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Pertanika is an international peer-reviewed journal devoted to the publication of original papers, and it serves as a forum for practical approaches to improving quality in issues pertaining to tropical agriculture and its related fields. *Pertanika* began publication in 1978 as the Journal of Tropical Agricultural Science. In 1992, a decision was made to streamline *Pertanika* into three journals to meet the need for specialised journals in areas of study aligned with the interdisciplinary strengths of the university. The revamped Journal of Science & Technology (JST) aims to develop as a pioneer journal focusing on research in science and engineering, and its related fields. Other *Pertanika* series include Journal of Tropical Agricultural Science (JTAS); and Journal of Social Sciences and Humanities (JSSH).

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Pertanika is the official journal of Universiti Putra Malaysia. The abbreviation for Pertanika Journal of Science & Technology is *Pertanika J. Sci. Technol.*

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EDITORIAL OFFICE
JOURNAL DIVISION
Office of the Deputy Vice Chancellor (R&I)
1st Floor, IDEA Tower II
UPM-MTDC Technology Centre
Universiti Putra Malaysia
43400 Serdang, Selangor Malaysia.
Gen Enq.: +603 8947 1622 | 1619 | 1616
E-mail: executive_editor.pertanika@upm.my
URL: www.journals-jd.upm.edu.my

PUBLISHER
Kamariah Mohd Saidin
UPM Press
Universiti Putra Malaysia
43400 UPM, Serdang, Selangor, Malaysia.
Tel: +603 8946 8855, 8946 8854
Fax: +603 8941 6172
E-mail: penerbit@putra.upm.edu.my
URL: http://penerbit.upm.edu.my

ABSTRACTING/INDEXING
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Selected Articles From the International Conference on Agricultural and Food Engineering for Life 2012 (CAFEi 2012)

Foreword

Welcome to the **Second Issue 2015** of the Journal of Science and Technology (JST)!

JST is an open-access journal for studies in science and technology published by Universiti Putra Malaysia Press. It is independently owned and managed by the university and is run on a non-profit basis for the benefit of the world-wide science community.

In this issue, **15 articles** are published, **two** are review articles, **one** is short communication and **six** are regular articles. **Six articles** are from the International Conference on Agricultural and Food Engineering for Life 2012 (CAFEi 2012). The authors of these articles vary in country of origin, coming from **Malaysia**, **India**, **Ethiopia**, **Bangladesh** and **Japan**.

The first review article in this issue discusses on Activin A: Its role and involvement in inflammatory diseases (*Tie Tung Hing, Rusliza Basir, Chuah Yaw Kuang, Herni Talib* and *Norshariza Nordin*). Second review article discribe in details on the ballistic impact performance of the layered and laminated composites (*Kannan Rassiah, M.M.H Megat Ahmad* and *Aidy Ali*) and the short communication paper in this issue reports on the dose optimisation of ^{18}F-Flurodeoxyglucose for whole body PET Oncology examination in CDNI of UPM (*Hishar, H., Salasiah, M., Fathinul Fikri, A. S.* and *Nordin, A. J.*).

The first regular article in this issue is on the incinerated domestic waste sludge powder as sustainable replacement material for concrete (*Kartini, K., Dahlia Lema, A.M., Dyg. Siti Quraisyah, A.A., Anthony, A.D., Nuraini, T.* and *Siti Rahimah, R.*). The following article look at: The mapping of spatial patterns of violent crime in peninsular Malaysia: Normal mixture model approach (*Syerrina Zakaria* and *Nuzlinda Abdul Rahman*); evaluation of performance measures of two-lane highways under heterogeneous traffic (*Pritam Saha, Ashoke Kumar Sarkar* and *Manish Pal*); analysis of absorber and buffer layer band gap grading on CIGS thin film solar cell performance using SCAPS (*Nima Khoshsirat, Nurul Amziah Md Yunus, Mohd Nizar Hamidon, Suhaidi Shafie* and *Nowshad Amin*); a comparative study on standard, modified and chaotic firefly algorithms (*Hong Choon Ong, Surafel Luleseged Tilahun* and *Suey Shya Tang*); and pathway of continuous professional development among physiotherapists: a qualitative study (*Ayiesah Ramli* and *Marzzatul Farhana Maslan*).

I conclude this issue with six articles arising from the CAFEi 2012 international conference: total phenolic content and antioxidant activity of *Quercus infectoria* galls using supercritical CO_2 extraction technique and its comparison with Soxhlet extraction (*Hasmida, M. N., Liza, M. S., Nur Syukriah, A. R., Harisun, Y., Mohd Azizi, C. Y.* and *Fadzilah Adibah, A. M.*); drying performances and milling quality of rice during industrial fluidized bed drying of paddy in Malaysia (*Ibrahim, M. N., Sarker, M. S. H., Ab. Aziz N.* and *Mohd Salleh, P.*); effects of cooking temperature in repetitive cooking-chilling cycles on

resistant starch content and quality characteristics (*Nor, M. Z. M., Talib, R. A., Noranizan, M. A., Chin, N. L.* and *Hashim, K.*); effects of milling methods on tensile properties of polypropylene / oil palm mesocarp fibre biocomposite of fish crackers (*Nordin, N. I. A. A., Ariffin, H., Hassan, M. A., Ibrahim, N. A., Shirai, Y.* and *Andou, Y.*); the effects of airflow on oven temperatures and cakes qualities (*Shahapuzi, N. S., Taip, F. S., Ab. Aziz, N.* and *Ahmedov, A.*); and quantification of gallic acid and tannic acid from *Quercus infectoria* (manjakani) and their effects on antioxidant and antibacterial activities (*Ab. Rahman, N. S., Md. Salleh L., Abd. Majid F. A.* and *Harisun, Y.*).

I anticipate that you will find the evidence presented in this issue to be intriguing, thought-provoking, and, hopefully, useful in setting up new milestones. Please recommend the journal to your colleagues and students to make this endeavour meaningful.

I would also like to express my gratitude to all the contributors, namely, the authors, reviewers and editors for their professional contribution towards making this issue feasible. Last but not least, the editorial assistance of the journal division staff is fully appreciated.

JST is currently accepting manuscripts for upcoming issues based on original qualitative or quantitative research that opens new areas of inquiry and investigation.

Chief Executive Editor
Nayan Deep S. KANWAL, FRSA, ABIM, AMIS, Ph.D.
nayan@upm.my

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Review Article

Activin A: Its Role and Involvement in Inflammatory Diseases

Tie Tung Hing[1], Rusliza Basir[1*], Chuah Yaw Kuang[1], Herni Talib[2] and Norshariza Nordin[3]

[1]Department of Human Anatomy, Faculty of Medicine and Health Sciences, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

[2]Department of Pathology, Faculty of Medicine and Health Sciences, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

[3]Department of Obstetric and Gynaecology, Faculty of Medicine and Health Sciences, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

ABSTRACT

Activin proteins are members of the transforming growth factor-β family. Activin A is involved in several biological responses including wound repair, cell death, proliferation and differentiation of many cell types. Biologically active activins consist of homodimers or heterodimers of two beta (β) subunits that are linked together by a single covalent disulphide bond. The subunits in humans are βA, βB, βC and βE. As an example, a combination of two βA subunits will produce a unit of activin A. These proteins are found in most cells of body such as macrophage and activated circulating monocytes. Their role in inflammation can be categorised into two types, either pro- or anti-inflammatory agents, depending on the cell type and phase. Activin signals are kept in balance by antagonist follistatin (Fst), which is a glycoprotein expressed in tissues and encoded by the follistatin gene in humans.

Keywords: Activin, transforming growth factor, activin subunit, inflammation, follistatin.

INTRODUCTION

Activin A is a multifunctional growth factor that is a functionally and structurally different member of the transforming growth factor-beta (TGF-β) superfamily of proteins (Chang, Brown & Matzuk, 2002; Griselda, Anayansi, & Iván, 2012). Precursor proteins are the forms of activin produced after cleavage from bioactive ligands. They were given the name activin after the first identification as

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E-mail addresses:
carlzman.tie@gmail.com (Tie Tung Hing),
rusliza@upm.edu.my (Rusliza Basir),
acsonchuah@gmail.com (Chuah Yaw Kuang),
herni@upm.edu.my (Herni Talib),
shariza@upm.edu.my (Norshariza Nordin)
*Corresponding Author

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activators of follicle-stimulating hormone (FSH) secreted from the pituitary gland (Ling *et al*., 1986). These proteins also play an important role in regulating various biological functions in a many types of tissues and cells which include apoptosis, wound repair, differentiation of cells, survival of neural cell and inflammation (Yndestad *et al*., 2004).

Activins are involved in inducing mesodermal tissue differentiation in embryonic *Xenopus* explants (Griselda *et al*., 2012). Given their ability to accumulate and stimulate haemoglobin differentiation in erythroleukemia cell lines in vitro, these proteins were discovered through isolation from a monocytic leukaemic cell line derived from human bone marrow. The very first name given to activins was erythroid differentiating factor (EDF). Description of activin and its ligand as survival factors for nerve cells was the result of a search for nerve growth factors. Activin A was able to support the survival of the central nervous system (CNS) neurons in culture, as well as to protect neuronal injury from neurotoxicity due to its role as a neurotrophic and neuroprotective factor (Woodruff, 2000). Moreover, activin A affects liver homeostasis and may stimulate hepatocyte cell death (Woodruff, 1998). It is produced and secreted quickly into the bloodstream from the endothelium of the vascular system and other tissues during inflammation. Mediators such as nitric oxide and pro-inflammatory cytokines, as well as leucocyte activation, play a crucial role in numerous aspects of the inflammatory response. As seen in patients with either acute or chronic inflammation (e.g., septicaemia and inflammatory bowel disease), the concentrations of activin A were observed to have increased in the serum and tissues (Hubner, Brauchle, Gregor & Werner, 1997; Michel, Ebert, Phillips & Nau, 2003a).

Bioactive activins comprise either homodimers or heterodimers of two beta (β) subunits, with four distinct subunits of activin identified to date as being involved in the development of these proteins. These subunits in humans are βA, βB, βC and βE, which are able to produce activin A (βA/βA), activin B (βB/βB) and activin AB (βA/βB). Activin A homodimer is produced by merging two subunits of βA. This homodimer is produced by the creation of either an intermolecular disulphide bond or a covalently dimerised bond between the 6th (of the nine) conserved cysteines in the established proteins. The entire nine cysteines, excluding the 6th, are implicated in the production of a cysteine knot by means of disulphide bonds within the molecule. The disulphide bonds are typical for members of the TGF-α family and are fundamental for their effects on living tissues (Griselda *et al*., 2012; Kreidl, Oztürk, Metzner, Berger & Grusch, 2009).

SOURCE OF ACTIVIN A

Activin A is synthesised and secreted in almost every cell type and tissue. For the action of an autocrine or paracrine mechanism to be carried out, the tissues must be equipped with many receptors of activin A. Tissue macrophages and activated circulating monocytes can synthesise and release activin A after stimulation by inflammatory stimuli. A large amount of activin A is secreted by bone marrow stromal cells in response to inflammatory mediators (Phillips, Jones, Scheerlinck, Hedger & de Kretser, 2001; Welt, Sidis, Keutmann & Schneyer, 2002). Activin A is released from bone marrow stromal cells after stimulation of the vascular endothelium by monocytes and bone marrow stromal fibroblasts once T-cells have interacted with cytokine.

Therefore, Bone marrow is another effective source of activin A and also a source of activin A during inflammation (Wu, Chen, Winnall, Phillips & Hedger, 2013). Additionally, strong stimulation by lipopolysaccharide (LPS), interleukin (IL)-1β or IL-6 also leads to the production of activin A by macrophages, monocytes and endothelial cells. In systemic inflammation, endothelial cells may be an essential depot of activin A activity (Phillips, Jones, Clarke, Scheerlinck & de Kretser, 2005; Phillips *et al*., 2001). Furthermore, secretion of activin A can be enhanced by mutual action between monocytes and activated T-cells via CD-40 (Abe, Shintani, Eto, Harada, Kosaka & Matsumoto, 2002). Moreover, mast cells and neutrophils are other potential sources of activin A during inflammation, while activin A can be produced after treatment with interferon (IFN)-γ and CD40L, or LPS when dendritic cells interact with activated T-cells (Phillips *et al*., 2005; de Kretser, O'Hehir, Hardy & Hedger, 2011).

ACTIVIN A SUBUNIT

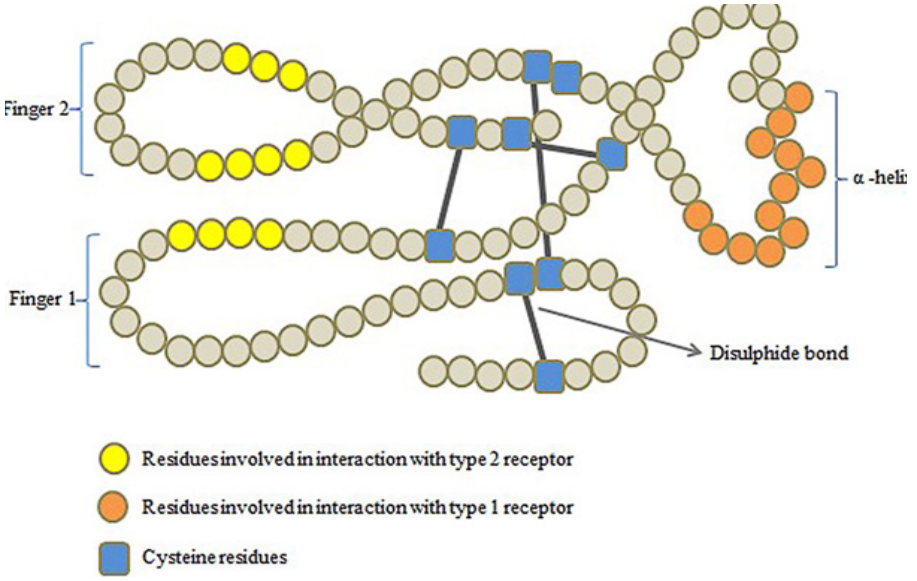

Fig.1: A subunit of activin A (Adapted from *Activin-A Binds Follistatin and Type II Receptors through Overlapping Binding Sites: Generation of Mutants with Isolated Binding Activities* by Harrison, C. A. & Chan, K. L., 2008, *Endocrinology, 147*(6), 2744-2753).

The binding sites for type II receptor are located at finger 1 (Phe^{17}, Ile^{30}, AL^{31}, Pro^{32}, His^{36}) and finger 2 (Arg^{87}, Pro^{88}, Ser^{90}, Leu^{92}, Tyr^{94}, Ile^{100}, Lys^{102}, Glu^{111}) of activin A. Follisatin can bind to contiguous site on activin A. The first is the convex outer side of activin β strand (Ile^{30}, AL^{31}, Pro^{32}, Leu^{92}, Tyr^{9}, Ile^{100} and Lys^{102}) and in β strand fingertip (Asp^{32} – Asn^{99}). The second is the binding site of type I receptor that is formed by the concave activin β strand of one subunit (Trp^{25}, Trp^{28}, Met^{91}, Tyr^{93} and Ile^{105}) and helical wrist region on the other β subunit (His^{47}, Ile^{48}, Gly^{50}, Thr^{51}, Ser^{52}, $Phe^{58,}$ Thr^{61} and His^{65}) (Harrison, Chan & Robertson, 2006).

ACTIVIN A SIGNALING PATHWAY

Since activins are members of the TGF-β family of signaling molecules, mature activins are believed to signal through single-pass transmembrane serine-threonine kinase receptors type I and type II, both of which are crucial for activin-mediated biological activities. The cytoplasmic region of both type I and type II receptors is the region where the serine-threonine activity takes place, which also serves as the docking stations. The signalling pathway started by the attachment of activin A to dimers of the activin type-II receptors ActR-II, which are also known as ACVR2 or ActR-IIB (ACVR2B).

Consequently, dimers of the activin type I receptor (activin receptor-like kinase [ALK] 4) are recruited and phosphorylated in their type-II serine-threonine kinase domain. The primary residues within a glycine- and serine-rich (GS) domain of the type I receptor are phosphorylated by the assembled active type II receptor, as a consequence of the formation of the heteromeric ligand-receptor complex, which brings together the cytoplasmic region of the two types of receptors. The activation of intracellular protein Smads is the result of the activation of activin type I receptors. It was found that the mutually dependent characteristic of activin type I and type II receptors is due to the need of activin type I receptors to bind their ligand to activin type II receptors, whilst the association of type II receptors to type I receptors is important to signal the nucleus (Bilezikjian & Vale, 2011). Although the binding of type II receptors to ligands can take place without the presence of type I receptors, they still need the latter to signal and function well.

Receptors of activin are regularly internalized immediately after the binding of ligand. Immediately after receptor activation, phosphorylation of receptor-regulated Smad (R-Smad) proteins by the activin type I receptors takes place after their recruitment to the receptor complex. Molecular complexes are formed after the common mediator Smad 4 approaches the recruited and phosphorylated R-Smads (Smads 2 & 3). Modulation of gene expression is initiated by direct binding to DNA or association with other transcription factors by the molecular complexes that come together with cofactors. The phosphatidylinositol-3′-phosphate-binding protein, known as Smad anchor for activation of the receptor (SARA), is responsible for phosphorylation of Smads 2 and 3. Smads 2 and 3 are bound together to the receptor complex with the aid of SARA. R-Smads and SARA are cleaved from the receptor complex once R-Smad proteins are phosphorylated. Gene expression is initiated following the translocation of phosphorylated R-Smads after the recruitment of the cytoplasmic common mediator Smad, the co-Smad 4 (Schmierer, Schuster, Shkumatava & Kuchler, 2003; Kreidl *et al*., 2009; Griselda *et al*., 2012; Cárcamo *et al*., 1994; Wieser, Attisano, Wrana & Massagué, 1993).

The Smad proteins are generally divided into two subcategories: R-Smads (receptor-activated) or co-Smad (not receptor-activated) (Bilezikjian & Vale, 2011). Smad proteins have an N-terminal against decapentaplegic homology domain 1 (MH1) and a C-terminal against decapentaplegic homology 2 (MH2) linked by a proline-rich region. Binding of DNA to the CAGA sequence, as well as to some GC-rich sequences, is mediated by the MH1 domain, which contains a nuclear localisation signal. Transcription activation only happens when the MH2 domain is fused to the Gal4 DNA-binding domain. For MH2-specific association with the L45 loop of activin type I receptors, as well as in the homo- or hetero-oligomerisation of Smads,

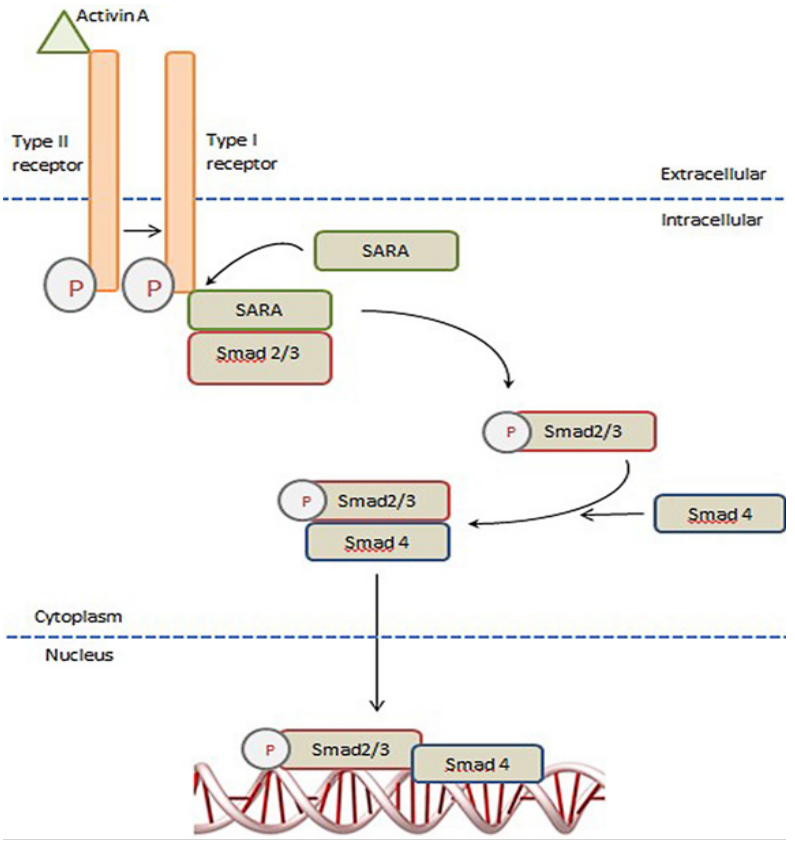

Fig.2: Activin/ Smad signalling pathway

the presence of L3 loop containing in MH2 is important (Bilezikjian & Vale, 2011; Ten Dijke, Miyazono & Heldin, 2000). The conserved C-terminal SSXS motif in R-Smads is recognized by the tail of activin type I receptors in the cytoplasmic region. The association between Smad 4 and R-Smad is initiated after the cleavage of R-Smad from activin type I receptors due to the phosphorylation of the C-terminal SSXS. Consequently, this initiates translocation of the heteromeric Smad complex to the nucleus (Attisano & Wrana, 2000; Bilezikjian & Vale, 2011).

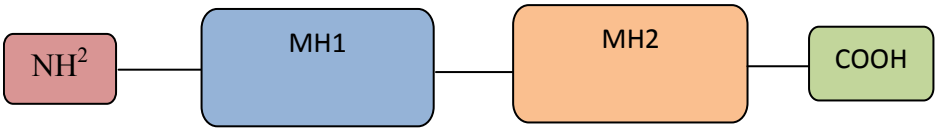

Fig.3: A schematic diagram of the Smad protein with an N-terminal Mad Homology 1 (MH1) and a C-terminal Mad Homology 2 (MH2) domain linked by a proline-rich region.

Smad 3 recognises the two inverted repeats of GTCT on DNA. However, the single copy of this core sequence only bound by the MH1 of Smad 3. The MH1 of Smad 2 cannot bind to DNA by itself. In addition, the repeat sequence of GNCN in the promoter are not sufficient for binding to Smad-dependent targeting of specific genes, but it is enough for Smad-DNA binding.

The most comprehensively characterised DNA-binding partners of the Smad proteins are the forkhead activin signal transducer (FAST) family of DNA-binding proteins. Furthermore, the family has been identified in humans and mice as well. The TGF-β activin-responsive region of promoters acts mutually with FAST in order to activate gene transcription downstream of activin signaling (Attisano & Wrana, 2000).

ACTIVIN AS A MEDIATOR IN INFLAMMATION: ACUTE AND CHRONIC

At the site of injury, inflammation quickly develops as the consequence of the secretion of inflammatory cytokines (IL-1, IL-6 and tumour necrosis factor-alpha (TNF-α)] from cells such as macrophages and stromal cells. In succession, acute phase response (APR) is stimulated after these cytokines act at systemic sites such as the liver to trigger febrile responses and gene expression (de Kretser, Hedger & Phillips, 1999). Sites of the injury are protected by innate response by APR to prevent host from continuous tissue damage. Some physical changes are protected by innate response, which in turn, suppresses actions of serum acute phase proteins and cytokines (Steel & Whitehead, 1994). Damage of lung during LPS-induced inflammation occurs after neutrophils translocate from bone marrow. Activin A is secreted during inflammation by neutrophils following direct stimulation by TNF-α; serum TNF-α is reduced if activin A is blocked. After 4 and 5 hours of LPS introduction, serum activin A level is greatly elevated (Wu *et al*., 2013; de Kretser *et al*., 1999). The activin A released during acute inflammation is very early and biphasic. Its release during early acute inflammation is rapid and therefore from pre-stored materials. Its secretion during the later phases of inflammatory responses is from newly synthesised inactive material hours after stimulation such as by LPS. The cells that are responsible for the production of activin A at the later stage are monocytes, macrophages, dendritic cells and endothelial cells. Neutrophils and epithelial cells are responsible for activin A secretion such as those from the stomach and lungs (Philips *et al*., 2009).

Upregulation of activin A expression at the sites of injury is either as protein or mRNA in synovial fluid or in chronic inflammatory bowel disease (IBD) in patients with inflammatory arthropathy (de Kretser *et al*., 1999). In chronic inflammation, comparing patients with gout and rheumatoid arthritis with those with non-inflammatory osteoarthritis, activin A levels were elevated in synovial fluid, as well as expression of activin βA in synovial membranes. Activin A has been implicated in IBD. The ulcerative colitis or Crohn's disease from IBD patients, inflamed mucosal and sub-mucosal tissues in the intestinal wall were observed, together with high IL-1β and activin βA subunit mRNA levels. In normal tissues, expression of IL-1β was low (Philips *et al*., 2009). This same group of patients also displayed very high level of plasma activin A level and βA mRNA in the intestinal wall. Moreover, in areas where IL-1β mRNA was highly expressed, activin βA subunit mRNA was found in the mucosa and sub-mucosa of tissues at sites of inflammation, whereas low levels of expression were found in healthy tissues; for example, the activin A-stimulated production of IL-6 and IL-8 in amniotic membranes. The action of activin A is believed to have two phases; inhibitory as the activin A concentration is high and vice versa (Wu *et al*., 2013).

ACTIVIN A: A PRO-INFLAMMATORY AGENT

A pro-inflammatory role for activin A has been observed in diverse cell types (Jones, de Kretser, Patella, & Phillips, 2004). The role of activin A as a pro-inflammatory mediator is proved by the upregulation of activin expression by pro-inflammatory cytokines (granulocyte monocyte colony-stimulating factor (GM-CSF) and interferon γ) and/or LPS, as well as down regulation by glucocorticoids. The role of activin A during pro-inflammatory activities was confirmed by its pro-migratory on monocytes. Additionally, metalloproteinase expression can be induced by activin A in macrophages during the inflammatory response. Migration and infiltration by macrophages through the basement membrane during inflammation are also triggered by activin A. Taking malarial infection as an example, it is important that cells are first activated by parasite-specific molecules to initiate immune responses against the infection. This is then followed by the activation of *mitogen-activated protein kinase* (MAPK) and nuclear factor (NFκB) signalling pathways, resulting in expression of cytokine genes (El-Gendi *et al*., 2010; Zhu, Krishnegowda, Li & Gowda, 2010).

In malarial infection, macrophages phagocytose the invading parasites. During this process, the release of pro-inflammatory cytokines such as TNF-α, IL-6 and IL-1β can be stimulated by activin A (Rapeah, Dhaniah, Nurul & Norazmi, 2010; Phillips *et al*., 2005). In bone marrow-derived macrophages, TNF-α and IL-1β are also produced following the stimulation of activin A. Moreover, activin A increases the expression of pro-inflammatory mediators such as prostanoids, prostaglandin and thromboxane, cyclo-oxygenase 2 (COX-2), and inducible nitric oxide synthase (iNOS) (Jones *et al*., 2004; Nusing & Barsig, 1999). The pro-inflammatory effects of activin A can also be seen in LPS-induced inflammation process in sheep. This involves the pro-inflammatory cytokines TNF-α and IL-6, which are secreted following the release of activin A. Activin A has also been implicated in stimulating the production of IL-6 and IL-8 in amniotic membranes, where its role as a pro- or anti-inflammatory agent depends on its concentration. When the activin concentration is low, it acts as a pro-inflammatory factor, and when it is high, an anti-inflammatory agent (Jones *et al*., 2004). Additionally, IκB degradation, nuclear translocation of NFκB and phosphorylation of Extracellular signal-regulated kinases 1/2 (ERK 1/2) and p38 MAPK lead to the production of TNF-α, IL-1β and IL-6 by activin A (Phillips, de Kretser & Hedger, 2009).

ACTIVIN A: AN ANTI-INFLAMMATORY AGENT

Activin A has been well documented an anti-inflammatory agent, with evidence that activin A can limit the secretion and actions of IL-1β and IL-6 (Phillips *et al*., 2001). A study on monocytic cultures uncovered the ability of activin A to keep down the production of IL-1β by inhibiting the alteration of the IL-1β precursor into its secreted biologically active form, most likely by blocking caspase-1 (Philips *et al*., 2009; Phillips *et al*., 2011). In LPS-stimulated macrophages, activin A can suppress the secretion and expression of IL-1β and IL-10 (de Kretser *et al*., 2011; Wang *et al*., 2008). During the early stage of inflammation induced by macrophage, the pro-inflammatory effect of activin A is turned into anti-inflammatory at the later stage (de Kretser *et al*., 2011). During APRs, activin A is able to initiate anti-inflammatory effects at local site of infection or injury and to repress unnecessary inflammation at the pheripheral sites such as

liver (Smith *et al*., 2004). Moreover, theactivity of activin A in a hepatoma cell line (HepG2) can be inhibited by a secretion of acute phase inflammatory proteins mediated by IL-6 (Werner & Alzheimer, 2006). With the aid of activin A, the pancreatic islet cells and endothelial cells of humans are able to produce insulin and anti-inflammatory effects, as well as anti-oxidative and anti-inflammatory effects. In patients with coronary artery disease (CAD), anti-inflammatory effects on previously activated peripheral blood mononuclear cells are also displayed by activin A (Andersen *et al*., 2011). The anti-inflammatory effects of activin A are also proven, with antagonistic action on the secretion of IL-6 and IL-11 in myeloblasts (Sugama, Takenouchi, Kitani, Fujita & Hashimoto, 2007). During the early phase of cardiopulmonary bypass (CPB), the anti-inflammatory effects of activin A only occur after the production of pro-inflammatory cytokines such as TNF-α and IL-8 (Sablotzki *et al*., 1997).

ACTIVIN IN ORGAN SPECIFIC FUNCTIONS

In the brain, differentiation of cerebrocorticol neural progenitor cells (NPC) into neuronal phenotype is driven by activin A. In proliferating NPC, activin can induce discrete but significant rises in the proportion of neurons. The neurogenesis by activin is not due to its role as regulator but as an instructor on NPC. In addition, inhibition of activin can cause reduced neurogenesis and development of anxiety-related behavior (Rodríguez-Martínez, Molina-Hernández & Velasco, 2012).

Activin A is upregulated in heart failure but treatment with anti-activin A antibody can restore growth hormone (GH) which improves heart function by normalizing GH level (Fukushima *et al*., 2011). Activin A has also been identified to play a role in healing after myocardial infarction, but a continuous increase in activin A levels can lead to myocardial remodelling and eventually heart failure (Yndestad *et al*., 2004). Recovery could be due to the presence of high serum activin levels that cause a reduction by upregulating expression of B-cell lymphoma 2 (Bcl-2) protein (Oshima *et al*., 2009).

Activin A has been proven to function as a negative regulator for liver growth. It can inhibit mitogen-induced DNA synthesis and induce apoptosis in cell lines of hepatoma and in hepatocytes *in vivo* as well as *in vitro*. Overproduction of activin A due to inhibin deficiency can cause hepatocyte destruction. Activin A remained at low levels at the first 12 hours of partial hepatectomy (PHX) and increased by three times higher at 168 hours, which suggests that it acts as regulator of liver regeneration. Furthermore, hepatocyte replication increased following the elevation of follistatin at 24-48 hours of post PHX (Rodgarkia-Dara *et al*., 2006).

In kidney, activin A is important as a negative regulator in the growth of ureter bud (UB) from Wolffian duct (WD) during the development of metanephric kidney. UB outgrowth is in response to glial-cell-derived neurotrophic growth factor (GDNF). Thus, ectopic bud formation can be controlled by inhibiting GDNF by activin A. Activin A is also involves in branching morphogenesis of UB by acting as negative regulator (Maeshima *et al*., 2004). Activin A in pancreas is crucial for glucose metabolism by stimulating differentiation of β cells to produce insulin, which allows insulin target cells to act efficiently in glucose uptake (Hashimoto & Funaba, 2011; Ueland *et al*., 2012).

ROLE OF ACTIVIN IN DISEASES

During malaria parasite infection, pro-inflammatory cytokines like TNF-α and IFN-γ, and anti-inflammatory cytokine like IL-10 are produced to provide protection against the parasites. In malarial infection, activin A initiates the release of pro-inflammatory cytokines such as TNF-α, and IFN-γ and IL-10 production is activated upon enhancement of inflammation. These cytokines are important in protection against malaria parasites (Semitekolou *et al*., 2009; Phillips *et al*., 2005; Gribi, Tanaka, Harper-Summers & Yu, 2005; Robinson *et al*., 2009).

Activin A has been identified to be involved in the pathogenesis of inflammation bowel disease (IBD), enhancing the migration instead of proliferation of intestinal epithelial cells, as well as stimulating inflammation during colitis (Huber *et al*., 1997; Zhang, Resta, Jung, Barrett & Sarvetnick, 2009). In patients with ulcerative colitis or Crohn's disease, strong expression of the activin βA subunit was detected, but not in people with a healthy digestive tract. Moreover, the higher the expression of activin βA mRNA was, the more severe the degree of inflammation would be. Increased expression of activin A mRNA has been identified in highly inflamed tissues such as the mucosa and sub-mucosa of injured intestinal epithelium (Hubner *et al*., 1997). Furthermore, increased expression of type I and type II activin receptors has also been reported in patients with IBD but not in healthy people, indicating that activin signalling contributes to the enhanced receptor expression (Zhang *et al*., 2009).

In patients with asthma, increased concentration of activin A in bronchoalveolar lavage fluid is due to its secretion from cells lining the alveoli, which are epithelial cells and activated human lung mast cells (Philips *et al*., 2009; Werner & Alzheimer, 2006). Alveolar cells, endothelial cells and fibroblasts were found to respond to activin in inflamed lung tissues, which was supported by the strong ActR-IB expression in these cells. Mice deficient in mast cells shows lower secretion of activin A; therefore, mast cells are an important source of activin in the airway of mice with asthma. It is believed that mast cell-derived activin A could promote airway tissue remodelling, as activin A was found to enhance the proliferation of airway smooth muscle (ASM) cells in humans. The action is completed with paracrine signalling to enhance the proliferation of ASM cells. Activin increases the differentiation and migration of mast cell progenitors but inhibits growth. For that reason, activin A may regulate mast cells as the effector cell of the immune system, which could further contribute to the pathogenesis of asthma (Werner & Alzheimer, 2006).

CURRENT RESEARCH FINDINGS AND NEW TREND FOR ACTIVIN RESEARCH

Activin has been identified to be involved in the control of biological systems and wide interaction with other members of TGF-β superfamily, as well as other hormones/peptides. The motivation underlying the study by Makanji *et al*. (2011) is the apparent role of activin in promoting cachexia in patients with a variety of tumours and promoting development of gonadal tumours. However, the value of this study is much broader.

The non-specific interaction of activin and other TGF-β superfamily, together with regulation and signalling of other family members, makes the identification of specific action and the development of specific agonists and antagonists very challenging. In this study, genetic

manipulations (transgenic animals or in vitro small interfering RNA gene knockdown) are useful only with the aid of specific agonistic and antagonistic compounds for the peptide of interest. The system's biology framework was used to create specific antagonist (activin propetide [AT propetide]) that inhibits activin A stimulation of FSH release. Specific domains for protein folding were determined by study on the molecular two- and three-dimensional structures of activin and TGF-β1 peptide so that the structure that binds and inhibits mature TGF-β1 can be created. The inhibition of activin action by chimeric peptide was achieved through the linking of C-terminal portion of activin with the N-terminal portion of TGF-β1. The specificity of AT propeptide was important in genetic manipulation. The AT propeptide allows insight into functions of activin and mechanisms of interaction with other TGF-β1 members such as inhibin. New therapies for cancer and other diseases could be developed from this chimeric propeptide. Hence, this study is valuable and may be rewarding upon the proper application of the 21st century systems biology thinking (Nielsen & Torday, 2011).

FOLLISTATIN: AN ANTAGONIST OF ACTIVIN A

The single-chain glycoprotein and structurally different follistatin was recognised as an extracellular antagonist of activin that could reduce its biological activities. The LPS- or IL-1β dosed sheep has proven the involvement of follistatin during inflammation. Follistatin is a monomeric protein and is structurally different to the TGF-β superfamily of proteins. Most organs that express follistatin also express activin; consequencely, follistatin activities in nature likely to have an autocrine or paracrine characteristic. The bond between follistatin and activin is very strong, with Kd 50-680 pmol/L. In addition, the local diffusion of activin A from the site of action can be controlled by follistatin (Deli *et al*., 2008; Welt *et al*., 2002; Oshima *et al*., 2009). The bond between follistatin and activin is virtually irreversible; however, activin must be cleaved by proteolytic mechanism to reveal its endocrine signalling effects. Such a mechanism has been identified for the related bone marrow macrophage protein (BMP), in which bioactive BMP is released from chordin, its binding protein, by metalloproteinase (Welt *et al*., 2002).

A third of activin residues are buried at the binding sites of receptor as soon as one activin dimer binds to two follistatin molecules. Three distinct types of follistatins are produced through the protein processing and splicing of a single follistatin gene. They are proteins with 288, 303 and 315 amino acids. Three homologous domains are contained in each of these three follistatins. In case of inhibition of activin, only two domains are involved in the binding process (Deli *et al*., 2008). The inhibitory action occurs only when the receptor binding site of activin A is obstructed by complex formation with follistatin (Kreidl *et al*., 2009). Follistatin has an important role in suppressing activin A, as it can reduce mortality rate by reducing the excessive production of activin A during inflammation (Wu *et al*., 2013; Jones *et al*., 2004). For example, over scarring as well as formation of granule and re-epithelisation on wounded skin is under the influence of a high concentration of activin A but healing quality can be restored with the aid of follistatin (Werner & Alzheimer, 2006). Furthermore, liver apoptosis induced by activin A can be inhibited with follstatin as well (Oshima *et al*., 2009).

CONCLUSION

Activin A has been linked to inflammation, cells growth, tissue repair, metabolism and apoptosis. The focus of this review is on its role in inflammatory response. Its expression is significantly upregulated during acute and chronic inflammation. Activin can be involved in both pro- and anti-inflammatory responses depending on the cell type and stage of its expression. Inflammation has always been a major biological response related to activin, such as that in malaria infection, IBD and asthma. Besides, activin has different functions in organs, while in brain, it has a role in neurogenesis. It also improves heart failure and healing after myocardial infarction and acts as a regulator of liver regeneration after PHX in the liver, growth of UB from WD and branching of UB, and glucose metabolism in pancreas. Nonetheless, further studies are required to identify and understand the roles of activin in other pathological conditions.

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Review Article

Ballistic Impact Performance of the Layered and Laminated Composites: A Reviews

Kannan Rassiah[1, 2*], M.M.H Megat Ahmad[1] and Aidy Ali[1]

[1]*Department of Mechanical Engineering, Faculty of Engineering, Universiti Pertahanan Nasional Malaysia, Kem Sg. Besi, Kuala Lumpur, Malaysia*

[2]*Department of Mechanical Engineering, Politeknik Merlimau Melaka, KB 1031, Pejabat Pos Merlimau, Melaka, Malaysia*

ABSTRACT

This paper presents an overview topic of layered and laminated fibre composites. The review presents an investigation on the effect of varying the properties of fibre and the matrix of layered and laminated composites and identifies the fundamental parameters determining ballistic impact protection. The advantages of layered and laminated reinforced composites with different thicknesses for further enhancing ballistic penetration resistance of the laminated fibre composite have been reviewed. Lamination of multiple layers of composite material can give better ballistic performance.

Keywords: Layer, laminate, ballistic impact, composites

INTRODUCTION

A variety of fibre composite materials are found to be increasingly used in different industries including concrete structural, automotive, aerospace and defence applications. The characteristics of composites, such as strength and stiffness, allow for the structure of safe automobiles, aircraft with high scope and light-machine components (Naik *et al*,. 2005; Castillo *et al*., 2012; Borvik *et al*., 2009). Today, this material is an increasingly important component used in many defence and commercial systems where it is a component of structural panels, control arrangements, configurations of insulated armour and in reinforcement parts.This is due to its mechanical and physical properties compared to the properties of the components taken separately (Kuan *et al*., 2009). Another

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E-mail addresses:
kannan@pmm.edu.my, kannan780915@gmail.com (Kannan Rassiah),
megat@upnm.edu.my(M.M.H. Megat Ahmad),
aidyali@upnm.edu.my (Aidy Ali)
*Corresponding Author

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reason for its high demand is due to its reduced structural weight that makes it suitable for use in lightweight materials (Grujicic *et al*., 2006). Here, the design of composite systems for enhanced material performance in engineering has created lightweight structures.

Moreover, composites used in reinforced plastics possess the benefits of fabrication advantage, crushing steadiness and high energy absorption recital. This makes the material suitable for use in defence application as the criteria for defence application call for materials that are durable, strong and ableto absorb impact energy (Kuan *et al*., 2009; Carrillo *et al*., 2012; Dimeski *et al*., 2011). High mechanical properties are extensively used for military purpose, for instance, in personnel armour, vehicles and arms, which are all important and necessary for the protection of military personnel. The selection of material used for defence protection, however, is affected by different criteria such as cost and weight.

Although the use of composites is gaining interest, it faces several challenges. The first is that laminate structural composites are formed not only by the class of matrix and reinforcement materials. Second, it is dissimilar from composition design of the reinforcement. Third, it depends on how to lay up conventional kevlar, fibreglass, nylon, ramie and aluminium in laminate structural and functional stability during storage and use. Several studies have shown that a composite of kevlar, fibreglass, natural rubber, ceramics or aluminium provides a method to improve ballistic impact and effect in different configurations (Carrillo *et al*., 2012;Naik *et al*., 2008;Kyziol, L, 2007; Huang *et al*., 2007 and Ali *et al*.,2010).

This paper reviews research work published in the field of ballistic impact layered and laminated composites with special reference to the laminate thickness ballistic impact and the properties of the composites. The single, combining two or more layers of material together and the process of producing a laminar by stacking a number of thin layers of fibres and matrix and consolidating them into desired thickness is considered.

LAYERED AND LAMINATED PLATE COMPOSITES

The laminate fibre reinforced composites appealing to the military, the navy and the construction sector (Nunes *et al*., 2004; Ramadhana *et al*., 2012; & Deka *et al*., 2006). The study considered the stacking of a number of layers with different forms of reinforcement fibres and matrix in the desired thickness. The layered and laminate investigation gives the benefit ballistic protection. Iqbal *et al*.(2011) investigated ballistic limit, which increased with an increase in the thickness of the monolithic and layered target plate. Besides that, according to Huang *et al*.(2007), depth of penetration was dependent on ceramic thickness. Carrillo *et al*. (2012) found that adding a thermoplastic layer matrix increased ballistic performance. Table 1 shows the experiment results of ballistic limit velocity for all specimens, which correlated well with layered application and thickness (Buitrago *et al*., 2010; Sabet *et al*., 2011; Iqbal *et al*., 2011).

DESIGN OF BALLISTIC IMPACT THROUGH THICKNESS IMPROVEMENT

During ballistic impact tests the resolution of ballistic limit is of leading consequence indevising protective structures, military vehicles and body armour. The influence of target thickness can be one of the factors in impact resistance application. The thickness and layeredness of the body play an essential role in energy absorption or delamination area of materials and by increasing

the thickness (Barcikowski, 2008); the body structure can endure more load and more energy absorption (Balakrishnan *et al*., 2012; Demir *et al*., 2008; & Satoto *et al*,. 2009). Three parts were observed in this study: i) single layer, ii) composite laminate, iii) hybrid laminate.

TABLE 1: Ballistic Impact Test

Fabric type	Laminate Thickness (mm)	Ballistic Limit (m/s)
Monolithic	3	212
Monolithic	6	332
Monolithic	12	550
5 layer Cross Play	5	95
6 layer Cross Play	6	98.4
10 layer Cross Play	10	140
2 Layer Plate	0.5	51.22
3 Layer Plate	0.71	64.52

Single Layer of Thickness

Balakrishnan etal. (2012) studied the effect of plasma-transferred arc hard-faced interlayer thickness on ballistic performance using three different thicknesses (4, 5.5 and 7 mm) hard-faced middle layer. The researchers found that all the three joints with three different thicknesses of hard-faced material successfully stopped the projectile and the bullets were shattered. Meanwhile, according to Sabouri *et al*. (2011), using different aluminium thickness in varies locations shows better ballistic resistance with the thickness of glass/epoxy layers remaining stable.

Sheikh *et al*. (2009) investigated the performance of single and multiple laminated panels with simple layered shell elements. The result shows that the particular energy absorption ability of the thin laminate with five layers is found to be more than that of the thick laminate of 10 layers; however, the double laminated panel with two five layers has enhanced energy absorption capacity than that of the thick laminate of 10 layers.

However, according Iqbal and Gupta (2011) the nose-shaped projectile affected the thickness of the plate whether in single or multiple layers. The result shows that the thin monolithic target plates as well as the layered connection of plates presented the lowest ballistic resistance.

A similar method conducted by Babu *et al*. (2007) used hard steel cylindro-conical projectiles for unidirectional glass-fibre reinforced epoxy composite plates. Table 2 shows that the changeable stacking sequence, thickness, layered laminated and symmetric laminated has been tested. The ballistic limit for thin targets plays a major role with the rising thickness of the laminates and with nose geometry of weighty projectile that has less control of the energy absorption.

TABLE 2 : Ballistic Limit of Laminates for Different Nose Geometry and Thickness

Projectile Nose Geometry	Ballistic Limit (m/Sec)				
	4 layers (0,90)	6 layers symmetric	6 layers symmetric	6 layers symmetric	6 layers symmetric
	2.3 mm	3.2 mm	4.9 mm	3.2 mm	4.9 mm
Sharp nose	30	67	67	45	46
Blunt nose	32	40	39	50	49
Truncated nose	36	46	45	58	58

Madhu and Bhat (2003) studied the standard and oblique impacts on single and layered mild steel and aluminium plates in the range of 10 mm to 40 mm. The proportion of plate thickness to the diameter of the projectile is in the range of 1.5 to 13.0 with of an ogive-shaped, hard steel projectile. The residual velocities for combined target in plates of intermediate thicknesses were of the same order as in the case of a monolithic target.

In addition, the thicker single or monolithic plate can improve ballistic impact limit and perform as the ballistic penetration resistance. The ballistic limit increased with an increase in the thickness of the monolithic target plate.

Laminated Composites

Demir *et al*. (2008) studied the laminated composite performance of the mechanical properties of the support plate and the areal density. The composite targets were subjected to the thickness of the alumina tiles as 6 mm and steel backing plates of five different thicknesses (4, 5.9, 7.8, 9.7 and 11.6) were used. The ballistic recital of the laminated alumina steel composites increased with respect to the areal density and hardness of the composite.

Hohler *et al*. (2001) investigated the recital of oblique, ceramic/metal, bilayer composite armour by two ceramics, alumina and silicon carbide and two metallic backings, rolled homogeneous armour steel and aluminium, in a variety of combinations. The results show that areal densities of the depth of penetration targets decreased with increasing ceramic thickness.

Deka *et al*. (2008) examined the performance of plain-weave laminated composites of changeable thicknesses under high velocity impact. When the layer content increased from 8, 12 and 16 the velocity was 209.02 ms^{-1}, 294.45 ms^{-1}, and 304.1 ms ^{1}, respectively. The finding shows that ballistic limit is linearly correlated to the thickness of the laminated composites where the impact velocity was decreased from the full penetration order to a partial penetration order in unity of V_{50}.

Besides that, according to Patel *et al*. (2004), the penetration occurrence of a cylindro-conical impactor on the Kevlar/ epoxy-laminated composites with thickness of 10, 15 and 20 layered cross ply laminate plates with total thickness of 6.35 mm, 9.525 mm and 12.7 mm correspondingly affect the velocity limit. Here, the results show that ballistic limit velocity is 106.95 m/s, 121.3 m/s and 136.68 m/s, respectively.

Ahmad *et al*. (2007) explored the ballistic impact recital of high strength, high modulus composite coated with natural rubber. The result of different coating techniques and natural

rubber modulus were studied. The result showed that ballistic limit of 2 neat layers and 2 coated layers composite was 241 m/s and energy absorption was 31.9 J with areal density 1076 g/m^2. While for the 4 neat layers, the ballistic limit 200 m/s and energy absorption was 22.0 J with areal density of 808 g/m^2, respectively. This shows that energy absorption at the ballistic limit by the combined fibre was higher that by the neat system. The result indicates that the combination of 2 neat and 2 natural rubber coated Twaron composite in the 4-layer composite absorbed more ballistic impact energy than all-neat.

Lee *et al*. (2003) reported on the ballistic penetration of six various configurations of 4 layers Kevlar and 8 ml of shear thickening fluid. The equal target weights and the test result showed that 8 ml shear thickening fluid impregnated in 4 layers of Kevlar had a higher impact velocity of 253 m/s and the lowest penetration depth. The addition of fluid with kevlar fibre enhances the ballistic penetration resistances.

This also shows that ballistic limit is linearly related to the thickness and layeredness of the laminated composites. Laminate of the target plate has considerable influence on the ballistic limit and thickness of the ballistic limit decreased with an increase in the number of layers.

Laminated Hybrid Composites

Hybrid composites composed of two or more different reinforcements in a single matrix dominate over ordinary composites. The impact specimens were fabricated using layers of two types as shown in Figure 1. Material parameters are grave for the ballistic recital of hybrid laminate systems.The researcher evaluated the function of poly methyl methacrylate on the impact response of composites consisting of poly methyl methacrylate and polycarbonate as well as on the polycarbonate - Glass - poly methyl methacrylate - polycarbonate laminates. The outcome was that increasing the thickness of PMMA improved the overall impact capability of these laminates (Hsieh *et al*. 2004).

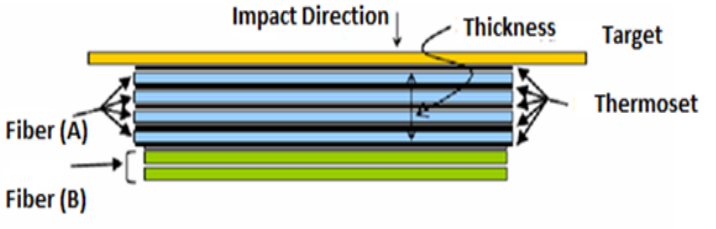

Fig.1: Laminated composites with several layers.

Kyziol (2007) studied the shooting resistance of ceramics, rubber, glass reinforced plastic, natural and modified wood. The outcome was that glass reinforced plastic samples of 16 mm thickness sheltered with ceramic plates of 10 mm thickness can be measured as bullet-proof against bullets of 7.62 mm calibre. The results obtained from the shooting tests of glass reinforced plastic laminate plates point out that the thickness of the ballistic shield plays a key role.

Satoto *et al*. (2009) studied the ballistic penetration show of a composite material collected of woven Kevlar fibre, woven nylon fibre, woven ramie fibre impregnated with compatible

resins and hybrid composite construction of these three structures. The researchers found that although the material was composed of 4 layers of Kevlar – A363F showed better performance than the 32-layer composites of nylon-Oxford X7 and 48 layers of ramie fibres, but the ballistic penetration resistance of Kevlar, nylon and ramie fibre is enhanced by impregnation of the fibre with the compatible resin. Impregnated resin fibre composite is shown to give better ballistic defence as contrasted with simple stacks of neat fibre.

In addition to the evolution of new materials that have a higher ballistic impact limit to the thickness and better ballistic penetration resistance, using hybrid materials can be improved through the design process to create materials that are sufficiently stiff, strong, flexible and lightweight. Similar results were obtained by Fink (2000) and Kaufmann *et al*. (2003) in their study of the performance of each layer where it was found that the material significantly influenced the overall performance of the armour. Enhancing performance could be achieved by cross configuration of the fibre layers without increasing panel weight and thickness. Based on that, various configuration analyses can be studied such as shown in Fig. 2, which considers four different hybrids composites (Synthetic fibre A / Synthetic fibre B / resin) in different thickness configuration.

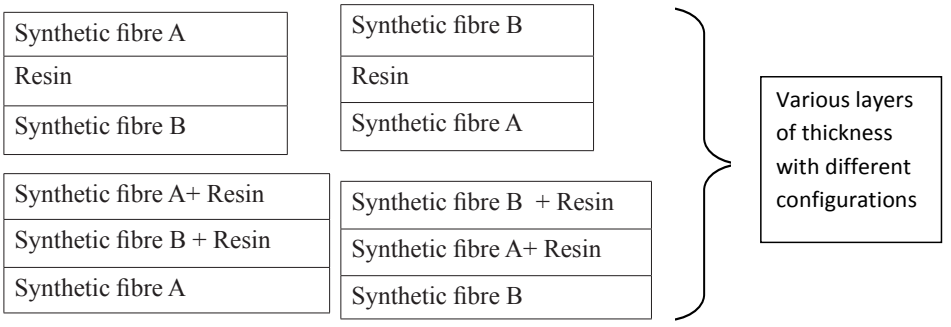

Fig.2: Different configurations.

FUTURE APPLICATIONS AND RESEARCH

The use of layered and laminated hybrid composites in ballistics will ensure optimisation of each subsystem module in composite personnel armour, vehicles and other systems subject to small panels. Using hybrid material design can provide additional important design variables such as thickness, size, materials, shape, distribution and orientation as well as cost (Rao *et al*., 2009; Ma *et al*., 2006). This can be identified by looking at the laminates of the hybrid system as they are superior to multiple with low resin content and can be improved ahead by hybrid systems using fibre layers on the impact side and respectively close fitting composites between the layers.

CONCLUSION

Ballistic impact engineering materials should be considered in selection of the most acceptable armour materials for use by the military. They provide good protection, superior ballistic penetration resistance and high flexibility. The most complicated part of this analysis was

increasing the number of fibre layers because of the comprehensive new energy dissipation mechanisms and increased interactions between the different plies of the composite and between the target and projectile. The literature shows that through experimentation, improved ballistic performance can be achieved through the thickness design process. In addition, they also provide better performance.

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Short Communication

Dose Optimisation of ^{18}F-Flurodeoxyglucose for Whole Body PET Oncology Examination in CDNI of UPM

Hishar, H.*, Salasiah, M., Fathinul Fikri, A. S. and Nordin, A. J.

Centre for Diagnostic Nuclear Imaging, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

ABSTRACT

A shift to administration of optimal dose of ^{18}F-FDG between 4 and 5 MBq/kg from the current practice of higher doses potentially yields a reasonable-to-excellent PET image. For this purpose, whole-body MIP images of 32 patients (23 men, 9 women, age 51.9 ± 13.7 years), administered with ^{18}F-FDG (activity 5.3 ± 0.5 MBq/kg, 45 minutes uptake time) for whole-body PET/CT examinations, were evaluated. Image quality was assessed visually by two radiologists using a three-point scoring scale: poor, reasonable and excellent. The interobserver agreement revealed a kappa value higher than 0.7. Therefore, the utilisation of ^{18}F-FDG dose between 4 and 5MBq/kg is considered an optimum dose for whole-body PET/CT examination.

Keywords: ^{18}F-FDG, PET/CT, PET image quality, optimum dose, administered dose/body weight (kg), MIP

INTRODUCTION

The Centre for Diagnostic Nuclear Imaging (CDNI) of Universiti Putra Malaysia (UPM) is equipped with a Siemens® Biograph 64 True-V Positron Emission Tomography (PET)/Computed Tomography (CT) scanner (Germany) operating on a 64-multislice CT detector system. This Siemens® Biograph 64 system has the latest technology of lutetium oxyorthosilicate (LSO) for its camera detector. LSO offers greater sensitivity and improves dead time and counting rate performance, which allow it to handle even very high activity levels as compared to traditional bismuth germanate (BGO) detector (Nagasaki *et al*., 2011; Everaert *et al*., 2003; Nutt, 2002).

However, the recommended doses of ^{18}F-FDG are inconsistent among countries due to facility setup, protocol and patient

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E-mail addresses:
hishar.hassan@gmail.com (Hishar, H.),
salasiah.mustafa@yahoo.com (Salasiah, M.),
ahmadsaadff@gmail.com (Fathinul Fikri, A. S.),
drimaging@yahoo.com (Nordin, A. J.)
*Corresponding Author

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factors. Although the current practice in CDNI employs the administration of ^{18}F-FDG dose between 6 and 8 MBq/kg,the author reiterates that, with a greater sensitivity of LSO camera detector equipped in PET/CT system at CDNI, an optimal dose of ^{18}F-FDG between 4 and 5 MBq/kg administered to the patient is sufficient to achieve a reasonable-to-excellent image quality without compromising the medical diagnostic aspect. Interestingly, dose optimisation of ^{18}F-FDG has been proven to significantly reduce the total effective dose received by patients undergoing PET/CT examination and also technologists during the dose preparation.

MATERIALS AND METHODS

Patient Preparation and Administration of ^{18}F-FDG

A total of 32 patients were recruited in this study. Prior toundergoing the PET/CT whole-body examination,patients were required to fast for at least 6 hours before the scheduled appointment time. Upon admission at the centre, the patients' body weight and height were taken (average of 61.7 ± 6.2 kg, BMI averaged, 22.7 ± 1.2 kg/m^2) and blood glucose level (average, 5.10 ± 0.9 mmol/L) was recorded in their respective examination forms. Nonetheless, patients with hyper or hypoglycaemia conditions were excluded from the study to eliminate any possible bias on the uptake of ^{18}F-FDG. The patients were then briefed by a radiologist on the complete procedure and also possible adverse effects prior to giving and signing an informed consent.

^{18}F-FDG dose ranging from 4 MBq to 5 MBq/kg (average of 5.3 ± 0.5 MBq/kg), which had been prepared in the hot laboratory, was intravenously administered to the patients. The dose prepared was lower than the usual practice as to prove that a lower dose of ^{18}F-FDG was sufficient to yield a reasonable-to-excellent image quality. The total dose given was calculated in accordance to the patients' body weight. Then, the patients were asked to lie down on a bed in a dimly lit room for approximately 45 minutes to minimise brain and muscle stimulations during the ^{18}F-FDG uptake.

Image Acquisition

Quality Assurance and Quality Control of PET/CT Scanner

Routine quality control procedures were performed by a radiographer on the PET/CT scanner in accordance to IAEA Human Health Series No. 1, Quality Assurance for PET and PET/CT Systems prior to the examination. This was to ensure that the system was operated within the tolerance level without affecting the image quality of PET/CT, patients' dose of CT, accuracy of CT-based attenuation corrections and accuracy of CT and PET co-registration (IAEA 2009).

Imaging Protocol

Following the 45-minute uptake time, the patients were brought to the PET/CT suite. The radiographer assisted the patients during the procedure. The patients were positioned with their arms above the head, lying supine on the scanning table throughout the examination. A low dose (150 mAs, 120 kVp) axial CT topogram scan was performed from the base of skull to the mid-thigh region using a modulated 4D care dose system for the purpose of study

planning. An Optivantage™ dual head injector was used to deliver about 80 to 100 ml of contrast (Omnipaque), with the delivery rate set at 2 to 3 ml per second. This was done to encourage renal excretion and enhance the contrast of structures or fluids in the body for the purpose of diagnostic imaging.

Upon completion of the CT image acquisition, the second phase of the study was commenced with PET image acquisition. The PET images were acquired in 3-dimensional (3D) mode on all patients in 5 different bed positions at 2 minutes per bed position. At the end of the examination procedure, all the images were reconstructed using the iterative algorithm technique. CT data fused to PET data was used for attenuation correction of the PET images. Meanwhile, a3D maximum intensity projection (MIP) and 2D multiplanar image in axial, coronal and sagittal were made available for reviewing. Each of the 3D MIP images of the 32 study subjects were then analysed and scored visually using a 3-score system: poor, reasonable and excellent by two nuclear radiologists.

Statistical Analysis

The interobserver of the agreement between two nuclear radiologists was evaluated using kappa measure of agreement (K). Kappa values of >0.8 indicate almost perfect agreement while kappa values of 0.61 to 0.80 indicate substantial agreement (Viera *et al*., 2005).

RESULTS

Patients' body weight was recorded and an average of 61.7 ± 6.2 kg was attained, with average BMI value of 22.7 ± 1.2 kg/m^2 and average blood glucose level of 5.10 ± 0.9 mmol/L.

The visual analysis of MIP PET images for each of the image was given the scores of "excellent", "reasonable" and "poor", as shown in Fig.1.

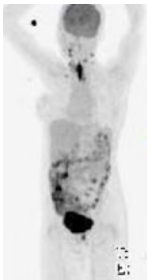
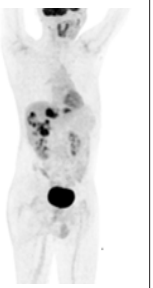
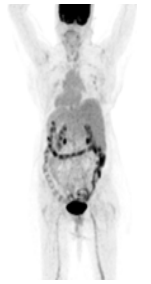
"Poor"		*"Reasonable"*		*"Excellent"*	
					
*(a) F, 54 yr old, **6.35 MBq/kg*	*(b) M, 51 yr old, **7.60 MBq/kg*	*(c) F, 50 yr old, 4.67 MBq/kg*	*(d) M, 47 yr old, 4.59 MBq/kg*	*(e) M, 48 yr old, 4.97 MBq/kg*	*(f) F, 65 yr old, 4.75 MBq/kg)*

Fig.1: Representative MIP whole body PET/CT images quality scoring system

As observed in Figures 1(e) and (f), the MIP images were considered as excellent by nuclear radiologists based on the low background noise to the image sharpness and the intensity of ^{18}F-FDG in the organ of interest: the brain, liver and urinary bladder. In contrast to Figures 1(a) and (b), the region of interest drawn was rather faint. Meanwhile, the MIP images of 1(a) and 1(b) were from the patients who had been administered with ^{18}F-FDG doses at 6 to 8MBq/kg.

The cross tabulation of the visual analysis scores of PET image quality given by two nuclear radiologists in relation to the activity administered per kg of body weight of ^{18}F-FDG is summarised in Table 1.

TABLE 1 : Cross Tabulation on Interobserver Agreement of Visual Analysis Scores of PET Scan Image Quality in Relation to Administered Activity of 18F-FDG (4 to 5 MBq/kg)

Cross tabulation on visual analysis scores of PET image quality

Nuclear Radiologist 1	Image quality	Nuclear Radiologist 2			Total
		Poor	Reasonable	Excellent	
	Poor	2	0	0	2
	Reasonable	1	8	3	12
	Excellent	0	1	17	18
	Total	3	9	20	**32**

The tabulated scores of the PET image quality presented in Table 1 show that out of the 32 images analysed by two nuclear radiologists, 30 images (94%) were classified as reasonable and excellent by Nuclear Radiologist 1 as compared with only 29 images (91%) by Nuclear Radiologist 2. The interobserver agreement between the two nuclear radiologists on the PET image scoring was evaluated in the statistical analysis using the kappa measure of agreement (K). The agreement between the two nuclear radiologists for the complete group of PET image quality was apparently substantial, with a kappa value of 0.709 ($p < 0.05$).

DISCUSSION

A shift to an optimum dose by means of lowering the current dose practice from 6-8 MBq/kg to 4-5 MBq/kg could also potentially yield a reasonable-to-excellent PET/CT image. It is known that the uptake time period, patient's blood glucose level, patient motion, patient comfort and inflammation are the biological and physical factors that attribute to the estimation of standardise uptake value (SUV) and image quality (Boellaard *et al*.,2008). In this study, however, the researchers ensured that all the above-mentioned factors were minimised and controlled to reduce any potential statistical bias.

Referring to Fig.1, the "gold standard" of characterising the excellent quality of the PET image is by looking at the sharp contrast between the organs of interest (brain, liver and urinary bladder) and the low level of background noise (Everaert *et al*.,2003). As observed in Figures 1(e) and (f), the intensity of ^{18}F-FDG in structures of organ or interest appeared to be sharp and homogenous.

The interobserver agreement on the PET images from both the nuclear radiologists in the form of kappa value, 0.709 ($p < 0.05$), revealed the interobserver agreement was substantially good. This finding indicates a good level of agreement during the independent assessment on PET image (Viera *et al*.,2005). As the assessment of the PET image quality given the ^{18}F-FDG dose at 4 to 5 MBq/kg revealed a reasonable-to-excellent quality image, it is recommended that a shift from the current practice of 6-8 MBq/kg to 4-5 MBq/kg of ^{18}F-FDG dose be administered to patients.

Moreover, this recommended low dose is also in compliance with the recommendation by the International Commission on Radiological Protection (ICRP) on dose optimisation (Hishar *et al*., 2014; Hishar *et al*., 2013; Everaert *et al*., 2003). Lowering the current practice dose would directly reduce the effective dose (internal exposure) received by the patients. The effective dose for ^{18}F-FDG is calculated via the amount of radioactivity administered and the entrance exposure (IAEA 2008). The effective dose of internal exposure from intravenous administration of an ^{18}F-FDG activity can be estimated from Eint = Γ. A, where Γ is dose coefficient (18F = 19 μSv/MBq) and A is the administered activity (ICRP 1999). With the suggested dose administration of 4 MBq/kg compared to the current dose practice of 8 MBq/kg, the effective dose received by the patients would be reduced by half.

As shown in Fig.1, even at 4 MBq/kg the ^{18}F-FDG dose has yielded a reasonable-to-excellent image; therefore, the current practice of giving more than 4 MBq/kg to patients should be questioned. Even though some might refute that the radiation exposure received by the patient is outweighed by its justified benefit for medical diagnostic purpose, it is still notright to do so when a better and safer solution does exist. In addition, this approach will also help to reduce the radiation burden, not only to the patient but also to the personnel involved during the preparation of ^{18}F-FDG dose.

As for the expenditure aspect, the operational cost will be greatly reduced because CDNI purchases the ^{18}F-FDG dose from the suppliers in the market. This is due to the optimal dose that will be adjusted, which may directly influence the purchase of ^{18}F-FDG compound. With the optimal dose suggested, the author foresees that the operational cost (particularly the expenditure on ^{18}F-FDG purchase) will be reduced by at least 10 to 15 %. The author is also confident that with the dose suggested, together with the efficiency in the management of patients (patient's scheduling and proper scanning plan), there is no doubt that the operational cost of CDNI will be reduced with the added benefit of reduced radiation exposure to the patients and staff.

Limitations of the study

A limitation of this current study is the small number of patients involved as its sample, with only 32 images analysed. The study was intended to be a preliminary study to prove that with an LSO camera detector of greater sensitivity equipped in PET/CT system at CDNI, ^{18}F-FDG dose activity ranging between 4 and 5 MBq/kg is sufficient for a PET/CT whole–body examination. Thus, the extent of the study may be conducted using a larger number of patients in the future.

CONCLUSION

This short communication is to suggest the implementation of ^{18}F-FDG dose between 4 and 5 MBq/kg for standard diagnostic whole-body PET/CT scans. This preliminary study revealed that the recommended lower dose of ^{18}F-FDG activity is able to yield satisfactory image quality, which does not compromise the diagnostic ability of the test and radiation protection benefits nor incur any additional cost.

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Incinerated Domestic Waste Sludge Powder as Sustainable Replacement Material for Concrete

Kartini, K.*, Dahlia Lema, A.M., Dyg. Siti Quraisyah, A.A., Anthony, A.D., Nuraini, T. and Siti Rahimah, R.

Faculty of Civil Engineering, Universiti Teknologi MARA, Shah Alam, Selangor, Malaysia

ABSTRACT

Sludge is an unavoidable product of wastewater treatment that creates problems of disposal. Increasingly, strict environmental control regulations have resulted in limitations on sludge disposal options.Disposal by incineration has been found to be a good option. In this research, application of domestic waste sludge powder (DWSP) was used as cement replacement in concrete mix. This study utilised replacement of 3 %, 5 %, 7 %, 10 % and 15 % by weight of OPC with water binder (w/b) ratio of 0.60, 0.55 and 0.40 for Grade 30, Grade 40 and Grade 50 respectively. The performance of DWSP concrete in terms of its compressive strength, water absorption, water permeability and Rapid Chloride Ion penetration were investigated. All values of compressive strength for DWSP concrete were lower compared to the OPC control, and the strength decreased as the percentage of replacement with DWSP increased for Grade 30 and Grade 50, except for Grade 40 at replacement of 7 %. Meanwhile, water absorption and water permeability for the DWSP concrete increased as the replacement increased. Overall, with further research in producing quality DWSP, the potential of using this waste as a cement replacement material is very promising.

Keywords: Domestic Waste Sludge Powder, compressive strength, water absorption, water permeability, Rapid chloride Ion penetration

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E-mail addresses:
ce_kartini2002@yahoo.com (Kartini, K.),
DahliaLemaM@yahoo.com (Dahlia Lema, A. M.),
Q_raisyahdsq89@yahoo.com.my (Dyg. Siti Quraisyah, A. A.),
crnls_anthony@yahoo.com (Anthony, A. D.),
nanie_nuraini@yahoo.com (Nuraini,T.),
emarosseli@yahoo.com (Siti Rahimah, R.)
*Corresponding Author

INTRODUCTION

In recent decades, disposal of dry sludge have been an important problem of sewage treatment plants due to environmental restrictions. The material is not usually permitted to be buried in soil or used as agricultural fertiliser because of its high heavy metal content. For highly urbanised

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cities, sludge disposal by land filling might not be appropriate due to limitation of land. Some investigations on concrete mix designs showed that the properties of sludge have been undergoing changes through technological advancement (Zeedan, 2010; Cyr *et al*., 2007; Monzo *et al*., 2003). The increasing demand for cement and concrete can be made possible with the introduction of cement replacement. The use of dry sludge as an alternative for cement replacement as a means of waste disposal and resource recovery for sustainable and inexpensive raw materials should be looked into. It was reported that the volume of sludge in Malaysia is expected to rise to 7.0 million m^3 annually with a typical wastewater treatment plant (WTP) producing about 200,000 m^3 of sludge per day (Chiang *et al*., 2009). Abdul Jalil (2010) reported that for Kuala Lumpur itself, the per capita domestic waste generated was approximately 0.8-1.3 kg per day, with 50 % of the waste being organic as cited in Bavani and Phon (2009). With these statistics, it is expected that large volumes of dry sludge produced and finding areas for disposal will be a problem. Increasingly strict environmental control regulations have also resulted in limitations on sludge disposal options. Disposal by incineration has been found to be a good option. The product of incineration will be utilised or recycled into building and construction materials, resulting in economical, technological, ecological and sustainable advantages. This will dramatically reduce or overcome the current sludge disposal problem.

Reuse of water treatment sludge has received considerable attention recently, and the reuse of sludge in the production of construction materials has been thoroughly investigated. According to Chiang *et al*. (2009) and Deng and Chih (2001), dried sludge could be used as brick-making material. Matar (2008) commented that not more than 10 % of sludge can be added to make concrete Grade 40; any more will cause compressive strength to drop.Monzo *et al*. (2003) reported that incinerating the sludge up to 800°C can produce an amorphous SSA, while Chih *et al*. (2003) stated that sludge ash collected after incineration could be used as brick-making material. Fontes *et al*. (2004) in their investigation on the potential use of sewage sludge ash suggested that the sludge should be burnt to a temperature of 550°C for 3 hours for it to be used as cement replacement, while Deng and Chih (2001) revealed that the performance of sludge concrete is related to the amount of sludge ash added to the mixture. An increase in the amount of sludge in the internal pores of cement also increases the percentage of water absorption. A study conducted by Jamshidi (2011) on sludge concrete with w/b ratio between 0.45 and 0.55 showed that a 5 % addition of sludge in concrete gave the lowest water absorption, beyond which water absorption increased. Another form of sludge utilisation was the production of lightweight concrete aggregate from a mixture of clay and sludge (Chen *et al*., 2006). Preliminary results from Tay (1986) showed that sludge ash could also be used as filler in concrete and as brick-making material. Knowing the potential of this dry sludge as building material, a study was initiated to investigate the potential use of the locally available dried wastewater sludge as partial replacement for cement in Grade 30, Grade 40 and Grade 50 concrete in terms of its compressive strength and durability index performance.

METHODOLOGY

Materials

Domestic Waste Sludge Powder (DWSP) was produced from sludge obtained from KLIA Wastewater Treatment Plant. The wet digested sludge cake with its odour of tar was allowed to dry under the hot sun for a week to remove some of the moisture, and later burnt under uncontrolled burning in a ferrocement furnace for 72 hours. This was to ensure that some of the water in the sludge was removed so as not to add to the total amount of water required (i.e. water binder ratio; w/b) in the concrete mix. The dry sludge cakes of about 5 kg were ground using the Los Angeles (LA) Abrasion machine. Inside the LA drum, there were 45 ball bearings, each of diameter 25 mm. The drum was rotated at 5000 revolutions using an electric motor at a speed of 25.7 rpm (revolutions per minute). After grinding, in ensuring fineness, the crushed dry sludge was sieved through a 90 µm sieve in order to produce DWSP. The fineness of 100 gram DWSP passing a 90 µm sieve was 25 %. Fig.1 shows the process of obtaining the DWSP. Other materials used in the concrete mixture were crushed stone granite of 20 mm maximum size and mining sand passing a 5 mm BS 410 sieve.

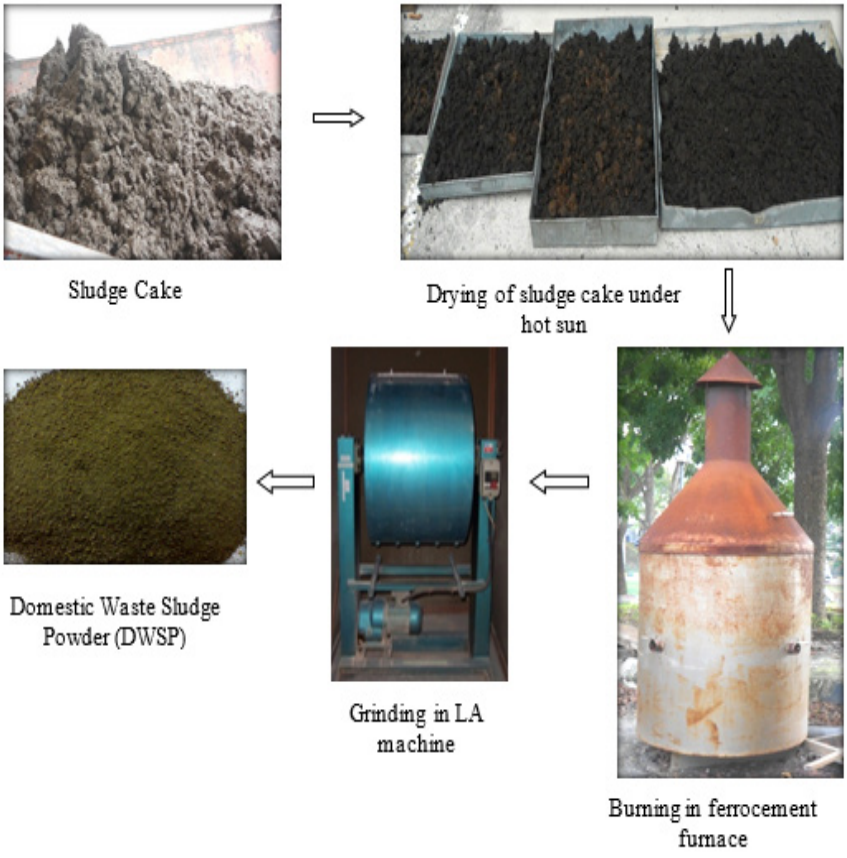

Fig.1: Process involved in the production of Domestic Waste Sludge Powder (DWSP)

The chemical composition test on DWSP and OPC was conducted and tabulated as shown in Table 1. The table shows that the oxide compositions of OPC confirmed the results obtained from the Energy Dispersive X-ray Spectroscopy (EDAX) test conducted (Fig.2). The OPC constituted of calcium oxide (CaO) and silicon dioxide (SiO_2) of 65 % and 21 %, respectively. The alkali expressed as sodium oxide (Na_2O) was 0.05%. While, SiO_2 in DWSP was 19.4 % and CaO was 5.93 %. Silicate is the main component of sludge; it is what makes sludge applicable for use as raw material in ceramic production. Sulphur trioxide (SO_3), that is, gypsum was quite high in DWSP, i.e. 8.53 %. This SO_3 is used to retard quick setting in cement. Phosphorous pentoxide (P_2O_5) content is quite high (8.77 %), and the amount is the critical factor limiting the feed rate as cement quality is adversely affected if its concentration in cement is too high. It is to be noted that sludge components vary as they depend on local circumstances and the treatment methods.

TABLE 1: Chemical Composition of DWSP and OPC

Chemical Composition	Content in %	
	DWSP	OPC
Silicon dioxide (SiO_2)	19.4	21.38
Aluminium oxide (Al_2O_3)	6.74	5.6
Ferric oxide (Fe_2O_3)	5.86	3.36
Sulphur trioxide (SO_3)	8.53	N/A
Calcium oxide (CaO)	5.93	64.64
Magnesium oxide (MgO)	0.93	2.06
Potassium oxide (K_2O)	1.69	N/A
Sodium oxide (Na_2O)	0.10	0.05
Phosphorous pentoxide (P_2O_5)	8.77	N/A
Titanium oxide (TiO_2)	0.50	N/A
Manganic oxide (Mn_2O_3)	0.06	N/A

Mix Proportion

The mix design adopted for the preparation of the concrete specimens in this research was based on methods used by the British Department of Environment (1986). Table 2 gives details of the series of the mix proportion prepared.

In this present study, six (6) series of concrete specimens were prepared based on replacement level of 0 %, 3 %, 5 %, 7 %, 10 % and 15 % of DWSP to the OPC by weight. These mixes are designated as OPC, DWSP3, DWSP5, DWSP7, DWSP10 and DWSP15, representing concrete made of OPC plain, 3 %, 5 %, 7 %, 10 % and 15 % of DWSP to the OPC respectively. As the grade of concrete varies, each series is composed of a different w/b ratio of 0.4, 0.55 and 0.6, with cement content of 475 kg/m^3, 350 kg/m^3 and 320 kg/m^3 respectively. The mixes of 100 % OPC were used as control reference. All the requirements for making the specimen were in accordance with BS EN 12390-1:2000.

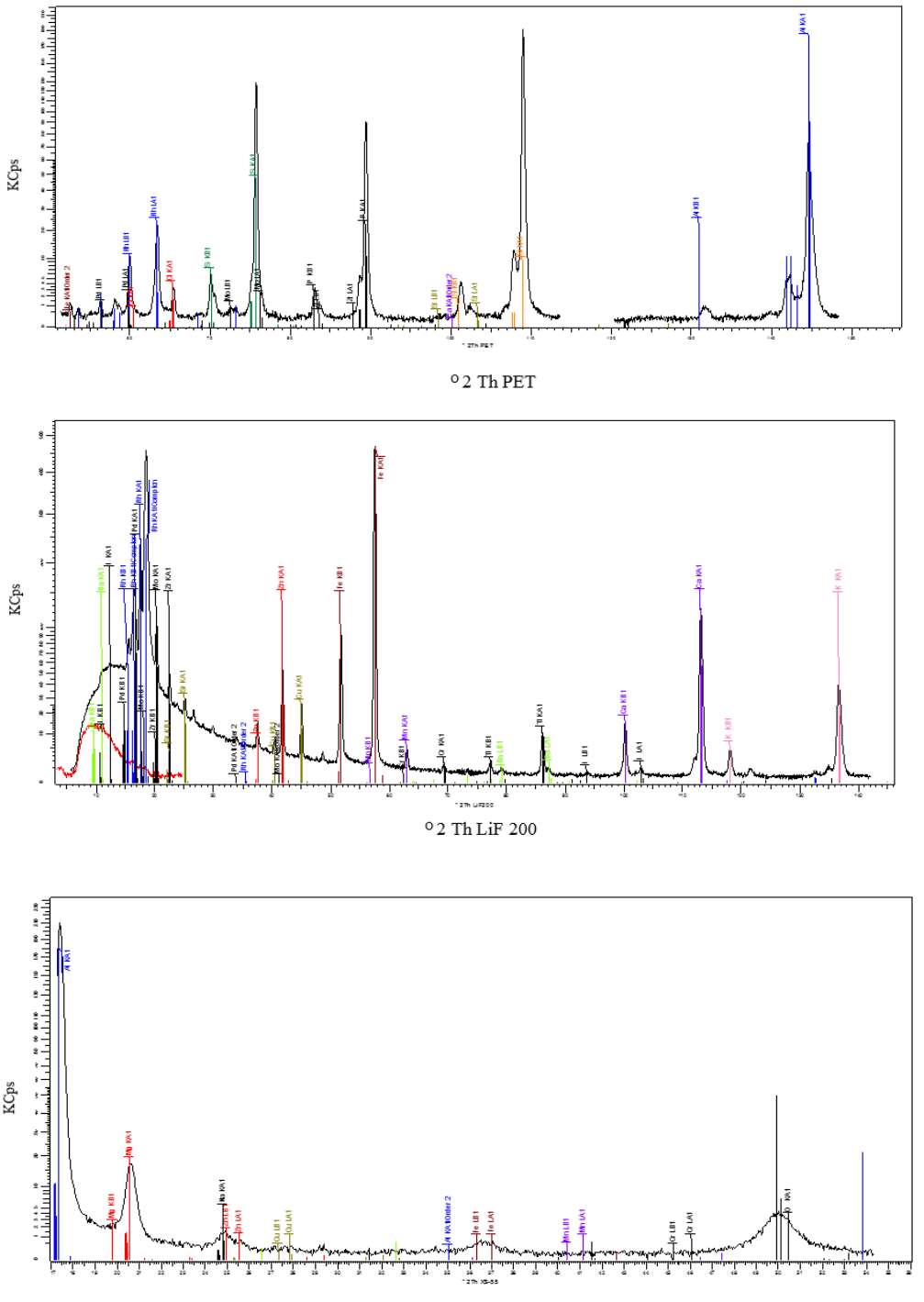

Fig.2: Energy Dispersive X-ray Spectroscopy (EDAX) test

TABLE 2 : Mixture Proportion of DWSP concrete

Mixes	Concrete Grade	Mass per Unit Volume of Materials (kg/m^3)					w/b
		Cement	DWSP	Water	Aggregate		
					Fine	Coarse	
OPC	30	320	-	190	1170	850	0.6
DWSP3		310	10	190	1170	850	
DWSP5		304	16	190	1170	850	
DWSP7		297	23	190	1170	850	
DWSP10		288	32	190	1170	850	
DWSP15		272	48	190	1170	850	
OPC	40	350	-	190	835	980	0.55
DWSP3		340	10	190	835	980	
DWSP5		333	17	190	835	980	
DWSP7		326	24	190	835	980	
DWSP10		315	35	190	835	980	
DWSP15		298	52	190	835	980	
OPC	50	475	-	190	645	1095	0.40
DWSP3		460	15	190	645	1095	
DWSP5		450	25	190	645	1095	
DWSP7		440	35	190	645	1095	
DWSP10		430	45	190	645	1095	
DWSP15		405	70	190	645	1095	

Test Methods

Several tests were performed to determine the chemical composition, fineness of DWSP and hardened DWSP concrete. For hardened concrete, the compressive strength of 100 mm cube specimens was conducted based on BS EN 12390-3:2000. The water-cured specimens were tested at the age of 7, 28 and 60 days.

For determination of the durability properties of the concrete specimens, the water absorption test was conducted on the cylindrical specimens of 50 mm in diameter by 100 mm height. The specimens were oven-dried to constant mass at 105 ± 5ºC for 72 ± 2 hours and then stored in air-tight containers as stipulated in BS 1881-122:2011. The specimens were water-cured until ages of 28 and 60 days before testing, and the specimens were weighed before the immersion in water for 30 minutes, 60 minutes, 120 minutes and 240 minutes. To determine the water permeability of the concrete, the test was based on BS EN 12390-8:2000. In Rapid Chloride Penetration Test (RCPT), three (3) water-cured specimens of size 100 mm x 50 mm dia. cylinder from each selected mix were tested for chloride ion penetration at 28 and 60 days age of testing. The test was conducted according to the standard procedure of ASTM C1202: 1997.

Table 3 summaries the types of test conducted in the research and also the numbers of specimens prepared for each test.

TABLE 3: Types of test conducted and number of specimens prepared for OPC and DWSP Concrete

Mixes	Test Conducted and Number of Specimens								
	Compressive Strength Test (Cube –100 x 100 x 100 mm)			Water Permeability Test (Cylinder –150 mm dia. x 150 mm)		Water Absorption Test (Cylinder –50 mm dia. x 100 mm)		Rapid Chloride Penetration Test (Cylinder –50 mm dia. x 100 mm)	
	Curing Period (Days)								
	7	28	60	28	60	28	60	28	60
OPC	5	5	5	5	5	5	5	3	3
DWSP 3	5	5	5	5	5	5	5	3	3
DWSP 5	5	5	5	5	5	5	5	3	3
DWSP 7	5	5	5	5	5	5	5	3	3
DWSP 10	5	5	5	5	5	5	5	3	3
DWSP 15	5	5	5	5	5	5	5	3	3
	90			60		60		36	

RESULTS AND DISCUSSION

Compressive Strength

Table 4 shows the results of compressive strength and the percentage remained of the compressive strength obtained for different concrete grades, mixes and ages of water curing. For Grade 30 concrete, the 28-day strength of all the DWSP concrete was below the control concrete (OPC) for replacement of 3 % to 15 %. It also shows that as period of curing increased, strength also increased. This is true as there is still reaction of the organics sludge with cement that occurs at a slow rate.

TABLE 4 : Compressive strength of OPC and DWSP concretes of various mixes

Grade	w/b	Compressive Strength (N/mm^2)			Percentage Remained Comp. Strength (%)		
		7 days	28 days	60 days	7 days	28 days	60 days
30	0.6	-	31.77	35.28	-	100.0	100.0
		-	26.33	30.49	-	82.9	86.4
		-	25.28	27.35	-	79.6	77.5
		-	21.76	24.40	-	68.5	69.2
		-	20.52	22.81	-	64.6	64.7
		-	18.88	20.23	-	59.4	57.3

TABLE 4 : (Cont)

40	0.55	20.63	40.24	45.54	100.0	100.0	100.0
		18.33	31.90	33.96	88.9	79.3	74.6
		22.77	37.76	40.18	110.4	93.8	88.2
		26.59	42.75	44.61	128.9	106.2	2.04
		12.63	27.46	29.55	61.2	68.2	98.0
		9.33	23.84	23.03	45.2	59.2	50.6
50	0.40	46.1	53.5	67.6	100.0	100.0	100.0
		18.1	27.3	28.4	39.3	51.0	42.0
		14.6	21.8	21.9	31.7	40.7	32.4
		7.93	16.1	20.6	17.2	30.0	30.5
		-	4.9	8.6	-	9.2	13.0
		-	0.99	3.1	-	2.0	5.0

For concrete Grade 40, it can be seen that the compressive strength of DWSP concretes decreased compared to OPC control concrete, and increased as the period of curing prolonged. However, increase in the percentage of replacement in the DWSP concrete from 5% to 7% resulted in increase in strength, beyond 7 % the compressive strength reduces. This might be because by replacing more than 7 % of cement with sludge powder, the cement reaction was lower in the concrete mass due to the decrease of the CaO ratio as a result of higher replacement of DWSP. Besides this, the oxide composition of SiO_2 in DWSP was only 19.4 % (low quality of sludge), which was much lower than OPC (21.38 %) and thus, produced slow setting, hence low strength. In Grade 50 concrete, the compressive strength of DWSP showed very low strength. In fact, with 10 % and 15 % replacement, total collapse was at 7-days' strength, while prolong curing (28 days and 60 days) gave a very small increase in strength. This might also be due to a high content of SO_3 in DWSP (8.53 %), which retarded the quick setting in cement, thus slowing the rate of hydration taking place in the matrix during the fresh state due to a high content of SO_3. A study by Cyr *et al*. (2007) on the hydration time of sludge ash with respect to compressive strength showed that as the amount of sludge ash increased, strength was reduced and at early hydration time (1 day), strength was much lower compared to longer hydration time (28 days). The high amount of sludge in the concrete mix delayed the setting time and subsequently, the concrete mechanical properties were reduced significantly due to the presence of organic material in sludge. The results obtained from this study were also in agreement with a study conducted by Matar (2008), in which the compressive strength of Grade 40 concrete decreased when the percentage of sludge content was 10 % or more.

Water Absorption

Table 5 records the percentage by weight of water absorbed for each concrete mix for Grade 30, Grade 40 and Grade 50 for four (4) hours of immersion. Based on the results obtained for each concrete grade, water absorption for the control concrete (OPC) taken at 28 days curing were 4.11 %, 4.18 % and 4.20 %, respectively. Neville (2008) revealed that concrete can be considered good concrete if the percentage of water absorption was below 10 % by mass of concrete.

For the DWSP concrete, it can be seen that as the percentage of replacement of OPC with DWSP increased, the percentage water absorption for all mixes also increased. For Grade 30, Grade 40 and Grade 50 concretes taken at 28 days of curing, the percentage of water absorption ranged between 4.30 % and 4.80 %, 4.35 % and 4.92 % and 4.40 % and 4.90 % respectively. The results obtained also confirmed the finding of Valls *et al*. (2004), which showed that absorption capacity increased with an increase in sludge content. This might be due to capacity to hold water increase together with a certain increase in the number of cavities inside the concrete. A study by Jamshidi *et al*. (2011) on sludge concrete with w/b ratio between 0.45 and 0.55 showed that a 5 % addition of sludge in concrete gave the lowest water absorption, beyond that water absorption increased. A study by Deng and Chih (2001) revealed that the ability of the concrete mixture was apparently related to the amount of sludge ash added to the mixture. They further commented that as the adhesivity of the mixture decreased, the internal pore of the cement increased when the mixture contained a high percentage of sludge (Deng & Chih, 2001). As a result, the quantity of absorbed water increased.

TABLE 5 : Water absorption characteristics of OPC and DWSP concretes

Mixes	Concrete Grade	w/b	Absorption, %	
			28 days	60 days
OPC	30	0.6	4.11	3.85
DWSP3			4.30	4.11
DWSP5			4.52	4.34
DWSP7			4.64	4.46
DWSP10			4.75	4.61
DWSP15			4.80	4.67
OPC	40	0.55	4.18	3.85
DWSP3			3.73	3.52
DWSP5			4.51	4.21
DWSP7			4.61	4.44
DWSP10			4.87	4.50
DWSP15			4.92	4.75
OPC	50	0.40	4.20	3.98
DWSP3			4.40	4.12
DWSP5			4.56	4.20
DWSP7			4.66	4.31
DWSP10			4.73	4.63
DWSP15			4.90	4.80

On the other hand, prolonged curing to 60 days resulted in a lower degree of water absorption. This might be due to the fact that proper hydration had taken placed, which over time, decreased the number of pores. However, all the mixes with the replacement of sludge could still be considered as having average water absorption value as the value was within the range of 3-5 % as stipulated in BS 1881: Part 122: 2011.

Water Permeability

Fig.3 shows the depth of penetration of water into the concrete mixes taken at 28 days and 60 days of water curing. Fig.3 also shows that 3 to 7 % of DWSP in concrete gave lower depth of penetration compared to OPC control mix concrete for Grade 40 concrete. The reason might be that DWSP material occupied the empty space in the pore structure and substantially reduces the permeability of the concrete. This resulted in reduction in the porosity of the concrete and, subsequently, the pores. This is in accordance with a study by Sandrolini and Franzoni (2003), in which they commented that porosity increased with an increase of sludge content and over periods of time (age of concrete), the number of pores decreased, thus reducing the depth of water penetration. However, further increase in the percentage of replacement with DWSP resulted in higher depth of water penetration into the concrete compared to the control OPC concrete. This is in line with Valls *et al*. (2005), who reported that higher concentration of sludge resulted in high porosity of concrete, where the pores were interconnected and contributed to the transport of fluids through the concrete.

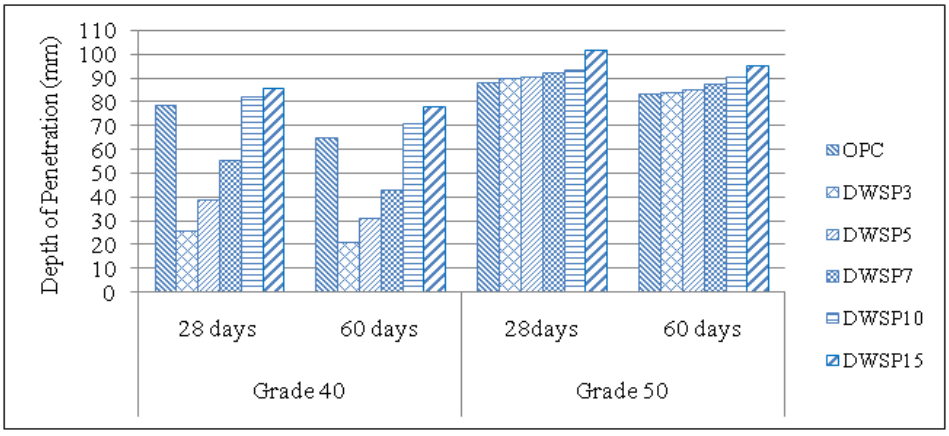

Fig.3: Depth of Penetration of water in OPC and DWSP concretes

Fig.3, which shows results for Grade 50 concrete, reveals that the depth of penetration for DWSP concrete for all series was very high compared to the control (OPC) concrete. This was again in accordance with the finding of Valls *et al*. (2005), which showed that the internal pores of the cement matrix increased when the mixture contained a high percentage of sludge. Thus, the more permeable the concrete, the lower will be its resistance to deterioration; therefore, it can be said that the durability of concrete decreases with increased in DWSP in concrete.

Chloride Ion Penetration

Table 6 shows the coulomb charge of the DWSP concrete of Grade 40 taken at age of 60 days. Reference to ASTM C1202: 1997 in determining the degree of chloride permeability (charge passed) of the concrete is also shown in the table. These coulomb charge values were obtained when the specimens were subjected to 60 V applied DC voltage for 6 hours.

TABLE 6 : Coulomb Charge of OPC and DWSP concretes

Mixes	Concrete Grade	w/b	Chloride Permeability (Coulomb)			
			28 days	ASTM C1202 (1997)	60 days	ASTM C1202 (1997)
OPC	40	0.55	4326	High	2775	Medium
DWSP3			3510	Medium	1693	Low
DWSP5			2812	Medium	1403	Low
DWSP7			1940	Low	1135	Low
DWSP10			1955	Low	1329	Low
DWSP15			2202	Medium	1477	Low

The table shows that the OPC concrete has high charge passed values at the age of 28 days and the values obtained from the OPC concrete with reference to ASTM C1202: 1997, indicated a rather high chloride penetrability characteristic, while a prolonged curing period resulted in the charge coulombs for the concrete being improved. However, for the DWSP concretes, it was seen that with the increase of sludge up to 7 % resulted in the charge passed value being reduced. Increasing the percentage of sludge beyond 7 % in the concrete resulted in an increase in the charge passed, even though according to ASTM C 1202: 1997, it still gave low-to-medium chloride permeability.

CONCLUSION

From the investigation carried out, increasing the replacement of OPC with DWSP in the concrete mixes resulted in lower compressive strength. The water absorption values of DWSP concrete were higher than the OPC control concrete.However, all the mixes with the replacement of sludge could still be considered as having average water absorption value as the values were still within the range of 3-5 % as stipulatedin BS 1881: Part 122: 2011.

OPC control concrete was more permeable than the DWSP concretes for Grade 40concrete; however, for Grade 50, the DWSP concrete gave higher depth of penetration (more permeable). The resistance to chloride ion penetration of concrete as measured by the charge coulomb drastically enhanced resistance to chloride permeability with incorporation of DWSP up to15 %. This suggested that the presence of DWSP (Grade 40) resulted in lower coefficient of permeability.

Overall, there is potential for using DWSP as partial cement replacement. However, more detailed research should be conducted to yield methods for producing quality powder.

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The Mapping of Spatial Patterns of Violent Crime in Peninsular Malaysia: Normal Mixture Model Approach

Syerrina Zakaria[1*] and Nuzlinda Abdul Rahman[2]

[1]*Department of Mathematic, Faculty of Science and Technology, Universiti Malaysia Terengganu, 21030 Kuala Terengganu, Terengganu, Malaysia*

[2]*School of Mathematical Sciences, Universiti Sains Malaysia, 11800 Penang, Malaysia*

ABSTRACT

The objective of this study was to explore the geographic distribution and temporal patterns of violent crime cases in Peninsular Malaysia by using the tools and techniques for spatial analysis. This study will also provide a general picture of violent crime patterns in Malaysia. The unit of analysis is district and the violent crime data from the year 2000 until 2009 were used in this study. In order to obtain the optimum number of components of crime in the space-time period, the space-time Normal Mixture Models were used. Based on the results of this model, the mapping of the crime occurrences was made. This map displays the spatial distribution of crime occurrence in 82 districts of Peninsular Malaysia. From this analysis, more violent crimes were shown to have occurred in developed states such as Selangor, Wilayah Persekutuan Kuala Lumpur and Johor. The findings of this study could be used by policy makers or responsible agencies to take any relevant actions in terms of crime prevention, human resource allocation and law enforcement so as to overcome this important issue in future.

Keywords: Violent crime, normal mixture, model mapping

INTRODUCTION

Crime is one of the major problems faced by most countries in the world. The most common indicator used to measure crime cases is the crime rate. The pattern of this social problem can be detected differently based on specific types of environment. Crime is believed to be higher in the more developed and densely populated areas such as at large cities, towns or urban areas compared with undeveloped areas such as the rural areas. This situation happens because of several factors such as environmental characteristic, economic, social, political, demographics, and so forth. The results of a study by Gyamfi (2002) parallel this theory. The author reported that crime was highest in

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E-mail addresses:
syerrina_zakaria@yahoo.com (Syerrina Zakaria),
nuzlinda2001@yahoo.com, nuzlinda@usm.my
(Nuzlinda Abdul Rahman)
*Corresponding Author

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southern Ghana, where it is the more developed and densely populated region. In addition, crime cases increased from the northern to southern Ghana, with a heavy concentration in Ashanti, the most populous region. Savoie *et al*. (2006) also found that property crimes highly happened in the city centre of the Island of Montreal. Meanwhile, Perreault *et al*. (2008) found that youth crime is distributed over many small hot spots across the entire island of Montreal. Crime situation was also found to be affected by global economic situation (Sidhu, 2005). During an economic downturn or recession, unemployment rate rises and people face difficulty finding jobs. This scenario will increase crime rates. The labour market conditions are also believed to have links with crimes. This association was studied by Lee and Slack (2008), who found that an index of low hour and seasonal employment had negative relationship with crime rates.

Recently, the opportunities for using social science data (i.e., crime data, disease data, accident data) to estimate spatial patterns and relationships increase because of the rapid development in the tools and methods in the field of spatial analysis. Spatial analysis is a method that is widely used to study issues related to space, place, or geographical circumstances. In spatial analysis, the location where an event occurs may provide an indication of the reason why that particular event occurs. In the beginning, spatial analysis involves mapping methods, reviews, and geographic location without formal techniques.

At the beginning of 21st century, spatial modern analyses were widely developed and focused on specific use of computer-based techniques. Mapping is one of the most widely used techniques in spatial analysis. It is very useful especially in identifying the relationship between exposure and the cases concerned. Later, spatial analysis becomes more widely used in many areas. It is not only used by researchers in the field of geography but also applied in many fields of study such as epidemiology (Forand *et al*., 2002; Kandala *et al*., 2006), biology, demography (Osei & Duker, 2008), health (Gatrell *et al*., 2004; Holt & Lo, 2008; Waller &Gotway, 2004), sociology, statistics, information technology, safety (Erdogan, 2009; Erdogan *et al*., 2008), Mathematics (Kakamu *et al*., 2008), Science and Computer Science (Corcoran *et al*., 2007).

The traditional mapping method frequently used is the percentile method. However, the classification based on percentile is arbitrary because it cannot be confirmed if this classification is able to detect high-risk or low-risk areas properly.Analternative method that can be used for classifying the risk of each study area using the mixture model (Bohning & Seidel, 2003; Schlattmann & Böhning, 1993; Schlattmann, 2003). Based on the simulation study carried out by Böhning and Schlattman (1993), that aimed to compare the mapping based on the percentile method with mixture model methods,found that the mixture model approach provided a high percentage of correct classification than the traditional methods.

In crime study, combinations of theoretical and statistical analyses are believed to be very useful. A study by Ackerman and Murray (2004) used this combination to study on assessing the spatial patterns of crime cases in Lima, Ohio, by using geographical information systems (GIS) and quantitative techniques. They analyzed the violent and property crimes at the macro, meso, and micro levels. Apart from that, several studies also investigated the impacts of factors on crime cases, while suggestions for strategic planning to overcome the problems were also provided. For example, Kanyo and Norizan (2007) studied the trend of crimes by investigating the factors that are believed to have significant effects on the crime cases in Penang, Malaysia. They also provided several suggestions related to the actions that could be taken to overcome

crime problems. Meanwhile, Felson and Poulsen (2003) studied the simple indicators of crime by the hour in 13 middle-sized American cities by using robbery data for 1999 to 2001. However, these studies were analyzed without taking into consideration the spatial effects. The analyses of crime cases by using spatial technique are growing from time to time. Rogerson and Sun (2001) analyzed the geographic patterns of crimes using the spatial method by combining nearest neighbour statistic and cumulative sum method. They described a new procedure for detecting changes over time in the spatial pattern of crime point events in Buffulo, New York. Collins *et al*. (2006) examined the basic theory and methods used to analyze the spatial lattice data and applied the method in the crime cases analysis. In their study, they used two forms of simultaneous autoregressive model known as spatial lag and spatial error model. Other studies by Ackerman and Murray (2004) used geographic information system, GIS and quantitative techniques to explore spatial characteristic of crimes. Meanwhile, Gyamfi (2002) examined the socio spatial environment factors that might have significant effects on macro geographic crime trends and patterns in Ghana.

Problems due to crimes have also become a major concern for policy makers in Malaysia. This situation can be seen from crime data given by Royal Malaysia Police (PDRM). These problems will cause not only the loss of property, lives, and misery, but also large impacts on many aspects such as psychological, economical and so on. Sidhu (2005) and Sidhu (2006) reported that crime trends developed from simple crimes which became more complex global crimes. This situation makes the work more difficult, especially for police department to detect and solve the crime cases reported by the public.

In Malaysia, limited studies have been done on crime data using statistical method. Most of the studies done focused more on experts' opinion or knowledge and discussed about the causes and effects of crimes, such as the studies by Kanyo and Norizan (2007), Sidhu (2005) and Sidhu (2006). The investigation of the crime situation can be more significant using a combination of qualitative and quantitative techniques. The objective of this study was to explore the geographic distribution and temporal patterns of violent crime cases in Malaysia using the Normal Mixture Model approach. This study is hoped to provide a general picture of the violent crime patterns in Malaysia.

RESEARCH HIGHLIGHTS

1. Instead of using commonly used crime rates based on population, this study uses crime rates based on the total number of crimes.
2. The optimum number of components of space-time mixture model for crime data from 2000 to 2009 is five.
3. Application mixture model produce smoother map compared to traditional methods.

DATA

Malaysia comprises two major parts known as Peninsular Malaysia and East Malaysia. Based on the 2000 census, Malaysia is divided into 14 states, 82 administrative districts in Peninsular Malaysia and 53 administrative districts in East Malaysia. Due to the difficulty in obtaining data in East Malaysia, this study only included districts in Peninsular Malaysia. In this study, administrative district was used as the unit of analysis. There are twelve states located in Peninsular Malaysia, consisting of a total of 82 districts.

The number of crime cases employed in this study was obtained from Royal Malaysia Police (PDRM). Two categories of crime cases were included in index crime statistics known as violent and property crimes. The definition of index crime statistics is the crimes that are reported with sufficient regularity and with sufficient significance to be meaningful as an index to the crime situation (Sidhu, 2005). It is important to note that this study only considered violent crimes.

The violent category includes murder, gang robbery with firearm, gang robbery without firearm, robbery with firearm, robbery without firearm, rape, and voluntarily causing hurt. The data from 2000 to 2009, according to each district reported in Peninsular Malaysia, were analyzed in this study.

Violent crime rate (vr) standardized by total crime cases is defined as the total number of violent crime divided by the total number of crime, T. Then, the value of rate takes a logarithmic transformation. This transformation ensures that the variables are on the same scale. The values of the log of rate, x_i follows approximately normal distribution that takes any real number (Ceccato & Dolmen, 2011; Cole, 2009; Collins, Babyak & Moloney, n.d.; Wang, 2005). Further analyses are implemented by using statistical techniques based on assumption of normal distribution. The formula for rate of violent crime is given as follows:

$$VR = \frac{V}{T}$$

where

V is the number of an event occurred and is the total number of crimes occurred. In this study, event is referred to the number of violent crime cases and T is the number of violent crime coupled with property crime cases. Instead of the common rate known as crime rate based on population at risk, this rate is used as an alternative solution for the small number problem (Waller & Gotway, 2004). Rates based on the small populations with small numbers of violent crime cases will be high than the rate based on large populations with small number of violent crime cases. Let say, there are two district with 100,000 population and 1000 population. These two districts recorded the same number of violent crime cases, which is 45. The rate is 0.00045 for the first district and the rate for the second district is 0.045. The rates do not reflect the true risks based on the crime incidents.

METHODOLOGY

Mixture Model of Normal Distribution

The mixture model assumes that the population under study comes from a certain distribution but it consists of components with different levels of risk incidence, which then becomes a heterogenous case (Lindsay, 1995; Rattanasiri, Böhning, Rojanavipart & Athipanyakom, 2004). Each component follows a normal distribution with mean, $\hat{u}_k$ and standard deviation, $\hat{\sigma}_k$, and represents a certain proportion, $\hat{p}_k$, of the total district unit.

As suggested by Everitt and Hand (1981), the parameter estimate is done by using Maximum Likelihood Estimation (MLE). The probability density functions for vector random variable, x, of dimension *n* have the following form:

$$f(x;p,\sigma,\mu)=\sum_{k=1}^{c} p_k g_k(x;\sigma_k,\mu_k) \tag{1}$$

In equation (1), $p=(p_1,p_2,...,p_{c-1})$ are the $c-1$ independent mixing proportions of the mixture where

$$0<p_k<1 \quad \text{and} \quad p_c=1-\sum_{k-1}^{c-1} p_k \ , \tag{2}$$

with μ_k and σ_k as the mean and standard deviation vector, respectively. The log of the likelihood function is given by:

$$L=\sum_{i=1}^{n} \log_e\{\sum_{k=1}^{c} p_k g_k(x_i;\sigma_k,\mu_k)\} \ . \tag{3}$$

The maximum likelihood equations are obtained by equating the first partial derivatives of (3) with respect to p_k , the elements of each σ_k , and those of each vector, μ_k , to zero.

Let the probability of observations x_i belong to the component *s* denoted by $P\left(s\middle|x_i\right)$, where

$$P\left(s\middle|x_i\right)=\frac{p_S g_S\left(x_i;\sigma_S,\mu_S\right)}{f\left(x_i;p,\sigma,\mu\right)} \tag{4}$$

Using equation (4), the estimated value of parameters are given in the following form:

$$\hat{p}_k=\frac{1}{n}\sum_{i=1}^{n}\hat{P}(k|x_i) \quad \text{where} \quad \begin{cases} p_k, & k=1,...,c-1 \\ 1-\sum_{1}^{c-1} p_k, & k=c \end{cases} \tag{5}$$

$$\hat{\mu}_k=\frac{1}{n\hat{p}_k}\sum_{i=1}^{n}\hat{P}(k|x_i) \quad \text{where} \quad k=1,...,c \tag{6}$$

$$\hat{\sigma}_k=\frac{1}{n\hat{p}_k}\sum_{i=1}^{n}\hat{P}(k|x_i)\left(x_i-\hat{\mu}_k\right)^2 \quad \text{where} \quad k=1,...,c \tag{7}$$

The next step after parameter estimation is to determine the optimum number of components that are compatible with the data. This can be done by computing Likelihood Ratio Statistic (LRS) for testing the hypothesis:

H_0 : number of components = k

against

H_a : number of components = k + 1

The likelihood ratio test is defined as

$$LRS = -2 \times [L_k - L_{k+1}],$$

where L_k is the maximum likelihood estimator under the null hypothesis and L_{k+1} is the maximum likelihood estimator under the alternative hypothesis. The LRS test has an asymptotic null distribution χ^2 of with degree of freedom equal to the difference number of parameters under the null and alternative hypothesis. However, McLachlan (1987) reported that the regularity conditions of conventional LRS result do not hold for this distribution. In order to solve this problem, several previous studies have proposed a method known as parametric bootstrap to obtain a critical value. McLachlan (1987) and Shlattmann and Bohning (1993) have provided more details on bootstrap procedure.

By applying parametric bootstrap, B bootstrap samples are generated randomly from the mixture density $f(x; p, \sigma, \mu)$ of the original sample based on the number of components under the null hypothesis. In this study, a total bootstrap samples of 200 replications were generated. Parameter estimation using the MLE method was applied for the null and alternative hypotheses for each bootstrap sample. Then, the value of LRS was computed for these bootstrap samples to get the B replicated values of LRS. Hence, the distribution of LRS under the null hypothesis is assessed. Hypothesis testing of the null hypothesis versus alternative hypothesis was tested with a bootstrapped critical value and compared with the LRS value from the original sample.

Space Time Mixture Modelling

The basic idea of space-time mixture model technique was to consider the space-time data as one dataset which consists of data combination for several years. Let be the violent crime rate for area i, i, $i = 1,...,n$ and time t, $t = 1,...,T$, the mixture probability density of normal distribution is defined as:

$$f(x; p, \sigma, \mu) = \sum_{k=1}^{c} p_k g_k(x_{it}; \sigma_k, \mu_k) \qquad (8)$$

where

$$0 < p_k < 1 \qquad p_c = 1 - \sum_{k-1}^{c-1} p_k \ .$$

The log of the likelihood function is given by

$$L = \sum_{t=1}^{T} \sum_{i=1}^{n} \log_e \{ \sum_{k=1}^{c} p_k g_k (x_{it}; p, \sigma_k, \mu_k) \} \quad . \tag{9}$$

The maximum likelihood equations were obtained by equating the first partial derivatives of (3.8) with respect to p_k , the elements of each matrix σ_k and those of each vector μ_k , to zero. One advantage of using the space-time mixture model is that it provides fewer parameters for comparison (Rattanasiri *et al*., 2004). For example, in this study, only 1 set of parameters was estimated instead of 10 sets of the parameters that were needed to be estimated for the mixture model applied separately for each year of the study period. For the purpose of computational simplicity, this study assumed that all components have the same standard deviation ($\sigma_1 = \sigma_2 = ... = \sigma_c$).

RESULTS AND DISCUSSION

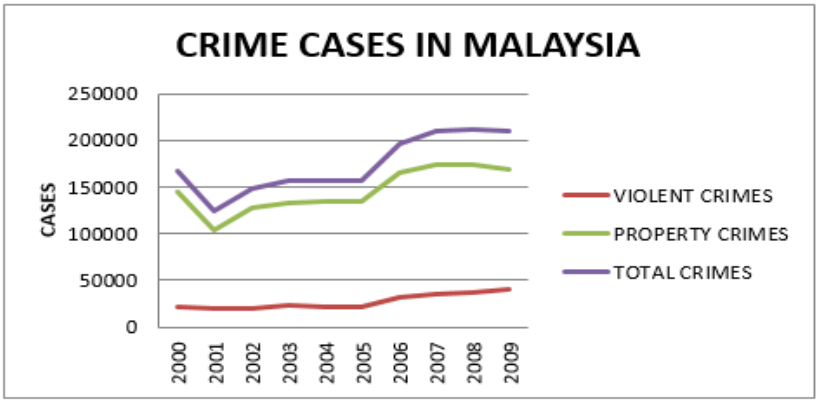

Fig.1: Crime cases in Malaysia, 2000 - 2009

As a preliminary analysis, Fig.1 shows the line graph of crime cases in Malaysia from 2000 to 2009. It shows that the violent crime cases remained along the same gradual growth from 2000 to 2005. However, the crime incidents were slightly higher from 2005 to 2009. On the other hand, the property crime cases showed fluctuation trends throughout the study period. From year 2000 to 2001 and between 2008 and 2009, the property crime cases showed a downward trend, whereas for 2002 to 2007, it showed an upward trend. The figure also showed that the property crime cases were the main contributor to the total crimes since the patterns of this crime were very similar to the pattern of the total crime cases. Throughout the study period, there were increases of 88.9% in violent crimes and 15.9% in property crimes as compared to 2000 to 2009. Although the number of cases for violent crime was less than the number of cases for property crimes, violent crimes grew at a faster rate than property crimes.That was one ofthe reasons why only the analysis of violent crime was discussed in this study. Overall, the total crime cases increased about 25.3% from 2000 to 2009.

For further analysis, the normal space-time mixture model as discussed in the methodology section was be applied to violent crime data in Peninsular Malaysia from 2000 to 2009. This space time method has made comparisons of the map much easier because it has the same scale, whereby each year has the same number of components and parameter estimates. On the contrary, a separate mixture model gives different numbers of the component for each year with different parameter estimates. This is rather arbitrary that has made map comparison difficult to do. For illustration purposes, Table 1 shows the results of the estimated parameters value for the space-time mixture model for data of 2000 to 2009.

TABLE 1 : Result of fitting space time mixture model to violent crime rate (from 2000 to 2009)

Component	Proportion, p	Log Mean, μ	Mean	Standard Deviation, σ	Log Likelihood, L	LRS , $-2(L_k - L_{k+1})$
k=7	0.1036113	-0.9825961		0.7343305	-543.9397	
	0.146502	-1.16606		0.6774542		
	0.1619918	-1.173779		0.5938236		
	0.1655543	-1.15195		0.5237591		
	0.1610484	-1.112987		0.4557219		
	0.1494789	-1.060338		0.3917262		
	0.1118132	-0.9064786		0.2710334		
k=6	0.1263943	-1.016897		0.7384063	-543.5343	-0.810929
	0.1762872	-1.175104		0.6527572		
	0.1917415	-1.168136		0.5613894		
	0.1910558	-1.131699		0.4786827		
	0.1788031	-1.076412		0.4055494		
	0.135718	-0.9255937		0.2802739		
k=5	0.159318	-1.05329	0.348788	0.7292025	-542.6071	-1.854387
	0.2189217	-1.181914	0.306691	0.6220058		
	0.2311252	-1.155231	0.314984	0.5163202		
	0.2200448	-1.09602	0.334199	0.4206643		
	0.1705903	-0.9487246	0.387235	0.291106		
k=4	0.2107838	-1.09205		0.7218557	-540.9602	-3.293775
	0.2824057	-1.181832		0.573576		
	0.2822723	-1.124204		0.4497419		
	0.2245381	-0.9774026		0.3041401		
k=3	0.3011373	-1.140015		0.6958411	-537.3221	-7.276182
	0.3810857	-1.167653		0.4984847		
	0.3177769	-1.013429		0.3199025		
k=2	0.4727242	-1.194746		0.4700092	-675.598	276.5518
	0.5272758	-1.100158		0.3820215		
k=1	1	-1.150677		0.4213953	-680.0797	8.963265

The main question arising in this analysis is whether the risk of crimes for each district in the study area is homogeneous or not. If the risk is homogeneous, the number of components is one. The hypothesis testing to test k=1 against k=2 is done. From the table, it can be seen that the improvement in log-likelihood model with two components is quite large. Thus, the two-component modelis better than a model with only one component. Large improvement was also found in the log-likelihood value when the number of components is 3 and 4. However, the log-likelihood improvement showed a small difference between the model with 4 components and the model with 5 components.

The first hypothesis tested the number of components k = 4 against k = 5. From the bootstrapped result, the 95% confidence interval of LRS distribution is (-2.828311, 1.53165). Meanwhile, the value of LRS from the original sample is -3.293775. The value of LRS from the original sample is not in the confident interval. Therefore, there is sufficient evidence to reject the null hypothesis.

The second hypothesis tested the hypothesis of k = 5 against k = 6. From the bootstrapped result, it was found that the 95% confidence interval of LRS distribution is (-2.289859, 0.9772211). The value of LRS for the original sample is -1.854387, which means that the value falls within the confidence interval. Therefore, there is no evidence to reject the null hypothesis. Since the null hypothesis cannot be rejected, the optimum number of components of space-time mixture model for crime data from 2000 to 2009 is five.

For the application of the mixture model to the space time period of 2000-2009, the optimum number of categories obtained was five components. The first component has a mean of 0.348788 [exp (-1.05329)] and standard deviation of 0.729202. This means, 0.3487 of 1 total crime is violent crime. In other words,there were approximately 4 cases of violent crime occur every 10 criminal cases. For this space time period, 15.9% of the districts were allocated in the first component, 21.9%, 23.1%, 22.0%, and 17.1% in the next category, respectively.There are more districts included in thecomponent with lower violent crime rates for components 2, 3 and 4 compared to components 1and 5 with have high crime rates.

The map was constructed based on the mean of violent crime rates where the darker areas have higher crime rates. From the map, we can see the changes of crime occurrence for each district. For illustration purposes, four maps for 2001, 2003, 2006 and 2009 were provided. For example, from Fig.2, the darkest area shave the rate of 0.387235 while the most promising areas have a mean rate of 0.306691. Whereas for every 10 cases of total crimes that have occurred, approximately 4.0 cases of violent crimes occur in the riskier areas.

As shown in Fig.2 to Fig.5, the number of districts with higher mean (shown by darker colour on the map) increased from 2001 until 2009. The obvious changes can be seen at the area of southern Peninsular Malaysia. For 2001, 2003 and 2006, it showed that no district in that area fell within the components with the highest crime rates. However, almost all the districts in the south fell into the category with the highest crime rates in 2009. The same scenario also happened in the state of Selangor, whereby more districtswere in the categories with high crime rate values in 2009 compared to the previous years. A similar situation occurred in Wilayah Persekutuan Kuala Lumpur which fell within the component with the highest crime rates in 2009.

This situation is believed to be attributed by the unemployment rate in Malaysia. For example, the unemployment rate in Johor in 2006 was 2.1%, and this rate increased to 3.2% in 2009. Meanwhile,the unemployment rates of unemployment in Selangor in 2001, 2003, 2006 and 2009 were 2.4%, 3.2%, 3.2% and 3.6%, respectively. It can be seen that the violent crime rate is affected by unemployment rate. When the unemployment rate increased, violent crime rate will also increase. This factor was discussed in this study because it is the common factor which had mostly been considered inprevious studies (see Amar & Sidhu, 2005, 2006; Andresen, 2012; Baller, Anselin, Messner & Hawkins, 2001; Ceccato & Dolmen, 2011; Kakamu, Polasek & Wago, 2008; Lee & Slack, 2008; Tsushima, 1996). However, a detail analysis should be done to check for the significant relationship between violent crime rates and the unemployment rate using a statistical analysis such as by using the spatial modelling.

Based on the map of the four-year study, there were three districts in the category with the highest crime rates, namely, Gua Musang, Sepang and Cameron Highland for all the four years. Meanwhile, most districts of the state of Johor were in the category of low crime rates in 2001, although these districts were in the riskiest category of crime rates in 2009 such as Segamat, Kluang, Mersing, Kota Tinggi, Johor Bharu, Pontian, Muar and Batu Pahat. Meanwhile, some other areas that were categorised in the less risky category in 2001 were found to be in the highest crime rates category in 2009. The districts are Manjung, Hilir Perak, Seremban, Tampin and Port Dickson.

It can be seen that the application ofthe mixture model to crime data by district in Peninsular Malaysia was able to produce a clearer map compared to the traditional method known as the choropleth method. It removes the random variability from the map (Rattanasiri *et al*., 2004; Schlattmann & Böhning, 1993; Schlattmann, 2003). Forexample, maps constructed based on the choropleth method, that is, quintiles (divided into 5 groups) of the crime rates from originaldata are presented in Fig.7 to Fig.10. As shown in those figures, the map based on the mixture model provides a clearer picture of the high and low risk areas.

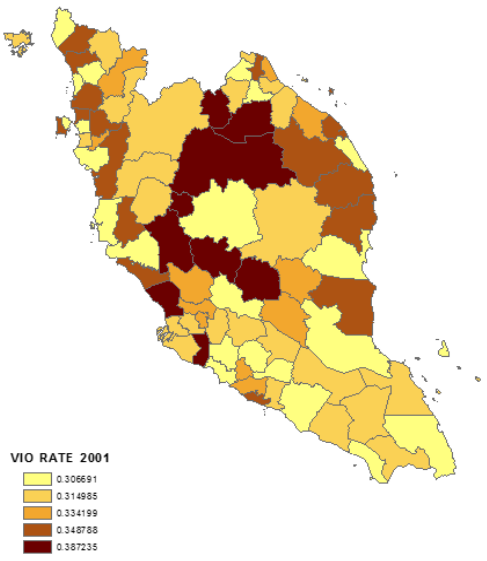

Fig.2: A map showing violent crime rate using the space-time mixture model for 2001

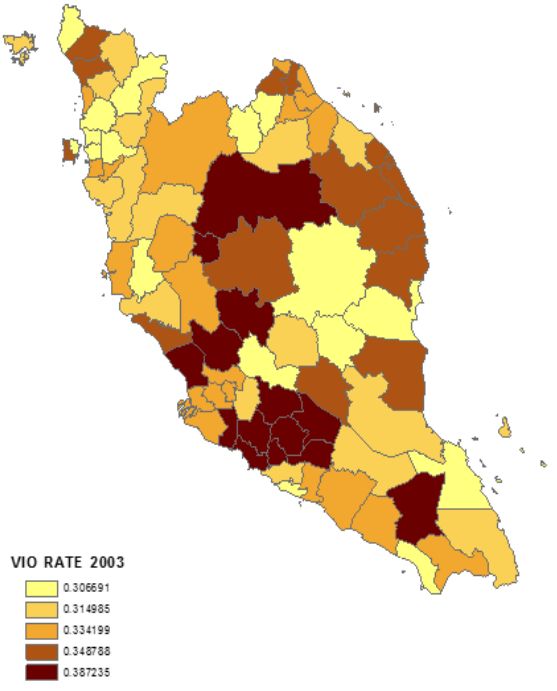

Fig.3: A map showing the violent crime rate using space-time mixture model for 2003

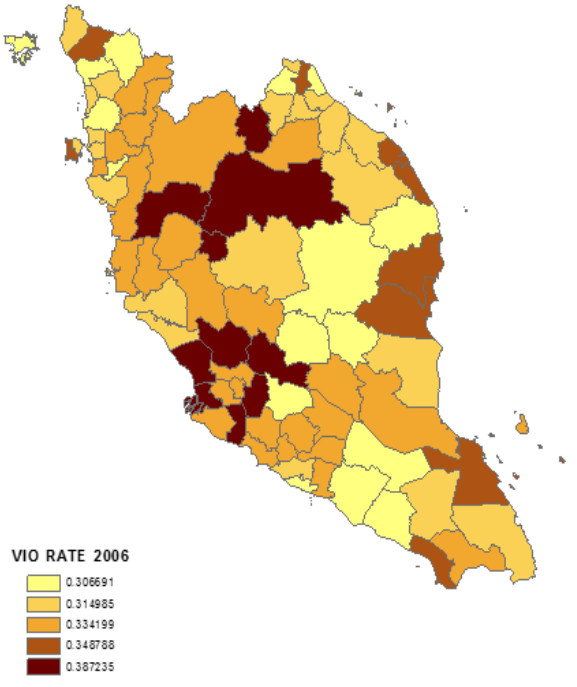

Fig.4: A map showing the violent crime rate using the space-time mixture model for 2006

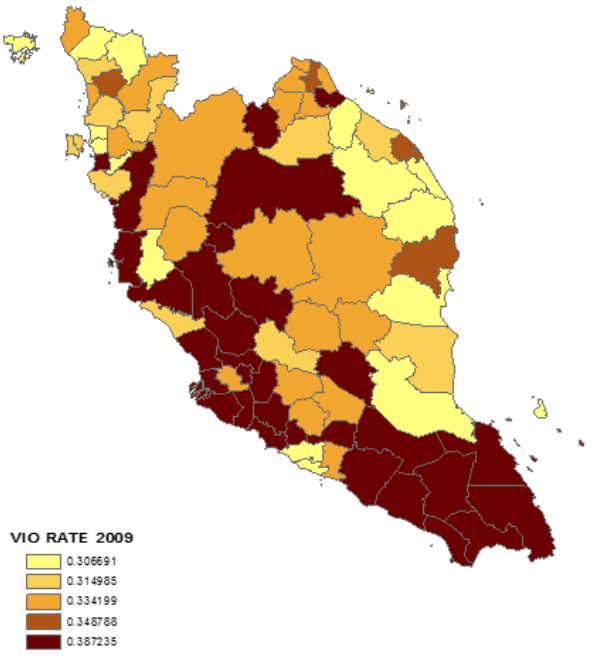

Fig.5: A map showing the violent crime rate using space-time mixture model for 2009

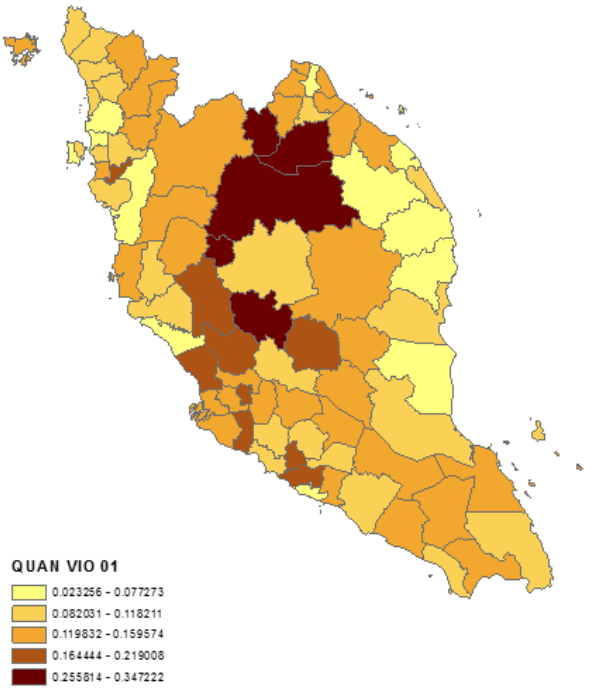

Fig.6: A map showing the violent crime rate using the percentiles method for 2001.

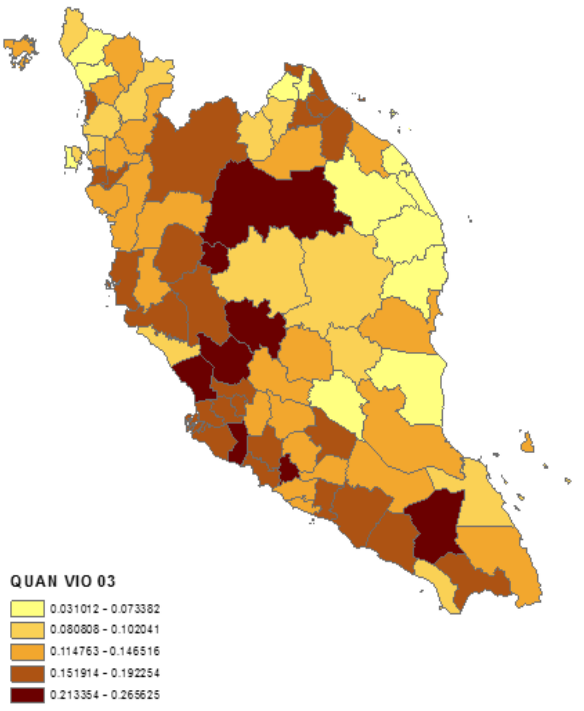

Fig.7: A map showing the violent crime rate using the percentiles method for 2003

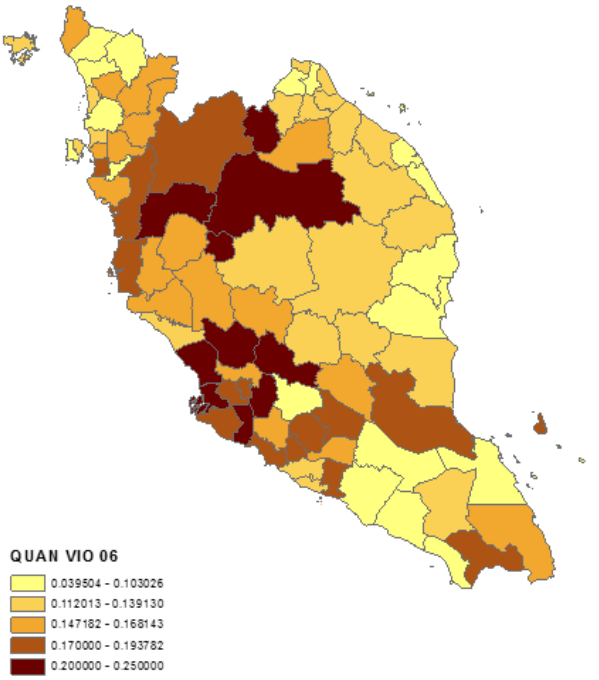

Fig.8: A map showing the violent crime rate using the percentiles method for 2006

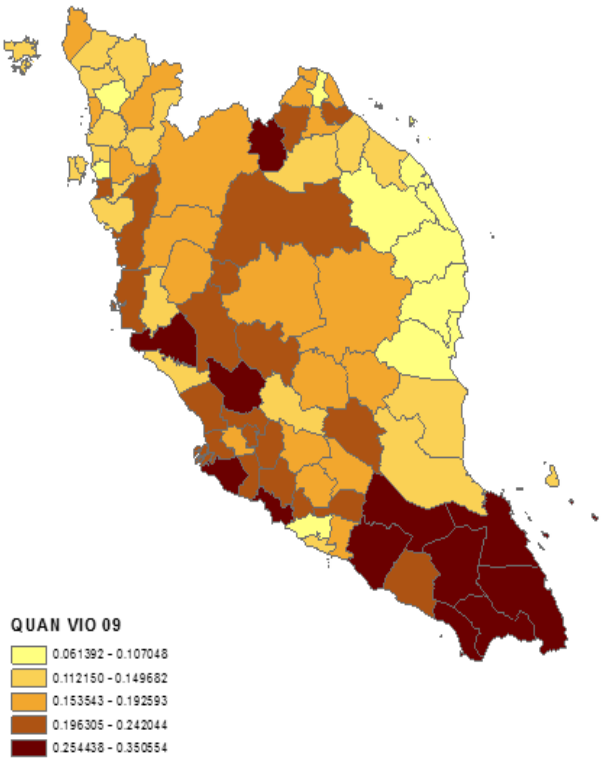

Fig.9: A map showing the violent crime rate using the percentiles method for 2009

CONCLUSION

Crime is one of the major problems faced by most countries in the world including Malaysia. This social problem causes not only losses of property, lives, and misery but also has large impacts on many aspects such as psychological, economical and so on. Mass media play an important role in the public's perception of the incident of crimes. The sensational news of crimes widely disseminated by the press and media can cause more fear amongst the public compared to the official statistics reported by the police. From this analysis, more violent crimes were found to be occurring in developed states such as Selangor, Wilayah Persekutuan Kuala Lumpur and Johor. The resultsobtained are consistent with several previous studies which reported that developed states have higher crime rates compared to developing and under developed states (Gyamfi, 2002; Savoie *et al*., 2006; Perreault *et al*., 2008).

It is impossible to provide crime forecasts with a high degree of accuracy because it is dependent upon socioeconomic, demographic and environment factors, among other. In this study, it seems that there is a relationship between violent crime rates and unemployment rates. However, for more detailed analysis, some statistical modelling should be done such as spatial regression modelling to measure the relationship between these variables. Although there is no intelligence formula that can be used to reveal future crime situations, information of the past reported crime cases can be one of the sources to predict the situation of crimes in the future.

Crime mapping is one of the methods that can be used to analyze the crime situation in a country. Based on the maps obtained, the high and low crime areas can be identified. Therefore, the authorities should pay more attention to the high crime areas so that necessary actions can be taken to overcome the crimes. The main concern is usually to reduce both crimes because they affect not only the people in particular, but also the nation in general.

It is believed that a study on the spatial and demographic patterns of crimes in Malaysia can provide useful information for the government, safety department and policymakers to make appropriate planning, especially in resource allocation and to develop suitable strategies for future plans to overcome problems pertaining to crimes. The forecast information of the future trends by using past data is a valuable tool to policy makers in developing crime prevention programmes. These outcomes can be used to target particular populations and develop interventions so that suitable actions are better adapted to the environmental and socioeconomic settings. However, well-organized data collection and computer storage are very important as these allow spatial pattern analysis over space and time to make them easier and more effective. Thus, more accurate and appropriate action plans can be done to overcome the crime situation in Malaysia.

ACKNOWLEDGEMENTS

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Evaluation of Performance Measures of Two-Lane Highways under Heterogeneous Traffic

Pritam Saha[1*], Ashoke Kumar Sarkar[2] and Manish Pal[1]

[1]*Civil Engineering Department, National Institute of Technology Agartala, Tripura, India*

[2]*Civil Engineering Department, Birla Institute of Technology and Science Pilani, Rajasthan, India*

ABSTRACT

This paper evaluates the performance measures indicated by the Highway Capacity Manual (HCM) to assess the level of service (LOS) on two-lane highways under heterogeneous traffic conditions. The study is based on field observations and, accordingly, traffic data were collected on a two-lane National Highway in India. The measures, percent time-spent-following (PTSF) and average travel speed (ATS) were estimated using easily collectible field data considering both two-way and directional segments. The actual PTSF values were observed to be lower than the expected values. This lack of conformity is probably because of drivers' impatience that increases with the platoon length and delay. The ATS was also measured in the field and compared to those obtained using HCM. ATS was observed to be insensitive to flow in mixed traffic situations because of frequent platoon formation. In addition, a direct assessment of LOS in the field indicates over-prediction, which is attributed to the presence of a sizable proportion of slower vehicles based on users' opinion poll.

Keywords: Average travel speed, heterogeneous traffic flow; level of service, percent time-spent-following, platooning, two-lane highways

INTRODUCTION

Performance evaluation on two-lane highways has always been a complicated issue because of distinct operational characteristics manifested by the utilisation of a single lane for traffic operation in each direction of travel. This results in frequent interaction between vehicles travelling in the same as well as in the opposite directions. Quite often, faster vehicles get delayed when they approach a slower one, because they are forced to travel behind the impeding vehicle while searching for an acceptable passing gap. As a result, they start moving in a platoon. The amount of platooning depends on the passing opportunity and the impedance caused by the slower vehicles. Clearly, this increases with the increase of traffic flow as well as the proportion of slower vehicles in

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E-mail addresses:
saha.pritam@gmail.com (Pritam Saha),
asarkarbits@gmail.com (Ashoke Kumar Sarkar),
mani_nita@yahoo.co.in (Manish Pal)
*Corresponding Author

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the traffic stream. These factors collectively make it difficult to maintain the desired travel speed in the traffic stream, which is a function of passing frequency on two-lane roads. Thus, the amount of platooning, in turn, significantly affects the quality of service.

The perceptions of the users of traffic facilities are expressed in terms of performance measure to describe the quality of traffic flow quantitatively. The Highway Capacity Manual (HCM) 2010 provides methods for obtaining performance measures as well as a level of service (LOS), which describes the performance of a traffic facility under given conditions. This recommends two performance measures: average travel speed (ATS) and percent-time spent following (PTSF) to assess LOS of two-lane highways. However, there are two major problems associated with these measures, which accordingly make LOS assessment inappropriate: LOS is not sensitive to speed on two-lane highways, direct measurement of PTSF is not easy, and empirical model is resorted to. In addition, a number of researchers (Luttinen, 2001; Harwood *et al*., 2003; Pollatschek & Polus, 2005) reported that the HCM empirical model overestimates the actual PTSF values. This is reasonably detectable, particularly if the traffic stream consists of a sizable percentage of slower vehicles.

It is therefore necessary to evaluate the effectiveness of the method suggested by HCM when the traffic is heterogeneous in character. To meet this objective, an attempt was made in the present study to suggest an alternative method of determining performance measures directly from field data that assess LOS under heterogeneous traffic conditions and compares the obtained values with those obtained according to HCM guidelines.

LITERATURE REVIEW

HCM Performance Measures: An Overview

HCM 1950 provided an analytical procedure for capacity analysis on two-lane highways. The manual introduced three different types of capacity: basic, possible and practical, representing capacity under ideal, prevailing and reasonable driving conditions, respectively. The manual recommended operating speed as performance measure for practical capacity to account for the "quality of service" on two-lane highways because engineers were concerned about the traffic conditions experienced by road-users; they were not interested in the maximum performance of the system only.

HCM 1965 extended the idea of practical capacity to the well-known LOS concept. Six LOSs (A–F) were defined therein; the concept is still in use today. LOS was expressed in terms of two performance measures: operating speed as the governing performance measure and volume-to-capacity ratio as the supplementary service measure.

Average speed was considered as an inadequate measure of the balance between passing demand and passing supply while developing the third edition of HCM. In addition, despite the previous presumption, this measure was found to be less sensitive to traffic flow rate. HCM 1985 thus introduced a new performance measure, percent time delay (PTD), on two-lane highways in addition to ATS. PTD was defined as "the average percent of time that all vehicles are delayed while travelling in platoons due to inability to pass." Vehicles are considered delayed while moving inside a platoon due to reduction of speed from their desired speed. HCM 1985 suggested the use of percent of vehicles with headways less than 5 s as a surrogate measure of PTD in field studies.

The phrase "percent time delay" was somewhat inappropriate because the criterion was not delay but time spent while travelling in platoons. HCM 2000 defined a more descriptive term, PTSF, which explains better the platooning effect and lack of passing opportunities on two-lane highways. The PTSF is defined as "the average percentage of travel time that vehicles must travel in platoons behind slower vehicles because of an inability to pass." To estimate PTSF, the HCM recommends a surrogate field measure of the percentage of vehicles in the traffic stream with headways less than 3 s. ATS was considered an auxiliary performance measure for high-class (Class I) highways as it makes LOS sensitive to design speed. For Class I highways, LOS is defined by threshold values of both PTSF and ATS, whereas for Class II highways, on which motorists do not necessarily expect to travel at high speeds, the performance measure is PTSF alone. Because motorists' expectation is considered lower on Class II highways than on Class I highways, a further increase of 5% to the PTSF threshold was made for Class II highways. The flow rate at the upper limit of a given LOS is higher in HCM 2000 than in HCM 1985, as the headway criterion was lowered from 5 to 3 s. Analysis procedure laid down for level and rolling terrain was extended by two-way segment methodology whereas, for specific upgrades and downgrades, it was directional segment methodology.

The most recent edition, HCM 2010, addressed directional segments analysis procedure in general terrain (level or rolling) as well. Based on the wide range of functions served, two-lane highways were classified into three categories: Class I, Class II and Class III. The first two classes, analogous with the earlier classification, explain rural two-lane highways, while Class III highways are defined as the portions of rural highways that serve moderately developed areas (small town or developed recreational areas). The Florida Department of Transportation developed the analysis approach for these highways by modifying the rural highway method. This edition also provided distinct performance measures taking automobile and bicycle mode into consideration. Prevalent ATS and PTSF conception continued unaltered, except that an introduction of a new measure, percent of free-flow speed (PFFS), was found appropriate on Class III highways, where passing restrictions are not a major concern, but drivers are expected to make steady progress at or near the speed limit. However, bicycle levels of service for two-lane highway segments are defined based on a traveler-perception model and assessed by bicycle LOS (BLOS) score. Threshold values of PTSF and ATS correspond to the previous edition but the adjustment factors to evaluate these measures were appropriately modified.

Evaluation of HCM Measures: Study Records

Following the tradition of HCM, a number of researchers (De Arazoza & McLeod, 1993; Brilon *et al*., 1994) suggested the use of ATS as a service measure in assessing LOS. In developed areas of the United States, De Arazoza and McLeod (1993) suggested ATS as the main LOS criterion for uninterrupted flow conditions. Brilon *et al*. (1994) reported that the LOS assessment on German two-lane highways is based on ATS. In Finland, Luoma and Jaatinen (1999) considered both ATS and the predictability of travel times as performance measure for goods transport. However, a couple of international studies (Brilon & Weiser, 2006; Al-Kaisy & Karjala, 2008) made it evident that lack of a benchmark point along a performance scale is a major limitation of using ATS and makes performance comparison across sites impractical.

In support of the German experience on two-lane highways, Brilon and Weiser (2006) reported the use of average speed of passenger cars as a significant performance measure. This could be estimated over a longer stretch of highway, considering the average of both directions. This was further investigated by Al-Kaisy and Karjala (2008) on four two-lane highway study sites in the state of Montana. Moreover, they proposed the use of ATS as PFFS and ATS of passenger cars as PFFS of passenger cars. The functional relationship of these measures with the platooning variables was apparently not observed to be strong.

The headway criteria adopted in the third edition of HCM (HCM 1985) was investigated further for more practical estimation of PTD. Guell and Virkler (1988) suggested headways not larger than 3.5 or 4 s and Luttinen (1992) suggested headways not greater than 3 s for the estimation of proportion of delayed vehicles. Besides this, Botha *et al*. (1994) reported the use of PTD as a measure to describe service quality on two-lane rural highways. However, the subsequent edition of HCM considered PTSF to be more appropriate as performance measure and was given more attention in later research. Several studies (Luttinen, 2001; Harwood *et al*., 2003; Pollatschek & Polus, 2005) reported that the method suggested by HCM to determine PTSF overestimates the values. Luttinen (2001) proposed several models to estimate PTSF based on the total flow, percentage of no-passing zones and directional distribution of traffic. These models gave lower value of PTSF than that estimated by HCM. Based on a study conducted on uncertainty in the operational analysis of two-lane highways, Luttinen (2002) showed that limitations in the accuracy of the analysis procedures cause errors and reduce the usefulness of the LOS concept. Similarly, Dixon *et al*. (2002) also evaluated the HCM 2000 two-lane highway analysis procedures using the TWOPAS simulation model and field data collected from northern Idaho. The PTSF values of one-directional procedure were observed to be overestimated by both simulation models: about 10 % overestimation and about 30 % overestimation in the field data. However, the values of two-way procedure were observed to be more accurate compared to those of one-directional method. Subsequent to this research, Harwood *et al*. (2003) conducted the National Cooperative Highway Research Program Study on the HCM's two-lane road analysis methodology and indicated an overestimation of PTSF by HCM. Accordingly, they developed a revised set of curves to estimate PTSF. Based on an analysis of drivers' impatience on two-lane rural highways, Pollatschek and Polus (2005) developed theoretical models for reducing the critical passing gap with longer delays prior to the passing maneuver. The impatience of the driver may cause willingness to accept more risk because delay increases, which eventually reduces PTSF. This could be one reason for overestimating the PTSF parameter by HCM. In a paper on the German experience, Brilon and Weiser (2006) reported that, in Germany, the PTSF has never been considered a substantial measure of effectiveness as it does not directly express the degree of efficiency of traffic operation. Polus and Cohen (2009) later developed a queuing model to estimate PTSF from the field data. This was used in a study conducted on 15 two-lane rural highway sections in northern Israel, and the actual PTSF values obtained from the study were also observed to be considerably lower than the corresponding HCM values. In another study, Cohen and Polus (2011) observed similar lower values of PTSF and provided improved relationship between PTSF and two-way flow by fitting the new estimates by means of the least-squares method. Bessa Jr and Setti (2011) recalibrated the HCM 2000 ATS and PTSF functions for Brazilian

roads using a genetic algorithm. These new models were able to better represent the behaviour of traffic streams. In a recent paper, Rozenshtein *et al*. (2012) reported that they calibrated PTSF models using actual field data collected on 84 one-way segments of two-lane rural highways in Israel and compared those with HCM and other empirical models proposed in the past. Following the trend of past research, they also reported significant overestimation of the PTSF values, particularly during moderate- and low-flow condition. Meanwhile, Morrall and Werner (1990) proposed the use of overtaking ratio, which is obtained by dividing the number of passing achieved by the number of passing desired, as an indicator of LOS on two-lane highways; Romana and Pérez (2006) suggested an alternative way of using the HCM 2000 performance measures, that is, ATS and PTSF for evaluating the LOS on two-lane highways. On the basis of a study conducted in Egypt on evaluation of operational performance, Hashim and Abdel-Wahed (2011) defined seven performance measures and three platooning variables. They found follower density performance measure to have the strongest correlations to platooning variables.

The above studies, however, do not consider the effect on performance measures when the traffic is heterogeneous in character. This is significant in developing countries, where heterogeneity in traffic mix is prevalent. Even the traffic composition occasionally consists of countable percentage of non-motorized traffic. As a consequence, large speed variation in the traffic stream becomes one of the essential factors for frequent platoon formation and affects performance measures. The present study evaluates the HCM measures using actual field data comprising considerable proportion of slower vehicles collected on two-lane highways in northeast India; this has been a matter of great concern to the engineers analysing traffic flow on these roads.

STUDY DESIGN

An approach toward ATS measurement

On a two-lane highway segment, HCM recommends deriving ATS from free-flow speed (FFS) after incorporating appropriate adjustments for demand flow, heavy vehicle and grade and percentage of no passing zones, respectively. At free-flow condition, ATS corresponds to FFS, and the value starts decreasing with the increase of flow mainly because of the inability to pass slower vehicles. However, there is a difficulty in estimating average FFS at heterogeneous traffic because of the different perceptions of drivers and vehicular characteristics, even under similar roadway conditions. Noticeably, because of larger speed difference, standard deviation is observed to increase with the increase of the average FFS, thus causing error in the estimation (Luttinen, 2002).Therefore, in order to examine the suitability of ATS as a performance measure on two-lane highways if the prevailing traffic is heterogeneous in character, evaluation needs be resorted to using actual field data at different flow levels.

An approach toward PTSF estimation

The flow of traffic on two-lane highways is different basically because of the on-coming traffic in the opposite lane. Thus, the faster vehicles may get delayed due to impedance caused by slower ones and the lack of passing opportunities. This results in the formation of platoons.

On two-lane highways, this can be explained as a queuing process, wherein the second vehicle just after the lead one, i.e. the vehicle forced to move at a slow speed, is considered as the first vehicle in the "queue," and, normally, this second vehicle gets the first opportunity to overtake through gap acceptance process. Additional vehicles in the queue get their opportunity to pass slower ones as and when they reach the second position in the platoon. This flow characteristic is illustrated schematically in Fig.1.

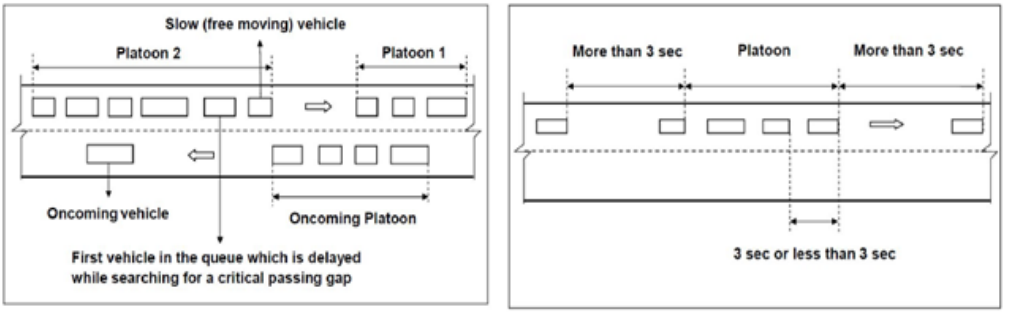

Fig.1: Schematic presentation of platooning on two-lane highways.

The determinants of PTSF on two-lane highways is certainly the amount of impedance to faster vehicles caused by slower one and the availability of passing opportunities, the effect of which is manifested in the amount of platooning. Therefore, to estimate this measure directly from the field, expected time spent by the faster vehicles while in platoon definitely needs to be measured, which is indeed not easy. Polus and Cohen (2009) developed a method to obtain PTSF as a function of average number of headways inside and outside a platoon. Adopting the same approach, Saha and Sarkar (2013) however incorporated minor modification and obtained the following equation (Eq. 1).

$$PTSF=100Q/(Q_o+N_o) \qquad (1)$$

The average values of both Q_o, average number of headways inside platoons and N_o and average number of headways between platoons are very simple to observe in the field, making field estimation of PTSF easy.

Study sites and field data

Field studies were conducted to observe ATS and the average number of headways inside and between platoons on NH-44 (two-lane highway; 7 m wide with 1 m shoulder on either sides of the road), a national highway passing through Tripura, a state in northeast India. Eight segments were selected along the 20-km highway section for conducting the study at different traffic flow levels. Due consideration was given in selection of the study locations so that they would be free from the effect of intersection, curvature and ribbon development. Pavement conditions were good and uniform in all the stretches and the posted speed was 50 kmph. Based on the functions served, the section could be categorised as Class I highway. However, shoulder conditions were not good and therefor, the same road space is used by both

non-motorised and motorised traffic. In addition, there was no access control along the road stretch, resulting in a mixed traffic situation on the road. Video photographic survey technique was adopted to observe the platooning phenomenon appropriately. Two reference lines, 500-m apart, were marked, and four observation points were selected for installing the video cameras: two in each direction for recording the entry and exit of vehicles, respectively. The recordings were synchronised so that extraction errors due to time lag could be avoided. The video files were played on a computer to extract the traffic data. The necessary readings, i.e. vehicle type, registration number and the time, were noted down independently for the four specified locations when a vehicle just crossed the reference line. Presence of car and two-wheelers in the traffic stream was observed to be significant. Proportion of non-motorised traffic including both bicycle as well as paddle tricycle, three wheelers and heavy vehicles were observed to be about 15, 13 and 10 % respectively. From the extracted data, the average number of headways inside and between platoons (Q_o and N_o) were determined, and then the PTSF values were calculated using Eq. (1). Moreover, the average travel time taken by vehicles to traverse the reference lines was recorded to compute the ATS.

To estimate FFS in accordance with the HCM method, it was required to identify the time of the day as well as the duration of low flow condition (two-way flow ≤200 pc/h). Therefore, traffic volume count was made at two locations in order to determine the hourly traffic variation. The study was conducted only during the day time for 10 h (8:00 AM to 6:00 PM) because at night-time traffic flow reduces drastically and the variety of vehicle categories in the traffic stream cannot be clearly observed. Subsequent to this, the spot speed study was conducted at four locations in the study stretch by adopting video photographic survey technique only during identified time periods. A video camera was placed away from the longitudinal trap of 10-m length made on the carriageway to avoid any influence in the operating speeds of vehicles. Also, the video camera was mounted on a stand, the height of which was adjusted in such a way that it covered the entire trap length and kept some margin on both sides. The desired data were extracted from the recorded video in the laboratory using a computer. The time taken to cover the trap length by each vehicle was measured with an accuracy of 0.01 s. This time factor was used to calculate the spot speed of vehicles passing through the section. The observed speed was analysed and adjusted in accordance with the HCM guidelines to obtain FFS. ATS values were then determined as per HCM 2000 and HCM 2010 methodology at different flow levels to compare with the values obtained from the actual field data.

STUDY RESULTS

The highway section selected for the study connects the capital city Agartala of Tripura, a state in northeast India at its western end. Factually, as the highway approached the city, the traffic flow was found to be high and more heterogeneous. Thus, to cover a wide range of traffic flow level, data were collected from eight segments selected along the study section at different times of the day in accordance with the hourly traffic variation obtained from traffic volume count. Directional split in the traffic stream was observed to be nearly 50/50 in all the segments. The raw data was analysed and a wide spectrum of traffic flow was identified, which corresponded to volume by capacity ratios (v/c ratio) from 0.2 to 1.0. Both two-way

segment and directional segment (west-bound and east-bound traffic) according to HCM (2000 and 2010 editions, respectively) was considered in the analysis. ATS was measured separately for motorised and non-motorised traffic in the field (Table 1). In addition, to compute Q_o and N_o by simple counts of headways inside and outside platoons, the phenomenon of platooning was studied for the study sections.The number of platoons and average platoon length (APL) observed during the field study at different flow level is shown in Table 2.

TABLE 1: Average Travel Speed of Motorized and Non-Motorized Vehicles Observed at Different Volume by Capacity Ratios

v/c ratio	Average Travel Speed (kmph)					
	West-bound traffic		East-bound traffic		Both directions of travel	
	Motorised vehicles	Non-motorised vehicles	Motorised vehicles	Non-motorised vehicles	Motorised vehicles	Non-motorised vehicles
0.2	40.00	13.70	36.79	12.50	38.09	12.15
0.3	36.92	13.82	37.66	12.67	37.27	13.15
0.4	33.10	14.02	36.45	13.00	35.17	13.32
0.5	25.96	15.42	29.29	13.59	28.13	13.83
0.6	25.56	13.01	28.01	13.47	26.85	14.52
0.7	27.04	14.08	28.91	14.81	27.77	13.35
0.8	24.97	13.06	27.53	16.32	26.17	14.60
0.9	28.46	14.83	19.28	10.83	26.12	12.92
1	26.64	12.80	25.26	17.50	26.36	14.86

TABLE 2: Observed Number and Average Length of Platoon at Different Volume by Capacity Ratios

v/c ratio	Number of platoons observed			Average platoon length (vehicle)		
	West-bound	East-bound	Both directions of travel	West-bound	East-bound	Both directions of travel
0.2	5	4	9	2.20	3.25	2.73
0.3	89	86	175	2.46	2.55	2.50
0.4	91	154	245	2.48	2.64	2.56
0.5	116	202	318	2.96	2.93	2.95
0.6	82	91	173	3.10	3.20	3.15
0.7	154	85	239	3.34	3.32	3.33
0.8	119	89	208	3.70	3.88	3.79
0.9	118	22	140	3.53	6.00	4.77
1	87	13	100	3.90	5.23	4.57

FFS values were derived from the observed field data using HCM procedure. The total two-way flow during the study was observed to exceed 200 pc/h even under low-flow condition. Accordingly, flow rate correction was applied to the arithmetic mean of the observed speed data to compute the FFS values. The coefficient of variation of the speed data was also estimated at each study site and was observed to be in the range of 0.29 to 0.35, which is in contrast to the estimated range of 0.11 to 0.14 by McLean (1989) for desired speed. This indicates an error in the estimated FFS and thus affects the values of ATS. At different demand flow levels, ATS values were obtained using both two-way and directional segment methodology of HCM 2000and 2010. The values thus obtained were plotted against v/c ratio (Fig. 2) to observe the trend and pattern and were compared to the actual field values. The figure shows a consistent decrease of the values obtained using the HCM method with the increase of v/c ratio, whereas no significant variation was observed when measured in the field. Even during low-flow condition, ATS measured in the field was around 20 % less than those obtained using HCM. This could be attributed to the frequent interaction with the slower vehicles. The ATS measured in the field was observed to decrease at a gradual rate of up to v/c ratio 0.5, beyond which it remained unchanged. To investigate the reason, this was plotted against APL (Fig. 3). Eq. 2 presents the exponential relationship that best fit (minimum sum of error squares) the data points.

$$ATS = 17.82 * \left(\frac{1}{1-e^{\left(-\frac{APL}{3.58}\right)}}\right) \quad (2)$$

$R^2 = 0.88$

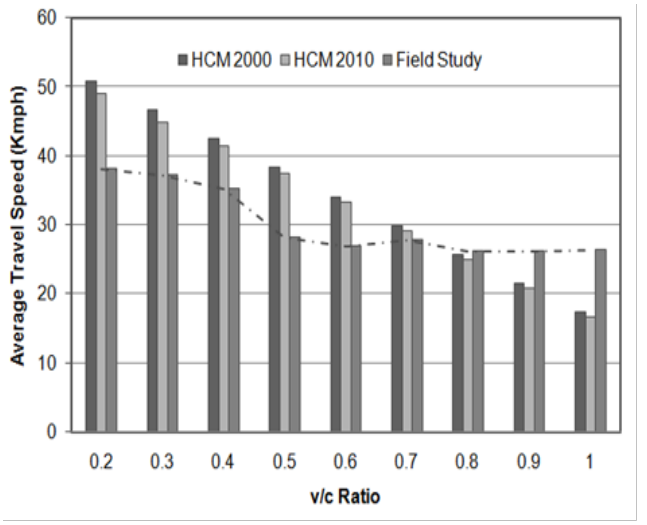

Fig.2: Comparison of average travel speed obtained from field study and Highway Capacity Manual (HCM) methods.

It may be noted from Fig.3 that ATS does not decrease sharply with the APL when its value is 3 or above. Also, an APL value of approximately 3 and above was observed against v/c ratio of 0.5 or more (Table 2). The hypothesised reason is passing opportunity reduces considerably if the queue length is 2 and above because vehicles inside the platoon must wait until the first vehicle in the queue gets an opportunity to pass the slower one; consequently, they follow the impeded vehicle. Thus, the effect of flow on speed significantly decreases as the APL increases to 3 and above. However, this hypothesis needs further investigation under heterogeneous traffic consisting of a sizeable percentage of slower vehicles. In addition, it is difficult to compare performances across sites without a reference point because of the wide range of geometric standards (Al-Kaisy & Karjala, 2008). Thus, the use of ATS as a performance measure without specifying the prevailing roadway and traffic condition is impractical.

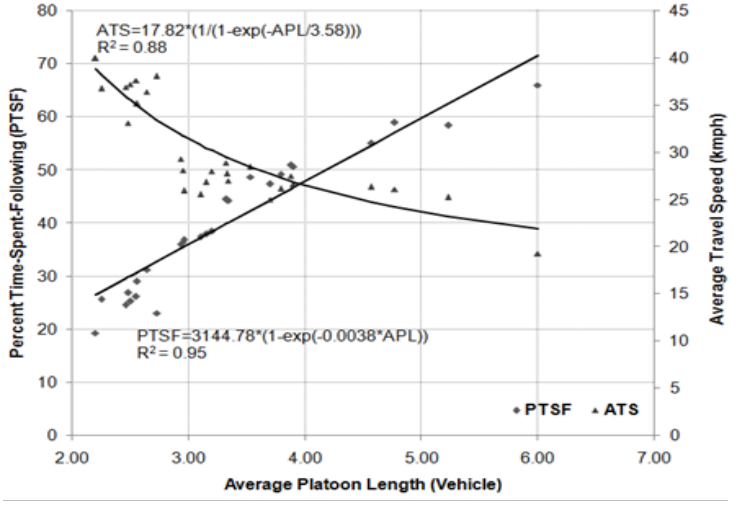

Fig.3: Effect of average platoon length on average travel speed and percent time-spent-following at study sites.

In plain terrain, HCM 2000 recommended two-way segment analysis procedure, which was subsequently modified to directional segment analysis to obtain a full estimate of operating conditions in the recent edition, HCM 2010. Thus, both two-way and directional segments were taken into consideration while computing PTSF values using field data. The actual PTSF values were plotted against v/c ratio (separately for two-way and directional segments); the relationships are presented in Fig. 4 and Fig. 5. The best-fit method was used to calibrate the relationship and was obtained as follows:

$$PTSF = 80.28 * \left(1 - e^{\left(-1.218 * \left(\frac{v}{c}\right)\right)}\right) \quad (3)$$

$R^2 = 0.97$ (For Two-way segment)

$$PTSF = 64.74 * \left(1 - e^{\left(-1.5656 * \left(\frac{v}{c}\right)\right)}\right) \quad (4)$$

$R^2 = 0.98$ (Directional segment: West-bound traffic)

$$PTSF = 95.30 * \left(1 - e^{\left(-1.0101*\left(\frac{v}{c}\right)\right)}\right) \quad (5)$$

$R^2 = 0.94$ (Directional segment: East-bound traffic)

It is evident from the observation that PTSF is sensitive to traffic flow and increases with it. This could be attributed to APL as the possibility of passing slower vehicles becomes more and more restricted with an increase in traffic flow, resulting in longer platoons i.e. higher number of following vehicles (Table 2). Figure 3 shows the relationship of observed APL and the corresponding actual PTSF values, in which PTSF increases exponentially with APL.

The calibrated curves based on HCM 2000 and HCM 2010 procedures (Figs. 4 and 5) show an overestimation of PTSF when compared to the field model. While approaching capacity, HCM model values were observed to be close to 90 % whereas field models indicate about 50–60 %. Similar lower values were also obtained by Luttinen (2001), Harwood *et al*. (2003) and Pollatschek and Polus (2005). In order to investigate the reason of overestimation, it is essential to identify the flow level in which the percent difference of HCM and field models is largest. Therefore, these values were also plotted against v/c ratio (Fig. 6), and the percent difference was observed to decrease consistently as the flow increases. However, significant variation was not observed between two-way and directional segments and a maximum value of about 50 % was found during low flow.

Study of overestimation of HCM model indicates that drivers become impatient while travelling in a platoon for a long time and sometimes take considerable risk to pass slower vehicles, disobeying lane discipline. The passing frequency thus increases, which in turn results in reducing the estimated value. At high flow level, despite the increasing risk, a few drivers even dare to overtake from the lower positions in the queue, which is explained with evidence in the subsequent development. This may also cause reduction in platoon length and thus affect PTSF. However, because of limited passing opportunities, the percent difference decreases with the flow. Driver impatience was also considered as one of the important factors that lessen actual PTSF values (Pollatschek & Polus, 2005; Rozenshtein *et al*., 2012).

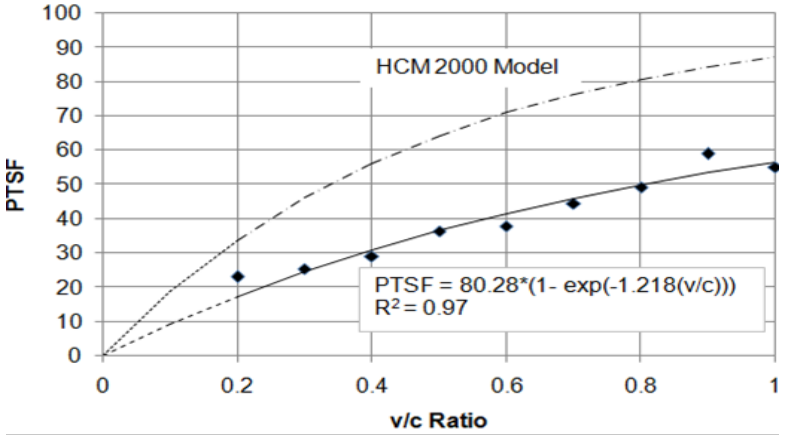

Fig.4: Comparison of percent time-spent-following (PTSF) obtained from field measurement and Highway Capacity Manual (HCM) 2000 model.

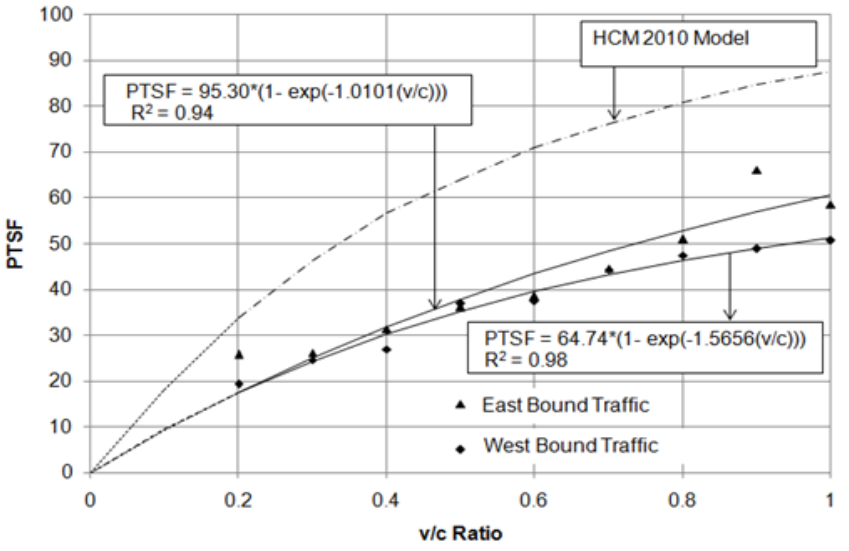

Fig.5: Comparison of percent time-spent-following (PTSF) obtained from field measurement and Highway Capacity Manual (HCM) 2010 model.

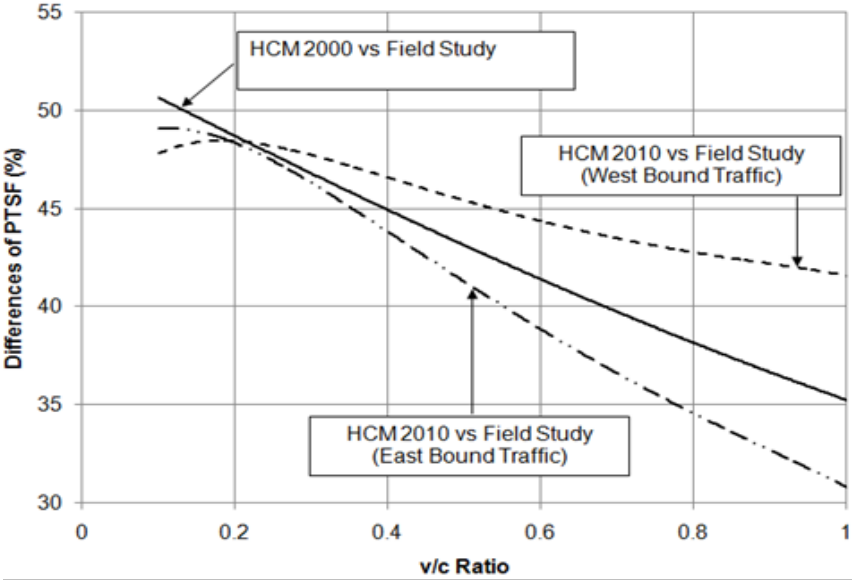

Fig.6: Differences in percent time-spent-following (PTSF) values, field study vs. HCM 2000 and HCM 2010.

The operating condition of a heterogeneous traffic stream at high flow was also investigated assuming M/M/1 queuing model (Poisson arrivals at the back of the queue and a single Poisson server for passing maneuver). For two-lane highway (single Poisson server), traffic intensity (ρ) can be expressed as a function of the number of headways inside platoons (Q_o) (Eq. 6).

$$\rho=1-1/Q_o \quad (6)$$

The traffic intensity (ρ) is defined as the ratio of average time spent by a vehicle in the first position while waiting for an acceptable gap and average inter-arrival times at the back of the queue, and it is definitely <1. The ρ values were derived from the field data using Eq. 6 at different traffic flow level, independently for both the directions (west- and east-bound traffic). The values were then plotted against v/c ratio (Fig. 7) and Eq. 7 and Eq. 8 were calibrated accordingly using the best-fit method.

$$\rho = 0.909 * \left(1 - e^{\left(-1.342*\left(\frac{v}{c}\right)\right)}\right) \quad (7)$$

$R^2 = 0.98$ (West-bound traffic)

$$\rho = 0.817 * \left(1 - e^{\left(-2.172*\left(\frac{v}{c}\right)\right)}\right) \quad (8)$$

$R^2 = 0.72$ (East-bound traffic)

At high flow, growth of queues should not be sharp because reduced passing gaps restrict the rate at which a vehicle departs from one platoon and joins the next one. The traffic intensity, therefore, should also not grow sharply at high-flow level (Polus & Cohen, 2009). However, an apparent growth of traffic intensity with respect to flow was observed in the present study (Fig. 7), and both the models (Eq.7 and 8) were asymptotic to 0.817 and 0.909, respectively. This signifies that the average waiting time may reach about 80–90 % inter-arrival times in the queue, which consequently grounds the driver's frustration. This expected behavioural change was also noticed during the study. This change eventually results in willingness of the drivers to take more risk, which could perhaps be one of the reasons for PTSF overestimation even during high flow.

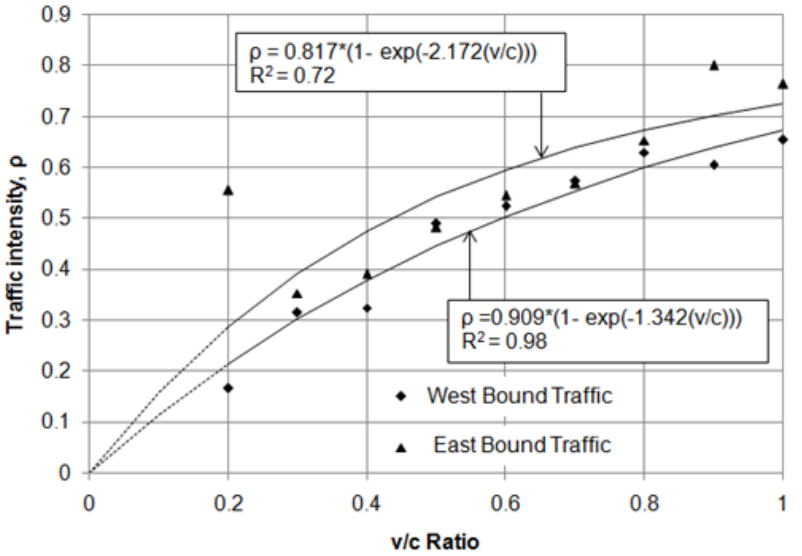

Fig.7: Relationship between observed traffic intensity (ρ) at study sites and volume by capacity (v/c) ratios.

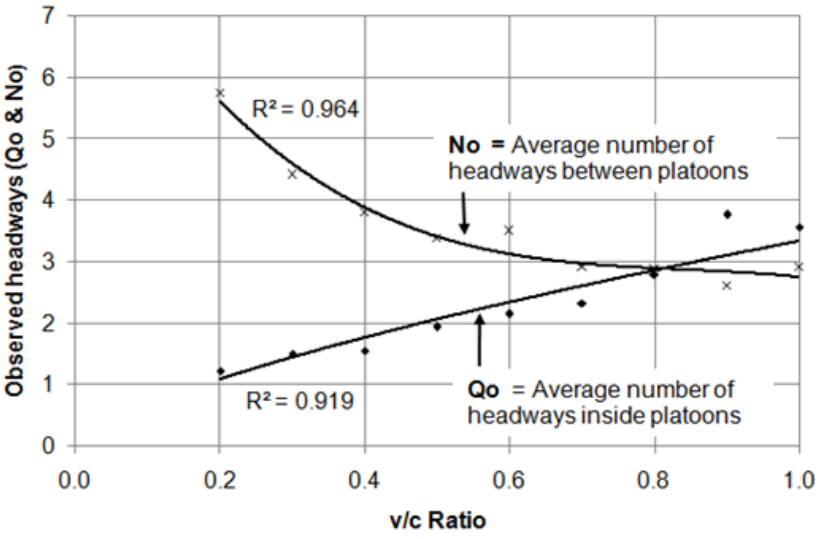

Fig.8: Average number of headways inside and outside platoons over different volume by capacity (v/c) ratios.

The endeavour of the study was to realise the root cause of the inadequacy of HCM measures in assessing LOS of two-lane highways. The method to assess LOS using field data proposed by Polus and Cohen (2009) was adopted in the present study, and the evaluation was subsequently compared with the results that could have been obtained using the HCM criteria. The method defines LOS C as the flow condition when the average number of headways inside and outside platoons is approximately equal. Figure 8 shows the headways inside and between platoons observed in the field, which points to equality of headways at v/c ratio of 0.8. However, this flow level corresponds to LOS in the range of E to F when assessed according to the HCM criteria. This implies that a large proportion of slower vehicles is present in the traffic stream, the speed of which is not impeded. These vehicles were categorically identified and about 50 road-users (including drivers and passengers of these vehicles) were invited for informal discussions; however, their responses indicated that the speed was more or less similar to their expectations. They also opined that the difference between the average times spent between and inside the platoon was relatively less for such vehicle categories. HCM performance measures, therefore, lead to an inappropriate assessment of service quality, particularly if the heterogeneity in the traffic stream is prevalent.

SUMMARY AND CONCLUSION

Quality of service is greatly compromised on two-lane highways when the traffic is heterogeneous in character. This is predominant in developing countries, where the prevailing traffic consists of a wide range of vehicle category, including a substantial percentage of slower vehicles. The evaluation of HCM performance measures is therefore imperative to assess the effectiveness of those measures under this condition. The present study therefore attempted to estimate PTSF and ATS directly from field observations and compared them with those

obtained using HCM method. Both two-way segment and directional segments were examined. It was observed that the estimated PTSF was considerably lower than those expected from HCM empirical models. However, the percent difference between field and HCM models was observed to decrease with the flow. The reason for this is acceptance of reduced passing gap during low and moderate flow, which eventually increases the passing frequency. However, during heavy flow, when drivers were forced to move in a platoon, few drivers were observed to take increased risk to overtake and thus disobey the lane discipline. Therefore, drivers' impatience may be considered as one of the possible reasons of lack of conformity between the estimated and expected values, which are perhaps not considered by the HCM models.

Usually, the average FFS becomes erroneous when the speed variation in the traffic stream is large and standard deviation of the speed data is high. This eventually affects ATS values when estimated using HCM guidelines. As a result, these values do not come in agreement with those measured directly in the field. Also, the field observation shows that speed is insensitive to flow, particularly when the platoon length becomes 3 or more. This is due to the fact that, with the increase of platoon length, the percentage of vehicles that follow increases and the average speed corresponds to the speed of the slower vehicles. At this stage, the average speed becomes insensitive to the flow. Thus, the use of ATS as a performance measure could be impractical if the formation of platoon is frequent; this is expected in mixed traffic situation. In addition, it is also important to specify the roadway and traffic condition as a benchmark and to use ATS as a performance measure.

In the present study, it was also attempted to assess LOS using direct field observations of headways. An equality of headways (inside and between) concept that defines LOS C was used for direct LOS assessment and, subsequently, compared to that obtained using HCM criteria. Based on the headway data, it was observed that LOS C corresponds to very high flow (v/c ratio 0.8) whereas it is in the range of E–F when assessed according to HCM at this flow level. This was followed by informal discussions with about 50 road-users, who indicated that the facility more or less met their expectations. Evidently, the presence of a large proportion of slower vehicles, speed of which is certainly not impeded, is the root cause of over-prediction as per HCM methodology. Further research is, however, required to understand the traffic behaviour on two-lane highways in mixed-traffic conditions for assessing LOS.

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Analysis of Absorber and Buffer Layer Band Gap Grading on CIGS Thin Film Solar Cell Performance Using SCAPS

Nima Khoshsirat[1], Nurul Amziah Md Yunus[1*], Mohd Nizar Hamidon[1], Suhaidi Shafie[1] and Nowshad Amin[2]

[1]Micro and Nano Electronic Systems Unit, Department of Electrical and Electronic Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

[2]Photovoltaic Systems and Advanced Solar Cells Research Group, Solar Energy Research Institute, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

ABSTRACT

A numerical simulation and analysis was performed to investigate the effect of absorber and buffer layer band gap grading and on a Copper-Indium-Gallium-Diselenide (CIGS) solar cell. The software used is the Solar Cell Capacitance Simulator (SCAPS). The absorber and buffer layer energy band structures' effect on the cell's output parameters such as open circuit voltage, short circuit current density, fill factor and efficiency were extensively simulated. Two structures of the energy band gap were simulated and studied for each of the absorber and buffer layer. The simulation was done on the uniform structure in which the energy band gap is constant throughout the layer. It was then continued on the cell with graded band structure, where the energy band gap of the material is varied throughout the layer. It was found that the cell with graded band structure in absorber and buffer layer had demonstrated higher efficiency and better performance in comparison with the cell with uniform band gap structure.

Keywords: Band gap, CIGS absorber, Grading, SCAPS

INTRODUCTION

Copper-Indium-Gallium-diSelenide (CIGS) thin-film solar cell is known as a promising alternative to expensive conventional silicon-based solar cells. Its characteristics for high performance and low cost have led to the direct increase in the interests in this type of thin film solar cells. The cell, consisting of the CIGS layer, is a p-type semiconductor material. The role of this layer that is to absorb incoming photons which reach the layer. According to the theoretical considerations proposed by Loferski and also based on Shockley–Queisser limit, the maximum theoretical efficiency of a solar cell can be obtained by the absorber

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E-mail addresses:
khoshsirat@ieee.org (Nima Khoshsirat),
amziah@upm.edu.my (NurulAmziah Md Yunus),
mnh@upm.edu.my (Mohd Nizar Hamidon),
suhaidi@upm.edu.my (Suhaidi Shafie),
nowshad@eng.ukm.my (Nowshad Amin)
*Corresponding Author

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layer band gap in the range of 1.2 eV to 1.5 eV (Loferski, 1956; Shockley & Queisser, 1961). This has led to the interest in using CIGS material due to its energy band gap, which is in the range of 1.06 eV to 1.7 eV. This feature firmly makes CIGS as a promising semiconductor material to be used as an absorber layer in a thin film solar cell.

The other important layer of the CIGS solar cell structure is the buffer layer. The role of a buffer layer in a heterojunction is to form a junction with the absorber layer, while leading maximum amount of incoming light to the absorber layer. The buffer layer should have minimal absorption losses, low surface recombination and minimal electrical resistance when transporting the photo generated carriers to the outer circuit (McCandless & Hegedus, 1991). The most important features of the buffer layer are protecting the junction against the chemical reactions and mechanical damage, as well as optimizing the band alignment of the cell, electrical properties and making a wide depletion region with the p-type absorber layer. This eventually can minimize the carriers tunneling and maintain higher open circuit voltage value to establish higher contact potential (Contreras *et al*., 2002). In order to satisfy such desired features, the buffer layer should have a wider band gap in comparison with the CIGS layer. The most common used material as a buffer layer in CIGS solar cells is Cadmium Sulfide (CdS). Recently, the efficiency of a CdS/CIGS solar cell was recorded at 20.4% and this was achieved when it was fabricated on flexible polymer substrates (Tiwari, 2013). This achievement was compared to the previous 20.3% efficiency which was gained with a CdS/CIGS solar cell on glass substrate (Jackson *et al*., 2011). Despite the highest efficiency of CIGS cells recorded, the cell was with CdS buffer layer. This has been a contrast to some developments of solar cells with Cd-free buffer layer which is important because of CdS toxicity was found to be harmful to human health (Siebentritt, 2004). Thus, the Indium Sulfide is one of most promising alternative materials to be used as the buffer layer in CIGS cells due to its band gap that can vary in the range of 2 eV to 2.9 eV (Ernits *et al*., 2007; Revathi, Prathap, Subbaiah, & Reddy, 2008; Saadallah, Jebbari, Kammoun, & Yacoubi, 2011). The highest efficiency level that has been achieved up to now from a CIGS thin film solar cell with In_2S_3 buffer layer is 16.4% (Naghavi, Spiering, Powalla, Cavana, & Lincot, 2003). Accordingly, there is still a gap between the highest reported efficiency of cells with In_2S_3 and CdS buffer layers.

One technique that can be used to improve the efficiency of In_2S_3/CIGS cell performance is band gap grading. The band gap grading usually is done on absorber layer band gap. Since the band gap of In_2S_3 can vary in a certain range, this technique can be done on buffer layer band structure in an In_2S_3/CIGS cell as well. In this study, the effects of absorber and buffer layer band gap grading were investigated separately.

MATERIALS AND METHODS

SCAPS Numerical Simulation Programme

SCAPS (Solar Cell Capacitance Simulator) software was designed and introduced by the University of Gent (Burgelman, Nollet, & Degrave, 2000). It is used for numerical analysis of solar cell performance and characteristics. Multiple measurements of solar cells' output parameters can be done through SCAPS. It can simulate the open circuit voltage (Voc), short circuit current density (Jsc), output J-V characteristic, fill factor (FF), quantum efficiency (QE),

cell's output efficiency (η), generation and recombination profiles, band alignment, etc. SCAPS simulation software does all these measurements by solving the fundamental semiconductor equations such as the Poissons equation and the continuity equation for the electrons and holes. These equations are solved via numerical techniques. It needs an initial prediction for the calculations and to get the solution. Thus, SCAPS will calculate several situations at the first measurement point and under the set working point. Fig.1 is a flow chart that shows the strategy of SCAPS performed for getting to the working point and the first calculation point. Each calculation begins at the start point. The initial assumption to get to the equilibrium situation is zero quasi-Fermi levels throughout the structure and no potential drop over the structure. In the case of the calculation under dark condition, this equilibrium is used as an initial condition to calculate the solution. While under light condition (with illumination), the short circuit situation is calculated in an intermediate step to serve as the next initial guess. The convergence of the Gummel type iteration scheme with Newton-Raphson algorithm is used in SCAPS for numerical calculations. This scheme's parameters can be set in the numerical setting panel as convergence settings. In order to improve the convergence, the number of iteration steps can be increased. Obviously, this can decrease the calculation speed and consequently increase the processing time, especially in batch calculations.

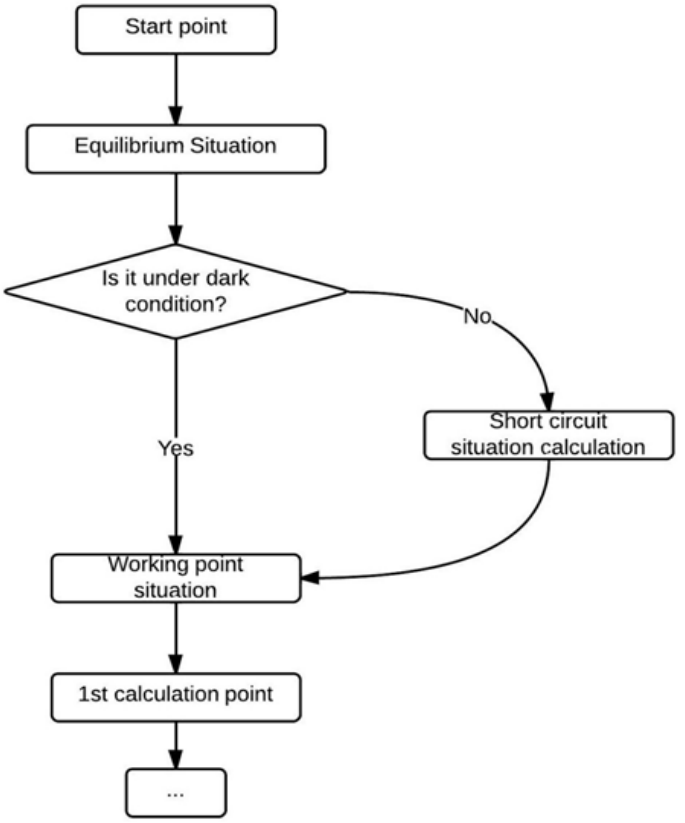

Fig.1: Strategy of getting to the working point and first calculation point

One of the most important facilities available in SCAPS is the possibility of defining layers with graded parameters. The mechanism of a graded layer definition is that the layer is considered to have composition $A_{1-y}B_y$. The properties of pure compounds A (y=0), B (y=1) and the composition grading y(x) over the thickness of the layer should be set as input values. The material properties that may vary from pure A to pure B in the layer thickness are defined as a function of y, as shown in equation (1):

$$P = f[y(x)] \quad (1)$$

Several grading functions available in SCAPS that can be used for grading over the layer are linear, logarithmic, parabolic (two laws), power law, exponential, effective medium, etc. These significant features of SCAPS are used to investigate the effects of absorber and buffer layers' band gap grading on CIGS solar cell's output performance.

Cell Structure and Materials Properties

The cell structure simulated is shown in Fig.2. The TCO window layer is actually a bi-layer made by an intrinsic zinc oxide (i-ZnO) and an aluminium- doped zinc oxide (ZnO:Al) deposited on In_2S_3 buffer layer. The absorber layer is $Cu(In_{1-x}, Ga_x)Se_2$ (CIGS) which is a compound and direct band gap semiconductor material. The band gap and consequently the electron affinity of $Cu(In_{1-x}, Ga_x)Se_2$ vary in the range of 1.06 eV- 1.7 eV and 4.6eV - 3.4 eV, respectively, due to the variation of Gallium ratio in the layer composition (x) (Saji, Lee & Lee, 2011). As mentioned in the introduction section, the band gap and electron affinity of In_2S_3 can vary in the range of 2 eV to 2.9 eV. The adjustable properties of these materials give the opportunity of band gap grading in absorber and buffer layers.

In this study, the absorber and buffer layer band gap and their corresponding electron affinity are varied in the mentioned range at individual simulation steps. There are other electro-optical properties of materials need to be set for each layer in SCAPS. Table 1 shows the summary of material properties that are used in SCPAS for this simulation. All the parameters are extracted from reliable numerical models and experimental studies (Barreau, Marsillac, & Berne, 2001; Gloeckler, Fahrenbruch & Sites, 2003; Rau & Siebentritt, 2006; Robles *et al*., 2005; Shan & Yu, 2004).

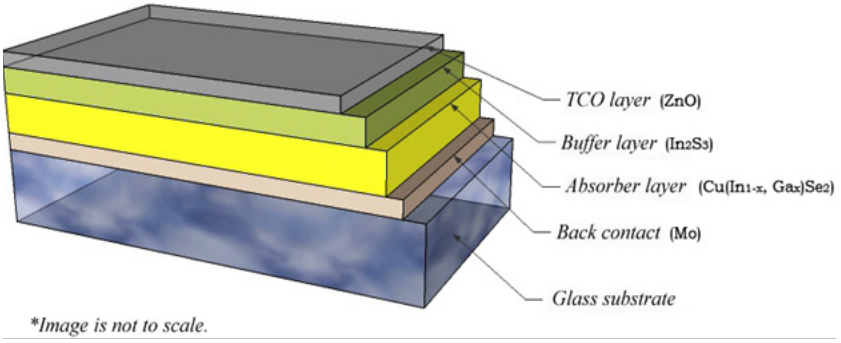

Fig.2: The CIGS thin film solar cell's structure (Khoshsirat & Md Yunus, 2013)

TABLE 1 : Summary of the parameters used in SCAPS for CIGS solar cell simulation

	Layers			
Properties	CIGS	In_2S_3	i-ZnO	ZnO:Al
Thickness (μm)	2	0.03-1	0.1	0.8
Energy Band gap (eV)	1.2	2.1-2.9	3.3	3.3
Electron Affinity (eV)	4.25	4.65-3.85	4.6	4.6
Dielectric permittivity	13.6	13.5	9	9
Conduction band effective density of states ($1/cm^3$)	2.2E+18	1.8E+19	2.2E+18	2.2E+18
Valance band effective density of states ($1/cm^3$)	1.8E+19	4.0E+13	1.8E+19	1.8E+19
Electron mobility (cm^2/Vs)	1.0E+2	4.0E+2	1.0E+2	1.E+2
Hole mobility (cm^2/Vs)	2.5E+1	2.1E+2	2.5E+1	2.5E+1
Acceptor concentration ($1/cm^3$)	1.0E+16	1.0E+1	0	0
Donor concentration ($1/cm^3$)	0	1.E+16-5E+18	1.0E+16	1.0E+18

RESULTS AND DISCUSSION

The Effect of Absorber Layer Band Gap Grading on Cell Performance

In this stage of simulation, the effect of using non-uniform band absorber layer was investigated as the band gap of CIGS absorber layer varied. In other words, the goal of this step is to study the effect of absorber layer band gap grading due to Ga content grading on the cell performance. There are several models for graded band CIGS absorber layer. One of them is the partially back grading, where the absorber layer is divided into two parts; the first part is near to the p-n junction which has uniform band gap and the second part is thinner and closer to the back contact and it has linear graded band gap and the highest band near back contact. In this simulation, the band gap of uniform part is considered 1.2 eV corresponding to the optimum energy band gap of CIGS layer. The graded part's band gap appears to increase on one occasion from 1.2 eV to 1.4 eV and next from 1.2 eV to 1.7 eV. Meanwhile, the thickness of the graded band section (d_{grd}) also varied from 0.1μm to 0.5μm and the thickness of uniform band section was kept constant at 1μm. At the last step a cell with whole back graded band absorber layer was simulated and the results of the simulation are shown in Table 2. The band diagram of cells with partially and whole back graded absorber layers are shown in Fig.3. As shown, the lower edge of conduction band varies with the variation of band gap due to Ga grading in the absorber layer. The difference between the minimum and maximum conduction band edges is shown by ΔE_g.

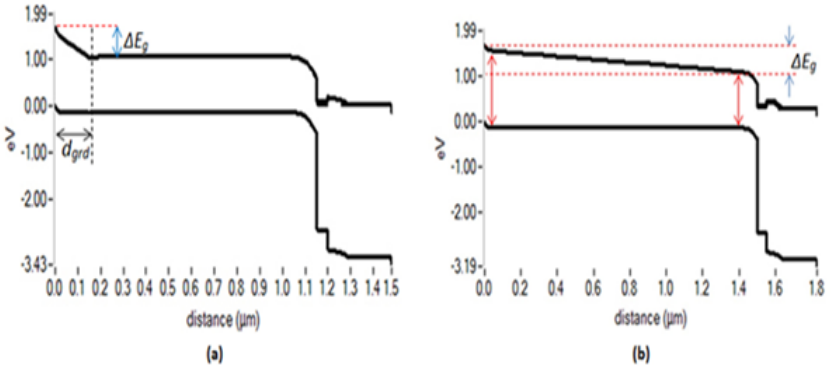

Fig.3: Band diagram of the cell with graded band absorber layer; (a) Partially back graded, (b) Whole back graded

According to the simulation results shown in Table 2 the short circuit current density is more sensitive to the variations of the graded band section's thickness and ΔEg. However, generally the cell performance will be improved by using back graded band absorber layer.

TABLE 2 : Comparison of the cells with uniform and graded band absorber layer

Band structure	ΔE_g	d_{grd}	V_{oc}	J_{sc}	FF%	η%
Cell with uniform Band absorber layer	-	-	0.7188	27.62	68.51	13.6
Cell with partially back graded band absorber layer	0.2	0.1	0.7213	29.11	68.72	14.43
Cell with partially back graded band absorber layer		0.5	0.7516	29.08	68.67	15
Cell with whole back graded band absorber layer		1.5	0.7528	32.1	74.73	18.06
Cell with partially back graded band absorber layer	0.5	0.1	0.7218	29.09	68.72	14.43
Cell with partially back graded band absorber layer		0.5	0.7219	29.214	68.69	14.49
Cell with whole back graded band absorber layer		1.5	0.7806	35.46	77.71	21.51

The results revealed that the enhancement of the cell output parameters at higher ΔE_g is more obvious. This is due to an increase of conductivity that leads to the increase of short circuit current density. Fig.4 shows the increase of cell conductance due to increase of graded section thickness. The most important benefit of using a whole graded band configuration for the CIGS absorber layer is its lower back contact recombination probability. Based on the simulation results in a cell with 1.5μm uniform band gap of 1.2 eV, the back contact recombination current density at the maximum power point voltage (V_{mpp}) is 3.66 (mA/cm^2), whereas it is 7.77E^{-4}

(mA/cm^2) in a cell with 1.5µm absorber layer that its band gap is graded linearly from 1.4 eV to 1.2 eV. This significant reduction in the difference of back contact recombination current density gives the possibility to reduce the thickness of absorber layer. Thus, higher levels of efficiency can be obtained from the cells with thinner absorber layer (<1.5 µm). This will lead to the reduction in material usage and consequently the cell fabrication cost.

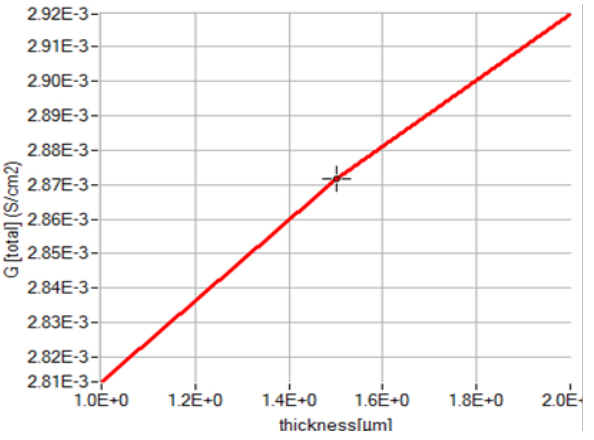

Fig.4: Increase of cell conductance due to increase of graded section thickness

Effect of Buffer Layer Band Gap Grading on Cell Performance

In this step, the effect of buffer layer band gap grading was simulated. Table 3 shows the output parameters of a cell with 0.04 µm graded band buffer layer. The electron affinity of the layer starts from 4.05 eV at areas that are closed to the absorber and increases linearly to 4.65 eV at the end of the buffer layer. As shown in Table 3, the variation of the J_{sc} resulting from the changes in the buffer layer's band gap structure is small. Therefore, J_{sc} can be considered almost constant. Both V_{oc} and FF% increased due to the buffer layer band gap grading. As tabulated, the effect of using graded band buffer layer on the cell's fill factor is significant, and this could be attributed to the lower series resistance in the cell with graded band buffer layer. The cell's output efficiency increased in consequence of increase of open circuit voltage and Fill-Factor. Accordingly, the buffer layer band gap grading could improve the performance of the cell. Fig.5 shows the difference in the J-V curve of two cells; one with a uniform band buffer layer and the other with a graded band buffer layer which refers to the series resistance difference of two structures.

TABLE 3 : Comparison of the cells with uniform and graded band buffer layer

Band structure	V_{oc}(v)	J_{sc}(mA/cm2)	FF%	η%
Cell with graded band buffer layer	0.7566	26.43	82.44	16.49
Cell with uniform band buffer layer	0.7515	26.53	78.78	15.6

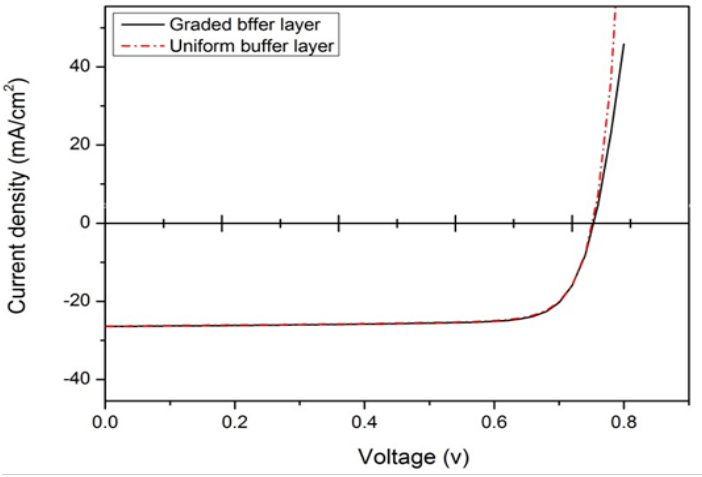

Fig.5: The J-V characteristic of cells with uniform and graded band buffer layer

As mentioned in the introduction section, the maximum efficiency reported for a CIGS thin film solar cell with In_2S_3 buffer layer is 16.4%, which was obtained from the cell with a uniform band structure for both absorber and buffer layer. Nevertheless, based on the Loferski's theoretical concerns, the maximum possible theoretical efficiency of the cell with CIGS absorber layer is about 25%, which is considered high for this kind of cell. This is still 5% lower than Shockley–Queisser limit for the single-junction solar cell efficiency.

The simulation results of this study show that an increase of the cell efficiency is possible without the growth of absorber layer thickness using the graded band structure for the absorber and buffer layer band gap. This can reduce the gap between the current efficiency record of a CIGS/In_2S_3 solar cell and the maximum theoretical efficiency of the CIGS cell.

In this study, the simulation results (see Tables 2 and 3) show that by using graded band absorber and buffer layer, losses due to back contact recombination and parasitic resistive can be reduced. Therefore, higher levels of efficiency closer to the theoretical limits are achievable. These results are exclusive and in a good agreement with experimental studies, which have been done on CIGS/CdS solar cell with graded band absorber layer (Dullweber, Anna, Rau, & Schock, 2001; Lundberg, Edoff & Stolt, 2005).

CONCLUSION

The cell performance was analyzed and simulated by the functions of absorber and buffer layer band gap grading. The numerical simulation results obtained with SCAPS showed that the cell with graded band structure in absorber and buffer layer gave higher performance as compared to the cell with uniform band gap structure. Besides, the effect of the absorber layer band gap grading on cell performance was found to be more significant. It is shown that the cell with back graded band absorber layer represents higher conductivity, lower back contact recombination and thus higher efficiency. Hence, as a result found in this structure, this study has proposed the back graded band configuration to be used for thin and ultra-thin CIGS solar cells.

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A Comparative Study on Standard, Modified and Chaotic Firefly Algorithms

Hong Choon Ong[1*], Surafel Luleseged Tilahun[2] and Suey Shya Tang[1]

[1]*School of Mathematical Sciences, Universiti Sains Malaysia, 11800 USM, Pulau Pinang, Malaysia*

[2]*Computational Science Program, College of Nature Sciences,Addis Ababa University, 1176, A.A., Ethiopia*

ABSTRACT

Many studies have been carried out using different metaheuristic algorithms on optimisation problems in various fields like engineering design, economics and routes planning. In the real world, resources and time are scarce. Thus the goals of optimisation algorithms are to optimise these available resources. Different metaheuristic algorithms are available. The firefly algorithm is one of the recent metaheuristic algorithms that is used in many applications; it is also modified and hybridised to improve its performance. In this paper, we compare the Standard Firefly Algorithm, the Elitist Firefly Algorithm, also called the Modified Firefly Algorithm with the Chaotic Firefly Algorithm, which embeds chaos maps in the Standard Firefly Algorithm. The Modified Firefly Algorithm differs from the Standard Firefly Algorithm in such a way that the global optimum solution at a particular iteration will not move randomly but in a direction that is chosen from randomly generated directions that can improve its performance. If none of these directions improves its performance, then the algorithm will not be updated. On the other hand, the Chaotic Firefly Algorithm tunes the parameters of the algorithms for the purpose of increasing the global search mobility i.e. to improve the attractiveness of fireflies. In our study, we found that the Chaotic Firefly Algorithms using three different chaotic maps do not perform as well as the Modified Firefly Algorithms; however, at least one or two of the Chaotic Firefly Algorithms outperform the Standard Firefly Algorithm under the given accuracy and efficiency tests.

Keywords: Metaheuristic, optimisation, Standard Firefly Algorithm, Modified Firefly Algorithm, Chaotic Firefly Algorithm

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E-mail addresses:
hcong@cs.usm.my (Hong Choon Ong)
surafelaau@yahoo.com (Surafel Luleseged Tilahun)
shyatang@yahoo.com (Suey Shya Tang)
*Corresponding Author

INTRODUCTION

Generally, optimisation refers to maximisation or minimization of an objective function by finding suitable values for the variables from a set of feasible values. Optimisation solution methods can be categorised into two categories: deterministic algorithms and non-

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deterministic algorithms. Heuristic or metaheuristic algorithms fall under non-deterministic algorithms. The word 'heuristic' comes from a Greek word 'to find' or 'to discover' while metaheuristic combines the words 'meta' and 'heuristic' where 'meta' means a higher level. However, no difference is recorded between metaheuristic and heuristic; researchers seem to use both names interchangeably. Heuristic algorithms are usually experience-based. Simply put, they are a 'common-sense' approach to problem solving (Luke, 2013). They are used to speed up the process of finding a good enough solution based on an educated guess, an intuitive judgment or expertise (Suh *et al*., 2011). Metaheuristic algorithms are iterative procedures that combine the concepts of exploration and exploitation within feasible regions (Osman & Laporte, 1996). Learning strategies are used to organise information in order to generate near-optimal solutions. Based on previous knowledge and most of the simulated real-life experiments done in this area, heuristic algorithms try to get solutions which are effective to dismantle the problems. They can be applied to many problems as they do not rely on rigorous mathematical characteristics of the problems and may be generally used on global optimisation problems(Fink & Voβ, 1998; Fu *et al*., 2005; Tilahun & Ong, 2012a; Kopecek, 2014; Maknoon *et al*., 2014; Cui, 2014).However,they do not guarantee the generation of an exact optimal solution within acceptable timescales (Hopper & Turton, 2000). On the contrary, metaheuristics are likely to generate near-optimal solutions within acceptable timescales. Nowadays, many studies are found to group all stochastic algorithms within random variables and global exploration into metaheuristic algorithms.

Two major search components of metaheuristics are exploration and exploitation. Exploration means looking for diverse solutions by going around entire new regions in a particular search space while exploitation means getting the current best solution by focusing on those found regions. In a case where there is too little exploration and too much exploitation, the algorithm may get trapped within local optima resulting in difficulty in finding the global optimum. Therefore, we need to achieve a balance between these components to improve the convergence of the algorithm (Creppinsek *et al*., 2000; Yang, 2011). Most metaheuristic algorithms are nature-inspired and they mimic biological, physical or natural phenomena from the real world. Some popular nature-inspired algorithms are the Particle Swarm Optimisation (PSO) algorithm, the Prey-Predator Algorithm (PPA), the Cuckoo Search (CS) algorithm, the Bat Algorithm (BA) and the Firefly Algorithm (FFA). They have different strengths and better performances for a certain class of optimisation problems. It is quite challenging to select the suitable or best algorithm for a specific problem, and this by itself is another optimisation problem.

Nowadays, there are trends to introduce new algorithms, extend and modify the existing algorithms, compare the performance of different algorithms, apply the algorithms in multiple disciplines for optimisation purpose and combine any two algorithms in hybridisation (Fisher *et al*., 2013). Even though the Firefly Algorithm is one of the recently introduced metaheuristic algorithms (Yang, 2009), it has been used extensively for many applications and has been modified and hybridised for different classes of optimisation problems (Apostolopoulos & Vlachos, 2011; Basu & Mahanti, 2011; Gandomi *et. al*, 2013; Tilahun & Ong, 2013). In this study, we compared the performances of the Standard (FFA) algorithm, the Modified Firefly Algorithm (MFFA) and the Chaotic Firefly Algorithms (CFFAs) based on eight chosen test

functions in terms of their accuracy and effectiveness. Three chaotic maps, namely, the Iterative map, the Chebyshev map and the Sinusodial map for the Chaotic Firefly Algorithms, were chosen based on their statistical results and success rate, which can improve the reliability of the global optimality and enhance the quality of the results (Gandomi *et al*., 2013).In the chaotic map, we varied the parameters, β (the attractiveness coefficient) and γ (light absorption coefficient). If γ is too small and tends to zero, attractiveness becomes constant; and if γ is too large, attractiveness will decrease dramatically, as will be discussed later. Therefore we needed to tune the γ parameter used for all the Firefly Algorithms tested within these two extreme γ values.

BASIC CONCEPTS

Optimisation Problem

Optimisation is a mathematical method to find a maximum or minimum value of an objective function *f(x)* by choosing a variable from a feasible region, Ω. x^* is said to be a solution for a minimisation problem if and only if $x^* \in \Omega$ and $f(x^*) \leq f(x)$, $\forall \in \Omega$. We can express the typical minimisation problem in equation (1) and its equivalent maximisation problem in equation (2).

$$\min_{x \in \mathbb{R}^n} f(x)$$

$$\text{s.t.} \quad x \in \Omega \subseteq \mathbb{R}^d \tag{1}$$

Hence $f(x^*) \leq f(x)$ for all $x \in \Omega$

$$\max_{x \in \mathbb{R}^n} -f(x)$$

$$\text{s.t.} \quad x \in \Omega \subseteq \mathbb{R}^d \tag{2}$$

Hence $f(x^*) \geq f(x)$ for all $x \in \Omega$

Optimisation algorithms are useful tools for different applications including experimental design, parameter estimation, model development and statistical analysis; process synthesis, analysis, design and retrofit; model predictive control and real-time optimisation. They are also useful in the planning, scheduling and integration of process operations into the supply chain for manufacturing and distribution (Biegler, 2010).

Standard and Modified Firefly Algorithm (FFA, MFFA)

The Modified Firefly Algorithm is an extension or improvement of the Firefly Algorithm (FFA), which is a nature-inspired algorithm that imitates the behaviour of fireflies of flashing light within their bodies to attract mating partners and potential predators (Yang, 2010).

There are three assumptions made in adapting this behaviour of fireflies for use in an algorithm:

i. All fireflies are unisex; therefore, a firefly may be attracted to any other firefly;

ii. The attractiveness of fireflies is proportional to their brightness, and for two fireflies, x_i and x_j, if the brightness of $x_j \geq$ the brightness of x_i, then x_i will move towards x_j. At the same time, attractiveness decreases as distance increases.

iii. The brightness of a firefly is affected or determined by the landscape of the objective function to be optimised

There are two important components affecting the movements of fireflies: the variation of light intensity and the formulation of the attractiveness. As mentioned above, light intensity I decreases as the distance r increases. We can express $I(r)$ as given below:

$$I(r) = I_0 \, e^{-\gamma r} \tag{3}$$

By combining this with the inverse square law, *I (r)* can be expressed as:

$$I(r) = I_0 \, e^{-\gamma r^2} \tag{4}$$

As the attractiveness is proportional to the light intensity, we now define the attractiveness β as:

$$\beta = \beta_0 \, e^{-\gamma r^2} \tag{5}$$

where β_0 is the attractiveness at r=0. Computationally, it is harder to calculate the exponential function than $1/(1+r^2)$, hence we approximate the equation of computing the attractiveness β as:

$$\beta = \frac{\beta_0}{1 + \gamma r^2} \tag{6}$$

Suppose we have a firefly *i* located at x_i as the brighter firefly while another less bright firefly *j* is at x_j; the firefly *j* will move towards the firefly *i*. The location is then updated using the process given below:

$$x_j \leftarrow x_j + \beta_0 \, e^{-\gamma r_{ij}^2} \left(x_i - x_j \right) + \alpha(rand) \tag{7}$$

where *rand* is a vector of random numbers $0 \leq \alpha \leq 1$ and $0.01 \leq \gamma \leq 100$ (Yang, 2009).

The distance between two fireflies, namely, firefly *i* and firefly *j*, r_{ij}, can be computed using Euclidean distance as:

$$r_{ij} = \| x_i - x_j \| \tag{8}$$

While the same in all the issues discussed above, the Standard and Modified Firefly Algorithms mainly differ in the updating process of the brightest firefly; instead of letting this brightest firefly, x_B, which serves as the current global best solution to move randomly, we set the movement direction for it. The purpose for doing so is that we know that if this brightest firefly moves randomly, it may move to a region where its brightness may decrease i.e. the performance of the global best solution for the algorithm may decrease. In order to do so, we generate m unit vectors, $u_1, u_2, \ldots, u_m$, and choose one in which the brightness of the current global best solution will increase if it moves in the particular direction, say U (Tilahun & Ong, 2012b). The movement direction of the brightest firefly can be expressed as below:

$$x_B \leftarrow x_B + \alpha U \qquad (9)$$

where α is the random step length and U is the unit vector chosen from the m directions. If there is no direction from the m unit vectors for the current brightest solution to move in order to increase its performance, the current brightest solution will remain at its existing position. Furthermore, unlike the Standard Firefly Algorithm, in the Modified Firefly Algorithm, β_0, which is the attractiveness at r=0, for a firefly this is not taken as 1 but can be expressed as:

$$\beta_o = \frac{{I_0}^i}{{I_0}^j} \qquad (10)$$

where I_0^i is the intensity at r=0 for firefly i while I_0^j is the intensity at r=0 for firefly j and $I_0^j \neq 0$. The modified firefly algorithm procedure is shown in Fig.1.

Chaotic Firefly Algorithm (CFFA)

Chaos is a type of unique deterministic nonlinear dynamic behaviour and it has been applied widely in communication, automation, pattern recognition and other fields. A dynamic system may be mathematically expressed either by a continuous set of equations or by a discrete system, called a map, as follows:

$$x_{k+1} = f(x_k), 0 < x_k < 1, \qquad k = 0, 1, 2 \ldots \qquad (11)$$

running in chaotic state. The chaotic sequence

$$\{ x_k : k=0,1,2\ldots \}$$

can be used as a spread-spectrum sequence and as a random number sequence. These sequences have already been proven for their easy and fast generation and storage (Heidari-Bateni & McGillem, 1992; Nguyen *et al*., 2013).

The main idea of using chaotic maps or systems in a Firefly Algorithm is to replace the random variables used in the firefly algorithm with chaotic variables so as to increase its mobility for robust global optimisation. To fulfil this purpose, we tune the parameters β and γ for the attractive movement in chaotic firefly. Tuning the parameters β and γ will affect the results of the convergence rate and the number of algorithm iterations (Arora & Singh, 2013; Yang, 2011).

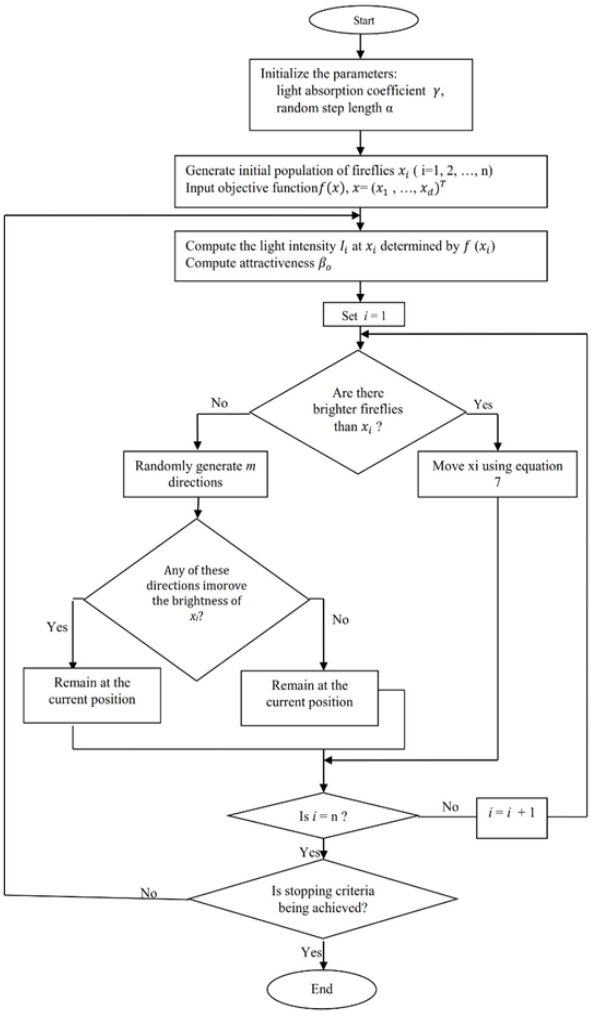

Fig.1: The Modified Firefly Algorithm Procedure

Although firefly algorithms are efficient enough in optimisation problems, in this study we noticed that the solutions kept on changing as they approached the optimum. Gandomi *et al*. (2013)suggested to tune the light absorption coefficient, γ, and the attractiveness coefficient, β, using chaotic maps so as to increase the mobility of the solution in the algorithms. The chaotic Firefly Algorithm procedure is shown in Fig.2.

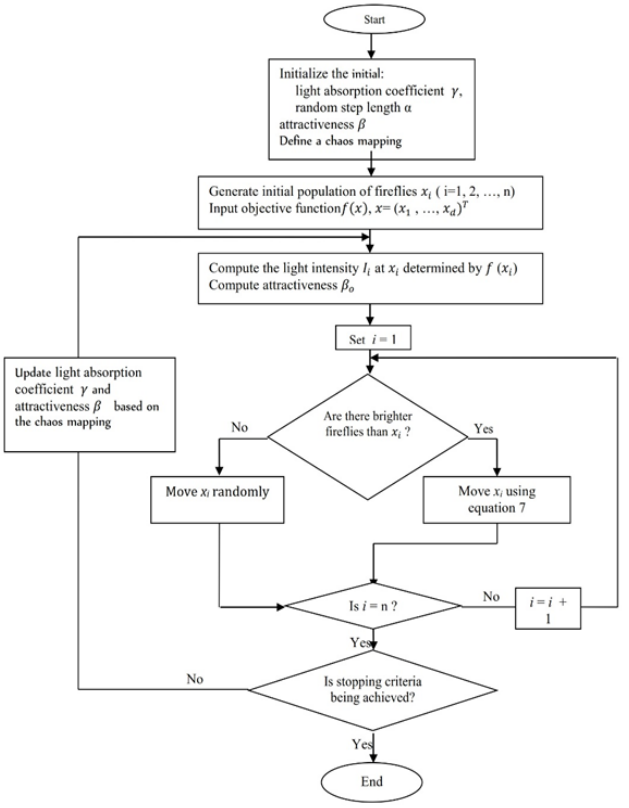

Fig.2: The Chaotic Firefly Algorithm Procedure

Chaotic solutions are affected by three properties: sensitivity of parameters, sensitivity of initial points and randomness (Sharkovsky *et al*., 1997). For the sensitivity of parameters, slight changes in the parameters or initial values for the data will lead to vastly different future behaviour. On sensitivity of initial points, we can see that if an initial point x_0 varies slightly, two sequences found from repeated calculations on a chaotic map with a parameter finally become quite different. For randomness, solutions starting from almost all x_0 in [0, 1] wander in [0, 1] as the random number is taken from a uniform distribution.

The characteristic of non-repetition of chaos enables the algorithm to carry out overall searches at a higher speed than stochastic ergodic searches, which depend on probabilities. A random local search may cause the algorithm to be easily trapped in the local minima but Chaotic maps are useful in helping the algorithm to escape this condition.

One-dimensional maps are the simplest systems with the capability of computing a chaotic process (Ram, 2009).In this study, we choose three chaotic maps that have good performance in terms of success rate and statistical analysis which was studied by Gandomi *et al*. (2013).The maps chosen are the Iterative map, the Chebyshev map and the Sinusodial map. Gandomi *et al*. (2013) suggested to normalise the chaotic maps between 0 and 2 to produce the simulation results. We embedded these maps into the firefly algorithms for it to become Chaotic Firefly Algorithms (CFFA).

The three chosen chaotic maps were one-dimensional and non-invertible maps. The first map was an Iterative map. It is a mapping function that maps a region back onto itself and is defined as below:

$$x_{k+1} = \sin\left(\frac{a\pi}{x_k}\right) \tag{12}$$

where $a \in (0, 1)$ is the suggested range for the parameter. In this study, we used a=0.5 for all the firefly algorithms being tested.

Secondly we had a Chebyshev map, which is a typical chaotic map called an identity map, defined as:

$$x_{k+1} = \cos\left(k\cos^{-1}(x_k)\right) \tag{13}$$

The third chaotic map we tested was a Sinusodial map. It is defined as below:

$$x_{k+1} = a{x_k}^2 \sin\left(\pi x_k\right) \tag{14}$$

Gandomi *et al*. (2013) suggested to use a=2.3 and x_0=0.7, which gave us a simplification of the equation as:

$$x_{k+1} = \sin\left(\pi x_k\right) \tag{15}$$

In Gandomi *et al*. (2013), the success rate for evaluating the performance of the algorithms can be calculated using this equation:

$$S_r = 100 \times \frac{N_{successf\ ul}}{N_{all}} \quad (16)$$

where N_{all} is the number of all trials, $N_{successful}$ is the number of trials in which is found a successful solution. Here, a run in which the results are close to the global optimum would be considered a successful run. A successful run can be expressed as:

$$\|x^{gb} - x^*\| \leq (UB - LB) \times 10^{-4} \quad (17)$$

where x^{gb} is the global best obtained by the proposed algorithms and *UB* is the upper bound while LB is the lower bound of the variable tested.

TEST FUNCTIONS

Standard benchmarks or test functions are useful and important in evaluating the reliability, efficiency and validation of optimisation algorithms. The test functions can be categorised according to their types of continuity, modality and dimensionality. On the continuity of test functions, we used both continuous and discontinuous functions. A function *f(x)* is said to be continuous at a point *c* if the limit of the function as *x* approaches *c* is the same as the functional value of the function at *x*=*c*. If this property is not fulfilled at any point *x*=*c*, the function *f(x)* is said to be discontinuous at *c*. For the modalities of test functions, we used both unimodal and multimodal functions.

A unimodal function has only one optimum while a multimodal function may have many local optima and global optima. Multimodal functions are useful in testing the ability of optimisation algorithms to escape from a local minimum. In this study, we chose eight test functions under different categories of continuity, modality and dimensionality. One of the selected test functions was a four-dimensional objective function while the rest of the functions chosen were two-dimensional.

Beale Function

The Beale Function is a continuous and unimodal function with sharp peaks at the corners of the input domain. It is a two-dimensional function.The function is defined as:

$$f_1(x)=(1.5 - x_1+ x_1 x_2)^2+(2.25 - x_1+x_1 x_2^2)^2+(2.625-x_1+x_1 x_2^3)^2)$$

The variables x_1 and x_2 are both defined on the interval [-4.5, 4.5]. It is unimodal and contains only one optimum i.e. a global minimum at $(x_1,x_2)=(3, 0.5)$ giving the value of $f_1(x)=0$ (Momin & Yang, 2013). Figure 3 shows the graph of $f_1(x)$.

Leon Function

The Leon Function is a continuous and unimodal function. It is a two-dimensional function. The function is defined as:

$$f_2(x)=100(x_2-x_1^2)^2+(1-x_1)^2$$

The variables x_1 and x_2 are both defined on the interval [-1.2, 1.2]. The global minimum of the Leon Function at $(x_1,x_2)= (1, 1)$ gives the value of $f_2(x) = 0$ (Momin & Yang, 2013). Figure 4 shows the graph of $f_2(x)$ plotted for two-dimensional graph.

Matyas Function

The Matyas Function is a continuous and unimodal function. It is a two-dimensional function. The function is defined as:

$$f_3(x)=0.26(x_1^2+x_2^2)+0.48x_1 x_2$$

The variables x_1 and x_2 are both defined on the interval [-10, 10]. The global minimum of the Matyas function at $(x_1,x_2)= (0, 0)$ gives the value of $f_3(x) = 0$ (Momin & Yang, 2013). Figure 5 shows the graph of $f_3(x)$ plotted for a two-dimensional graph.

Goldstein Price Function

Goldstein Price Function is a continuous and multimodal function. It is a two-dimensional function.The function is defined as:

$$f_4(x)=[1+(x_1+x_2+1)^2(19-14x_1+3x_1^2-14x_2+6x_1x_2+3x_2^2)]\times [30+(2x_1-3x_2)^2(18-32x_1+12x_1^2+48x_2-36x_1x_2+27x_2^2)]$$

The variables x_1 and x_2 are both defined on the interval [-2, 2]. The global minimum of the Goldstein Price function at $(x_1,x_2)= (0, -1)$ gives the value of $f_4(x) = 3$ (Schonlau, 1997). Not far from the global minimum, there are another three local minima. Therefore, it is said to be a difficult problem for minimisation methods due to its having more than one local minimum. Figure 6 shows the graph of $f_4(x)$.

Hosaki Function

The Hosaki Function is a continuous and multimodal (bimodal) function. It is a two-dimensional function.The function is defined as below:

$$f_5(x) = (1 - 8x_1 + 7x_1^2 - \frac{7}{3}x_1^3 + \frac{1}{4}x_1^4)(x_2^2 e^{-x_2})$$

The variable x_1 is defined on the interval [0, 5] while x_2 is defined on the interval [0, 6]. The global minimum of the Hosaki function at $(x_1,x_2)= (4, 2)$ and the local minimum of the Hosaki function at $(x_1,x_2)= (1, 2)$ give the value of $f_5(x) = -2.3458$ (Duan *et al*., 1992). Figure (7) shows the graph of $f_5(x)$.

Price 1 Function

The Price 1 Function is a continuous and multimodal function. It is a two-dimensional function. The function is defined as:

$$f_6(x)=(|x_1|-5)^2+(|x^2|-5)^2$$

The variables x_1 and x_2 are both defined on the interval [-500, 500]. There are four global minima of the Price 1 Function at (x_1,x_2)= {(-5, -5), (-5, 5), (5, -5), (5, 5)} which give the value of $f_6(x) = 0$ (Price, 1976). Figure 8 shows the graph of $f_6(x)$.

Bird Function

The Bird Function is a continuous and multimodal function. It is a two-dimensional function. The function is defined as:

$$f_7(x) = \sin(x_1)e^{(1-\cos(x_2))^2} + \cos(x_2)e^{(1-\sin(x_1))^2} + (x_1 - x_2)^2$$

The variables x_1 and x_2 are both defined on the interval $[-2\pi, 2\pi]$. The global minimum of the Bird Function at (x_1,x_2)= (4.70104, 3.15294) and (-1.58214, -3.13024) gives the value of $f_7(x) = -106.764537$ (Momin & Yang, 2013). Figure 9 shows the graph of $f_7(x)$ plotted for a two-dimensional graph.

Cosine Mixture Function

The Cosine Mixture Function is a discontinuous and multimodal function. This function can be either in two or four dimensions.The function is defined as:

$$f_8(x) = -0.1\left[\sum_{i=1}^{n}\cos(5\pi x_i) - \sum_{i=1}^{n} x_i^2\right]$$

The variable x_1 is defined on the interval [-1, 1]. In this study, we fixed the number of dimensions to four. The minimum of the Cosine Mixture function is found at $x_1 = 0$ to give the values of $f_8(x) = -0.2$ and $f_8(x) = -0.4$ for the two-dimensional and four-dimensional cases respectively. (Momin & Yang, 2013). Figure 10 shows the graph of $f_8(x)$ plotted for the two-dimensional graph.

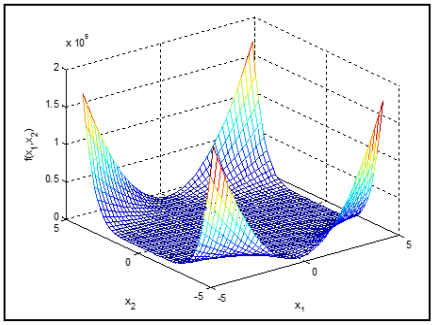

Fig.3: Beale Function

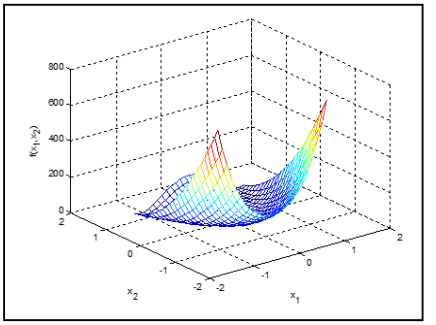

Fig.4: Leon Function

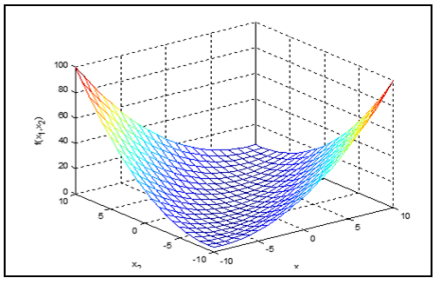

Fig.5: 5 Matyas Function

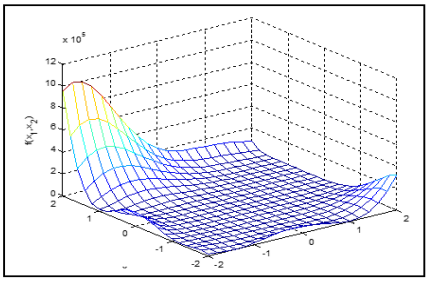

Fig.6: Goldstein Price Function

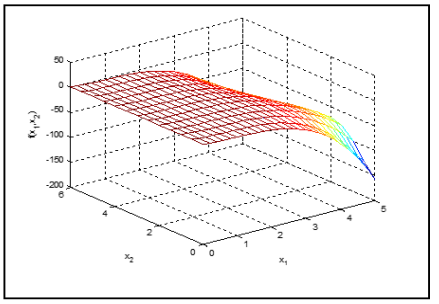

Fig.7: Hosaki Function

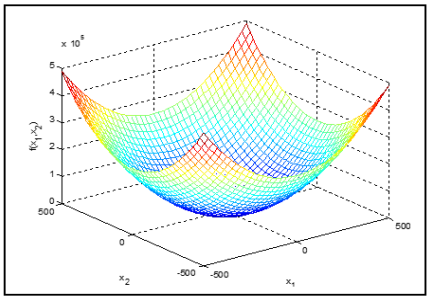

Fig.8: Price 1 Function

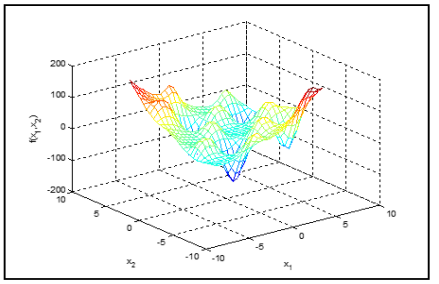

Fig.9: Bird Function

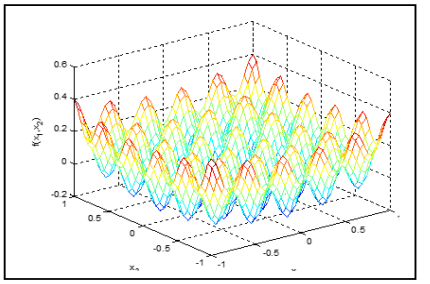

Fig.10: Cosine Mixture

TABLE 1 : Simulation Results of the 5 Different Firefly Algorithms Tested (j=1, 2, …, 5) on 8 Chosen Test Functions (i=1, 2, …, 8) Based on Maximum Number of Iterations as the Termination Criterion

Test Function	Test Result	Metaheuristic Optimisation Algorithms														
		FFA			MFFA			CFFA1			CFFA2			CFFA3		
		n=50	n=100	n=150	n=50	n=100	n=150	n=50	n=100	n=150	n=50	n=100	n=150	n=50	n=100	n=150
$f_1(x)$ $\min f(x)=0$	$\hat{\mu}_{1jk}$	0.0168	***0.0152***	0.0125	***0.0032***	0.0025	0.0022	0.0181	***0.0131***	0.0111	0.0197	0.0177	0.0122	0.0147	***0.0129***	0.0109
	$\hat{\delta}_{\beta 1jk}$	0.0173	0.0153	0.0137	0.0040	0.0025	0.0021	0.0203	0.0142	0.0146	0.0233	0.0196	0.0121	0.0167	0.0148	0.0132
	CPU_{1jk}	0.7413	2.8330	6.0502	0.7738	2.8675	6.1215	0.7655	2.8509	6.0539	0.7735	2.8568	6.1057	0.7279	2.7826	5.9865
	σ_{CPU1jk}	0.0975	0.2309	0.3284	0.1081	0.2613	0.3491	0.1113	0.2790	0.3086	0.1220	0.2317	0.3288	0.0961	0.1752	0.3503
$f_2(x)$ $\min f(x)=0$	$\hat{\mu}_{2jk}$	***0.0007***	***0.0005***	***0.0004***	***0.0000***	0.0000	0.0000	0.0009	0.0006	0.0004	0.0070	0.0055	0.0046	0.0038	0.0033	0.0022
	$\hat{\delta}_{\beta 2jk}$	0.0007	0.0006	0.0005	0.0000	0.0000	0.0000	0.0009	0.0006	0.0004	0.0051	0.0043	0.0038	0.0037	0.0039	0.0027
	CPU_{2jk}	0.6363	2.5177	5.7592	0.6593	2.5399	5.7683	0.6407	2.5119	5.7572	0.6472	2.5475	5.8246	0.6351	2.5102	5.7188
	σ_{CPU2jk}	0.0196	0.0892	0.1879	0.0329	0.1211	0.1995	0.0276	0.0701	0.2686	0.0322	0.0759	0.2393	0.0171	0.0683	0.1747
$f_3(x)$ $\min f(x)=0$	$\hat{\mu}_{3jk}$	0.0012	0.0008	0.0006	***0.0001***	0.0001	0.0001	0.0011	0.0007	0.0007	0.0007	0.0006	0.0006	0.0010	0.0009	0.0008
	$\hat{\delta}_{\beta 3jk}$	0.0013	***0.0008***	0.0006	0.0001	0.0001	0.0001	0.0010	***0.0007***	0.0006	0.0007	***0.0006***	0.0005	0.0010	0.0010	0.0008
	CPU_{3jk}	0.6229	2.4016	5.8260	0.6263	2.4040	5.8936	0.6198	2.4110	5.9596	0.6185	2.3873	5.9198	0.6126	2.3594	5.7717
	σ_{CPU3jk}	0.0390	0.0638	0.2689	0.0299	0.0750	0.3292	0.0372	0.0811	0.4025	0.0349	0.0482	0.4616	0.0350	0.0508	0.3422
$f_4(x)$ $\min f(x)=3$	$\hat{\mu}_{4jk}$	3.3556	***3.3074***	3.2528	***3.0262***	3.0256	3.0211	3.1999	***3.1805***	3.1405	3.6163	3.5624	3.4656	3.6351	3.5980	3.4614
	$\hat{\delta}_{\beta 4jk}$	0.3710	0.2561	0.3861	0.0227	0.0233	0.0179	0.2232	0.1974	0.1212	0.6187	0.5688	0.4166	0.6445	0.5464	0.4766
	CPU_{4jk}	0.7922	2.8402	5.6012	0.8139	2.8561	5.6195	0.7930	2.8319	5.6291	0.7972	2.8469	5.6430	0.7834	2.7955	5.6207
	σ_{CPU4jk}	0.0476	0.3582	0.1263	0.0354	0.3621	0.1246	0.0355	0.3588	0.0883	0.0492	0.3980	0.1022	0.0397	0.3515	0.2423
$f_5(x)$ $\min f(x)=-2.3458$	$\hat{\mu}_{5jk}$	-2.3437	***-2.3439***	-2.3440	***-2.3455***	-2.3455	-2.3455	-2.3443	***-2.3447***	-2.3447	-2.3437	***-2.3443***	-2.3446	-2.3435	-2.3436	-2.3438
	$\hat{\delta}_{\beta 5jk}$	0.0019	0.0018	0.0019	0.0002	0.0003	0.0002	0.0014	0.0013	0.0010	0.0019	0.0015	0.0012	0.0025	0.0019	0.0020
	CPU_{5jk}	0.6867	2.6726	5.8906	0.7036	2.7082	5.9441	0.6830	2.6885	5.9463	0.6973	2.7247	5.9349	0.6799	2.6508	5.8383
	σ_{CPU5jk}	0.0327	0.0466	0.1512	0.0245	0.0619	0.1970	0.0221	0.0688	0.2731	0.0251	0.0972	0.2566	0.0215	0.0572	0.1675
$f_6(x)$ $\min f(x)=0$	$\hat{\mu}_{6jk}$	0.8153	***0.3164***	0.2142	***0.0014***	0.0014	0.0014	0.8943	***0.2840***	0.2230	0.7857	***0.2927***	0.2237	0.7911	0.3174	0.1936
	$\hat{\delta}_{\beta 6jk}$	0.8663	0.3447	0.1845	0.0014	0.0015	0.0013	0.8022	0.2764	0.2098	0.8092	0.2673	0.2364	0.8558	0.2770	0.1880
	CPU_{6jk}	0.7346	2.7748	5.3622	0.7493	2.8104	5.3987	0.7413	2.8283	5.3546	0.7486	2.8001	5.3480	0.7423	2.8138	5.3611
	σ_{CPU6jk}	0.0696	0.7754	0.1722	0.0774	0.8176	0.1803	0.0772	0.8563	0.1808	0.0801	0.8563	0.1912	0.0730	0.8446	0.1809
$f_7(x)$ $\min f(x)=-106.7645$	$\hat{\mu}_{7jk}$	-106.4223	***-106.4639***	-106.5784	***-106.7020***	-106.7075	-106.7165	-106.4271	***-106.5059***	-106.5596	-106.4044	***-106.5120***	-106.5237	-106.4653	***-106.5546***	-106.5547
	$\hat{\delta}_{\beta 7jk}$	0.3314	0.3352	0.2073	0.0898	0.0813	0.0648	0.3900	0.2514	0.2101	0.3773	0.2691	0.2651	0.2983	0.3172	0.2334
	CPU_{7jk}	0.7185	2.6916	6.3377	0.7296	2.7364	6.3970	0.7181	2.7246	6.3762	0.7176	2.7016	6.4642	0.7065	2.6523	6.2098
	σ_{CPU7jk}	0.0584	0.1894	0.6835	0.0574	0.2010	0.6793	0.0644	0.2403	0.6441	0.0612	0.1706	0.6374	0.0636	0.1947	0.5772
$f_8(x)$ $\min f(x)=-0.4$	$\hat{\mu}_{8jk}$	-0.2457	***-0.2622***	-0.2638	***-0.3346***	-0.3378	-0.3381	-0.2678	***-0.2689***	-0.2744	-0.2486	-0.2553	-0.2622	-0.2499	-0.2537	-0.2708
	$\hat{\delta}_{\beta 8jk}$	0.0447	0.0465	0.0418	0.0163	0.0169	0.0185	0.0487	0.0412	0.0446	0.0480	0.0442	0.0425	0.0475	0.0456	0.0511
	CPU_{8jk}	0.4636	1.9318	4.0674	0.4750	1.9157	4.0551	0.4647	1.9154	4.0996	0.4650	1.9256	4.0701	0.4585	1.8807	4.0223
	σ_{CPU8jk}	0.0083	0.0086	0.0070	0.0108	0.0173	0.0135	0.0087	0.0069	0.0081	0.0082	0.0076	0.0061	0.0088	0.0078	0.0065

** $\hat{\mu}_{ijk}$ is the mean functional value generated from 100 trials; $\hat{\sigma}_{\mu 1jk}$ is the standard deviation of the mean functional value from 100 trials; h_{ijk} is the hypothesised value accepted at 95% confidence interval; CPU_{ijk} is the mean CPU time needed for each trial; i is the i^{th} test function; j is the j^{th} algorithm and k is the number of solutions set (n=50, n=100 and n=150).

** FFA (Firefly Algorithm), MFFA (Modified Firefly Algorithm), CFFA1 (Chaotic Firefly Algorithm with the Iterative Map), CFFA2 (Chaotic Firefly Algorithm with the Chebyshev Map) and CFFA3 (Chaotic Firefly Algorithm with the Sinusodial Map)

TABLE 2: Simulation Results of the 5 Different Firefly Algorithms Tested (j=1, 2, …, 5) on 8 Chosen Test Functions (i=1, 2, …,8) Based on Tolerance (tol) as the Termination Criterion

Test Function	Test Result	Metaheuristic Optimisation Algorithms														
		FFA			MFFA			CFFA1			CFFA2			CFFA3		
		n=50	n=100	n=150	n=50	n=100	n=150	n=50	n=100	n=150	n=50	n=100	n=150	n=50	n=100	n=150
$f_1(x)$ $\min f(x)=0$ *tol*=0.01	Itr_{1jk}	42.9700	34.3700	***26.1400***	35.1300	24.6400	20.3600	44.1100	36.2500	27.2500	41.2300	31.4600	***23.6200***	54.2000	33.1000	***23.3800***
	δ_{Itr1jk}	35.8195	32.4332	22.4563	31.0401	25.0613	23.8965	36.7706	28.6633	25.0438	35.8493	32.5953	24.6989	50.2177	32.2908	22.2083
	CPU_{1jk}	0.2288	0.6730	1.0800	0.1961	0.4781	0.8516	0.2300	0.7200	1.1228	0.2153	0.6216	0.9730	0.2742	0.6305	0.9470
	σ_{CPU1jk}	0.2048	0.6613	0.9251	0.1789	0.4780	0.9923	0.1896	0.5866	0.9510	0.2006	0.6875	1.0160	0.2450	0.6086	0.8983
$f_2(x)$ $\min f(x)=0$ *tol*=0.001	Itr_{2jk}	27.8100	14.0800	***9.1700***	13.2200	8.9800	6.7200	22.6300	11.3400	***8.7200***	15.4900	9.6800	***7.4800***	28.3100	18.5900	15.3700
	δ_{Itr2jk}	20.0890	9.5205	7.1096	9.5278	6.0753	4.4383	16.7794	8.3014	5.3069	10.6747	6.8812	5.4300	20.4735	12.3387	12.4411
	CPU_{2jk}	0.1306	0.3095	0.4019	0.0697	0.1928	0.3019	0.1069	0.2419	0.3856	0.0739	0.2145	0.3292	0.1344	0.4102	0.6859
	σ_{CPU2jk}	0.0951	0.2154	0.3041	0.0479	0.1219	0.2031	0.0796	0.1727	0.2555	0.0505	0.1660	0.2582	0.0971	0.2802	0.5901
$f_3(x)$ $\min f(x)=0$ *tol*=0.001	Itr_{3jk}	38.9400	23.1800	***22.7100***	18.2400	15.9200	15.6900	31.2500	21.7200	***18.1800***	35.6600	27.2400	22.6500	36.9100	24.8600	***21.8400***
	δ_{Itr3jk}	37.1323	21.4478	25.1803	16.6976	13.6394	15.0861	23.6418	17.6756	15.4961	30.8326	21.6338	20.9388	31.5191	24.0353	19.2510
	CPU_{3jk}	0.1923	0.4797	0.9433	0.0970	0.3236	0.6628	0.1580	0.4536	0.7741	0.1798	0.5509	0.9530	0.1839	0.5022	0.9106
	σ_{CPU3jk}	0.1802	0.4605	1.0402	0.0835	0.2623	0.6412	0.1242	0.3922	0.6814	0.1556	0.4565	0.8825	0.1770	0.4873	0.8623
$f_4(x)$ $\min f(x)=3$ *tol*=3.1	Itr_{4jk}	52.1800	39.9400	***34.4900***	18.5200	17.1800	16.6000	44.3900	28.7200	***18.6000***	43.4700	25.3700	***24.5200***	56.2500	42.7800	***34.2300***
	δ_{Itr4jk}	39.1058	34.9617	33.3544	15.1427	14.8633	16.1627	32.1121	22.1547	14.8565	36.1203	20.2388	24.2681	42.5166	38.4281	29.8558
	CPU_{4jk}	0.2528	0.7738	3.2248	0.0983	0.3359	1.5286	0.2184	0.5523	1.7123	0.2117	0.4916	2.2916	0.2706	0.8070	3.2697
	σ_{CPU4jk}	0.1898	0.6814	3.4070	0.0797	0.2844	1.5094	0.1573	0.4302	1.4172	0.1793	0.3929	2.3343	0.2044	0.7120	3.1594
$f_5(x)$ $\min f(x)=-2.3458$ *tol*=-2.34	Itr_{5jk}	30.5500	18.2500	***17.6300***	16.5700	13.7600	13.7500	30.5100	25.5200	20.7500	27.4900	21.3700	***17.2800***	35.1800	27.4300	21.3000
	δ_{Itr5jk}	31.4843	18.6463	15.7606	16.3702	14.7868	15.3783	30.6223	24.9968	20.4551	24.8277	22.9680	15.2256	35.1353	28.9766	23.1676
	CPU_{5jk}	0.1452	0.3373	0.7231	0.0839	0.2550	0.5753	0.1444	0.4705	0.8525	0.1322	0.3909	0.7073	0.1645	0.4952	0.8564
	σ_{CPU5jk}	0.1486	0.3442	0.6471	0.0788	0.2719	0.6362	0.1468	0.4561	0.8404	0.1191	0.4192	0.6233	0.1659	0.5254	0.9304
$f_6(x)$ $\min f(x)=0$ *tol*=0.1	Itr_{6jk}	***51.9800***	35.5000	29.3900	***38.3900***	25.1100	21.1000	***60.7300***	41.3700	32.4500	***55.2500***	42.6100	34.5000	***54.2300***	42.8000	42.5000
	δ_{Itr6jk}	37.4009	30.1617	26.3848	20.9212	15.5660	11.8709	35.4190	32.2423	27.9252	37.8944	34.3529	35.6780	32.9284	33.1083	38.7487
	CPU_{6jk}	0.2466	0.6714	1.2195	0.1881	0.4883	0.8773	0.2841	0.7995	1.3519	0.2628	0.8234	1.4228	0.2553	0.8063	1.7316
	σ_{CPU6jk}	0.1745	0.5677	1.0727	0.0970	0.3113	0.5132	0.1644	0.6277	1.1821	0.1799	0.6838	1.4853	0.1570	0.6227	1.5657
$f_7(x)$ $\min f(x)=-106.7645$ *tol*=-106.5	Itr_{7jk}	22.9400	18.5600	***12.8100***	14.5400	12.0300	11.0000	22.5300	16.5600	13.9900	22.6700	15.6300	***12.3800***	29.3800	28.3400	20.8400
	δ_{Itr7jk}	17.6840	17.3224	9.7636	11.2586	10.9622	9.7203	21.4963	11.4224	12.5927	16.1508	12.0191	10.3872	27.8375	24.8125	17.6164
	CPU_{7jk}	0.1161	0.3513	0.5316	0.0822	0.2316	0.4625	0.1178	0.3127	0.5784	0.1144	0.2963	0.5166	0.1469	0.5400	0.8570
	σ_{CPU7jk}	0.0900	0.3263	0.4046	0.0641	0.2092	0.4072	0.1228	0.2143	0.5176	0.0832	0.2257	0.4331	0.1391	0.4765	0.7271
$f_8(x)$ $\min f(x)=-0.4$ *tol*=-0.3	Itr_{8jk}	72.0300	62.5200	***52.4400***	34.5100	31.4500	27.3600	55.7200	54.2500	***46.2300***	69.0100	62.5100	***48.9900***	54.2400	39.7200	***38.4100***
	δ_{Itr8jk}	77.0973	69.0998	55.5391	23.9636	32.4571	29.3779	49.9345	43.1603	34.5773	52.6666	58.1844	48.3534	46.5182	42.2130	43.2428
	CPU_{8jk}	0.3959	1.3702	2.5859	0.1973	0.6877	1.3323	0.3061	1.1823	2.2256	0.3816	1.3411	2.3911	0.2913	0.8586	1.8223
	σ_{CPU8jk}	0.4262	1.5138	2.7758	0.1317	0.7012	1.4088	0.2795	0.9388	1.6506	0.2912	1.2396	2.3707	0.2502	0.9521	2.0178

** Itr_{ijk} is the mean number of iterations needed for convergence in each trial; CPU_{ijk} is the mean CPU time needed for each trial; i is the i^{th} test function; j is the j^{th} algorithms and k is the number of solutions set (n=50, n=100 and n=150).

** FFA (Firefly Algorithm), MFFA (Modified Firefly Algorithm), CFFA1 (Chaotic Firefly Algorithm with the Iterative Map), CFFA2 (Chaotic Firefly Algorithm with the Chebyshev Map) and CFFA3 (Chaotic Firefly Algorithm with the Sinusodial Map)

SIMULATION RESULTS

We carried out tests for the eight functions for the algorithms under two termination criteria, which are the maximum number of iterations and pre-given tolerance. The maximum number of iterations was set as 200 iterations and was tested at 100 trials, with the same random initial solution passing to the entire algorithm in each trial. We recorded the mean functional values and their standard deviations as well to examine the accuracy of the tested functions on different algorithms. At the same time, we compared the mean CPU time needed per trial on the FFA, MFFA and CFFA. Using the second termination criterion, we set a tolerance which was close to the global minimum value. We got the mean number of iterations needed for convergence when the termination criterion was met and the mean CPU time needed per trial for the algorithms respectively. For both tests, we set the number of solutions at 50, 100 and 150 to see whether the number of solutions affected the simulation results. The simulation results for the first test are shown in Table 1 while the simulation results for the second test are shown in Table 2.

Under the maximum number of iterations as termination criteria, the number of iterations for the inner loop was set at 200 and these were tested 100 times repeatedly in the while loop until the best solution (optimal value) was obtained. The results in Table 1 show the sample mean, sample standard deviation and sample mean CPU time for eight test problems *(i*) under five algorithms (*j)* in three sets of numbers of fireflies (*k*). Under these criteria, the accuracy of the algorithms can be compared.

In the first test, for the mean functional values, MFFA, outperformed the other algorithms for all eight test functions i.e. the functional values were the closest to the global minimum values. For instance, when n=50, MFFA outperformed the other algorithms (shown in bold and italics) since its sample mean optimal value was the nearest to the established optimum in all the eight test functions carried out where μ_{121}=0.0032, μ_{221}=0.0000, μ_{321}=0.0001, μ_{421}=3.0262, μ_{521}=-2.3455$\mu\sigma$621=0.0014, μ_{721}=-106.7020 and μ_{821}=-0.3346. We noticed that the MFFA achieved the highest accuracy. Besides that, we also observed that at least one of the CFFA (when we see the three chaotic mappings as a group) performed better than the FFA. For instance, when n =100, 7 out of 8 test problems in the CFFAs performed better than FFA (shown in bold and italics) where μ_{132}=0.0131 and μ_{152}=0.0129 compared to μ_{122}=0.0152;μ_{332}=0.0007and μ_{342}=0.0006 compared to μ_{312}=0.0008; $\mu_{\_432}$=3.1805 compared to μ_{412}=3.3074; μ_{532}=-2.3447 and μ_{542}=-2.3443 compared to μ_{512}=-2.3439; μ_{632}=0.2840 and μ_{642}=0.2927compared to μ_{612}=0.3164; μ_{732}=-106.5059, μ_{742}=-106.5120 and μ_{752}=-106.5546 compared to μ_{712}=-106.4639; μ_{832}=-0.2689 compared to μ_{812}=-0.2622.

Besides comparing the sample mean optimal value, we can also compared the sample standard deviation for the sample mean value obtained from the 8 test functions. Overall, the sample standard deviation for the MFFA was the smallest among all the five algorithms tested regardless of the number of fireflies set. It indicated that the results from the MFFA were more constant and stable. On the other hand, the performances of the sample standard deviations and CPU times of the FFA and the CFFA depended on the performance of their sample means respectively. For instance (as underlined), $\sigma\mu_{121}$ = 0.0040, $\sigma\mu_{221}$ = 0.0000, $\sigma\mu_{321}$ = 0.0001, $\sigma\mu_{421}$= 0.0227, $\sigma\mu_{521}$= 0.0002, $\sigma\mu_{621}$ = 0.0014, $\sigma\mu_{721}$ = 0.0898 and $\sigma\mu_{821}$ = 0.0163 for n=50; $\sigma\mu_{122}$)=0.0025, $\sigma\mu_{222}$= 0.0000, $\sigma\mu_{322}$ = 0.0001, $\sigma\mu_{422}$ = 0.0233, $\sigma\mu_{522}$ = 0.0003, $\sigma\mu_{622}$ =0.0015,

$\sigma\mu_{722}$)=0.0813 and $\sigma\_(\mu_{822})$ = 0.0169 for n=100; and $\sigma\mu_{123}$ =0.0021, $\sigma\mu_{223}$=0.0000, $\sigma\mu_{323}$=0.0001, $\sigma\mu_{423}$ =0.0179, $\sigma\mu_{523}$ =0.0002, $\sigma\mu_{623}$)=0.0013, $\sigma\mu_{723}$ =0.0648 and $\sigma\mu_{823}$ =0.0185 for n=150. A low standard deviation obtained in MFFA indicated that the optimal sample mean of the optimal solutions obtained for each trial was distributed very close to the optimal sample mean of the optimal solutions i.e. the variation between the means was rather small.

Under the tolerance value accepted according to the established optimum of the eight test problems as termination criteria, the number of iterations for the inner loop was set at 10000 number of times and these iterations were tested 100 times repeatedly until a termination criterion, which achieved a very close solution to the actual optimal solution based on the tolerance given, was obtained.The results in Table 2 show the number of iterations needed for the convergence in each trial with its sample standard deviation and the mean CPU time needed for each trial and its sample standard deviation. Under these criteria, the effectiveness of the algorithms can be compared.

In the second test, for the mean number of iterations, the MFFA also outperformed the other algorithms for all the eight test functions i.e. it converged faster than others and had the least number of iterations. For instance, under f_6(shown in bold and italics), when n=50, Itr_{621}=38.3900 compared to Itr_{611}=51.9800, Itr_{631}=60.7300, Itr_{641}=55.2500 and Itr_{651}=54.2300. As was the situation for the first test, there was at least one CFFA (when we saw the three chaotic mappings as a group) converged faster than the FFA. For instance, when n=150, 7 out of 8 test problems in the CFFAs performed better than the FFA (shown in bold and italics) where Itr_{143}=23.6200 and Itr_{153}=23.3800 compared to Itr_{113}=26.1400;Itr_{233}=8.7200 and Itr_{243}=7.4800 compared to Itr_{213}=9.1700; Itr_{333}=18.1800 and Itr_{353}=21.8400 compared to Itr_{313}=22.7100; Itr_{433}=18.6000, Itr_{443}=24.5200 and Itr_{453}=34.2300 compared to Itr_{413}=34.4900;Itr_{543}=17.2800 compared to Itr_{513}=17.6300; Itr_{743}=12.3800compared to Itr_{713}=12.8100; Itr_{833}=46.2300, Itr_{843}=48.9900 and Itr_{853}=38.4100 compared to Itr_{813}=52.4400.

Furthermore, the MFFA needed the least CPU time to converge, followed by both the CFFA and the FFA simultaneously. The sample standard deviations of the number of iterations and the CPU times for convergence depended on the performance of the number of iterations spontaneously. We noticed that the sample standard deviation for MFFA was the least and its CPU time was the least as well since the MFFA had the smallest number of iterations needed to reach the convergence criteria. For instance, under f_7 (as underlined), Itr_{722}=12.0300 at σItr_{722}=10.9622 with CPU_{722}=0.2316 (MFFA) when n = 100 has the least number of iterations, standard deviation and CPU time if compared to the results obtained in the FFA and the CFFAs.

Most of the time, when the number of fireflies increased, the optimal values got closer to the established optimum. For instance,μ_{211}=0.0007 for n = 50 had improved to μ_{212}=0.0005 for n = 100 and then further improved toμ_{213}=0.0004 for n = 150. We could see the sample mean was approaching the established optimum when the number of fireflies increased from 50 to 100 and then to 150. This indicated that the number of fireflies had a positive relationship with the optimal sample mean value. Therefore, if we wished to get the best optimal value, what we needed to do was to increase the sample size. We also noticed that the mean number of iterations was actually affected by the tolerance value set for the functions; the smaller the difference between the global minimum value and the tolerance, the fewer the number of iterations were needed for convergence. For instance, f_6 was set at the tolerance of 0.1, f_1

was set at the tolerance of 0.01, f_3 was set at the tolerance of 0.001 where the tolerance in f_3 had the smallest different with a global minimum ($f_{3\min}$=0). When n=100, we observed that (underlined) the number of iterations decreased from Itr_{612}=35.5000 to Itr_{112}=34.3700 and then Itr_{312}=23.1800 under the FFA.

Theoretically, exploration is done using a big-step length which can take the solution away from its current neighbourhood. Both of the three versions of Firefly Algorithms have the same exploration property. However, due to the chaos representation of the algorithm parameters in the CFFA, Gandomy et. al. (2013)claimed that it was faster than the Standard Firefly Algorithm. In terms of exploitation, the MFFA has the advanatge of tracking its best and not leaving it unless a better solution is found. Unlike the MFFA, the performance of the other two versions usually fluctuates with the iterations; hence, the MFFA outperformed the other two.

CONCLUSION

This paper discusses the recent versions of Firefly Algorithms, the Chaotic Firefly Algorithm and the Modified Firefly Algorithm, along with the Standard Firefly Algorithm. The Firefly Algorithm is a metaheuristic optimisation algorithm in which randomly generated feasible solutions are assigned as fireflies with a light intensity based on the objective function. A firefly tends to follow brighter fireflies and where no brighter firefly exists, if it is the brightest one, it will move randomly. This random movement is modified in the Modified Firefly Algorithm so that the performance of the algorithm will not fluctuate through iterations and it keeps the best solution throughout the iteration, only replacing it if a better solution is found. On the other hand, by incorporating chaos in determining the values of the algorithm parameter rather than taking it as a constant number, is how the Chaos Firefly operates. Incorporating chaos helps in the fast convergence of the algorithm. In this study, these three versions of Firefly Algorithms were studied and compared based on eight selected test problems of different types. The simulation results for the Standard Firefly Algorithm, the Modified Firefly Algorithm and the Chaos Firefly Algorithm, using the three types of chaos models, suggested that the Modified Firefly Algorithm outperformed the other versions in average performance and also had a smaller standard deviation. Hence, based on the selected test problems, the MFFA was seen to be the most accurate and effective algorithm compared to the Chaos and Standard Firefly Algorithm while at least one mapping of the Chaotic Firefly Algorithm performed better than the Standard Firefly Algorithm.

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Pathway of Continuous Professional Development Among Physiotherapists: A Qualitative Study

Ayiesah Ramli* **and Marzzatul Farhana Maslan**

Physiotherapy Program, School of Rehabilitation Science, Faculty of Health Sciences, Universiti Kebangsaan Malaysia, Jalan Raja Muda Abdul Aziz, 50300 Kuala Lumpur, Malaysia

ABSTRACT

Continuous professional development (CPD) has gained prominence in the last decade to meet improved self-development and health care services among health professionals. Being practitioners serving clients in health care, therefore, necessitates the importance of the physiotherapist's participation in activities of CPD. This paper aims to identify how physiotherapists view CPD, barriers to its progress and its impact on healthcare practice. This is a qualitative study with one-to-one interview sessions involving open-ended questions to facilitate free flow of idea that are rich with information. Twenty-two physiotherapist (17 females and 5 males) participated. Four main themes were generated following analysis: (i) comprehension of what is CPD (ii) outcome of CPD (iii) barriers to undertaking CPD and (iv) strategies to improve participation in CPD. Further sub-themes were generated from the themes suggestive of physiotherapists' awareness and concerns related to CPD activities and problems encountered when embarking on CPD participation. In conclusion, physiotherapists should recognise the importance of participation in CPD activities either for self-development or to provide effective health care services. The main barrier to CPD activities that was identified was a support system that facilitates enhancement in such activities. This has major implication such as mandatory participation in CPD among staff and for managers to ensure that an effective mechanism is in place such as funding, schedule events and moral support.

Keywords: Barriers, continuous professional development, clinical competency, perception, physiotherapist, strategies

INTRODUCTION

Continuous professional development (CPD) has evolved as an important tool in improving the physiotherapist's knowledge regarding patient's health (Brown *et al*., 2002). It is a process of learning to keep up to date with current practices in health care services (French & Dowds, 2008). The vast media

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E-mail addresses:
kamalia086@yahoo.com (Ayiesah Ramli)
marzzatul@gmail.com (Marzzatul Farhana Maslan)
*Corresponding Author

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explosion has provided patients with knowledge of expansion of health care services that are of high quality. The maintenance and improvement of the health professional's knowledge, practical skill and personal qualities are necessary to meet current patient demand of good health care services (Aris *et al*., 2010).

In physiotherapy, CPD includes areas of personal and professional development that begins with undergraduate education (Yeomans, 1995). CPD activities can occur on the job through day-to-day experiences, performance reviews, journal clubs, peer discussion, in-service training, critical reading and personal reflection (Blake & Cooney, 2000). Clinical supervision, lecturing, clinical teaching, writing reports, significant incident analysis and research are identified as other CPD activities (Chartered Society of Physiotherapy, 2005). Among the many types of CPD activity, reflective practice or portfolio is the most effective way to meet CPD goals (French & Dowds, 2008).The written work during reflection can benefit physiotherapists when reflecting on their past experiences and learning from them (Sturrock & Lennie, 2012).

In most professions, activities of CPD are seen as an instrument for professional development to specialisations and maintenance of high-quality services (Cole, 2000). Similarly, in Malaysia physiotherapists are responsible to provide better management for patient care, and this was observed to be the strongest motivating factor for undertaking CPD activities. The awareness of the importance of updating knowledge, improving competency and collegial support can encourage heatlh care professionals to involve themselves in CPD activities (French & Dowds, 2008). It is necessary for managers to encourage their staff (Adanu, 2007) as governmental bodies under the Ministry of Health make it mandatory to be involved in CPD activities or as a criterion for promotion (Aris *et al*., 2010). It is believed that through participation in CPD, healthcare professionals would improve their practice and clinical reasoning when deciding priorities in patient care (Ahuja, 2011).

Since CPD in most professions is a criterion for professional development, it would be advantageous to identify among physiotherapists the reasons for their participation in CPD activities, the barriers as well as its impact on their practice. Such findings are justifiable as no previous studies have been carried out that can be used to look into the problems encountered in the local context to improve practice. These findings can also provide suggestions to higher authorities and policy makers on measures to take for improved participation in CPD activities in the future among staff.

MATERIALS AND METHODS

Design

This is a qualitative study through semi-structured interviews (Levy, 2006) providing an in-depth understanding of personal thoughts and individual experiences (Ryan *et al*., 2007). A phenomenological approach was chosen rather than the close-ended questionnaire (Bolton 2002) to understand the experiences of the physiotherapists as phenomena (Hancock, 1998). Purposive sampling was chosen to elicit the true experiences of the participants and to prevent data pollution and biases. The interview session was held in Kuala Lumpur during a workshop on "Future Direction of Physiotherapists" funded by the Ministry of Higher Education (NNP-087).

Participants

The participants involved in the study came mainly from West Malaysia, both from the academic sector (5) as well as practice -- clinical physiotherapists working in both public (12) and private hospitals (5). The workshop was held over three days. These 22 physiotherapists (17 females and 5 males) participated in a one-to-one semi-structured interview lasting between 30 and 45 minutes with10 participants interviewed on each day of the workshop. Their mean age was 43 years. English was chosen as the medium of communication during the interview sessions. A maximum variation sample was chosen to ensure maximum diversity of the sampling group as it represented the different experiences and perception of physiotherapists in the country (Gunn & Goding, 2009). The characteristics of the maximum variation sampling included physiotherapists working more than 3 years either in government or private hospitals, full-time staff as physiotherapists and representatives from both academic institutions as well as clinicians from various hospitals throughout the country.

Ethical Consideration

Permission for this study was obtained from the Ethics Committee of a government university. All of the research materials were handled exclusively by the first researcher and no names were associated with the final written materials.

Procedure of Study

A pilot semi-structured interview session was initially carried out among the interviewers to familiarise themselves with possible questions raised during the interview sessions that lasted 30 to 45 minutes. Four interviewers underwent training to ensure that the interview sessions would be consistent among the interviewers. Each interviewer was given guidelines and could leave the guidelines whenever appropriate (Cohen & Crabtree, 2006). The purpose of this pilot interview was to expose the interviewers with the interview process and to verify the interview questions. This would enable them to comprehend the issues under discussion and carry out the interviews within the available time period (Bradford, 2011). The interview questions were open-ended to facilitate free flow of ideas to generate data that was rich with information (Kurasaki, 2000).

Prior consent was obtained from the participants and an explaination was done regarding the objectives of study. Each participant was then interviewed and the interview sessions recorded on audiotape to ensure accuracy of the data collected. The interview was done the responses reached saturation, which is central to qualitative sampling. Interview sessions was done in a quiet room carried out by a trained interviewer to avoid disturbance and noise. There were 4 trained interviewers and each of them was given the task to interview only 5 to 6 participants. During the transcription process, the subject's name and other information related to the subjects was blinded by the researcher.

Data Analysis

The interview audiotapes were transcribed into words following a standardised set of typing procedure (Kurasaki, 2000). The transcript was read several times to ensure understanding of its content and the codes were assigned to the text segments. To aid in coding and retrieval of data, computer-based qualitative analysis software (CQDAS) Nvivo 9 was used. The advantage of using NVivo version 9 software is its ability to do sophisticated data coding (Levy, 2006). It was more efficient in processing the grouping of data that was further categorised (Ping, 2008). Each interview transcript was converted into rich text file and imported into Nvivo.

Validity and Reliability

To enhance reliability, the first text was coded independently by the first author and a second coder (Miles & Huberman, 1994). Codes were compared, differences in opinion were discussed, and if necessary, codes were changed. Code checking with the second coder was performed for approximately one fifth of the second text. The third and fourth texts were coded by the first author. For the text that was coded by two coders, the same codes were applied by both most of the time and a few differences were observed in the interpretation that required discussion; the differences were subsequently resolved (Schreiber, 2009). To increase the study's credibility, member validation was performed (Bryman, 2008). These were senior staff who was not involved in the the process of data collection but acted as moderator to agree on the themes generated in the findings. Following this, the participants were invited to react to the accuracy and completeness of the preliminary findings through e-mail (Chioncel *et al*., 2003) and given one week to provide feedback. This validation process did not lead to any changes.

RESULTS

Four main themes were generated following analysis: (1) understanding of CPD activities (2) outcomes of CPD (3) barriers undertaking CPD and (4) strategies to improve participation in CPD. Sub-themes were identified further under the main themes to provide broader perspective of the physiotherapist's perception of CPD activities (Table 1).

TABLE 1: Knowledge and Barriers to Continuous Professional Development Among Physiotherapists

Theme	Sub-themes	Number of participants	Percentage (%)
Understanding of CPD	The process of lifelong learning	15	31
	Keep up to date	13	27
	Towards specialisation	10	21
	Involvement in formal or informal activities	10	21
Outcome of CPD	Knowledge exploration	18	35
	Clinical competency	14	27
	Developing self-development	10	20
	Acknowledgement	9	18

TABLE 1 : (Cont.)

Barriers to undertaking CPD	Financial problems	14	32
	Demanding workload	11	26
	Staff insufficiency	11	26
	Distance	7	16
Strategies to improve participation in CPD	Organisational role	15	32
	Relevancy of CPD activities	10	22
	Cost efficient	10	22
	Well trained coordinator	11	24

Theme 1: Understanding CPD

(i) Process of lifelong learning

All the participants had a clear understanding of the term CPD. However, there were differences in definition based on individual experience of previous CPD activities. Some highlighted that CPD was a process of lifelong learning, which is a continuous process of upgrading and maintaining professional knowledge and skills towards self-development. It was regarded as a platform to gain promotion and an avenue to hold better positions. It was emphasised that the physiotherapists could improve their skills and knowledge for direct patient-care capabilities through CPD programmes.

> *CPD means on-going education so that physiotherapist can learn throughout their working life for self-development and earned paper qualification. With CPD, they can update their knowledge, and skills in preparation of giving better treatment to patient care. They can gain monetary rewards and promotion if they followed through structures CPD programs like postgraduate studies. (Participant 7)*

> *I consider CPD as any activity that involves improvement in the individual's knowledge above the basic knowledge. (Participant 6)*

(ii) Keeping up-to-date

It was perceived that through CPD, their knowledge can be brought up-to-date and upgraded to different levels, for example, knowledge on patient management, clinical skills, research development and advancement of physiotherapy practice.

> *We have to know what physiotherapists in other countries are doing to ensure we are also up-to-date with the latest trend of patient-care management through CPD activities…this information can be either obtained locally or internationally. Through self-development, attending both short courses and postgraduate studies can enhance our professional knowledge, attribute and physiotherapy practice in clinical skills or research methods. (Participant 3)*

(iii) Towards specialisation

Continuous professional development is a means of improving and gaining knowledge for areas of specialisation.

> *Every health profession needs to involve in upgraded courses related to specialisation for in-depth understanding of an area of specialisation so that they can become more confident to apply new theories or concepts. (Participant 10)*

> *CPD activities, for novices like us, include training in a specialized area, for example, musculoskeletal, spinal and critical care (ICU). (Participant 4)*

B: Types of CPD commonly carried out in physiotherapy departments

There were different types of CPD activity identified that were either formal or informal activities related to the health profession.

(i) Formal activities

Attending conferences, seminars, workshops and the structured activities carried out are considered the formal activities of CPD. These activities were carried out monthly or twice per week to provide an opportunity to share knowledge with peers following attendance at courses or conferences.

> *In our CME, the staffs which attended courses are required to share with other staff members and present what they have learnt...even though it might be just 50 % of the material that they learnt. This can even be a technique that we had previously learnt." (Participant 17)*

> *The other type of CPD in my department is when someone carries out certain research projects and presents it to their other colleagues to look into the effectiveness of an intervention programme. In this case, we tend to work with other health personnels who have the expert knowledge in research to guide us through the research process. (Participant 6)*

(ii) Informal activities

The informal activities include regular discussion among staff and students, for example, in a case study presentation by students or a process of reviewing an article. The participants agreed that in the discussions process, there was learning taking place between the students, young graduates and the senior staff.

> *In our CPD activities weekly, during student case study presentation, everyone benefited from the sessions -- students, young graduates, senior therapists and lecturers especially during the questions-and-answer sessions. (Participant 11)*

I realized that my knowledge from the previous schools of thought is no more relevant... the CPD sessions with the students make me aware of new knowledge… that I was not aware of. (Participant 5)

Theme 2: Outcome of CPD

(i) Knowledge exploration

The participants recognised the benefits of participating in CPD activities. They identified that the learning process includes learning from one another and refreshing previously gained knowledge.

…in CPD, we can learn the latest techniques. CPD is a platform for sharing our experiences, among other staffs who attended courses. So when we sent one staff to attend the courses, she is expected to share her knowledge with us when she comes back to department. (Participant12)

(ii) Clinical competency

They acknowledged that CPD activities could improve their competency and clinical skills, especially through workshops given by foreign guest speakers who presented the latest updates with new skills in patient management.

Especially when there are new treatment techniques, I will try to apply such treatment techniques to patients who come to the department for physiotherapy services. (Participant 17)

(iii) Self development

Most of them acknowledged that the structured courses are worth pursuing. Attending post-graduate courses brought in-depth understanding of an area of specialisation exposure to appraisal of literature review and the critical reasoning process to become reflective practitioners. The co-coordinators of such programmes needed to provide certification or eventually provide credentials for certain skills and performance that would be a boost to the staff involved in the CPD activities.

My participation in post-graduate studies was an eye-opener for me to venture into research. I am able to think critically and provide alternative therapy to the patient's needs. Because of my speciality in manual therapy, I could gain patients' confidence, and they were impressed with my approach of treatment technique…. (Participant 8)

I could reflect when handling patient because I learnt it in my post-graduate study on how to carry out reflection. This has helped me appreciate reflection and indirectly made me think critically. This is definitely useful….I was then able to appreciate problem-based learning and how to translate it into practice and the relevancy of evidence-based practice… (Participant 1)

For me if you undergo certified CPD courses, it gives you more weightage. My organisation provides special allowances and recognition to individuals who have undergone certified specialised courses. (Participant 4)

Theme 3: Barriers undertaking CPD

(i) Financial constraints

Challenges such as financial constraints and high workload demand are limiting factors to CPD participation. Some staff that attended courses were sponsored by their workplace organisation whilst others needed to pay themselves. These courses were generally expensive, especially if it involved foreign guest speakers, with added costs for the fee, transport and lodging.

The fee is expensive and is one of the main reasons why I am not able to attend good courses. This year the allocation of the budget was limited and my manager only allows one sponsored staff to attend the course. Also these courses…were mostly held in Kuala Lumpur, which required me to pay more for lodging and travelling. (Participant 17)

Budget is the number-one factor why I cannot attend good courses and able to attend only certain courses, which are cheaper.(Participant 5)

Most of the courses were held in Kuala Lumpur, which requires me to book a flight to travel besides paying extra costs for the lodging. I have fears travelling in a plane, and the other modes of transport would require me to travel long hours since I am living in Sabah. (Participant 8)

(ii) Demanding workload

Workload demands were indicated as barriers, especially among participants holding managerial positions. The staff shortage in managing patients in the ward setting and the demand of supervisory clinical workload among students were identified as barriers to participation in CPD activities.

From my experience, if you are involved in management, it becomes even more difficult to attend courses, especially if it exceeds more than three days. This is made worse in bigger hospitals where the departments would undergo a series of auditing from certified auditors like SIRIM as well as student's supervisory clinical workload. (Participant 4)

There is not enough staff…. One of the staffs is on emergency leave, one is on maternity leave….. There are also the outpatients who needed to be attended…,so we have to cancel our CPD sessions, as I have to replace some of the staff to go to the wards. In fact, the new knowledge I learnt could not be applied in the clinical setting due to constraint of time. (Participant 5)

(iii) Personal attitude

Attitudinal factors are other contributory factors for poor participation in CPD activities. Some staff was not motivated to improve themselves and are in the "comfort zone." They do not feel the social pressures to develop continuously, even though they are aware that some CPD activities are more rewarding than others. They described the structured courses, for example, a master's programme, as bringing more recognition, a higher salary scheme and promotion, and therefore, worth participation. The senior participants expressed readily that CPD are only for the younger staff, even though they expected to be given equal opportunities like the junior staff for promotions despite their poor enrolment for courses in CPD activities.

> *Some of the senior physiotherapists prefer to let the junior physiotherapists to attend courses due to their family commitments and inability to cope with the changing trend of patient management. However, for some senior physiotherapists who are willing to proceed with CPD, they are encouraged to do so. (Participant 1)*

Theme 4: Strategies to improve participation in CPD

(i) Organisational role

Mandatory participation is encouraged for renewal of the license in ensuring the professionalism of the profession. A mechanism that monitors participation in various CPD activities should be enhanced with incentives and rewards. There were suggestions to professional bodies to play a major role in promoting CPD activities.

> *...I feel remuneration or incentives should be given to those who have undergone further studies, or some kind of specialisation, for example, in Cardiorespiratory, Musculoskeletal or Neurology or value added courses like reflexology or specialised massage technique. They should be given recognition by the professional bodies as well as a mechanism to trace them as experts.... (Participant 1)*

Managers must take the front line in ensuring involvement of CPD among staff. They are responsible for allocating specific time for CPD activities to encourage more participation. In fact, the managers should participate and contribute to CPD activities themselves and set an exemple for other staff.

> *My boss should have strategies, and allocate time frame for CPD activities, perhaps every three months, one physiotherapist in the department should attend CPD courses....(Participant 8)*

> *They should play an important role or else their staff will not take seriously if they themselves did not participate in CPD activities. The leaders need to encourage their staff by taking alternative measures so that CPD programmes are taken seriously among the staff. (Participant17)*

(ii) Relevancy of CPD Activities

The contents of the CPD activities should be relevant for current practices as well as non-clinical activities that are relevant for professional development such as courses related to information technology, management approaches, leadership and communication skills.

> *The CPD activities should also include other non-clinical issues like communication skills, work culture or team work and I'm a bit disappointed, that none of these areas were brought up in the department.(Participant 13)*

A scheduled programme should be identified to ensure continuous flow of events to avoid repetition of the similar topics during CPD activities.

> *Maybe a survey is necessary to ask physiotherapists related to the areas of interests rather than repeating the same topics' several times, for example, lymphedema has been covered on several occasions…. this will bore the participants (Participant 11)*

(iii) Cost-effectiveness

The provision of funding for staff development should be allocated by departmental heads in the planning of the yearly budget. The course organisers should ensure a reasonable fee is charged when organising courses or a mechanism in the provision of subsidies by departments should be considered.

> *Whoever is organising courses should look into the costs so that the participant can afford it. Lately, these courses usually exceed more than RM500 to RM1000. Thus, few would be able to attend, unless it is paid for by the institution. (Participant 3)*

> *…they shouldn't make a profit from CPD courses, imagine if one have to pay RM1500 for a three-day course…. This is too much especially for the younger groups who are only earning between RM1000 to RM2000. Imagine ...if we have to pay RM1500 for a three-day course, this will take away more than half of my pay…because of this. I am not able to attend CPD activities. (Participant 8)*

(iv) Guidelines for CPD activities

A suggestion for the courses offered to demonstrate practical application of theory into practice was made by the participants to ensure benefits for application to practice. Preferably, these courses should be conducted by the best speaker with vast experience to ensure the quality of the CPD contents with distribution of lecture notes during or after registrationfor the courses attended.

> *Sometimes the speakers do not demonstrate their skills but focuses only on the theory... I am not convinced by the speaker that the technique works. They should demonstrate their skills and be confident when answering questions. (Participant 3)*

I think….the slides or notes should be given to the participants to be used as a reference in the future.(Participant 6)

DISCUSSION

It is well documented that continuous professional development (CPD) is important for role expansion of a profession and its integrity (Cooney & Blake, 2000).The physiotherapists agreed that it is through CPD that professionals undergo a process of educational activities to maintain and improve their competency towards a career goal (Pool *et al*., 2012). In fact, it is an integral part of one's working life, where the theories are put into practice (Murphy *et al*., 2006). They demonstrated understanding of the concept of CPD as a process of lifelong learning and a platform for health professionals to gain new knowledge throughout their working life consistent with findings of previous studies (Bolderston, 2007; Gunn & Goding, 2009).

As a consequence, keeping up-to-date with the current evidenced-based practice would lead to quality service in patient care among health professionals (Lee *et al*., 2010). The process of CPD not only involves improving knowledge but specialisation in niche areas of interests, from basic knowledge to advanced knowledge (Bolderston, 2007). Practising new skills through evidence-based practices with new modalities can indirectly provide effective patient care (Bolderston, 2007). Similar to our findings, the physiotherapist perception of CPD was to ensure that they were current in their practices and at par with physiotherapists elsewhere.

There were several advantages in participating in CPD activities identified by the participants that were consistent with previous findings (Blake & Cooney, 2000; Bolton, 2002). This included upgrading of knowledge, sharing of knowledge between colleagues, an opportunity to refresh previous knowledge and acquire the latest information and recognition of practice. As previously acknowledged by Chong *et al*. (2011), knowledge or information that is not updated by learning programmes would result in "deskill" because basic knowledge can last until a half-life of about 2.5 years (Chong *et al*., 2011). The relevancy for CPD participation is also seen in other professions. Similarly, as emphasised by other professionals, most chiropractors agreed that there is a need for participation in CPD activities although it is non-mandatory for them as they discovered that CPD can enhance their professional competencies in providing improved health care services (Bolton, 2002). The nurses improved their competency in practice and developed proper professionalism in their career through CPD activities (Chong *et al*., 2011). This knowledge transfer following participation in CPD activities would increase awareness between the old practices with new practices learnt in a conducive environment (O'Sullivan, 2003;8:107-22).

Our findings revealed that CPD activities have been a pathway among participants to develop critical thinking and become reflective in their practice due to the new knowledge (Cole, 2000) that improved their self-confidence to excel. The enrolment in post-graduate studies provides an avenue for self-development and an opportunity to understand arising problems and create a new interest to develop (Bolton, 2002). The participation in CPD of staff indirectly promotes a working environment that is translated to deliver high-quality health care services (Martin *et al*., 2008). In some organisations, for example, in the Ministry of Health, the staff are awarded credit points to meet the department's key performance indicators (KPI) that can be translated into recognition and salary advancement (Bloom, 2005). The provision

of incentives and remunerations are motivating factors for involvement in CPD activities (Landers *et al*., 2005) among staff.

Likewise, the participation in CPD activities is not without its challenges. There were similar challenges observed in relation to compliance with CPD activities, which includes the demand for extra time as well as the need for considerable effort when undertaking CPD activities (Tennant & Field, 2004). The lack of financial support by a hospital or institutions to provide an opportunity to attend CPD activities were similar findings (Brown *et al*., 2002; Murphy *et al*., 2006). Some physiotherapists had to invest their own money to attend courses for payment of fees, lodging and travelling expenses as reported by Murphy *et al*. (2006), who cited one instance when participants had to allocate 30 % of their income for further education (Murphy *et al*., 2006). Furthermore, the good courses are expensive (Lee *et al*., 2010) especially if external speakers are brought in from overseas. The increased workload at the workplace is another contributory factor that hinders participation in CPD activities, especially that of those with multiple roles, for example, administrative duties, clinical work and student supervision (Wood, 2008). Managers have encountered problems when giving time off to staff for attending CPD activities (Chong *et al*., 2011) as the time taken off could not be replaced by other staff members (Sturrock & Lennie, 2012). The inadequate staff numbers in the workplace can prevent the application of the new knowledge into practice due to time constraint and non-familiarity with the new knowledge (Jull, 2009).

Other barriers to CPD participation were attitudinal factors such as lack of interest, demotivation, comfort zone and family commitments (Hancox, 2002). Personal attitude is crucial as CPD requires the individuals to take personal responsibility for identifying their own learning needs and to evaluate if these needs are met (Donen, 1999). The poor motivation to meet a learning need would prevent them from travelling long distances to attend a workshop (Lee *et al*., 2010). This is similar to the finding of Jukkala *et al*. (2008), who found that 40 % of the subjects in their study agreed that they needed to travel long distances just to gain new knowledge.

Generally, this study showed that the physiotherapists interviewed were motivated to be involved in CPD activities but constrained by a demanding workload, low or no budget and attitudinal factors, poor recognition and acknowledgement. Despite this, they acknowledges that there was a need to create an environment that fosteredCPD through departmental plans that recognise the gaps in knowledge and co-ordinate CPD activities to match clinical needs either through informal or formal activities.

CONCLUSION

Overall, this study managed to identify the physiotherapists' perception of CPD, impact on their practice, the barriers to CPD participation and the strategies to improve CPD participation. Generally, they agreed that CPD was relevant and that every individual needed to update him/herself regularly by attending workshops, seminars or conferences to ensure professional survival and competency. Heads of department or managers should demonstrate flexibility to provide staff with resources and opportunities despite constant budgetary pressures. This has major implications for managers to ensure that an effective mechanism is in place to allow

recognition and rewards for staff who are constantly seeking new knowledge for the growth of their professional development. Such findings can provide suggestions to higher authorities and policy makers to take measures for continuous support of CPD activities in the near future.

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Total Phenolic Content and Antioxidant Activity of *Quercus infectoria* Galls Using Supercritical CO_2 Extraction Technique and Its Comparison with Soxhlet Extraction

Hasmida, M. N. [1*], Liza, M. S. [1,2] , Nur Syukriah, A. R. [2], Harisun, Y. [3], Mohd Azizi, C. Y. [1] and Fadzilah Adibah, A. M.[2]

[1]*Centre of Lipids Engineering and Applied Research, Ibnu Sina Institute for Scientific and Industrial Research, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Malaysia*

[2]*Department of Bioprocess Engineering, Faculty of Chemical Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Malaysia*

[3]*Institute of Bioproduct Development (IBD), Universiti Teknologi Malaysia, 8131 UTM Johor Bahru, Malaysia*

ABSTRACT

Quercus infectoria gall, which is known as manjakani in Malaysia, was traditionally used in treating diseases. The bioactive compounds from the galls can be extracted using various extraction methods. In this study, supercritical carbon dioxide (SC-CO_2) extraction was used to study the effects of CO_2 flow rate on the yield, total phenolic content and antioxidant activity of *Q. infectoria* extract by fixing the pressure and temperature at the highest density (P: 30 MPa, T: 40°C). The results were compared with those acquired from the Soxhlet extraction method. The results showed that the Soxhlet extraction had a higher percentage of extraction yield than SC-CO_2 extraction. The selectivity of *Q. infectoria* extracts using SC-CO_2 extraction was better than the Soxhlet extraction method. Meanwhile, the extraction efficiency using the SC-CO_2 extraction ranged from 46% to 53%. The SC-CO_2 extraction also yielded higher total phenolic content than using the Soxhlet extraction method when 2 mL/min of CO_2 flow rate was applied (203.53 mg GA/g sample). This study also revealed that the extracts from the SC-CO_2 extraction showed a better radical scavenging activity compared to the Soxhlet extraction when analysed using DPPH (2,2-diphenyl-1-picryl hydrazyl) radical scavenging activity assays.

Keywords: *Quercus infectoria*, radical scavenging activity, Soxhlet extraction, supercritical carbon dioxide extraction, radical scavenging activity, total phenolic content

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E-mail addresses:
hasmidanasir@gmail.com (Hasmida, M. N.)
i.liza@cheme.utm.my (Liza, M. S.)
syukriah_org@yahoo.com (Nur Syukriah, A. R.)
harisun@ibd.utm.my (Harisun, Y.)
azizi@cheme.utm.my (Mohd Azizi, C. Y.)
adibah@cheme.utm.my (Fadzilah Adibah, A. M.)
*Corresponding Author

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INTRODUCTION

Quercus infectoria, which is known as Manjakani in Malaysia, is a small tree native to Greece, Asia Minor and Iran (Basri & Fan, 2005). The attack by the gall-wasp *Adleria gallae-tinctoria* results in the galls arising on the young branches of this tree. The galls of *Q. infectoria* are usually used in herbal drink as a remedy for women after their childbirth and to restore the elasticity of the uterine wall. Besides, these galls are also traditionally used as dental powder and in the treatment of toothache and gingivitis. Inflammatory disease has traditionally been cured by using *Q. infectoria* extract. The potentials of *Q. infectoria* in medical and nutraceuticals areas as reported by Kaur *et al*. (2004) have encouraged other researchers to further study and investigate the usage and application of the galls of *Q. infectoria*.

In the galls of *Q. infectoria*, the bioactive compounds that have medicinal properties are tannin (50-70%), small amount of gallic acid, and ellagic acid (Wiart & Kumar, 2001). There are other constituents such as syringic acid, β-sitosterol, amentoflavone, hexamethyl ether, isocryptometrin, methyl betulate, methyl oleanate, and hexagalloyglucose (Hwang *et al*., 2000). Tannin, which is derived from phenolic compounds, has been found to have antioxidant activity and antimicrobial (Everest & Ozturk, 2005), antibacterial (Hamid *et al*., 2005), and antifungal (Yamunarani *et al*., 2005) properties.

Recently, supercritical fluid extraction has gained much interest as an alternative way to replace the conventional methods for the extraction of phytochemicals from plant matrix. Theoretically, Pourmortazavi and Hajimirsadeghi (2007) have reported that supercritical fluid extraction, which operates based on the utilisation of a fluid under supercritical conditions, is a technology suitable for extraction and purification of a variety of compounds especially those that have low volatility or that are susceptible to thermal degradation. This is because supercritical fluid has gas-like characteristics that help the fluid diffuse to the matrix and access the phytochemicals, while its liquid-like characteristics provide good salvation power. Furthermore, diffusivity, density, surface tension and viscosity of supercritical fluids can be varied by altering the operating conditions. Therefore, these properties of supercritical fluids can give advantages in controlling the extraction process. Additionally, using supercritical carbon dioxide (SC-CO_2) as a solvent gives advantages to the extraction process due to non-toxic and non-flammable characteristics of CO_2. This method is inexpensive and can be used under mild operating conditions, namely 7.4 MPa critical pressure and 31°C temperature of CO_2.

This study investigated the effects of CO_2 flow rate on the yield, total phenolic content, and antioxidant activity of *Q. infectoria* extract. Then, all the properties were compared with those acquired from the Soxhlet extraction method.

MATERIALS AND METHODS

Preparation of Material

The *Q. infectoria* galls were prepared by rinsing the galls with tap water in order to remove unwanted material from the samples. Then, the galls were subsequently dried overnight in an oven at 50°C. Before the extraction was done, the galls were crushed by using a mechanical mortar. The prepared galls were stored in a dark place at room temperature.

Supercritical Carbon Dioxide (SC-CO_2) Extraction

SC-CO_2 extraction was performed using the method proposed by Mandana *et al*. (2011a) with some modifications. The SC-CO_2 system comprised a 50-mL extraction vessel, a high-pressure pump, an automated back pressure regulator and an oven. CO_2 in liquid form was supplied from a gas cylinder. 15.00 ± 0.05 g of *Q. infectoria* was extracted using CO_2 flow rate of 2, 3, and 4 mL/min. The fractionation was done for every 10 min time interval of 2 h. The pressure and temperature were fixed at the highest possible density, which was 0.92 g/mL with the pressure of 30 MPa and the temperature of 40°C in order to determine the effect of CO_2 flow rate for the extraction process.

Soxhlet Extraction Method

Soxhlet extraction was carried out to compare the extraction performances with the SC-CO_2 extraction. In order to prepare the sample extract, 5.00 ± 0.05 g of powdered *Q. infectoria* galls was inserted in the thimble, while 150 mL of 100% methanol (boiling point, bp = 64.7°C) was placed in the flask of Soxhlet apparatus. The temperature of the process was corresponded to the boiling point of solvent used and the extraction time was set for 6 h. At the end of the process, the solvent was removed from the yield by using a rotary evaporator at 40°C. All the steps were repeated by using 100% ethanol (b_p = 78.37°C), acetone (b_p = 56.0°C) and water (b_p = 100°C) as the extraction solvents.

Yield Calculation

The yield of the extract defined on 1 g of *Q. infectoria* galls basis was calculated by using the following equation:

$$\text{Percentage extract yield} = \frac{m_1}{m_0} \times 100\% \qquad [1]$$

Where m_1 is the mass of the extract in gram and m_0 is the mass of the sample in gram.

Total Phenolic Content Analysis

Total phenolic content (TPC) in the *Q. infectoria* extracts was analysed by using Folin-Ciocalteu (FC) reagent. The solution was prepared by mixing 20 µL of 1 mg/mL plant extract, 1.58 mL of distilled water, and 100 µL of FC reagent (diluted ten-fold) thoroughly in a test tube. The solution was left at room temperature for 7 min in order for the reaction to take place. Then, 300 µL of 75 g/L sodium carbonate (Na_2CO_3) solution was added into the sample solution and the tube was kept in a dark place for 30 min at room temperature. The absorbance of the solution was measured at 765 nm. The calculation of TPC was done on the basis of the gallic acid standard curve, which was constructed by using the same procedures and concentrations of 0, 50, 100, 150, 250, and 500 mg/mL. The results were expressed as gallic acid equivalents (mg GAE/g extract sample).

Antioxidant Activity Assay

Assay for antioxidant activity of the extract was done by dissolving 77 µL of 2.5 mg/mL extracts in 3 mL of 6×10^{-5} M methanolic DPPH solution. DPPH or 2,2-diphenyl-1-picrylhydrazyl is a stable free radical that forms a purple-coloured solution when dissolved in methanol. Antioxidant components can scavenge this stable free radical and therefore the purple colour will be bleached. The mixture was vortexed at room temperature for 30 s. The control sample absorbance ($A_{control}$), which contained methanolic solution of DPPH, was also carried out. All of the mixtures were placed in a dark place for 30 min at room temperature. The absorbance of all the sample solutions (A_{sample}) was measured at 517 nm using UV-Vis spectrophotometer. Antioxidant activity was calculated by using the following equation:

$$\text{Antioxidant activity (\%)} = \frac{A_{control} - A_{sample}}{A_{control}} \times 100\% \qquad [2]$$

Statistical Analysis

Results were expressed as the mean ± S.D. of duplicate independent experiments. Data were analysed using SPSS 16.00 for Windows (SPSS Inc., Chicago, IL). The significant differences between the data were analysed by using one-way analysis of variance (ANOVA) at 95% confidence level. P values of <0.05 were considered to be significant.

RESULTS AND DISCUSSION

Comparison of Extraction Yield from Soxhlet Extraction and Supercritical Carbon Dioxide Extraction

In order to get the percentage yield of *Q. infectoria* extracts using SFE, a set of extraction experiments was done at 30 MPa and 40°C, while the flow rate of CO_2 varied from 2 to 4 mL/min. Table 1 shows the effects of CO_2 flow rate on the extraction yield of the *Q. infectoria* extract.

TABLE 1 : Comparison of percentage yield of Q. infectoria by using supercritical CO_2 extraction

Type of plant	CO_2 Flow Rate (mL/min)		
	2	3	4
Q. infectoria	0.3652*	0.3060	0.2940

*Extraction yield expressed as % dry weight.

The percentage yield of the *Q. infectoria* extract at 2 mL/min (0.37%) is the highest value compared to others, whereas the lowest percentage yield is 4 mL/min (0.29%). This finding revealed that the solute-solvent saturation was achieved when lower flow rate was applied (Ana Najwa, 2008). When this happened, the increase in residence time would increase the solubility of the solute in the solvent. Moreover, King (1997) mentioned that extracting seeds with high oil content is best when a low solvent flow rate is used in order to hinder compaction of the

sample in the vessel that may obstruct complete extraction of the oil. In addition, Kumoro and Hasan (2007) stated that the cumulative yield of the extract is improved as the flow rate of the solvent increases.

The results obtained by using the Soxhlet extraction method are shown in Fig.1. Generally, each solvent used gave high percentage yield in the range between 45.71% and 80.03%. Based on the findings, the use of 100% aqueous as a solvent showed the highest percentage yield for the extraction of *Q. infectoria* by using the Soxhlet extraction method. On the other hand, the lowest extraction yield for *Q. infectoria* was found when 100% acetone was used, suggesting that polar compounds in biological plant are easier to extract with more polar solvents, while the less polar solvent allows the extraction of the less polar compounds (Mandana *et al*., 2011b). This is based on the theory of 'likes dissolve likes'. The results showed that pure aqueous gave higher yield compared to others because of the higher polarity of the solvent. The preceding findings were opposite to the results by Wang *et al*. (2011) who found that pomegrated peel was extracted effectively using methanol, followed by water, ethanol, and acetone. This contrary finding might be caused by the different characteristics of the desired compound, extraction method and the origin of the raw material used (Caldera *et al*., 2012).

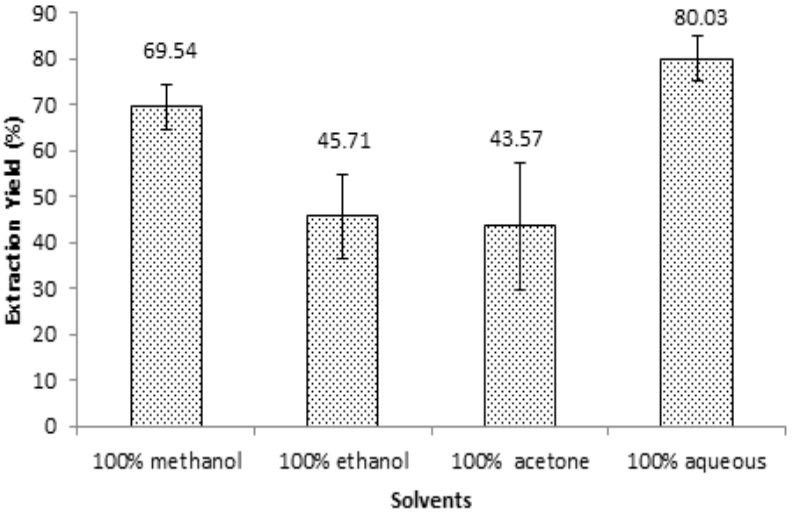

Fig.1: Percentage yield of Q. infectoria by using the Soxhlet extraction method

From these results, the performance of SC-CO_2 extraction was low as it obviously showed that the yield obtained from the Soxhlet extraction was higher than the SC-CO_2 extraction with $P<0.05$. Nevertheless, the selectivity of the extracts using SC-CO_2 extraction offers considerable advantages such as clear colour of the extracts attained from the SC-CO_2 extraction process. This observation was due to the less impurity or organic matrices which were extracted during the process. However, the extracts of the Soxhlet method were dark in colour and highly turbid. Similar findings were found by Berglö *et al*. (1999) and Hawthorne *et al*. (2000). The performance of the SC-CO_2 extraction could also be explained by several analyses done on the *Q. infectoria* extract, which were total phenolic content and antioxidant analysis.

Total Phenolic Content

Total phenolic content (TPC), as determined by using the Folin Ciocalteu method, was reported as gallic acid equivalents (mg GAE/g sample). This analysis was used to examine its contribution in the antioxidant activity of the plant extracts. The total phenolic content of the extract is shown in Table 2.

TABLE 2 : Total Phenolic Content of *Q. infectoria* for different extraction methods

Extraction Method	Total Phenolic Content ± SD (mg GA/g sample)
SC-CO_2 extraction	
Flow rate: 2 ml/min	203.53 ± 10.56[a]
Flow rate: 3 ml/min	186.13 ± 7.46[a]
Flow rate: 4 ml/min	193.60 ± 9.88[a]
Soxhlet extraction	
100% methanol	95.86 ± 2.02[b]
100% ethanol	109.78 ± 6.57[b]
100% acetone	107.64 ± 0.50[b]
100% aqueous	95.86 ± 1.01[b]

SD: standard deviation

a,b shows significantly different ($P<0.05$)

The total phenolic content in the extracts when 2, 3, and 4 mL/min of CO_2 flow rate were used was found out to be 203.53, 186.13, and 193.60 mg GAE/g sample, respectively. The CO_2 flow rate of 2 mL/min showed the highest phenolic content, suggesting the highest solubility of the sample in the solvent. The solvent can easily access the solute when the solubility is high; hence, the amount of the desired compound extracted increases and the extraction of impurities can be avoided.

As for the Soxhlet extraction method, the best content of phenolic compound was found in 100% ethanol extract (109.78 mg GAE/g sample), but the difference with the other solvents was shown to be insignificant ($P>0.05$). These findings clarified that the amount of yield extracted did not affect the content of phenolic compound in the extract.

Based on the findings, the SC-CO_2 extraction gave significantly higher total phenolic content ($P<0.05$) in the *Q. infectoria* extract as the efficiency of SC-CO_2 extraction ranged from 46% to 53% compared to the extracts of Soxhlet extraction method. Basically, a higher amount of phenolic compound is useful for the prevention of oxidative activities of the plant's extract.

DPPH Radical Scavenging Activity of Q. infectoria

The DPPH radical scavenging activity analysis was accomplished to investigate the ability of *Q. infectoria* to scavenge free radicals in vitro by improving the percentage of the scavenging activity. For the extraction using SC-CO_2, all of the extracts using various CO_2 flow rates gave significantly high activity, as established in Table 3. The table clearly shows that the highest

DPPH radical scavenging activity was obtained by using 3 mL/min (96.96%), but the difference with the other CO_2 flow rate was very low (P>0.05). The extracts from the Soxhlet extraction showed that the water extract (94.55%) gave the highest antioxidant activity, but with a slight or no significant difference with the others (P>0.05). These findings revealed that the types of solvent used in the Soxhlet extraction method did not give significant effect on DPPH radical scavenging activity of the extracts. In addition, the results from these two extraction methods could be explained by the fact that the extract is rich in phenolic compounds which always play an important role in the radical scavenging activity of the plant (Poumorad *et al*., 2006).

TABLE 3: DPPH radical scavenging activity of *Q. infectoria* for different extraction methods

Extraction Method	Antioxidant activity ± SD (%)
SC-CO_2 extraction	
Flow rate 2 mL/min	96.93 ± 0.92[a]
Flow rate 3 mL/min	96.96 ± 0.01[a]
Flow rate 4 mL/min	95.84 ± 0.15[a,b]
Soxhlet extraction	
100% methanol	93.38 ± 0.18[b]
100% ethanol	92.60 ± 1.28[b]
100% acetone	92.83 ± 1.61[b]
100% aqueous	94.55 ± 0.37[a,b]

SD: standard deviation

a,b shows significantly different (P<0.05)

CONCLUSION

In conclusion, the *Q. infectoria* extracts from supercritical carbon dioxide extraction undoubtedly gave better total phenolic content and DPPH radical scavenging activity compared to the Soxhlet extraction method. The findings showed that there was a significant difference between these two extraction methods even though the Soxhlet extraction showed better result in terms of extraction yield. Furthermore, supercritical carbon dioxide extraction was found to be more efficient than the Soxhlet extraction method as the supercritical carbon dioxide extraction took lesser time consumption and amount of solvent. Thus, supercritical carbon dioxide extraction is more suitable for extracting food material than the Soxhlet extraction method.

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Drying Performances and Milling Quality of Rice during Industrial Fluidized Bed Drying of Paddy in Malaysia

Ibrahim, M. N.[1*], Sarker, M. S. H. [1, 2], Ab. Aziz N.[1] and Mohd Salleh, P.[3]

[1]Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

[2]Department of Food Engineering and Technology, Hajee Mohammad Danesh Science and Technology University, Dinajpur-5200, Bangladesh

[3]Deputy Director (Packaging and Handling Programme), Food Technology Research Centre, MARDI, 43400 Serdang, Selangor, Malaysia

ABSTRACT

Field investigation on the operation of an industrial fluidized bed paddy dryer of 25 t/h capacity available in a processing complex of Padiberas Nasional Berhad (BERNAS) of Malaysia was carried out to assess its drying characteristics, energy consumption and quality of product during two paddy harvesting seasons. A grain drying simulation model was used to predict dryer performance which can be used as a basis for improving drying operations. For the first season (August-September), average drying rate was found to be 538 kg moisture/h to reduce moisture content (mc) from 36.98± 0.89% dry basis (db) to 27.58±0.79% (db) at 100-120°C of drying air temperature with a feed rate (capacity) of 7.75 t/h. In the second season (February-March), average drying rate was found to be 435 kg moisture/h to reduce mc from 28.14 ±0.68% (db) to 22.54 ± 0.69% (db) at 78-90°C drying air temperature with a feed rate of 9.5 t/h. The thermal and electrical energy consumptions were obtained as 7.57 and 0.97 MJ/kg water removed, respectively, for the first season, while 5.92 and 1.2 MJ/kg water removed for the second season. Higher head rice yield and whiteness and lower milling recovery were achieved during the first season than the second season at acceptable milling degree and transparency. Meanwhile, simulation results indicated that the dryer performed better in terms of increased drying capacity during the second than the first season; the dryer could be operated at 150°C to achieve almost double throughput capacity up to 20 t/h for the second season, while for the first season, high mc hindered the capacity to be at or below 7.75 t/h even when using higher a temperature of 160°C to reduce moisture to the desired final moisture of 24-25% (db). proportion of slower vehicles based on users' opinion poll.

Keywords: Drying simulation, drying rate, industrial fluidized bed paddy dryer, milling quality, energy consumption, head rice yield, drying performance

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E-mail addresses:
nordinib@upm.edu.my (Ibrahim, M. N.),
mshsarker_hstu@yahoo.com (Sarker, M. S. H.),
norashikin@upm.edu.my (Ab. Aziz N.),
msp@mardi.gov.my (Mohd Salleh, P.)
*Corresponding Author

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INTRODUCTION

Reduction of high moisture in paddy rapidly to a safe level of 22-23% (the moisture content is expressed as the percentage dry basis throughout this paper unless stated otherwise) has been suggested by many researchers (Igathinathane *et al*., 2008; Soponronnarit, 1997; Tirawanichakul *et al*., 2004; Poomsa-ad *et al*., 2001). Sutherland and Ghaly (1992), the pioneers of fluidization technique for paddy drying, showed that head rice yield was higher (i.e., between 58-61%) when paddy moisture content was reduced from 28.2% to 20.5 % but it was lower (i.e., between 15% to 24%) when the final moisture content was 19%. Tumambing (1993) developed a mathematical model and conducted an experiments on continuous fluidized bed drying of paddy with experimental conditions of 40-100°C drying air temperature, 25-20 cm bed thickness and 1.5-2.5 m/s of air velocity. The researcher claimed that a fluidized bed dryer offers promising alternative to be used for rapid pre-drying of paddy. Feasibility of paddy drying by fluidization technique also was conducted by Soponronnarit and Prachayawarakorn (1994), who reported the drying capacity of a dryer increased with specific air flow rate and drying air temperature, while energy consumption was reduced when specific air flow rate was decreased or fraction of recycled air was increased. Soponronnarit *et al*. (1995) described the design and testing of a prototype fluidized bed paddy dryer with a capacity of 0.82 t/h. They used air temperature of 100-120°C, fraction of recycled air of 0.66, specific airflow rate of 0.05 kg/s-kg dry matter, superficial air velocity of 3.2 m/s, and bed depth of 0.1 m to reduce paddy moisture contents from 45% to 24%. They also found that electrical and thermal energy consumptions in terms of primary energy were 0.53 and 1.79 MJ/kg water evaporated, respectively. Soponronnarit *et al*. (1996a) also developed a cross flow fluidized bed paddy dryer with a capacity of 200 kg/h and suggested that the final moisture content of paddy should not be lower than 23% so as to maintain quality in terms of both whiteness and head rice yield. They further added that energy consumption to reduce moisture from 30 to 24% was minimum at the drying air temperature of 115°C, air speed of 2.3 m/s, bed thickness of 10 cm and fraction recycled air of 0.8, while the drying capacity was near maximum. Soponronnarit *et al*. (1996b) investigated the performance of commercial fluidized bed dryer with capacities of 1-2, 2.5-5.0 and 5-10 t/h having the provision of recycling the exhaust air while the heat source was from burning diesel or oil fuel. They reported that energy consumption decreased with increasing moisture content of paddy and drying temperature. In order to reduce paddy moisture down to 22% (db) in a single pass, they recommended the use of maximum drying temperature of 150°C for acceptable quality of product. Based on the foregoing studies, the feasibility of fluidized bed dryer for drying of high moisture paddy is favourable because of its compact size, fast drying rate and low energy consumption with acceptable quality (Soponronnarit, 1999). Prachayawarakorn *et al*. (2005a) studied the performances of industrial pulsed and traditional fluidized bed dryers and reported that head rice yield and whiteness were similar in both dryers. In terms of energy consumption, they found that the pulsed fluidized bed dryer was more economical than the conventional dryer. Although extensive research has already been performed on batch fluidized bed paddy drying on laboratory scale, limited published works focusing on the performance of large scale industrial paddy dryer quantifying the criteria such as drying characteristics, energy consumption and final quality of dried product with comprehensive statistical analysis are

available at present. Meanwhile, performance evaluation of a dryer promotes its successful and economic operation. Before starting the operation of any dryer, it is very important to select and fix the possible suitable air velocity, particularly air temperature and weir height, for a constant feed rate in order to minimize energy consumption and maximize drying capacity. Computer simulation is a cheap and time-saving method to predict drying parameters for designing and optimizing the operation of a dryer. The simulation approach is able to select suitable drying air temperature at a particular throughput capacity that meets the intended moisture reduction from freshly harvested wet paddy to achieve the desired level of the final moisture content, which will not otherwise be possible without extensive experimental works. The factors that limit the dryer operation can easily be sorted out. Hence, the present attempt was undertaken to obtain the practical information on industrial fluidized bed paddy drying in the Complex of BERNAS and to further suggest for improvement of the dryer operation to achieve quality rice.

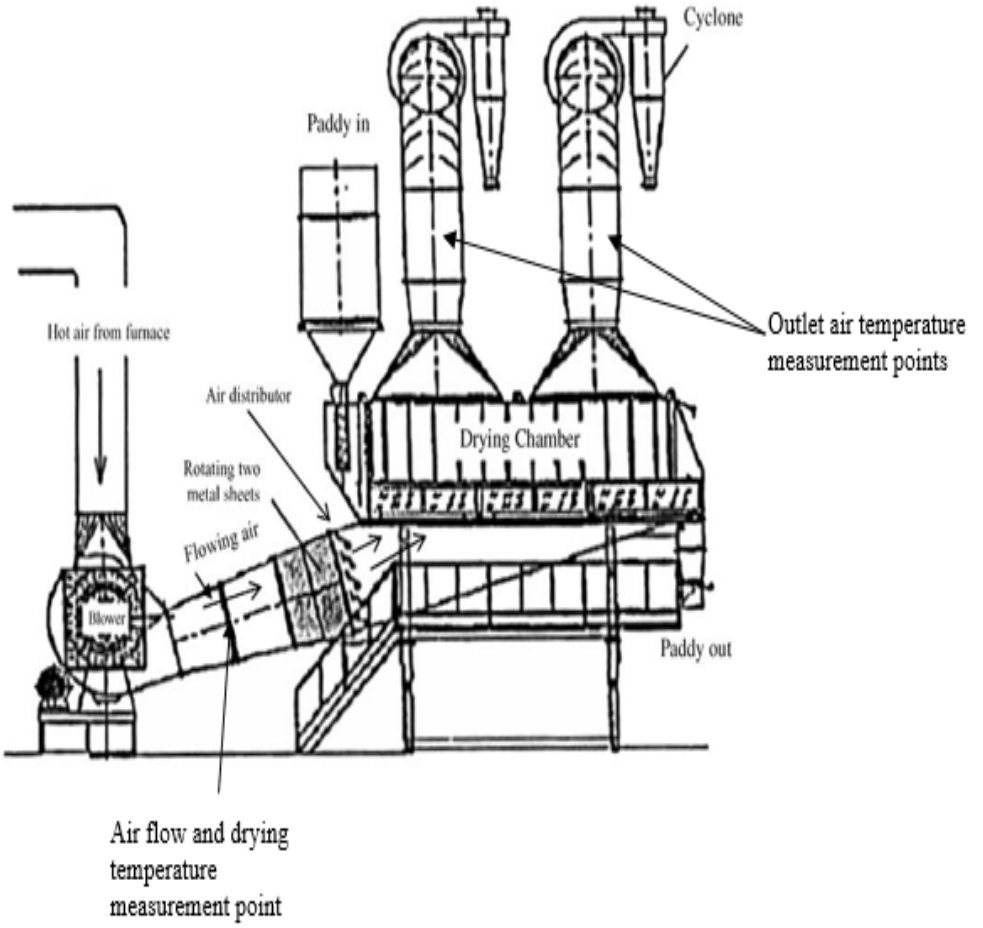


Fig.1: Schematic diagram of an industrial pulsed fluidized bed dryer
(Adapted from Prachayawarakorn et al. (2005a)

MATERIALS AND METHODS

Operating the Industrial Fluidized Bed Dryer

For the purpose of this study, an industrial pulsed fluidized bed dryer (FBD) with 5.0 m × 1.22 m bed area and 25 t/h capacity available at paddy processing plant of BERNAS, Simpang Empat in Perlis, Malaysia, was operated in a continuous mode during two usual paddy drying seasons. A schematic diagram indicating temperature and air flow measurement points is presented in Fig.1. This is almost similar to the dryer proposed by Prachayawarakorn *et al*. (2005a). The average initial moisture content was 36.98 ± 0.89 and 28.14 ± 0.68 % db during the first season (August-September) and the second season (February-March), respectively. The freshly harvested paddy of MR219 variety was dried in both the seasons. Drying temperature and air flow rate were 100-122°C and 13.66 m^3/s, respectively, during the first season while these values were 78-90°C and 13.66 m^3/s in the second season. All data were recorded when steady state condition of the operation of the dryer was observed. Dryer was operated in two seasons to take into account the seasonal variation in the operating parameters of the dryer, which were obviously found to be changed due to different initial moisture contents of the paddy. The drying air was from the heat produced by the combustion of rice husk in cyclonic furnace.

The collected data were impurities and initial moisture contents of paddy, drying air velocity, temperature, as well as relative humidity of air and moisture change of the paddy during drying. During operation of the dryer, samples were taken simultaneously from inlet and outlet of dryer at an approximately 60 min interval, while moisture reduction was determined in three replications. Control paddy samples were dried using ambient air, while paddy was spread at 1-2 cm bed thickness on plastic mat under shed. Besides paddy drying with industrial dryer, control drying was carried out so as to compare rice quality in terms of head rice yield, whiteness, milling recovery, milling degree and transparency with fluidized bed drying. It is important to note that after fluidized bed drying, the paddy samples were further dried to around 16% using ambient air for the present work. It is also worth mentioning that bulk paddy after fluidized bed drying was further dried using inclined bed dryer in the plant as per usual drying practice. The moisture content of the paddy was measured by the Satake digital grain moisture meter model "SS-6" with an accuracy of ±0.5%. The moisture meter was previously calibrated with standard oven method (at the temperature of 103°C for 24 hours) through determining paddy moisture content in the laboratory. Drying air temperature and relative humidity were measured by K-type thermocouple (HANNA Co. with ± 0.5°C accuracy) connected with data logger and Thermo Hygrometer (H19564, HANNA), respectively. Air velocity at dryer inlet was measured using Thermal Anemometer (TESTO 4235 with ± 0.03 m/s). Knowing the cross-section area at the point of air velocity measurement, the volume of air was calculated by continuity equation (Eq. 1). Bed air velocity was calculated using the same equation. The collected data were used for calculating actual energy consumption during paddy drying operation using Equations 2, 3,4 and 5 according to Jittanit *et al*. (2010) and Sarker *et al*. (2013a & 2014).

$$Q = A \times V \quad [1]$$

$$E_{Total} = 2.6E_{elec} + E_{heat} \quad [2]$$

$$E_{elec} = P \times t \quad [3]$$

$$E_{heat} = m_a C_a (T_i - T_{mix}) \quad [4]$$

$$m_a = Q \times \rho_a \times t \quad [5]$$

Where, Q is the drying air volume (m^3/s), A is the cross-sectional area of air inlet (m^2), V is the mean velocity of the drying air across the air inlet section (m/s), E_{total} is the total primary energy consumption (kJ), E_{elec} is the electrical energy consumption by the blower fan of the dryer (kJ), E_{heat} is the thermal energy consumption for heating the drying air (kJ), P is the power of the blower fan motor (kW), t is the total drying time (hour), m_a is the mass of the drying air (kg), ρ_a is the air density (kg/m^3), C_a is the specific heat of drying air (kJ/kg °C), T_i is the ambient air temperature and T_{mix} is the drying air temperature after heater. Drying time (residence time) of FBD was calculated as the hold-up capacity divided by feed rate according to Soponronnarit *et al*. (1996a), while feed rate was calculated by dividing the total amount of paddy dried by total time of operation of FBD. The dried paddy samples were stored in a refrigerator at 4-6°C in sealed poly packages for 3-4 weeks for further quality testing. In addition, a computer drying simulation model (Sarker *et al*., 2013b) was used for the calculation in predicting dryer performances. The simulation programme was run at various feed rates using different drying temperatures ranging from 90 to 160°C, supposed to be the suitable drying temperature for fluidized bed drying of paddy (Poomsa-ad *et al*., 2001; Soponronnarit *et al*., 1994, 1996a; Sutherland & Ghaly, 1990) for selecting the possible maximum dryer throughput capacity for reducing paddy moisture content to 24-25% db, the safe level of moisture content after the fluidized bed drying for maintaining quality of rice.

Assessment of Rice Quality

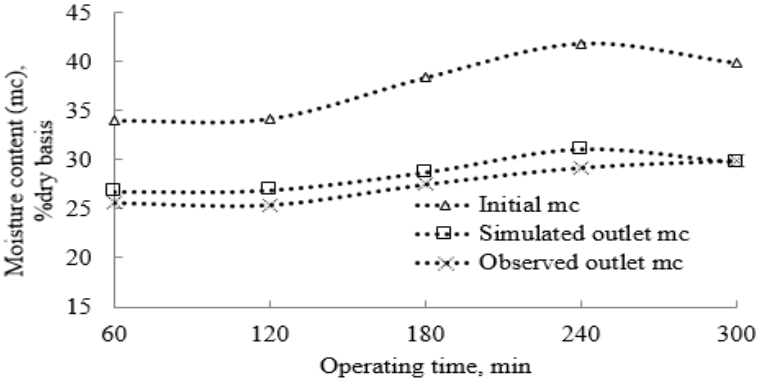
For head rice yield (HRY) determination, 125 g dried and cleaned paddy sample, with two replications, was dehusked with a Testing Husker (THU-35A, Satake Engineering Co., Ltd.), while the bran was removed with a Satake Testing Mill (TM 05C) running for 45 sec for each amount of dehusked brown rice samples. Head-rice was separated by Satake Test Grain Grader (TRG 05B) using 5.2 mm S-type identical cylinder. HRY was defined in this study as the ratio of head-rice mass to original cleaned dried paddy mass. Whiteness, milling degree and transparency were measured using a Satake whiteness meter with four replications as double sub-samples obtained from each sample of HRY. Percentage milling recovery was calculated as the weight of total milled rice (including head rice and broken rice) divided by the weight of dried paddy sample and multiplied by 100. Rice milling degree is defined as the extent to which the bran layers of rice have been removed during the milling process.

Statistical Analysis

The statistical analysis was carried out by using a single factor experiment in completely randomized design (CRD). The only factor was drying method (two drying methods: Control drying and fluidized bed drying). Each drying method was replicated twice. The statistical software package SAS 9.2 version was used to calculate the mean values, standard error mean (SEM) and the analysis of variance of obtained values on head rice yield, whiteness, milling recovery, milling degree and transparency. Meanwhile, Duncan's Multiple Range Test analysis was employed to determine the differences in the rice quality parameters between control drying and fluidized bed drying in case of each season. In addition, analysis was done to compare the quality of milled rice between two seasons with only fluidized bed drying method was considered as the factor.

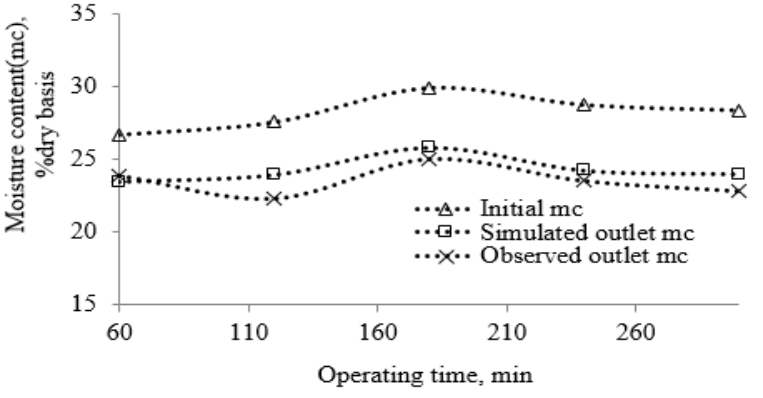
RESULTS AND DISCUSSION

Simulation Results Compared with Observed Results



a. First season

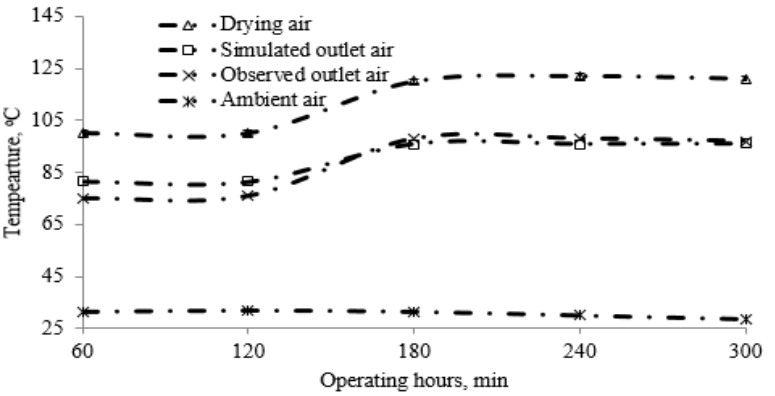
[Feed rate=7.75 t/h, drying air temperature=100-120°C, air velocity= 2.24 m/s and weir height=10 cm]



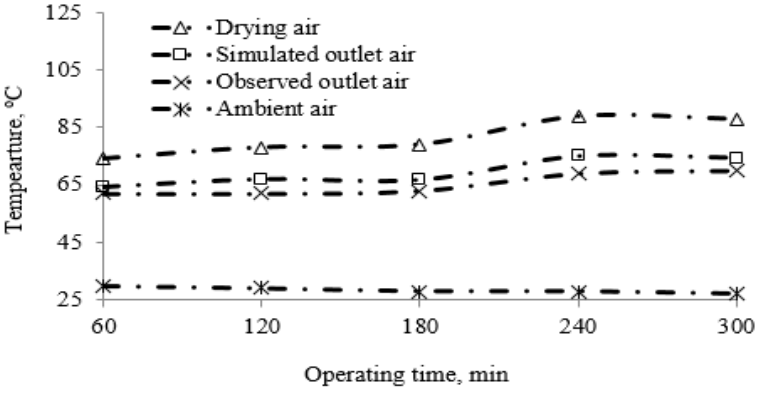
b. Second season

[Feed rate=9.5 t/h, drying air temperature=78-90 °C, air velocity= 2.24 m/s and weir height=10 cm]

Fig. 2: Comparison of simulated and observed moisture content



a. **First season**
[Feed rate=7.75 th^{-1}, Paddy initial moisture content=34-41.84% (db), air velocity= 2.24 ms^{-1} and weir height=10 cm]



b. **Second season**
[Feed rate=9.5 th^{-1}, paddy initial moisture content=30.77-33 % (db), air velocity= 2.24 ms^{-1} and weir height=10 cm]

Fig. 3: Comparison of simulated and observed outlet air temperature.

Fig.2 and Fig.3 represent moisture reduction and evolution of temperature, respectively, during drying of high moisture paddy by the fluidized bed paddy dryer. The simulated outlet moisture content and outlet air temperature were also plotted to indicate a very good agreement with the observed results. Moreover, the mean relative deviation (MRD) values were obtained as 4.86 and 7.62 % for the outlet moisture content during the first and second seasons, respectively, and 11.01 and 10.59% for the outlet air temperature for the two seasons also proved the accuracy of prediction. From Fig.2, it is clearly noticed that moisture drop ranged from 8.42 to 10.1% in the first season operation when drying paddy of 34-40% initial moisture content at the drying temperature of 100-120°C and bed air velocity of 2.24 m/s. Moisture drop during the second season operation was obtained in the range of 2.83 to 7.2% from the initial moisture content of 27-30% at the drying temperature of 78-90°C and the bed air velocity of 2.24 m/s. The residence time was calculated as 2.75 min and 2.24 min for the first and second seasons, respectively. The simulated outlet paddy moisture was slightly overestimated. The drying air

temperatures for drying of various initial moisture content paddy by the dryer were between 78 to 120°C in the two operational seasons. Consequently, both corresponding observed and simulated outlet air temperatures were found to be varied in each season, as shown in Fig.3. Although the drying air temperature was noticed to be changed with the initial paddy moisture content by the operator during dryer operation at the plant site, it is crucial to emphasise that selecting appropriate operating temperature of the dryer in order to achieve possible maximum throughput capacity. This will be further discussed in the following section in relation to the application of simulation approach in determining suitable drying parameters.

Drying Rate

The drying rate obtained during the operation of fluidized bed dryer with corresponding operating parameters used in the two seasons are presented in Table 1. It revealed that the higher drying rate of 538 kg moisture/h was achieved at feed rate of 7.75t/h during the first season, whereas comparatively lower drying rate of 435 kg moisture/h was achieved at the higher feed rate of 9.5t/h in the second season. These drying rates are much lower compared to the results reported by Prachayawarakorn *et al*. (2005). It is noted that higher drying rate was achieved in the first season due to higher drying temperature used for the drying of comparatively higher initial moisture content paddy, as shown in Table 1. A similar phenomenon during fluidized bed drying was also reported by the other authors (e.g. Tatemoto & Sawada, 2012; Kozanoglu *et al*., 2012; Srisang *et al*., 2011). The dryer was found to be operated at a much lower capacity than the design capacity, and this justified its lower performance. During the operation of the dryer, it was noticed that the paddy flow through the intake elevator and pre-cleaner was not consistent, and this reduced the throughput capacity. In addition, high moisture and high impurities in the paddy had caused problems due to clogging and thus hindering smooth and steady throughput. However, a consistent paddy flow could be ensured through continuous inspection and necessary adjustments to the elevator and pre-cleaner.

TABLE 1: Drying rate of an industrial fluidized bed dryer with capacity of 25 t/h during two operating seasons

Operational season	Paddy initial mc (%db)	Paddy mc after FBD (%db)	Drying temperature (°C)	Bed air velocity (m/s)	Feed rate (t/h)	Drying rate (kg/h)
First	34-40	25-30	100-120	2.24	7.75	538
Second	27-30	20-24	78-90	2.24	9.5	435

Energy Consumption

The specific electrical and thermal energy consumption calculated from the observed data is presented in Fig.4. The specific electrical energy consumption in terms of primary energy was found as 0.97 MJ/kg water evaporated during the first season and 1.2 MJ/kg water evaporated in the case of the second season. On the other hand, the specific thermal energy consumptions were found to be 5.91MJ/kg water evaporated and 7.58 MJ/kg water evaporated, respectively,

during the first- and second-season drying processes. In almost similar drying conditions, higher electrical energy consumption was noticed in this case as compared to the results reported by other authors such as Soponronnarit *et al*. (1996b) and Prachayawarakorn *et al*. (2005a, 2005b), while thermal energy consumption was comparable with the values reported by Prachayawarakorn et al. (2005a, 2005b). Hence, it might be possible to minimize the energy consumption in the FBD of the present setup by ensuring that its operation is attained at a possible maximum capacity using suitable drying temperature.

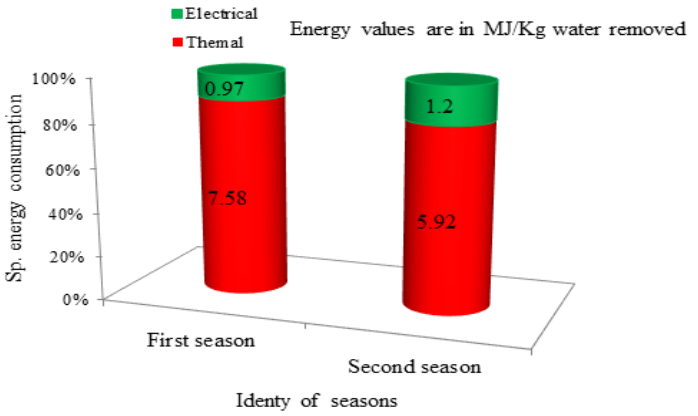


Fig. 4: Energy consumption during industrial fluidized bed paddy drying.

Paddy Quality Assessment

Table 2 presents the means with standard error mean (SEM) values of the head rice yield, whiteness, milling recovery, milling degree and transparency from the fluidized bed drying and control drying. Head-rice yield, whiteness and milling recovery of the rice samples dried by fluidized bed dryer were almost identical at acceptable milling degree and transparency to those which were dried using ambient air as the control drying over both seasons. From the statistical analysis shown in Table 2c, it is clear that the head rice yield of paddy dried by FBD during the first season was comparatively higher (around 3%) than that obtained during the second season. Higher initial moisture content paddy (34-40%) dried by FBD using higher air temperatures 100-120°C resulted in higher head rice yields, which are similar with the previous findings reported by Taweerattanapanish *et al*. (1999). Lower drying temperature (78-90°C) used by FBD for the drying of paddy having relatively lower initial moisture content (27-30%) during the second season yielded lower head rice yield and whiteness. It is noted that the use of higher drying temperature (above 90°C) has a great advantage in drying high moisture paddy by fluidization technique. Unfortunately, the head rice yield achieved from this industrial dryer is still lower than the results obtained by other researchers (see Sutherland & Ghaly, 1992; Soponronnarit*et al*., 1998). However, if tempering, the important step after the high temperature fluidized bed drying as reported by many researchers (Soponronnarit *et al*., 1999; Taweerattanapanish *et al*., 1999; Poomsa-ad *et al*., 2001; Prachayawarakorn *et al*. 2005b) could be applied, rice quality might be improved. Therefore, tempering could be adopted after fluidized bed drying in the paddy processing plant.

TABLE 2: Comparison of the milling qualities of rice

a. Milling qualities during first season					
Drying method	HRY (%)	Whiteness (%)	Milling recovery (%)	Milling degree (%)	Transparency
Control	*46.5±0.64[a]	39.3±0.21 [a]	63.6±0.91[a]	87±0.95[a]	1.56±0.03[a]
FBD	48.9±0.17 [a]	39.2±0.18 [a]	64.1±0.47 [a]	86±1.0[a]	1.56±0.09 [a]
b. Milling qualities during second season					
Drying method	HRY (%)	Whiteness (%)	Milling recovery (%)	Milling degree (%)	Transparency
Control	44.6±0.20 [a]	37.1±0.15 [a]	67.8±0.36 [a]	77±0.29 [b]	1.90±0.0 [a]
FBD	46.1±0.29 [a]	37.7±0.58 [a]	67.3±0.49 [a]	86±1.0 [a]	1.60±0.03 [b]
c. Comparison of milling qualities of fluidized bed paddy drying between two seasons					
Drying method	HRY (%)	Whiteness (%)	Milling recovery (%)	Milling degree (%)	Transparency
FBD-first season	48.9 ±0.16 [a]	39.33 ±0.21 [a]	64.06 ±0.47[ba]	86 ±1[a]	1.56 ±0.09[a]
FBD-second season	46.1 ±0.29 [b]	37.7 ±0.58 [b]	67.03 ±0.49 [a]	84.7 ±0.25[a]	1.65 ±0.02[a]

FBD, Fluidized bed drying.*Mean values ± standard error mean. a-d The test values: Same letters for the different quality attributes in each column mean that the values are not significantly different ($p > 0.05$).

TABLE 3: Simulated final moisture content during drying with industrial fluidized bed dryer of 25 t/h design capacity

Drying air temperature, °C	First season			Second season		
	At capacity (feed rate)			At capacity (feed rate)		
	7.75 t/h	10 t/h	15 t/h	10 t/h	15 t/h	20 t/h
90	*-	-	-	24.88	26.02	26.69
100	-	-	-	24.32	25.52	26.25
110	-	-	-	23.80	25.05	25.82
120	26.67	27.77	29.33	23.32	24.60	25.82
130	26.16	27.26	28.83	-	24.19	25.40
140	25.70	26.78	28.37	-	23.79	25.00
150	25.28	26.35	27.92	-	23.42	24.25
160	24.92	25.69	27.51	-	-	-

(Usual average initial paddy moisture content (mc) during first and second season were taken as 36% (db) and 30 % db, respectively, while bed height of 10 cm and bed air velocity of 2.25 m/s were maintained for all the simulation runs in both seasons;

*- values were not calculated as these are either much more higher or lower than the desired final moisture (bolded values) of 24-25%)

Application of Simulation Approach in Determining Suitable Drying Parameters

Previous researchers (Pomona-ad *et al*., 2001; Soponronnarit *et al*., 1994; 1996a; Sutherland & Ghaly, 1990) reported that for quality rice after fluidized bed drying, paddy final moisture content should not be lower than 24-25 %. Hence, in order to achieve this desired level of the final moisture content, a good number of drying simulation runs were done at different feed rates and drying air temperatures. The simulation results are shown in Table 3. It must be noted that the approach used in determining suitable operating parameters was based on simulation results. During the first season, when the initial moisture content was very high (36 %), it is clearly noticed that the dryer could reduce moisture content to 23-24 % when operating at higher drying temperatures of up to 160°C with low throughput capacity (i.e., feed rate) of less than 7.75 t/h. However, if the final moisture content to be achieved ranged from 24 to 25%, the dryer could then be operated at the maximum throughput of 7.75 t/h. By using higher drying temperature of 160°C, throughput capacity could be increased in the range of 10 and 15 t/h to result in the final moisture content of 26% and 27.51%, respectively, as can be seen at the bottom rows of Table 3. In the case of the second season, when the initial moisture content is comparatively lower (30 %), the dryer could reduce moisture content of paddy to 23-24%, even at 110°C. Although the capacity achieved was only 10 t/h, it is still higher than the drying capacity of the first season with higher initial moisture. From Table 3, it is clear that the dryer could be operated at higher capacity of 20 t/h to reduce moisture content to around 24-25% from 30% by using the drying temperature of 150°C. Consequently, if the dryer is run at its maximum possible throughput capacity, both specific electrical and thermal energy consumptions would be minimal. However, this simulation approach can be used to select suitable combination of operating parameters for possible maximum throughput capacity through determination of expected final moisture with reasonable energy consumption.

CONCLUSION

An assessment on the actual operating status and final quality of product during paddy drying with the commercial fluidized bed dryer was carried out. The actual throughput capacity was found to be less than half of its design capacity, thus exhibiting lower performance of the dryer. Nevertheless, the dryer performed better in yielding around 3% higher head rice yield during drying paddy with higher initial moisture content of 34-40% using higher temperature of 100-120°C in the case of the first season than that of the low initial moisture paddy of 27-30% using lower temperature of 78-90°C in the second season. The dryer exhibited the drying rates of 538 kg moisture/h and 435 kg moisture/h for the first and second seasons, respectively. The thermal and electrical energy consumptions in terms of primary energy in MJ/kg water removed were obtained as 7.57 and 0.97, respectively, for the first season and 5.92 and 1.2 for the second season. Hence, when the initial moisture content is higher than 28% dry basis, it is recommended that inlet-air temperature should be higher than 100°C in order to achieve higher head rice yield. Also, throughput capacity of the dryer could be increased by using higher drying air temperature based on the initial paddy moisture content. In addition, continuous supervision of intake elevator, pre-cleaner and rotary feeder is necessary to avoid any inconsistent paddy flow due to variation of impurities and moisture content in freshly harvested paddy. Simulation

approach, which was found to be reliable in predicting the average outlet paddy moisture content and outlet air temperature during the drying process, could be readily used in determining suitable operating parameters to achieve possible maximum throughput capacity of industrial fluidized bed dryer as it allows rapid calculation of the required changes in the manipulated variables in response to any sudden change of dryer condition.

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Effects of Cooking Temperature in Repetitive Cooking-Chilling Cycles on Resistant Starch Content and Quality Characteristics of Fish Crackers

Nor, M. Z. M.[1], Talib, R. A.[1,4*], Noranizan, M. A.[2], Chin, N. L.[1] and Hashim, K.[3]

[1]*Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia*

[2]*Department of Food Technology, Faculty of Food Science and Technology, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia*

[3]*Malaysian Nuclear Agency, Bangi, 43000 Kajang, Selangor, Malaysia*

[4]*Halal Products Research Institute, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia*

ABSTRACT

This work describes the effects of different cooking temperatures in repetitive cooking-chilling (RCC) process on resistant starch (RS) content in fish crackers prepared in a ratio of 1:1 fish to sago starch formulation. In this work, three sets of four RCC cycles were performed on fish crackers, in which each set was cooked at fixed temperatures of 100, 115 and 121°C, respectively. The chilling temperature was fixed at 4°C in all cases. Subjecting the fish crackers to a higher cooking temperature for up to 4 cycles of RCC can increase the RS content. However, quality degradation was observed in the characteristics of the fish crackers. During the first RCC cycle, cooking at a higher temperature had caused the crackers to crack and burst. Besides, defragmentation to the shape of the fish cracker gels was also observed during the first RCC cycle, coupled with softer texture and high moisture content. When the products were subjected to frying, their linear expansion decreased, the texture became harder and the colour turned darker. This work demonstrated that the application of higher cooking temperature up to 4 RCC cycles was able to enhance the RS content in the fish crackers, but it was less able to attain the product's perfect shape. On the contrary, fish crackers that were exposed to lower cooking temperatures contained lower RS but with less shape damage.

Keywords: Resistant starch, fish cracker, cooking, chilling, temperature

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E-mail addresses:
zuhair@upm.edu.my (Nor, M. Z. M.),
rosnita@upm.edu.my (Talib, R. A.),
noraadzahan@upm.edu.my (Noranizan, M. A.),
chinnl@upm.edu.my (Chin, N. L.),
kbhashim@nuclearmalaysia.gov.my (Hashim, K.)
*Corresponding Author

INTRODUCTION

Fish crackers or "keropok" are well-known amongst Malaysians as one of the most popular snacks. Fish crackers consist of dough formed from a mixture of starch, fish flesh and water, which is then shaped and boiled to gelatinize the starch, cut into thin slices

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and dried before marketed. It is commonly fried before being served. Resistant starch (RS) is a type of starch that passes through small intestine without being digested, similar to dietary fibre. It is reported that the content of RS can be improved in fish cracker production via a repetitive cooking-chilling (RCC) process. However, there are some minor drawbacks of the quality characteristics of chilled and fried products that have been reported (Nor *et al*., 2014). The repeated cooking-chilling cycles at the cooking temperature of 100°C and chilling of 4°C increased the extent of starch gelatinization for each of the successive cooking cycles and promoted retrogradation upon cooling. This promoted the formation of resistant starch type 3. Resistant starch type 3(RS3) is a resistant starch formed from retrograded starch during food processing. Other types of RS include resistant starch type 1 (RS1; i.e. physically inaccessible to digestive enzymes found in whole or partly ground cereal grains, seeds and legumes), resistant starch type 2 (RS2; i.e. a natural crystalline resistant starch granules found in unripe banana and some legumes), and resistant starch type 4 (RS4; i.e. a chemically modified resistant starch). In another study, Nor *et al*. (2012) reported that fish crackers formulated from sago starch had the highest RS content in the dried samples as compared to tapioca and wheat starches due to higher amylose content. There is a possibility to improve the RCC by increasing its cooking temperature to above 100°C in order to obtain better RS yield in the treated products. This approach is based on the previous studies which reported that high temperature treatment via autoclaving and pressure cooking produces better RS yield in the food processed. The RS yield from wheat starch increased progressively as the autoclaving temperature was increased from about 2.5% at 100°C to about 9% at 134°C (Berry, 1986). Parchure and Kulkarni (1997) further reported that a 10-minute pressure cooking of native rice and amaranth produced RS content which was 1.7 times higher compared to those boiled for 15 minutes. Meanwhile, the repetition of heat treatment and cooling at different heating temperatures was undertaken by Sievert and Pomeranz (1989) who reported that by 20 repetitions of autoclaving (121°C, 134°C and 148°C for 1 hour) and cooling (4°C for overnight) of amylomaize VII starch, the RS level was raised from 20% to over 40%. Considering the potential of producing fish crackers with high RS content via higher cooking temperature in the RCC treatment, the objective of this study was therefore to investigate the effects of different cooking temperatures in the RCC treatment on the RS content and other quality characteristics of fish crackers.

MATERIALS AND METHODS

Materials

Fresh sardines (Sardina pilchardus) were purchased from a wet market in Serdang, Malaysia, while salt, sugar and monosodium glutamate (MSG) were purchased from a grocery store. Sago starch (Metroxylon sagu) was purchased from Songiing Holding Sdn. Bhd. in Sibu, Malaysia, whereas filtered ice water was obtained from the laboratory.

Preparation of the Fish Crackers

Fig.1 illustrates the fish cracker preparation methods using a modified version of the procedure previously described by Siaw *et al*. (1985). The fish cracker formulation is as follows: 1:1

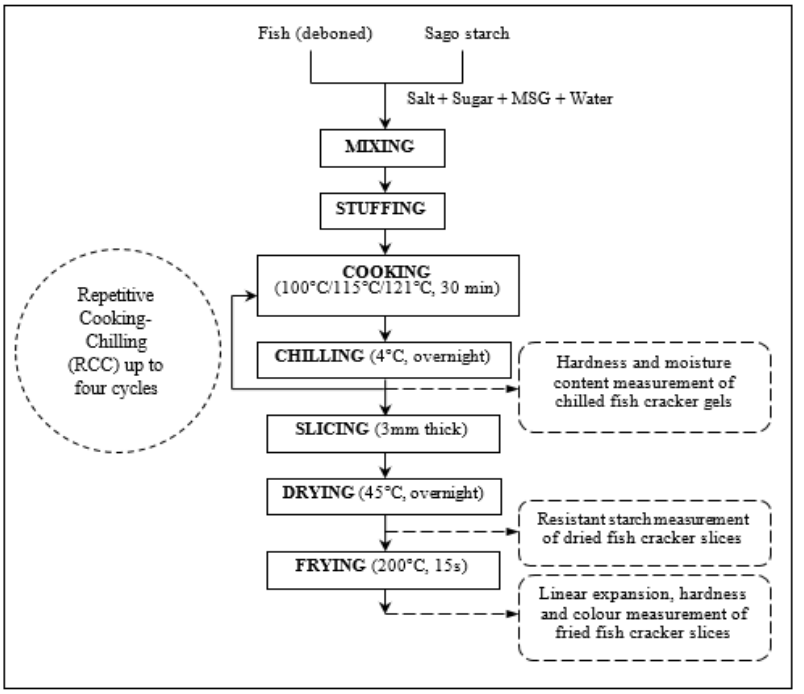

Fig.1: Application of different cooking temperatures in the RCC cycles in fish cracker production

fish to sago starch ratio (1kg: 1kg), 2% salt, 1% sugar, 0.1% monosodium glutamate (MSG) and 30% ice water (% weight based on the total weight of starch and wet fish). Firstly, the fresh sardines were manually beheaded, degutted and deboned using a knife to obtain fresh flesh after the fish had been transported from the local wet market to the laboratory. The fish flesh was ground using a meat grinder (National, Japan) before being transferred into a silent cutter (Khin Shang Hoo Iron Works, Taiwan) and mix it with salt, sugar and MSG. Then, ice water and sago starch were added until the mixture formed homogenous dough. This mixing process took approximately 20 minutes. Meanwhile, a sausage stuffer (F-Dick, Germany) was used to stuff the dough-like mixture into circular cellulose casings (2.5 cm diameter and 12 cm length). Both ends of the stuffed casings were tied with cotton strings, and the stuffed rods were cooked for 30 minutes at fixed temperatures of 100°C, 115°C and 121°C, respectively (Kyaw *et al*., 2001). After cooking, the cooked gels were immediately immersed in ice water to prevent shrinkage and to facilitate separation from the casings. The gels were then chilled overnight in a cold room at 4°C to complete one cooking-chilling cycle. The cooking-chilling cycle was repeated up to four times. The chilled gels were manually cut into 3 mm slices, which were later oven dried at 45°C overnight until the final moisture content was around 8–10%. The dried crackers (already considered a half finished product) were deep fried in palm oil at 200°C for 15 seconds until they fully expanded. These fried crackers were considered as the final products. Samples were taken after chilling (in gel form), after drying (in dried slice form) and after frying (in expanded cracker form) to obtain measurements. The protein contents for the single cooking-chilling fish crackers with similar formulation of tapioca, wheat and sago starches were found to be 15.6%, 24.9% and 15.7%, respectively, with a standard fat content of 0.4% (Yu, 1991).

Measurement of Resistant Starch in the Dried Fish Crackers

Protein removal was performed prior to the RS analysis (Goñi *et al*., 1996), while the Resistant Starch Assay Analysis (Megazyme International Ireland Ltd. Co., Ireland) was used for the RS content determination. The method included the incubation with α-amylase (37°C, 16 hours) to hydrolyse the digestible starch, the solubilisation of precipitate using 2M KOH, the incubation with amyloglucosidase (50°C, 30 minutes) and the quantification of glucose using glucose oxidase/peroxidase reagent.

Measurement of Instrumental Hardness and Moisture Content in the Chilled Fish Crackers

The chilled gels were maintained at room temperature (32°C) for 2 hours prior to the analysis (Cheow *et al*., 2004) and cut into cylinders (25 mm diameter and 25 mm length). The hardness test was performed using a texture analyser (Stable Micro System, TA.XT.Plus, UK). The samples were placed on a heavy-duty platform and deformation was applied onto the sample using a compression probe (P/36R) 35 mm in diameter at a constant speed of 2 mm/s. The compression tests were set at a distance of 50% deformation. Force-deformation data were recorded to determine the textural characteristics of the samples. The hardness force was obtained from the absolute value of the peak height and the hardness measurements were performed in quadruplicate.

The moisture content was determined using the oven drying method (AOAC, 2000). The chilled gels were divided into two sections corresponding to the centre and periphery of the cylindrical rods and each section was used to determine the moisture content of the gel. The centre portion was 15 mm in diameter. A balance of the centre portion was used to determine the peripheral portion and three samples were used for each measurement.

Measurement of Linear Expansion, Hardness and Colour in Fried Fish Crackers

The linear expansion percentage of the fried fish cracker sample was determined according to Yu *et al*. (1981). Three parallel lines were drawn on the dried fish crackers using a marker. The length of each line was measured before and after frying. The calculation for the linear expansion is as follows:

$$\% \text{ linear expansion} = \quad \times 100 \qquad [1]$$

The textural analysis of the fried fish cracker sample was performed using a texture analyser (Stable Micro System, TA-XT2i, United Kingdom). The samples were placed on a covered hollow cylindrical base (25 mm inner diameter, 1.5 mm thickness, stainless steel). Deformation was applied using a 5 mm spherical compression probe (P/0.25S) at a constant speed of 3 mm/s until the sample cracked. The maximum force of break was used as an indicator of the sample's hardness. The average value was calculated from 25 measurements. The colour of the fried samples was determined using a colour reader (Konica Minolta, CR-10, Japan). Ten measurements were used to determine lightness (L*), redness (a*) and yellowness (b*), and the average value was recorded for each sample.

Observation of the Physical Condition after Cooking

Observations were carried out on the physical condition of the chilled fish cracker gels subjected to a single cycle of repetitive cooking-chilling (RCC). Each sample was classified according to the attributes listed in the Appendix.

Data Analysis

All the tests were conducted in triplicates, and a statistical analysis was performed using Microsoft Excel 2007 (Vista Edition, Microsoft Corporation, USA) and Statistical Analysis System (SAS) software (Version 9.2, SAS Institute, Inc., USA). A significance level of $\alpha = 0.05$ was used throughout the analysis, and p-values larger than α ($p>0.05$) indicated that the difference between the treatments was not significant. When a significant difference was found from the ANOVA results, the treatments were compared using the t-test.

RESULTS AND DISCUSSION

Effects of Different Cooking Temperatures in RCC on the Resistant Starch Content of Dried Fish Crackers

The results of one to four RCC cycles on the RS, which yielded dried fish crackers under different cooking temperatures are shown in Table 1. The one-way ANOVA test performed showed that the RCC treatment indicated significant differences ($p<0.05$) in resistant starch formation in each cycle for the samples at different cooking temperatures. Similar trends also reported by Nor *et al*. (2014) for different formulation samples cooked at 100°C. The repeated cooking and chilling processes facilitate further gelatinization and retrogradation of the fish crackers, thus promoting the formation of RS.

TABLE 1: Average RS values in the dried sago starch based-fish crackers for each cooking-chilling cycle at different cooking temperatures

Temperature	Resistant starch in each cycle (%, dwb)			
	1	2	3	4
100°C	3.07 ± 0.02^{a1}	3.44 ± 0.05^{b1}	3.68 ± 0.11^{bc1}	3.81 ± 0.12^{c1}
115°C	3.04 ± 0.07^{a1}	3.56 ± 0.26^{b1}	3.74 ± 0.20^{bc1}	3.99 ± 0.11^{c23}
121°C	3.36 ± 0.10^{a2}	3.79 ± 0.20^{b1}	3.99 ± 0.23^{bc1}	4.23 ± 0.20^{c3}

*Mean of triplicate determinations ± standard deviation

**Means within a row with different letters are significantly different ($p<0.05$)

***Means within a column with different numbers are significantly different ($p<0.05$)

When the cooking temperatures were compared, the ranking of the RS content in the dried samples exposed to the RCC treatment is as follows: 121°C > 115°C > 100°C. Sago starch-formulated fish cracker cooked at 121°C up to four cycles of RCC exhibited the highest RS yield, which was 4.23% (dwb). This value is higher as compared to the RS content in selected commercial fish crackers available in the Malaysian market ranging from 0.91%to 1.77% (dwb)(Nor *et al*., 2012).

The comparison between different cooking temperatures showed that higher temperatures would produce appreciably higher RS. This is in agreement with Shin-Kyung *et al*. (1997) who reported that the heating of corn starches at 121°C provided higher yield of RS than it was at 100°C. In addition, Berry (1986) reported that the yield of RS from wheat starch increased progressively as the autoclaving temperature increased, i.e. from about 2.5% at 100°C to about 9% at 134°C.

Sievert and Pomeranz (1989) reported that although the differences were small, amylomaize VII starch autoclaved at 134°C had a higher RS content as compared to the same samples autoclaved at 121°C after completing four autoclaving-cooling cycles. Nevertheless, it was also reported that at a higher autoclave temperature (148°C), the amount of RS obtained was lowered. However, this finding was not further discussed.

The increase in RS as the cooking temperature increased might be due to more amylose leached out from the starch granules. Mohd Adzahan *et al*. (2010) reported that approximately 96% of amylose leached out at 120°C compared with approximately 88% amylose being leached out at 100°C when solubilised sago starch was heated for 1 hour. In addition, higher cooking temperature also caused thermal degradation of amylopectin into shorter amylopectin chains that could be attributed to the formation of RS since the entanglement of retrograded amylopectin molecules might reduce enzyme susceptibility (Eerlingen & Delcour, 1995). Jiranuntakul *et al*. (2012) observed thermal degradation of amylopectin molecules when subjected to heat-moisture treatment at 120°C and 140°C as compared to that at 100°C in waxy starches. The degradation of amylopectin is indicated by smaller molecules in the starch chain distribution. However, this is not shown in the current work.

Since the current study only used a small scale autoclave which has safety limit of 121°C, the treatment at a higher autoclaving temperature could not be performed. Despite this limitation, the application of higher temperature would lead to a higher processing cost, so limiting the autoclaving temperature at 121°C would be the best choice. The effects on other quality characteristics are also a major concern.

Effects of Different Cooking Temperatures in RCC on the Hardness and Moisture Content of Chilled Fish Cracker Gels

Fig.2 displays the effects of the repetitive cooking-chilling (RCC) treatment on the hardness of chilled fish cracker gels at different cooking temperatures. There were increases in the hardness values from cycle one to cycle two when the samples were cooked at 115°C and 121°C. However, the values decreased in the subsequent cycles, leaving the hardness values after completing the four cooking-chilling cycles to be lesser than the values in the first cycle. However, the values are still higher than the samples that were cooked-chilled for four cycles at 100°C. In overall, the samples cooked at the temperatures higher than 100°C produced higher hardness values. Kyaw *et al*. (2001) reported an increase in the gel strength of the fish cracker gels made from wheat starch when cooked under the cooking temperatures ranging from 100°C to 121°C. They proposed that this finding might be due to the swelling and water uptake of starch during gelatinization upon heating. In contrast, as the fish cracker gels were made based on tapioca starch, they found a decrease in the strength of the fish cracker gels,

although being cooked in the same range of the cooking temperatures. This could possibly be due to the population of fragmented starch granules that increased as the cooking temperature increased. However, their study only focused on a single cooking cycle.

The moisture contents in the centre and periphery parts of each sample treated with RCC at different cooking temperatures are displayed in Fig.3 (a) and Fig.3 (b), respectively. The RCC treatment caused a decrease in the moisture content for each sample from cycle one to cycle four. It was observed that the samples cooked at the temperatures higher than 100°C had lower moisture content (centre and periphery), which was probably due to their compact structure that supports the findings of hardness values in Fig.2.

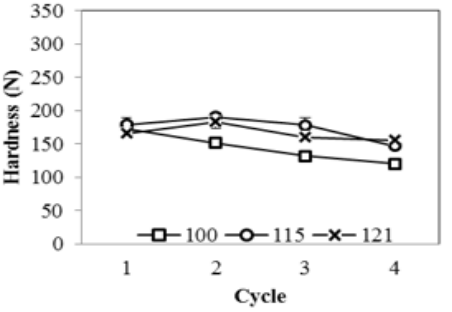

Fig.2: Hardness values of chilled sago starch based-fish cracker gels when subjected to cooking-chilling repetitions at different cooking temperatures

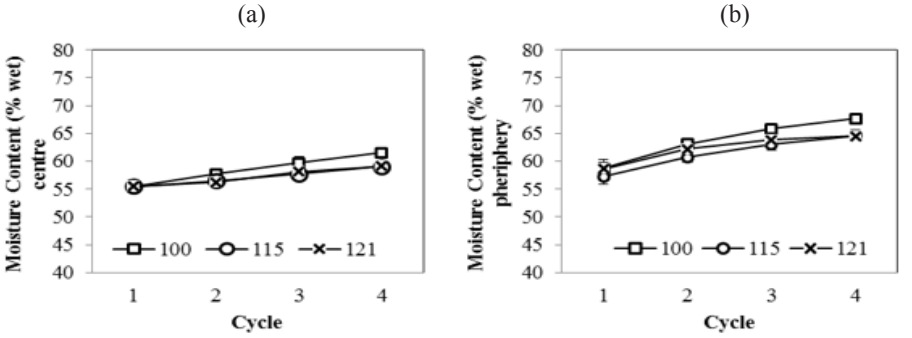

Fig.3: Moisture content values–centre (a) and periphery (b) of the chilled sago starch-based fish crackers when subjected to repetitive cooking-chilling cycles at different cooking temperatures

Effects of Different Cooking Temperatures in RCC on the Linear Expansion, Hardness and Colour of Fried Fish Crackers

Table 2 shows the linear expansion values of the fried fish crackers treated with RCC at different cooking temperatures. Similar to the findings of Nor *et al*. (2013), the RCC treatment caused a decrease in the linear expansion of the treated fish crackers. At higher cooking temperatures (115°C and 121°C), the samples became less expanded after frying. This could probably be due to the extreme effects of the high temperature treatment that caused more damages to the starch granules. Kyaw *et al*. (2001) reported that the cooking of fish crackers made of tapioca starch at a temperature above 108°C would cause a reduced expansion due to the water being released into the fused fish and starch structure.

TABLE 2: Average linear expansion values in the fried sago starch based-fish crackers for each cooking-chilling cycle at different cooking temperatures

Temperature	Linear expansion in each cycle (%)			
	1	2	3	4
100°C	77.60±2.95^{a1}	71.88±1.32^{b1}	66.34±4.49^{c1}	59.45±1.91^{d1}
115°C	73.10±5.77^{a1}	67.10±8.21^{ab12}	61.91±8.59^{ab12}	59.79±3.90^{b1}
121°C	73.79±1.51^{a1}	61.04±3.27^{b2}	54.58±2.66^{c2}	48.73±1.41^{d2}

*Mean of triplicate determinations ± standard deviation

**Means within a row with different letters are significantly different ($p<0.05$)

***Means within a column with different numbers are significantly different ($p<0.05$)

The most likely factor affecting the reduction in the linear expansion is the formation of resistant starch in the fish crackers. The treated samples had less expandability due to the strong hydrogen bonding involved in the RS formation. Furthermore, extensive heating in excess water through repetitions of the cooking-chilling process had caused the maximal swelling and the rupture of the starch granules which resulted in a reduced amount of expansion.

Fig.4 displays a comparison of the hardness values of fried sago-starch-based fish crackers treated at different cooking temperatures of RCC. Repetition of cooking-chilling cycles caused an increase in the hardness value of each sample. The samples cooked at higher cooking temperatures tended to become harder after frying. As hardness and expansion are inversely proportional (Yu & Low, 1992), the same trend was also observed by comparing the LE values in Table 3 with the hardness values shown in Fig.4. Sago starch fish crackers treated with four cycles of RCC at 121°C demonstrated the lowest linear expansion value (48.79%) although they had the highest hardness value (21.1 N). Hardness has often been related to the crispiness in crispy foods. Low hardness shows high crispiness score and this meets consumers' preferences in the selection of fish crackers (Huda *et al*., 2009).

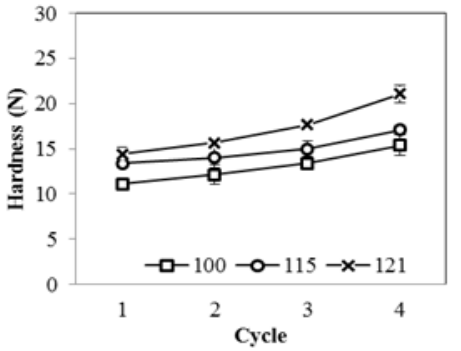

Fig.4: Hardness values of the fried sago starch based-fish crackers when subjected to cooking-chilling repetitions at different cooking temperatures

Fig.5 (a), (b) and (c) show the comparison of colour values for the fried sago starch fish crackers treated by the RCC at different cooking temperatures. RCC caused a reduction in L* for all the samples from the first to the fourth cycle, but no significant changes were observed for a* and b*. It was also observed that higher cooking temperatures produced fish crackers with lower L* and higher a*. All the changes in the colour of the products were mainly associated with the expansion ability of the samples (Altan *et al*., 2008). Darker fish crackers were obtained from less expandable products which could be related to their compact structure. To date, there has been no report on assessing consumers' preferences for specific colour values of fish crackers although some researchers have observed that lighter coloured fish crackers are generally preferred (Huda *et al*., 2010).

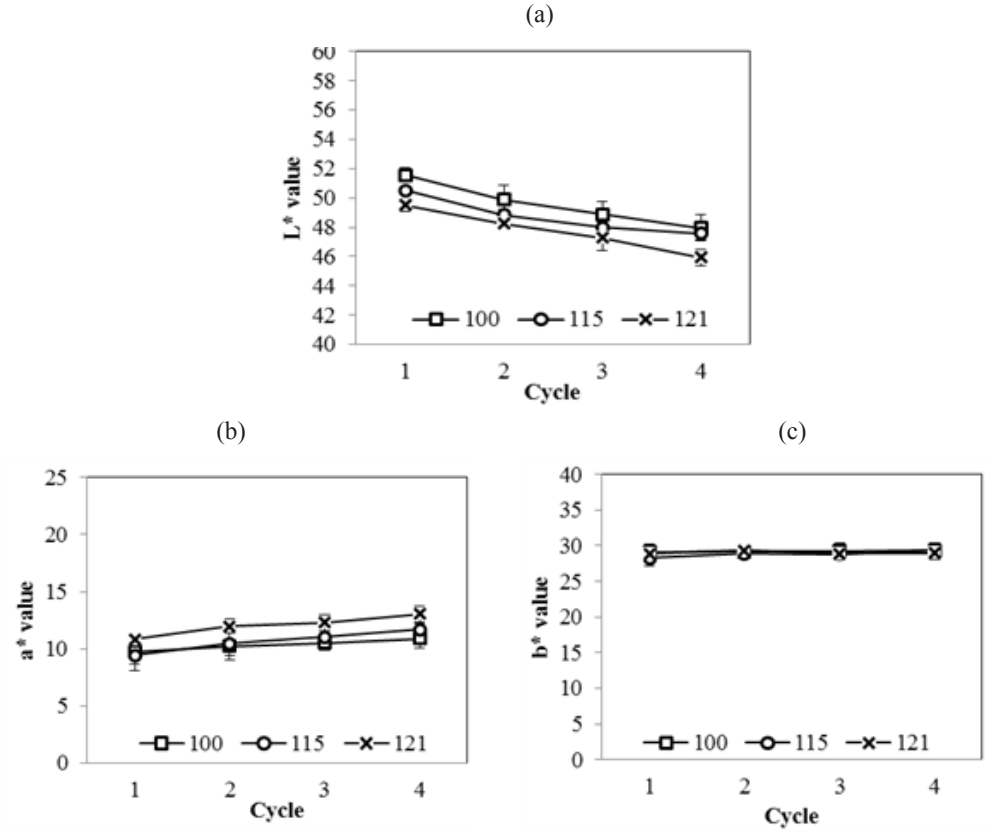

Fig.5: (a), (b) and (c). Colour values of the fried sago starch-based fish crackers when subjected to cooking-chilling repetitions at different cooking temperatures

Effects of Different Cooking Temperatures in RCC on the Physical Conditions of the Chilled Fish Cracker Gels

Table 3 shows the percentage of different physical conditions of the chilled fish cracker gels after being subjected to the first cycle of the RCC treatment. The percentage was calculated based on the number of samples identified in each attribute over the total samples cooked

in the same batch. From the observation made, it could be seen that the chilled fish cracker gels cooked at 100°C were almost in perfect condition after the treatment compared to those cooked above 100°C. This finding is important because gels in a perfect condition will facilitate further processes.

Fish crackers cooked at 100°C were observed to have almost a perfect shape. Meanwhile, higher cooking temperatures that require higher pressure above the atmospheric pressure during cooking may cause physical damage to the gels. Fish cracker gels cooked at 115°C (10 psi) produced ~77% perfect condition gels, whereas only 36% of the samples were in the perfect condition after being subjected to cooking at 121°C (15 psi). It is believed that any further increase of the cooking temperature may cause more damages to the shape of the samples. Kyaw *et al*. (2001) studied the effects of pressure cooking on the microstructure and expansion of the fish crackers cooked up to 121°C. They also highlighted the gradual increases of the tapioca and wheat starch swelling power with an increase in the cooking temperature before the granules were hydrated with simultaneous loss of their polarization crosses and burst. In this study, the observation was done only during the first RCC cycle because the gels were initially formed at this stage, while the subsequent cycles had lesser effects on the shape formation of the gels.

TABLE 3 : Conditions of the chilled fish cracker gels after the first repetitive cooking-chilling cycles

	Physical condition of the gels after the first RCC cycle (%)		
Attribute	100°C	115°C	121°C
Perfect	98.3	77.0	36.1
Minor Crack	0.0	0.00	27.7
Major Crack	0.0	7.5	14.1
Minor Burst	1.7	7.5	12.4
Major Burst	0.0	4.0	4.8
Fragmented	0.0	4.0	4.9

CONCLUSION

From the experiment, it is apparent that as the cooking temperature increases, the RS content in the fish crackers also increases. The cooking temperature at 121°C with the repetition of four cycles resulted in the highest RS content. However, it could be seen that there were some defects on the physical characteristics of the fish crackers such as cracks and burst. Despite these, the process of cooking at high temperature with the repetition of four cycles is still meaningful to further promote the nutritional aspects of the fish cracker, apart from highlighting the fact that fish crackers are processed products that contain high protein, high carbohydrate and high calcium.

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APPENDIX

List of physical condition attributes and descriptions of the chilled fish cracker gels after single RCC

No.	Attribute	Description	Illustration
1	Perfect	Sample was in perfect shape after cooking and chilling without any obvious deformation	
2	Minor crack	Sample was found to have cracks not more than 50% of the whole body	
3	Major crack	Sample was found to have a crack more than 50% of the whole body	
4	Minor burst	Sample which burst less than 50% of the whole body	
5	Major burst	Sample which burst more than 50% of the whole body	
6	Fragment	Sample was found to be fragmented at some parts/whole part of the body	

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Effects of Milling Methods on Tensile Properties of Polypropylene / Oil Palm Mesocarp Fibre Biocomposite

Nordin, N. I. A. A.[1,2], Ariffin, H.[1,3*], Hassan, M. A.1, Ibrahim, N. A.[4], Shirai, Y.[5] and Andou, Y.[5]

[1]Department of Bioprocess Technology, Faculty of Biotechnology and Biomolecular Sciences, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

[2]Faculty of Chemical and Natural Resources Engineering, Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300 Kuantan, Pahang, Malaysia

[3]Laboratory of Biopolymer and Derivatives, Institute of Tropical Forestry and Forest Products, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

[4]Department of Chemistry, Faculty of Science, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

[5]Graduate School of Life Science and System Engineering, Kyushu Institute of Technology, 2-4 Hibikino, Wakamatsu, Fukuoka 808-0196 Japan

ABSTRACT

The objective of this study was to evaluate the effects of milling methods on tensile properties of polypropylene (PP) / oil palm mesocarp fibre (OPMF) biocomposites. Two types of mills were used; Wiley mill (WM) and disc mill (DM). Ground OPMF from each milling process was examined for its particle size distribution and aspect ratio by sieve and microscopic analyses, respectively. Results showed that DM-OPMF had smaller diameter fibre with uniform particle size compared to the WM-OPMF. Surface morphology study by SEM showed that DM-OPMF had rougher surface compared to WM-OPMF. Furthermore, it was found that PP/DM-OPMF biocomposite had higher tensile strength compared to PP/WM-OPMF, with almost two-fold. Thus, it is suggested that small diameter and uniform size fibre may improve stress transfer and surface contact between the fibre and polymer matrix and cause well-dispersion of filler throughout the polymer resulted in better tensile strength of PP/DM-OPMF compared to PP/WM-OPMF biocomposite. Overall, it can be concluded that disc milling could serve as a simple and effective grinding method for improving the tensile properties of biocomposite.

Keywords: Biocomposite, disc mill, oil palm mesocarp fiber, polypropylene, tensile properties, Wiley mill.

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E-mail addresses:
idamalina@yahoo.com (Nordin, N. I. A. A.),
hidayah@upm.edu.my (Ariffin, H.),
alihas@upm.edu.my (Hassan, M. A.),
alihas@upm.edu.my (Ibrahim, N. A.),
alihas@upm.edu.my (Shirai, Y.),
alihas@upm.edu.my (Andou, Y.)
*Corresponding Author

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INTRODUCTION

Malaysia is the largest producer of palm oil contributing to 46% of the total world palm oil supply (MPOC, 2010). From the palm oil industries, about 77 million tonnes (dry weight) of biomass are generated annually consisting of 45 million tonnes of frond, 7 million tonnes of empty fruit bunch (EFB), 14 million tonnes of trunk and 11 million tonnes of mesocarp fibre (Ng *et al*., 2010). Research has shown that oil palm biomass, especially EFB, has potential for biocomposite production (Khalid *et al*., 2008; Rozman *et al*., 2003). However, studies by Phattaraporn *et al*. (2011) and Thawien (2009) showed that oil palm mesocarp fiber (OPMF) also appeared to hold great potential in biocomposite industries, despite of less attention being given for effective utilization of OPMF. Currently, OPMF is treated as a waste product, which is burnt as fuel boiler inside the palm oil mill and as soil mulching in the plantation.

Grinding process is crucial in biocomposite production, especially if the biocomposite is to be prepared by compounding using internal mixer or extruder. This is done to allow a better wetting to the filler during processing. Shinoj *et al*. (2011) reported that a better interaction between fibre particles and polymer matrix could improve mechanical properties of the biocomposites. Mechanical properties of biocomposite reinforced by short lignocellulosic fibres can be contributed by several factors such as fibre particle size, aspect ratio and fibre/matrix adhesion. Fibre with high aspect ratio, i.e. long fibre with small diameter will give a higher surface area and hence, provide a larger contact area for the interaction of fibre particles and polymer matrix. High aspect ratio fibres bind better to the polymer compared to thick and short fibres. A typical sized fibre used for making biocomposite is less than 500 μm (Siyamak *et al*., 2012; Bledzki & Faruk, 2006; Mikushina *et al*., 2002).

Grinding is a common unit process to produce fine particles. Such fine particles are required for the utilization of natural lignocellulosic materials to enhance the accessibility of reactive agents (Bitra *et al*., 2009) and to strengthen the interaction between different materials due to a higher surface area (Juliana *et al*., 2012). There are different types of grinder with different fibre size reduction mechanisms, which result in different sizes of fibre particles. The common milling methods used are Wiley mill (WM) and disc mill (DM). Both the mills have different modes of action; WM is used to reduce the size of fibre by cutting, while disc milling involves a combination of actions which include compression, rubbing action, shearing and cutting.

As different mills may have different fibre breaking mechanisms, it is interesting to study the effects of fibre milling methods on the properties of the fibre and subsequently, its biocomposites. In this study, the effects of OPMF milling by WM and DM were investigated by evaluating the physical changes of OPMF in terms of particle size distribution, aspect ratio and morphological changes. Milled-OPMF obtained from both the milling techniques were then used for biocomposite preparation, while the effects of particle size distribution, aspect ratio and morphological surface on the tensile strength of the biocomposites were clarified.

MATERIALS AND METHODS

Raw Materials

OPMF was obtained from FELDA Serting Hilir Palm Oil Mill located in Negeri Sembilan, Malaysia. OPMF was disintegrated by manual washing and sorting. The OPMF was then sun dried. Samples were stored in sealed plastic bags at room temperature (±24°C) prior to further use. Polypropylene (PP) pellet (606251G112) was supplied by Polypropylene (Malaysia) Sdn. Bhd. and used as binding material.

Preparation of Oil Palm Mesocarp Fibre

Original OPMF used in this study had an average length of 20 – 30 mm. The size of OPMF was then reduced by using two different grinding methods. The first method involved the use of Wiley-type mill (Taiwan) Model CW-1 at the frequency of 50 Hz, with 5 rotating blades and a 0.5 mm screen. The second method used a disc mill (Ishiusu, Japan) Model RD 1-15, a stone type grinder with the gap between the stone fitted at the scale of 2 and operated in wet condition.

As for DM, the samples were wetted with distilled water at the ratio of 1:9 (fiber: water) in order to assist the disc-milling process. OPMF was pretreated by using the DM for 10 cycles. This was to ensure that only certain sizes of the fibre would be able to pass through the gap between the discs. After milling, DM-OPMF in slurry form (Fig.1a) was dried in an oven at 60°C to become a solid cake (Fig.1b). Dried solid cake was then crushed using stainless steels Waring blender to powderize the solid cake.

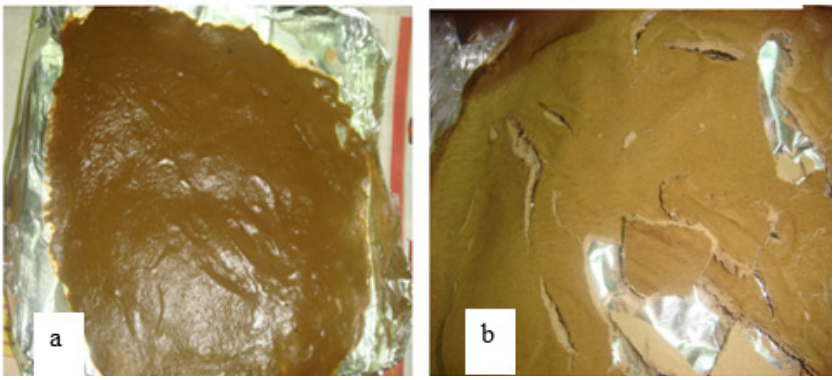

Fig.1: (a) DM-OPMF in slurry form; (b) Oven-dried DM-OPMF in the form of solid cake

Fabrication of the PP/OPMF Biocomposites

Only fibres that passed through 150 μm mesh size were used as biocomposite filler. Prior to blending, milled-OPMF were kept in an oven at 50 °C for 24 h. The blending of PP with WM-OPMF and DM-OPMF was carried out using twin screw internal mixer model Haake Rheomix Polydrive at 170°C and 50 rpm rotor speed for 10 min. The biocomposites were prepared at 20 and 50% (w/w) of WM-OPMF and DM-OPMF based on weight ratio.

Characterization

Particle Size Analysis

Particle size distribution (PSD) of ground OPMF was determined by sieve analysis. The sample was divided into fractions with different particle sizes by sieving it. Ground WM- and DM-OPMF were sieved using a vibrating screener to pass through various mesh sizes of 150, 125, 100 and 75 µm for 20 min. The sieved samples were then weighed and subjected to particle size distribution. The percentage of PSD was calculated using equation 1 below.

$$\text{PSD}\ (\%) = \frac{\text{Weight of fraction (g)}}{\text{Weight of initial sample (g)}} \times 100\% \qquad [1]$$

After sieving, WM-OPMF and DM-OPMF which passed through mesh size of 150 µm were analyzed by using microscope to observe their particle size geometry (length and diameter), followed by aspect ratio determination (length: diameter). A light microscope (Motic: Model BA310, China) provided with an image analyzer (Motic Images plus 2.0) was used to measure the length and diameter of the fibre particles.

Thermogravimetric Analysis of OPMF

Thermogravimetric analysis (TGA) was conducted on a TG analyzer model EXSTAR6000 TG/DTA6200 in order to confirm the compositional change of ground OPMF. OPMF powder sample (6 – 8 mg) was placed on an aluminium pan. The sample was heated from 50 – 550°C at a heating rate of 10ºC/min under a nitrogen flow of 100 ml/min. The corresponding weight loss (µg) and its derivative, DTG (µg/min), were recorded.

Morphological Characterization

Surface morphology of WM-OPMF and DM-OPMF samples was observed under a scanning electron microscopy (SEM, model LEO 1455 VPSEM with Oxford Inca EDX). For the SEM analysis, oven-dried samples were mounted on the stub and gold-coated for 180 sec prior to the SEM observation. The SEM micrographs were obtained with an acceleration voltage of 20 kV.

Tensile Test of PP/OPMF Biocomposite

PP/OPMF blends were moulded in a mould with the dimension of 10 x 8 cm and a thickness of 1 mm. The PP/OPMF biocomposite was compressed with electrically heated platen pressed at 170°C at a pressure of 110 kg/cm2 before it was cooled for 5 min at 50°C. The biocomposite sheet was cut into dumb-bell shape following the ASTM method D-638 type-V. Tensile test was carried out using Instron Universal Testing Machine (Model 4302 Series IX) based on ASTM D638. The test was conducted at a constant crosshead speed of 5 mm/min, load cell of 1 kN and a gauge length of 10 mm. An average of five readings was taken as the resultant value.

RESULTS AND DISCUSSION

Particle Size Analysis

PSD of WM-OPMF and DM-OPMF is shown in Fig.2. DM-OPMF showed a higher percentage of small sized particles (<75 µm) compared to WM-OPMF by almost three folds. From the same figure, it can be seen that disc milling was able to reduce the diameter of OPMF, with more than 50% of the OPMF had a particle size of <100 µm. This is in contrary with WM, whereby only 20% of the fibres had a particle size of <100 µm. This observation was contributed by the mode of action of each milling process, whereby WM was only able to shorten the length of the OPMF by cutting knife/teeth without affecting the diameter of the fibre. On the other hand, DM reduced the size of OPMF by milling between the rotating disc resulting cutting to short length, imparting and shearing to a smaller diameter and exposing microfibrils of the OPMF. From the physical appearance of the OPMF powder with size <150 µm, it can be observed that DM-OPMF had finer powder compared to WM-OPMF (see Fig.3). This observation is in agreement with the result shown in Fig.2.

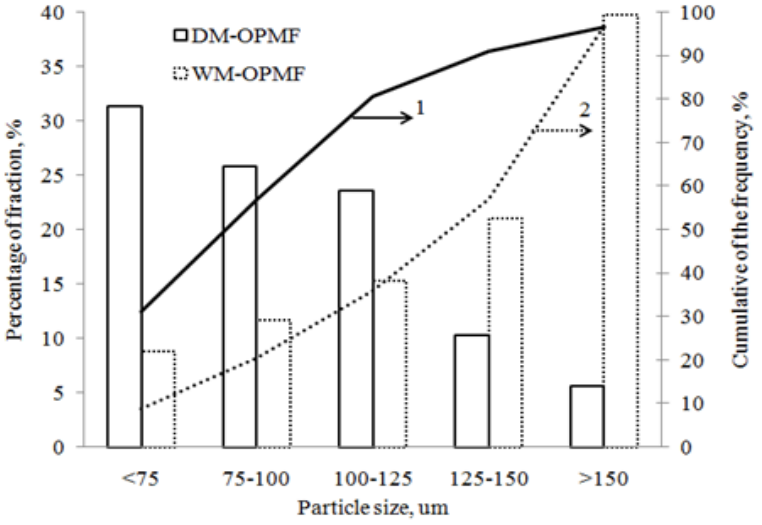

Fig.2: Particle size distribution of DM-OPMF and WM-OPMF. Bar graphs indicate fraction of particle size of the fibre (%) and lines show cumulative of the fraction (%) for, (1) DM-OPMF and (2) WM-OPMF.

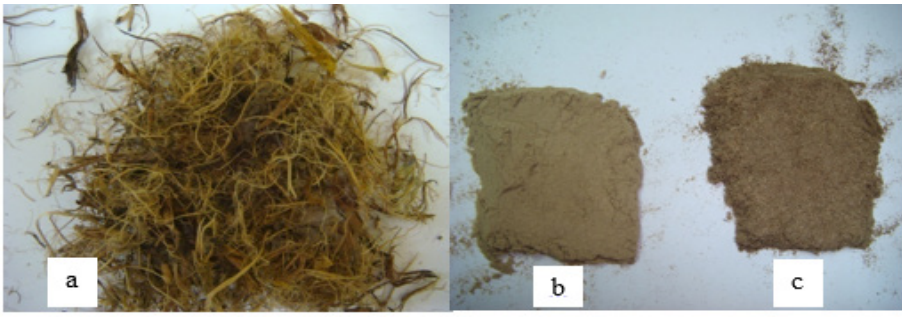

Fig.3: Physical appearance of: (a) raw OPMF; (b) powdered DM-OPMF; (c) ground WM-OPMF; after sieving to pass mesh size of 150µm.

Ground fibres were also measured for their length, L and diameter, D by microscopic analysis. The distribution of the L and D of the milled-OPMF is shown in Fig.4. A small diameter fibre indicates that the cellulose macrofibrils have been ripped into microfibrils due to mechanical action. Overall, it is seen that both L and D of the DM-OPMF converged towards the lower size with average L and D values of <200 and <50 µm, respectively. WM-OPMF, on the other hand, had a wider distribution of fibre L and D. This observation contributes to the aspect ratio values of DM-OPMF and WM-OPMF (Fig.5). Since DM-OPMF had low L and D values, this contributed to the low aspect ratio of DM-OPMF, with an average aspect ratio of 5. Even though WM-OPMF seemed to have high aspect ratio with average value of ≈10, the average length and diameter of the fibres are relatively high for biocomposite preparation (Montano-Leyva *et al*., 2013). Juliana *et al*. (2012) discussed that thin and long particles might increase the contact area between the fibres and polymer matrix, resulting in a better adhesion of the two materials. In this study, disc milling was used to get thin fibres. Overall, the particle size of disc milled-OPMF is much smaller compared to that obtained from Wiley mill.

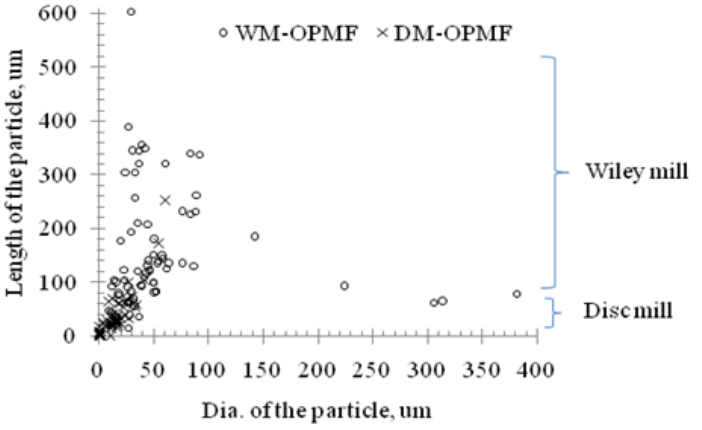

Fig.4: Distribution of diameter and length of DM-OPMF and WM-OPMF particles

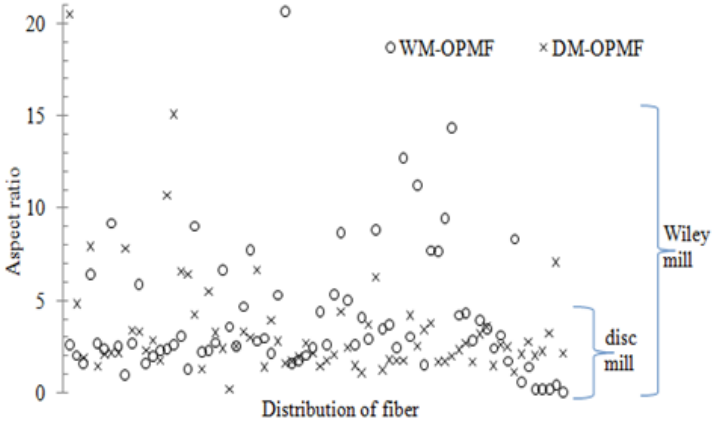

Fig.5: Distribution of the aspect ratio of DM-OPMF and WM-OPMF

Morphological Analysis

SEM analysis was done to support the above findings, as shown in Figures 6a–e. Fig.6a shows the SEM micrograph of original OPMF fibre (not ground), whereby it can be observed that silica bodies are deposited on the entire of fibre surface. Similar observations were also reported by Chua *et al*. (2009) and Nik Mahmud *et al*. (2013). Figures 6b–e show the SEM micrographs of DM- and WM-OPMF. Overall, it is shown that DM-OPMF had smaller fibre size (Fig.6c) compared to WM-OPMF (Fig.6e). This observation can be explained by the mode of action of disc milling which involves crushing, shearing and cutting of the fibre samples (Vishwanathan *et al*., 2011). The microfibrillated structure of DM-OPMF can be clearly observed at a higher magnification (see Fig.6b). The shearing force by rotating disc of DM was able to open the pores of the fibre, causing PP to penetrate within the OPMF fibrils easily.

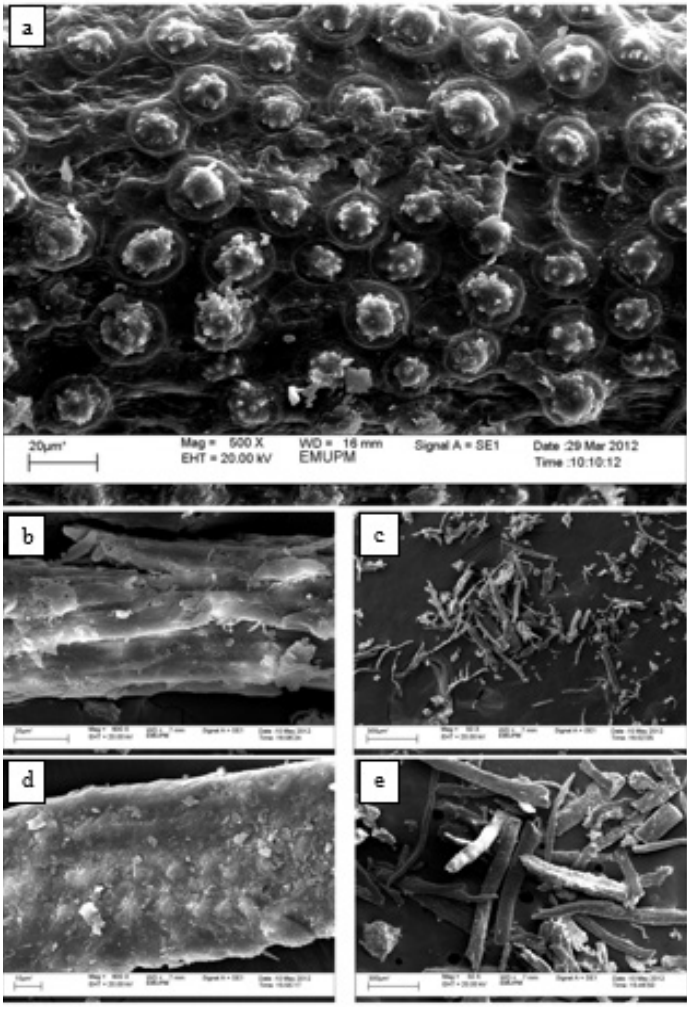

Fig.6: The SEM micrographs of OPMF: (a) original OPMF (not ground); (b) DM-OPMF (800x magnification) (c); DM-OPMF (50x magnification) (d) WM-OPMF (800x magnification), and (e) WM-OPMF (50x magnification). Silica bodies were found deposited on the surface of original OPMF fibre

It is interesting to observe that silica bodies were removed from the surface of milled-OPMF. Both DM- and WM-OPMF showed no silica bodies on their surface. According to Shinoj *et al*. (2011), removal of silica bodies could create perforated silica craters, which would result in a better fibre-matrix interfacial adhesion. This can be explained by the creation of rougher fibre surface which is more advantageous in helping the penetration of melt polymer into the fibre (Shinoj *et al*., 2010).

Thermal Stability of DM-OPMF and WM-OPMF

Thermal analysis of the milled fibres was done to evaluate the thermal stability of the samples. Fig.7 shows TG and DTG curves of DM-OPMF and WM-OPMF. From the TG curves, both DM-OPMF and WM-OPMF had almost similar thermal degradation trend with three-step degradation indicating the three major components in lignocellulose; hemicellulose, cellulose and lignin. Incomplete decomposition at 550ºC indicates that some lignin fractions were degraded at a higher temperature range (Yang *et al*., 2007). On the other hand, DTG curves showed two apparent peaks at the temperature range of 250–320°C and 320–390°C, indicating the degradation of hemicellulose and cellulose, respectively.

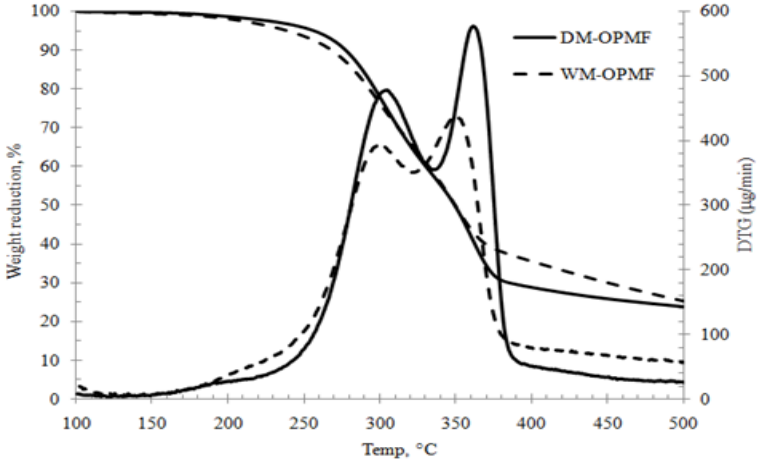

Fig.7: TG and DTG curves of DM-OPMF and WM-OPMF

TABLE 1: Summary of degradation temperature at 5, 20 and 50% obtained by TGA of DM-OPMF and WM-OPMF

Sample	$T_{5\%}$ (°C)	$T_{20\%}$ (°C)	$T_{50\%}$ (°C)	T_p (°C)	Weight loss (%) Temp region (°C) 150-250	Weight loss (%) Temp region (°C) 300-450	Residue at 550 °C (%)
DM-OPMF	256.5	296.6	349.0	299.7, 359.3	4	52	23
WM-OPMF	238.8	292.8	349.2	295.4, 346.6	7	46	24

$T_{n\%}$ represents the onset decomposition temperature of 5, 20 and 50% weight loss, T_p represents peak temperature of DTG.

A summary of the thermal decomposition profile of each sample is presented in Table 1. The results of decomposition temperature at different weight losses (5%, 20% and 50%), the onset temperature of TGA and peak temperature of DTG are tabulated. From the results, it is seen that DM-OPMF had higher thermal stability compared to WM-OPMF. For example, T5% of DM-OPMF was recorded at a higher temperature (256.5ºC) compared to that of WM-OPMF (238.8ºC). This indicates that some components in WM-OPMF degraded at a lower temperature. Lignin has been reported to have a wide thermal degradation temperature range between 150 – 900°C (Yang *et al*., 2007; Bachtiar *et al*., 2013). Therefore, based on the weight loss at the low temperature as shown in TG results, it is suggested that some lignin fractions were removed during disc-milling, causing the thermal stability of DM-OPMF to improve. This is supported by the residual weight of OPMF at 370ºC onwards, which showed that DM-OPMF had lower residual weight compared to WM-OPMF. This indicates that lignin content in DM-OPMF was lower as compared to WM-OPMF.

Lignin loss during disc-milling can be explained by shear effect during disc-milling. It is suggested that the shear and friction during disc milling disrupted lignocellulosic structure, as proven by the formation of microfibrillated structure. This is supported by Fig.4 which shows that DM-OPMF has a smaller fibre diameter (~ <50 μm) compared to that of WM-OPMF (up to 400 μm). In lignocellulose material, lignin acts as glue in between the cellulose fibrils (Reddy & Yang, 2005) causing them to arrange tightly in stacks and hence giving strength to the material. The disruption of lignin causes the cellulose fibrils to tear apart and form microfibrils. Moreover, Mikushina *et al*. (2002) highlighted that milling is expected to cause chemical changes in lignin. Additionally, the presence of lignin in fibre sample is usually shown by the dark appearance of the fibre. Herewith, it is shown in Fig.3 (b) and (c) that DM-OPMF had lighter colour compared to that of WM-OPMF.

Thermal and Tensile Properties of OPMF/PP Biocomposite

DM-OPMF and WM-OPMF fibres in the particle size of <150 μm were blended with PP in the mixer at 20 and 50% (w/w) of fibre loading. PP/OPMF biocomposites were tested for their thermal stability and tensile strength.

Generally, PP has higher thermal stability compared to lignocellulose, while the biocomposite samples had better thermal stability compared to OPMF alone, as seen in Figures 7 and 8. Bachtiar *et al*. (2013) described the degradation temperature for biocomposite to commonly fall between the degradation temperature of polymer matrix and the filler. From the results obtained, the biocomposites prepared from DM-OPMF and WM-OPMF at 20 and 50% (w/w) of fibre loading showed almost similar trend in thermal degradation from the beginning to completion, indicating that alteration in lignin content in DM-OPMF did not really affect the biocomposite thermal property. Fig.8 shows the TG and derivative TG (DTG) curves of DM- and WM-OPMF biocomposite at 50% (w/w) of fibre loading. From the DTG curve in Fig.8, three maximum peaks can be observed. The first peak temperature for DM- and WM-OPMF biocomposite was the same at 292°C. This peak indicated hemicelluloses decomposition as reported by Yang *et al*. (2007); degradation temperature of hemicelluloses ranged from 220 to 318°C. The second peak was degradation of cellulose that could be observed at 359°C

for DM-OPMF which was slightly higher than WM-OPMF at 356°C. Polypropylene in the biocomposite was decomposed at the peak temperature of 473 and 471°C for DM- and WM-OPMF, respectively.

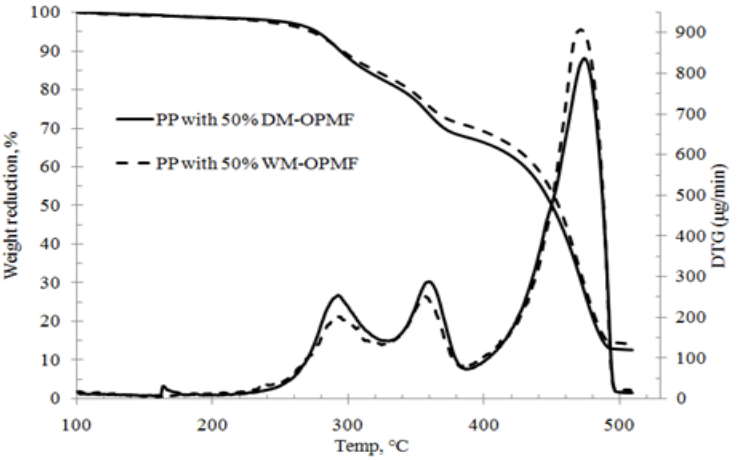

Fig.8: The TG and DTG curves of PP/DM-OPMF and PP/WM-OPMF biocomposites

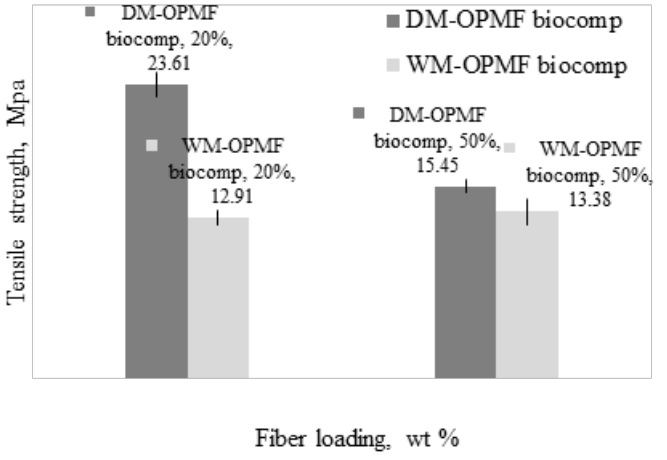

Fig.9: Tensile strength of DM-OPMF and WM-OPMF biocomposites prepared from fibre with particle size <150 µm with 20 and 50 % (w/w) fibre loading

The tensile strength of WM-OPMF and DM-OPMF biocomposites were tested in order to evaluate the performance of the biocomposites. Fig.9 shows the tensile strength of PP/WM-OPMF and PP/DM-OPMF biocomposites at 20 and 50% (w/w) fibres loading. There were marked differences in the tensile strength value for biocomposite prepared from DM-OPMF with that of WM-OPMF, especially for the sample with 20% (w/w) of fibre loading. Biocomposite with 20% (w/w) DM-OPMF had a tensile strength of 23.6 MPa, which is 83% stronger compared to the biocomposite with 20% (w/w) WM-OPMF. The same observation could be seen for the biocomposite prepared with 50% (w/w) fibres, even though the difference

in the tensile strength value was not as high as that for the biocomposite with 20% (w/w) fibres. The higher tensile strength demonstrated by the biocomposite prepared from DM-OPMF could be contributed by the smaller size of the fibre particles depicted in Figures 2, 4 and 6. Even though the OPMF used in this study were obtained after sieving to pass through 150 μm mesh, the microscopic analyses by light microscope and SEM revealed that the fibres with larger particle size (long fibres) could still pass through the mesh since the fibres passed through the mesh vertically. Based on the observation, it can be concluded that DM-OPMF had smaller fibre size compared to WM-OPMF and hence, it gave a higher surface area per volume which then improved the surface contact between fibres and polymer matrix. The higher surface contact between fibres and polymer improved the physical adhesion between the two materials. This explained why PP/DM-OPMF had a better tensile strength compared to PP/WM-OPMF. Moreover, the small size of DM-OPMF might have improved the stress transfer between polymer matrix and fibres compared to longer fibres. Fibres with small diameter also cause them to be arranged in the polymer matrix easily, leading the tensile strength to be improved in the PP/DM-OPMF biocomposite.

CONCLUSION

Two different grinding techniques were used to grind OPMF for biocomposite production. DM-OPMF showed a higher percentage of small sized particles (<75 μm) compared to WM-OPMF by almost three folds. Disc milling was able to disrupt lignin structure and hence unmasked the macrofibrils to form microfibrils. This eventually affected the tensile strength of PP/OPMF biocomposites. PP/DM-OPMF biocomposite with 20% (w/w) fibres had a tensile strength of 23.6 MPa, which is 83% higher compared to the PP/WM-OPMF biocomposite. This study revealed that the milling method has pronounced effects in improving the mechanical property of biocomposites. Overall, disc milling yielded smaller sized fibre particles compared to Wiley mill and hence, enhanced the surface contact between fibres and polymer matrix.

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The Effects of Airflow on Oven Temperatures and Cakes Qualities

Shahapuzi, N. S., Taip, F. S.*, Ab. Aziz, N. and Ahmedov, A.

Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia

ABSTRACT

The presence of airflow during heating process is expected to increase heat uniformity in a closed heating chamber. Circulation of hot air increases the percentage of convective heat transfer. In this study, effects of airflow on oven temperature, cake temperature and several cake qualities were investigated. Experimental studies were conducted in convective oven using two different baking modes; with and without airflow. During baking, oven temperatures and internal cake temperature were measured, and images of cake expansion were captured. Results of the study showed that the presence of airflow could maintain the oven temperature within a small range of set point temperature. Temperature in the oven exhibited ±5.5°C fluctuation, approximately 3.5% overshoot that occurred continuously during baking with airflow. On the contrary, higher overshoot (ranging from 15 to 30%) was observed in oven temperature without airflow. Airflow also showed a significant effect ($p<0.01$) during the second stage of baking. The presence of airflow increased the heating rate and resulted in a faster volume expansion, which was 3.21mm/min, as compared to 2.88mm/min without the airflow. However, airflow dried off the cake surface, resulted in quicker browning, higher weight loss and lower moisture content of cakes.

Keywords: Cake baking, airflow, heat transfer, internal cake temperature, volume expansion

INTRODUCTION

Oven conditions contribute to effectiveness of heat transfer to the product. Appropriate oven temperature, air circulation, humidity, baking time and oven load should be determined before setting up oven condition. There are a series of physical, chemical and biochemical changes involved during baking process. The changes can be seen on cake expansion, moisture content, surface colour and texture. These changes are usually evaluated as the final product quality.

During baking, heat is mainly transferred to the product surface through three mechanisms of heat transfer. The first mechanism is the radiation from oven walls, followed by the convection of hot air flowing inside the oven, and finally the conduction

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E-mail addresses:
nursyafikah@upm.edu.my (Shahapuzi, N. S.)
farahsaleena@upm.edu.my (Taip, F. S.)
norashikin@upm.edu.my (Ab. Aziz, N.)
anvar@upm.edu.my (Ahmedov, A.)
*Corresponding Author

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from the surface to the core of the product. A homogenous heating of product is crucial to prevent microbial risk and to maintain the uniformity of product quality (Verboven *et al*., 2000). For that reason, a uniform heating medium is required to obtain a homogeneous heating to the product. Thus, operating the oven with airflow mode could be a reasonably good option to accomplish a uniform oven temperature (Spence *et al*., 2007; Therdthai *et al*., 2004; McFarlane, 2006; Xue & Walker, 2003).

The presence of airflow generally influences heat exchange in the oven chamber, in which the percentage of convective heat transfer will be increased to some extent. Therefore, it is interesting to distinguish how these changes may influence oven temperature, baking process and finally product properties. Some previous studies have reported the effects of process condition in tunnel type oven (Baik *et al*., 2000a), pilot plant oven (Zareifard *et al*., 2009; Khatir *et al*., 2012), electric oven (Lostie *et al*., 2002) and infrared-microwave combination oven (Turabi *et al*., 2007), to name a few. However, the effects of the airflow to the oven temperature, process condition and product quality during cake baking have not been substantially reported.

The present study investigated how the variations in temperature and the presence of airflow affect the profile of oven temperature. This study also evaluated the effects of oven temperature and internal cake temperature towards cake expansion and product qualities.

MATERIALS AND METHODS

Cake batter was prepared by means of standard creaming method using a hand mixer (Panasonic, MKGH1, Osaka). Major ingredients are listed in Table 1. An aluminium baking pan with dimension of 15 cm x 15 cm x 7.5 cm (L x W x H) was modified to have transparent glass at the front side. The batter (ranged from 444 g to 448 g) was poured into the baking pan, and tapped three times before the top surface was smoothed with an offset spatula. The initial height of cake batter ranged from 28 mm to 30 mm.

TABLE 1: Major ingredients of butter cake based on flour weight

Ingredients	Wt. (%)
Butter	65.5
Castor sugar	87.0
Sifted cake flour	100.0
Baking powder	3.5
Eggs	67.8
Fresh milk	69.6
Vanilla essence	4.3

Baking experiments were carried out in an electrical convective oven Brio-Inox (Gierre, Milano). The cake was baked at three different oven temperatures (160°C, 170°C and 180°C) under forced convection (with airflow mode) and static air (without airflow). The overall schematic diagram of the experimental apparatus is illustrated in Fig.1. Wire K-type thermocouples were used to measure the temperature at four different positions identified as

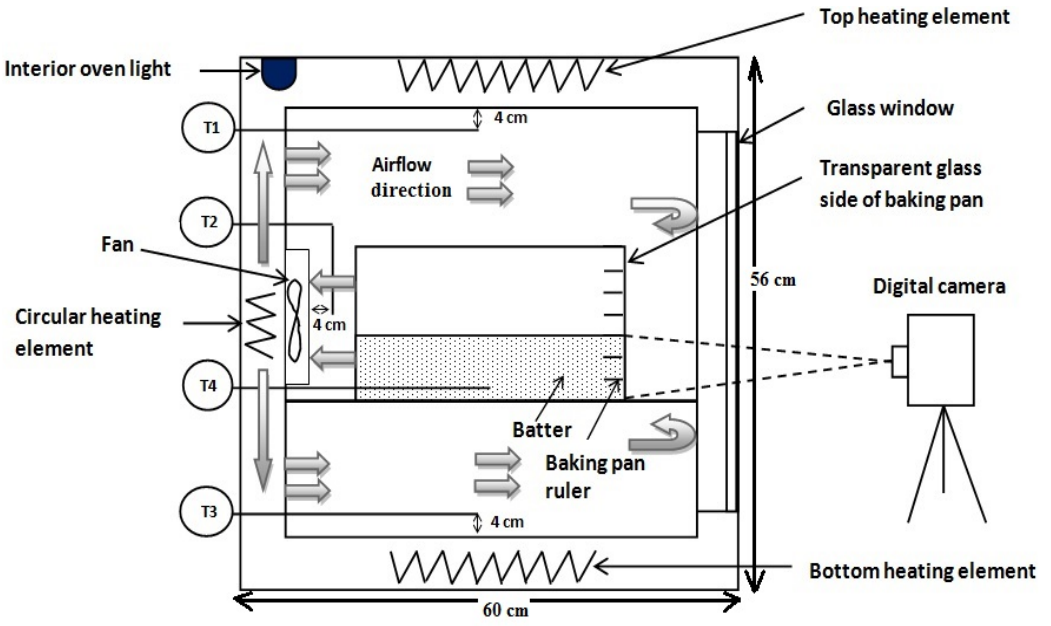
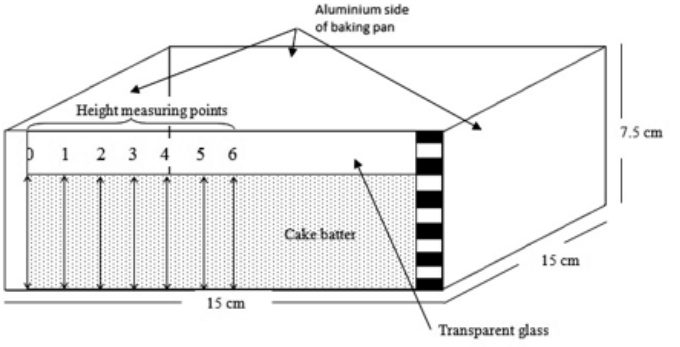


Fig.1: Experimental baking apparatus

(a)



(b)

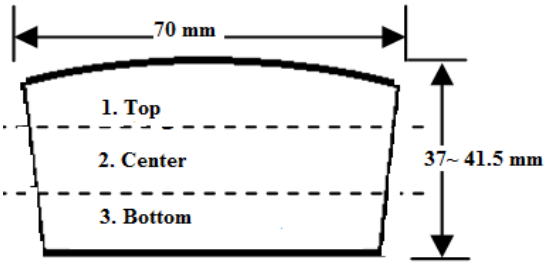


Fig.2: Experimental cake baking pan and sectioning of cake samples for the analysis of moisture content

T1 (top), T2 (center), T3 (bottom) and T4 (internal cake temperature). All thermocouples are connected to a data logger (Monarch Instrument, Monarch 309, USA) for temperature display and recording.

A digital camera (Sony, DSC-H50) was used to photograph the cake at 3 min intervals during baking. The distance of the camera tripod was 0.5 m from the oven glass window. Height of the cake was measured at every 1 cm interval from the left side until the centre point, as shown in Fig.2a. The measurement of the height of the cake was done from the digital image after the calibration of the images using the stainless steel ruler marking at the right side of the baking pan. Shrinkage during baking was calculated as the difference in height at its maximum height and the final height at the end of baking for each point. The difference in the cross sectional area of the sliced cake after baking and after 1 hour of cooling was measured to calculate the shrinkage during the cooling. This method was adopted from the previous work of HadiNezhad and Butler (2010). The crust and crumb thickness were also measured for every baked cake. The data of three replicates were recorded for each experiment.

The initial and final moisture contents of cake were analyzed by means of moisture analyzer (Infrared moisture balance, MX-50 AND Weighing, Adelaide) under standard drying, medium accuracy (0.05%/min) based on wet sample mass. The initial moisture content of batter was analyzed by spreading 2 g batter sample evenly on the aluminum foil before the start key was pressed to heat it. The cake was then cut into halves and a 2-mm sample was sliced from the centre part of the first half. The sample was then divided into three portions; top crust, crumb center and bottom crust, as shown in Fig.2b. The samples were placed in an air tight container to prevent moisture loss prior to the analysis. The moisture content was measured according to the sample portion.

Statistical analysis was conducted to study the effects of airflow and temperature on the oven temperatures profile and the quality of the final cake using GraphPad Prism 6 (GraphPad Software, Inc., 1992-2014). The data were analyzed using a two-way ANOVA analysis of data.

RESULTS AND DISCUSSION

Effects of Airflow on Oven Temperatures

The temperature profiles in the oven chamber during the cake baking process for with and without airflow at the set point temperature 170°C are shown in Fig.3. The oven temperatures were measured at the top, centre and bottom of the oven chamber, and identified as T1, T2 and T3, respectively. Three different points were chosen to measure the oven temperatures.

A distinct temperature difference could be seen between the top, centre and bottom of the oven during the baking process without the airflow mode. The temperature at the top (T1) was always higher than the bottom (T3), followed by the centre of oven (T2), giving the standard deviation range of 1.7°C to 10.2°C. This could be due to the location of the thermostat in the oven and the ineffective heat transfer that resulted from the lack of air circulation. On the contrary, with the presence of airflow in the oven chamber, all the three measured locations showed relatively similar temperature ($p<0.05$), whereby the standard deviation was between 0.4°C and 2.9°C.

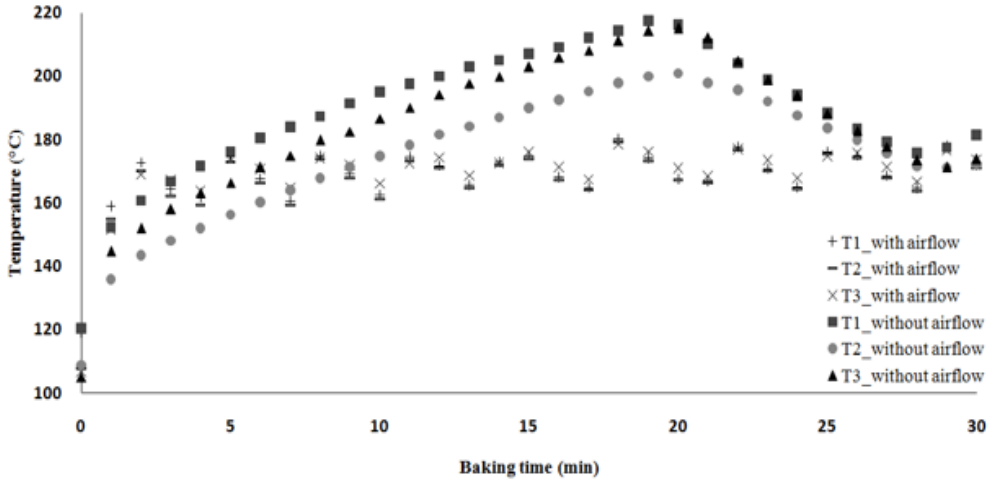


Fig.3: Oven temperature profiles during cake baking in convective oven for with and without airflow modes. T1, close to the top oven surface, T2, the centre location of oven, T3, close to the bottom oven surface

Although the set point is 170°C, the actual temperatures in each location were higher with overshoots between 19-30% without the airflow mode. The temperature at the top (T1) reached up to 217°C, which was 47°C higher than the set point (see Fig.3). A smaller overshoot (3.54%) was observed with the presence of airflow. The average temperature of the oven during baking with airflow was nearly to the set point temperature even though there was a fluctuation (±5.5°C) in the oven temperature.

The presence of airflow can also maintain the temperature within a small deviation of the set point. However, the temperature varied according to the locations. The bottom part of the oven chamber showed a slightly higher temperature than the centre and the top surface. This could be due to the variation in the circulation pattern of hot air in the oven chamber, as shown in Fig.1. Initially, the air in the oven chamber was sucked into the fan. Then, the air was heated by a circular heating element before travelling behind the panel to re-enter the oven chamber. The measurement of air velocity at the top, the centre and the bottom of the oven's right rear panel showed average readings of 0.34 m/s, 0.35 m/s and 1.88 m/s, respectively, measured by the thermal anemometer (Testo 425, USA). Higher hot air velocity at the bottom of the oven chamber generated the turbulence air condition in the oven, hence increased the convective heat. A relatively similar result was found by Spence *et al* .(2007) for the flow field in the domestic kitchen oven.

Effects of Baking Temperature on Oven Temperature

The oven temperature was affected by the variation of the set point temperature for both baking modes. Fig.4 shows the oven temperature profile at the top (T1) during the baking temperature of 160°C, 170°C and 180°C. The temperature variations during baking without airflow required a longer time to reach the maximum oven temperature than baking with airflow ($p<0.01$). In contrast, the increasing temperature during baking with airflow mode had slightly decreased the

time taken to complete one cycle. This could be due to the physical properties of air that varied with temperature. Increasing the baking temperature would increase the velocity of hot air. It therefore caused the heat transfer mechanism to speed up. Thus, the maximum temperature could be reached faster. Hence, a complete cycle was accomplished earlier.

In contrast to baking with airflow, the temperature variation during baking without the airflow mode resulted in a quite a high percentage of overshoots ($p<0.01$). The maximum temperature overshoots were in the ranged of 28-30% for the top of the oven chamber, 16-19% for the centre of the oven chamber and 25-28% for the bottom of the oven chamber.

Effects of Baking Temperature on Internal Cake Temperature

The internal cake temperature for the three different baking temperatures is illustrated in Fig.5. There were three distinct stages during cake baking, namely, initial heat penetration, heating up and crust and crumb formation. The initial heat penetration stage occurred in the first one sixth of baking time. There was a relatively small temperature gradient. This was followed by a heating up stage where cake temperature increased rapidly. The internal cake temperature reached a pseudo-plateau during crust and crumb formation at an approximated temperature of 99.9°C. By increasing the baking temperature, shorter time was needed to reach pseudo-plateau temperature. The pseudo-plateau temperature occurred as a result of structure stiffening and formation of superficial dry crust layer that limit heat transfer through the crust and water vaporization (Fehaili *et al*., 2010; Lostie *et al*., 2002; Ousegui *et al*., 2010). Therefore, the temperature of crumb would not exceed the water boiling point temperature.

Temperature variation did not have significant effects on the internal cake temperature during the first and third stages ($p>0.05$). This was closely related to the slow heat penetration mechanism in the first stage and maximum heat occupancy in the cake during the third stage. Nevertheless, the temperature variation had given a direct impact on the second stage of baking ($p<0.01$). This could be due to the linear relationship between the oven temperatures to the heating rate in the cake. Higher oven temperature means more heat is transferred to the cake surface. Therefore, a steeper slope representing higher rate could be seen as the oven temperature increased from 160°C to 180°C, as shown in Fig.5. The temperature increment

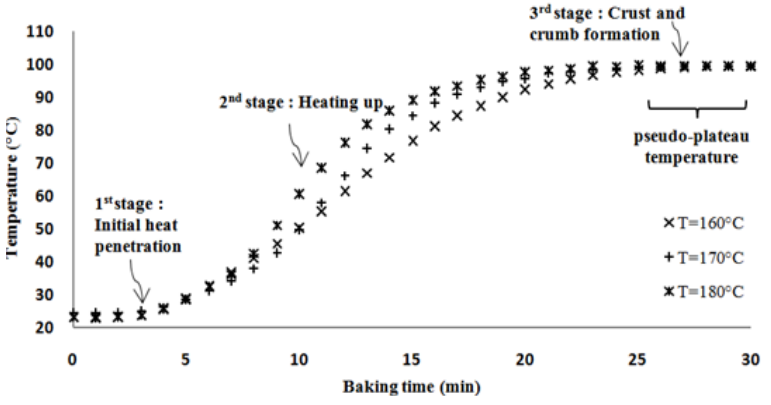


Fig.5: Internal cake temperature for the increasing of baking temperature with airflow mode

during the second stage of baking is critical since it is responsible to initiate several reactions such as caramelization, Maillard reaction, starch gelatinization, protein denaturation, water movement, cell structure formation and enzyme activities (Chang, 2006).

The findings from the present study corroborate with the results obtained by Lostie *et al*. (2002) who found that increasing the baking temperature would increase the rate of internal cake temperature during the second stage of baking. The heating rate during second stage increased more than half, i.e. from 5.17°C/min to 7.96°C/min, as the temperature was increased from 160°C to 180°C.

Effects of Airflow on Internal Cake Temperature

Fig.6 shows distinct three stages of internal temperature of the cake baked with and without airflow modes. The significant difference ($p<0.01$) could be seen on the thermal gradient during the second stage. The airflow significantly influenced the second stage by increasing the thermal gradient thus produced a sharper slope and hence shorter time to accomplish the second stage. The circulation of hot air increased the convective heat transfer to the cake surface. On the contrary, the second stage of baking without airflow mode showed slow but steady increase in the temperature until it reached a plateau in the third stage.

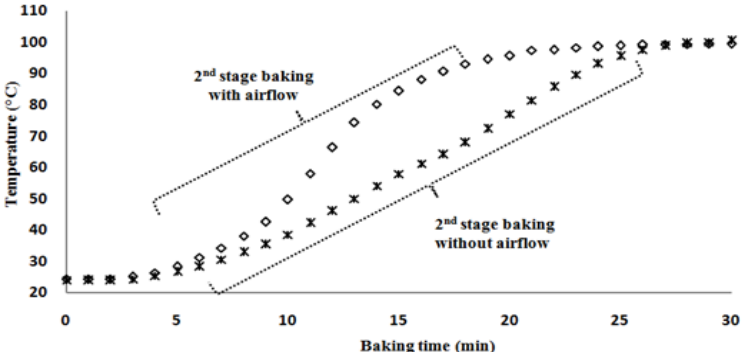


Fig.6: Internal cake temperature for baking in different modes at 170°C

TABLE 2: Heating rate of cake baking at 170°C

Time (min)	Without airflow mode(°C/min)	With airflow mode(°C/min)
0	0.33	0.48
5	2.10	3.20
9	3.74	7.52
14	3.45	3.20
18	4.36	1.04
23	2.63	0.33
26	0.70	0.05

The heating rates in both baking modes are tabulated in Table 2. Slower heating rate occurred at the first stage of baking in both the baking modes. The second stage started

approximately at minute 5, where batter temperature started to increase gradually. Rapid heating could be seen in baking with airflow mode where the heating rate was doubled from 3.2°C/min to 7.52°C/min, as compared to without the presence of airflow. The presence of airflow reduced the total baking time by 20% as compared to without airflow.

Effects of Baking Temperature and Airflow on Cake Expansion

The images of a typical cake expansion that occurred during baking at 170°C with airflow mode are shown in Fig.7. The appearance of the brown surface colour gave an indication that the expansion process was slowing down and would end soon.

The profile of cake height changes during baking is plotted in Fig.8 based on the centre point of the cake height. Increasing the baking temperature slightly increased the percentage of height increment. This is similar to the findings in Fig.5 which showed that the increasing of baking temperature did not have any significant effect on the gradient of internal cake temperature.

The percentage of cake shrinkage after baking with airflow for 160°C, 170°C and 180°C are 11%, 14.9% and 17%, respectively. These results are based on the total cross sectional area of a sliced cake. In contrast, the cake shrinkage after baking without airflow was 12.5%, 21.9% and 22.7% for 160°C, 170°C and 180°C, respectively. Cake baked at a high temperature shrank the most after cooling. This might be the results of cake contraction during cooling which was caused by the release of gas in the bubbles. Higher baking temperature produced a higher pressure of gas in the bubble, and therefore caused greater cake contraction.

Apart from baking temperature, the airflow inside the oven chamber resulted in significant differences in the expansion of cakes ($p<0.05$). Cakes expanded at different rates during the second stage of baking period in both the baking modes (see Fig.9). Higher percentage of height changes could be seen in the baking process with the airflow mode. In particular, the rate of cakes height increased approximately 3.21mm/min during the second stage of baking with airflow as compared to 2.88mm/min while baking without airflow.

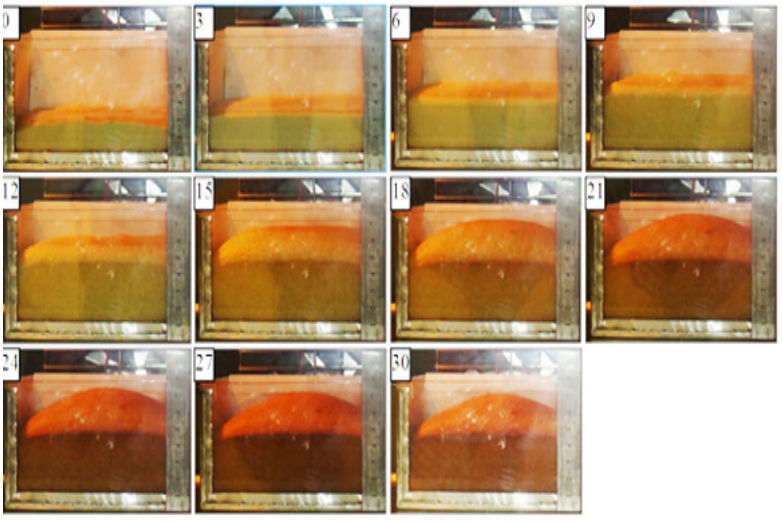


Fig.7: Successive images taken during batter expansion. The number on each image represents the time (min) of baking at which the image was taken

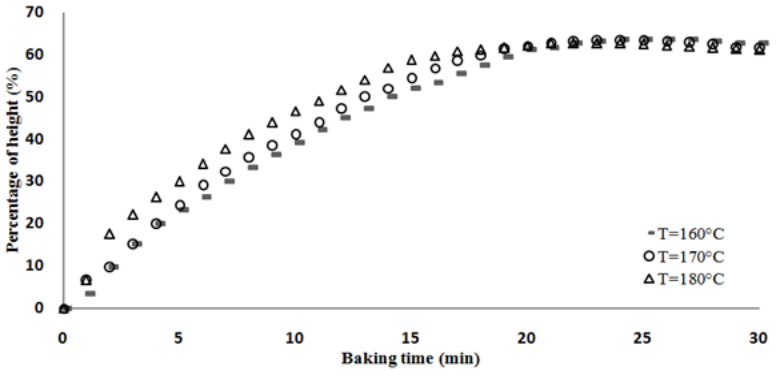


Fig.8: The profile of cake height changes for low (160°C), medium (170°C) and high (180°C) baking temperature baking with airflow mode

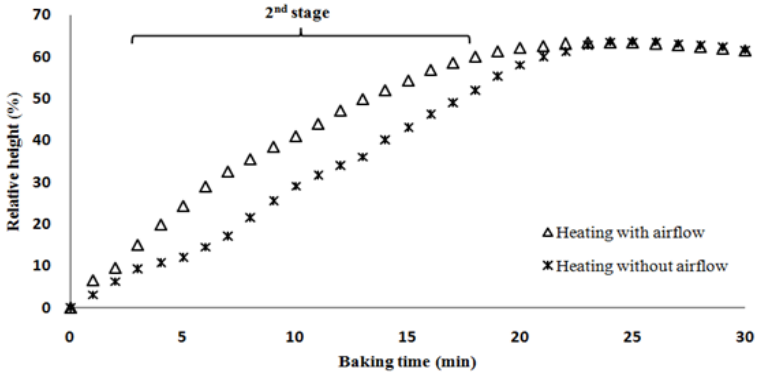


Fig.9: The profile of cake height changes for different baking modes at baking temperature 170°C

Fig.8 and Fig.9 can be used to emphasize the findings on the effects of temperature variation and baking mode towards baking process. Hence, airflow plays an important role in cake baking by having a higher percentage of cake height increment.

Moisture Content of Cakes

Water movement during cake baking process occurs when the moisture in the batter moves to its surrounding and then evaporates. Water movement also contributes in heat transfer and diffusion. However, cake will become dry and large crust portion will be formed if too much water evaporates and thus reduces the taste and cake quality. Therefore, the moisture content becomes one major concern since it greatly influences the quality and acceptance of a product (Chang, 2006).

The initial moisture content of batter was 34.06± 0.12% in wet basis. Table 3 shows the percentage of moisture content of cake after baking ended with the range of 25.27% to 33% in this experiment. Generally, the moisture content was lowest on the top surface (portion 1), highest in the middle (portion 2) and intermediate moisture content in the bottom portion (portion 3) where the significance level is $p<0.01$. This is approximately similar to that found

in Baik *et al*. (2000b) for thick cake layer baked in a tunnel type industrial oven. Therefore, the findings in this study can support the theory that temperature distribution in the product (generally, surface>bottom>centre) can affect moisture movement, as suggested by Baik *et al*. (2000b).

TABLE 3 : Percentage of moisture content in wet basis of cake after baking

Portion	Baking without airflow mode (%)			Baking with airflow mode (%)		
	160°C	170°C	180°C	160°C	170°C	180°C
Top	25.49±1.56	27.19±0.93	27.27±3.21	26.12±0.85	25.27±0.45	25.27±0.01
Center	32.08±2.18	33±0.44	32.38±0.32	31.92±0.54	32.33±0.94	32.1±0.38
Bottom	29.06±0.95	29.45±0.73	27.97±0.19	29.05±0.01	27.69±0.12	27.14±0.02

Cakes baked with airflow showed a lower percentage of moisture content as compared to baking without airflow ($p<0.05$). The thermal gradient in cake might influence the percentage of the final moisture content. As shown in Fig.6, baking with airflow resulted in a higher thermal gradient. The higher thermal gradient in the cake might enhance the migration of moisture from the hotter to colder region.

The moisture loss during baking might contribute to the weight loss of cake. By increasing the baking temperature, it causes the higher percentage of weight loss, as illustrated in Fig.10. Besides, the cake baked with airflow mode resulted in a higher weight loss compared to those without airflow mode ($p<0.05$). This might be due to the enhanced airflow velocity by the fan to distribute heat throughout the oven chamber and thus increased heat penetration to the cake.

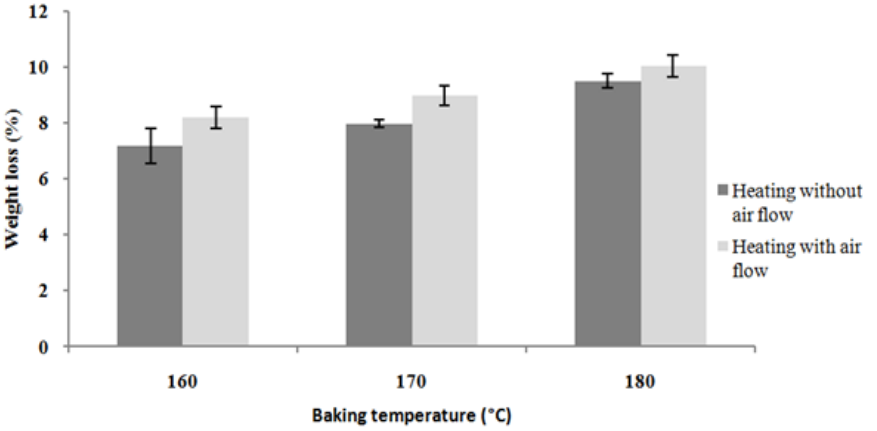


Fig.10: The percentage of weight loss with an error bar for low (160°), medium (170°C) and high temperature (180°) under different heating modes

Cake Properties and Qualities

Several factors have effects on the texture of cake such as ingredient, mixing process, baking temperature, baking time, air velocity and type of oven. The variation in the baking temperature caused the surface colour to become dark brown and resulted in thick cake crust. The presence

of airflow during baking resulted in a uniformly brown surface colour of the cake. In contrast, baking without airflow mode produced a cake that has a pale surface colour with some dark spot, cracked surface and compact texture.

After cooling to room temperature for an hour, the cakes undergoes a slight shrinkage. Cakes baked with airflow mode shrunk lesser (about 11%-17%) than those without the airflow mode (12.5% to 22.7%), while higher baking temperature increased shrinkage in both the baking modes. The final height of the cake baked with airflow was also lower. The reduction in the cake height is mainly due to the contraction of cake. Baking with airflow causes bubble expansion to become faster and gas pressure in the bubbles becomes higher, causing the cake contraction to occur earlier.

CONCLUSION

The presence of airflow during baking process highly influences the oven temperature as the hot air can be circulated throughout the oven chamber resulting in a uniform oven temperature profile. It can also maintain the temperature of the oven very closely to the set point temperature. A uniform heat in the oven chamber increases the heat received by the product and affects its end quality. The cake baked with airflow showed a higher rate of volume expansion but a lower percentage of moisture content as a result of the higher heating rate in the cake.

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Quantification of Gallic Acid and Tannic Acid from *Quercus infectoria* (Manjakani) and their Effects on Antioxidant and Antibacterial Activities

Ab. Rahman, N. S.[1,2], Md. Salleh L.[1,2*], Abd. Majid F. A.[2] and Harisun, Y.[3]

[1]*Centre of Lipid Engineering Applied Research, Faculty of Chemical Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia*

[2]*Department of Bioprocess Engineering, Faculty of Chemical Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia*

[3]*Institute of Bioproduct Development, Faculty of Chemical Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia*

ABSTRACT

Effects of different types of solvent on the antioxidant and antibacterial activity of *Quercus infectoria* extract have not been well documented. Therefore, extraction process was conducted using conventional Soxhlet extraction with six different types of solvent (100% methanol, ethanol, acetone, water and 70% methanol, and ethanol). High performance liquid chromatography was implemented to identify gallic acid and tannic acid in the extracts. Water extracts contained the highest concentration of both gallic acid and tannic acid compared to other types of solvent; 51.14 mg/g sample and 1332.88 mg/g sample of gallic acid and tannic acid. Meanwhile, antioxidant and antibacterial activity were tested using DPPH free radicals scavenging and disc diffusion assay. Results demonstrated that water extracts gave the highest antioxidant activity (approximately 94.55%), while acetone extract gave the largest inhibition zone for disc diffusion assay (19.00mm respectively). The results also revealed rich sources of gallic acid and tannic acid in Q.infectoria which might provide a novel source of these natural antioxidant and antibacterial activity.

Keywords: Bioactive compound, solvent, antioxidant activity, antibacterial activity, gallic acid, HPLC, tannic acid, and *Q. infectoria.*

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E-mail addresses:
syukriah_org@yahoo.com (Ab. Rahman, N. S.),
i.liza@cheme.utm.my (Md. Salleh L.),
adibah@cheme.utm.my (Abd. Majid F. A.),
harisun@ibd.utm.my (Harisun, Y.)
*Corresponding Author

INTRODUCTION

Quercus infectoria has been extensively used as a medicinal plant since ancient time because it is reported to contain large amounts of bioactive constituents such as tannin, gallic acid, syringic acid, ellagic acid, β-sitosterol, amentoflavone, hexamethyl ether,

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isocryptometrin, methyl betulate, methyl oleanate, hexagalloyglucose, etc. (Dar *et al*., 1976; Ikram & Nowshad, 1977; Hwang *et al*., 2000). The main constituents found in the galls of *Quercus infectoria* are tannin (50-70%), free gallic acid and ellagic acid (Evans, 1996; Wiart & Kumar, 2001). Tannic acid and gallic acid, which are derivatives of tannins, have been reported to have antioxidant activity and the ability to become antimicrobial (Everest & Ozturk, 2005), antibacterial (Hamid *et al*., 2005) and antifungal agent (Yamunarani *et al*., 2005). Extraction of bioactive compounds from medical plants has enabled demonstration of their physiological activity by medical researchers.

Quercus infectoria gall (QI), which is widely known as manjakani, is a small tree native to Greece, Asia Minor and Iran. It is also popularly known as oak tree. Galls arise on young branches of this tree because of attack by gall wasp called Adleriagallae-tinctoria. In Malaysia, it is known as a herbal drink to cure uterine wall of women after their childbirth. In India, it is better known as Majuphal and has been used as dental powder and in the treatments of toothache and gingivitis. In Asian countries, it has been used for centuries as a traditional medicine for inflammatory disease (Galla, 1911; Kaur *et al*., 2004). Hemorrhoids caused by inflammation of the skin can be treated by applying powdered *Quercus infectoria* in the form of ointment on the skin. Its potential medical and cosmeceutcal areas have greatly induced researchers to study its scientific values in further detail.

Most works on *Quercus infectoria* have been done on identification and isolation of its biological and pharmacological properties (Asghari *et al*., 2011; Basri *et al*., 2005; Kaur *et al*., 2005). However, none of the study has reported on the effects of different solvents on antioxidant and antibacterial activities. Therefore, the objectives of the present study were to identify and quantify gallic acid and tannic acid from the gall extracts using six different types of solvents (100% methanol, 70% methanol, 100% ethanol, 70% ethanol, 100% acetone and water) by using HPLC, and to evaluate the antioxidant and antibacterial activities using DPPH free radicals scavenging and disc diffusion method.

MATERIALS AND METHODS

Materials

Methanol (MeOH 100% and 70%), ethanol (EtOH 100% and 70%), Acetone 100% and 2, 2-diphenyl-1-picrylhydrazyl (DPPH), Gallic acid and Tannin acid were purchased from Sigma Aldrich (M) Sdn Bhd Chemicals.

Plant Material

The galls of *Quercus infectoria* were purchased from a local herbal market in Johor Bahru, Malaysia. The galls were crushed into fine powder and washed under tap water to remove undesired particles. After that, all the samples were dried in an oven at 50ºC.

Extraction Preparation

5 g of powdered galls were weighted and placed in the timber, while 150ml of methanol (100%) was placed at the bottom of the apparatus. The extraction process was done for 6 hours to achieve a complete extraction process..The extraction yield was put in the rotary evaporator at 400C to remove the solvent. All the steps were repeated using 70% methanol, 100% ethanol, 70% ethanol, acetone and water.

High Performance Liquid Chromatography (HPLC)

1. Determination of Gallic Acid

Determination of active constituents from the extracted compounds was done using high performance liquid chromatography as described by Pin *et al*. (2006) with slight modification. Waters 600E System Controller combined with Waters 996 Photodiode Array Detector was used and C18 column was selected as stationary phase. Meanwhile, 0.1% orthophosphoric acid (H_3PO_4) was consumed as solvent A and 100% and 100% acetonitrile (100%) as solvent B. Then, the flow rate of mobile phase was adjusted at 1 ml/min at 280nm and every injection was set until 10 µL was achieved.

2. Determination of Tannic Acid

The identification of tannic acid from the extracts was done according to Asghari *et al*. (2011), with slight modification. High performance liquid chromatography was performed by reversed-phase HPLC on a C18 column using a binary gradient elution consisting of an aqueous methanol eluents at low pH as mobile phase. The gradient system consisted of solvent A (25ml acetic acid and 975ml distilled water) and solvent B (99.8% methanol) pumped at 1mL/min. The gradient started with 100% solution A and ended with 100% solution B at 30 min. The column temperature was maintained at 30°C. The sample peaks were identified by comparing with standard solution of tannic acid at 280nm. The percentage of the tannin acid was calculated using the appropriate calibration curves.

Antioxidant Activity

This assay was carried out according to the method proposed by Miliauskas *et al*. (2004) with a slight modification. Extract solution was prepared by dissolving 0.025 g of the dry extract in 10ml of methanol to give a final concentration of 2.5mg/ml. After that, 77µL of the extract solution was mixed with 3ml of 6 x 10^{-5} M methanolic solution of DPPH. The mixture was placed in the dark for 30 minutes at room temperature and the decrease in the absorption was measured at 517nm by using a spectrophotometer. Radical scavenging activity of the samples was calculated by using following formula:

$$\text{DPPH quenched (\%)} = \frac{A_{\text{Control}} - A_{\text{Sample}}}{A_{\text{Control}}} \times 100 \qquad [1]$$

Antibacterial Assay

The extracts from the galls of *Quercus infectoria* at 50mg/ml were screened against two-gram positive bacteria (*Staphylococcus aureus* and *Bacillus Subtilis*) and two-gram negative bacteria (*Escherichia Coli* and *Pseudomonas aeruginosa*). 50µL of bacteria suspension from the culture suspension was applied to the nutrient agar plate. Then, it was swabbed to the entire surface of the agar by using a sterile hockey stick. After that, sterile filter paper disc (Whatman No.1, 6mm) was impregnated with 20µl of each of the extracts (50mg/ml). Then, streptomycin (10µg/ disc) was used as standard to confirm that the entire microorganism tested was inhibited by the antibiotic and sterile distilled water used as the negative control. All the plates were incubated for 24 hours at 37°C. Then, the antibacterial activity was interpreted from the size of diameter of zone inhibition measured to the nearest millimetre (mm), as observed from the clear zone surrounding the disc (Basri & Fan, 2005). The inhibition zone was measured after 24 hours.

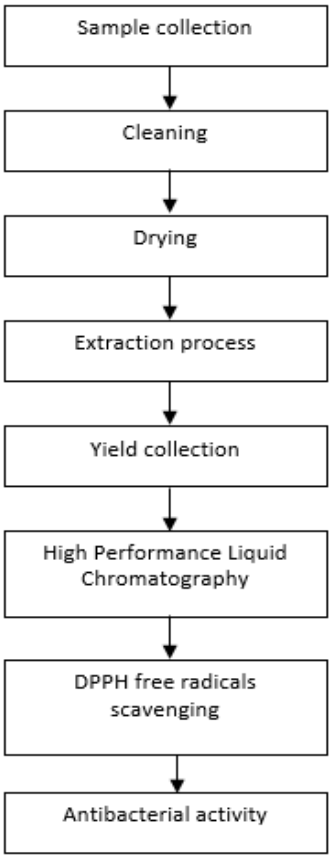

Fig.1: The flowchart of research activity

RESULTS AND DISCUSSION

Yield of Extracts

Table 1 shows the percentage yield of the *Quercus infectoria* extract using different types of solvents. The highest extraction yield was found with water extract (76%±0.084) and with a slight difference, followed by 70% methanol (74%±0.050) and 70% ethanol (71%±0.047). The findings indicated that *Quercus infectoria* was most soluble in polar solvents and most components were hydrophilic or water-soluble. On the other hand, 100% acetone resulted in lowest extraction yield suggesting that polar compounds in a biological plant were easier to extract with more polar solvents, whereas the less polar solvent was unable to extract the polar bioactive compounds. Tian *et al*. (2009) have reported similar findings, whereby the highest yield of *Galla chinensis* was obtained from aqueous solvent.

TABLE 1: Yield of Quercus infectoriaextract based on different types of solvent

Types of solvents	Yield of extract (% g/g sample)	Synder's solvent polarity index[c]
100% methanol	70±0.054[b]	6.6
70% methanol	74±0.050[b]	7.32
100% ethanol	52±0.147[a]	5.2
70% ethanol	71±0.047[b]	6.34
100% acetone	46±0.149[a]	5.4
100% aqueous	76±0.084[b]	9.0

Each bar represents means ± SD of three replicates. Different letters on the bars indicate groups are significantly different from each other according to Tukey's test ($p<0.05$).
[C] Synder's solvent polarity index cited from Markom *et al*. (2007).

From the ANOVA analysis, 100% ethanol and acetone were statistically significant compared with other solvents ($p<0.05$). Even though 100% ethanol and aqueous contain hydroxyl group which can create hydrogen bonding with the solute, aqueous solvent is proven to be more efficient in extracting the solute due to its higher polarity and shorter chain (Pin *et al*., 2006). These characteristics enhance its ability to extract polar compounds which thus explains the significant differences observed between the yields of 100% ethanol water.

The results above indicated that the mixture of the organic solvents (70% methanol and ethanol) produced a higher extraction yield compared to pure solvent (100% methanol and ethanol) alone, while the pure solvent of methanol gave a higher yield compared to absolute ethanol. As can be obtained from Table 1, the polarity of the mixture (70% methanol and ethanol) is higher compared to pure solvents (100% methanol and ethanol), which explains the higher yield obtained from those solvents. This finding is compatible with the previous findings documented by Markom *et al*. (2007) who found that the addition of water in ethanol significantly increased the extraction yield of *Phyllanthus niruri* Linn.

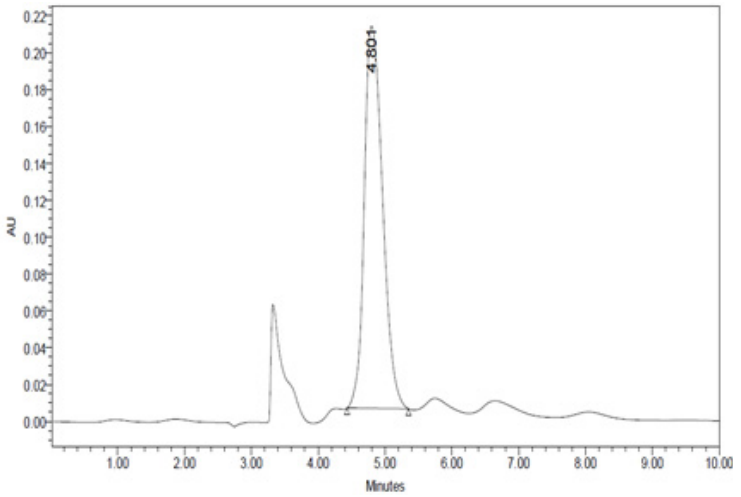

Fig.2: HPLC chromatogram of standard Gallic acid

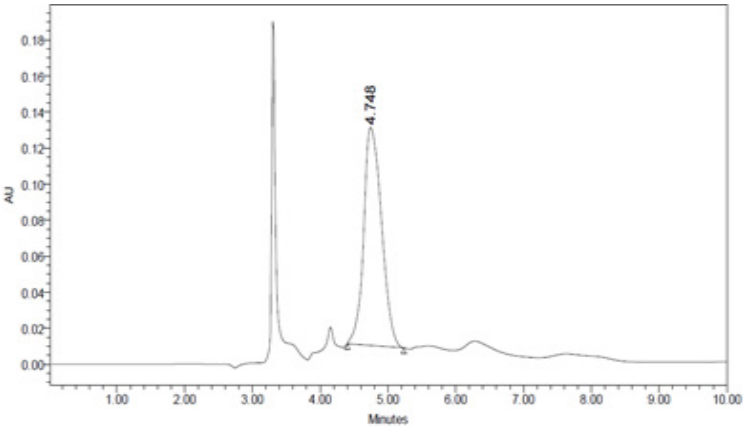

Fig.3: The HPLC chromatogram of Gallic acid from *Q. infectoria* galls extract

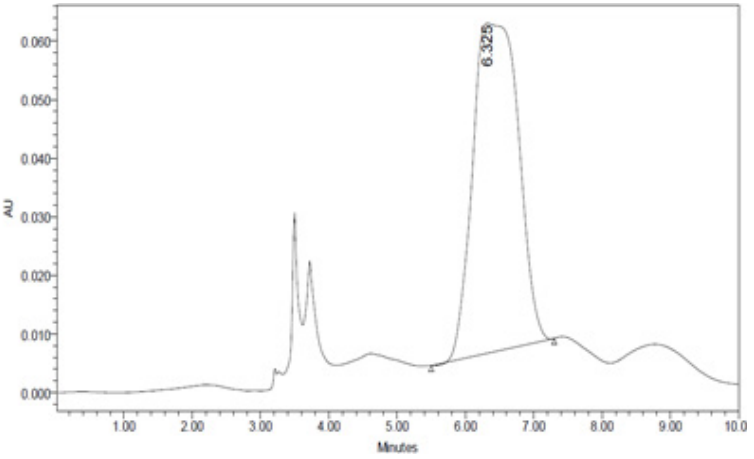

Fig.4: The HPLC chromatogram of standard Tannic acid

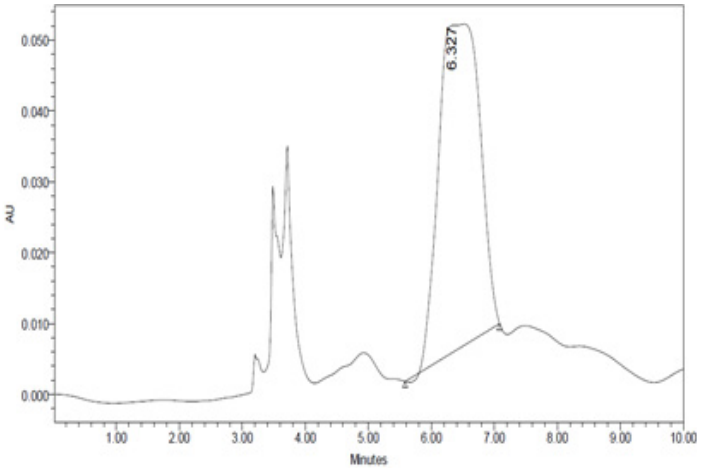

Fig.5: The HPLC chromatogram of Tannic acid from *Q. infectoria* galls extract

Identification and Quantification of Gallic Acid and Tannic Acid by HPLC Assay

The concentrations of the bioactive compounds were determined by using the peak area from the calibration curves (Song & Barlow, 2006) and are shown in Table 1. Fig.2 to Fig.5 show the chromatogram of standard and sample of gallic acid and tannic acid. HPLC analysis produced the similar trend in both gallic and tannic acid where 100% aqueous> 70% ethanol> 70% methanol> 100% methanol> 100% ethanol> 100% acetone. Aqueous extracts contain the highest concentration of gallic acid (101 mg/g sample) and tannic acid (2975 mg/g sample) compared to the other solvents used for the extraction process. This result indicates that aqueous solvent is a better extraction solvent compared to other solvents for the extraction of both gallic acid and tannic acid from the *Quercus infectoria* extracts.

A previous study reported lower concentrations of gallic acid and tannic acid from ethanolic extract of *Quercus infectoria*, which are 87 mg/g and 199 mg/g, respectively (Kaur *et al*., 2008). These different results might be contributed by the different solvent and solid to solvent ratio used.

Scavenging Antioxidant Activity

DPPH is a potent tool to ascertain the antioxidant capacity of the extracted compound. Fig.6 depicts the DPPH free radical scavenging by the *Quercus infectoria* extracts using different types of solvent.

In Fig.6, water extract (94%) gives the highest DPPH scavenging activity with a slight difference from 70% methanol (94%) and 100% methanol (93%). In addition, all the extracts showed higher scavenging activities compared to the control (BHA). The finding documented previously on the contradictive result revealed that the ethanol extract of *Galla chinensis* gave a higher reduction activity compared to the aqueous extract (Tian *et al*., 2009). The different findings are due to the ability of the solvent to extract the different bioactive compounds for different plants. From the ANOVA, types of solvents do not give significant differences in the scavenging of free radicals ($P > 0.05$).

This finding is compatible with that of Jain *et al*. (2011) who found that *Quercus infectoria* possesses antioxidant activity by scavenging free radicals about 90%, while Kaur *et al*. (2008) reported lower antioxidant activity, which is about 71%. The variation in the findings is due to the different concentrations and solvents used. In addition, the different origins of the raw material might have influenced the growth of the plant itself because different soil compositions would also yield different vegetative traits (Devkota & Jha, 2009).

The observation showed that the extract from 70% methanol and ethanol gave higher antioxidant activities compared to the absolute solvent. Turkmen *et al*. (2006) reported the same finding whereby 50% and 80% of solvent mixtures exhibited considerably higher DPPH radicals scavenging activity compared to the pure solvent. The high antioxidant activity of *Quercus infectoria* is due to the presence of gallic acid and tannic acid, which are proven to possess antioxidant activity (Govindarajan *et al*., 2005; Robert *et al*., 1999).

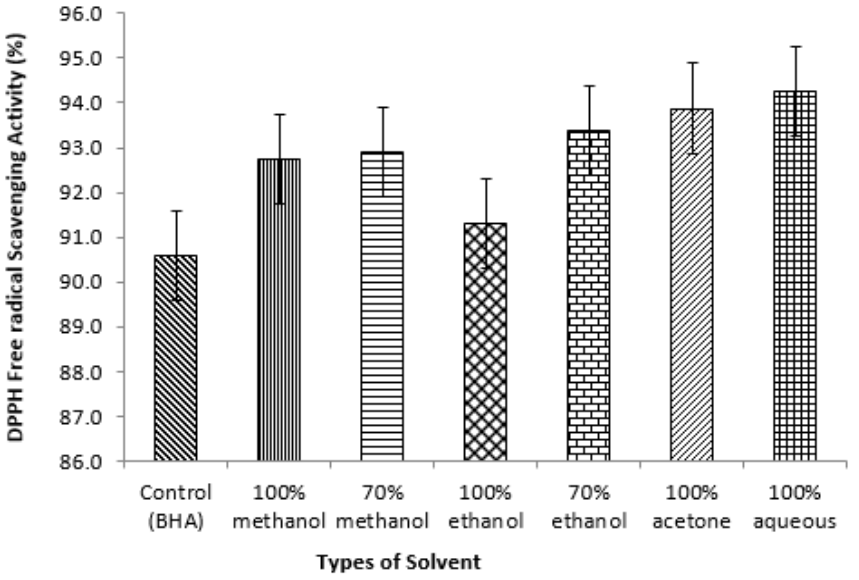

Fig.6: The DPPH scavenging activity of different types of solvent

Antibacterial Activity

The antibacterial activity of *Quercus infectoria* gall extracts using different solvents was studied by using disc diffusion method and the results are tabulated in Table 2 and Fig.7. All the extracts showed inhibitory effects that are not significantly different ($P>0.05$) against each bacterial species tested. After 24 hours, the largest inhibition zone (19±0.14mm) was shown by the extract of 100% acetone against *B.subtilis* and it was comparable with the commercial antibiotics (19±0.07.mm). However, other samples also showed inhibition zone that varied from 12±0.21mm to 18±0.21mm. *B. subtilis* was found to be the most susceptible towards all extracts. The smallest inhibition zone was exhibited by 100% acetone extract against *P. aeruginosa*.

TABLE 2 : Concentrations of Gallic Acid and Tannic Acid from Quercus infectoria galls extract

Types of solvent	Gallic acid (mg/g sample)	Tannic acid (mg/ g sample)
100% Methanol	51	1332
70% Methanol	71	1823
100% Ethanol	37	954
70% Ethanol	99	2512
100% Acetone	34	949
100% Aqueous	101	2975

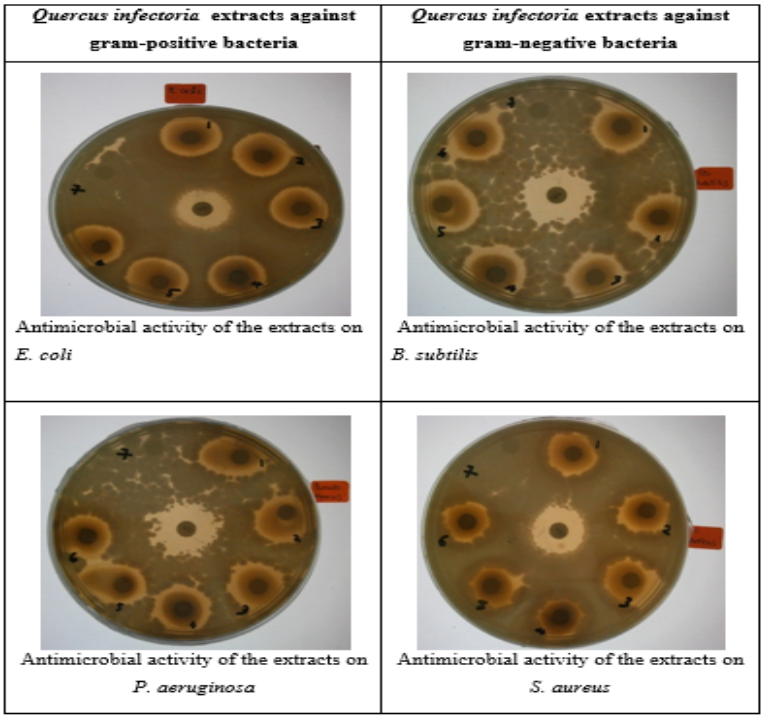

Fig.7: The antimicrobial activity of the extracts on *E. coli*: Disc diffusion test for the effect of *Quercus infectoria* against *E.coli* grown on nutrient agar medium. 1:100% methanol, 2:70% methanol, 3:100% ethanol, 4:70% ethanol, 5:100% acetone, 6:100% distilled water, 7: Negative control.

From Table 3, the antimicrobial properties of alcoholic and acetone extract were found to be superior compared to aqueous extract for those selected bacteria. This finding is in accordance with the result of a previous research which reported that the plant extracts using organic solvent exhibited more antibacterial activity compared to the extract using aqueous as a solvent (Turkmen *et al*., 2006; Parekh *et al*., 2005). Similarly, Basri and Fan (2005) have also documented the potential of aqueous and acetone extracts of galls as antibacterial agents. The most interesting finding is that the inhibition zone showed by all the bigger extracts compared to the positive control against *E. coli*. This suggests that *E. coli* is more susceptible to the extracts compared to commercial antibiotics (streptomycin).

TABLE 3: Antibacterial activity of different extracts of Manjakani galls at 24 hours

Types of Solvent	Diameter of inhibition zone (mm)± S.D			
	E. coli	*P. aeruginosa*	*B. subtilis*	*S. aureus*
100% methanol (1)	17±0.00[a]	13±.0.14 [a]	16±0.07 [a]	16±0.21 [a]
70% methanol (2)	16±0.07 [a]	15±0.14 [a]	17±0.00 [a]	15±0.21 [a]
100% ethanol (3)	17±0.00 [a]	14±0.21 [a]	18±0.21 [a]	15±0.21 [a]
70% ethanol (4)	17±0.14 [a]	15±0.14 [a]	17±0.07 [a]	14±0.21 [a]
100% acetone (5)	18±0.14 [a]	12±0.21 [a]	19±0.14 [a]	14±0.14 [a]
100% aqueous (6)	15±0.00 [a]	13±0.14 [a]	17±0.14 [a]	14±0.21 [a]
Neg. control (7)	-	-	-	-
Streptomycin	15±0.07 [a]	15±0.14 [a]	19±0.07 [a]	17±0.21 [a]

Each bar represents means ± SD of three replicates. Different letters on the bars indicate that groups are significantly different from each other according to Tukey's test ($p<0.05$).

Meanwhile, the antibacterial activity of the extracts might be due to the presence of gallic acid and tannic acid, which are derivatives of tannins. Tannins are reactive with the secreted extracellular enzymes and bacteria cell wall. This bioactive compound will destroy the cell walls of the bacteria and alter the transportation of nutrients into the cell to hinder the growth of this microorganism. This fact can explain the significant inhibitory effect of the *Quercus infectoria* extracts and this current finding is in accordance with the previous study reported by Li *et al*. (2011) who claimed that tannins from *Terminalia chebula Fructus Retz.* contain antibacterial activity.

CONCLUSION

In this study, the HPLC analysis had identified and quantified the gallic acid and tannic acid in the *Quercus infectoria* (manjakani) gall extract. The results demonstrated that conventional soxhlet extraction is efficient to extract the bioactive compounds from the plants. The strong antioxidant and antibacterial activities of the *Quercus infectoria* extracts are probably due to the bioactive compounds that are present in the plants. However, further investigation of individual phenolic compounds, their in vivo antioxidant and antibacterial activity, is necessary before advocating the application of *Quercus infectoria* in pharmaceutical and food products.

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Definition: Significant new information to readers of the Journal in a short but complete form. It is suitable for the publication of technical advance, bioinformatics or insightful findings of plant and animal development and function.

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      George Swan[1] and Nayan Kanwal[2]
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      [2]Office of the Deputy Vice Chancellor (R&I), Universiti Putra Malaysia, Serdang, Malaysia.

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   Swan and Kanwal (2007) reported that …
   The results have been interpreted (Kanwal *et al.* 2009).

   - References should be listed in alphabetical order, by the authors' last names. For the same author, or for the same set of authors, references should be arranged chronologically. If there is more than one publication in the same year for the same author(s), the letters 'a', 'b', etc., should be added to the year.

   - When the authors are more than 11, list 5 authors and then et al.

   - Do not use indentations in typing References. Use one line of space to separate each reference. The name of the journal should be written in full. For example:

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     - Jalaludin, S. (1997b). The use of different vegetable oil in chicken ration. *Malayan Agriculturist, 11,* 29-31.
     - Tan, S. G., Omar, M. Y., Mahani, K. W., Rahani, M., & Selvaraj, O. S. (1994). Biochemical genetic studies on wild populations of three species of green leafhoppers *Nephotettix* from Peninsular Malaysia. *Biochemical Genetics*, *32,* 415 - 422.

   - In case of citing an author(s) who has published more than one paper in the same year, the papers should be distinguished by addition of a small letter as shown above, e.g. Jalaludin (1997a); Jalaludin (1997b).

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   Proceedings: Kanwal, N. D. S. (2001). Assessing the visual impact of degraded land management with landscape design software. In Kanwal, N. D. S., & Lecoustre, P. (Eds.), *International forum for Urban Landscape Technologies* (p. 117-127). Lullier, Geneva, Switzerland: CIRAD Press.

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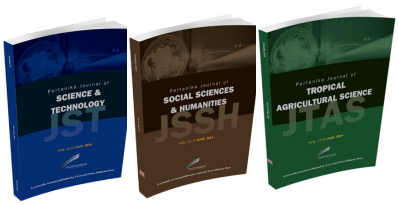

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