# Flow Properties of Novolak-Type Resin Made from Liquefaction of Oil Palm Empty Fruit Bunch (EFB) Fibres Using Sulfuric Acid as a Catalyst

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Novolak-type resins were produced via liquefaction of oil palm empty fruit bunch (EFB) fibres with various liquefaction and resinification parameters such as temperature, time, catalyst concentration, and phenol-to-EFB ratios, and investigated in terms of flow properties (melt viscosity and melt flow rate) of the phenolated EFB (PEFB) and resinified PEFB (RPEFB). The results showed that the phenol/EFB ratio was the most effectual parameter in the variation of melt viscosity and melt flow rate (MFR) of PEFB. The melt flow rate of the PEFB was tremendously lower than that of the commercial novolak resin. The results showed that the activation energy for flow, which was obtained using the Arrhenius equation, increased when the liquefaction temperature increased. The results also demonstrated that the molten PEFB showed a shear thinning or pseudo-plastic behavior.

*Keywords: Empty fruit bunch; Liquefaction; Flow properties; Novolak resin*

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## INTRODUCTION

EFB (oil palm empty fruit bunch) is one of the major lignocelluloses waste products in Malaysia. In fact, EFB is one of the major byproducts from the oil palm industry. In 2011, it was estimated that 70 to 80 million tonnes of oil palm biomass were generated from this industry (MPOB 2011). To date, this large amount of EFB has not been fully utilized commercially.

Numerous studies have attempted to produce products using EFB fiber. Such products have included reducing sugars (Chin *et al.* 2013), composite boards (Chai *et al.* 2009), activated carbon (Alam *et al.* 2009), bio-oil (Fan *et al.* 2011) and bio-absorbent (Sajab *et al.* 2013). In addition, the EFB fiber is also used in the production of phenolic resins through the liquefaction process. Liquefaction is a thermochemical process in which biomass or wood reacts with an organic liquid with or without acid at a medium temperature range of 100 to 250 ºC. Through this process, wood or biomass will be de-polymerized into a low molecular weight product and can be used in a variety of polymeric materials depending on the specific active groups (Pan 2011).

Various types of materials have been successfully liquefied in the liquefaction process, such as wood (Pu and Shiraishi 1993; Lin *et al.* 1994), bark (Yuan *et al.* 2009; Zhao *et al.* 2010), and lignocellulosic material (Yamada and Ono 1999; Alma and Basturk 2006; Bakarudin *et al.* 2012). Recently, the preparation of novolak-type resin by liquefaction of EFB in the presence of phenol and resinification of phenolated EFB mixture has been carried out (Ahmadzadeh *et al.* 2008, 2009). The results showed that the majority of EFB can be successfully converted to phenolic resin with this method.

However, assessment of the physical and mechanical properties of the phenolic resin produced via this method and comparison with commercial novolak resin is necessary.

Viscosity is perhaps the most important property of flowing fluids. The viscosity of a liquid is proportional to the resistance exhibited by its molecules to align with the flow and to deform under shear stress. Flow properties of plastics and resin are one of the most important properties for molding and its processes. However, no attempt has been made so far to investigate the effect of reaction parameters on the flow properties (melt viscosity and melt flow rate) of this type of phenolic resin. Therefore, this work aimed to study the effect of various reaction parameters on the flow properties of phenolated EFB (PEFB).

## EXPERIMENTAL

### Materials

EFB was used as a raw material and obtained from Szetech Engineering. The air-dry EFB was ground to 60 to 70 wire mesh by using a ball mill. Then, it was dried at 105 °C for 8 h in an air-circulated oven. Phenol and sulfuric acid 97% were used as a liquefaction reagent and catalyst, respectively. Hexamethylenetetramine (HMTA) (curing agent), zinc stearate (lubricating agent and starter), and calcium hydroxide (accelerating agent) were used as molding components. All of the chemicals were extra pure grade and were used without further purification. Commercial novolak resin (Sumbi-PR-F-63) obtained from Indopherin Jaya (Indonesia) was used as a comparison to the lab novolak resin produced.

### Liquefaction, Resinification, and Sample Preparation

The liquefaction process was started by charging EFB, phenol (1: 3 ratio), and sulfuric acid as a catalyst (3%, 5%, and 8% base on the phenol) 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 of 110 ºC, 130 ºC, and 150 ºC. The main product produced upon completion of this reaction was phenolated EFB (PEFB).

After the liquefaction process, the temperature of the phenolated mixture was allowed to decrease to 100 ºC. Then, 37% formaldehyde solution was reacted with the phenolated mixture at different weight ratios, which are 55%, 70%, and 85%. The resinification reaction was carried out for 80 min at 105 ºC with constant stirring. The product produced in this process was called resinified phenolated EFB (RPEFB).

After the completion of each phenolation or resinification process, the resulting mixture was diluted by the addition of methanol and then filtered with a glass-fiber filter to separate the liquefaction residue (methanol-insoluble part) from the methanol-soluble part. The sulfuric acid remaining in the methanol-soluble part was neutralized with magnesium oxide (MgO), and the filtration process was once again repeated on the neutralized solution in order to remove the salt produced from neutralization. Subsequently, the methanol was evaporated from the solution at 80 °C under vacuum, and the free phenol (unreacted phenol) was distilled under reduced pressure at 190 °C to get the condensed PEFB and RPEFB into a solid form. Then these solid products were ground in mortar for further analysis or application.

### Gel Permeation Chromatography (GPC)

The PEFBs molecular weights and their distributions of were determined using a gel permeation chromatography (GPC) Tosoh. GPC.HLC-8020 device equipped with TSK – gel columns NO: GRC X LH, GMH X L and GMH X L. The column temperature was 35 ºC and the oven temperature was 40 ºC. Tetrahydrofuran (THF) was used as a mobile phase. The molecular weights of the samples were calibrated based on the monodisperse polystyrene standards. The sample 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.

### High Performance Liquid Chromatography (HPLC)

The amount of free phenol in soluble part was measured using high performance liquid chromatography (HPLC) (Walter 1515 isocratic HPLC pump) equipped with a Walter 2487 Dual λ Absorbance detector and Walter $C_{18}$ 5 μm symmetry column ( 4.6 mm diameter x 150 mm length). The chromatographic zones were visualized under UV light (254 nm). Tests were performed at a flow rate of 1 mL/min using a methanol /water solution (1/2 v/v) as the mobile phase. In addition, the solution of pure phenol at different concentration at methanol was used as a standard solution for calibration. Phenolated EFB samples were dissolved in methanol at a suitable concentration and were injected into the HPLC apparatus in the amount of 10 μL. Later, the amount of free phenol using of calibration curve was calculated.

### Measurement of Flow Properties (Viscosity)

The melt viscosity and rheological properties of phenolated EFB were measured by a capillary rheometer (Shimadzu model CFT-500D). The measured diameter and length of die used were 1 mm and 2 mm, respectively. The apparent melt viscosity and shear rate were measured at constant temperature (175 ºC) under shear stress ranging from $1.839 \times 10^5$ to $7.355 \times 10^5$ (Pa) after a preheating time of 10 s. The apparent melt-viscosity of the sample was automatically calculated as a ratio of shear stress to shear rate. The melt flow rate also was automatically calculated by CFT.

## RESULTS AND DISCUSSION

### Effect of Reaction Temperature on the Average Molecular Weight

Table 1 shows the average mass 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 ($\overline{M}_w / \overline{M}_n$). 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. A possible explanation for this 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. In addition, the $\overline{M}_w$ and $\overline{M}_w / \overline{M}_n$ of phenolated EFB samples increased when the reaction temperature increased. The molecular weight of phenolated EFB increased rapidly as the reaction temperature increased up to 130 ºC. After that, the molecular weight slightly increased when the reaction temperature increased from 130 ºC to 150 ºC. A previous report shows that the reaction parameters such as phenol/wood ratio and catalyst concentration should be taken into account as the effective parameter on the molecular weight and its distribution. Moreover, the changes in the molecular weight depend upon the amounts of combined phenol which are not significant at the different phenol/wood ratio levels (Alma *et al.* 1995).

**Table 1.** Effect of Reaction Temperature on the Average Molecular Weight

| 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 |

Other liquefaction conditions: time: 60 min, EFB/phenol ratio: 1/3

## Effect of Phenol/EFB (P/E) Ratio on the Liquefaction Process

Table 2 shows the effect of an initial input of phenol/EFB (P/E) ratio in the liquefaction reaction on the melt viscosity (η) and melt flow rate (MFR) of phenolated EFB (PEFB). These results explicitly show that the melt viscosity of PEFB increased when the ratio of phenol to EFB decreased. The fluidity of the PEFB becomes difficult as the viscosity increases, and the MFR value also decreases by increasing the melt viscosity, which is shown in Table 1. The MFR value decreased when the melt viscosity increased; this was due to the increase of the P/E ratio and resulted in a decrease in the average molecular weight ($\overline{M}_w$) of the PEFB. A possible explanation for this might be that the effect of the P/E ratio on the liquefaction of EFB was similar to the effect of the phenol/formaldehyde (P/F) ratio in the production of the novolak resin. In the production of the novolak resin, as the ratio P/F increased, the average molecular weight ($\overline{M}_w$) of the resin decreased. This was due to the reduction of active $CH_2$ groups from formaldehyde. Meanwhile, in the liquefaction of EFB, the role of liquefied EFB is similar to the role of active $CH_2$ groups in the production of novolak resin. This explanation was also confirmed when the liquefaction of cellulose or other biomass was carried out, where the increase of the phenol/biomass ratio can significantly restrain the re-condensation reaction by blocking the reactive sites on the liquefied component, consequently preventing a further increase in molecular weight (Lin 1996; Lin *et al.* 1997; Zhang *et al.* 2005). Furthermore, when the P/E ratio increased to 4, the melt viscosity value of the PEFB was almost the same as the melt viscosity of the commercial novolak resin.

**Table 2.** Effect of Initial Input P/E ratio on the Melt Viscosity and Melt Flow Rate of PEFB

| Sample | P/E | R.t (min) | R.T (°C) | Catalyst (%) | *η* (Pa.S) | MFR(g/10 min) |
|---|---|---|---|---|---|---|
| Novolak | — | — | — | — | 41 | 344 |
| 1 | 4 | 30 | 130 | 5 | 53 | 205 |
| 2 | 4 | 90 | 130 | 5 | 62 | 194 |
| 3 | 3 | 90 | 130 | 5 | 465 | 34 |
| 4 | 1 | 90 | 130 | 5 | 9300 | 1.16 |

P/E: phenol/ EFB; R. t: reaction time; R.T: reaction temperature; *η*: viscosity; MFR: melt flow rate

## Effect of Liquefaction Reaction Parameters

Table 3 shows the effect of liquefaction reaction parameters (such as temperature, time, and catalyst concentration) on the melt viscosity and melt flow rate of PEFB. These results indicated that the melt viscosity and melt flow rate were changed by varying the reaction parameters. It must be noted that the differences were not significant. Many parameters such as molecular weight, molecular weight distribution and polymer structure, the amount of free phenol, and moisture content (Alma 1996; Masica 1982) affect the melt viscosity of the phenolic resin. Therefore, it is difficult to explain the reason for this variation at this stage without having sufficient information about the structure of the phenolated EFB. However, the results indicated that the viscosity of the PEFB was higher than for the commercial novolak. Comparison of the data in Table 3 with the data in Table 2 shows that the initial input of the phenol/EFB ratio in the liquefaction reaction will affect the melt viscosity ($\eta$) and MFR. This comparison clearly indicates that the phenol/ EFB ratio has a great effect on the melt viscosity of PEFB.

**Table 3.** Effect of the Liquefaction Reaction Parameter on the Melt Viscosity and Melt Flow Rate of PEFB

| Sample | P/E | R.t (min) | R.T ($^oC$) | Catalyst (%) | $\eta$ (Pa.S) | MFR(g/10 min) | F.P (%) |
|---|---|---|---|---|---|---|---|
| 1 | 3 | 60 | 110 | 5 | 440 | 25 | 5.5 |
| 2 | 3 | 60 | 150 | 5 | 420 | 26 | 5.2 |
| 3 | 3 | 90 | 150 | 5 | 287 | 38 | 5.8 |
| 4 | 3 | 90 | 150 | 8 | 240 | 45 | 4.5 |
| 5 | 3 | 90 | 130 | 8 | 520 | 21 | 5.3 |
| 6 | 3 | 90 | 130 | 5 | 465 | 34 | 6.5 |
| 7 | 3 | 30 | 130 | 5 | 431 | 25 | 5.8 |
| 8 | 3 | 120 | 130 | 5 | 602 | 18 | 5.6 |
| Novolak | — | — | — | — | 41 | 344 | 1.4 |

P/E: phenol/EFB; R.t: reaction time; R.T: reaction temperature; $\eta$: viscosity; MFR: melt flow rate; F.P: free phenol

## Effect of the Formaldehyde/PEFB (F.P) Ratio on the Resinification Process

Table 4 shows the effect of resinification (reaction between formaldehyde, phenol, and polyphenol after liquefaction) on the melt viscosity and melt flow rate of PEFB. The results showed that when the amount of formaldehyde added to the free phenol (F/P) was 55% and 70%, the viscosity of resinified PEFB (RPEFB) was lower than the viscosity of the PEFB. However, the viscosity of RPEFB was higher than the viscosity of PEFB when the ratio F/P was 85%. A possible explanation is that the decreased in the PEFB viscosity might be due to the fact that 63% of the aqueous formaldehyde is made up of water. Water from aqueous formaldehyde act as a solvent to the PEFB, hence decreasing the viscosity of the PEFB in the early stage of reaction. During the polymerization process between PEFB and formaldehyde, it is expected that only a small amount of phenols or polyphenols react with formaldehyde due to insufficient amount of formaldehyde in the reaction mixture. This can be seen in Table 3, when the amount increased as the viscosity of RPEFB increased. This could be due to the liquefied component from the EFB fibres and also the changes in the degree of polymerization. It is known that in the production of novolak resin, the molecular weight and viscosity of resin are increased by increasing the F/P ratio (Brydson 1975). Increasing the F/P ratio resulted in the increase of the viscosity for each resin produced because the extra amount of formaldehyde tended to speed up the degree of polymerization or the curing rate. In addition, increasing the F/P ratio to more than 70% resulted in a high molecular weight resin being produced, or in other words one with a high melt viscosity. This can also be seen when the viscosity of RPEFB 3 was higher than the viscosity of PEFB.

**Table 4.** Effect of Resinification on the Melt Viscosity and Melt Flow Rate of PEFB

| Sample | F/P | $\eta$ (Pa.S) | MFR(g/10 min) |
|---|---|---|---|
| PEFB | 0 | 465 | 34.00 |
| RPEFB1 | 55 | 123 | 87.80 |
| RPEFB2 | 70 | 250 | 43.18 |
| RPEFB3 | 85 | 876 | 12.35 |

F/P: formaldehyde/phenol; $\eta$: viscosity; MFR: melt flow rate

### Plots of ln *η vs.* $T^{-1}$ for Phenolated EFB Samples

Figure 1 shows the plots of ln $\eta$ *vs.* $T^{-1}$ obtained under a constant shear stress ($1.839 \times 10^5$ Pa). Under the given shear stress, the apparent viscosities of the molten phenolated EFB decreased by increasing the temperature. The relationship between ln $\eta$ and $1/T$ is linear, though the data is somewhat scattered. It means that the dependence of $\eta$ on temperature roughly obeys the Arrhenius equation,

$$\eta = A \exp(-E_a / RT) \tag{1}$$

where $A$ is a constant which is related to material properties, $E_a$ is the activation energy for thermal flow (kJ/mol), $R$ is the universal gas constant (8.314 kJ/mol K), and $T$ is the absolute temperature (K).

The relationship between the values of the activation energy of the phenolated EFB (calculated based on Fig. 1 and Eq. 1) and the reaction yield and liquefaction temperature can be observed in Table 5. It is evident that the activation energy to flow the phenolated EFB increased as the reaction temperature increased. This seems to be in agreement with other researchers who found that the activation energy for flow increases when the combined phenol increases (Alma 1996; Lin *et al.* 1994). This result may be explained by the fact that increasing the reaction time or temperature leads not only to the increase in the reaction yield but also changes in the structures of the liquefied material (Lin *et al.* 2004; Zhang *et al.* 2005). The ortho-substitution phenol band in the liquefied product increases with increasing liquefaction temperature which leads to a high sterical advantage (Zhang *et al.* 2005). Furthermore, it can be suggested that an increase in liquefaction temperature leads to a pronounced increase in steric hindrance of molecular motions. Alma (1996) and Lin *et al.* (1994) reported that the above phenomena can be explained by the increase of combined phenol of phenolated wood due to the increase in liquefaction temperature. The increase of the combined phenol will increase the steric hindrance which creates difficulties for molecular motion. Difficulties in the molecular motion resulted in the increase of activation energy for flow.

**Table 5.** Relationship of the Activation Energy of the PEFB to the Reaction Yield and Liquefaction Temperature

| R.T (ºC) | Equation | $R^2$ | $E_a$ (kJ) | Reaction Yield (%) |
|---|---|---|---|---|
| 110 | $\ln(\eta) = 45762T^{-1} - 112.47$ | 0.9883 | 380.465 | 31.5 |
| 130 | $\ln(\eta) = 56891T^{-1} - 138.10$ | 0.9774 | 472.992 | 42.5 |
| 150 | $\ln(\eta) = 106369T^{-1} - 266.70$ | 0.9735 | 884.342 | 47.1 |

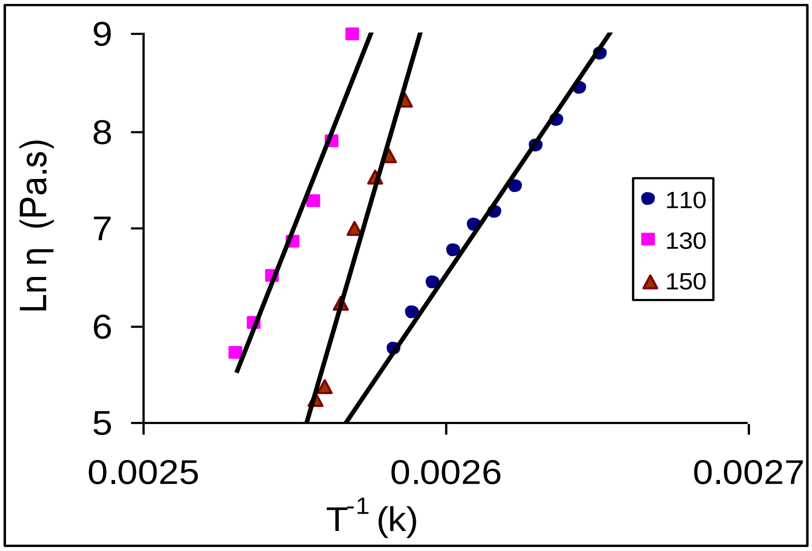

**Fig. 1.** Plots of ln *η* *vs.* $T^{-1}$ for phenolated EFB samples obtained at various liquefaction temperatures under a shear stress of 1.839 × $10^5$ Pa

## Plots of ln *η* for the Molten PEFB as a Function of Shear Rate (*γ*)

The viscosity ($\eta$) of the molten phenolated EFB as a function of shear rate ($\gamma$) at a constant temperature (110 ºC) and shear stress ($\tau$ =1.839 × $10^5$ Pa) for various liquefaction conditions are shown in Fig. 2 (liquefaction condition, considering the sample number, reported in Table 1 and Table 2).

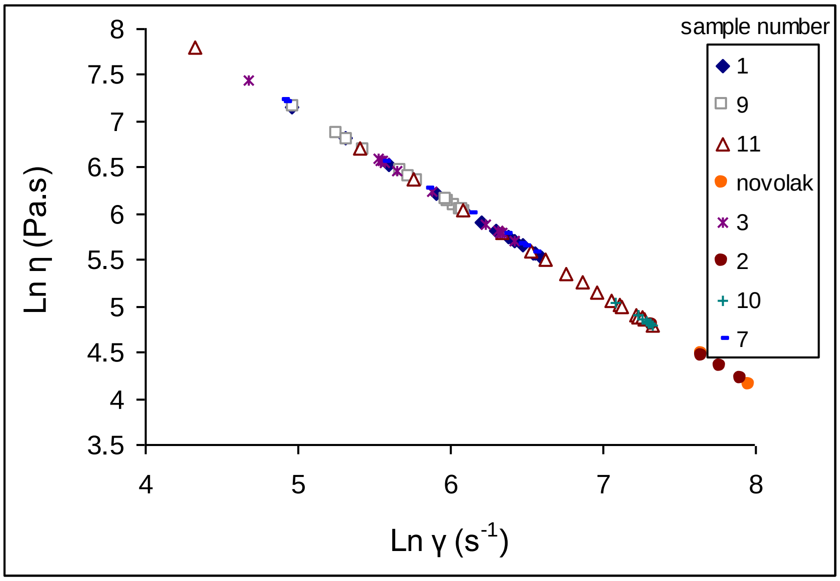

**Fig. 2.** Plots of ln *η* for the molten PEFB as a function of shear rate (γ) at 110 °C and shear stress of 1.839 × $10^5$ Pa

It can be seen from Fig. 2 that ln *η* decreased linearly along with increasing ln *γ*. This indicates that the dependence of *η* on *γ* obeyed the power law equation. These results also demonstrate that the molten phenolated EFB showed a shear thinning or pseudo-plastic behavior.

## The Apparent Flow Curves, Shear Stress (*τ*) *vs*. Shear Rate (*γ*), and Viscosity (*η*) *vs*. Shear Rate (*γ*)

The apparent flow curves, shear stress (*τ*) *vs*. shear rate (*γ*), and viscosity (*η*) *vs*. shear rate (*γ*) for neat phenolated EFB at constant temperature (110 ºC) are shown in Fig. 3. The curves show that the flow of molten phenolated EFB is shear thinning and obeys the Ostwald de Waele power law:

$$\tau = k\,\gamma^{n} \tag{2}$$

$$\eta = k\gamma^{n-1} \tag{3}$$

In this law, *η* is the apparent viscosity, *γ* is the shear rate, *τ* is the shear stress, *k* is the viscosity coefficient, and *n* is the power law exponent. The value of the power law exponent (*n*) is evaluated by regression analyses of the data. The obtained equations from the plot in Fig. 3 are:

$$\tau = 14351\,\gamma^{0.561} \tag{4}$$

$$\eta = 13109\gamma^{-0.4253} \tag{5}$$

These equations indicate that the amount of *n* for the sample is less than 1, which reveals that the phenolated EFB melts have a pseudoplastic nature.

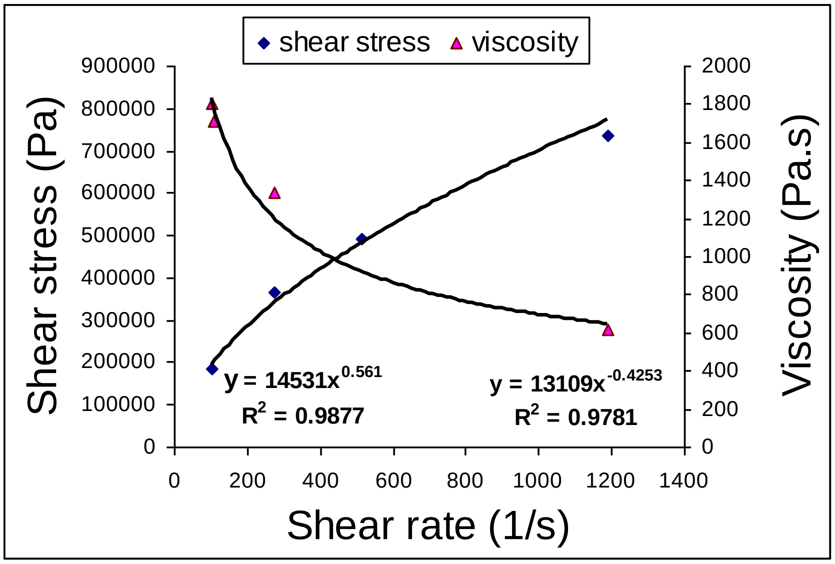

**Fig. 3.** The apparent flow curves, shear stress (*τ*) *vs*. shear rate (*γ*), and viscosity (*η*) *vs*. shear rate (*γ*) for PEFB at constant temperature (110 °C)

## CONCLUSIONS

1. The results showed that the phenol/EFB ratio is the most effectual parameter on the variations of melt viscosity and melt flow rate (MFR) of PEFB.
2. Viscosity of PEFB decreases as formaldehyde is introduced in the resinification, which may be due to the presence of large amounts of water from formaldehyde, but when formaldehyde ratio increases, viscosity of the RPEFB also increases due to the change in the degree of polymerization.
3. The appearance melt viscosity of PEFB was higher than for the commercial novolak resin, but the appearance melt viscosity ($\eta$) of PEFB was close to the appearance melt viscosity ($\eta$) of commercial novolak resin when the phenol/ EFB ratio was 4.
4. The rheological study showed that molten PEFB has a pseudoplastic nature and obeys the power law equation. It was also found that the dependence of $\eta$ on temperature roughly obeys the Arrhenius equation.

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