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Preparation of Novolak Type Resin by Liquefaction of Palm Oil Empty Fruit Bunch (EFB) Using Sulphuric Acid as a Catalyst

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ABSTRACT

The liquefaction of palm oil empty fruit bunch (EFB) in phenol was carried out in the presence of sulphuric acid as a catalyst in the reflux-condenser system. The effects of the reaction temperature and sulphuric acid concentration on the liquefaction reaction, physical, and mechanical properties of phenolated EFB (PEFB) resin were studied. The reaction yield increased as the reaction temperature and catalyst concentration increased from 110ºC to 150ºC and 3% to 8%, respectively. However, no significant difference in results was observed between samples reacting at 130ºC and 150ºC using 5% and 8% catalyst. The thermal gravimetric analysis tests have indicated that the thermal stability of PEFB increases by increasing the catalyst concentration. However, the results do not show any important variation in the thermal stability by increasing the liquefaction temperature. From studies of differential scanning calorimetry it was found that the curing enthalpy of PEFB with hexamethylenetetramine increases by increasing the liquefaction temperature.

Key Words:

liquefaction;
palm oil empty fruit bunch;
novolak resin;
molecular weight;
thermogravimetric analysis.

INTRODUCTION

It is not an exaggeration to say that wood and agriculture products are some of the most important products of nature. In recent years, there has been a dramatic increase in the consumption of natural resources in a general sense, the growth of population as well as the high consumption of agricultural and wood products. Consequently, the amount of wood and lignocellulosic wastes generated is alarmingly high. The municipal waste stream largely consists of wood-based wastes and other lignocellulosic followed by plastics. Meanwhile, a huge amount of lignocelluloses wastes is produced

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every year from the wood-related industry, forest, and agricultural application, etc. However, large quantities of lignocellulosics such as woody wastes from building materials, sawdust, waste papers, and barks are still incinerated or discarded in the environment [1]. On the other hand, new landfill sites and petroleum resources become scarce and costly, especially in the industrialized countries. Simultaneously, the threat to environment caused by a huge amount of accumulating debris grows seriously [2]. Therefore, recycling of the municipal wastes, as an important alternative, has nowadays gained importance in handling the problem. In other words, recycling is the most suitable form of disposal in the long run, beneficial not only ecologically but also economically and environmentally.

Lignocellulosic materials are natural composites. These materials depend on species, consisting of approximately 40-50% cellulose, 20-30% hemicellulose and 20-30% lignin. Cellulose has a similar structure in all the lignocellulosic materials, but the lignin and hemicellulose exist in various structures. Cellulose is a thermoset linear high-molecular weight polymer, which is exclusively built up of β-D glucose. The glucose molecule has three hydroxyl groups, and therefore it tends to form significant number of hydrogen bonds. Lignin has major phenolic groups and along with hemicellulose are thermoplastics [3].

In Malaysia, palm oil empty fruit bunches (EFB) are the major byproducts of the palm oil industry and one of the major lignocelluloses wastes. They are produced after being milled at a palm oil mill. Every ton of fresh fruit bunches produce approximately 0.22 tons of EFBs after being milled. Total amount of EFBs are approximately 10 million tones per year [4]. To date, this large amount of EFBs has not been fully utilized commercially.

Our previous results showed that novolak type resin can be successfully prepared by liquefaction of EFBs in phenol using acid as a catalyst. It was also found that, sulphuric acid is the best catalyst for liquefaction of EFBs [3,5]. However, no attempt has been made on the effects of reaction temperature and catalyst concentration on the reaction yield and the physical and mechanical properties of PEFB. Therefore, the aim of this study is to investigate the effect of reaction temperature and catalyst concentrations on the liquefaction reaction of EFB and the physical and mechanical properties of PEFB.

EXPERIMENTAL

Materials

EFB was used as a raw material obtained from Szetech Engineering Sdn. Bhd, Malaysia. The air-dry EFB was ground to 60-70 wire mesh by a ball mill. Then, it was dried at 105ºC for 8 h in an air-circulated oven. Phenol and sulphuric acid (97%) were used as a liquefaction reagent and catalyst, respectively. Hexamethylenetetramine, HMT (curing agent), zinc stearate (lubricating agent and starter) and calcium hydroxide (accelerating agent) were used as moulding components. All chemicals, of extra pure grade were supplied by Hamburg Merck chemical and used without further purification. Commercial novolak resin (Sumbi-PR-F-63) which was used as a reference was obtained from PT. Indoperin Jaya. Indonesia.

Liquefaction and Sample Preparation

EFB, phenol (1:3 ratio) and sulphuric acid as a catalyst (3-8% of phenol weight) were charged into a four-neck glass reaction flask (500 mL) equipped with a reflux condenser, thermometer and an electrical stirrer. The liquefaction reaction was carried out in an electrical heating mantle at various temperatures. The main product of this reaction is a phenolated EFB (PEFB).

After completion of each phenolation process, the resulting mixture was diluted with methanol and then filtered with a glass-fibre filter (3.1 μm particle-retainable) to separate the residue of biomass (methanol-insoluble fraction) from methanol-soluble fraction. The resulting residue was oven dried and weighed, and its amount was recorded in weight percentages. The sulphuric acid remaining in the methanol-soluble fraction was neutralized with magnesium oxide (MgO) and the neutralized solution was once again filtered with a glass-fibre filter to remove the salt produced by neutralization. Subsequently, methanol was evaporated from the solution at 80°C under vacuum and the free phenol (unreacted phenol) was distilled under a reduced pressure at 190°C to obtain condensed phenolated EFB (PEFB) in a solid

form.

The methanol-insoluble fraction, residue (R), and the amount of reaction yield (Y) were calculated by the following equations:

$$R(\%) = (W_R/W_E) \times 100 \quad (1)$$

$$Y(\%) = [W_{PEFB}/(W_{P0}+W_E)] \times 100 \quad (2)$$

where, W_E is the weight of oven-dried initial input EFB used (g) and W_R is the oven-dried weight of the solid methanol-insoluble fraction (g), W_{PEFB} is the weight of phenolated EFB (g) obtained after removing the free phenol from methanol soluble fraction, and W_{P0} is the weight of initial input phenol (g).

In thermal gravimetric (TGA) and differential scanning calorimetric (DSC) analysis tests, CPEFB, which is the mixture of PEFB and cross-linking agent (hexa-methylenetetramine) has been used to investigate the cross-linking reaction and its effect on the thermal properties.

Gel Permeation Chromatography

The PEFBs molecular weights and their distributions were determined using a Tosoh GPC HLC-8020 gel permeation chromatography (GPC) equipped with TSK-gel columns Nos: GRC X LH, GMH X L, and GMH X L. The column temperature was 35ºC and the oven temperature was 40ºC. Tetrahydrofuran (THF) was used as a mobile phase. The molecular weights of the samples were calibrated based on the monodisperse polystyrene standards. The samples dissolved in THF in a concentration of 0.01(g/mL) were injected in the amount of 150 μL and eluted at a flow rate of 1.0 mL/min.

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) and differential thermogravimetric (DTG) analysis tests were carried out using a Mettler Toledo SDTA 851e thermal analyzer, at heating rate of 10ºC /min in a temperature range of 30-600ºC in a static atmosphere. The weights of the samples were between 7-11 mg.

Differential Scanning Calorimetry

DSC measurements were analyzed on a Mettler Toledo thermal analyzer model DSC 882. Around 2 mg of samples was placed in aluminium cups that were subsequently crimped hermetically. The sample and reference, which consisted of an empty aluminium cup similarly crimped, were heated to 350ºC. The DSC curves were obtained as heat flow versus temperature plots by heating from room temperature to 350ºC at a rate of 10ºC/min under N_2 atmosphere.

Compounding and Hot Press Moulding

The PEFB resin was dissolved in acetone and mixed with HMT (25% of the resin weight), zinc stearate (2.5% of the resin weight), calcium hydroxide (6% of the resin weight), and 50% of EFB fibre. After mixing, the mixture was oven dried at 70ºC for 2 h to remove acetone, and then ground in an electrical grinder to make a fine powder.

The obtained powder was compression moulded into sheets in a die with a dimension of 130×130×3.4 mm. The moulding conditions were as follows: temperature: 185±5ºC; pressure: 100 kg/cm^2; preheating time: 20 s; venting time: 20 s; curing time: 330 s and cooling under a slight pressure to ambient temperature. Then, this moulding board was cut to the test specimen size.

Moulding Shrinkage

Moulding shrinkage is the difference of the dimensions of a cold die and a cold moulded sample [6]. It was calculated according to the following equation:

$$\text{Moulding shrinkage } (\%) = [(L_0-L_1)/L_1] \times 100 \quad (3)$$

Where, L_0 and L_1 were the lengths of cold die and cold moulded samples (mm) respectively.

RESULTS AND DISCUSSION

According to the results obtained and presented in Table 1, the effect of liquefaction temperature on the solid residue and reaction yield as a function of catalyst (sulphuric acid) percentage is evident when the liquefaction time was 60 min. This table indicates that generally the solid residue decreases with the increase of the liquefaction temperature at various concentrations of sulphuric acid. The data in Table 1 illustrate that the residue decreases around 17% by increasing

Table 1. Effect of liquefaction temperature on the solid residue and reaction yield.

Liquefaction temperature (ºC)	Catalyst (%)	Residue (%)	Yield (%)
110	3	55 ± 0.8	18.2 ± 0.1
	5	37 ± 0.8	31.5 ± 0.2
	8	25.8 ± 0.7	43.1 ± 0.2
130	3	38.3 ± 0.8	30 ± 0.2
	5	15.5 ± 0.7	42.5 ± 0.2
	8	11 ± 0.7	48.3 ± 0.2
150	3	20.8 ± 0.7	32.2 ± 0.2
	5	5.2 ± 0.7	46.1 ± 0.2
	8	3.8 ± 0.7	48.9 ± 0.2

Note: other liquefaction condition: time 60 min, phenol/EFB = 3

Table 2. Effect of liquefaction temperature on the solid residue and reaction yield.

Liquefaction temperature (ºC)	Catalyst (%)	Residue (%)	Yield (%)
110	3	53.1 ± 0.8	20.7 ± 0.1
	5	28 ± 0.8	37.6 ± 0.2
	8	20.9 ± 0.7	45.4 ± 0.2
130	3	35.9 ± 0.8	31.3± 0.2
	5	7.8 ± 0.7	45.8± 0.2
	8	7.5 ± 0.7	48.6± 0.2
150	3	17 ± 0.7	33.1± 0.2
	5	3.8 ± 0.7	47.4± 0.2
	8	3.2 ± 0.7	50± 0.2

Note: other liquefaction condition: time 90 min, phenol/EFB = 3

the reaction temperature from 110ºC to 130ºC, using 3% catalyst. The residue decreases the same (17%) when the reaction temperature increases further from 130ºC to 150ºC. This indicates that the effect of increasing temperature either from 110ºC to 130ºC or from 130ºC to 150ºC using 3% catalyst is similar on solid residue. Table 1 also shows that the residue decreases around 21% and 15% by increasing the reaction temperature from 110ºC to 130ºC in reactions with 5% and 8% of catalyst, respectively. The residue decreases around 10% and 7% when the reaction temperature increases from 130ºC to 150ºC. This reveals that the effect of above mentioned temperature raise on solid residue becomes insignificant using higher catalyst concentration (5% and 8%). Table 1 also indicates that the reaction yield increases by around 12%, 11%, and 5% by increasing the reaction temperature from 110ºC to 130ºC using 3%, 5%, and 8% of catalyst. While it increases by around 2%, 3%, and 1% when the temperature raises from 130ºC to 150ºC using 3%, 5%, and 8% catalyst, respectively.

Table 2 shows the effect of the liquefaction temperature on the solid residue and reaction yield as a function of catalyst percentage when the liquefaction time is 90 min. Table 2 also shows similar phenomenon as Table 1. The solid residue decreases and reaction yield increases, with the increase of the liquefaction temperature at various concentrations of sulphuric acid. Table 2 indicates that the reaction yield reaches around 10%, 8%, and 4% by increasing the reaction temperature from 110ºC to 130ºC using 3%, 5%, and 8% catalyst. While the reaction yield increases by around 2%, 1.5%, and 1% when the temperature is raised from 130ºC to 150ºC using 3%, 5%, and 8% catalyst, respectively. Therefore, it can be explained that the effect of former increase in temperature on the reaction yield is stronger than that of the latter.

Table 2 also clearly shows that there is not much difference in the amount of solid residue for the liquefaction with 5% and 8% catalyst when liquefaction temperature is 130ºC or 150ºC. Solid residue decreases less than 1% in both temperatures when the catalyst moves up from 5% to 8%. Considering the amount of residue and reaction yield, it can be said that the temperature between 130ºC and 150ºC is the optimum temperature for this process.

Table 3 shows the effect of sulphuric acid concentration on the percentage of residue and reaction yield at 130ºC as a function of reaction time. The data in Table 3 illustrates that the reaction residue decreases with increasing the concentration of catalyst from 3% to 8% in all reaction time lengths. The decrease in the rate of reaction residue diminishes significantly when the catalyst concentration increases above 5%. The rate of raising amount of reaction yield follows a similar trend as well. This is due to the utilized optimum

Table 3. Effect of catalyst concenteration on the solid residue and reaction yield.

Catalyst (%)	Liquefaction time (min)	Residue (%)	Yield (%)
3	30	40.2 ± 0.8	27.5 ± 0.1
	60	38.3 ± 0.8	30 ± 0.2
	90	35.9 ± 0.8	31.3 ± 0.2
	120	35.4 ± 0.8	32.4 ± 0.2
5	30	33 ± 0.8	35.8 ± 0.1
	60	15.5 ± 0.7	42.5 ± 0.2
	90	7.8 ± 0.7	45.8 ± 0.2
	120	6.4 ± 0.7	48.6 ± 0.2
8	30	23 ± 0.7	41.7 ± 0.2
	60	11 ± 0.7	48.3 ± 0.2
	90	7.5 ± 0.7	48.6 ± 0.2
	120	5.8 ± 0.7	49.2 ± 0.2

Note: other liquefaction condition: temperature 130ºC, phenol/EFB=3

amount of catalyst which results in the reduction of reaction activation energy [7].

Considering the corrosive effect of sulphuric acid on the reactor, it can be concluded from the results that the sufficient amounts of reaction yield as well as a minimum amount of residue are required targets. Hence, as this study shows, 5% concentration of sulphuric acid seems to be the optimum level for the liquefaction process.

Table 4 shows the effect of reaction temperature on the weight-average molecular weight ($\overline{M}_w$) and the number-average molecular weight ($\overline{M}_n$) of the phenolated EFB (PEFB) as well as their distribution or dispersity ($\overline{M}_w/\overline{M}_n$). Through the liquefaction of wood in the presence of phenol, large molecular weight components of wood (cellulose, hemicellulose, and lignin) are known to be reduced to small molecular masses [8].

As shown in Table 4, the M_w and $\overline{M}_w/\overline{M}_n$ of phenolated EFB samples increase when the reaction temperature increases. The molecular weight ($\overline{M}_w$) of PEFB increases rapidly as the reaction temperature increases to 130ºC. After that, the molecular weight increases slightly by increase in reaction temperature from 130ºC to 150ºC.

Alma et al. [9] reported that the reaction parameters such as phenol/wood ratio and catalyst concentration should be taken into account as the effective parameters on the molecular weight and its distribution. Their report says that changes in the molecular weight depend upon the amounts of combined phenol which are not significant at the different phenol/wood ratio levels. The $\overline{M}_w$ and $\overline{M}_w/\overline{M}_n$ of phenolated wood samples increase obviously with increase in acid catalyst concentration. The phenol acts as a protective agent for recondensation during liquefaction.

As can be seen in the Table 4, the $\overline{M}_w/\overline{M}_n$ values obtained for the phenolated EFB are higher than $\overline{M}_w/\overline{M}_n$ values of commercial novolak resin (reported specifications). The possible explanation for this observation might be that the EFB is composed of three different main components. Therefore, the dissolved products after liquefaction have rather broad molecular weight distribution in comparison with that of the commercial novolak resin.

Figure 1 shows the general DSC thermograph of the mixture of phenolated EFB and HMT, which is known as cured PEFB (CPEFB). The PEFB was obtained at different liquefaction temperatures; sample CPEFB1 at 110ºC, sample CPEFB2 at 130ºC, and sample CPEFB3 at 150ºC as are shown in Figure 1. It can be seen in Figure 1 that the first peak (as a shoulder, no dip) is an endothermic peak which appears at

Table 4. Effect of reaction temperature on the average molecular weights.

Reaction temperature (ºC)	Catalyst (%)	$\overline{M}_w \times 10^{-3}$	$\overline{M}_n \times 10^{-2}$	$\overline{M}_w/\overline{M}_n$
Novolak	-	1.850	9.120	2.03
110	5	1.594	3.629	4.38
130	5	2.111	3.904	5.41
150	5	2.204	3.746	5.88

Note: other liquefaction condition: time 60 min, phenol/EFB = 3

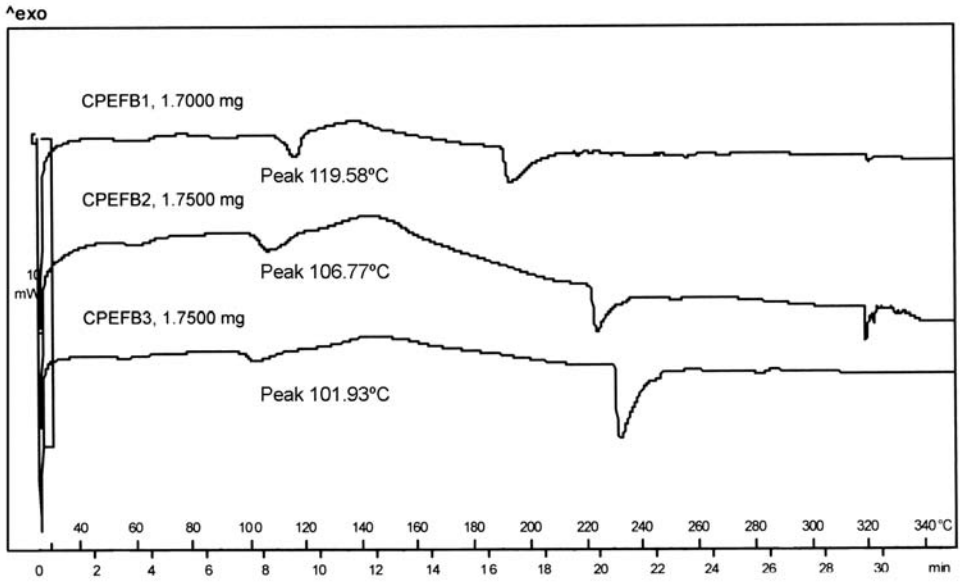

Figure 1. DSC Thermograph of CPEFB which obtained at different liquefaction temperature, CPEFB1 at 110ºC, CPEFB2 at 130ºC, and CPEFB3 at 150ºC.

a temperature around 60ºC for all samples. This peak is attributed to the glass transition temperatures of samples before any cross-linking occurs [10].

The second peak indicates the endothermic phenomenon. This peak is due to the evaporation of moisture from CPEFB and also melting of PEFB.

The next peak is an exothermic peak which is observed around 140ºC. This is due to the effect of HMT on the phenolated EFB. Therefore, this exothermic peak is the result of curing or cross-linking reaction of PEFB with HMT. The curing time is reported as the time between the starting point of this peak and its maximum point. The curing reaction enthalpy can also be calculated by the area of this peak.

The effects of liquefaction (phenolation) temperature of EFB on the curing behaviour (melting peak temperature (T_m), exothermic curing peak temperature (T_p), the curing reaction enthalpy (ΔH) and curing time (Δt) of phenolated EFB) are observed in Table 5 which were obtained from DSC thermograph.

The results indicate that the increase of liquefaction temperature from 110ºC to 150ºC leads to a decrease in melting temperature by 17.5ºC, an increase in the peak temperature by 6.8ºC, and an increase in the curing enthalpy by 69.8 J/g as well as 0.8 min decrease in the curing time. It was found that the curing enthalpy of phenolated wood is increased with increasing the combined phenol [9]. The com-

Table 5. Effects of liquefaction temperature and catalyst in the curing behaviour of PEFB using 25% of HMT.

T_l (ºC)	Catalyst (%)	T_m (ºC)	T_p (ºC)	ΔH (J/g)	Δt (min)
110	5	119.5	137.62	61.2	1.6
130	5	106.7	143.8	114.1	2.3
150	5	101.9	144.4	131.4	2.4
130	8	104.2	144.0	112.58	3.2

T_l = liquefaction temperature, T_m = melting point, T_p = curing peak temperature, ΔH = curing reaction enthalpy, Δt = curing time

Table 6. Thermal weight losses of PEFB and CPEFB as a function of liquefaction temperatures.

Reaction temperature (ºC)	HMT (%)	Weight loss at first stage (%)	Weight loss at 2nd stage (%)	Weight loss at 3rd stage (%)
110	0	8	53	57
130	0	8	53	57
150	0	7	53	58
110	25	25	35	40
130	25	24	36	41
150	25	25	34	40

Liquefaction condition: time (60 min), catalyst (5%), and phenol/EEB = 3

bined phenol is increased by increasing the liquefaction temperature [11]. The DSC results clearly show that the variation in T_l, T_m, T_p, ΔH, and Δt is higher when the temperature increases from 110ºC to 130ºC in comparison with increase of temperature from 130ºC to 150ºC.

The result also indicates that when the catalyst of liquefaction reaction increases from 5% to 8%, among other parameters the curing time varies while the variation of other parameters is not so significant. The results in Table 5 also indicate when the concentration of catalyst at liquefaction reaction increases from 5% to 8%, among the curing thermodynamic parameters (T_l, T_m, T_p, ΔH, and Δt) while the variation of other parameters is negligible, the curing time (Δt) varies more than other parameters. A possible explanation for this phenomenon might be referred to the curing time of phenolic resin with HMT which extends in the acidic condition.

Alma et al. [9] reported that the curing enthalpy, curing peak temperature, and curing time of phenolated wood with sufficient amount of combined phenol is 128 J/g, 167.2 ºC, 1.99 min, respectively. This is, in fact, close to the result of commercial novolak resin. As Table 5 shows, the result of PEFB which was phenolated at 130ºC and 150ºC are similar to the results of phenolated wood as mentioned above.

Table 6 summarizes the weight losses of PEFB and CPEFB as a function of liquefaction temperature at several degradation temperature ranges: the first stage is a temperature range below 200ºC, the second stage is a temperature range between 200ºC and 500ºC, and the third stage is a temperature range between 500ºC and 600ºC. As shown in Table 6, the weight loss of PEFB and CPEFB at temperature below 200ºC is about 8% and 25%, respectively. This result indicates that the weight loss of CPEFB is approximately 17% higher than the weight loss of PEFB at first stage (temperature below 200ºC). Fedtke [12] found that during the cross-linking reaction of novolak with HMT, a considerable amount of gas consisting of ammonia is produced. Therefore, this extra weight loss might be due to the cross-linking reaction (a reaction between HMT and PEFB) and also to the degradation of unreacted HMT. The weight losses of CPEFB at second and third stage are clearly lower than the PEFB which reveals an efficient cross-linking reaction [10]. This result also indicates that the thermal stability of PEFB increases due to cross-linking reaction. The results also denote that the weight losses of PEFB and CPEFB at all three stages of degradation are almost equal. This reveals that the liquefaction temperature does not affect the thermal stability of PEFB and CPEFB.

Table 7 illustrates the weight losses of PEFB as a function of catalyst concentration in liquefaction

Table 7. Thermal weight losses of PEFB as a function of catalyst concentration in liquefaction reactions.

Catalyst (%)	Weight loss at first stage (%)	Weight loss at 2nd stage (%)	Weight loss at 3rd stage (%)
3	4	60	66
5	7	56	58
8	9	39	44

Liquefaction condition: time (90 min), phenol/EFB (3), and temperature (130ºC)

Table 8. Effect of liquefaction reaction parameters on the shirinkage of board.

Phenol/EFB	Reaction time (min)	Temperature (ºC)	Catalyst (%)	Shrinkage (%)
Novolak	-	-	-	0.22
3	90	110	5	0.35
3	90	130	5	0.36
3	90	150	5	0.38
3	90	130	3	0.34
3	90	130	5	0.36
3	90	130	8	0.33
3	90	130	8	0.40
2	90	130	5	0.20
3	90	130	5	0.36
4	90	130	5	0.52

reaction. The results indicate that the weight loss at the first stage increases when the catalyst concentration goes up. The weight loss at the first stage is due to the removal of free phenol and the moisture content. Thus the amount of free phenol and the moisture content of samples that were prepared at lower catalyst concentration are lower compared with samples prepared at higher catalyst concentration.

It may be seen from the data in Table 7 that the amounts of weight loss at the second and third stages decrease as the catalyst concentration is increased. The results indicate that the thermal stability of PEFB increases by increasing the catalyst concentration. This finding is consistent with the findings of Alma and Kelley [10]. They found that the thermal stability of cured phenolated chestnut bark increases by the increase of catalyst concentration. This might be due to a higher cross-linking ratio when a higher catalyst concentration is used.

Table 8 shows the effect of liquefaction condition on the shrinkage of the PEFB-base moulding boards. Data in Table 8 indicate that the degree of shrinkage in these samples is roughly similar when the phenol/EFB ratio is 3. This amount is slightly higher than the commercial novolak resin. Table 8 also indicates that the increase in the phenol/EFB ratio increases the amount of shrinkage. This finding might be explained by reduction of $\overline{M}_w$ of PEFB due to the increase of phenol/EFB ratio. Lin [13] and Zhang et al. [14] have found that the increasing of the phenol/wood ratio decreases the $\overline{M}_w$ of phenolated wood.

CONCLUSION

Using sulphuric acid as the catalyst, the results of this investigation showed that in general, the liquefaction yield increases and the EFB residue decreases, by increasing the reaction temperature and catalyst concentration. The examinations of the physical properties of PEFB showed that the $\overline{M}_w$ and $\overline{M}_w/\overline{M}_n$ of PEFB samples increase along with increasing of the liquefaction temperature. The molecular weight ($\overline{M}_w$) of PEFB increases rapidly as the reaction temperature increases up to 130ºC and after that the molecular weight slightly increases by increasing the reaction temperature. The TGA studies showed that the thermal stability of the PEFB increases by increasing the catalyst concentration. It was also shown that the effect of liquefaction temperature and catalyst concentration on the variation of shrinkage in the PEFB-base board is not significant.

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