POLYLACTIC ACID/THERMOPLASTIC TAPIOCA STARCH BLEND INCORPORATED WITH SELECTED ESSENTIAL OILS FOR ACTIVE FOOD PACKAGING

Adamu Dahiru Ajiya

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ABSTRACT

The environmental effect of petroleum-based polymers and the activity of food spoilage microorganisms made researchers work on alternative bioactive packaging materials. This work aimed at producing sustainable thermoplastic tapioca starch (TPTS) and its polylactic acid (PLA) blend with introduction of antimicrobial (AM) activity via incorporation of lemongrass (LG), lemon balm (LB) and pandan (PA) essential oils (EOs) on the films. The plasticising effect of glycerol and water and compatibilising effect of vinegar loadings on the properties of TPTS were tested. So also the impact of TPTS loading on the PLA/TPTS blend properties. Furthermore, the AM activity of TPTS incorporated with the EOs on *E. coli*, *B. cereus* and *S. marcescens* was evaluated by inhibitory zone; and packaging effect of PLA/TPTS films coated with the EOs on death rate of microorganisms inoculated on *Bahulu* cake was investigated. The TPTS was formed by tape casting method, while PLA/TPTS film by hot pressing. The results indicated an increase in crystallinity and elongation at break with increase in plasticiser loading in TPTS and percentage TPTS loading in PLA/TPTS blend; both were accompanied by a decrease in T_g and tensile strength. Continuous surface morphology was seen at high plasticiser loading in TPTS, while high TPTS loading manifested a phase separation in PLA/TPTS blend. The results show that 125 °C processing temperature, 15 wt% glycerol, 45 wt% water and 3.5 wt% vinegar loading were optimum parameters for TPTS production. Whereas, 185°C processing temperature and 25 wt% TPTS loading are optimum parameters for PLA/TPTS blend production. Citral was found as the common active compound among the EOs. LG and LB were active against all the tested microorganisms, while PA was very mild at higher concentration. For active TPTS and PLA/TPTS films activity, *E. coli*: LB>LG>PA; *B. cereus*: LG>LB>PA; *S. marcescens*: LB>LG>PA. The TPTS and PLA/TPTS active films produced have shown improved properties for food packaging and effective AM activity against the selected microorganisms.

ABSTRAK

Kesan alam sekitar daripada polimer berasaskan petroleum dan aktiviti mikroorganisma yang membinasakan makanan membuat penyelidik bertumpu pada bahan pembungkusan bioaktif alternatif. Kajian ini bertujuan menghasilkan adunan lestari termoplastik kanji ubi kayu (TPTS) dan asid polilaktik (PLA) dengan aktiviti antimikrob (AM) yang diperbaiki menerusi gabungan minyak pati (EOs) *serai* (LG), *lemon balm* (LB) dan *pandan* (PA). Kesan pengekstrakan gliserol dan air dan kesan keserasian kandungan cuka ke atas sifat-sifat TPTS telah diuji. Begitu juga kesan kandungan TPTS pada sifat gabungan PLA/TPTS. Tambahan pula, aktiviti AM TPTS yang digabungkan dengan EOs pada *E. coli*, *B. cereus* dan *S. marcescens* dinilai oleh zon penghalang; dan kesan pembungkusan filem PLA/TPTS yang disalut dengan EOs pada kadar kematian mikroorganisma yang diumpukkan pada kek Bahulu telah disiasat. TPTS terhasil menerusi kaedah tuangan pita, manakala filem PLA/TPTS terhasil menerusi penekanan panas. Hasil dapatan menunjukkan peningkatan dalam kehabluran dan pemanjangan patah dengan pertambahan kandungan pemplastik dalam TPTS serta peratus kandungan TPTS dalam adunan PLA/TPTS, kedua-duanya dituruti dengan pengurangan T_g dan kekuatan tegangan. Morfologi permukaan yang berterusan dilihat pada kandungan pemplastik yang tinggi dalam TPTS, manakala kandungan TPTS yang tinggi menunjukkan pemisahan fasa dalam adunan PLA/TPTS. Keputusan menunjukkan bahawa pada suhu pemprosesan 125°C, 15 wt% berat gliserol, 45 wt% air dan 3.5 wt% cuka adalah parameter optimum untuk pengeluaran TPTS. Sementara itu, suhu pemprosesan 185 °C dan 25 wt% TPTS adalah parameter optimum untuk pengeluaran PLA/TPTS. Citral dijumpai sebagai sebatian aktif yang biasa di kalangan EOs. LG dan LB aktif terhadap semua mikroorganisma yang diuji, sementara PA kurang berkesan pada kepekatan yang lebih tinggi. Untuk aktiviti filem aktif TPTS dan PLA/TPTS, *E. coli*: LB>LG>PA; *B. cereus*: LG>LB>PA; *S. marcescens*: LB>LG>PA. Filem aktif TPTS dan PLA/TPTS yang terhasil telah menunjukkan sifat yang dipertingkatkan sebagai pembungkus makanan dan aktiviti AM berkesan terhadap mikroorganisma terpilih.

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LIST OF SYMBOLS AND ABBREVIATIONS

μ	-	death rate
α	-	Alpha crystalline structure
ε	-	Elongation at break (%)
σ	-	Tensile stress (MPa)
°C	-	Degree Celsius
μm	-	Micrometre
ANOVA	-	Analysis of variance
Au × nm	-	Area under curve × nanometer
ASTM	-	American society for testing and materials
BCM	-	Bacillus cereus medium
CEN	-	European standardisation committee
DIN	-	German institute for standardisation
DMA	-	Dynamic mechanical analysis
DSC	-	Differential scanning calorimetry
DTG	-	Derivative thermogravimetry
E’	-	Storage elastic modulus
E”	-	Loss viscous modulus
EMB	-	Eosin methylene blue
FTIR	-	Fourier transform infrared
g	-	gram
GCMS	-	Gas chromatography mass spectrometry
HPMC	-	Hydroxypropyl methyl cellulose
ISO	-	International Standards Organisation
LAB	-	Lactic acid bacteria
MBC	-	Minimum bactericidal concentration
MIC	-	Minimum inhibition concentration
min	-	minute

mm - millimeter
N - Population
NA - Nutrient agar
NB - Nutrient Broth
ORCA - Organic reclamation and composting association
PDA - Potatoes dextrose agar
PLA - Polylactic acid
PVC - Polyvinyl chloride
rpm - Revolution per minute
s - second
SEM - Scanning electron microscopy
t - Time
Tan δ - Damping coefficient
T_g - Glass transition temperature
TGA - Thermogravimetric analysis
T_m - Melting temperature
TPS - Thermoplastic starch
TPTS - Thermoplastic tapioca starch
XLD - Xylose lysine deoxy-cholate
XRD - X -ray diffraction
UV - Ultraviolet

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CHAPTER 1

INTRODUCTION

1.1 Research background

The plastics have immense importance in modern life; its function is universal ranging from household articles, self-healing materials, clothes, shoes, packaging, up to auto parts. The primary source of plastics is the fossil fuel. The increased change of petroleum resource price in the 1970s extend the consciousness of researchers toward alternative sources for plastics, which is one of the significant products of fossil fuel (Wittcoff *et al.*, 2013). The rate of depletion of fossil fuel is among other factors of concern (Pervaiz *et al.*, 2014). This limited resource is bound to finish in the midst of an uneven price system. It is forecasted that fossil fuel will be depleted averagely by 2100 if the current rate of consumption is maintained (IEA, 2017). Secondly, the environmental pollution caused by the excessive volume of non-biodegradable plastic waste materials poses a threat to life and wellbeing of the environment. This issue is coupled with inadequate landfill space for disposal, the high maintenance cost of landfill, and non-degradability of the plastics for about a thousand years (Cressey, 2016).

The cumulative amount of plastics produced globally between 1950 and 2015 was around 7.82 billion tonnes. Most of these products were used in the field of packaging. One unique characteristic of plastic packaging is that it is used once after which it is discarded, particularly the food packaging where contamination by the microorganisms is involved (Geyer *et al.*, 2017). By 2015 the packaging sector accounted for 42 % (141 million tonnes) of global plastic produced. While during the same period, packaging constituted about 50 % of global plastic waste (Hannah & Max, 2018).

In Malaysia a similar trend was observed, packaging constituted 45 % of plastics produced. Also, about 24 % of municipal solid waste is made of plastic materials, even though, the percentage of packaging material was not quantified (Zainua & Songip, 2017). But research shows that about 22-55 billion shopping bags are sold per year in Malaysia (Sung, 2017). This activity constitutes a major environmental concern because only 5 % of the plastics are recycled, 42 % of the municipal waste is incinerated which brings about air pollution. On the other hand, the rest of the 53 % are dumped in landfills, and the plastics in this category does not degrade quickly, destroying the environment in open landfills (Zainua & Songip, 2017). Disposable food package provides the benefit and convenience of one time, sanitary use. Thin films used in such products are typically made from water-insoluble synthetic polymers or polymer blends. However, the disposal of these products is a concern due to limited landfill space. Incineration of such products is not desirable because of increasing alarm about greenhouse gases generation. Consequently, there is a need for bio-degradable products which may be quickly and conveniently disposed of without dumping or incineration (Gabor & Tita, 2012). It has been proven by series of researches (Iwate, 2015; Roshafima *et al.*, 2012; Seligra *et al.*, 2016) that petroleum-based plastics degrade eventually, but that process usually takes a very long time and contribute to global warming through the release of carbon dioxide and methane gas. Therefore, research in alternative renewable, biodegradable, eco-friendly and cost-effective raw material for the production of plastics flourish.

Biodegradable plastics are made out of ingredients that can be metabolised by naturally occurring micro-organisms in the environment. Bio-degradable films and coatings have been mainly considered in food preservation, because of their capability for improving global food quality (McKeen, 2012). Recently, innovative ways to inhibit microbial growth in the foods while maintaining quality, freshness, and safety have been investigated. One option is the use of packaging to provide protection, quality and longer shelf life (Priyaa *et al.*, 2014). Packaging is the most significant process aimed at giving the stable condition of food products for their storage, transportation, distribution, and consumption. The primary function of packaging is protection from mechanical damage and prevention or inhibition of chemical changes, biochemical changes and microbiological spoilage (Biron, 2007). Food products during packaging can be subjected to various type of contaminations including microbial contamination that is mainly caused by bacteria, yeasts and mould (Malhotra *et al.*, 2015; Prasad & Kochhar, 2014). Specifically, food products are sensitive and their shelf life is limited by their interaction with packaging materials that can alter their water activity, pH, added preservatives and also substances that can affect their temperature, relative humidity, light and gas composition (Lucera *et al.*, 2016; Miranda *et al.*, 2016). One of the significant possibility to extend the shelf life of the products is to develop packaging material with specific properties such as pH, water activity, respiration rate and existence of antimicrobial compounds which is referred to as bioactive packaging material and can be used to improve the quality of food products and to extend their shelf life (Du *et al.*, 2011). In food packaging, the goal is to use bioactive materials to get a desirable response, for example, the inhibition of microbial growth, barrier adjusting materials, as well as flavour maintenance and enhancing material properties. Most bio-based materials such as polysaccharides and protein-based polymers (starch, cellulose, alginate, collagen, gelatin, and proteins) are hydrophilic with a relatively high degree of crystallinity causing processing and performance difficulties. Therefore, antimicrobial (AM) packages made from such bio-based films demonstrate high moisture sensitivity, poor water barrier and poor mechanical properties compared to those made from synthetic polymers such as polyvinyl chloride (PVC), polypropylene (PP) and polyethylene terephthalate (PET) (Roshafima *et al.*, 2012). Packaging materials with suitable physicomechanical properties can be prepared from biopolymers such as starch-based materials when the contents are modified by physical, mechanical and chemical techniques or by blending with compatible plasticisers (McKeen, 2012).

The production of starch globally was estimated to be around 75 million tons in 2012, where maize, tapioca (cassava), wheat and potato were the major plants for starch production with an amount of rice and other starches being produced (Waterschoot *et al.*, 2015). Tapioca starch global production was estimated to reach a volume of 6.7 million tons, and the report forecast a 1.6 % growth annually where it will be 7.4 million tons by 2023 (IMARC, 2018). It was reported that Malaysia grows about 400,000 tonnes/year of tapioca, which is large enough to earn a sustainable income to farmers, and provide raw materials to the industries (Hillocks *et al.*, 2002; UFAO, 2012). But another report shows that Malaysia’s tapioca production in 2016 stood at 77,980 tonnes declining from 400,000 tonnes in 1989 due to market and other factors (Factfish, 2017).

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