Effect of Maleic Acid and Glycerol Concentrations on the Characteristics of Glycerol Ester of Maleic Rosin

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Abstract

Gum rosin, distilled from the resin of pine trees (*Pinus merkusii*), is highly susceptible to degradation and oxidation. To maintain its quality, modification by fortification among other methods should be made where a stable product named glycerol ester of maleic rosin (GEMR) is produced. In the experiment reported in this paper, rosin was prepared on the laboratory scale. Fortification was performed using maleic acid of varied concentrations (8%, 10%, and 12%, w/w), followed by mixing with glycerol through an esterification process (10%, 12%, and 14%, w/w). The average yield of GEMR produced was in the range from 67.11% to 79.30%. The average softening point, acid number, and ash content were in the ranges of 91.67–120.67°C, 5.84–9.82 mg KOH/g, and 5 x 10^{-3}–14.7 x 10^{-3}%, respectively. No significant effect of concentration was observed on yield and acid number. The GEMR product was completely soluble in toluene at all concentration levels. It was found that the increase of glycerol portion affected the properties of GEMR, i.e., lower ash content and acid number, while higher softening point. Based on the acid number, solubility in toluene, and softening point values, the GEMR obtained in this research fulfilled the requirements of Chinese GEMR standards.

Keywords: gum rosin, modified rosin, fortification, esterification, acid number

Introduction

Gum rosin is a solid material produced by distilling pine oleoresin, obtained by tapping the stem of pine trees. Indonesia is the third biggest gum rosin producer in the world after Brazil and China. In Indonesia, the total production of gum rosin reached ± 66 tons/year, ± 80% of which was exported to various countries (Perhutani 2018). In practice, before shipping, degradation of gum rosin (non-modified) usually occurs during the stockpiling stage. One of the weaknesses of non-modified gum rosin is that it is prone to oxidation in an open space. Such a condition causes rosin to be susceptible to crystallizing from a solid form and to easily react with heavy metal salts, especially in varnish products (Khadijah and Chumaidi 2022). Therefore, gum rosin modification is necessary to generate a stable product. One of the methods of gum rosin modification is to mix glycerol and maleic acid to produce glycerol ester of maleic rosin (GEMR).

Maleic rosin can be used as a mixture for the wood composite industry (Rongxian and Wen 2009). It is a derivate product produced by fortification using maleic acid. Meanwhile, glycerol ester is one of rosin derivatives produced by esterification (Prasetyo *et al.* 2012). Glycerol and maleic acid serve as agents in the modification process. In a previous work, the effect of maleic acid concentrations on maleic rosin was observed (Wiyono 2007). Esterification of maleic rosin was conducted to find out the optimum cooking conditions (Prakoso *et al.* 2021). In this study, rosin modification was carried out using an esterification process, which changed the reaction between carboxylic acid and alcohol or glycerol into GEMR, releasing water (H_2O) in the process.

Materials and Methods

Oleoresin Distillation

The starting gum rosin was obtained from *Pinus merkusii* oleoresin tapping (drilling method) in a forest stand at RPH Samudra, BKPH Lumbir, KPH West Banyumas. Hydro-distillation (100 g of oleoresin) was performed on the laboratory scale with a three-necked flask (300 mL) above the heating mantle. Heating was carried out with a resin-water ratio of 1:1.5 at a temperature of 150°C for approximately 2 hours. The obtained gum rosin (control) was then tested for its physicochemical properties.

Fortification Process

Rosin (30 g) was transferred to a distillation flask and heated at an initial temperature of 150°C. The temperature was then increased to 200°C until the rosin melted. Maleic acid (technical grade) was then mixed at different concentrations based on the rosin initial weight (8%, 10%, and 12%, w/w) to obtain a dark homogenous mixture. The temperature was maintained at 200°C for approximately 1 hour. After that, maleic rosin acid or maleic rosin mixture was obtained.

Esterification Process

An esterification process was carried out by further heating the obtained maleic rosin at 280°C to produce

GEMR. Glycerol (technical grade) was added at various concentrations based on the rosin initial weight (10%, 12%, and 14%, w/w). The temperature was maintained at 280°C for approximately 2 hours. The mixture was stirred to acquire a homogeneous material. The obtained mixture was then dissolved in toluene solution to remove the remaining maleic acid. Water was added, and the mixture was transferred to a separating funnel and shaken. After that, the layer of GEMR was poured to a container gradually. The yield (%) was calculated based on the initial gum rosin weight.

Physico-chemical Properties

The softening point (ring and ball apparatus), acid number, solubility in toluene, and ash content were measured according to the SNI 7636:2020 standard. These measurements were carried out with five replications.

Data Analysis

The variation of physico-chemical properties of rosin were analyzed by two-way analysis of variance (ANOVA) with general linear model procedure followed by Tukey's (HSD) post-hoc test (p = 0.05). All statistical calculations were conducted using SPSS-Win 18.0.

Results and Discussion

Rosin Characteristics

The rosin produced by hydro-distillation method was tested according to the Indonesian National Standard (SNI 7636:2020). The results of the measurement of rosin physico-chemical properties are presented in Table 1. Color measurement was carried out visually to the limitations of testing equipment in the laboratory and by comparison to commercial gum rosin obtained from PGT Cimanggu. Based on the color observed (Fig. 1), the rosin was designated to be of Water White (WW) quality.

The softening point was determined to measure the turpentine content remaining in the rosin. The obtained softening point value (75.07°C) did not fall in the standard range of WW quality, causing the rosin to be classified as N quality rosin. Technically, the higher the softening point, the smaller the turpentine content remaining in the rosin. Meanwhile, the acid number was 190.49 mg KOH/g, which fell in the standard range (160~200 mg KOH/g).

Ash content testing was carried out to indicate the level of impurities contained in the control gum rosin. The ash content (9×10^{-3}%) was within the standard range for WW class rosin. It is expected that lower ash content would produce esters of better quality in the gum rosin (Santosa 2010). This finding was supported by the fact that the rosin tested here was completely dissolved in toluene. Technically, solubility in toluene indicates the presence of foreign matters in rosin.

Table 1. Physico-chemical properties of control gum rosin

No	Assesments	Value
1	Yield (%)	63.66
2	Solubility in toluene (1:1)	dissolved
3	Acid number (mg KOH/g)	190.49
4	Softening point (°C)	75.07
5	Ash content (%)	9×10^{-3}

Figure 1. Control gum rosin

Characteristics of Glycerol Ester of Maleic Rosin

The physico-chemical properties of the GEMR obtained are presented in Table 2. The average yield value of the GEMR was in the range from 67.11% to 79.30%. The highest yield (79.30%) was found with maleic acid and glycerol, each at a 12% concentration. However, based on the results of the analysis of variance, there was no significant effect of concentrations on the yield. This indicates that the portion range used in this experiment did not affect the yield. Therefore, it becomes necessary to explore the optimum concentrations in the next trials.

The average softening point ranged from 91.67ºC to 120.67ºC. The results of the analysis of variance showed that the interaction between the amounts of glycerol and maleic acid had a significant effect (p = 0.008). It was observed that the addition in the portion of maleic acid from 8% to 12% increased the softening point. On the contrary, the increase in the portion of glycerol until 14% decreased the softening point. The highest softening point value (120.67ºC) was obtained from the interaction between 12% glycerol and 10% maleic acid. Based on the Tukey's test results, the combination of glycerol at the smallest percentage of 10% and maleic acid at 12% gave a value of 104.33ºC, which was not significantly different from the highest value. The lowest softening point value of GEMR was found by combining 8% maleic acid and 10% glycerol. The higher the contents of fat, wax, and oil in rosin, the lower the softening point (Coppen and Hone 1995). This indicates that the gum rosin derivates still contained traces of foreign matters during the washing process.

Table 2. Average values of physico-chemical properties and solubility in toluene of glycerol ester of maleic rosin

Concentration of Glycerol (%)	Concentration of Maleic Acid (%)	Yield (%)	Softening Point (°C)	Acid Number (mgKOH/g)	Ash Content (×10^{-3} %)
10	8	67.11	91.67 [a]	8.42	8.7
	10	74.87	93 [ab]	7.71	11.3.
	12	78.27	104.33 [abc]	9.82	14.7
	Average	73.42 ± 5.72	96.33 ± 6.96	8.65 ± 1.07	11.6 ± 3
12	8	76.69	106.67 [abc]	7.95	7.7
	10	78.09	120.67 [c]	7.48	11
	12	79.3	105.33 [abc]	9.12	13.3
	Average	78.03 ± 1.31	110.89±8.49	8.18±0.84	10.7 ± 2.9
14	8	73.42	104.67 [abc]	7.71	5
	10	74.44	104 [abc]	5.84	9.5
	12	75.42	113.33 [bc]	8.88	14.3
	Average	74.43 ± 1	107.33 ± 5.21	7.48 ± 1.53	9.6 ± 4,7

Remarks: The same letters on the same column are not statistically different at P < 0.05 by Tukey's test.

The acid number indicates the amount of free fatty acids in rosin products (Wiyono 2007). Gum rosin contains 90% resin acids and 10% non-acids; carboxylic acids mostly take the form of abietic acids (Hiller *et al.* 2007; Fiebach 1993). Reducing the acid number would improve the quality of the GEMR product. Less unreacted carboxylic acids would remain, and more polyester would be formed. Therefore, the product would become more stable and not easily damaged when exposed to alkaline chemicals (Purnavita *et al.* 2017). The results of the analysis of variance showed that glycerol and maleic acid concentrations did not have any significant effects. This indicates that the ester bonds formed were not sensitive to the portion range used in this study. In other words, the smallest portions of those reagents could be used to produce GEMR.

Ash content measurement was carried out to determine the levels of foreign materials, acid insoluble materials, calcium, potassium, and magnesium salts (Mocak *et al.* 1998). Ash content indicates the amount of mineral content in a product (Waluyo *et al.* 2012). The value is important for GEMR applied to paper products. The obtained average value of ash content ranged from 5 x 10^{-3}% to 14.7 x 10^{-3}%. The results of the analysis of variance showed that the only factor that had a significant effect was the maleic acid concentration (p = 0.007). Significant differences were observed at maleic acid concentrations of 8% and 12% (Fig. 3). It is assumed that the maleic acid contained some impurities during the production of GEMR.

The solubility in toluene was measured qualitatively. The GEMR product was completely soluble in toluene at all concentrations of glycerol and maleic acid. This indicates low levels of impurities in rosin (Khadafi *et al.* 2014). Organic solvents such as ethyl ether, benzene, and ethyl alcohol are able to dissolve gum rosin (Permatasari & Rahmatullah 2018). More polar alcohol solvents would dissolve rosin faster than toluene would.

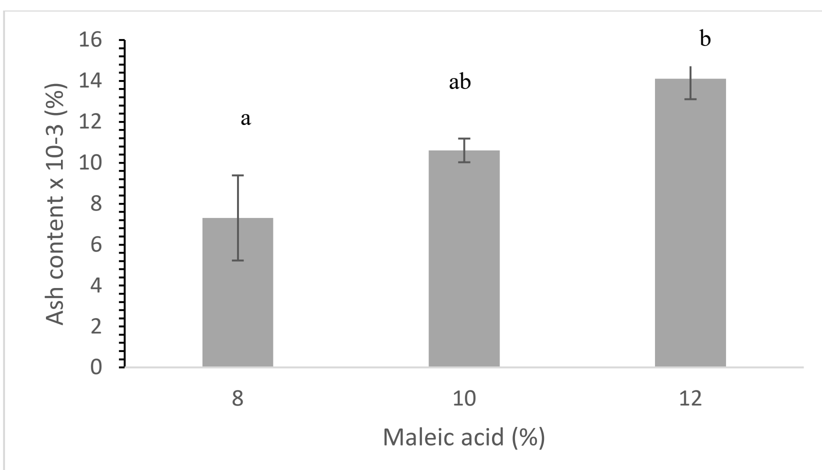

Figure 3. Effect of maleic acid percentage (based on rosin weight) on the ash content value. The same letters are not statistically different at P < 0.05 by Tukey's test.

Comparison of Glycerol Ester of Rosin Maleic with Control Rosin

Rosin derivatives were processed to overcome gum rosin weaknesses. These weaknesses could be reduced by gum rosin modification by methods such as esterification (Soliman *et al.* 2021) and fortification using maleic acid (Bildik *et al.* 2019). The comparison of statistics between the rosin starting material and the GEMR produced is presented in Table 3. There was no difference between both in the solubility in toluene. However, a considerable difference was observed in the acid number, in which case GEMR had significantly lower values (190.49 mg KOH/g vs 5.84~9.82 mg KOH/g). The esterification reaction could be evaluated by a decrease in the acid number (Rachmawati 2009). The acids in the rosin underwent a decrease in the number of H atoms due to binding to OH atoms during the esterification process (Kirk and Othmer 2004). On the other hand, the softening point of GEMR drastically increased compared to the rosin control (92~121ºC vs of 75.07ºC). This was because the GEMR had undergone a long cooking process at high temperatures. Thus, the turpentine amount diminished. A different trend was observed for the ash content. The GEMR showed slightly different values than those of the original rosin. The highest ash content level was 15 x 10^{-3}%, which indicates that the GEMR product contained small amounts of minerals. On the contrary, the lowest ash content value was 5 x 10^{-3}%, which suggests that some minerals were degraded during the heating process.

Table 3. Properties of glycerol ester of maleic rosin, control rosin, and gum rosin of SNI 7636:2020

Properties	Glycerol Ester of Rosin Maleic: Concentration of Glycerol (%)	Concentration of Maleic Acid (%): 8	10	12	Gum Rosin (Control)	SNI 7636:2020*				
Acid Number (mg KOH/g)	10	10.04	7.71	9.82						
	12	7.95	7.48	9.12	190.49	160~200				
	14	7.71	5.84	8.88						
Softening Point (ºC)	10	92	93	104						
	12	107	121	105	75.07	≥78	≥78	≥78	≥76	≥74
	14	105	104	113						
Ash Content (× 10^{-3} %)	10	8.7	11.3	14.7						
	12	7.7	11	13.3	9	≤ 20	≤ 20	≤ 40	≤ 50	≤ 80
	14	5.0	9.5	14.3						

Source: BSN (2020)

Comparison of Glycerol Ester of Rosin Maleic with Indonesian National Standard (SNI) for Gum Rosin

Color testing was carried out by comparing the color of the GEMR product with SNI requirements for gum rosin color. The dark brown color of GEMR (Fig. 4) was caused by lengthy heating during the fortification and esterification processes (Kencanawati *et al.* 2017). Higher maleic anhydride proportions yield a darker color (Prakoso *et al.* 2021). Thus, bleaching should be performed during the GEMR processing to obtain a clear color. With regard to the ash content, all treatments yielded GEMR products in the gum rosin range set out under the SNI (Table 3), but the softening point of the GEMR products was higher than required by the SNI. GEMR has a long carbon chain due to excessive heating during the fortification and esterification processes. The number of double bonds and the length of the carbon chain in the compounds would affect the softening point value (Ramadhiani *et al.* 2020). Furthermore, the acid number of GEMR were below the range prescribed by the SNI due to the esterification process. For food industry purposes, it is necessary to maintain the lower values of acid number (Hidayat *et al.* 2021).

Figure 4. Glycerol ester of rosin maleic product

Comparison Glycerol Ester of Rosin Maleic with Chinese derivative gum rosin

As China has been known as the world's biggest producer of gum rosin, the research results were further compared to Chinese standards (Table 4). An acid number test was carried out to determine the quality of the gum rosin derivative product. The esterification process primarily aims to reduce the acid number or increase the rosin ester conversion using heterogeneous catalysts (Mardiah *et al.* 2023). In a previous study, Mahendra (2019) produced glycerol ester of rosin with acid number in the range from 80 to 90 mg KOH/g. The average acid number obtained in this research was in the range 5.84~9.82 mg KOH/g, lower than the value range of the four types of Chinese quality standards (GER-100M, GER-120M, GER-130M, and GER-140M).

The softening point value indicates the ripeness of the gum rosin derivative product. In this research, the softening point values of GEMR did not meet the Chinese standards in all treatments. The treatment with 14% glycerol and 8% maleic acid met the Chinese GER-100M standard, and the treatment with 12% glycerol and 10% maleic acid met the Chinese GER-120M standard. Furthermore, the solubility in toluene results indicated no undissolved materials in this experiment.

Table 4. Properties of glycerol ester of maleic rosin from the experiment and Chinese standard

Properties	Concentration of Glycerol (%)	Concentration of Maleic Acid (%)			Chinese glycerol ester of maleic rosin			
		8%	10%	12%	GER-100M	GER-120M	GER-130M	GER-140M
Acid number (mg KOH/g)	10%	8.42	7.71	9.82	≤ 25	≤30	≤ 30	≤ 42
	12%	7.95	7.48	9.12				
	14%	7.71	5.84	8.88				
Softening Point (°C)	10%	92	93	104	102~108	120~126	130~136	135~145
	12%	107	121	105				
	14%	105	104	113				

Source: Wuzhou (2005)

Conclusions

From the results of this research, it can be concluded that esterification with mixes of maleic acid (at concentrations of 8%, 10%, and 12%) and glycerol (at concentrations of 10%, 12%, and 14%) had significant effects on ash content and softening points. The increase in the percentage of glycerol would result in decreases in ash content and acid number and increases in softening point (from 10% to 12% glycerol concentration). The increase in maleic acid concentration would significantly increase the ash content. No significant effect of concentrations was observed on the yield and acid number. Compared to the SNI standard for gum rosin, the GEMR produced here had lower acid number and higher softening point. It also had better quality in acid number compared to Chinese GEMR standards. Processing pine resin with the addition of maleic acid at 6% and glycerol at 10%, or with the addition of maleic acid at 6% and glycerol at 12%, fulfilled the requirement of Chinese GEMR standards for softening point.

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