



**UNIVERSITI PUTRA MALAYSIA**

***CLARIFICATION OF GUAVA JUICE USING MEMBRANE-BASED  
PROCESS***

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## ABSTRACT

Guava (*Psidium guajava* L.) is very popular in many tropical and subtropical countries. Due to its high nutritional values, guava has been processed and preserved into puree, canned slices in syrup and has high potential to be promoted as a healthy fruit juices. However, similarly to other typical fruit juices, guava juice contains high concentration of pectin and other carbohydrates such as cellulose, hemicellulose and etc that resulting the juice to be high in turbidity and viscosity. Membrane-based process especially ultrafiltration (UF) has a big potential in the guava juice processing since it is absence in phase transition, mild operating condition and easy scaling up. Thus, this study was aimed to explore the potential of applying the membrane-based process using ultrafiltration for guava juice clarification by observing its effect on the permeate flux behaviour and guava juice quality attributes. It was found that the highest steady-state flux which was  $17.93 \text{ kg/m}^2/\text{hr}$  has been achieved at operating pressure (OP) of 2.0 bar. Besides, 80% of the initial juice was recovered in permeate as clarified juice. In this study, a limiting flux was observed at OP of 2.0 bar where there will be no significant increase on permeate flux as further increases of pressure was applied. The decline in permeate flux was analysed using different membrane pore blockings. Intermediate pore blocking was identified as the best suited model. Moreover, the preservation of the physiochemical, nutritional and functional properties of guava juice has been achieved through the clarification process by ultrafiltration. The UF process managed to remove the components that contribute to turbidity, reduction in TPC and TSS and also stable pH and Vitamin C that leads to enhance the colour properties of the guava juice which indicating the successfulness of the clarification process. As the potential advantage of ultrafiltration in the fruit

juice processing industry is undeniable, this can support the clarified guava juice commercialization value as it is minimally processed food.



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## **LIST OF ABBREVIATIONS**

<b>AAC</b>	<b>ascorbic acid content</b>
<b>MF</b>	<b>microfiltration</b>
<b>UF</b>	<b>ultrafiltration</b>
<b>NF</b>	<b>nanofiltration</b>
<b>RO</b>	<b>reverse osmosis</b>
<b>MWCO</b>	<b>molecular weight cut off</b>
<b>OP</b>	<b>operating pressure</b>
<b>TSS</b>	<b>total soluble solid</b>
<b>TPC</b>	<b>total phenolic content</b>
<b>PES</b>	<b>polyethersulfone</b>

## CHAPTER 1

### INTRODUCTION

#### 1.1 Backgrounds

Guava (*Psidium guajava* L.) is very popular in many tropical and subtropical countries. It has been cultivated and distributed by man and bird where the place of origin is uncertain but it is believed to be an area extending from southern Mexico into or through Central America (Morton, 1987). Guava fruit usually consumed as fresh where it contains high vitamin C and lycopene compared to fresh orange juice (Sciences, Akesowan, & Choonhahirun, 2013).

Besides, it is rich in vitamin A, omega-3 and -6 polyunsaturated fatty acids, dietary fibre, potassium, magnesium and antioxidant pigments such as carotenoid and polyphenols (Sciences et al., 2013). Guava also being processed and preserved into puree, canned slices in syrup and juice. Due to its high nutritional values, the consumption of guava juice has been increased and also used as an alternative to other beverage such as coffee, tea and carbonated drinks.

However, fruit juices are naturally cloudy, high viscosity and turbidity due to the presence of polysaccharides (pectin, cellulose, hemicelluloses, lignin and starch), proteins and tannins (Sciences et al., 2013). This is because the conventional fruit juice processing was made by mechanical pressing of guava mash where the obtained juice is cloudy and low in ascorbic acid due to a high content of ascorbic acids remains in the pomace (Kuar *et al.*, 2009).

Generally, the traditional method of clarification of juice which contains pectin involves a number of steps, including centrifugation to remove suspended solid, hydrolysis of pectin with specific enzymes (depectinization), flocculation of turbidity with clarifying agents, and finally filtration by the diatomaceous earth to remove the fining agents (P. Rai, 2015). Besides, the uses of enzymes such as pectinase (Kime, 1985; Sciences et al., 2013), defecation, sulfitation, single carbonation and modified double carbonation (Saska, Zossi, & Liu, 2010) and uses of fining agents such as bentonite gelatin and silica sol (Uçan, Akyildiz, & Erdal, 2014) also had been used in the juice clarification. Moreover, traditional methods of processing fruit juices are usually based on the use of high temperatures in order to destroy spoilage bacteria, inactivate enzymes and produce concentrated juices (A Cassano, Marchio, & Drioli, 2007). Although, conventional thermal processing can ensures the safety and extends the shelf life of fruit juices but it leads to detrimental change in the sensoria and nutritional qualities of the product (A Cassano, Donato, & Drioli, 2007).

Compared with traditional juice processing methods, membrane processes are low-cost and a thermal separation techniques which involve no phase change or chemical agents. These features are becoming very important factors in the production of new fruit juices with natural fresh taste and additive-free (A Cassano, 2004). For example, UF membranes have been shown to be of potential interest for clarification

of fruit juices and have become a commercial success. They are able to retain large species such as microorganisms, lipids, proteins and colloids while small solutes as for example vitamins, salts, sugars flow together with water (A Cassano, Marchio, et al., 2007).

Membrane-based process is a method which selectively separate materials via pores and/or minute gaps in the molecular arrangement of continuous structure. Membrane separations are classified by pore size and by the separation driving force which can be classified as microfiltration (MF), ultrafiltration (UF), reverse osmosis (RO) and nanofiltration (NF) (Membrane separation technology primer, n.d.)

Membrane-based process especially ultrafiltration has a big potential in the guava juice clarification as there are a lot of case study has been done using this membrane separation. Membrane process has been applied successfully to clarify blood orange juice(A Cassano, Marchio, et al., 2007), Xoconostle jucie (Castro-mu, Fíla, & Barrag, 2017), melon juice (Fabrice Vaillant et al., 2005), apple juice (Bruijna, Venegasb, & Borquezc, 2002; Girard & Fukumoto, 1999; Onsekizoglu, Bahceci, & Acar, 2010), passion fruit juices (Oliveira, Docê, Teresa, & Barros, 2012), pineapple juices (Maria, Carvalho, & Alberto, 2010), bergamot juices (Carmela, Leonardo, Donna, & Cassano, 2018), watermelon juices (C. Rai, Rai, Majumdar, & Dasgupta, 2010) and etc.

## **1.2 Problem Statements**

A typical guava juice can be seen as highly in turbidity and viscosity that contains high concentration of pectin and other carbohydrates such as cellulose, hemicellulose and etc. This will subsequently lead to low consumption of guava juice as the consumers nowadays prefer a pleasant flavour and colour of fruit juices. Thus,

clarification of guava juices was importance in order to increase its commercial value as a high content of Vitamin C juices. A conventional method used for clarification of juices such as using fining agents, addition of enzymes, centrifugation and heating process was still lack in a few aspects although the process can ensure a clarify juice can be obtained. Traditional technology cannot remove microorganisms and bacteria by using centrifugation filter only thus its limits the shelf life of the juices. Besides, although heating process can ensured to remove all the microorganisms and bacteria however, it will degrade the nutritional contents in the juices and traditional technology also cost a lot for its complex process. Membrane-based technology has been widely used to solve all the problems above as it does not involve any heating process and in the same time can retained all the nutritional values and prolong the shelf life of juices. Thus, studies conducted was to address these problems associated with guava juice were summarized in this paper.

### **1.3 Objectives**

The objectives of this study are:

1. To explored the potential of applying the membrane-based process using ultrafiltration for the production of guava juice at different trans-membrane pressure (TMP)
2. To determine the effect of membrane processing conditions on the permeate flux behaviour and guava juice quality attributes.

### **1.4 Scope of Study**

The main focus on this study is on the white seeded guava fruit which covers the clarification of the juice by membrane-based process. The effect of membrane

processing conditions on the permeate flux behaviour and guava juice quality attributes are also being studied. Therefore, in this study, we examine how membrane pore size and trans-membrane pressure affect the flux performance and quality attributes on the guava juice.

### **1.5 Thesis structure**

The flow of this thesis structure consist of five chapters. Chapter 1 is about the introduction of guava fruit and its application, the problems associated with commercial guava juice processing using traditional method and the objective of this study. Chapter 2 is an organized sets of literature review which includes the overview of guava fruit, technique for juice clarification, membraned-based process, membraned-based application and membrane fouling. Chapter 3 is about the methods and materials used in the clarification of guava juice. Chapter 4 is the results and discussions of the experiment which contain figures, graphs and tables. Lastly, conclusion and recommendation of this study was stated in Chapter 5.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Overview of guava**

##### **2.1.1 World guava production**

Guava, *Psidium guajava* L., is one of the myrtle family (Myrtaceae) which is one of the most gregarious fruit tree which almost universally known by its common English name or its equivalent in other languages (Morton & Morton, 2004).

Guava (*Psidium guajava* L.) is a tropical fruit that usually consumed as fresh (Lee & Yusof, 2006). The guava tree can be easily recognize due to its smooth, thin and copper-coloured bark that flake off, showing the greenish layer beneath. The colour of the inner flesh can be classified into white, deep pink and salmon red (Castro-mu et al., 2017).

Guava (*Psidium guajava*) was one of the most popular fruits of India as it was a New World introduction that arrived in India from the Philippines as a result of the

Spanish conquest and from Portuguese incursions in India. It is known to be the best in the world for some of the cultivars developed in India. Guava is cultivated throughout the country and occupies an area of 155,000 ha with a total production of 1.8 million t (Issue & Highlights, 2007).

In addition, guava (*Psidium guajava*) fruit plant also grown abundantly throughout western Nigeria (Amusa, Ashaye, Oladapo, & Oni, 2005). Also, guava is one of the leading fruits of Mexico (Morton & Morton, 2004).

Guava fruit has two main cellular types which are parenchyma and stone cells. Stone cells are woody materials that are strongly lignified and resistant to degradation by pectinases. The mesocarp portion of the guava holds 90% of the total cell wall material of guava pulp where 77% are stone cells. The dominant structural feature in parenchyma cell walls is cellulose with side chains of glucans and xyloglucans and highly branched arabinans. Thus, a combination of enzymes such as pectinesterase, arabanase, hemicellulose, tannase and cellulose are used to improve the clarification of the guava juice (Kashyap, Vohra, Chopra, & Tewari, 2001).

### **2.1.2 Production of guava in Malaysia**

Guava (*Psidium guajava* L.), also known locally as jambu batu, is grown commercially and in many home gardens in Malaysia. The tree is very hardy and can grow to about 7-8 metres high with characteristic smooth, pale mottled bark that peels off in thin flakes. The fruits vary in size, shape and flavour depending on the variety. The better varieties are sweet while others may be astringent (Jhi, 2006).

There was two variety of guava which were seedless guava and with seed guava variety. However, the seedless variety was not many recommended for commercial growing due to its unstable characteristic and guava with seed was more

popular. The total area of guava in Malaysia was 1,440 hectare with an annual production of 18,880 mt in 2011 while area grown with guava was in Daerah Muar in Johor (213 ha), Daerah Batang Padang in Perak (185 ha) and Daerah Segamat in Johor (68 ha) (Anem, 2010).



Figure 1: Seed guava (left) and seedless guava (right)

### 2.1.3 Nutritional values of guava

The Table 1 shows that a local fruit such as guava has a high quantity of antioxidant such as phenols and ascorbic acid. The seeded guava contained higher ascorbic acid when compared to seedless guava. Guava also contains high primary

antioxidant potential when compared to other local fruits and an imported fruit such as orange. (Jhi, 2006).

According to Wern, K.H., Haron, H., Keng (2016), consumption of fresh juices or commercial 100% fruit juices both good since there was no significant difference between TPC and antioxidant activities.

Table 1: Comparison of antioxidant content and activities of extracts from tropical fruits

Fruits	TPC (mg GAE/100g)	AAC (mg/100g)	IC <sub>50</sub> (mg/mL)	AEAC (mg AA/100g)	FRAP (mg GAE/g)
Guava (seeded) <sup>8</sup>	138 ± 31a	144 ± 60	1.71 ± 0.61f	218 ± 79	2.09 ± 0.18
Guava(seedless) <sup>6</sup>	179 ± 44a	132 ± 46	2.11±0.63f,g	176 ± 54	1.65 ± 0.06
Banana (mas) <sup>6</sup>	51 ± 7c	4.9 ± 0.6	13.4± 2.5i	27.8 ± 5.5	0.18 ± 0.06
Dragon fruit <sup>3</sup>	21 ± 6d	8.0 ± 1.6	27.5 ± 3.9j	13.5 ± 2.1	0.07 ± 0.01
Star fruit <sup>7</sup>	131 ± 54a	5.2 ± 1.9	3.8 ± 2.1g,h	98 ± 55	0.83 ± 0.02
Sugar apple(brown) <sup>3</sup>	175 ± 36a	21.3 ± 2.1	3.9 ± 0.4g,h	82.1 ± 6.9	0.62 ± 0.10
Sugar apple(green) <sup>3</sup>	165 ± 18a	6.8 ± 0.8	4.6 ± 0.8h	71.4 ± 11.8	0.58 ± 0.04
Water apple <sup>4</sup>	35 ± 4e	4.1 ± 2.1	12.0 ± 3.8i	31 ± 10	0.30 ± 0.02
Orange <sup>5</sup>	75 ± 10b	67 ± 9	5.4 ± 1.3h	70 ± 17	0.61 ± 0.05

The superscript numerals in the fruits column are the number of samples studied.

Values having the same superscript within the same column (TPC, IC<sub>50</sub>) are not significantly different (p=0.05).

Where TPC-total phenolic content; AAC- ascorbic acid content; IC<sub>50</sub>, AEAC and FRAP are primary antioxidant properties.

Guava that are rich in antioxidants help to reduce the incidence of degenerative diseases such as arthritis, arteriosclerosis, cancer, heart disease, inflammation and brain dysfunction as in many other fruits and vegetables (Jhi, 2006).

Ascorbic acid which mainly in the skin, secondly in the firm flesh, and little in the central pulp-varies from 56 to 600 mg. It may range up to 350-450 mg in nearly ripe fruit. When specimens of the same lot of fruits are fully ripe and soft, it may

decline to 50-100 mg. However, as expected, canning or other heat processing destroys about 50% of the ascorbic acid (Morton & Morton, 2004).

Table 2: Nutrient composition in guava fruit (Morton & Morton, 2004)

<b>Food Value Per 100 g of Edible Portion*</b>	
Calories	36-50
Moisture	77-86 g
Crude Fiber	2.8-5.5 g
Protein	0.9-1.0 g
Fat	0.1-0.5 g
Ash	0.43-0.7 g
Carbohydrates	9.5-10 g
Calcium	9.1-17 mg
Phosphorus	17.8-30 mg
Iron	0.30-0.70 mg
Carotene (Vitamin A)	200-400 I.U.
Thiamine	0.046 mg
Riboflavin	0.03-0.04 mg
Niacin	0.6-1.068 mg
Vitamin B3	40 I.U.
Vitamin G4	35 I.U.

The approximate composition of the guava fruit was shown in Table 2. Although guavas are rich source of ascorbic acid, the stem and outer flesh contain most of the ascorbic acid. Its content achieved a maximum in green fully mature fruit and starts to decline rapidly as they ripen. However, colour of the flesh does not associated with ascorbic acid level (Menzel & Branch, 2017).

#### **2.1.4 Guava for processed food application**

Raw guavas are eaten out-of-hand, but was preferred seeded and served sliced as dessert or in salads (Morton & Morton, 2004). Besides, guava fruits has been process and preserve into several products such as puree, jam, syrup, paste and etc. In

order to obtain high yield of guava products during processing, the guava fruit should be ensure fully ripened and possess a thick outer flesh and small seed cavity (Charley, 1950).

A standard dessert throughout Latin America and the Spanish-speaking islands of the West Indies is stewed guava shells (*cascos de guayaba*) which the guava halves with the central seed pulp removed, strained and added to the shells while cooking to enrich the syrup. The canned product is widely sold and the shells can also be quick-frozen. They are often served with cream cheese. In addition, sometimes guavas are canned whole or cut in half without seed removal, while bars of thick, rich guava paste and guava cheese are staple sweets, and guava jelly is almost universally marketed (Morton & Morton, 2004).

Guava juice is now becoming popular due to its nutritional values (Issue & Highlights, 2007). A clear guava juice with all the ascorbic acid and other properties is made into syrup for use on waffles, ice cream, puddings and in milkshakes. Moreover, guava juice and nectar are among the numerous popular canned or bottled fruit beverages of the Caribbean area. There are a lot of recipes for utilizing guavas in pies, cakes, puddings, sauce, ice cream, jam, butter, marmalade, chutney, relish, catsup, and other products (Morton & Morton, 2004).

According to Morton & Morton (2004), in South Africa, a baby-food manufacturer markets a guava-tapioca product, and a guava extract prepared from small and overripe fruits is used as an ascorbic-acid enrichment for soft drinks and various foods. Dehydrated guavas may be reduced to a powder which can be used to flavour ice cream, confections and fruit juices, or boiled with sugar to make jelly, or utilized as pectin to make jelly of low-pectin fruits.

### 2.1.5 Guava juice production

There Based on the Figure 2, it showed the process flow of general juice production (Juices et al., n.d.). Firstly, for the preparation of raw material, the fruits were selected based on its matured and undamaged fruits. If there were mouldy or under-ripe fruits, it would be sorted and removed. Then, it will be cleaned using a water, peeled the fruits and removed stones or seeds. If necessary, the fruits will be chopped into pieces to fit into the liquidiser or pulper.

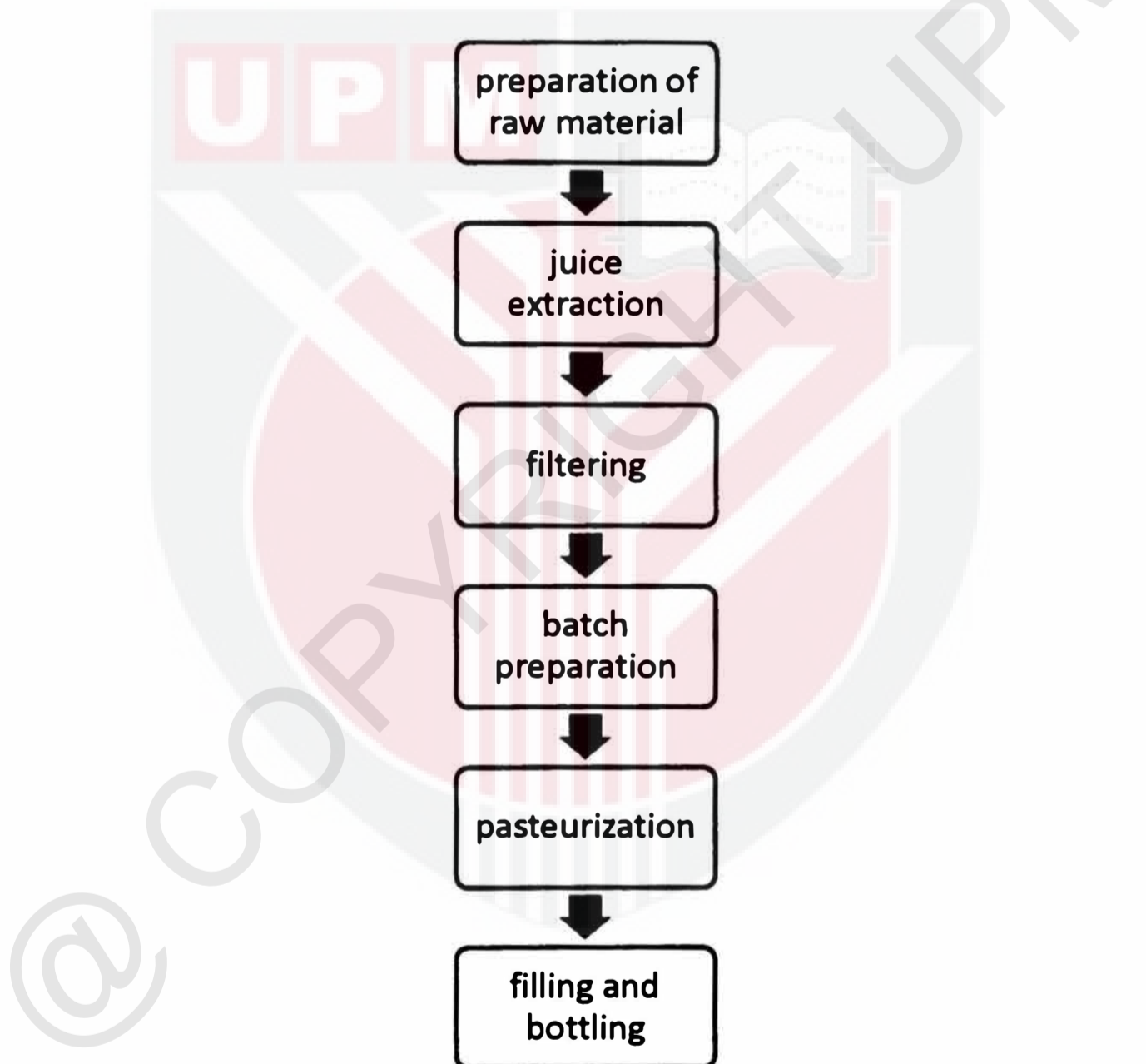


Figure 2: Process flow of juice processing

Next, the extraction method involves several methods depending on the type of fruit used. For citrus fruits which are naturally juicy, the best option is to use a hand

presser while some fruits such as melon and papaya were steamed to release the juice. Moreover, apples are pressed and fruits such as mango, guava, soursop, pineapple, strawberry must be pulped to extract the juice. The fruit pieces are pushed through a perforated metal plate that crushes and turns them into a pulp. Some fruits can be pulped in a liquidiser and then filtered to remove the fruit pieces. After that, the juice need to be filtered using muslin cloth or stainless steel filter in order to make a clear juice. Some of the larger filter presses have a filter included. Although juice is naturally cloudy, some consumers prefer a clear product. Thus, pectic enzymes may be necessary to help clear the juice by break down the pectin. However, it may be difficult to find and expensive and therefore should only be used if really necessary and readily available.

Then, when the juice or pulp has been collected, it is necessary to prepare the batch according to the chosen recipe either to make a fruit squashes, fruit nectar and etc. Next, all the products need to be pasteurized at 80-95 °C 1-10 minutes. This method of pasteurisation has its own advantage but also has problems. The use of high temperatures in juice processing was to destroy spoilage bacteria, inactivate enzymes and produce concentrated juices (A Cassano, Marchio, et al., 2007). However, it leads to detrimental change in the sensorial and nutritional qualities of the product (A Cassano, Donato, et al., 2007). Lastly, the products will be hot-filled into a clean and sterilised bottles. After filling hot, the bottles are capped and laid on their sides to cool prior to labelling.

According to (Viquez & Coo, 1981) a clear juice is usually more acceptable. Juices that have an unstable cloud or whose turbidity is considered 'muddy' or undesirable tend to be marketed as clear juices. This is because the general juice

processing had obtained juice that was cloudy and low in ascorbic acid due to a high content of ascorbic acids remains in the pomace (Kaur, Sarkar, & Sharma, 2009). Also, during early 1930s, when fruit processing industries began to produce juices, the yields were low, and many difficulties were encountered in filtering the juice to an acceptable clarity (Uhlig, 1998).

## **2.2 Techniques for juice clarification**

Conventional method for guava juice extractions involved boiling the fruit pulp with water and extracting (Sreekantiah, 1968). However, this method can affect the natural flavour of the guava and its nutritional values. Fruit juices was pasteurized and concentrated using thermal treatment was also one of the traditional method for juice preparation but it is also affect its quality attributes (Castro-mu et al., 2017).

Moreover, mechanical pressing of guava mash which is one of the conventional method for juice processing also results in cloudy and low in ascorbic acid due to its nutritional values remains in the pomace (Sciences et al., 2013). Thus, clarification steps was crucial in order to produce a 'clear' juices and in the same time preserve all the nutritional values.

Some common clarification process involves several steps which are centrifugation in order to eliminate suspended solids, hydrolysis of pectin with specific enzyme (depectinization), flocculation of turbidity with clarifying agents and filtration by diatomaceous earth to remove fining agents ( Rai & De, 2009).

Nowadays, the uses of enzymes treatment in juice extraction has been commonly used. It offers to increase juice recovery, TSS, clarity, decrease turbidity and viscosity by combination of enzymes (Sharma, Patel, & Sharma, 2014). Guava juice production using pectinase, apple juice clarification using honey and pectinase

(Kime, 1985), clarified lemon juice (Uçan et al., 2014) and etc were examples of using enzyme treatment in fruit juices of processing.

The combination effects of carbonation and sonication process have been evaluated on guava juice. Its results in enhanced the manifestation of cavitation and subsequently produced juice with higher ascorbic acid content, lower clarity and higher polyphenoloxidase polyphenoloxidase activity. However, these treatments do not have strong lethal effects on microorganisms (Cheng, Soh, Liew, & Teh, 2007).

In contrary, juice clarification using sulfitation had achieved comparable decolourisation and mud settling characteristics when compared to carbonation process. However, lower thermal stability of clarified juice and syrup from carbonation required more study (Saska et al., 2010).

As consumers are becoming more health conscious, demands for natural and fresh-like foods have increased interest in using non-thermal technologies in food processing to produce foods with minimal damage to nutritional and sensory properties. Several technologies are being explored as a potential alternative to thermal processing, among which are membrane filtration, osmotic dehydration, pulse electric field, ultrasound, irradiation and high pressure. (Cheng et al., 2007).

Membrane filtration such as ultrafiltration process were used in clarification of lemon, orange and carrot juices able to achieve a good level clarification and avoiding use of gelatines, adsorbents and other filtration coadiuvant (P. Rai, 2015). In addition, ultrafiltration and microfiltration had preserved the physiochemical, nutritional and functional properties through clarification using membrane filtration in clarification of Mexican Xoconostle fruit juice (Castro-mu et al., 2017), melon juice (Fabrice Vaillant et al., 2005), passion fruit juice (Oliveira et al., 2012), blood orange juice (A Cassano,

Marchio, et al., 2007), kiwi fruit juice (A Cassano, Donato, et al., 2007), mandarin juices (Alfredo Cassano, Tasselli, Conidi, & Drioli, 2009), pineapple juice (Maria et al., 2010) and etc. However, permeates from UF membranes were reported by panellists as “light” and “sparkling clear” of apple juice while in MF membrane, the apple juice permeates were “dark” (Wu, Zall, & Tzeng, 1990).

### **2.3 Membrane-based process**

Membrane processes gives a lot of advantages when compared to the traditional juice processing operations. This membrane process technology that has been widely used in dairy, food and beverage industries are microfiltration (MF) and ultrafiltration (UF) (Castro-mu et al., 2017).

Most of the researchers had found out that the capability of this technology is better than the conventional separation method (A Cassano, Marchio, et al., 2007; Girard & Fukumoto, 1999; Laorko, Li, Tongchitpakdee, Chantachum, & Youravong, 2010; Maria et al., 2010; Nor & Vasiljevic, 2017; Fabrice Vaillant et al., 2005). The comparison between different type of membrane separation which includes microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) was tabulated in the Table 3.

Advantages of the UF over conventional fruit juice processing are in terms of increased juice yield, possibility of operating in a single step reducing working times, possibility of avoiding the use of gelatines, adsorbents and other filtration aids, reduction in enzyme utilization; easy cleaning and maintenance of the equipment; reduction of waste products and elimination of needs for pasteurization (A Cassano, Marchio, et al., 2007).

Table 3: Comparison between the different pressure driven methods

Membrane	Reverse osmosis (RO)	Nanofiltration (NF)	Ultrafiltration (UF)	Microfiltration (MF)
	Asymmetric	Asymmetric	Asymmetric	Asymmetric symmetric
Thin film thickness	1 micron 150 micron	1 micron 150 micron	1 micron 150–250 micron	1–150 micron
Rejection	High and low molecular weight compounds, NaCl, glucose, amino acids	High molecular weight compounds, mono-, di- and oligosaccharides, polyvalent ions	Macromolecules, proteins, polysaccharides, vira	Particles, clay, bacteria
Membrane materials <sup>1</sup>	Cellulose acetate (CA) thin film	CA, thin film	Ceramic, polysulfonic (PS), poly vinylidene fluoride (PVDF) CA, thin film	Ceramic, PS, PVDF, CA
Pore size	<0.002 micron	<0.002 micron	0.02–0.2 micron	0.02–4 microns
Module configuration <sup>2</sup>	Tubular, spiral wound, plate-and-frame	Tubular spiral wound, plate-and-frame	Tubular hollow fiber spiral wound, plate-and-frame	Tubular, hollow fiber
Operating pressure	15–160 bar	6–35 bar	1–10 bar	<2 bar

Usually, membrane separation such as ultrafiltration, microfiltration and nanofiltration was applied in clarification and extraction of fruits and vegetables juice while reverse osmosis was proposed to concentrate juice extracts (Yilmaz, 2018).

Membrane separation technology has been widely used in industries such as dairy, food and beverage. This membrane process is highly beneficial in terms of its mild operating condition, no phase transition, separation efficiency, no addition of aqueous phase and easy scaling up compared to conventional methods (Castro-mu et al., 2017).

## 2.4 Membrane-based applications

Membrane-based technology especially ultrafiltration (UF) has been widely used in many industrial applications. Clarification of fruit juices is one of the example in beverage industry application where the fruits undergoes membraned-based technology such as Ultrafiltration (UF) and Microfiltration (MF) and in the same time preserved its quality and nutritional values.

There are a lot of successful research has been done on different fruit juices by using membrane process which tabulated in Table 4.

Table 4: Summary of successful study using membrane-based process

Type of membrane	Membrane material	Type of samples	Size of the filter	Findings
UF	Polysulfone	Xoconostle fruit juice (Castro-mu et al., 2017)	MWCO of 100kDa	It can preserved the physicochemical, nutritional and functional properties of the Xoconostle juice with high juice recovery
MF	Cellulose acetate	Watermelon juice (C. Rai et al., 2010)	0.2 $\mu\text{m}$	Clear permeate juice retained almost the total amount of soluble solids, ascorbic acid, and malic acid present in the feed. Antioxidant lycopene can be a by-product recovered from the retentate stream
UF	polyvinylidene fluoride (PVDF)	Blood orange juice (A Cassano, Marchio, et al., 2007)	NMWCO of 15kDa	Has comparable physicochemical and nutritional properties with fresh juice except for insoluble solids that remained in the retentate stream

MF	Tubular ceramic and hollow fiber poly(imide)	Passion fruit juice (Oliveira et al., 2012)	0.3 $\mu\text{m}$	Physicochemical and nutritional properties behaved as expected and best performance for ceramic tubular membrane at 0.5 bar TMP while for hollow fiber at 1.0 bar TMP
MF	Tubular polyethersulfone	Pineapple juice (Maria et al., 2010)	0.3 $\mu\text{m}$	Clarification of pineapple juice was effective when 1.5 bar TMP was applied and luminosity of permeate juice increase and turbidity was almost completely removed
MF	Ceramic multichannel membrane	Melon juice (Fabrice Vaillant et al., 2005)	0.2 $\mu\text{m}$	Physicochemical and nutritional properties that are comparable with fresh melon juice, except for the absence of suspended solids and carotenoids,

				<p>which remained totally concentrated in the retentate</p>
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## 2.5 Membrane fouling

The major components in the fruit juices that contributes to fouling were pectin, sugars, citric acid, cellulose and hemicellulose (A Cassano, Marchio, et al., 2007; Huang & Yu, 1987).

In addition, accumulation of macromolecular or colloidal species such as proteins, tannins and fiber also a key that responsible on the fouling membrane surface that lead to flux decline (Calabria, 1996).

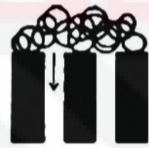
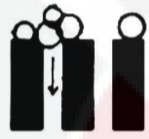


According to Huang & Yu (1987), fouling of the ultrafiltration in passion fruit juices was caused by the formation of a gel-like. Thus, fouling layers that formed by a combination of suspended particles and adsorbed macromolecules impurities will caused a flux decline (A Cassano, Marchio, et al., 2007). The result can be supported by Riedl, Girard, & Lencki (1998a), where they found that colloidal materials that aggregates on the membrane surface was strongly give impact on the flux resistance of the fouling layer.

On the other hand, a study has found that membrane morphology also had gave impact on the surface structure and flux resistance of the fouling layer (Riedl, Girard, & Lencki, 1998b). They concluded that “rough membranes produce a looser surface fouling layer that has a lower flow resistance per unit thickness of foulant than the dense fouling layer observed on smooth surfaces membrane”.

Barros, Andrade, Mendes, & Peres (2003) stated that designing systems for process engineer may be useful to classify fouling as in-depth pore fouling, pore plugging and cake formation.

There are four empirical models to present membrane fouling mechanisms in dead-end filtration based on constant pressure which are complete pore blocking, standard pore blocking, intermediate pore blocking and cake filtration which tabulated in Table 5 (Nor & Vasiljevic, 2017).

Table 5: Illustration of different fouling models

Fouling mechanism	Diagram	n	General description	Final equation	Equation number
Cake formation		0	Particles form a gel layer over the membrane surface	$J = J' + (J_0 - J')e^{-k_f t}$	(4)
Intermediate pore blocking		1	Particles settle on other that have already deposited on the membrane surface, which were already blocking some pores and/or directly blocks some membrane area	$J = \frac{J' J_0 e^{-k_f t}}{J' + J_0 (e^{-k_f t} - 1)}$	(5)
Internal pore blocking		1.5	Particles enter the membrane pores and deposited over the pore walls, reducing the membrane pore volume	$\frac{1}{Jt} = \frac{1}{J_0 t} + k_f t$	(6)
Complete pore blocking		2	Particles blocking the membrane surface by means of pore sealing	$t = \frac{1}{k_f J'} \ln \left[ \left( \frac{J_0}{J} - \frac{J_0}{J'} \right) - J' \left( \frac{1}{J} - \frac{1}{J_0} \right) \right]$	(7)

Taylor, Giorno, & Donato (2008) found that UF process was controlled by a “cake filtration” fouling mechanism. This can be further explained where the convective flux that moves from bulk solution toward the membrane, dominate on the rate of shear-induced back-diffusion of the rejected material that lead to the formation of a cake on the membrane surface.

## CHAPTER 3

### EXPERIMENTAL DESIGN & METHODOLOGY

The overall of this study is shown in Figure 3.

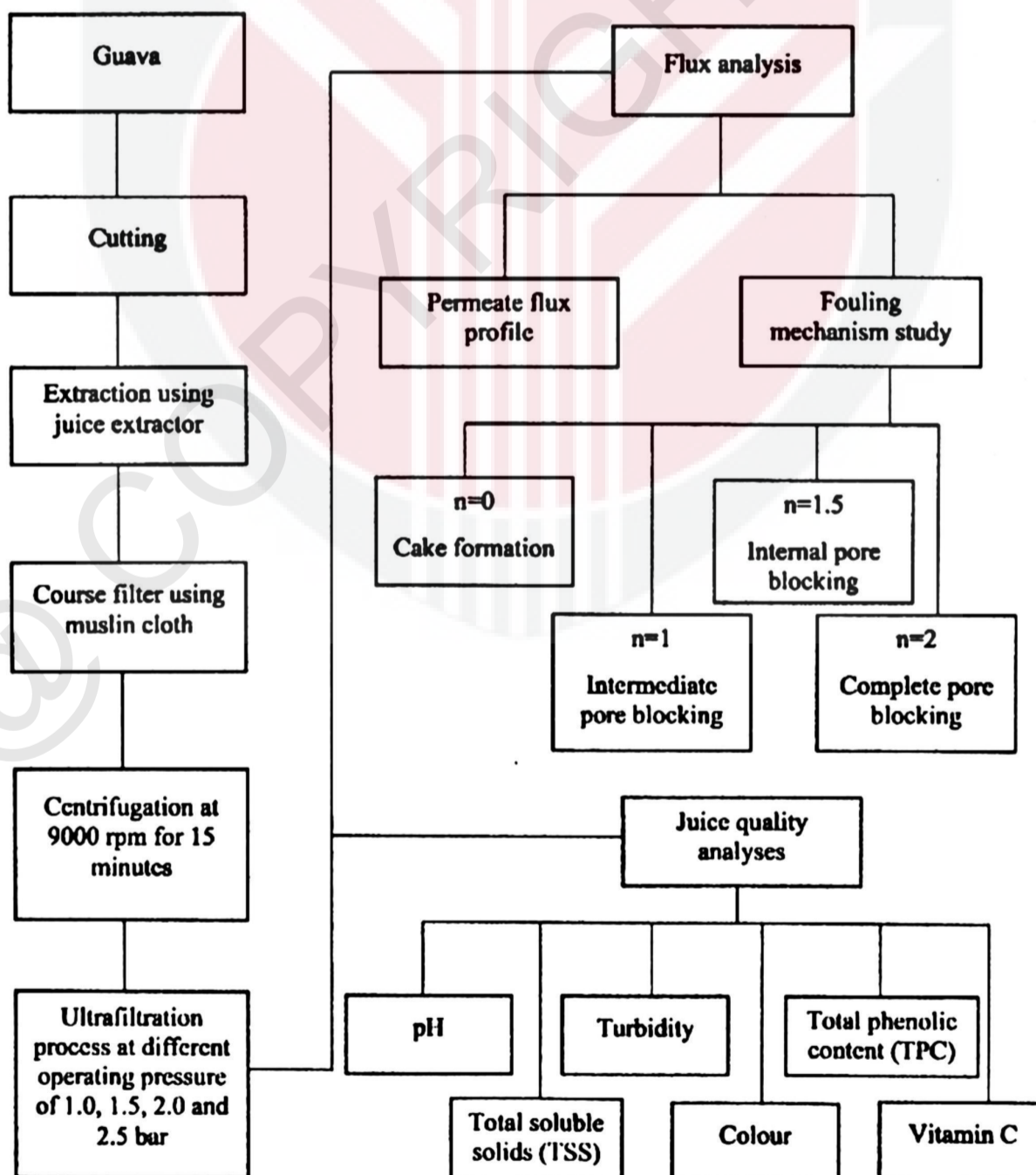


Figure 3: Overall process flow of clarified guava juice

### **3.1 Raw materials**

Ripe white seeded guava fruits with 80-90% maturity and free from visual blemishes and bruises were purchased from a local market at Serdang, Malaysia.

### **3.2 Guava juice preparation**

Ripe guavas were washed with tap water, peeled off the skin and cut into small pieces. The guavas were processed using juice extractor for a few seconds before being filtered with a nylon cloth. The extract was then centrifuged at 9000 rpm for 15 minutes. The obtained supernatant, known as the raw guava juice, was stored at -18 °C. For any subsequent process or analysis, the frozen juice was thawed with running water and re-centrifuged at 9000 rpm for 15 minutes to ensure the materials in suspension was removed as it was important for reducing clogging in membranes and results in best performance.

### **3.3 Membrane-based process**

This process was carried out in the laboratory scale of ultrafiltration unit. The system was equipped with a polyethersulfon dead end disc membrane with molecular weight cut-off (MWCO) of 100 kDa.

Figure 4 shows the schematic diagram for UF of guava juice. Each UF experiment was performed with capacity of 250 ml of raw guava juice as the feed. The process was performed at different operating pressure (OP) of 1.0, 1.5, 2.0 and 2.5 bar so that the limiting OP can be determined and able to give optimum permeate flux for the performance of juice clarification. The specification of the membrane used for guava juice clarification is shown in Table 6.

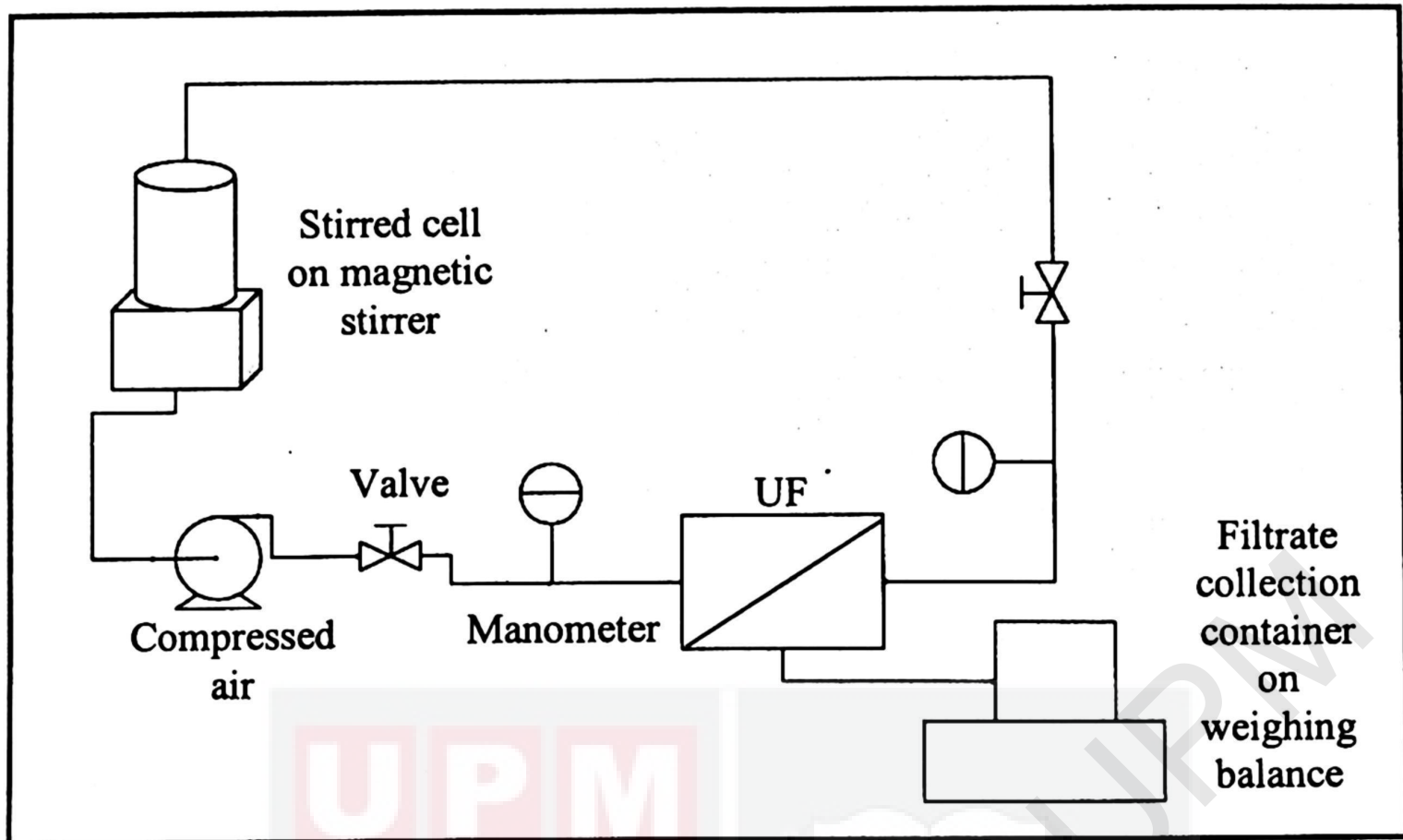


Figure 4: Schematic diagram for ultrafiltration of guava juice

Table 6: The specification of the membrane used for guava juice clarification

<b>Membrane</b>	Ultrafiltration (UF)
<b>Manufacturer</b>	EMD Millipore Corporation
<b>Membrane type</b>	PBHK06210
<b>MWCO (Da)</b>	100,000
<b>Filter diameter (mm)</b>	63.5
<b>Membrane material</b>	Polyethersulfon (PES)
<b>Configuration</b>	Dead end
<b>Operating pressure range (bar)</b>	1.0-2.5

Performance of the UF process was measured in terms of the permeate flux ( $J$ ) and permeate recovery (PR) as described in Eq. 1 and 2 below:

$$J = \frac{1}{A_m} \times \frac{\Delta W}{\Delta t} \quad (1)$$





$$PR(\%) = \frac{V_p}{V_f} \times 100 \quad (2)$$

where  $A_m$  is the effective membrane area ( $m^2$ ) and  $(\Delta W/\Delta t)$  is the permeate weight  $\Delta W$  collected over time  $\Delta t$  ( $kg \cdot h^{-1}$ ). While  $V_f$  and  $V_p$  are the volume (g) of the feed and permeate, respectively.

### 3.4 Analysis of the fouling mechanism

In order to understand the fouling mechanism during the process, four type of fouling was identified which were cake formation, intermediate pore blocking, internal pore blocking and complete pore blocking by using the equation from Table 5.

Table 7: Illustration of different fouling models

Fouling mechanism	Diagram	n	General description	Final equation	Equation number
Cake formation		0	Particles form a gel layer over the membrane surface	$J = J^* + (J_0 - J^*)e^{-k_f t}$	(4)
Intermediate pore blocking		1	Particles settle on other that have already deposited on the membrane surface, which were already blocking some pores and/or directly blocks some membrane area	$J = \frac{J_0 J^* e^{-k_f t}}{J^* + J_0 (e^{-k_f t} - 1)}$	(5)
Internal pore blocking		1.5	Particles enter the membrane pores and deposited over the pore walls, reducing the membrane pore volume	$\frac{1}{J} - \frac{1}{J^*} = k_f t$	(6)
Complete pore blocking		2	Particles blocking the membrane surface by means of pore sealing	$t = \frac{1}{k_f J^*} \ln \left[ \left( \frac{J_0}{J} \frac{J^* - J}{J^*} \right) - J^* \left( \frac{1}{J} - \frac{1}{J^*} \right) \right]$	(7)

The experimental data were fitted to each model using the Solver function in Microsoft Excel 2013 (USA) to identify the fouling mechanism involved during the ultrafiltration process. Four optimization runs were performed for each experimental flux profile by assigning  $n=0, 1, 1.5$  and  $2$  where the sum standard deviation (SSD)

between numerical predictions and experimental data for each run were compared for the lowest SSD to determine the main fouling mechanism.

### **3.5 Analyses of Juice Quality Attributes**

#### **3.5.1 pH**

pH was determined by using pH meter (PB-11, Sartorius, USA) which initially calibrated with buffer solution of pH 4 and 7.

#### **3.5.2 Turbidity**

Turbidity of the samples was determined using model Microprocessor Turbidity-meter (TN-100, Eutech Instrument, Singapore) and was reported as Nephelometric Turbidity Units (NTU). It was calibrated with patron solutions 0-1000 NTU.

#### **3.5.3 Total Soluble Solids (TSS)**

TSS was determined using Atago digital refractometer (PAL-3, Atago Co., Tokyo, Japan) in terms of Brix. The scale is ranging from 0-45 Brix.

#### **3.5.4 Color**

The colour measurement was determined with a Minolta Colorimeter (CS-2000 Konica Minolta Sensing Americas, Us). 30 ml sample was first placed in the petri dish and determined the colour of the CIELAB system. Data were expressed in terms of  $L^*$ ,  $a^*$  and  $b^*$  parameters.  $L^*$  represent the lightness index;  $a^*$  and  $-a^*$  redness and greenness, respectively;  $b^*$  and  $-b^*$  yellowness and blueness, respectively. Chroma was used to determine the degree of difference of a hue in comparison with a grey colour with the same lightness, and was considered the quantitative attribute of

colourfulness. The chroma (c) and hue (h) were calculated using the following Eq. 3 and 4:

$$h = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (3)$$

$$C = (a^{*2} + b^{*2})^{1/2} \quad (4)$$

### 3.5.5 Total Phenolic Content (TPC)

Total phenolic content was determined colorimetrically with Folin-Ciocalteu assay (Jhi, 2006) which depends on the reduction of Folin-Ciocalteu reagent by phenolic compounds under alkaline condition. 0.3 ml of guava juice sample was added into 1.5 ml of 10% Folin reagent (10 ml Folin reagent diluted with 90 ml of distilled water) and 1.2 ml of 7.5% sodium carbonate,  $\text{Na}_2\text{CO}_3$  (7.5 g of  $\text{Na}_2\text{CO}_3$  in 100 ml of solution). The absorbance of the solution was measured at 765 nm with Shimadzu UV-VIS spectrophotometer (Ultraspec 3100 Pro, Amersham Pharmacia Biotech, UK). The standard curve was established using gallic acid. The total phenolic content was expressed as gallic acid equivalent in mg/L.

### 3.5.6 Ascorbic acid

10 ml of guava juice sample was diluted with 10 ml of distilled water. The juice was titrated against the dichlorophenolindophenol (DCPIP) solution in the burette where 0.5 g of DCPIP was dissolved in 500 ml of distilled water. The endpoint of the titration was identified as the pink colour was observed which last at least for 30 seconds. Then, the mass of ascorbic acid (mg) in 100 ml juice was calculated. The reaction is

1:1 (based calculation). The mass of ascorbic acid were calculated using the following

Eq. 5 and 6:

Concentration of DCPIP (M) = Mass of DCPIP/Molecular weight (MW)of DCPIP/0.5

(5)

Mass ascorbic acid = MW DCPIP × Concentration of DCPIP (M) × VolumeDCPIP used × 1000

(6)



4.1 Evaluation of flux behavior

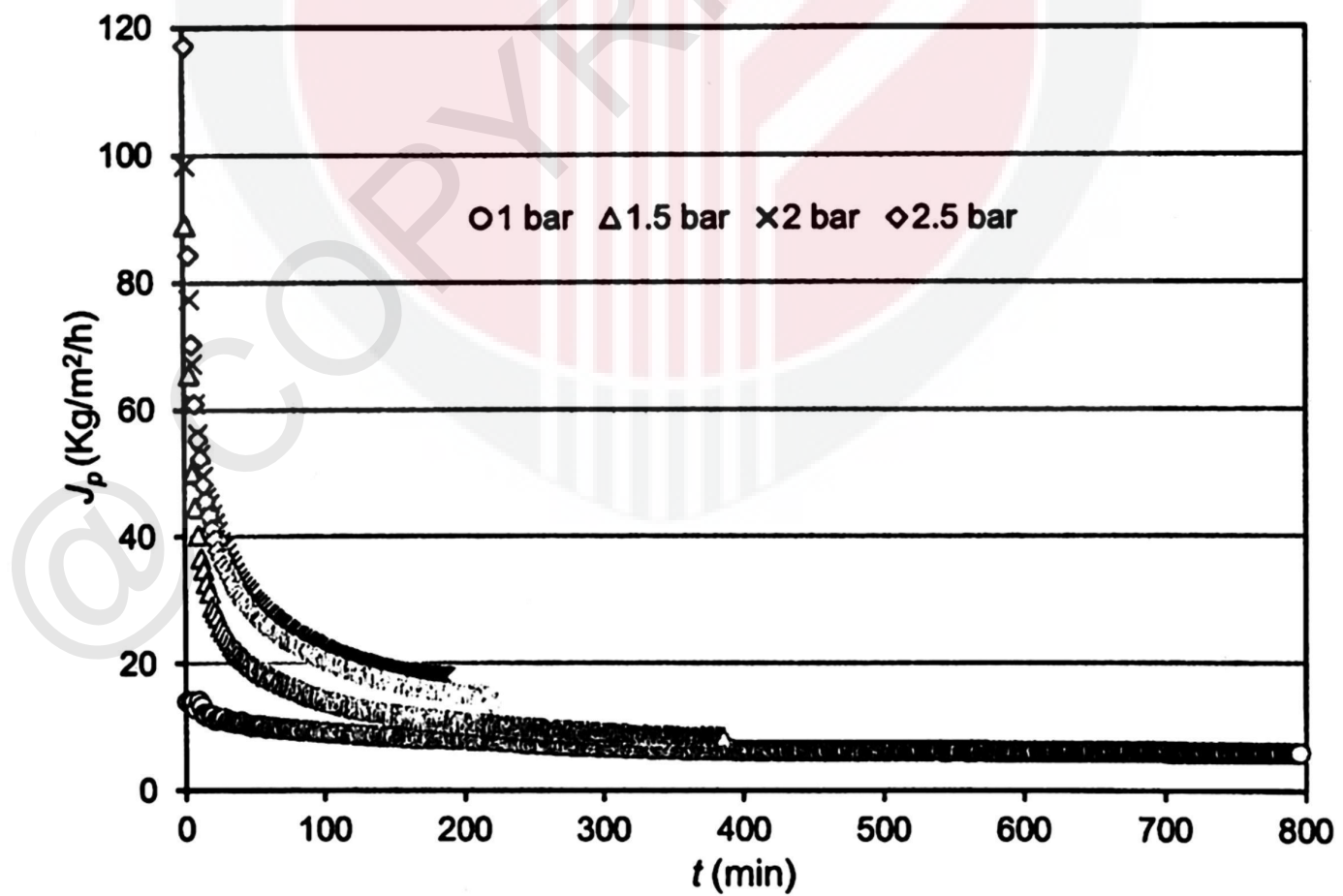


Figure 5: Permeate flux ( $J_p$ ) on function of time ( $t$ ) at different operating pressures

(OP)

Figure 5 shows the behavior of permeate flux ( $J_p$ ) as a function of time ( $t$ ) at different operating pressures. Based on the graph, the permeate flux at pressure of 1 bar showed the lowest initial flux which was 14.06 kg/m<sup>2</sup>/hr and took the longest time to achieve 80% of the permeate recovery (PR), which was about 796 minutes. Flux of 89.06 kg/m<sup>2</sup>/hr at pressure of 1.5 bar was observed in the graph that took 386 minutes to reach 80% of the PR while the permeate flux at pressure of 2.0 bar was 98.44 kg/m<sup>2</sup>/hr for about 186 minutes which was the shortest time to accomplished the 80% of the PR. Besides, the highest initial flux at OP of 2.5 bar was 117.19 kg/m<sup>2</sup>/hr can be seen from the graph and took about 220 minutes to complete 80% of the permeate recovery. The increase in permeate flux with trans-membrane pressure (TMP) was due to the higher driving force across the membrane (Nandi, Das, Uppaluri, & Purkait, 2009).

This findings can be supported by a previous study where they found out that the permeate flux increases with the increases of TMP in pineapple juice (Maria, Carvalho, & Alberto, 2010), passion fruit juice (Huang & Yu, 1987), mosambi juice (Nandi et al., 2009), Xoconostle juice (Castro-mu, Fíla, & Barrag, 2017), apple juice (Bruijna, Venegasb, & Borquezc, 2002) and etc.

As it can be seen from the graph, a continuous decreasing trend was observed up to a final steady state flux in Figure 5. The steady state flux decreased from 14.06 kg/m<sup>2</sup>/hr to 5.95 kg/m<sup>2</sup>/hr at operating pressure of 1.0 bar and from 89.06 kg/m<sup>2</sup>/hr to 8.35 kg/m<sup>2</sup>/hr at a pressure of 1.5 bar. The permeate flux at operating pressure of 2.0 bar was also observed to be decreased from 98.44 kg/m<sup>2</sup>/hr to 17.93 kg/m<sup>2</sup>/hr while for the highest permeate flux at pressure of 2.5 bars, it decreased from 117.19 kg/m<sup>2</sup>/hr to 13.64 kg/m<sup>2</sup>/hr. The reducing of flux is because of the increasing of solute

concentration on the surface of the membrane. Thus, it leads to development of gel type of layer over the membrane surface which causes a gradual increase in gel resistance. Subsequently, the permeate flux decline gradually. In addition, the decreasing on the permeate flux will eventually lead to fouling formation over the membrane surface.

Mirsaeedghazi, Emam-djomeh, & Mohammad (2010) had reported that decreased in permeate flux had a relationship with membrane fouling. The slope of the permeate flux decline during the primary stages was greater than at other times. This is because membrane fouling produced in early stages of testing was greater than in the final stage of microfiltration of pomegranate juice.

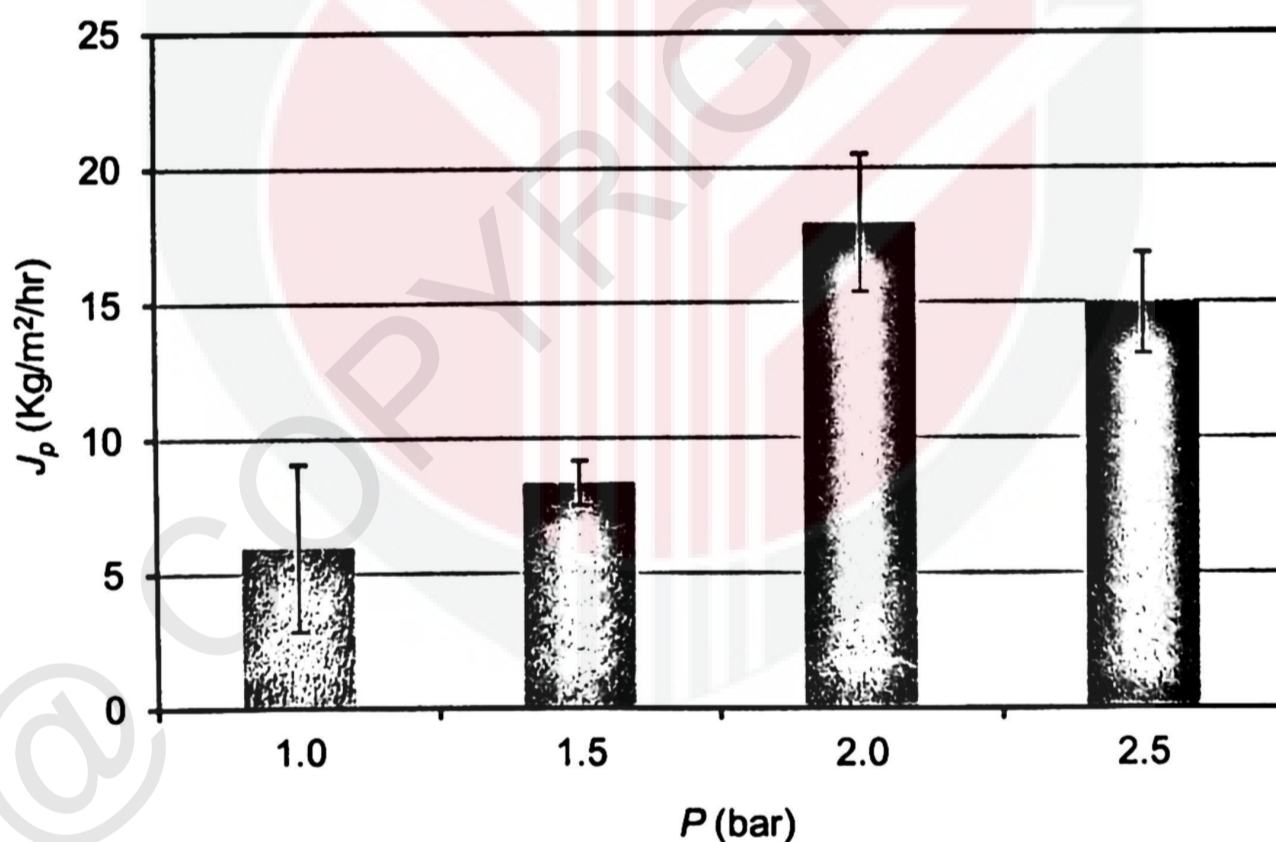


Figure 6: Permeate flux ( $J_p$ ) on function of different operating pressure ( $P$ ) at steady-state

Based on the figure 6, it clearly showed the permeate flux,  $J_p$  increased linearly as the operating pressure,  $P$  increases (from 1.0 to 2.0 bars) at steady-state and decreased back at a pressure of 2.5 bar. The permeate flux showed a deviation from a

linear flux-pressure behavior as the pressure increased and it became independent of pressure (Castro-mu et al., 2017). In this study, a limiting flux ( $J_{lim}$ ), the maximum flux that can be achieved at steady state in an operation, was observed at a pressure of 2.0 bars where there will be no significant increase on permeate flux as further increases of pressure was applied. Therefore, this pressure (2.0 bar) can be considered as the limiting operating pressure ( $OP_{lim}$ ).

After 2 bars, there was no significant incremental on *gap* can be observed at a pressure of 2.5 bar even though the pressure applied was higher as it already achieved the  $OP_{lim}$ . A similar study found that there is no significant increase in permeate flux when further pressure increase applied as it only increased about 4.13% at 72 kPa when compared to 138 kPa in clarification of Xoconostle juice (Castro-mu et al., 2017). Also, in the UF of passion fruit, it was found that when the pressure exceeded 12 bars, a further increase will not improve the flux (Yu & Chiang, 1978).

The  $OP_{lim}$  occurs when the permeate flux is not governed by the driving force, but it were governed by the fouling and polarization-concentration phenomena (A Cassano, Donato, & Drioli, 2007; A Cassano, Marchio, & Drioli, 2007). According to Castro-mu et al. (2017), the  $TMP_{lim}$  gives the maximum permeate flux (or limiting flux) produced by the driving force. The existence of a limiting flux is associate to the concentration polarization phenomenon that appears as the feed solution is passed through towards the membrane where the separation of suspended and soluble solids from the juice takes place.

In addition, the performance of pressure-driven membrane process can be influenced by the operating parameters during UF such as operating pressure, cross-flow velocity and temperature. However, one study has found that operating at high

pressure will not enhance the permeate flux. This is because the thickness of the gel layer will increase and results in more resistance as the driving force is increased (Porter & Porter, 1972).

Figure 7 displays the evolution of the permeate flux on function of time at selected conditions which was operating pressure of 2 bar. The initial productivity of the membrane in terms of permeate flux was 98.44 kg/m<sup>2</sup>/hr and a continuous decreasing was observed up to reach a final permeate flux of 17.93 kg/m<sup>2</sup>/hr. The decreasing trend in the permeate flux might be due to the fouling mechanism, the type of material membrane used and also the sizes of the molecular weight cut off (MWCO).

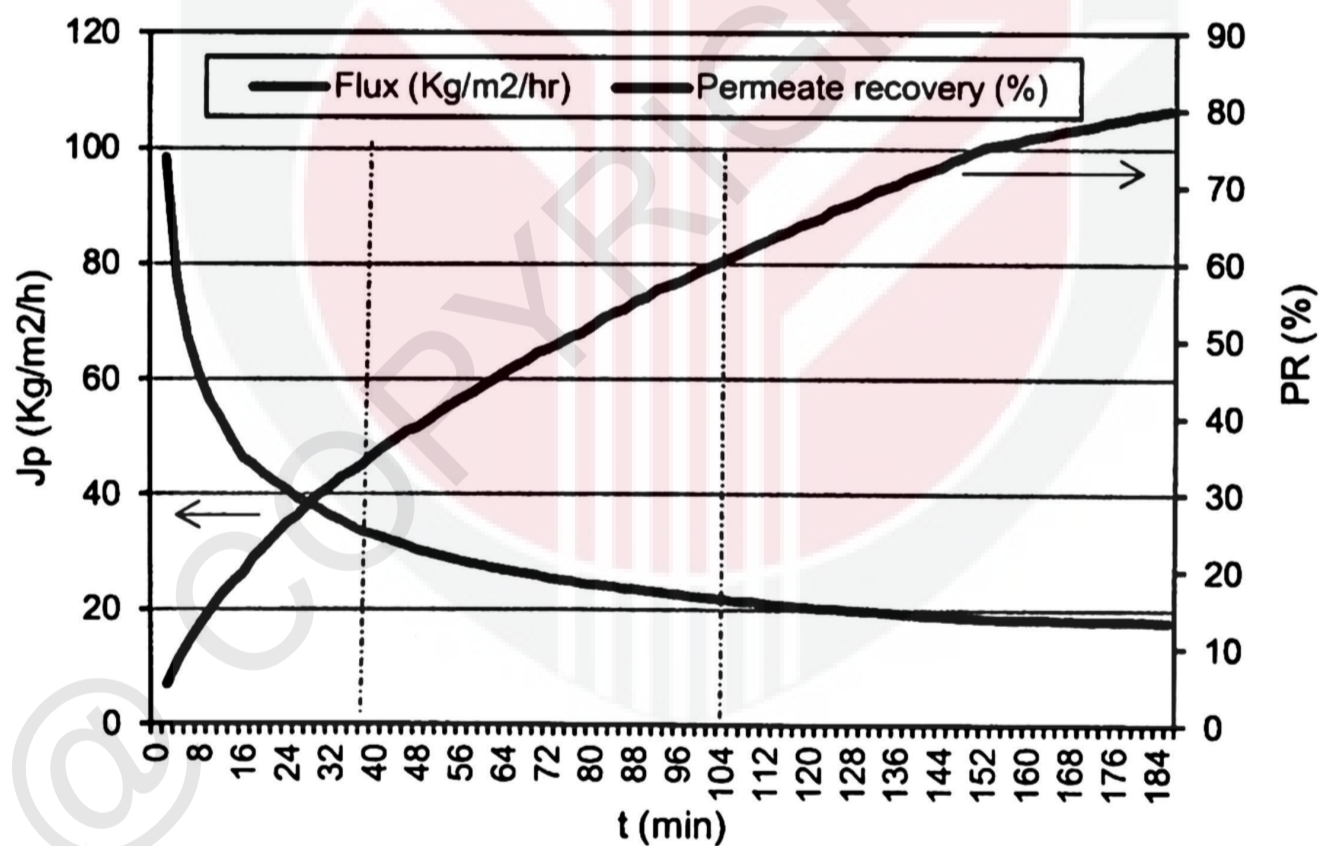


Figure 7: Behaviour of permeate flux ( $J_p$ ) and permeate recovery ( $PR$ ) on function of time ( $t$ ) at operating pressure of 2 bar

There are three states that can be observed on the Figure 7. The first state can be identified as extreme reduction state where it can be seen from the early 30 minutes as the flux decreased extremely from 98.44 kg/m<sup>2</sup>/hr to 37.50 kg/m<sup>2</sup>/hr. The second state was observed from 30-90 minutes where it is known as minor flux reduction state

and the third state was known as steady state flux after 90 minutes that represents non-variation of permeate flux as a function of time.

During the first state, it can be clearly seen that there was a drastic decrease in permeate flux and it started to decrease slowly in the second and third stage. This is due to the gel polarization phenomenon where the feed pass through the membrane and the separation of suspended solid from the guava juice occurs. Hence, when the rejected particles (suspended solids) start to deposit on the membrane surfaces, the rate of flux decreases (Castro-mu et al., 2017).

A similar findings were reported that the fouling and polarization-concentration phenomenon might be the reasons of decreasing trend in permeate flux (A Cassano, Donato, et al., 2007; A Cassano, Marchio, et al., 2007) and also the performance of the pressure-driven membrane process depends on many factors such as MWCO, configuration, membrane material, fouling phenomena and etc. Besides, operating conditions such as feed flow rate, processing pressure and temperature also play important role in the separation process (Boussu, Vandecasteele, & Bruggen, 2008).

In the same graph in Figure 7, it can be observed that the permeate flux decreased as the *PR* increased. This is because as the permeate flux was measured, the permeate recovery as a function of time was also been observed by measuring the mass of permeate was recovered. The clarification process need to be completed at 80% of *PR* before the process was stopped. Thus, at the end of the process, 160 g of permeate was recovered as clarified juice from 200 g of feed, with 40 g remaining as retentate in the system. This result can be supported by a study on clarification of Xoconostle

juice where 81.65% of the initial juice was recovered as clarified juice (Castro-mu et al., 2017).

#### 4.2 Identification of mechanism responsible for flux decline

Table 7 shows the summary of parameters associated to various pore blocking models. The cake formation represented  $n=0$ , intermediate pore blocking was  $n=1$ , internal pore blocking was  $n=1.5$  while  $n=2$  represented complete pore blocking.

At a pressure of 1.0 to 1.5 bars, the value for the lowest SSD was observed at  $n=1$  which was intermediate pore blocking while the process at a pressure of 2.5 bars can be observed exhibiting the complete pore blocking as the value of SSD was closed to  $n=2$ . As the SSD was used to identify the pore blocking mechanism that best represents the experimental flux curves throughout the filtration process, the overall process can be stated that it inhibited intermediate pore blocking.

One study had found that intermediate pore blocking followed with cake filtration represents the most competent combination of fouling mechanisms for the observed membrane flux decline in clarification of mosambi juice (Nandi et al., 2009). In addition, cake filtration fouling mechanism was found in the clarification of apple juice (Taylor, Giorno, & Donato, 2008).

Table 8: Summary of parameters associated to various pore blocking models

Operating pressure (bar)	Pore blocking model							
	Cake formation (n=0)		Intermediate pore blocking (n=1)		Internal pore blocking (n=1.5)		Complete pore blocking (n=2)	
	$K_a$	SSD	$K_b$	SSD	$K_c$	SSD	$K_d$	SSD
1.0	0.0046	32.1471	0.0005	18.9331*	0.0004	54.0851	0.0002	2674192
1.5	0.0568	843.0710	0.0011	311.199*	0.0022	2336.7690	0.0001	89487.15

2.0	0.0442	512.6527	0.0008	168.4934*	0.0015	1490.0970	5.43x10 <sup>-5</sup>	11312.56
2.5	0.0577	959.8069	0.0009	417.1894	0.0020	2153.6340	5.43x10 <sup>-5</sup>	406.919*

\* The smallest RSS for each experimental data block

The intermediate pore blocking occurs when the particles settle on others that were already deposited on the membrane surface, which were already blocking some pores and/or directly blocks some membrane area (Nor & Vasiljevic, 2017).

The fouling mechanism occurs when the solute deposited on the membrane surface as the particle sizes was bigger than the membrane filtration diameter. This finding can be supported by Brião & Tavares (2012) where they found that the fouling mechanism depends on the relationship between solute size and a diameter of the membrane pores. The composition of the fruit juice showed that the major foulants were pectin 34.6%; sucrose 21.6%; glucose 20.4%; fructose 10.8%; cellulose 3.5%; hemicellulose 1.5%, and citric acid 1.2%, on dry basis (Huang & Yu, 1987).

In order to remove the solute particles deposited on the membrane surface, cleaning using distilled water has been used. However, rinsing the membrane module with pure water could not restore the flux that indicated the low water solubility of the deposit. Thus, 0.1% NaOH solution had been identified to reduce the resistance of the deposit and restored the flux readily (Huang & Yu, 1987).

According to Mirsaeedghazi et al. (2010), a major component of fouling produced on the membranes was caused by the formation of a cake layer. However, reversible fouling that can be easily removed by washing with water, acid and alkaline solutions. The recovered membrane can then be reused, since its flux was shown to be similar to that of the first process.

**Figure 8 shows the best line fitting experimental flux curve vs time blocking models at operating pressure of 2 bar.**



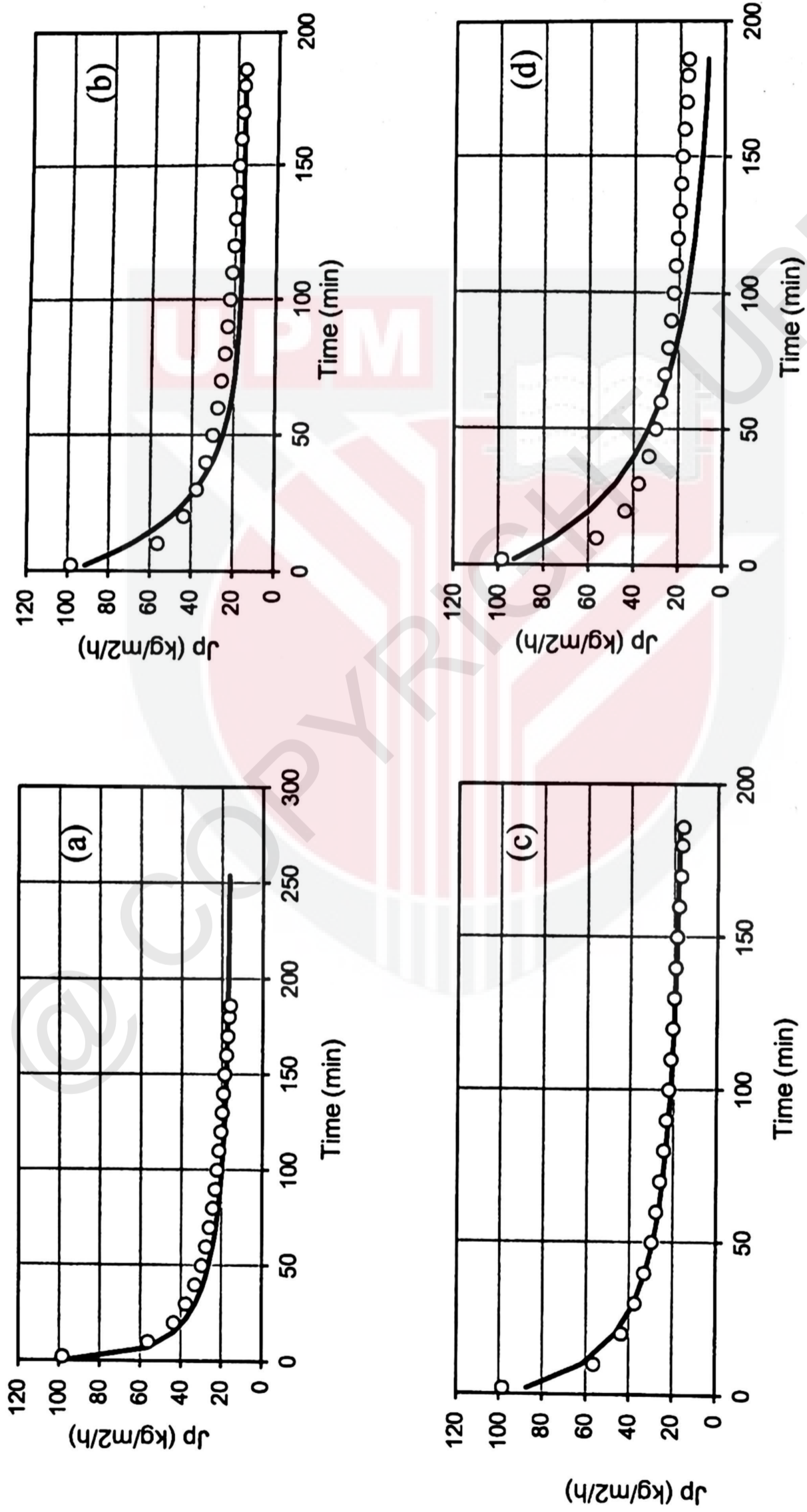


Figure 8: Experimental ( $\circ$ ) vs theoretical flux (-) for different pore blocking models at operating pressure of 2 bar (a) Cake formation model,  $n=0$ ; (b) Intermediate pore blocking model,  $n=1$ ; (c) Internal pore blocking model,  $n=1.5$ ; (d) Complete pore blocking model,  $n=2$

### 4.3 Evaluation of quality attributes of the clarified guava juice

#### 4.3.1 pH

Table 9: Analysis of pH on samples at different pressure

Pressure (Bar)	Samples		
	Feed	Permeate	Retentate
1.0	3.78±0.014 <sup>a</sup>	3.81±0.007 <sup>a</sup>	3.79±0.003 <sup>a</sup>
1.5	3.86±0.005 <sup>a</sup>	3.88±0.002 <sup>a</sup>	3.87±0.005 <sup>a</sup>
2.0	3.73±0.007 <sup>a</sup>	3.73±0.005 <sup>a</sup>	3.72±0.000 <sup>a</sup>
2.5	3.85±0.019 <sup>a</sup>	3.87±0.002 <sup>a</sup>	3.87±0.000 <sup>a</sup>

\* Different letters on the same row indicate the significant differences ( $p < 0.05$ )

Based on Table 8, it shows that there were no significant changes of sample pH after the membrane process at all different operational pressure.

There are no significant differences in pH values in the fresh (feed), clarified (permeate) and retained juice (retentate) at a pressure of 1.0, 1.5, 2.0 and 2.5 bar. This result indicates that the ultrafiltration process does not affect the pH value of the clarified guava juice. Similarly to the other clarification of fruit juices using UF, it shows that there are no obvious changes in pH value (Alfredo Cassano, Conidi, & Drioli, 2010; Alfredo Cassano, Tasselli, Conidi, & Drioli, 2009; Castro-mu et al., 2017).

Previous research has been done by Brasil, Maia, & Figueiredo (1995) shows that the clarification of guava juice using pectic enzyme and fining agents at different

stage of processing has a pH value of 3.8 for natural pulp, 3.75 for clear juice and 3.8 for cloudy juice which indicates that there are also no significant change in pH value. This previous study had very similar pH value obtained in the Table 4 which indicates that pH value would not be affected when undergoes UF process either by decreasing or increasing the operating pressure.

#### 4.3.2 Total Soluble Solids (TSS)

Table 10: TSS on samples at different operating pressure

Operating pressure (bar)	Feed (Brix')	Permeate (Brix')	Retentate (Brix')
1.0	5.30±0.283 <sup>a</sup>	4.92±0.071 <sup>a</sup>	5.48±0.306 <sup>a</sup>
1.5	7.15±1.155 <sup>a</sup>	5.62±1.061 <sup>a</sup>	7.60±0.943 <sup>a</sup>
2.0	6.30±0.189 <sup>a</sup>	5.20±2.121 <sup>a</sup>	7.65±0.778 <sup>a</sup>
2.5	7.25±0.825 <sup>a</sup>	6.73±0.849 <sup>a</sup>	7.97±0.283 <sup>a</sup>

\* Different letters on the same row indicate the significant differences (p<0.05)

In an analysis of TSS, UF did cause a slight reduction in permeate of guava juice which shown in Table 9. At an operating pressure of 1 bar, there was slightly decreased in total soluble solids from 5.3 to 4.92 in permeate when compared to the fresh guava juice (feed). The same condition was observed in other pressure where the TSS decreased about 21.4% for 1.5 bars, 17.5% for 2.0 bars and 7.2% for 2.5 bars. This is because during the UF process, it removed the suspended solids which might also remove some of the total soluble solids in the guava juice. This result can be supported by A Cassano, Marchio, et al. (2007), where they found out that UF process

was completely removed the suspended solids in freshly squeezed blood orange juice and there was a reduction in total soluble solids. TSS has depended on the low molecular weight compounds such as sugars (sucrose, glucose and fructose), mineral salts and some organic acids (Reiter, Neidhart, & Carle, 2003).

Besides, higher TSS content can be observed in the retentate when compared to feed and permeate for all pressure. For example, at a pressure of 2 bars, the TSS content increased about 17.6% in the retentate when compared to the feed stream. Higher suspended solid retained during UF process might be the cause of higher TSS content in the retentate stream.

This results can be supported by one study that had reported the higher TSS content in the retentate probably related to the presence of high suspended solids content in the pulpy products that can disturb with the measurement of the refractive index by confirming that sugars and organic acids were not being significantly different from those in the feed, permeate and retentate (Cisse, Vaillant, Perez, Dornier, & Reynes, 2005).

A similar findings where the TSS content was high in retentate was obtained in the clarification of melon juice (Fabrice Vaillant et al., 2005), clarification of blood orange juice (A Cassano, Marchio, et al., 2007), clarification of Xoconostle juice (Castro-mu et al., 2017) and ultrafiltration of kiwi fruit juice (A Cassano, Donato, et al., 2007) .

As the TSS were totally concentrated in the retentate stream by removal of the suspended solids, consequently, the flux decline during UF relate to fouling layers

formed by a combination of suspended solids and adsorbed macromolecular impurities (A Cassano, Marchio, et al., 2007).

### 4.3.3 Turbidity

Table 11: Turbidity at different operating pressure

Operating pressure (bar)	Feed (NTU)	Permeate (NTU)	Retentate (NTU)
1.0	20.01±5.037 <sup>a</sup>	0.99±0.500 <sup>a</sup>	33.32±16.051 <sup>a</sup>
1.5	56±9.522 <sup>ab</sup>	1.48±0.438 <sup>b</sup>	85.80±21.967 <sup>a</sup>
2.0	107.03±20.223 <sup>b</sup>	10.03±9.225 <sup>c</sup>	253.83±21.449 <sup>a</sup>
2.5	35.13±6.835 <sup>a</sup>	1.19±0.302 <sup>b</sup>	52.75±2.805 <sup>a</sup>

\* Different letters on the same row indicate the significant differences ( $p < 0.05$ )

According to the Table 10, a positive change in terms of physical properties of turbidity was observed. The turbidity of guava juice for all samples at a pressure of 1.0, 1.5, 2.0 and 2.5 bar was almost completely reduced when compared to permeate and the feed. 97% of turbidity was reduced in pressure of 1.5 bar while the other samples at a pressure of 1.0, 2.0 and 2.5 bar was also achieved at 90% and above. This indicated that the suspended solids in the guava juice were completely rejected by UF process and subsequently a higher level of clarity was achieved. A study had reported that clarified kiwifruit juice had a negligible turbidity as the suspended solids in the fresh kiwifruit juice was completely removed by UF (A Cassano, Donato, et al., 2007).

Based on previous studies, retention of 87.50% in turbidity was achieved in the clarified of Xoconostle juice through membrane technology, which gives positive

changes in terms of the physical aspect (Castro-mu et al., 2017). This means, a clarified Xoconostle juice rich in its nutritional components was obtained, whereas a concentration of the suspended solid was obtained in retentate stream.

The higher value of turbidity in all retentate streams for all operating pressure was also being observed in the Figure 7. This is due to the formation of a denser fouling layer that caused a higher solute retention over the membrane surface. The results can be supported by Castro-mu et al. (2017) where they found out the concentration of the suspended solids was obtained in the retentate stream by confirmed with the increases of density values in Xoconostle juice.

Similar results were obtained in the clarification of banana, pineapple, blackberry, melon and apple juice where the turbidity was greatly reduced in the permeate and high in the retentate stream (F Vaillant, Pérez, Acosta, & Dornier, 2008; Fabrice Vaillant et al., 2005; Wu, Zall, & Tzeng, 1990).


The positives changes in terms of physical aspect of turbidity was important in clarification of guava juice. This is because consumers nowadays had high demands on the good appearance of fruit juice. According to Castro-mu et al. (2017), a clear juice from Xoconostle fruit can be more attractive for humans in order to increase its consumption. Moreover, remarkable changes were observed on colour properties in the process juice through the removal of the turbidity.

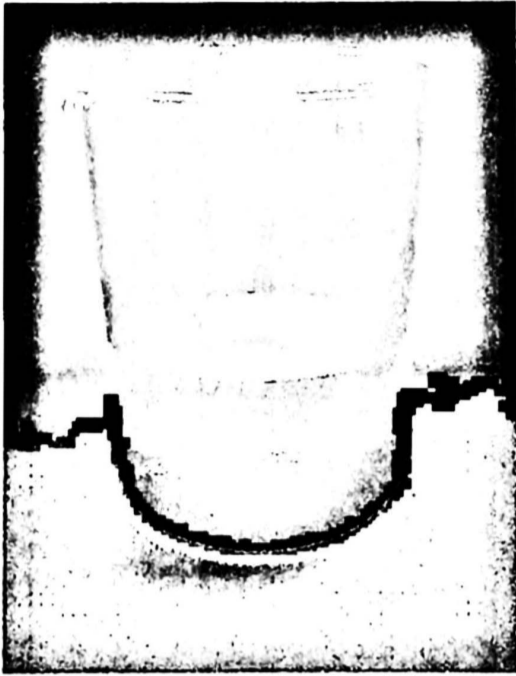


In addition, permeates from UF membranes were reported by panellists as “light” and “sparkling clear” of apple juice while in MF membrane, the apple juice permeates were “dark” (Wu et al., 1990).

#### 4.3.4 Colour characteristic

Table 11 showed the colour changes of guava juices before and after undergoes UF process. All samples at different operating pressure having colour changes and become less turbid. As can be seen, the guava juice that has undergone UF had more pleasant colour and appearance when compared to the raw guava juice obtained after extraction process.

Table 12: Colour changes of guava juice

Samples	Colour	Observations
<b>Before undergoes UF</b>		
Raw guava juice (fresh obtained from extractor)		The juice is cloudy and contain fibers, suspended solids and high molecular weight carbohydrates

<p>Fresh guava juice after centrifuge (feed)</p>		<p>The juice is less cloudy and turbid</p>
<p>After undergoes UF</p>		
<p>Clarified guava juice (permeate)</p>		<p>The juice is clear and not turbid</p>
<p>Guava juice that retained in the UF (retentate)</p>		<p>The juice in the retentate stream slightly cloudy and more turbid compare to the feed</p>

In order to prove the colour properties of guava juice had changed during UF, analysis on colour properties using colorimeter has been performed. The colour of guava juice was evaluated with tristimulus colour of system CIELab to obtain the C.I.E-L\*, a\* and b\* colour coordinates. The L\*, a\* and b\* represents lightness/darkness, greenness/redness and yellowness and blueness (Abid, Yaich, Hidouri, Attia, & Ayadi, 2018).

Table 12 exhibits the results analysis of colorimeter at different operating pressure. At a pressure of 2.0 bar, the feed presented a\* and b\* values of -0.14 and 1.25 indicated the juice exhibited greenness degree which also applied to the pressure at 1.0, 1.5 and 2.5 bar. The b\* value decreased up to 0.26 whereas decreases of a\* value to -0.38 was obtained during the clarification. Besides, the h° value increased from -84.12 to -52.43 which means that the greenness grade in the clarified juice was decreased by clarification as it behaved towards “clear” juice. Moreover, the brightness (chroma) value obtained was decreased from 0.80 to 0.26.

Table 13: Data analysis of colorimeter at different operating pressure

Operating pressure (bar)	Samples	Colour				
		L*	a*	b*	Hue angle (°)	Chroma
1.0	Feed	31.05±0.285	-0.05±0.049	0.68±0.064	-85.70	0.23
	Permeate	32.21±0.783	-0.25±0.014	0.22±0.066	0.03	0.06
	Retentate	31.85±0.170	-0.14±0.172	0.73±0.052	-79.34	0.28
1.5	Feed	31.05±0.285	-0.05±0.049	0.68±0.064	-85.70	0.23
	Permeate	32.21±0.783	-0.25±0.014	0.22±0.066	0.03	0.06

	<b>Retentate</b>	31.85±0.0170	-0.14±0.172	0.73±0.052	-79.34	0.28
<b>2.0</b>	<b>Feed</b>	32.27±0.038	-0.14±0.160	1.25±0.186	-84.12	0.80
	<b>Permeate</b>	32.93±0.754	-0.38±0.045	0.26±0.213	-52.43	0.26
	<b>Retentate</b>	31.80±0.082	-0.18±0.087	1.12±0.099	-80.90	0.64
<b>2.5</b>	<b>Feed</b>	31.73±0.040	-0.01±0.052	0.55±0.125	0.44	0.16
	<b>Permeate</b>	32.27±1.200	-0.25±0.259	0.80±0.137	-42.15	0.38
	<b>Retentate</b>	32.50±0.511	-0.26±0.108	-1.05±0.297	74.94	9.589

A clear guava juice is more preferable and high acceptance in consumer. These results were supported by Castro-mu et al. (2017) where they found that a clear Xoconostle fruit juice can be more attractive for human in order to increase its consumption while Mirsaeedghazi et al. (2010) stated that clarified pomegranate juice obtained from membrane processing has more desirable colour than fresh juice, and can improve the marketability of the product.

#### 4.3.5 Total Phenolic Content (TPC)

The total phenolic content from feed, permeate and retentate at different operating pressure was determined using Folin-Ciocalteu colorimetric method by manipulating the regression equation of gallic acid calibration curve ( $y=0.0111x - 0.0148$ ,  $R^2=0.9998$ ). The results obtained were tabulated in the Table 11.

Based on Table 11, it showed that the there was 19.74% reduction in TPC at 1 bar when compared in permeate and feed. About 47.25% reduction at 1.5 bars and 20.38% reduction at 2 bars was observed in Table 13 while at 2.5 bars, there was

38.91% reduction in TPC. Therefore, it can be stated that the higher the driving force, the greater the amount of reduction in TPC was observed. Thus, pressure at 2 bars showed the suitable pressure to run UF of guava juice as it gives a small reduction in TPC while giving a greater driving force.

Table 14: Total phenolic content of guava juice at different operating pressure

Operating pressure (bar)	Index	Absorbance (A <sub>765</sub> )	Total Phenolic Content (mg/L)
1.0	Feed	1.10±0.140 <sup>a</sup>	100.43
	Permeate	0.88±0.009 <sup>a</sup>	80.61
	Retentate	0.88±0.297 <sup>a</sup>	80.61
1.5	Feed	2.25±0.011 <sup>a</sup>	204.04
	Permeate	1.18±0.006 <sup>c</sup>	107.64
	Retentate	1.74±0.049 <sup>b</sup>	158.09
2.0	Feed	2.34±0.013 <sup>a</sup>	212.14
	Permeate	1.86±0.004 <sup>c</sup>	168.90
	Retentate	1.95±0.036 <sup>b</sup>	177.01
2.5	Feed	1.45±0.004 <sup>a</sup>	131.96
	Permeate	0.88±0.002 <sup>c</sup>	80.61
	Retentate	1.35±0.006 <sup>b</sup>	122.95

\* Different letters on the same row indicate the significant differences (p<0.05)

Acar (2005) stated that the main purpose of the clarification in pomegranate juice is to reduce the amount of phenolic compounds and reduce the astringency of the product.

Although, the decreases of total phenolic content in juice clarification reduced the astringency of the guava juices, but it is important in the contribution of antioxidant content. Thus, a small reduction of TPC in guava juice clarification showed that the membrane-based process does not reject the total phenolic content completely and still relatively high content of antioxidant. A study has found that only 6.1% of total phenolic content was reduced in clarification of pomegranate juice using UF. This is because the phenolic compounds cannot be retained by a UF membrane that caused the permeate samples had more phenolic contents (Acar, 2005).

As the fruits ripen, TPC decreases, but ascorbic acid content (AAC) increases. Both varieties of guava fruit contain relatively high quantities of antioxidants, as shown by the high amount of TPC and AAC recorded. In the case of AAC, guava can contain as much as ten times that of other fruits such as banana, dragon fruit, starfruit and sugar apple. In the case of TPC, only starfruit and sugar apple has a comparable quantity of the antioxidant. On the whole, the results suggest that guava is a healthy fruit to consume from the antioxidant viewpoint, and is better than the temperate fruit orange (Jhi, 2006).

The highest TPC value was detected for mango among all fresh juices while pomegranate among commercially 100% juices while guava among fruit drink (Wern, K.H, Haron, H., Keng, 2016).

The size of pore membrane gave an impact on clarifying juices of total phenolic content. This finding can be supported as in clarification of pineapple juice, the highest

value of total phenolic content as 69.34 mg GAE/100 ml using 0.2  $\mu$ m membrane while the lowest value of total phenolic content was obtained by 30 kDa membrane size (Laorko, Li, Tongchitpakdee, Chantachum, & Youravong, 2010). Thus, the membrane pore size play an important role as it will affect the reduction of total phenolic compound.

#### 4.3.5 Ascorbic acid

Table 15: Ascorbic acid content at different operating pressure

Operating pressure (bar)	Ascorbic acid (mg) in 100 ml		
	Feed	Permeate	Retentate
1.0	28.917 $\pm$ 0.000 <sup>ab</sup>	22.491 $\pm$ 1.515 <sup>b</sup>	31.595 $\pm$ 0.757 <sup>a</sup>
1.5	30.524 $\pm$ 0.757 <sup>a</sup>	23.562 $\pm$ 3.029 <sup>a</sup>	27.846 $\pm$ 0.000 <sup>a</sup>
2.0	28.917 $\pm$ 1.515 <sup>a</sup>	23.562 $\pm$ 1.515 <sup>a</sup>	27.846 $\pm$ 0.757 <sup>a</sup>
2.5	33.201 $\pm$ 1.515 <sup>ab</sup>	24.098 $\pm$ 0.757 <sup>b</sup>	29.988 $\pm$ 1.515 <sup>ab</sup>

\* Different letters on the same row indicate the significant differences ( $p < 0.05$ )

Table 14 showed the ascorbic acid content in 100 ml at different operating pressure (OP). There was 22.22% reduction in ascorbic acid at 1 bar, 22.81% reduction at 1.5 bar, 18.52% reduction at 2 bars and 27.42% at 2.5 bars when compared between feed and permeate. The small reduction in ascorbic acid content in guava juice showed that the UF does not highly affect the vitamin C content in guava juice. Also, it can be observed that at 2 bars, the reduction of ascorbic acid was only 18.52%, which give positive results as the high driving force can still manage to hold the vitamin C content of guava juice during UF process.

The ascorbic acid content in white seeded guava fruit was known to be high when compared to banana, dragon fruit and star fruit (Jhi, 2006). This claim can be proved from this study as the guava juice contained high ascorbic acid when compared to the banana (4.9 mg/100 g, dragon fruit (8.0 mg/100 g) and star fruit (5.2 mg/100 g).

Besides, the seeded guava contained high ascorbic acid when compared to seedless guava and contains high primary antioxidant potential when compared to other local fruits and an imported fruit such as orange (Jhi, 2006).

However, from this study, it can be observed that the fresh guava juice contained low ascorbic acid range from 28-33 mg/100 ml when compared to orange juice and guava fruit where Sciences, Akesowan, & Choonhahirun (2013) claimed that guava fruit has (100-200 mg/100 g) of ascorbic acid which higher than a fresh orange juice (60-80 mg/100 ml).

This might be due to the guava fruit itself, where it was not fully matured and all the skin was removed when extracted thus, it causes the low amount of ascorbic acid in guava juice. This finding can be supported by Menzel & Branch (2017) where they found out that the stem and outer flesh of guava fruit contain the most of the ascorbic acid. Its content achieved a maximum in green fully mature fruit and starts to decline rapidly as they ripen. However, colour of the flesh does not associate with ascorbic acid level.

## CHAPTER 5

### CONCLUSION AND RECOMMENDATION

#### 5.1 Conclusion

This study aims to explore the potential of applying the membrane-based process using ultrafiltration for the production of guava juice at different operating pressure (OP). Besides, it also determine the effect of membrane processing conditions on the permeate flux behaviour and guava juice quality attributes. The clarification of guava juice was performed via ultrafiltration with 100 kDa PES membrane at OP of 1.0, 1.5, 2.0 and 2.5 bar.

It was found that the highest steady-state flux which was 17.93 kg/m<sup>2</sup>/hr has been achieved at operating pressure (OP) of 2.0 bar. Besides, 80% of the initial juice was recovered in permeate as clarified juice. In this study, a limiting flux was observed at OP of 2.0 bar where there will be no significant increase on permeate flux as further increases of pressure was applied. The decline in permeate flux was analysed using different membrane pore blockings. Intermediate pore blocking was identified as the best suited model.

Moreover, the preservation of the physiochemical, nutritional and functional properties of guava juice has been achieved through the clarification process by

ultrafiltration. The UF process managed to remove the components that contribute to turbidity, reduction of TSS, reduction in TPC and a stable pH and vitamin C that leads to enhance the colour properties of the guava juice which indicating the successfulness of the clarification process. As the potential advantage of ultrafiltration in the fruit juice processing industry is undeniable, this can support the clarified guava juice commercialization value as it is minimally processed food.

## **5.2 Recommendations**

Since the fouling phenomenon occurs in every membrane based process, application of vibration and rotating membrane systems can be used to optimize the control of fouling phenomenon by create shear rate at the membrane interface.

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## APPENDICES

J	98.44		
J <sub>pss</sub>	16.13		
K <sub>c</sub>	0.044205923		
		SSD	512.652704
<b>2.0 bar</b>			
time (min)	J <sub>p</sub> (kg/m <sup>2</sup> /hr)	model	square dev
2	98.44	91.47287	48.506066
10	56.25	69.02964	163.319107
20	43.59	50.12886	42.7076936
30	37.50	37.98112	0.23147288
40	33.046875	30.17362	8.25559778
50	29.8125	25.15565	21.6862441
60	27.65625	21.93054	32.7837044
70	25.98214286	19.85773	37.5084179
80	24.375	18.52551	34.2165309
90	23.22916667	17.66928	30.9123803
100	22.21875	17.11896	26.0078076

Appendix 1: Raw data of fouling analysis at 2 bar



Appendix 2: UF laboratory scale set-up

Time (min)		Accum. ulated weight (g)	Separ. ated weight (g)	Flux (g/m <sup>2</sup> /min)	Flux (Kg/m <sup>2</sup> /hr)	Time (min)		Accum. ulated weight (g)	Separ. ated weight (g)	Flux (g/m <sup>2</sup> /min)	Flux (Kg/m <sup>2</sup> /hr)	Flux (Kg/m <sup>2</sup> /hr)		Mean	Std. Dev.	Std. Er.
												g 1	g 2			
0	0	0	0	0	0	0	0	0	0	0	0	0	0			
2	11					2	10	10	1062.5	33.75	33.75	103.13	33.75	38.44	6.63	4.89
4	24					4	18	5	1771.9	70.31	70.31	84.38	70.31	77.34	3.34	7.83
6	38					6	26	4	389.58	53.38	53.38	75.00	53.38	67.19	7.105	7.81
8	52					8	34	5	1132.81	87.97	87.97	87.97	53.91	80.94	3.34	7.03
10	66					10	42	6	1062.50	63.75	63.75	63.75	48.75	58.25	10.61	7.90
12	80					12	50	5	1075.83	60.94	60.94	60.94	45.31	53.13	11.05	7.81
14	94					14	58	3	337.50	56.25	56.25	56.25	42.88	43.95	9.47	6.70
16	108					16	66	2	853.38	51.98	51.98	51.98	41.02	46.23	7.46	5.27
18	122					18	74	6	888.05	52.08	52.08	52.08	38.94	45.31	9.58	6.77
20	136					20	82	3	828.13	43.63	43.63	43.63	37.90	43.93	6.82	6.09
22	150					22	90	4	735.45	47.73	47.73	47.73	36.85	42.19	7.83	5.64
24	164					24	98	4	781.25	46.88	46.88	46.88	35.18	41.02	8.29	5.88
26	178					26	106	2	745.19	44.71	44.71	44.71	33.89	33.30	7.85	5.41
28	192					28	114	3	725.45	43.83	43.83	43.83	33.48	38.90	7.10	5.02
30	206					30	122	3	708.33	42.90	42.90	42.90	32.90	37.90	7.07	5.00
32	220					32	130	2	663.53	41.02	41.02	41.02	31.64	36.33	6.63	4.89
34	234					34	138	3	670.98	40.28	40.28	40.28	30.88	35.57	6.63	4.89
36	248					36	146	2	651.04	39.88	39.88	39.88	29.83	34.38	6.63	4.89

Appendix 3: Raw data of flux analysis at 2 bar



Appendix 4: Condition of membrane after UF

## **CONFERENCE**

J. M. Omar and M. Z. M. Nor. *Clarification of Guava Juice Through Membrane-based Process*. Malaysian Society of Agricultural Engineers (MSAE) Conference 2019, Wisma Tani, Putrajaya, 21 Mac 2019. (Oral presenter)

## **ACHIEVEMENT**

J. M. Omar. *Clarification of Guava Juice Through Membrane-based Process*. Final Year Project Open Day, Faculty of Engineering, UPM, 28 Mei 2019. (Bronze award)

