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Effect of Phenolic Resin on Density, Porosity, Hardness, Thermal Stability, and Friction Performance as A Binder in Non-Asbestos Organic Brake Pad

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Abstract. Phenolic resin is the most common resin binder used in brake pad materials. Selection of the relative amount of phenolic resin is an important to maintain structurally intact with the other ingredients of brake pad composite. In this experimental study, the effects of phenolic resin as a binder in non-asbestos organic brake pad was investigated. The brake pad composite was made using a powder metallurgy technique. Four formulations with different volume fractions 10, 15, 20, and 25 were selected. Parameters applied were 47 MPa compaction pressure, 150 °C molding temperature, and 130 °C post curing temperature with 6 hours holding time. The properties of brake pad composite were examined density, porosity, hardness, thermal stability, and friction performance (refers to SNI 09-0143-1987). The results show the increase of phenolic resin content can decrease the density, porosity, thermal stability, friction coefficient, and volume wear rate by 1%, 16.79%, 2.39%, 42.5%, 24.16%, respectively. However, the hardness increases by 19.29 %. This investigation also recommends not to select 10 and 15 volume fraction of phenolic resin due to lower resistance to mechanical stresses although the thermal stability is better than the other studied composites.

Keyword : phenolic resin, density, porosity, hardness, thermal stability, friction performance

1. Introduction

Phenolic resin have some properties such as excellent thermal behavior, high strength level, long thermal and mechanical stability, and excellent cost performance characteristic. They are widely used in various application related to production of friction composite, brake pads, and clutch lining for the automotive industry and for brake elements in rail vehicles and machine [1]. Even today, phenolic resin still represent the most important binders in commercial brake pad composite because they offer resistance to temperature of 250 °C, good wetting capability, low cost, and good combination of thermal, mechanical, and tribology properties [2]. However, phenolic resin has limitations, such as high hardness which made the brake pad brittle, sensitive to humidity, and very low impact resistance [3]. Selection of the relative amount of phenolic resin in brake pad formulation as a binder is an important issue to maintain structurally intact with the other ingredients at various temperature, thus the brake pad composite is able to withstand under mechanical and thermal stresses during braking operation.



Furthermore, phenolic resins are also contribute to the friction performance. Jang *et al* [4] reported that the large of quantity phenolic resin was exhibited unstable friction coefficient as a result of the poor thermal stability phenolic resin. Kim *et al* [5] stated that increasing amount of phenolic resin with potassium titanate and CNSL were increase the hardness and the coefficient of friction at moderate temperature of about 100°C. While, increasing in amount of CNSL modified resin were decrease the friction coefficient, however the mechanical properties and wear were improve [6].

Recently, a lot papers reported the exploration of resin modified as a binder to improved mechanical properties and friction performance in fade and recovery condition [3,6–8]. However, a few modified resin are used in the commercial brake pad because of the cost factor that is not competitive [8], thus the commercial brake pad still used straight phenolic resin as a binder. Therefore, selection of the right amount of phenolic resin content is required. Phenolic resin will be effect to thermal stability of brake pad, ability to maintain mechanical properties, and ability to bind ingredients under braking condition from crumbling apart with other ingredients. In this study, the brake pad composites with a different of the volume fraction of phenolic resin were prepared. The effect of phenolic resin on density, porosity, hardness, thermal stability, and friction performance as a binder in non-asbestos organic brake pad were investigated.

2. Materials and Methods

The composition used in this study consisted of 10 ingredients, of which eight ingredients were kept constant (59%) and two ingredients, namely phenolic resin and BaSO₄ (41%) were varied in the volume fraction. All ingredients of the brake pad composite in the form of powder. Phenolic resin with the particle size of +270 mesh were used as a binder, and BaSO₄ with the particle size of 230 mesh used as an inert filler. The ingredients investigated in the formulation of brake pad composite as shown in Table 1.

Table 1. Ingredients of brake pad composites was used in this study

Sample	Phenolic resin (%)	BaSO ₄ (%)	Friction Dust (%)	Al ₂ O ₃ (%)	SiO ₂ (%)	MgO (%)	Copper (%)	MoS ₂ (%)	Graphite (%)	hBN (%)
PR-10	10	31	15	2	4	3	8	8	13	6
PR-15	15	26	15	2	4	3	8	8	13	6
PR-20	20	21	15	2	4	3	8	8	13	6
PR-25	25	16	15	2	4	3	8	8	13	6

Four different formulations, namely PR-10, PR-15, PR-20, and PR-25 were manufactured with powder metallurgy technique. The powder metallurgy stage consist of the following processes, namely, weighing, dry mixing, hot pressing, post-curing, and finishing. An analytical balance (Sartorius L 1201S) was used to weigh the ingredients sample. These ingredients were mixed in Rotation Drive STR4 for 60 minutes at 10 rpm in room temperature. Dry mixing was done to ensure the macroscopic homogeneity. Then, the powder mixture were hot pressed 47 MPa at a constant temperature of 150 °C for 15 minutes to obtained green body with the dimension of 25 mm x 25 mm x 7 mm. In the beginning of hot pressing process, the intermittent pressure was released 3-4 times to release the gas. Green body was post-cured in a furnace at a constant temperature of 130°C for 6 hours. The surface of the samples were then polished with the sand paper to obtain the smooth surface and desired thickness accordance with the dimensional of standard testing. The properties of the composite samples were examined density, porosity, hardness, thermal stability, and friction performance. The schematic of manufacturing brake pad composite was shown in Figure 1.

Characterization of the brake pad composite

Density test of composite sample was performed after post-curing process. A density of composite sample was measured using Archimedes' principle in accordance with the ASTM C-20, where the

difference between the sample weight in air (w_{air}) and its weight in a fluid (w_{fluid}), divided by the density of the fluid (ρ_{fluid}). The volume of the liquid displaced, which was identical to the volume of sample [9]. The density of composite sample can be calculated using the formula (1).

$$\rho = \frac{w_{air}}{w_{air} - w_{fluida}} \times \rho_{fluida} \quad (1)$$

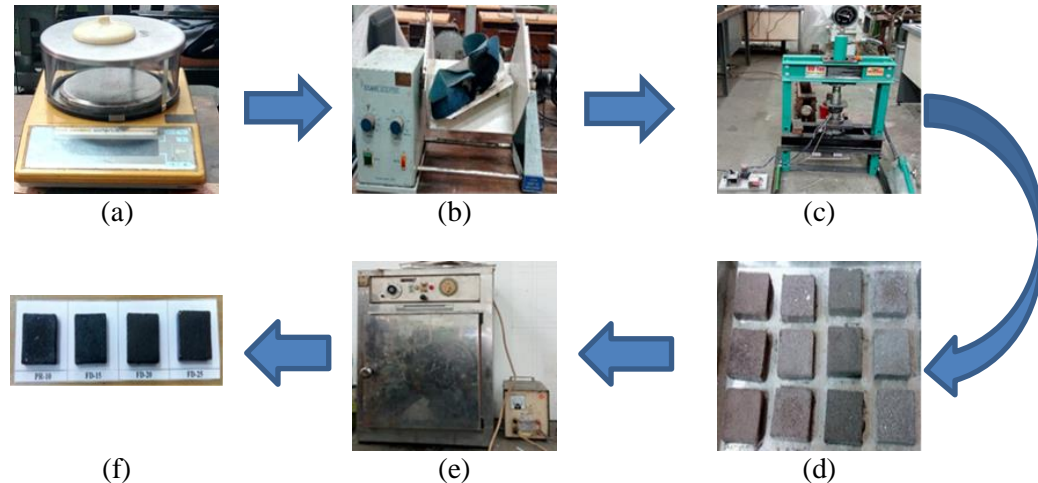


Figure 1. Stages process of making brake pad composite: (a) an analytical balance (Sartorius LC 1201S); (b) mixer (rotation drive STR4); (c) hand press hydraulic; (d) green bodies; (e) furnace (memmert 854 schwabach); (f) brake pad composites

The porosity measurement of composite sample was done based on Japanese Standard JIS D 4418: 1996. Composite samples with dimension of 25 mm x 25 mm x 5 mm \pm 0.5 mm were left in the desiccator for 24 hours at room temperature. All composite samples were weighted on the analytical balance (Sartorius LC 1201S) before the samples immersed in the oil container with the constant temperature of 90 \pm 10 $^{\circ}$ C for 8 hours. SAE 90 grade oil was used as the fluid of the test. Composite samples were left in container for 12 hours until the oil cools to the room temperature. Finally, all composite samples were withdrawn from the oil container and wiped with dry cloth for 5 times to remove oil from the sample surface. Composite sample was weighed again on the analytical balance (Sartorius LC 1201S). The porosity of composite sample was calculated using the following formula

$$\% \text{ porosity} = \frac{m_2 - m_1}{\rho V} \times 100 \quad (2)$$

Where m_1 is mass of composite sample before immersed in the oil container (mg), m_2 is mass of composite sample after absorbing oil (mg), ρ is density of oil (mg/cm³), and V is composite sample volume (cm³).

The hardness of composite sample was measured using a Shore D hardness tester according to the ASTM D2240. At least five replications of hardness test for each sample were made and the average value was reported.

Thermal stability is very important to determine the fade resistance on braking application. TG/DTA test was used to check change in weight and energy of the sample as a function of temperature [10]. The thermal stability of composite sample was measured using a Diamond TG/DTA analyzer PerkinElmer Instruments. Composite samples were heated at temperature of 30 $^{\circ}$ C to 1000 $^{\circ}$ C with a heating rate of 10 $^{\circ}$ C/minute in air condition. Alumina pan was used for this testing. TG/DTA test results are the graph of the temperature to weight loss and heat flow.

Friction and wear of composite samples were tested according to Indonesian National Standard (SNI 09-0143-1987) using a Friction Testing Machine JTM-1699 A (JTM Technology Co. Ltd). Two testing samples with dimension of 25 mm x 25 mm x 5 \pm 0.2 mm were used as a stator and disc made of cast

iron was used as rotor. Two samples were pressed against the disc at a contact pressure of 0.981 MPa. The disc temperature was maintained at 100, 150, 200, and 250 °C. The disc rotation and the number of rotation are 500 rpm and 1500 respectively at each temperature. The thickness of composite samples were noted before and after testing as much as 5 points to calculate the volume wear rate. The volume wear rate V ($\text{mm}^3/(\text{N m})$) was calculated using formulation (3).

$$V = \frac{1}{2\pi R} \frac{A}{n} \frac{d_1 - d_2}{f_m} \quad (3)$$

Where R is the distance between the center of sample and the center of the rotating disc (150 mm), n is number of rotation of disc (1500), A is the area of the sample (25 mm x 25 mm), d_1 is the average thickness of the sample before it is tested (mm), d_2 is the average thickness of sample after it is tested (mm), and f_m is the average force of sliding friction (N). Drawing scheme of the friction performance test was shown in Figure 2.

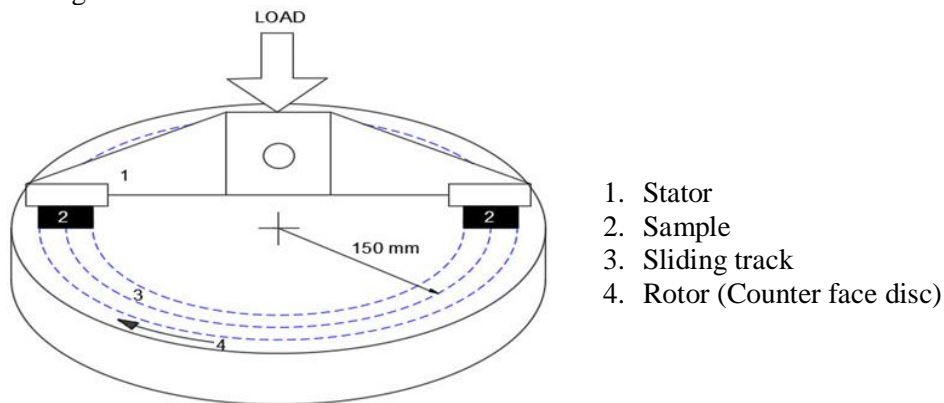


Figure 2. Drawing scheme of the friction performance test

3. Results and Discussion

3.1 Density, hardness, and porosity of brake pad composite

Figure 3 shows the result test of density, porosity, and hardness with on the varying of volume fraction of phenolic resin. Density sample depends upon the ingredients of brake pad composite. The result of density test exhibits that the density of composite samples decrease with increasing volume fraction of phenolic resin. Decreasing in composite density is attributed to replacement of low density phenolic resin ($\rho = 1.2 \text{ gr/cm}^3$) by high density of BaSO_4 ($\rho = 4.3 \text{ gr/cm}^3$) in the formulation of composite samples. In addition, density results show that the composite sample density complies with “the rule of mixture” of composite.

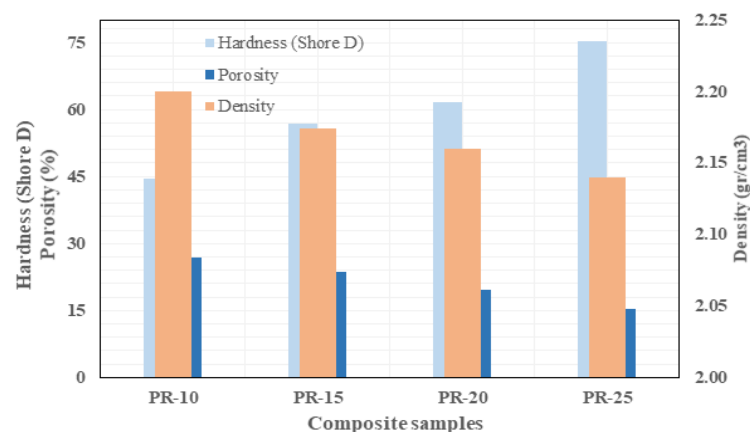


Figure 3. Density, porosity, and hardness of brake pad composites

The hardness result test of composite samples increase with increasing volume fraction of phenolic resin. Increasing volume fraction of phenolic resin will be increase amount of crosslinking during curing process thus the hardness improve. Kim *et al* [5] also reported that the hardness of brake pad increased with increasing phenolic resin. It was attributed to the high hardness of the thermosetting binder resin after curing. Meanwhile, porosity test results exhibit decrease with increasing in volume fraction of phenolic resin. Composite sample of PR-10 (filled 10 % vol phenolic resin) has the highest porosity. While, composite sample PR-25 (filled 25 % vol phenolic resin) has the lowest of porosity. The result of that porosity are caused by particle size of phenolic resin (about +270 mesh) fills the void space of particles during hot pressing process thus the contact area between particles increase and lead to decreasing in porosity. Brake pad porosity plays a role in the effectiveness of the brake system, because it absorbs heat and reduce brake noise. It is generally that the commercial products of brake pad need to maintain porosity about 10% to 14% for resulting in a good performing pads and the wear low [11].

3.2 The thermal stability of brake pad composite

The thermal analysis of four formulations, namely PR-10, PR-15, PR-20, and PR-25 were tested using Thermogravimetric and Derivative thermogravimetric Analysis (TG/DTA).

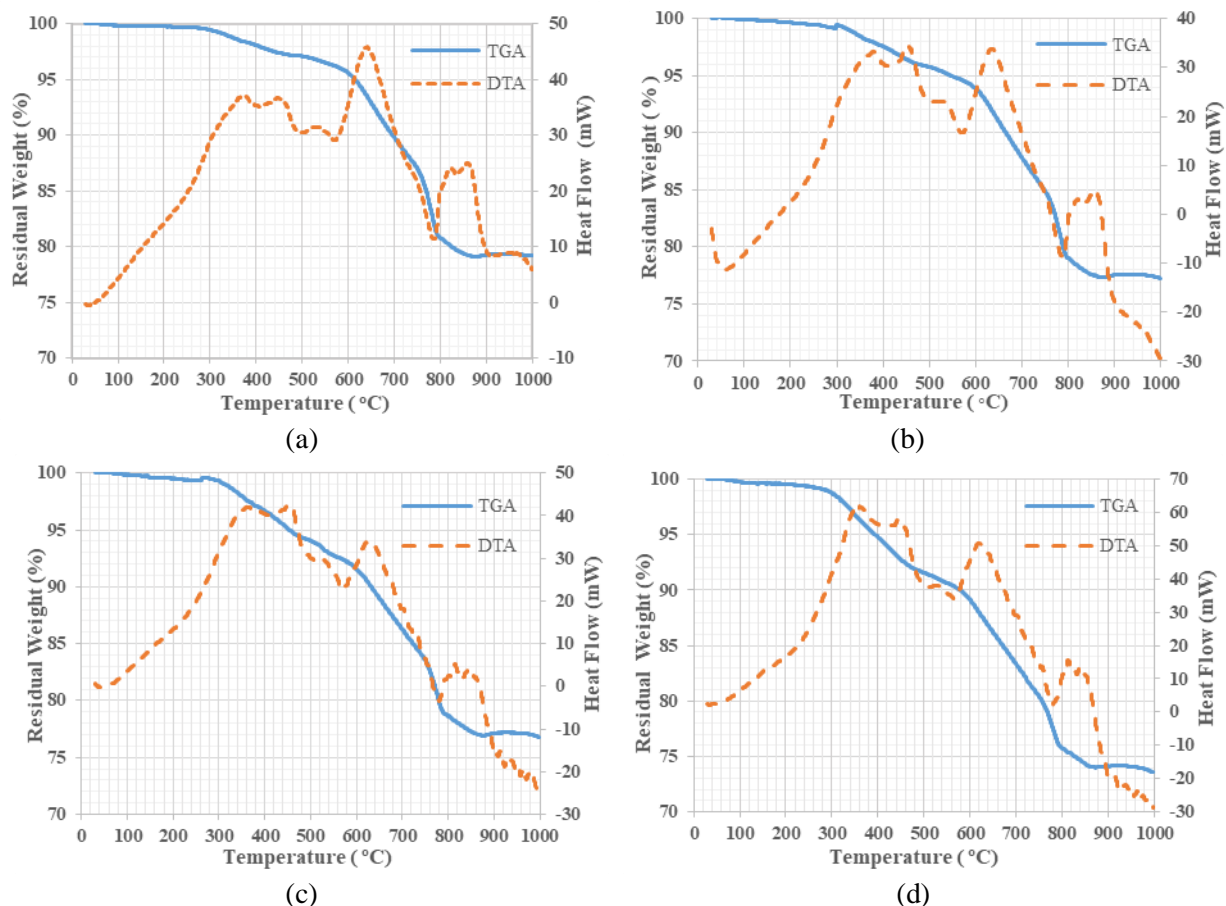


Figure 4. TG/DTA analysis of brake pad composites: (a) PR-10; (b) PR-15; (c) PR-20; (d) PR-25

Figures 4a-d show the TG/DTA curves for four composite samples with variation in volume fraction of phenolic resin. TG/DTA curves indicate the same weight losing trend, they have three weight loss steps, while the thermal decompositions occur in two stages. The initial weight loss ($\square 1.7\%$) occurs between the temperatures of 100°C to 200°C. It is attributed to the evaporation of water due to the reaction of chemical degradation phenolic resin. The degradation of four formulations are started at temperature of

above 275 °C. DTA curves show that the maximum thermal degradation are at 631 °C for PR-10, 624 °C for PR-15, 625 °C for PR-20, and 613 °C for PR-25. From the result of the maximum thermal degradation exhibits that composite sample of PR-10 has high degree of crosslinking thus it need more energy to decompose and improve the thermal resistance. In Figures 4a-d, there are a difference of slope in the TGA curves for four formulations, where the thermal degradation for PR-10 sample is more stable than the other studied composites. Thermal degradation (weight loss %) attributed to thermal stability, if the weight loss lesser meant thermal stability is better [12]. Table 2 shows that the weight loss (%) is follows: PR-10 < PR-15 < PR-20 < PR-25.

Table 2. Weight sample of brake pad composite in TG/DTA test

No	Sample Code	Initial Weight (mg)	Residue at 1000 °C		Maximum decomposition temperature (°C)
			(mg)	(%)	
1	PR-10	20.165	15.964	79.17	631
2	PR-15	22.264	17.199	77.25	624
3	PR-20	19.654	15.091	76.80	625
4	PR-25	23.720	17.448	73.56	613

TG/DTA results (Table 2) shows that the thermal stability of composite samples (PR-10, PR-15, PR-20, and PR-25) are decrease with increasing volume fraction of phenolic resin. Therefore, the composite sample of PR-10 has the best thermal stability than the other studied composites.

3.3 The friction and wear performance of brake pad composite

The friction coefficient and wear of brake pad composites are presented in Figure 6a-b. That graphs show that the results of friction test only performed on composite samples of PR-20 and PR-25. Where, the composite samples of PR-10 and PR-15 are damaged during the test run, as seen in Figure 5. The damage of composite samples PR-10 and PR-15 occur at temperature of 100 °C with the test duration of about 400 second for PR-10 and 700 second for PR-15. It is caused by phenolic resin is not sufficient binding all of the ingredients of composite, therefore composite samples have unable to against mechanical stresses during testing operation. It is also supported from the results of hardness value of composite samples PR-10 and PR-15. They have lower hardness value and have higher porosity than other studied composites (as seen in Fig 3).



Figure 5. Brake pad composites damaged during friction test: (a) PR-10; (b) PR-15

The results of friction coefficient and volume wear rate of composite samples PR-20 and PR-25 are shown in Figures 6a-b. Figure 6a shows that friction coefficient of composite sample PR-25 at temperatures of 100 °C to 250 °C is lower than composite sample of PR-20. It is attributed to composite sample of PR-25 has lower thermal stability than composite sample of PR-20. Cai *et al* [2] reported that the sample with poor thermal stability, causing a low magnitude of friction coefficient. In addition,

composite sample of PR-20 shows the friction coefficient decrease with increasing in temperature and more stable. However, the friction coefficient of composite sample PR-25 are fluctuated against temperature changes. These fluctuations are due to non-homogeneous heat loads on the friction surfaces changing during test period. Therefore, friction coefficient will be changes continuously [13].

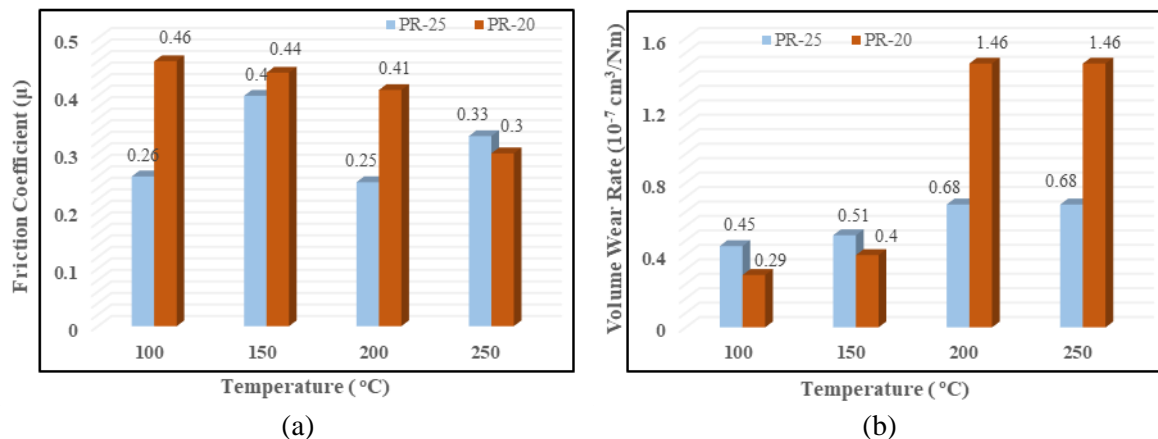


Figure 6. Friction performance of brake pad composites (PR-20 and PR-25) (a) friction coefficient versus temperature; (b) Volume wear rate versus temperature

Figure 6b shows the volume wear rate of composite samples PR-20 and PR-25. The graph shows that increase in temperature lead to increasing in the volume wear rate. The composite sample of PR-25 has higher volume wear rate than composite sample of PR-20 up to temperature of 150°C. On the contrary, the volume wear rate of composite sample PR-25 is lower than composite sample PR-20 at temperature of 200 °C to 250°C. Increasing in temperature allows phenolic resin to decompose thus they are not sufficient to bind all of ingredients composite. This conditions causes increasing in the volume wear rate. In addition, the thermal stability of composite sample PR-20 is better than composite sample PR-25 up to temperature of 150°C.

4. Conclusions

The density, porosity, hardness, thermal stability, and friction performance of non-asbestos organic brake pad with a different of the volume fraction of phenolic resin were investigated. Based on the results and discussion of this study the following conclusion can be made:

1. The density decreased with increasing of the volume fraction of phenolic resin. This is because of the replacement of low density phenolic resin by high density of BaSO_4 in the formulation.
2. The porosity decreased with increasing of the volume fraction of phenolic resin, while the hardness increased. This is attributed to the contact area between particles increased as a result of the phenolic resin particle fills the void space during hot press process. Thus, bonding strength and hardness of composite sample increase and the porosity of composite sample decrease.
3. The thermal stability of composite samples decrease with increasing of the volume fraction of phenolic resin. The composite sample of PR-10 exhibits better thermal stability than the other studied composites due to it has the lowest weight loss as compared to the other studied composites.
4. Increasing in the volume fraction of phenolic resin will be decreased in friction coefficient and increase in volume wear rate. The composite samples of PR-10 and PR-15 are not recommended to be selected in this formulation because they have lower resistance to mechanical stresses during friction performance test although the thermal stability is better than the other studied composites.

5. Acknowledgement

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