

## THE ROLE OF CAUSTIC LIME IN QUALITY IMPROVEMENT OF PALM SUGAR

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### ABSTRAK

Telah dilakukan penelitian mengenai peranan kapur tohor dalam memperbaiki mutu gula semut. Nira aren sebagai bahan dasar gula semut ditambahkan kapur tohor 0,1% dan dibiarkan selama 6 jam setelah penambahan, kemudian baru dilakukan pemasakan. Hasil penelitian menunjukkan bahwa penambahan kapur tohor dapat menahan penurunan pH dari nira. Disamping itu juga gula semut yang dihasilkan mempunyai mutu lebih baik, karena mengandung kadar air dan intensitas warna coklat yang lebih rendah jika dibandingkan dengan gula semut dari bahan dasar nira segar dan nira yang dicampurkan dengan bahan pengawet natrium benzoat.

### INTRODUCTION

If it is seen from geographic situation, where the province of West Sumatra is located on the back of Bukit Barisan and the area is relatively small, the development of agriculture is not beneficial. In order to accelerate the development, the industrial development is much more suitable for West Sumatra. Therefore the efforts in managing the industries, like agroindustry are necessary to be increased.

In West Sumatra a number of agroindustrial commodities, such as patchouly oil, cinnamon oil, tannin, palm sugar, etc play important roles in supporting the development. Palm sugar is an industrial product which produced from sap of palm (*Arenga pinmata saccharifera*). Palm is a kind of plant which is suitable to grow in tropical area such as Malaysia,

#### **Condition Test For Minimizing Intensity of Brown Colour Produced During Caramelization**

Sucrose solutions (17%) with various pHs, namely pHs 2,4,6,7 and 8 were heated varying temperatures (105,125 and 150 °C) and time (0 to 300 mins). Absorbance of solution was measured on each temperature of the experiment by using water as blank solution.

#### **The Test of Acidity Stability Level (pH) of Palm Sugar**

In this test three types of palm sap were used. They were fresh sap which was just taken from palm (A), palm sap which was mixed with 0.1% caustic lime (B) and palm sap which contained manggosteen peels. 100 ml of each palm sap (A,B and C) was placed in 250 ml beaker and the change of pH was checked in every two hours.

#### **Palm Sugar Preparation**

In palm sugar preparation the three types of palm sap were used, namely fresh sap which was just taken from palm, the sap which was mixed with 0.1% soda limes solution and the sap which was mixed with 0.1% sodium benzoate solution. The last two types of palm sap were left for 6 hours before cooked, while the first palm sap was cooked straight away after taking from palm.

The acidity level of the sap was measured with pH paper and filtered to remove the dirt. The clean sap was then put into container and heated until boiling. When the sap became viscous, the temperatur was lowered slowly. When the cooking finished the container was removed from fire and the formed sugar was crystallized by using wooden fork.

During cooking process the temperature was maintained 105-120 °C. If the temperatur is higher than 120 °C, the sugar produced is browner. Palm sugar produced was then filtered with 20 mesh filter to get uniform size.

#### **Test of Palm Sugar Colour Intensity**

Fifty grams of palm sugar was dissolved in 250 ml of water and filtered. The intensity of colour was measured with Bausch-Lamb Spectronic 20 by using water as blank solution.

#### **Determination of Sugar Content**

In determining sugar content three types of palm sugar have been analysed , namely sugar which was made of fresh palm sap, sugar which was made of

palm sap mixed soda limes solution and sugar which was made of palm sap mixed sodium benzoate solution.

100 grams of palm sugar was dissolved in methanol and applied on column chromatography of silica gel and eluted with 1% chloroform in methanol. Only sugar compounds were eluted, while the impurities which were brown coloured compounds left on the top of the column. After evaporating the solvent, the residue was redissolved in methanol and rechromatographed on column chromatography of silica gel and eluted with methanol. Every 50 ml of fraction was collected and it was separated into seven fractions. The first two fractions, namely fractions 1 and 2 and last two fractions, namely fractions 6 and 7 showed single spot on t.l.c.

Fractions 1 and 2 gave the same Rf and also did fractions 6 and 7. Fractions 3,4 and 5 showed two spots on t.l.c. with the same Rfs and all these fractions were combined and rechromatographed for separation. This separation gave two fractions and each fraction gave single spot.

The Rf of each fraction with single spot was compared with standard compounds, namely fructose, glucose, sucrose and maltose. After evaporating the solvent, each fraction was dried in desiccator for a few days and weighed.

#### **Determination of Moisture Content**

The clean crucibles were dried in oven for 1 hour at 105-110 °C and then kept in desiccator until weights were constant. 5 grams of palm sugar was placed in the crucibles and dried in oven for 8 hours at 105-110 °C and then kept in desiccator and weighted until the constant weight were obtained.

#### **Determination of Ash Content**

The clean crucibles were dried in oven for 1 hours at 105-110 °C and then kept in desiccator until the weights were constant. 5 grams of palm sugar was placed in crucibles and heated on burner until the disappearance of smoke and the heating was continued in furnace for 4 hours at 525 °C. The crucibles were then weighed until the weights were constant.

## **RESULT AND DISCUSSION**

#### **The Test of Colour Formation (Caramelization Reaction)**

It was reported that the heating of sugar solutions will produce brown coloured compound through two types of reaction, namely caramelization

and Maillard reactions (Whistler and Daniel, 1984 and Rhee and Rhee, 1981).

In order to investigate the caramelization reaction, a number of sugars, namely sucrose, glucose and fructose in various concentrations and temperatures have been heated. The experiments showed that the heating up to 300 °C for 5 hours for sucrose, glucose and fructose with concentrations 25 to 90% did not produce brown colour. However when these sugars were heated in the solid state (without water), the brown spots formed on heating at 250 °C.

This experiment showed that the the brown colour was easier to form when sugars were heated in the solid state. This can be understood that dehydration to form anhydro ring is easier obtained when sugars heated without water. This anhydro ring then forms double bonds which changes into unsaturated ring, such as furan compounds to give brown colour.

However, it has to be borne in mind that in palm sugar preparation, the sap used not only contained sugars, but also contained compounds other than sugars, such as protein, organic and inorganic salts (Miller, 1964; Abram and Romage, 1979).

Due to the sap contained compounds rather than sugars, caramelization reaction was then carried out with sugars which were dissolved in drinking water which contained inorganic salts.

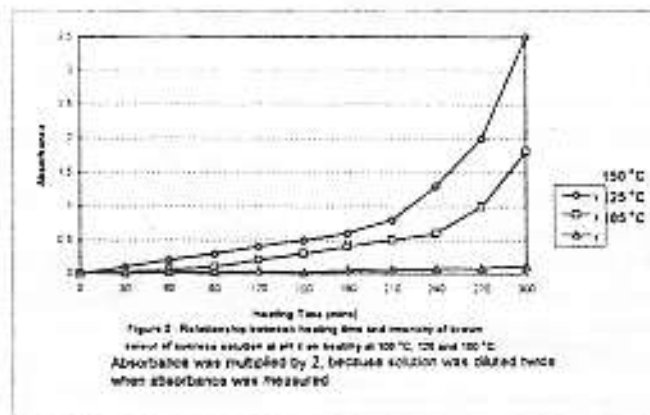
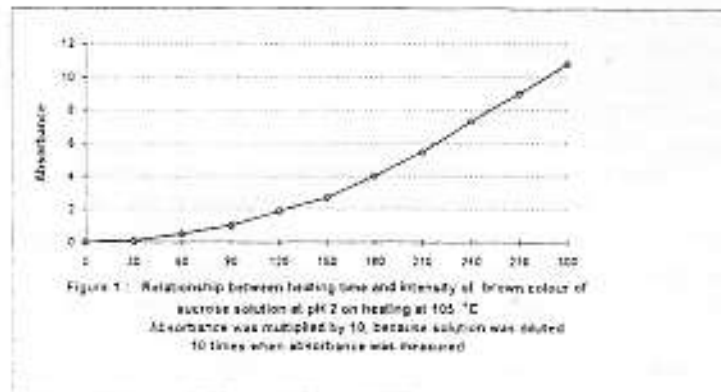
#### **Condition Test For Minimizing the Intensity Brown Colour Produced During Caramelization Reaction**

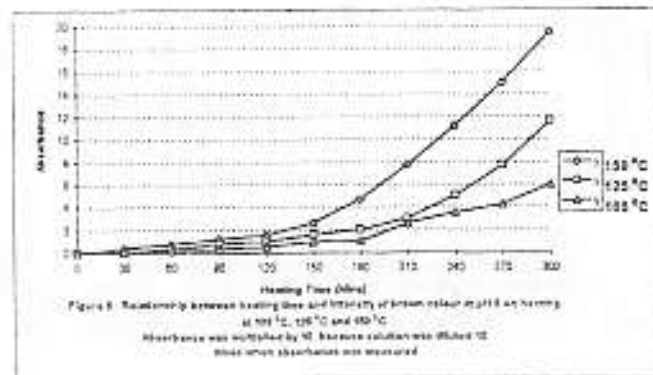
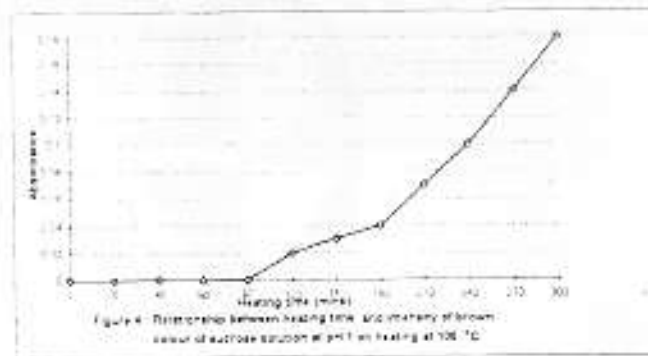
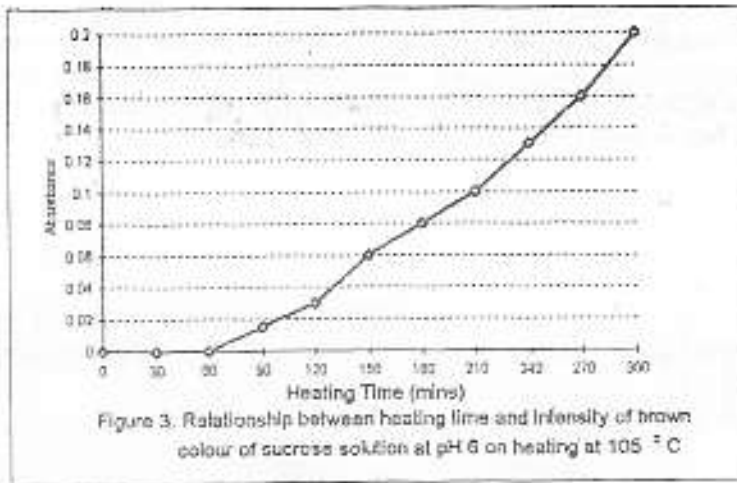
As mentioned above, the solution of sugars in double distilled water did not produce brown colour when heated at 300 °C with concentration up to 90%. Due palm sap used contained sugars and other compounds, the experiments was then carried out by heating sugar solution in drinking water which contained inorganic salts.

The experiment was carried out only for sucrose solution, because the experiment with double distilled water solutions which have been carried for sucrose, glucose and fructose gave the same results. The other reason to use sucrose solution, because palm sap contains sucrose which is much higher than other sugars. Concentration of sucrose was made 17%, because this concentration was close to concentration of sucrose in palm sap. In order to obtain condition with lowest intensity of brown colour pH of solution varied from 2 to 8.

The experiment showed that intensity of brown colour increased when temperature and heating time increased. The relationships were shown in Figures 1-5. The experiment also indicated that the formation of

brown colour was accelerated by ions which were present in water, since no brown colour was formed when sugar solutions in double distilled water were heated up to 300 °C. This result corresponds to the reports in a number of literatures explaining that caramelization reaction accelerated by acid or salts in the solution (Whistler and Daniel, 1984).





Moreover, the experiment also showed that the intensity of brown colour was affected by pH of solution. Figures 1-5 also showed the effect of pH on the intensity of brown colour. From 5 solutions examined, solutions with pHs 6 and 7 gave the lowest brown colour intensity. These data indicated that the intensity of brown colour is increased when the pH of solution is low (acid) or high (alkali).

Table 1 showed that the intensity of brown colour increased when pH of solution is lower or higher than 7. Moreover, Table 1 also indicated that the formation of brown colour is faster in solution with pH 8 than that of pH 6. This evidence corresponds to the experiment of Whistler and Daniel (1984) in which reported that the formation of brown colour at pH 8 is faster 10 times than that of at pH 6. Thus, the experiment concluded that in order to minimize the intensity of brown colour on heating process of sugar solution, the solution should be neutral or pH 7.

#### **The stability Test of Acidity Level (pH) of Palm Sap**

The acidity level (pH) of palm sap is very instable and can change easily, because of fermented process, by which sugar compounds in the sap are converted into acids and lower pH of solution. When the sap in acidic condition, reducing sugar content is increased due to the hydrolysis of sucrose into glucose and fructose or maltose into two molecules of glucose. The high content of reducing sugars in the sap will decrease the quality of palm sugar produced, because the moisture content of palm sugar will be increased due to hygroscopic behaviour of reducing sugars. The high content of reducing sugars will also increase the intensity of brown colour due to reaction of reducing sugar with protein to give brown colour compounds (Ellis, 1959).

To investigate the stability of acidity level (pH) of palm sap, three types of palm sap have been examined, namely fresh sap taken from palm, palm sap mixed with 0.1% soda limes and palm sap contained manggosteen peels. It can be seen in Table 2 that among three types of palm sap examined, palm sap mixed with soda limes solution had the most stable pH. The stability of pH is assumed due to reaction of soda limes with acids produced in the palm sap. Table 2 also showed that pH of sap mixed with manggosteen peel was more stable than that of pH of fresh sap. Palm sugar farmers always place manggosteen peel into bamboo tubes without which, the sap will become acid when the sap taken in the afternoon and can not be utilised for making palm sugar.

### Palm Sugar Preparation

The method of palm sugar preparation used by farmers is traditional method without development. The development of method is impossible to be carried out by the farmers, because most of them are uneducated. In order to improve the quality the method of preparation has to be evaluated. For this evaluation, three methods of palm sugar preparation have been examined, namely :

1. Using fresh palm sap taken from palm as raw material.
2. Using fresh palm sap taken from palm and mixed with 0,1% soda limes solution as raw material.
3. Using fresh palm sap taken from palm and mixed with 0,1% sodium benzoate solution as raw material.

Table 1 : Relationship between intensity of brown colour and pH as well as heating time on heating sugar solution (sucrose) at 105 °C

Heating Time (Mins)	ABSORBANCE				
	pH2	pH4	pH6	pH7	pH8
0	ud	ud	ud	ud	ud
30	0.1003	ud	ud	ud	0.0394
60	0.5173	0.0184	ud	ud	0.0115
90	0.9753	0.0547	0.0155	ud	0.1581
120	1.8145	0.0905	0.0324	0.0171	0.5906
150	2.5170	0.1517	0.0597	0.0289	1.2037
180	3.9980	0.1682	0.0785	0.0411	1.3041
210	5.2314	0.1768	0.1013	0.0623	2.5817
240	7.1130	0.1832	0.1316	0.0955	3.7767
270	8.8989	0.1926	0.1589	0.1386	4.1770
300	10.7611	0.2386	0.1986	0.1772	5.7814

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Table 2 : pH Changes of Palm Sap

Types of Palm Sap	pH With Time Interval (hrs)				
	0	2	4	6	8
The Fresh Sap	6.7	6.2	5.4	4.7	2.8
The Sap Mixed Soda Limes	7.0	7.0	6.8	6.5	5.8
The Sap Mixed With Mangosteen Peels	6.8	6.5	5.7	5.3	4.4

Table 3 : The quality of standard palm sugar recommended by SII-2043-87

No	Parameter	Unit	Condition
1	Form	-	Powder
2	Colour	-	Brownish Yellow
3	Taste	-	Normal
4	Sucrose	%	Minimum 80.0
5	Reducing Sugars	%	Maximum 6.0
6	Moisture Content	%	Maximum 3.0
7	Ash Content	%	Maximum 2.0
8	Undissolved Matter	%	Maximum 0.2
9	Polluted Metal	mg/kg	
	- Lead (Pb)		Maximum 1.0
	- Zinc (Zn)		Maximum 25
	-Mercury (Hg)		Maximum 0.05
	- Arsen (As)		Maximum 1.0

For making sugar from fresh palm sap the method used was the method used by the farmers. The sap was cooked soon the sap taken from palm without delaying time. Based on farmers experience, if the time is delayed, there is no good palm sugar produced, since the sap converted into acids due to the fermentation.

For making sugar from fresh palm sap mixed with caustic lime and that of mixed with sodium benzoate, the cooking was delayed for 6 hours. The experiment showed that sugar produced from the sap mixed with soda limes had good quality. The quality was better than those of the fresh sap

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and the sap mixed with sodium benzoate. This appraisal was based on chemical analysis of all sugars, namely test of brown colour intensity, sugar content, moisture content and ash content.

#### **The Test of Brown Colour Intensity**

The brown colour intensity of palm sugar will affect the buyer's demand. The darker the brown colour intensity of sugar the less the buyer's demand. In order to guarantee the quality of palm sugar distributed in the market, Indonesian government issued standard quality for palm sugar called SII 2043-87. According to this standard, the colour of palm sugar should be brownish yellow (Table 2). The result of investigation on three sugars was shown on Table 3.

This table showed that sugar produced from the sap mixed soda limes with the lowest brown sugar intensity, followed by sugar produced from the fresh sap and sugar produced from the sap mixed with sodium benzoate. The difference of colour intensity could be affected by reducing sugar content in each sugar. As described on Table 4, the highest reducing sugar content was found in sugar produced from the sap mixed with sodium benzoate, followed by sugar produced from the fresh sap and sugar produced from the sap mixed with soda limes. The higher reducing sugar content in palm sugar the higher the intensity of brown colour produced, because the brown colour was produced either by caramelization or reaction between reducing sugars and protein (Maillard, 1912). Protein which is distributed 0,20% in palm sap can react with reducing sugars to produce brown colour compounds (Rumokoi and Joseph, 1990).

#### **Determination of Sugar Content**

Sugar contents are important factor in palm sugar. Palm sugar with good quality contains high sucrose content and low reducing sugars. Glucose, fructose, lactose and maltose belong to reducing sugars. Sucrose is non reducing sugar. Palm sugar contains three reducing sugars, namely fructose (3,77%), glucose (2,50%) and maltose (3,68%) (Abram and Romage, 1979).

Sugar content was determined by column chromatography. For separating brown colour compounds, solution of palm sugar in methanol was chromatographed on silica column and eluted with 1% chloroform in methanol. The brown colour compounds left on the top of the column and sugar passed through the column were collected. The sugar solution in methanol was then chromatographed on the same column and eluted with methanol every 50 ml of fraction was collected and 7 fractions were

collected. Fractions 1 and 2 gave single spot with the same Rf on t.l.c. and identified as fructose by comparing with genuine material (co-chromatography). Both fractions were combined. After solvent evaporation, the residue was dried in desiccator and weighed.

Table 4 : Intensity of Brown Colour of Various Palm Sugars\*

No.	Sugar Produced From	Intensity of Brown Colour** (Absorbance)
1.	The Fresh Sap	1.24
2.	The Sap Mixed Soda Limes	0.79
3.	The Sap Mixed Sodium Benzoate	1.52

\* Concentration : 50 grams/250 ml water

\*\* Measured at 400 nm

Table 5 : Composition of Sugar Content

No.	Sugar Produced From	Sugar Content (%)			
		Sucrose	Maltose	Glucose	Fructose
1.	The Fresh Sap	85.30	2.80	3.40	2.50
2.	The Sap Mixed Soda	87.70	2.92	1.80	1.14
3.	Limes The Sap Mixed With Mangosteen Peels	79.17	2.63	4.45	3.66

Table 6 : Moisture Content of Various Palm Sugars

No.	Sugar Produced From	Moisture Content (%)
1.	The Fresh Sap	3.80
2.	The Sap Mixed Soda Limes	3.07
3.	The Sap Mixed With Mangosteen Peels	7.49

Table 7 : Moisture Absorption of Various Sugars

No.	Type	Moisture Absorption (%) on Various Relative Humidity and Time (20° C)		
		60 %, 1 hrs	60%,9 days	100%,25 days
1	Maltose, Hydrous	5.05	5.1	-
2	Maltose,	0.80	7.0	18.4
3	Anhydrous	0.28	0.63	73.4
4	Fructose	0.07	0.07	14.5
5	Glucose	0.54	1.20	1.4
6	Lactose, Anhydrous	5.05	5.10	-
7	Lactose, Hydrous Sucrose	0.04	0.03	18.4

Table 8 : Ash Content of Various Palm Sugars

No.	Sugar Produced From	Ash Content (%)
1.	The Fresh Sap	0.90
2.	The Sap Mixed Soda Limes	1.48
3.	The Sap Mixed With Mangosteen Peels	1.32

Each fraction 3, 4 and 5 gave two spots on t.l.c with the same R<sub>f</sub>s. All fractions were combined and rechromatographed on the same column to give 2 fractions identified as glucose and fructose based on t.l.c (co-chromatography). After solvent evaporation the residue was dried on desiccator and weighed.

Each fraction 6 and 7 gave single spot and the same R<sub>f</sub> on t.l.c and identified as sucrose by comparing with genuine material (co-chromatography). Both fractions were combined and after solvent evaporation, residue was dried in desiccator and weighed. Sugar content of each palm sugars was shown on table 5. The highest reducing sugar content was found in palm sugar produced from the sap mixed with sodium benzoate, while the lowest was in palm sugar produced from the sap mixed with soda limes.

Table 5 also showed that palm sugar produced from the sap mixed with soda limes had highest quality due to high content of sucrose and low

content of reducing sugars. High content of sucrose and low content of reducing sugars will reduce hygroscopic behaviour. Soda limes solution also reduced the decomposition of sucrose into glucose and fructose. This was indicated by higher sucrose content and lower glucose and fructose contents compared with palm sugar produced from the fresh sap.

#### **Determination of Moisture Content**

Moisture content is an important factor in palm sugar quality. High moisture content will accelerate the reaction of reducing sugar with protein to produce brown colour compounds as well as the sugar becomes soft and watery. Moisture content of three palm sugars, namely palm sugar produced from fresh sap, from sap mixed soda limes and from sap mixed with sodium benzoate have been investigated. The result of this investigation was shown in table 6. The table showed that the highest moisture content was obtained in palm sugar produced from the sap mixed with soda limes, followed by sugar produced from the fresh sap and sugar produced from the sap mixed with sodium benzoate. The different of moisture content was assumed due to the difference of reducing sugar content. As reported in literature reducing sugars are hygroscopic compounds (Browne, 1922). Table 7 showed moisture absorption of a number of reducing sugars.

#### **Determination of Ash Content**

Ash content shows metal content of a sample. The higher ash content the higher metal content in sample. Ash content is an important parameter to be detected in food, because high ash content in food will be harmful for consumer health. According to SII 2043-87, maximum ash content of palm sugar is 2%. Table 8 showed ash content of tree sugars investigated.

As shown by table 8, the highest ash content was found in sugar produced from the sap mixed with soda limes, followed by sugar produced from the sap mixed with sodium benzoate and sugar produced from the fresh sap. The high ash content of last two sugars due to the use of food additives contained metal, namely calcium for soda limes and sodium for sodium benzoate. However, ash content of three sugars was not harmful for health because it was lower than recommended by Government through SII 2043-87 (table 3).

## CONCLUSION

The experiment with a number of sugar solutions in water double distilled water (sucrose, glucose and fructose) in various concentrations showed that the brown colour was not obtained when heated up to 300 °C. However, when sugars were in solid state, brown colour was produced on heating at 250 °C. Sucrose solution produced low intensity of brown colour when heated at pH 6-7 and produced minimum intensity of brown colour at pH 7.

The experiment with tree types of palm sap, namely fresh sap, the sap mixed soda limes and the sap mixed with sodium benzoate showed that the best quality of sugar produced from the sap mixed with soda limes.

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