

# **STUDIES ON PRETREATMENT AND BIOSUGARS RECOVERY FROM OIL PALM FROND JUICE**

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## ABSTRACT

Oil palm (*Elaeis guineensis jacq*) frond (OPF) has a great potential to be commercialized due to its abundance as an oil palm solid wastes that are generated during oil palm agriculture and harvest. OPF juice has been identified as a good candidate to replace pure or technical grade sugars for the production of value-added products. Recently, it has been reported that OPF juice can be used as the sole renewable carbon source for the production of poly (3-hydroxybutyrate), P(3HB). In order to further evaluate the future prospect of OPF juice as fermentation feedstock, in-depth investigation on the bioconversion of OPF juice to produce other valuable products such as lactic acid and bioethanol will be the focus of future research project. Prior to that, OPF juice has to be treated to remove the precipitate as well as others impurities. This study has to be done at the first place in order to further enhance the sugars recovery to increase the concentration and purity of the renewable sugars contained in the OPF juice. In order to achieve this objective, three different pretreatment processes including chemical treatment with calcium chloride, heat and chemical treatment with hydrogen peroxide and activated carbon adsorption was applied. The process parameters obtained from sugar analysis provided an insight information about the effect of the different pretreatment methods on sugar content in OPF juice. The treated juice was subjected to further process which is purification and recovery using conventional method i.e., crystallization and spray drying.

## ABSTRAK

Pelepah kelapa sawit (*Elaeis guineensis Jacq*) (OPF) mempunyai potensi besar untuk dikomersialkan kerana banyak didapati sebagai sisa pepejal kelapa sawit yang dihasilkan semasa pertanian dan penuaian kelapa sawit. Jus OPF telah dikenal pasti sebagai calon yang baik untuk menggantikan gula gred tulen atau teknikal bagi pengeluaran produk nilai tambah. Baru-baru ini, telah dilaporkan bahawa jus OPF boleh digunakan sebagai sumber karbon baharu bagi pengeluaran poly (3- hydroxybutyrate), P(3HB). Untuk menilai lagi prospek masa depan jus OPF sebagai penapaian bahan mentah, kajian yang mendalam mengenai penukaran biologi jus OPF untuk menghasilkan produk bernilai yang lain seperti asid laktik dan bioethanol akan menjadi tumpuan projek penyelidikan masa depan. Sebelum itu, jus OPF perlu dirawat untuk menghapuskan mendakan serta lain-lain kekotoran. Kajian ini perlu dilakukan pada permulaan langkah bagi meningkatkan lagi pemulihan gula untuk meningkatkan kepekatan dan ketulenan gula boleh diperbaharui yang terkandung dalam jus OPF. Untuk mencapai matlamat ini, tiga proses pra-rawatan yang berbeza termasuk rawatan kimia dengan kalsium klorida, haba dan rawatan kimia dengan hidrogen peroksida dan penjerapan karbon aktif akan digunakan. Parameter proses yang diperolehi daripada analisis gula menyediakan gambaran maklumat mengenai kesan daripada kaedah rawatan awal yang berbeza pada kandungan gula dalam jus OPF. Jus yang telah dirawat seterusnya melalui proses iaitu penulenan dan perolehan menggunakan kaedah konvensional iaitu, penghabluran dan pengering semburan.

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## LIST OF ABBREVIATIONS

### *Latin*

*i.e* id est/ that is

### *Subscripts*

*hrs* hour

*g* gram

*rpm* rotation per minute



## **LIST OF ABBREVIATIONS**

<b>FFB</b>	<b>Fresh Fruit Bunch</b>
<b>HPLC</b>	<b>High Performance Liquid Chromatography</b>
<b>OPEFB</b>	<b>Oil Palm Empty Fruit Bunch</b>
<b>OPF</b>	<b>Oil Palm Frond</b>
<b>OPT</b>	<b>Oil Palm Trunk</b>

# 1 INTRODUCTION

## *1.1 Motivation and statement of problem*

It is generally acknowledged that the world today faced an unprecedented set of problems relating to the environment which includes issues relating to ecological destruction, resource depletion and atmospheric change, i.e. global warming. We are already using the planets renewable resources faster than what the planet can replenish. The world population at an estimated 7 billion people today is projected to grow to over 10 billion people as early as 2050. If the planet is already struggling to cope with the demands placed upon it by the human race currently, world needs to find a reliable source of energy urgently to ensure a sustainable source of energy demand. Renewable energy has been identified globally as the perfect solution to this matter. Renewable energy could be a key driver to achieve economic growth while ensuring minimal environmental harm. Besides that, food price is likely continue to rise for many years to come, making poor farmers, consumers and countries more vulnerable to poverty and food insecurity. Demand from consumers in rapidly growing economies will increase, the population continues to grow, and further growth in biofuels will place additional demands on the food system and more people will continue suffer from lack of food.

On the other hand, feedstock for microbial fermentation today is currently taken from edible food source, such as soy bean, malt and glucose that were also consumed by humans and animals. Competition on food consumption occurs between the needs for growth of human and animals and microbes may affect the food chain survival. Thus, studies on potential of biomass to be utilized as a source of fermentable sugars is carried out to reduce the production cost and the dependence on the food crops.

Presently, as the world second largest producer and exporter of palm oil in 2006 (USDA, 2007), million hectares of land in Malaysia is occupied with oil palm plantation generating huge amount of biomass, way much larger as compared to other types of biomass. Oil palm, known as *Elaeis guineensis jacq* is indigenous to West Africa but is now planted in all tropical areas of the world. Moreover, it has become the most important industrial crops especially in certain South East Asia countries like Malaysia, Indonesia and Thailand. Palm oil industry generates almost 94% of the biomass

feedstock while agricultural and forestry by-products such as wood residues, rice and sugar-cane contributes the remaining 6%. Due to the huge amount of biomass generated yearly, oil palm industries appears to be a very promising alternative as a source of raw materials for renewable energy in Malaysia.

### ***1.2 Problem statement***

Feedstock for microbial fermentation today is currently taken from edible food source that were also consumed by humans and animals. Competition on food consumption occurs between the needs for growth of human and animals and microbes. Moreover, market price for pure substrate is way expensive. Presently, millions of hectares of land in Malaysia is occupied with oil palm plantation generating huge quantities of biomass, especially oil palm fronds. Therefore, oil palm biomass is seen as good source due to its abundance and it is cheaper. The aim of this study was to develop an economical bioprocess to produce the renewable sugars as fermentation feedstock at laboratory scales using Oil Palm Frond juice (OPF). In order to further evaluate the future prospect of OPF juice as fermentation feedstock, in-depth investigation on pretreatment and sugar recovery on OPF juice will be carried out in order to further enhance the sugars recovery to increase the concentration and purity of the renewable sugars contained in the OPF juice.

### ***1.3 Objectives***

The following are the objectives of this research:

- i) To study the effect of pretreatments using activated carbon on OPF juice
- ii) To study the effect of different sugar recovery methods on optimum treated OPF juice

### ***1.4 Scopes***

The following are scopes of the contributions

- i) Investigate the effect of absorption using different activated carbon's size, dosage, contact time and agitation speed to remove precipitates and impurities during oil palm frond juice pretreatment.
- ii) Perform batch tests to determine equilibrium parameters for the activated carbon adsorption

- iii) Investigate the feasibility and optimal dosing for hydrogen peroxide pretreatment
- iv) Determine the effectiveness sugar recovery method on OPF juice
- v) Identify which method is the most effective method for sugars recovery, either crystallization or spray drier on optimum treated OPF juice.

### ***1.5 Expected outcome***

The process parameters obtained from sugar analysis will provide an insight information about the effect of the different pretreatment methods on sugar content in OPF juice. Parameters such as reaction temperature, reaction time and agitation speed, may differ from each methods; heat and chemical treatment, activated carbon. Besides, recovery process using crystallization and spray drying technique also expected to give distinct observation which can retain more amount of pure sugar between these two methods.

### ***1.6 Significant of proposed study***

The increasing price of fuel and abundance of biomass waste are global problem faced around the world. This study is significant to researchers, engineers, and manufacturer who interest in developing source of carbon production in a new approach which reduce the cost involving raw materials. Besides, this study can help to preserve the environment as well as maintain economic balance alongside the exponential rise of energy demand as it helps to reuse biomass waste as renewable, sustainable and cheap fermentation feedstock for value-added products.

### ***1.7 Organization of this thesis***

The structure of the reminder of the thesis is outlined as follow:

Chapter 2 provides a description on oil palm industry in Malaysia and the oil palm biomass production. In order to manage the abundance of oil palm biomass, many research projects have been carried out to utilize this biomass into renewable sources of energy. The aim of this study was to develop an economical bioprocess to produce the renewable sugars at laboratory scales using Oil Palm Frond juice (OPF). OPF waste generated by oil palm plantations is a major problem in terms of waste management.

However, this lignocellulosic waste material is a cheap source of cellulose. The high content of cellulose in OPF promises the high fermentable sugars production. However, OPF juice need to be treated in order to further enhance the sugars recovery to increase the concentration and purity of the renewable sugars contained in the OPF juice.

Chapter 3 will be focused on pretreatments and sugar recovery on OPF juice in order to further enhance the sugars recovery to increase the concentration and purity of the renewable sugars contained in the OPF juice. This objective can be achieved by several methods including chemical and heat treatment and activated carbon absorption. The conditions for the refinement of clarified sugarcane juice using different activated carbon's size, dosage, contact time and agitation speed to remove precipitates and impurities during absorption using activated carbon were investigated. The clarified juice obtained was analyzed using High Performance Liquid Chromatography (HPLC). The characteristics investigated included brix, polarity (sucrose percent), turbidity, color and purity. The treated juice will be subjected to further process which is purification and recovery using conventional method, i.e. crystallization and spray drier.

Chapter 4 discussed on the observation or any data from the preliminary works and some discussion over the results obtained. This chapter will be focusing on the studies that are carried out to find out the optimum pretreatment conditions for OPF juice. Response on OPJ juice's sugar content, colour, turbidity and Brix was observed for the heat and chemical treatment using hydrogen peroxide, calcium chloride and simultaneous analysis of pretreatment conditions for activated carbon adsorption like dosage, agitation speed and contact time. This section also deals with an important technique for handling solids that aren't pure. The technique is called crystallization, and it is the most widely used routine procedure for purifying solids. Other than that, spray drying methods which turns OPF juice to powder form.

## **2 LITERATURE REVIEW**

### ***2.1 Overview***

This paper presents the brief description on oil palm industry in Malaysia and the oil palm biomass production. In order to manage the abundance of oil palm biomass, many research projects have been carried out to utilize this biomass into renewable sources of energy. The aim of this study was to develop an economical bioprocess to produce the renewable sugars at laboratory scales using Oil Palm Frond juice (OPF). OPF waste generated by oil palm plantations is a major problem in terms of waste management. However, this lignocellulosic waste material is a cheap source of cellulose. The high content of cellulose in OPF promises the high fermentable sugars production. However, OPF juice need to be treated in order to further enhance the sugars recovery to increase the concentration and purity of the renewable sugars contained in the OPF juice.

### ***2.2 Oil palm industry***

Oil palm, known as *Elaeis guineensis jacq* is indigenous to West Africa where it grows in the wild and later was developed into an agricultural crop. Now it is planted in all tropical areas of the world. Moreover, it has become the most important industrial crops especially in certain South East Asia countries like Malaysia, Indonesia and Thailand (Shuit et al., 2008). Today, 4.49 million hectares of land in Malaysia are under oil palm cultivation; producing 17.73 million tonnes of palm oil and 2.13 tonnes of palm kernel oil. Malaysia currently accounts for 39 % of world palm oil production and 44% of world exports. If taken into account of other oils & fats produced in the country, Malaysia accounts for 12% and 27% of the world's total production and exports of oils and fats (Anthony, 2013). Besides, for the past 30 years Malaysia has been a world leader regarding organizing an efficient and highly productive commercial oil palm plantation sector, as well as in pioneering important agricultural research and varietal development that benefited palm oil producers throughout the region.

### ***2.3 Oil palm biomass***

In spite of the huge production, the oil consists of only about 10% of the total biomass produced on the plantation. The remainder consists of the huge amount of oil palm

wastes such as oil palm shells, mesocarp fibers and empty fruit bunch (from the mills) and oil palm fronds and oil palm trunk (from the field during replanting). It is estimated that the total potential oil palm biomass from 4.69 million hectares of oil palm in Malaysia in 2009 is 77.24 million tonnes per year comprising 13.97 million tonnes of oil palm trunks, 44.84 million tonnes of palm fronds, 6.93 million tonnes of EFB, 4.21 million tonnes of oil palm shell and 7.29 million tonnes of mesocarp (all dry weight), as shown in figure 2-1 below:

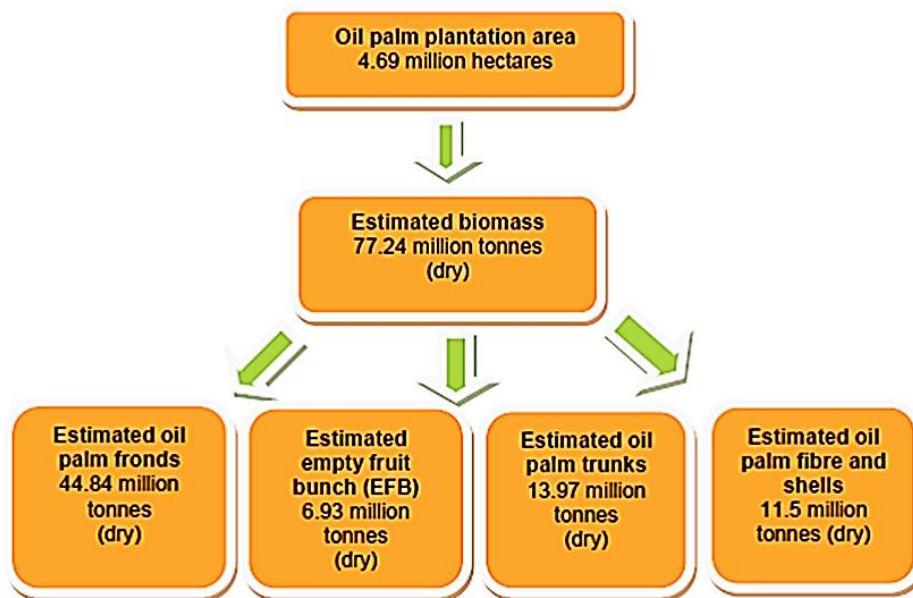


Figure 2-1: Potential availability of biomass from oil palm (Foo et al., 2011)



Figure 2-2: Oil palm trees

## 2.4 Oil palm fronds

Oil palm fronds (OPF) are lignocellulosytic biomass that is an inexpensive raw material containing 40 to 60 percent cellulose, 20 to 40 percent hemicellulose and 15 to 30 percent lignin. In the plantations, OPFs are available throughout the year as they are regularly cut during harvesting of fresh fruit bunches (FFBs) and pruning of the palm trees. The availability of fronds during the pruning activity was calculated using an estimate of 10.4 tonnes ha<sup>-1</sup>, which currently gives an average of 6.97 million tonnes per year. Meanwhile, it was estimated at an average of 54.43 million tonnes per year of oil palm fronds will be available during the replanting process in the years of 2007 - 2020. Additional fronds as well as oil palm trunks (OPT) become available in the plantations during the replanting of oil palm trees every 25 to 30 years. The OPF is obtained during pruning to harvest the fresh fruit bunch (FFB), therefore it is available daily. OPF is currently under-utilized as the plantation owners believe that all the OPF is necessary for nutrient recycling and soil conservation (Hassan et al., 1994; Wan Zahari et al., 2002). Hence, pruned fronds are just left on the plantation. However, our study shows that OPF does not contain high metal contents as widely thought, but contain high levels of carbohydrates in the form of simple sugars. Therefore, part of the OPF can be utilized for other purpose without scarifying the nutrient recycling process.

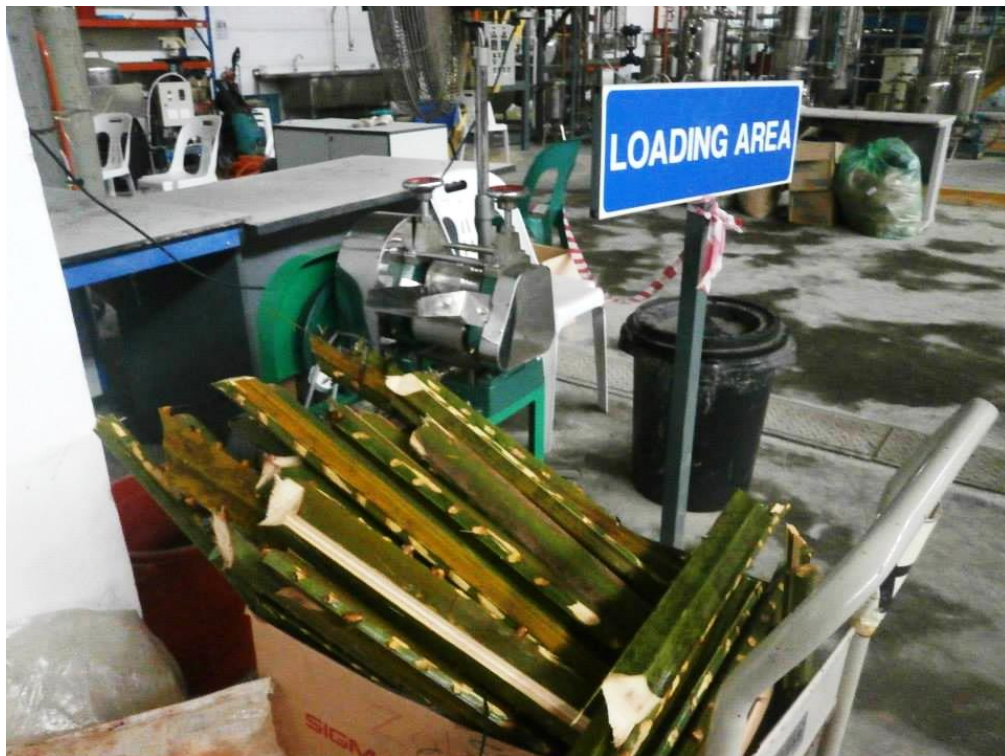


Figure 2-3: Oil palm fronds, without leaves



## ***2.5 Renewable sugars from oil palm biomass***

Studies have been done to utilize the oil palm biomass efficiently, for example to convert the oil palm empty fruit bunch (OPEFB) into fermentable sugars (Kader et al., 1999; Ariffin et al., 2006 and 2008a; Roslan et al., 2011), utilization of the OPEFB as substrate for enzyme production (Umikalsom et al., 1997; Ariffin et al., 2008b) and pulp preparation from OPEFB (Rushdan, 2002).

Apart from OPEFB, research has been done on sugars production from oil palm trunk (OPT). Recently, Kosugi et al. (2010) found that OPT contains high amounts of readily available sugars. Characterization of the sap from the inner part of the OPT revealed that a large amount of glucose (85.2 g/l) and some sucrose and fructose were present in the sap. In 2009, about 15.2 and 17.5 million tonnes (wet weight) of OPT and OPEFB were generated in Malaysia. However, the most abundant biomass from oil palm plantations is not OPEFB or OPT. The most generated oil palm biomass is oil palm frond (OPF), which amounted to 83 million tonnes (wet weight) annually (MPOC, 2010).

Production of sugars from dried OPF fiber has been recently reported by Fazilah et al., (2009) and Goh et al., (2010), involving the conversion of cellulose and hemicellulose into glucose and xylose through hydrothermal treatment followed by enzymatic hydrolysis. However, the methods used for converting OPF into sugars still involve the use of high temperature and pressure, and also cellulase enzymes. The overall process is costly. There is a need for an alternative approach of producing renewable sugars from this waste of the oil palm industry as fermentation feedstock, wherein the production method does not require harsh pre-treatment steps and the use of enzymes. This method will need to be cost-effective and sustainable to support the biotechnology industries in the long term.

## ***2.6 Renewable sugars from oil palm fronds juice***

Meanwhile, OPF juice has been identified as a good candidate to replace pure or technical grade sugars for the production of value-added products. Recently, it was reported that the pressed juice from oil palm frond (OPF) contained renewable sugars such as glucose, sucrose and fructose. By using a simple sugarcane press, 50% (wt/wt) of OPF juice was obtained from fresh OPF. The glucose content in the juice was 53.95

$\pm 2.86$  g/l, which accounts for 70% of the total free sugars. The sugars from the OPF juice can be used for various applications including the production of various products which includes polyhydroxyalkanoates such as polymer of hydroxyalkanoic acid, hydroxybutyric acid, hydroxyvaleric acid, and a copolymer thereof, wherein the copolymers maybe poly(hydroxybutyrate-co-hydroxyvalerate)(PHBV), poly (3-hydroxybutyrate-co-4-hydroxybutyrate) (P3HB4HB), polymers and/or copolymers of hydroxyterminated polyhydroxybutyrate (PHB-OH), heteropolymers thereof and any other polymers. In addition to that, biofuels and organic acids such as bioethanol, biobutanol, lactic acid, succinic acid, and all products that can be produced through chemical and biological synthesis from these sugars of OPF juice.

A closer look on the utilization of OPF juice can be seen through the production of the polyhydroxyalkanoate (PHA) as PHA can be conducted by incubating at least one strain of a PHA-producing microorganism in a culture medium comprising of the said sugars and/or a derivative thereof. The microorganisms used can be of bacteria, mold or yeast from the group Azotobacter, Pseudomonas, Coliform, Alcaligenes, Bacillus, Lactobacillus and genetically modified form thereof. More specifically, as reported by Zahari et.al (2013), the effect of various OPF juice concentrations on the production of poly (3-hydroxybutyrate), P (3HB) can be examined by *Cupriavidus necator* CCUG 52238T. The cell dry mass in shake flask experiment reached 8.42 g/l, with 32 wt % of P(3HB) at 30% (v/v) of OPF juice, comparable with using technical grade sugars. This result indicates that OPF juice can be used as an alternative renewable carbon source for P(3HB) production and has potential as a renewable sugar for microbes fermentation feedstock (Zahari et al., 2012).

In order to further evaluate the future prospect of OPF juice as fermentation feedstock, this research will be focused on pretreatments and sugar recovery on OPF juice in order to further enhance the sugars recovery to increase the concentration and purity of the renewable sugars contained in the OPF juice.

## ***2.7 Pretreatment methods***

Pretreatment process is the series of refining operations. “Refined” is a misunderstood word, especially when it comes to sugar. Somehow, over the years, refined has taken on the meaning of being overly processed and manipulated. In truth, the definition of refine

is “to make pure” by removing impurities or unwanted elements from (a substance), typically as part of an industrial process. The refining process for sugar production is the process of purification by simply separates natural sucrose from the plant material, without bleaching or heavy chemical manipulation.

In order to further purify the raw sugar, it is necessary to remove the maximum quantity of impurities from the expressed juice at the earliest to obtain pure crystallized sugar. Basically, as similar to sugarcane juice, OPF juice consists of a sucrose solution containing soluble and insoluble impurities (non-sucrose species). Apart from sucrose, raw sugar contains reducing sugars (glucose and fructose), inorganic ash (mainly calcium and potassium salts) and other organic matter which includes gums, amino acids and colour components, essentially from the cane. In sugar refining, glucose and fructose are regarded as impurities due to the difficulty in crystallizing them from solution. Strict process control, particularly of pH, must be maintained to avoid loss of sucrose in processing through its chemical hydrolysis to the unwanted sugars glucose and fructose. It is assumed that, the main impurities of sugarcane juice include polysaccharides, starches, waxes, proteins, fibers and colorful polymers (Ghosh et al., 2000) can be similarly found in OPF juice. Moreover, the fresh juice squeezed from the non-pretreated sugarcane was exposed to the peroxidase activity test. Its quality attributes were unstable and rapidly changed along the time due to the enzyme activities and high microbial growth rate. Thus, non-pretreated sugarcane juice is not suitable to be stored at room temperature to be used in the future use. Meanwhile, pretreated sugarcane juice had a higher clarity, lower viscosity and reduced color. Consequent benefits included higher crystallization yield, energy savings due to reduction of steam consumption by the evaporator and increased capacity of evaporators, vacuum pans, crystallizers and centrifuges.

This objective can be achieved by several methods largely classified as chemical and heat treatment and activated carbon adsorption. The treated juice was subjected to further process which is purification and recovery using conventional method, i.e. crystallization and spray dryer.

### ***2.7.1 Chemical treatment***

Clarification is carried out to remove impurities which inhibit the formation of the crystals and can discolour the final product. Clarification of the syrup is done to reduce turbidity. Clarification is therefore an essential step to obtain high yields and high quality of the sugar. The clarification process needs to remove components other than sucrose and, at the same time, minimize loss of sucrose and color formation. Approximately 40% of the remaining colorants are removed in the clarification step. There are two alternative types of defecation processes in use in cane refineries, carbonation, phosphatation and sulphitation.

For the production of crystal sugar, sulphitation most widely used process to clarify cane juice. It consists of sulphur dioxide (SO<sub>2</sub>) absorption by the juice. Currently method to clarify sugar cane juice in the manufacture of crystal sugar is carbonation, which generally employs treatment with lime and controlled addition of carbon dioxide (CO<sub>2</sub>). However these processes are typically not used in the manufacture of raw sugar or refinery sugar due to their complexity and expense (Dionisi et al., 2009).

Chemical clarification, based on modern cold lime, sulphitation, is carried out to remove impurities which inhibit the formation of the crystals and can discolour the final product. Lime also reduces the natural acidity of the cane juice. Batches of juice are treated simultaneously with lime (as a milky solution) and sulphur dioxide (by air forced through a sulphur furnace), after which the juice is transferred to an open boiling pan and quickly heated to 90°C or above. The lime and heat treatment form a heavy precipitant that flocculates, carrying with it most of the suspended impurities in the juice. The juice is then filtered and allowed to settle down, the clear juice is decanted and transferred to the boiling furnaces.

#### ***2.7.1.1 Carbonation***

Carbonation is used for the removal of insoluble matter that contributes to the turbidity of the sugar liquor, but also provides a degree of decolorization. Carbonation also called as carbonatation, generally employs treatment with lime and controlled addition of carbon dioxide (CO<sub>2</sub>) (Dionisi et al., 2009). Lime pre-filtration was necessary as a pretreatment for OPF juice because of the impurities and high silt in raw OPF juice that prevents sucrose degradation. In the books entitled 'Cane Sugar Refining with Ion Exchange Resins' published by The Purolite Company, carbonation involves adding

lime (CaO) to the melt liquor and then passing this juice through a carbonation vessel or boiler flue gas where carbon dioxide (CO<sub>2</sub>) is bubbled up through the juice. Any suitable source of lime as a chemical agent can be employed, but lime milk, Ca(OH)<sub>2</sub> or calcium saccharides is preferred. The addition of lime raises the pH of the

#### ***2.7.1.2 Phosphatation***

Another alternative which is similar to carbonation process is phosphatation. Phosphatation involves the addition of lime (CaO) and phosphoric acid (H<sub>3</sub>PO<sub>4</sub> or P<sub>2</sub>O<sub>5</sub>) to the malt liquor which results in the formation of a calcium phosphate precipitate. Color bodies adsorb onto the calcium phosphate precipitate and are removed during the subsequent clarification and filtration. The calcium phosphate particles entrap some impurities and absorb others, and then float to the top of the tank, where they can be skimmed off. Polymers are added to aid in the formation of a precipitate floc which is more easily filtered.

#### ***2.7.1.3 Sulphitation***

Other options for clarification of raw juice involve the addition of lime and sulfur dioxide, followed by boiling the treated juice ('liming-sulphitation process'). The sludge thus formed is separated by settling and vacuum filtration.

#### ***2.7.2 Chemical treatment with calcium chloride***

The OPF juice is further treated to remove precipitate formed after extraction process. The step of removing precipitates by exposing OPF juice to NaOH and CaCl<sub>2</sub>. The amount of sulphuric acid and NaOH used in the step of removing of precipitates produces a pH between pH 10 to 12. The amount of CaCl<sub>2</sub> added is between 0 to 1 % (w/v). The precipitates formed were glucan, pectin and other polysaccharides (Ariffin et al., 2013).

#### ***2.7.3 Heat and chemical treatment with hydrogen peroxide***

The main function of the heat pretreatments in this work was to diminish the enzyme activities that normally cause quality deteriorations of sugarcane juice such as degreening and/or browning during processing and storage. The OPF juice will be

heated at 70 °C for 10 minutes, as refer to the optimum heat treatment for sugarcane juice (Chauhan et al., 2002).

The use of oxidizing agents in sugar production has been discussed in several studies (Moodley, 1992; Mendoza, 2002; Bento, 2004). Hydrogen peroxide and ozone have both been used as decolorizing agents in refineries and mills. Bento (2004) describes the mechanism of decolorization. Peroxide cleaves unsaturated double bonds, forming carboxylic acids. Davis (2001) states that bleaching occurs via free radical addition at allylic site and double bonds, with eventual decomposition to acids and alcohols. The action of the oxidants decreases color and molecular weight, increasing efficacy of ion exchange. Bento also states that peroxide used in conjunction with ion exchange resins to decolorize sugar solutions can produce several benefits. Those applicable to this research include:

- Less contaminated resin
- Extension of resin cycles by 3-5 times
- Less effluent of a lower color produced
- Decreased consumption of chemicals and utilities
- Decreased costs

Juice from the clarifier or first stage evaporator is first pretreated with an oxidizing agent, such as hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>). The pretreated juice is then passed through columns containing granular activated carbon (GAC) for decolorisation. The first column acts as a guard column, where fines and suspended solids are removed (Raul, 2007).

#### ***2.7.4 Activated carbon adsorption***

In liquid sugar production, activated carbon is used for colour removal to produce the crystal syrups demanded by the food and beverage industries. In addition to decolourizing, activated carbon also removes taste and colour compounds by adsorption. Activated carbon may be used either alone or in conjunction with other decolourizing agents and systems.

Activated carbons function via adsorption Van der Waals forces cause solute particles to be pulled out of solution and adhere to the carbon surface. The undesired particle has a greater affinity for the carbon than for the solution. The non-polar characteristic of carbon allows for adsorption of similar compounds, but polar compounds are not typically adsorbed. However, chemisorption may occur due to oxygenated functional groups that remain from the activation process. This allows for polar molecules to also be removed from the solution. Activated carbons have been reported to be highly effective for phenol and flavonoid removal (Davis, 2001). The weakly acidic nature of colorants allows for electrostatic linkage to the adsorbent surface, while the neutral sugar remains unaffected (Ellis, 2004). Therefore, an 80% typical colorant removal can be achieved when filtering clarified sugar juice.

Activated carbon is used in sugar industries for the removal of colorants from sugar liquor and for treatment of drinking water and industrial wastewater. The food industry is also a major consumer of activated carbon, where it is used to remove compounds that adversely affect color, taste and odor. In the mineral industry activated carbon are used to recover gold from leached liquors. Medicinal uses and pharmaceutical industry is also another wide area for the utilization of activated carbon. In gas cleaning applications activated carbon are extensively used in air filters at industrial level as well as in general air conditioning application. Activated carbon is a good decolorizer.

After filtering any remaining solids, the clarified syrup is decolorized by filtration through activated carbon. Numerous types of activated carbon are available in the market place nowadays according to the precursor carbonaceous material (coal, wood, coconut, etc.) and their size. The most common types used for sugar juice decolorization being powdered activated carbon (usually termed as PAC) and granular activated carbon (GAC). Bone char or coal-based activated carbon, a pyrolyzed ground animal bones have a high surface area which adsorbs color and remove some ash is traditionally used in this role. Activated charcoal is added to the syrup, removing color-forming impurities and inorganic ash. Pretreatment with activated carbon improved color but impaired palatability via adsorption of flavor compounds. The relatively pure honey coloured liquor of sugarcane juice from the filtration stage, "raw liquor", is then subjected to final decolourization by contact with bone charcoal. The bone char consists of active carbon on a calcium phosphate skeleton. It has a high surface area and the unique ability to absorb colour and inorganic ash impurities from the sugar. Following

the decolourization cycle the bone char is revived first by water washing, to remove inorganic impurities, and then heating in the absence of air to 650°C to volatilise organic impurities. The decolourized 'fine liquor' is now ready for the final refining and recovery step, which is achieved by crystallization in vacuum pans (Armishaw, n.d).

## ***2.8 Evaporation of OPF juice***

The treated liquor, now called "fine liquor" is ready for crystallization. However, it must first be concentrated by having its excess water content removed. The purified syrup is then concentrated to supersaturation. The boiling operation is required to evaporate water and reduce the juice to a concentrated form usually called massecuit. For dewatering of OPF juice, different temperature for evaporation were used which are in the range of 60°C to 100°C and vacuum pressure between 0 mbar to 50 mbar. Concentrations of OPF juice for storage were in the range of 20% to 80% and for storage at temperature range -20°C to 30°C (Ariffin et al., 2013). In sugarcane industries, a falling film evaporator using as its heat source vapour supplied by a mechanical vapour re-compressor (MVR) or double effect plate evaporator (DEPE) was used to evaporate the treated sugarcane juice before undergoing the crystallization process.

In gur production, the most ancient sweetening agent in India mainly used in cooking, single pan furnace is generally used to boil the juice briskly at high temperature with the object of evaporating large quantity of water in it. During this period the temperature of boiling juice gradually rises from 100 to 105 °C. When the temperature is about 105°C the juice start frothing and from this time onwards the fire is to be regulated to prevent caramelization of the sugar. At 108 °C temperature stage, this syrup has to be constantly stirred to prevent charring and spilling over the sides of the pan. At this stage castor/ Mustard/ Groundnut oil 10 to 15 ml is added to every pan to keep down the froth. This prevents frothing to a certain extent, and also facilitates easy flow of gur from the pan to the cooling trough. The striking point corresponds to temperature range from 116 °C to 120 °C. The optimum temperature is found to be 118 °C once the striking point is achieved the pan is removed from the fire (or stop firing) and the content is transferred to a wooden , earthen or cemented trough which is called cooling trough for crystallization (Baboo and Soloman, 1995; Hunsigi, 2012).