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Study of Characterization of Pectin Gel Extracted from Malaysian Pomelo Fruit (*Citrus Maxima Merr.*) Peels as a Noble Food Ingredients

H. Chek Zaini, M. J. Noor Hanan and I. Suzila

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INTRODUCTION

POMELO FRUIT

- In Malaysia, approximately 1895 ha of pomelo (Citrus Maxima Merr.) trees are grown commercially, with an estimated annual production of 8830 metric tons of pomelo fruits. It is widely grown in the state of Perak, Kedah, Melaka, Kelantan and Johor. This largest citrus fruit is growing as large as 30 cm in diameter and weighing as much as 10 kg; the peel is very thick but soft and easy to peel away (Hameed *et al*., 2008).

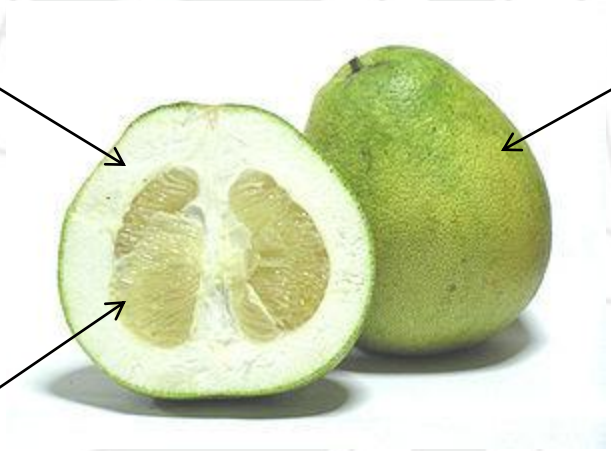
Pomelo fruit is commonly eaten fresh and available as food complements in desserts, salads, fruit cocktails, jam, juice combinations or food processing industries (Anon, 2011).

The thick peels of the pomelo are sometimes used to prepare marmalades and sweet candies (Hameed *et al*., 2008). However, the thick peels that surround the flesh of pomelo are bitter and usually discarded as waste.

POMELO FRUITS

Albedo

Flavedo



Flesh

Pomelo Fruit

POMELO PEELS

- The peels of citrus fruit are subdivided into the epicarp or flavedo and mesocarp or albedo. The part that is called flavedo is the colored peripheral surface of the peel while the part that is called albedo is the white soft middle layer of the peel (Ramful et. al, 2010).
- The pomelo fruit peel is reported to have antioxidant activity. The antioxidant activity in pomelo fruit peel (flavedo and albedo) have been studied by Matook and Fumio (2006) and the results strongly showed that the extract of flavedo and albedo can be used as easily accessible source of natural antioxidant and as a possible food supplement or in pharmaceutical industry. Figure 1 shows the albedo, flavedo and flesh of pomelo fruit.

ALBEDO FOR THE EXTRACTION OF PECTIN

PECTIN JELLY

- Pectin can have diverse functional properties, however the clearly known and common to various sources since the earliest studies (Broconnot, 1825; Vauquelin, 1970) is gel formation under specified conditions (Yapo, 2009a).

POMELO PECTIN

The major raw materials used for production of commercially acceptable pectin are citrus peel and apple pomace (May, 1990).

Pectin widely used in manufacturing jam, jellies, marmalades, preserves and function as a thickening agents for ketchup, sauces, and flavoured syrup. It is also useful as texturising agent in fruit-flavoured milk desserts (Madhav & Pushpalatha, 2002).

POMELO FLESH

END PRODUCT:POMELO JELLY

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OBJECTIVES

1. To determine ratio of albedo and water for pectin extraction.
2. To study the tenderness of the pectin jelly extracted from pomelo fruit peel.
3. To study the effect of sugar level in the production of pectin jelly.
4. To utilize pectin jelly for industrial application as an alternative to gelatin. Halal Issue

SOURCES OF PECTIN

- Found in most plants.
- The highest concentrations of pectin are found in the middle lamella of cell wall, with a gradual decrease as one passes through the primary wall toward the plasma membrane (Kertesz, 1951).
- The main source of pectin is from citrus fruit peel (Wang, *et al.,* 2002). Pectin obtained from citrus peels is referred to as citrus pectin.
- Industrial pectin's are commonly extracted from citrus peels and apple pomace with a multiple-stage physical-chemical process characterized by an extraction step with hot dilute mineral acid and recovery through alcohol precipitation (Daniel *et al.,* 1994; May 1990).

CLASSES OF PECTIN

- **Depending on the degree of methoxylation (DM), pectins are usually classified into two classes that are:**

1. High methoxyl (HM) pectin gel which is extracted normally and has more than 50% esterified acid units

2. Low methoxyl (LM) pectin gel where modification of extraction process or continued acid treatment will yield pectin with less than 50% methyl ester groups.

- **The degree of methoxylation governs the type of interactions in semidilute solutions and gelling effects of aqueous solutions of these systems (Schrieber et al., 2003).**

APPLICATIONS OF PECTIN

- Pectins have many applications in food science and nutrition, cosmetics and pharmacy. Due to its physical and chemical nature, pectin has high water-holding capacity. Because of that, pectin is isolated from apple pomace and citrus fruit peel and used as a gelling substances and thickening foods. Pectin is also used in traditional and modern medicine (Holloway *et al.*, 1983).

In pharmaceutical, pectin has been used as a matrix for prolonged drug release (Sungthongjeen *et al.,* 2004; Sriamornsak *et al*., 2007), as a mucoadhesive polymer for controlled drug delivery (Thirawong *et al*., 2007; Thirawong *et al*., 2008), as a carrier for floating drug delivery (Sriamornsak *et al*., 2005;Sriamornsak *et al.,* 2008), and as a carrier for colonic drug delivery (Sriamornsak, 1999; Sriamornsak *et al*., 2003

Pectin has been mainly used as a gelling agent in jams and jellies as well as a stabilizer in fruit juices and milk drinks. It has received great interest as a source of dietary fiber in foods (Tinker *et al*., 1994; Willats *et al*., 2006).

Table 1 : Pectin condition for gelation (Tony & Ming, 2003)
High methoxyl & Low methoxyl pectin

Conditions	HM Pectin	LM Pectin
pH	≤ 3.5 (range: 1.0– 3.5)	1.0 – 7.0 (or higher)
Soluble solids (S.S.)	≥ 55% (range: 55–85%)	0 – 85% (S.S. affects Ca2+ required)
Ca 2+	normally not a factor	required !!

GELATIN ISSUE

- One of the most controversial issues in food industry in Muslims world is gelatin based food products. Gelatin uses as value-added ingredient in foods because of its unique properties. Issues arise because of pig and cattles are the main sources of gelatin used in food industry (Mohamad Aizat *et al*., 2011).

There is an alternative of gelatin that is from fish. However, there are no gelatin substitutes from plant-based as yet being produced. Pectin and gelatin both are used as thickening agents in foods, but they give different results to the food products.

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MATERIALS AND METHODS

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MATERIALS

- Local Pomelo fruits with white pulp, Ipoh, Perak , Malaysia.
- The albedo parts is cut for pectin extraction.

Methods

a) Extraction of pectin (Pomelos)

- Washed
- Chopped
- Weighed
- Boiled
- Extracted the juices

Boiled chopped albedo

Methods

b) Procedure for making jelly

Boil extracted albedo juice and sugar

Add lemon juices

Boil until it reached gelling point (104.4˚C)

Keep in the container

Cooled

Keep in the chiller

POMELO JELLY

Analysis of Pectin Gel

1. Proximate Analysis

   Moisture content, protein, fat, ash, and fiber in jelly was determined in the proximate analysis according to the AOAC method. Carbohydrate content was calculated using the following calculation:

   % Carbohydrate = 100% - (% moisture + % ash + % fat + % protein + % fiber)

2. Determination of tenderness using percentage sag

Measurement of the tenderness of jelly is called percentage sag. The percentage (%) Sag is determined using the calculation below:

$$\% \text{ Sag} = \frac{\text{molded height} - \text{unmolded height}}{\text{molded height}} \times 100$$

Determining Effect of Sugar in Pectin Gel

Variation of sugar content	• 4 Samples of pectin jelly was prepared with different amount of sugar: Sample 1; 40g, Sample 2; 60g, Sample 3; 80g, and Sample 4; 100g.
pH measurement	• The pH of the juice is measured at room temperature using digital pH meter to determined the acidity of pomelo peels juice and the reading was recorded.
Determination of gelling point using Spoon Sheet Test	• Gelling point of pectin jelly was determined by using Spoon Sheet Test. • When it started to falling like a sheet, cooking was stopped (Madhav & Pushphalata, 2002).

Line Spread Test (Determination of spreadibility)

- An easy and portable tool that determines how far liquids flow
- A paper sheet was marked with concentric circles spread 0.5 inch apart from 0.5 inch to 3.5 inch radius. The hard plastic was covered the paper sheet that have been marked. The test was done by pouring the jelly at the center of line spread paper. Measurement of the jelly spread is taken and recorded.

Color measurement of jelly

- Measured by using Colorimeter
- The color is based on Hunter L, a, b color scale.

Acceptability test

- can be determined by sensory evaluation; scientific method used to evoke, measure, analyze and interpret those responses to products as perceived through the senses of sight, smell, touch, taste, and hearing (Stone & Sidel, 1993).
- The sensory analysis was done on QDA and Hedonic test to 10 people.

Statistical analysis

- Analysis of Variance (ANOVA) was performed to calculate the significant difference of the samples. One Way ANOVA was used to prove there are significant different of sample with different treatment.

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RESULTS AND DISCUSSION

Extraction of Pectin Juice

Table 3 :Formulation of extraction of pectin juice The different proportions of albedo and water used to extract pomelo peel juice

Formulation	1	2	3	4	5
Albedo (g)	40	152	260	500	500
Water (ml)	35	100	140	1000	500
Ratio of albedo to water	1:0.88	1:0.70	1:0.54	1:2.00	1:1.00

Jelly Making

Formulation	1	2	3	4	5
Juice Extracted (mL)	40	60	100	500	150
Sugar (g)	20	120	70	300	100
Citric Acid (mL)	4	6	16	15	50
Ratio pectin juice to sugar to citric acid	10:5:1	10:20:1	6.3:4.4:1	33.3:20:1	3:2:1
Yield of Jelly (ml)	42	62	120	784	160

- In this study, five formulations have been done to determine ratio of albedo to water for extraction.
- The different proportions of albedo and water used to extract pomelo peel juice that is reported to contain pectin. (Table 3)
- The ratio of albedo and water used in all five formulations are shown. The best ratio of water used is 1:1 that is Formulation 5. As compared with McWilliams (2005), the best ratio for making pectin jelly from apples is 1.92:1.
- The formulation was successful at the fifth formulation with the suitable ratio of juice, sugar and citric acid which is 3:2:1. The jelly from this formulation is semisolid and soft, but not spreading in texture, low sugar content, clear and translucent.

Characteristics of Pectin Gel

Table 4 Characteristics of pectin jelly

Characteristics	1	2	3	4	5
% Sag	14.30±0.00%	8.33±0.00%	25.00±0.00%	14.67±2.31%	3.03±0.00%
Texture	Concentrated	Weak Semisolid	Concentrated	Concentrated	Semisolid
Color	Pale Yellow	Pale Yellow	Yellow	Yellow	Yellow
Taste	Less Sweet, Bitter	Sweet, Bitter	Less Sweet, Bitter	Sweet, Bitter	Sweet, Bitter

Cont.

- **Percentage sag of all formulations have been detemined and shown in Table 4. The percentage sag is a convenient test for measuring gel strength. The larger the percentage score value indicates a very soft gel which means the stronger the jelly, the smaller percentage sag (McWilliams, 2005).**
- **In fourth formulation, which have high percentage score sag, resulted in concentrated form, it is not becomes a jelly. This is due to exceed amount of water used to extract pectin from pomelo peels. Fifth formulation has the smallest value of percentage sag, so that shows the jelly is strong.**

Proximate Analysis

Table 5 Proximate analysis of pectin jelly

Proximate Analysis	Pomelo Peel Pectin Jelly (%)
Protein	0.41±0.08
Fat	0.12±0.02
Fiber	0.46±0.47
Moisture	32.01±1.01
Ash	13.33±1.15
Carbohydrate	67.46±0.41

Cont.

- The proximate analysis is only performed on pectin jelly from fifth formulation. Analysis for protein, fat, fiber, moisture, and ash has been done according to AOAC method. The results of proximate analysis of pectin jelly are shown in Table 5.

Effect of sugar on pectin jelly

Table 6 Formulation of pectin jelly with different amount of sugar

Sample	Albedo (g)	Water (mL)	Juice extracted (mL)	Sugar content (g)	Citric acid (mL)
1	300	300	100	40	30
2	300	300	100	60	30
3	300	300	100	80	30
4	300	300	100	100	30

Cont.

- One of the important ingredients in pectin jelly is sugar. Four samples of Pomelo pectin gel were done with different amount of sugar and compared to find the best amount of sugar for obtaining good yield and quality pectin jelly. The formulation is shown in Table 6.
- Tarr (1926) reported that the jellying strength of the pectin is lessened if boiled before the addition of sugar. Yet, if the sugar is added to the pectin and acid prior to boiling, the strength of the jelly is not decreased even when boiled as long as 42.8 minutes.

Physical characteristics of pectin jelly using different amount of sugar

Table 7 Physical characteristics of pectin jelly samples with different amount of sugar

Physical Characteristics	Sample			
	1	**2**	**3**	**4**
Line Spread Test	1.1 cm	1.0 cm	1.0 cm	0.3 cm
% Sag	3.85±0.00%a	2.99±0.06 %a	7.32±0.00%a	42.51±4.53%a
Setting time (s)	12.65	10.22	10.03	2.79
Setting temperature (°C)	90.00	75.00	87.2	67.4
Yield of jelly (mL)	54	74	92	95
Texture	Semisolid	Semisolid	Weaker Semisolid	Concentrated

Sample

Sample 1

Sample 2

Sample 3

Sample 4

Cont.

- Physical characteristics of pectin jellies that contain different amount of Sugar were determined. Table 7 shows the result of physical test on pectin jelly.
- Line spread test is a measurement of viscosity of jelly. The distance of jelly flowed was read immediately after the elapsed time. The thinner the viscous liquid being tested, the higher will be the score of line spread test which means the less thick liquid will spread at longer distance compared to thick liquid.

Cont.

- Sample 3 have the shortest distance for line spread test; however the jelly results in liquid but stiff, so that it does not flow when we do the line spread test. In contrast, Sample 1 have the longest distance of line spread test because it contains less sugar that make it thin liquid and the gel setting takes longer time compared to other samples.
- Setting time and temperature of pectin jelly was decreasing with the increasing of amount of sugar. This has been reported by Lowe (1943) probably due to increased dehydration.

Cont.

- Percentage sag of pectin jelly with different amount of sugar was determined in triplicate. Sample 4 have the highest amount of percentage sag because it is concentrated liquid, so that when we do the sag test, the jelly will drop
- For Sample 3, the jelly form is semisolid, but still the jelly will drop a little bit when we do the percentage sag test because the high amount of sugar will make jelly becomes more tender.
- Generally, with increasing amount of sugar is finally reached those results in fluid syrup instead of a jelly (Lowe, 1943). Figure 2 shows the graph of the tenderness of the pectin jelly.

Cont.

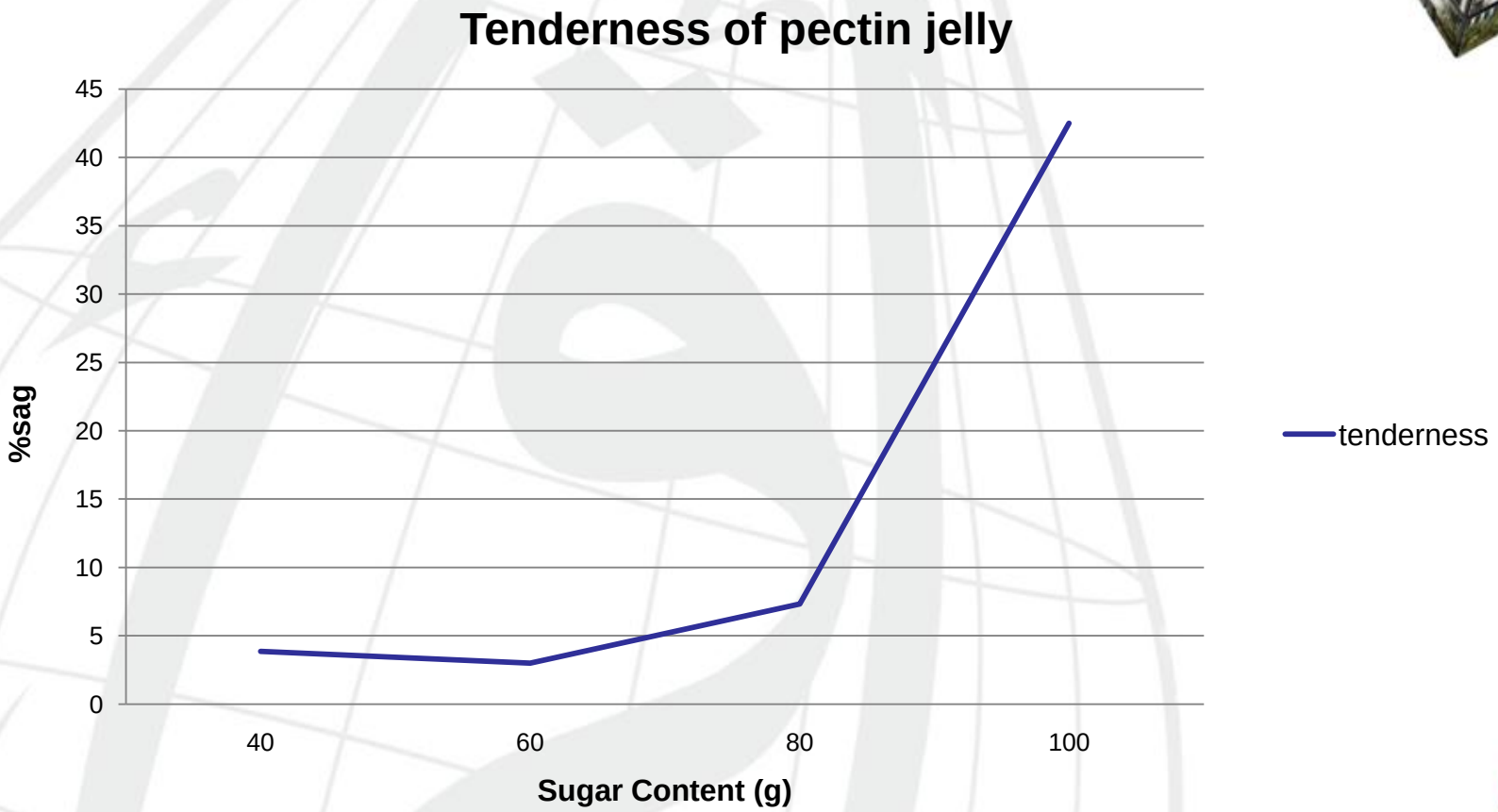

Figure 2 Graph of tenderness of pectin jelly

Colour of pectin jelly

- The color of the jelly was measured using Colorimeter. The Hunter L, a, b color scale is visually uniform. Table 8 shows the value of "L", "a", and "b".

Table 8 Colour of pectin jelly

Colour	Sample			
	1	2	3	4
L	44.57±0.77a	36.68±0.32a	38.99±0.68a	30.96±0.69a
a	-2.07±0.13a	-0.66±0.23a	-1.11±0.04a	-1.43±0.04b
b	17.64±0.76a	19.28±1.43a	16.52±0.47a	11.29±0.86b
Polar	Lighter, Greener, More Chromat	Lighter, Greener, More Chromat	Lighter, Greener, More Chromat	Lighter, Greener, More Chromat

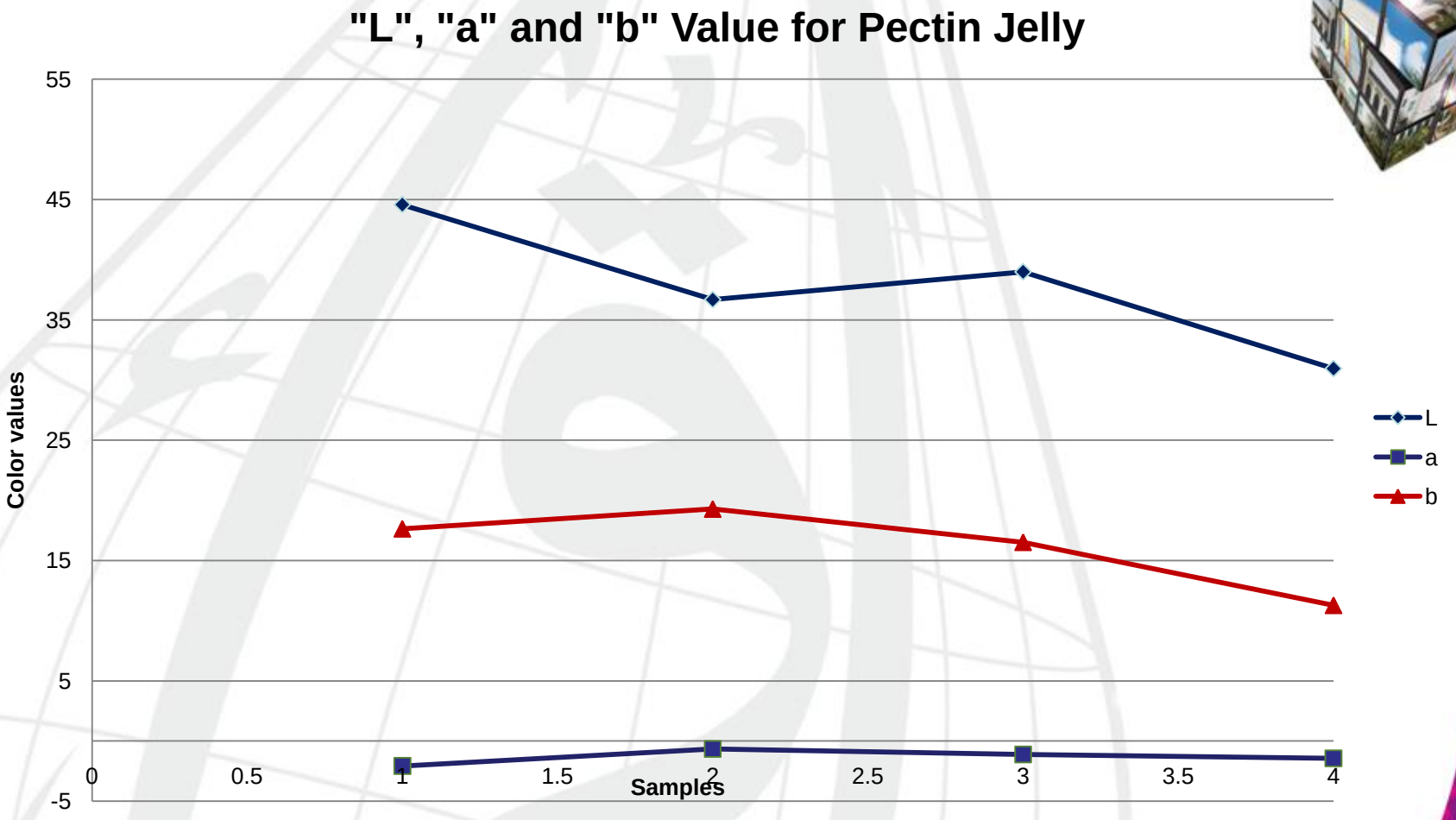

Figure 3: L", "a" and "b" values of pectin jelly

- According to table the “L” values observed in this study ranged from 30.96 to 44.57. Statistical test showed that there is significant difference between “L” color of the samples because p-value is less than α-value = 0.0.5.
- The “a” values ranged from -2.07 to -0.66. There is significant different for Sample 4.
- The “b” values ranged from 11.29 to 19.28. Sample 4 shows significant different among the samples for “a” and “b” values.

Sensory Evaluation

QDA Test QualitativebDescriptive Test

- Sensory evaluation is a technique in which numerical data are collected to establish lawful and specific relationships between product characteristics and human perception (Wang et al., 2007). Results of all sensory attributes are shown in Table 9.

Table 9 Attributes scores for pectin jelly variation

	ABC	CAB	BCA	CBA
Colour	4.9±1.91a	6.6±1.65b	7.7±1.06b	7.2±1.62b
Aroma	4.8±1.40a	5.7±1.06a	6.8±1.14a	6.5±1.58a
Tenderness	3.4±1.35a	5.2±0.92b	7.3±0.82c	8.7±0.82d
Sweetness	3.9±1.60a	4.9±1.52a	7.9±0.74b	8.6±0.70ac
Bitterness	8.6±1.07a	8.2±0.92a	4.8±0.63b	4.3±1.06ac

Cont.

- For colour intensity, BCA have the highest value. This is due to Maillard reaction while boiling sugar with juice extract. BCA also have high aroma compared to other samples.
- For tenderness attribute, CBA have the highest score because it contains lot of sugar and the jelly becomes weak and tenderer compared to others.
- The most sweetness sample is obviously CBA because of high sugar content.
- Then, ABC has highest value of bitterness due to less sugar content that make it unable to hide the bitter taste that naturally produced by pomelo peels.
- There are significance differences between samples for the sensory attributes of color, aroma, tenderness, sweetness and bitterness.
- From the results, we can conclude that the amount of sugar does affect the physical characteristics of pectin jelly.

Cont.

Hedonic Test

- Hedonic test is also known as Liking test is used to measure the level of liking for a product. A 9-point Hedonic scale ranging from dislike extremely (1) to like extremely (9) was used in evaluating the sensory attributes. Table 10 shows hedonic scores of variations.

Table 10 Hedonic scores of pectin jelly variation

Sample	Hedonic score
1	2.5±0.97a
2	6.2±2.35b
3	5.9±1.20c
4	4.9±1.10c

Cont.

- As shown in Table 10, Sample 2 has the highest score of 6.2 for hedonic test. Therefore, the most likeable pectin jelly is Sample 2. The panelists have chosen Sample 2 because of the attributes from the sample.

Alternatives of gelatin in food products

Mixed high methoxyl and low methoxyl pectin gels

- The two classes of pectin; high methoxyl (HM) and low methoxyl (LM) pectin both have their own limitation with respect to requirements for gelation.
- HM pectin is not considered a good candidate as gelatin alternative because it forms thermally irreversible gel, in addition to the street requirements of low pH and high soluble solids.
- Conversely, LM pectin appears to be more flexible in terms of manipulation of gelling conditions but a high sucrose concentrations LM too tend to pregel (May, 1990).

- The mixtures of pectins are often used in food applications to obtain products with certain functionalities. So, modulation of gel properties could be achieved perhaps through mixtures of HM and LM pectin at certain proportion, together with judicious control of $Ca^{2+,}$ sugar, pH, and types (degree of esterification) of HM pectin.

Confectionary products

- Gelled confections such as gummies, gum drops, jelly beans or Turkish delight can be manufactured using a range of gelling agents to create different textures and characteristics. It is usually made from pure gelatin gels.
- Poppe (1995) have study about improving and modifying texture of gelled confections by using pectin is believed to shorten the elastic texture of gelatin-based gummy confections; however, the resulting textures have never been adequately defined or quantified.
- To improved and modified the texture of the gelled confections, DeMars and Ziegler (2001) have studied about the texture and structure of gelatin/pectin-based gummy confections. They reported that the mixed gels were more fruity, sweet, and tart compared to pure gelatin gels.

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CONCLUSION AND RECOMENDATION

Conclusion

- This study was focused on characteristics of pectin jelly extracted from pomelo peels with the presence of water, sugar and citric acid. The effect of sugar to the characteristics of pectin jelly in terms of tenderness, color intensity, setting time, setting temperature, spreadibility and yield of the jelly were also determined.

- The best ratio of water for extraction of pectin was 1:1 and it was evident that the effect of sugar is significant on the characteristics of the final product. There are significant different between the samples in terms of tenderness (percentage sag)

- From proximate analysis result, it shows that pectin jelly contains 32.01% moisture, 13.33% ash, 0.46% fiber, 0.41% protein, 0.12% fat, and 67.46% carbohydrate.

- The ratio of pomelo peel juice to sugar to water is 3:2:1, considering all physicals, QDA and liking test. It can be stated that pomelo peel does contain pectin and it is potential to be used commercially in the food industry.

- As a conclusion, pectin jelly from this study can be developed into product such as pastilles, soft sweet jelly, thickeners, cosmetics, plant-based alternative to gelatin capsules for medicinal products.

Recommendations

Study have proved that pectin from pomelo peels can be extracted and that the pectin jelly is potential as gelatin substitutes especially for The **HALAL ISSUE** can be solved.

It is recommended that the jelly extracted from Pomelo cold be processed into powdered form for :

- easy handling
- Long shelve life.
- To study on the possibility of substituting the gelatin from pomelo peels in various product in food industry.

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