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The Carbon of *Swietenia Machrophylla* Fruit Peel and Coal Fly Ash as Bio-Composite Brake Ingredients

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Abstract

The eco-friendly brake composite has been still an interesting issue in the development of brake friction materials. Wastes of *S. Macrophylla* (mahogany) fruit skin and coal fly ash are available as organic ingredients of bio-composite brakes. In this research, we investigated the effects of both ingredients on the brake composite properties which were fabricated using hot isostatic pressing at temperature 200 °C and pressure 5 kN for 3 h. The specimens were prepared in some volume fractions of carbon (2 vol% - 12 vol%). As a result, several tested specimens containing mahogany fruit skin carbon revealed maximum Rockwell hardness 69 HRB, wear $2.49x10^{-4}mm^2/kg$, and water absorption 2.72 %, while specimens containing coal fly ash showed 78 HRB, $1.1x10^{-3} mm^2/kg$, and 3.5 %, respectively. The brake composites containing coal fly ash performed better than ones containing mahogany fruit skin carbon. The hardness and wear of these two types of brake composite friction materials meet the minimum criteria required by SAE, JA661, and are close to the quality of the brake pads of two commercial brake composite materials. Water absorption in the brake lining specimens with mahogany leather carbon showed that the addition of the volume fraction caused an increase in water absorption, while the specimen containing coal fly ash showed that the increase in the carbon volume fraction caused a decrease in water absorption.

INTRODUCTION

The brake composites are multiphase materials that serve to stop the rotor movements of vehicles and should have standard characteristics namely stable friction, low wear rate, noise resistance, ecofriendly, and high thermal stability. In the fabrication process, the superiority of these material properties could be achieved by the usage of about 7-15 ingredients (Singaravelu et al., 2019; Maleque & Atiqah, 2013). Meanwhile, the uniform materials could not meet the minimum requirements of a commercial brake friction material because the parameters like safety and comfort of brake performance were not achieved (Pujari & Srikiran, 2019). In recent years, many organics ingredients and recyclable wastes were used to manufacture green brakes in avoiding the use of asbestos material (Pujari and Srikiran, 2019; Singh et al., 2017). The bio-composite brake was selected as an alternative in recent years to substitute the conventional brake because it is lighter, stronger, higher wear resistance, higher corrosion resistance, higher durability so that it has been still attracting the interest of researchers to develop it (Wasilewski, 2017). In this research, the mahogany fruit skin carbon and the coal fly ash were selected as brake composite ingredients. because of their abundances in nature and its recyclability. Based on the data in the Indonesian Central Bureau of Statistics, Indonesia's forest area is around 144 million hectares, and 1.3 million hectares of land are in the form of industrial forest plantations. In detail, eight percent of industrial plantations are sources of mahogany. The carbon either in fiber or powder form is one of the

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important ingredients in bio-composite brake and it plays a role as a lubricant in the friction mechanism (Ahmadijokani et al, 2019). In the previously published work, the carbon of coconut chars was used as one ingredient of brake composite (Sutikno et al., 2010). Th carbon fiber can enhance the wear resistance drastically in comparison with aramid fiber (Ahmadijokani et al., 2020) and the chopped carbon fibers are used as reinforcement material for brake composite (Dang et al. 2018). The carbon is also available to make composites for integrated electrodes as electrochemical detectors in microchips (Arvinte et al., 2020; Adam et al., 2012). Some amazing characteristics in the carbon-based brake composites such as low density, high thermal capacity, low thermal expansion, and outstanding friction property are found in recent years by many researchers (Hao et al., 2014). To produce a stable friction coefficient and low wear loss, the content and dimension of carbon in the brake lining composite need to be adjusted (Hao et al, 2014; Wasilewski 2017). The shape of ingredients influences the tribological properties (Singh et al., 2017) and for example, the hardness and flexural strength of brake composites are improved steadily by increasing the carbon fiber content (Ahmadijokani et al., 2019).

The usage of wastes such as metals, glasses, and organic materials to fabricate brake friction material has been developed by the material society around the world (Sutikno et al., 2013). Coal fly ash is a result of the industry which is using coal as a fuel for energy-generating such as electricity, metallurgy, cement, and ceramic industries. These waste abundances have not recyclable and give a negative impact on society and environmental health. It contains a high content of alumina and silica and available as reinforcement particles for polymer matrics composite to replace ceramics materials for example MgO and Al_2O_3 (Chen et al., 2005). Coal fly ash fulfills the criteria as pozzolanic materials because it has cylindrical particles and very smooth (Külaots et al., 2004), it is appropriate used as one of brake composite ingredients. Another application is as solid sorbent to reduce sulfur dioxide. The tribo-study proved that the fly ash significantly increased the friction coefficient of brake composite specimens (Ahlawat et al., 2020; Ahlawat et al., 2019).

Their contents of mahogany fruit skin and coal fly ash were optimized at each specimen and their effects on the water absorption, mechanical properties, and thermal properties were investigated. The wear resistance and hardness of brake composite specimens are performance parameters of brake composite that must be fulfilled following the minimum criteria. Based on the Indonesian National Standard (SNI), good brake linings have hardness specifications (68-105 HRB), wear ($5x10^{-4}$ - $5x10^{-3}$ mm^2/kg), friction coefficient (0.14 - 0.27), and fracture strength (480 - 1500 N/cm^2).

MATERIALS AND METHODS

Materials

This fabrication process of specimens included the preparation of mahogany fruit skin and coal fly ash preparation. The mahogany rind was taken from a plantation in Gunungpati Sub-district, Semarang City, Indonesia, while coal fly ash was supplied from the waste of the Cilacap steam power plant, Central Java, Indonesia. The other ingredients consisted of epoxy, magnesium oxide, calcium carbonate, metal mix, zinc oxide, stearic acid, glass powder, bagasse, bakelite, and sulfur. These materials were technical grade materials taken from the local market. The used materials were calculated based on their volume fractions, initiated by determining the total volume of the mixture as a basis of calculation. In detail, the fabrication route included sequential dry mixing, pre-forming, hot pressing (curing process, post-curing), and finishing (Singh & Patnaik, 2015; Sugözü, 2018).

The basic composition of brake composite was fixed the same as in the previous article (Sutikno et al., 2012; Madnasri et al., 2013) by adding new ingredients as listed in Table 1 that including carbon mahogany rind and coal fly ash. Meanwhile, the rubber material was not used. All ingredients were mixed and ignored in a petri dish up to homogeneous. A mixing variation of carbon content and coal fly ash in the specimen was made in 2 vol% - 12 vol% and it was pressed by hot isostatic pressing method at 200 °C, for 3 h, and pressure 5 kN (Shojaei et al., 2007).

The testing of specimens was done to determine the effects of carbon volume fraction of mahogany fruit peel or coal fly ash on the hardness, wear resistance, water absorption, thermal resistance, and to observe the microstructures of the composite brakes. Additionally, the hardness testing was done using Rockwell hardness tester with load 981 N and ball indentor 2.5 mm. The scratch diameter of the indentation trace was observed using a microscope in magnification of 100x (1 strip = 38 microns) and used to determine Rockwell hardness using Eqs. (1) .

$$HRB = 100 - \frac{t}{0{,}002} \quad (1)$$

$$t = D - \sqrt{D^2 - d^2} \quad (2)$$

Where, HRB is the Rockwell hardness number, t is the indentation depth (mm), D is the diameter of indentor (mm) and d is the indentation diameter on the specimen (mm). Their hardness values of both specimens were compared to study the effects of carbons.

Table 1. The composition of developed brake composites.

Ingredient	Volume fraction (vol%)	
	Mahogany fruit skin	Coal fly ash
Epoxy	8.50	8.50
Metal powder	38.30	38.30
Carbon	2.84	2.84
Magnesium oxide	7.80	7.80
Calcium carbonate	7.80	7.80
Zinc oxide	2.13	2.13
Stearic acid	2.13	2.13
Glass powder	11.35	11.35
Bakelite	17.02	17.02
Sulfur	2.13	2.13

The specimen wear resistances were tested using Ogoshi High-Speed Universal Testing Machine (Type OAT-U) with load (P_0) of 6.36 kg, wearing distance (L_0) 200 mm, wearing plate length (B) of 3 mm, radius length of wearing plate (r) of 14 mm, and wearing time for 60 s. Scratches of revolving disc friction were measured by using an optical microscope in magnification of 100x (1 strip = 38 microns). Next, the wear resistance of both brake composite specimens was calculated using Eq. (3) and compared,

$$W_s = \frac{B\ (B_0)^3}{8r\ P_0 L_0} \quad (3)$$

where W_s is the wear resistance (mm^2/kg) and B_0 is the indentation length on the specimen (mm).

In this study, three brands of commercially available brake linings (Asmoto, Honda genuine part, and Indopart) were tested for their mechanical properties. These three types of commercial brake pads are original disc brakes for 125 cc class motorcycles. The testing of the mechanical properties of the three commercial brake linings includes the Rockwell hardness test, wear test, and tensile strength test. In the Rockwell hardness testing, some indentation loads were adjusted in adaptation with the specification of the test specimens. The results of testing the mechanical properties of commercial brake pads were compared with the test results of brake pads made of fly ash and brake pads made of carbon mahogany fruit skin.

Next, the water absorptions on both specimens were determined to obey their applicabilities in the wet environment. The specimens were sliced into 1 cm x 1 cm and their masses were weighed before and after the drying process at 50 °C for 2 h. After the specimens dried, we soaked it in a beaker glass filled with 100 mL water for 10 mins, and then it was taken to be re-weighed. The percentages of water absorption were calculated using Eq. (4) and then it was compared and analyzed,

$$Water\ Absorption = \frac{m_{wet} - m_{dry}}{m_{dry}} \quad (4)$$

where m_{dry} is mass of specimen before soaked (g) and m_{wet} is mass of specimen after soaked (g). We observed the microstructures of specimens (1 cm x 1 cm) surfaces and the chemical elements using a Scanning Electron Microscope (SEM) integrated with Energy Dispersive X-ray Spectroscopy (EDS).

To study the thermal properties and phase change of composite brake due to enthalpy change, the specimens were tested using differential thermal analysis (DTA). Several thermogravimetric analysis (TGA) parameters for different brake composites were set up at temperatures (T_{onset}) 0 °C - 450 °C, delta temperature (ΔT) 25 °C, and operation duration 120 mins. Many samples for TGA analysis were prepared of 1 g powders and the TGA instrument was operated in the temperature range of 25 °C – 425 °C for 2 h. Finally, a maximum operating temperature (T_{max}) was achieved at 425 °C.

RESULTS AND DISCUSSION

Several data on physical and mechanical properties are reported and analyzed in some tables and graphs. The brake composites containing mahogany fruit peel seem black and have an average density of 0.419 g/mL, while those containing coal fly ash were brown and have a mean density of 1.1915 g/mL. Figure 1 exhibits some Rockwell Hardness numbers scale B. The brake friction material containing mahogany peel has Rockwell hardness between 51 HRB - 69 HRB. On the other hand, brake friction material containing coal fly ash has Rockwell hardness in the range of 66 HRB - 78 HRB. It is found that the coal fly ash contributes to the hardness of friction material more dominant than the mahogany carbon shell. Both materials can produce brake linings that meet Rockwell hardness standards, namely greater than 68 HRB.

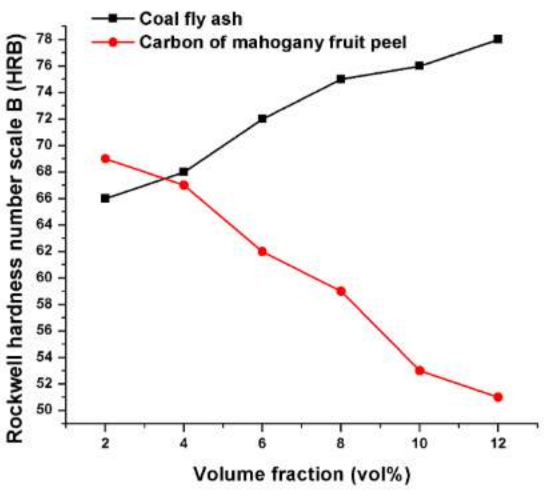
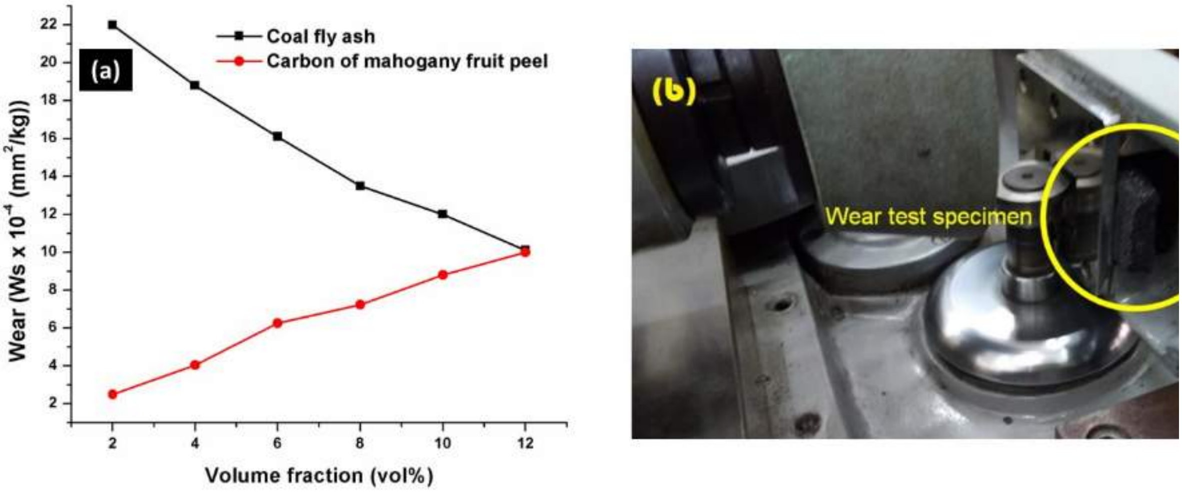


Figure 1. Rockwell hardness number of composite brake to the increase of volume fraction: (a) Mahogany fruit skin carbon and (b) coal fly ash.

At the testing of specimens containing mahogany rind carbon, the increase of volume fraction of mahogany rind carbon has decreased the hardness number. Inversely, at the testing of specimens containing coal fly ash, the increase of volume fraction of coal fly ash has improved the hardness number. The composite brake pads containing mahogany rind carbon have an optimum hardness number of 69 HRB, found in samples with 2 vol%, while those containing coal fly ash have an optimum hardness number of 78 HRB, found in samples with 12 vol%. This is because the size of coal fly ash particles (about 7.44 µm) is greater than that of the carbon particles of mahogany skin (about 3.53 µm). Large particles contribute to the nature of the material in resisting the movement of deformed structures. Coal fly ash particles have a denser structure and have stronger bonding between particles so they can withstand deformation during hardness testing. The addition of coal fly ash increases the hardness of the composite material of the brake lining due to the increased bonding strength (polymer) against plastic deformation. The smaller particles caused the density of the material to become higher and it plays a significant role in increasing the hardness of composite friction material. The carbon density of mahogany rind equals 35 % of the density of coal fly ash.

For the wear test of the brake lining composites, its results are plotted in Figure 2 and getting information that the addition of the volume fraction of the mahogany skin carbon causes the wear value increases as well, whereas the addition of the coal fly ash ingredient leads to a smaller wear value of the composite brake lining. The increasing trend in the wear of brake friction composites is proportional to the rising content of coal fly ash in the composite, it is consistent with the results of Dadkar et al's study in 2009 (Dadkar et al., 2009). Carbon-based brake composites provide excellent wear resistance and braking performance at high-energy braking conditions, larger than 5 MJ (Prabhu, 2016). The brake lining from coal fly ash has better wear resistance than the brake lining of the material of mahogany rind carbon. This is because coal fly ash has a specific gravity greater than carbon of the skin of mahogany so that composite samples containing coal fly ash have given a significant contribution to the densification process of the brake pad composite. The sample becomes harder and higher wear resistance. On the other hand, the composite of brake lining containing carbon of mahogany rind, the process of densification (compaction) of its constituent materials when mixing is not evenly distributed so that the sample is softer and has low wear resistance. It is thought a breaking process of a little coal fly ash particles is the cause.

A comparison of water absorption characteristics for two brake composites is exhibited in Figure 3. The addition of a volume fraction of coal fly ash causes a decrease in water absorption, in contrast to the addition of a volume fraction of the mahogany rind carbon causing an increase in



(a) (b)

Figure 2. (a) Wear of brake friction composite versus volume fraction increase of (i) mahogany rind carbon, and (ii) coal fly ash; and (b) picture of wear testing instrument of brake composite.

Table 2. Data on water absorption of specimen containing mahogany rind carbon.

The volume fraction of carbon (vol%)	Specimen immersion (10 min)		Water absorption of the specimen (%)
	The initial mass of specimen (g)	End mass of specimen (g)	
2	1.10	1.13	2.72
4	1.20	1.24	3.33
6	1.29	1.35	4.65
8	1.34	1.41	5.22
10	1.35	1.43	5.92
12	1.68	1.78	5.95

Table 3. Water absorption test data of specimens containing coal fly ash.

The volume fraction of coal fly ash (vol%)	Composite immersion (10 mins)		Water absorption (%)
	Initial mass (g)	End mass (g)	
2	1.30	1.40	7. 69
4	1.47	1.57	6.80
6	1.50	1.58	5.33
8	1.59	1.66	4.40
10	1.95	2.03	4.10
12	2.00	2.07	3.50

the water absorption rate. This is because coal fly ash has a smaller particle size so that it can fill smaller pores compared to carbon made of the mahogany rind. Thus, the produced composites of coal fly ash seem denser and solid. According to (Erol et al., 2008), the finer shapes of coal fly ash particles can reduce the composite porosities. The porosity at various sintered temperatures has shown a decrease due to the increase in the weight fraction of coal fly ash up to 5 %, where at the weight fraction of coal fly ash above of 5 wt%, the composite porosity with aluminum matrix material tended to be constant. Low porosity (relatively high density) was found in composites with a weight fraction of coal fly ash lower or equal to 5 wt%. The porosity is influenced by the weight fraction and the distribution of coal fly ash particles in the matrix. If the weight fraction of coal fly ash on the composite is low (less than or equal to 5 wt%), the distribution is evenly distributed. This makes the interaction or

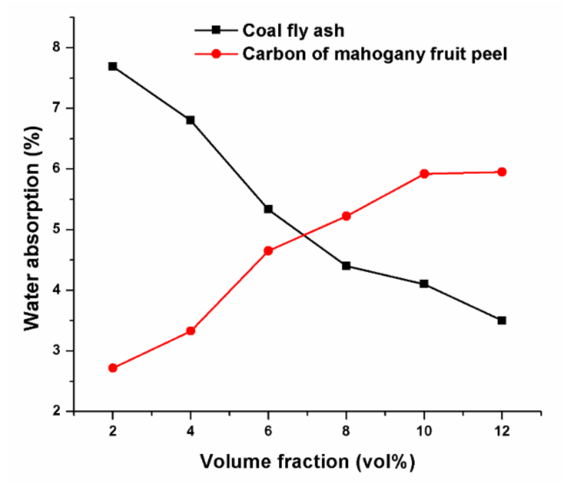


Figure 3. Water absorption for each composite brake linings on the different volume fraction: (a) Mahogany rind carbon and (b) coal fly ash.

bond between the coal fly ash particles and the matrix very good. Conversely, if the weight fraction of coal fly ash on the composite is more than 5 wt%, then some coal fly ash particles coincide or group together, so the bond between the coal fly ash particles and the matrix is imperfect. This will bring up the cavity so that its porosity increases. A previously published research result stated that the use of coal fly ash in composites can increase the compressive strength and water absorption rate (Ilic et al., 2003). The impact strength increases in the brake composite containing coal fly ash with smaller particle sizes which confirms that vacuum has existed in the composite with the larger particles of coal fly ash. This composite brake is better used in wet conditions because it can absorb water well.

Figure 4 shows some microstructures of composite brake lining containing mahogany leather and that of coal fly ash. The SEM images of composite brake pads containing carbon mahogany rind where the particle shapes are not clearly visible and very small while containing coal fly ash materials have the round particle shape and look very clear. Many pores found on the surfaces are in the separated positions because of the uneven mixing process and the particle size of each material is different.

The EDX curves of the composite brake linings are revealed in Figure 5. Table 4 exhibits the results of the weight percentages of chemical elements contained in the composite brake lining. The composite sample of brake lining with one of the ingredients of mahogany rind and coal fly ash has the same elemental content, but the amount of weight percentage of each chemical element contained is different. This can be seen in the carbon content of mahogany rind of 58.9 wt%, while the carbon content of coal fly ash material is 66.2 wt%. It is known that the specimen of brake composite made using mahogany fruit skin as one of the ingredients, containing carbon less than that of coal fly ash for the same formula mixing.

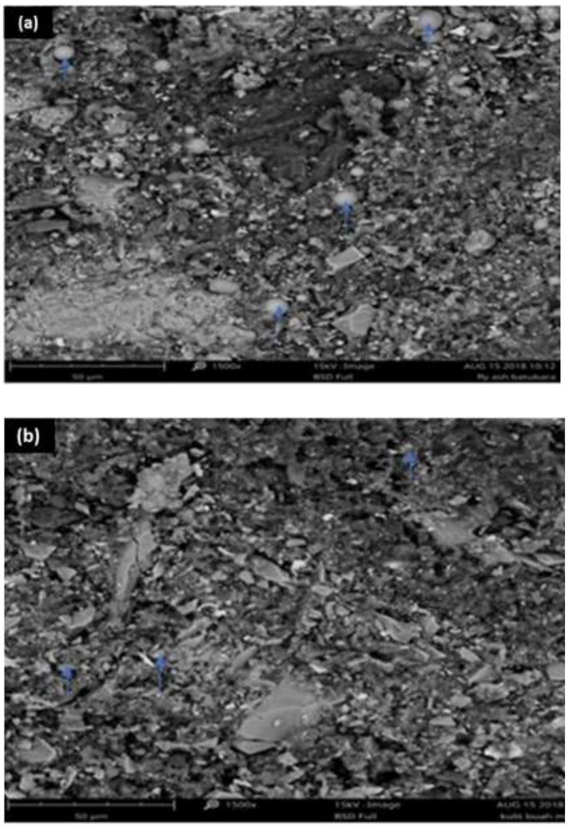


Figure 4. SEM images in magnification of 1500X of brake composite containing: (a) Mahogany rind carbon and (b) coal fly ash.

As a result of thermal characterization using Differential Thermal Analysis (DTA), its properties of composite materials from the carbon of mahogany rind and coal fly ash can be seen in Figure 6. The composite brake pads with mahogany rind carbon as one of its ingredients begin to experience enthalpy changes (delta m) of 1.75 mg when the temperature reached 3000 °C, whereas composite brake linings containing the coal fly ash material begin to suffer enthalpy changes (delta m) of 2.50 mg when the temperature reached 2500 °C. Meanwhile, the results of thermal testing revealed a difference in enthalpy change, which for brake lining with coal fly ash material has a greater enthalpy change (delta m) compared to that of with mahogany rind carbon material. A weight loss of

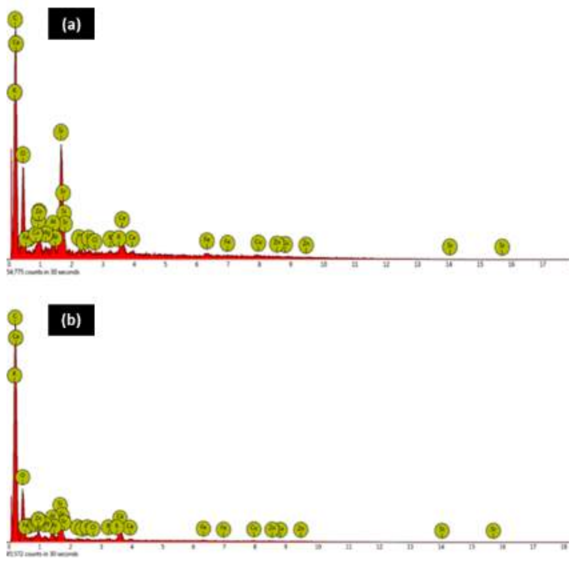


Figure 5. EDX curves of samples containing: (a) Mahogany rind carbon and (b) coal fly ash.

Table 4. Chemical elements in brake composite based on the EDX spectra.

Element	Weight in percentage (wt%)	
	Mahogany rind carbon	Coal fly ash
C	58.9	66.2
Si	3.6	1.3
Sr	3.3	1.5
O	25.9	26.2
Ca	1.8	1.4
Na	2.0	1.1
Al	0.9	0.8
Mg	0.4	0.4
S	0.2	0.1
Fe	0.9	0.3
Cl	0.2	0.1
K	0.2	0.1
Cu	1.0	0.3
Zn	0.6	0.3

mahogany rind carbon-based brake composite occurs at 400 – 125 °C as much as 1.75 mg, on the other hand, a weight loss of coal fly ash-based brake composites several 2.5 mg significantly occur at 325 – 150 °C. The weight loss characteristics of composite brake linings with different materials have been reported by Cong et al. in 2012 (Cong et al., 2012). Jeganmohan and Sugozu reported that heat treatment could improve both the physical properties and the friction properties of the composite brake pads (Jeganmohan & Sugozu, 2020). The DTA data are available to study the thermal properties of brake composite. In the real condition, the braking temperature for a motorcycle can achieve about 315 °C. Here, we simulate the real temperature of braking will increase up to 415 °C. It is expected, the brake material has been still good working at that temperature. The maximum temperature, 425 °C, is achieved after 23 mins.

The results of testing the mechanical properties of three types of commercial brake pads, namely Asmoto, Honda, and Indopart brands are shown in Table 5. These values are also compared with the brake pads made of coal fly ash and the brake pads made of charcoal mahogany fruit peel and Society American Engineers (SAE) Standard, J661. The three reported mechanical properties of brake lining to consist of material hardness, strength, and wear. Based on Table 5, the Asmoto brand brake pads show the most superior mechanical properties, namely hardness 134.2 HRB, tensile strength 1227 N/mm^2, and material wear 2.86x10^{-4} mm^2/kg. These values are compared with the experimental results of two types of fabricated brake lining specimens namely coal fly ash-based brake composite and mahogany fruit peel-based brake composite. Based on the superiority of its mechanical properties, it can be ranked from the most superior to the lowest as follows Asmoto brake composite, Honda brake composite, coal fly ash-based brake composite, mahogany fruit peel-based brake composite, and Indopart brake composite. The hardness values of the two types of brake lining composites of this study, coal fly ash-based brake composite (66 - 78 HRB) and MFP (51 - 69 HRB) meet SAE Standard J661, namely 68 - 105 HRB. Another fact is that the hardness value of Indopart brake pads is around 61 HRB, still below the minimum value set by SAE. In general, all tested specimens meet the wear requirements under SAE, J661 (5x10^{-4} – 5x10^{-3}mm^2/kg). Both coal fly ash-based brake pads and mahogany fruit peel-based brake pads meet the minimum wear requirements of the SAE, J661. Asmoto brake linings and Honda genuine parts also demonstrate superior tensile strength and meet SAE standards. The tensile strength of the Indopart brake pads (approximate 379 N/mm^2) is still below the SAE minimum standard (480 N/mm^2) and that of the coal fly ash-based brake composite specimen, 480 N/mm^2, is close to the minimum value of tensile strength according to SAE. Meanwhile, the tensile strength of the mahogany fruit peel-based

Table 5. Comparison of the assessed characteristics of fabricated brake composites with commercially available brake linings.

Test Specimen (TS)	Rockwell hardness number scale B (HRB)	Strength (N/mm^2)	Wear (mm^2/kg)
Asmoto	134.2	1227	2.86x10^{-4}
Honda genuine part	98.4	776.43	1.06x10^{-3}
Coal fly ash	66 – 78	385 - 480	1x10^{-3} – 2.2x10^{-3}
Mahogany fruit peel	51 – 69	300 - 400.33	2.49x10^{-4} – 1x10^{-3}
Indopart	61	379	1.18x10^{-3}
SAE J661	68-105	480 – 1500	5x10^{-4} – 5x10^{-3}

brake pads specimen, 400.33 N/mm^2, is still below the minimum value of tensile strength according to SAE.

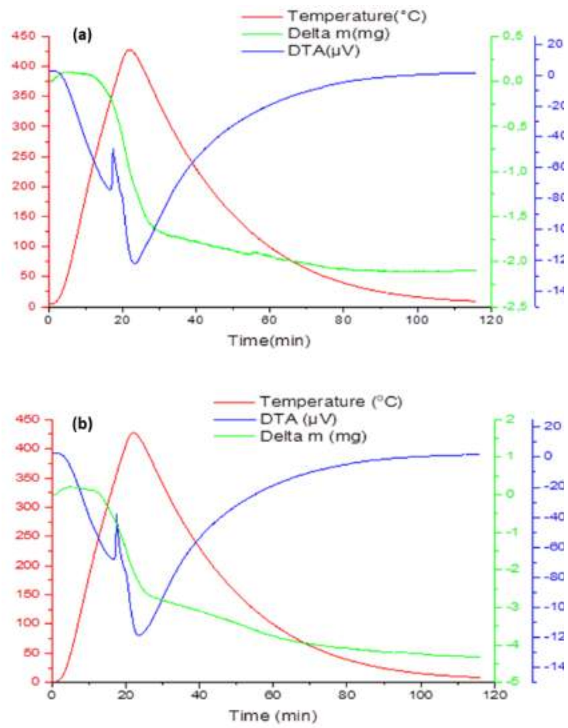


Figure 6. Graph of differential thermal analysis (DTA) of brake composite at 0°C – 450 °C for 120 minutes.

CONCLUSION

In conclusion, the composite brake pads containing carbon of mahogany peel revealed the best hardness of 69 HRB, while those containing coal fly ash indicated the best hardness of 78 HRB. We have found the best wear of composite brake lining with mahogany rind carbon correspond to 2.49x10^{-4} mm^2/kg, while those containing coal fly ash revealed the best wear of 10.1x10^{-4} mm^2/kg. The hardness and wear of these two types of brake composite materials meet the minimum requirements applied by SAE, JA661, and are close to the quality of Honda Genuine Parts and Asmoto Composite brake pads and exceed the quality of Indopart brand brake pads. Next, the water uptake on brake pad specimens with mahogany rind carbon confirmed that the addition of volume fraction has caused an increase in water absorption, whereas specimens containing coal fly ash showed that the increase of carbon volume fraction could cause the water absorption decrease. This recommends that a composite brake lining with coal fly ash material is more appropriate used in wet environments. Changes in enthalpies (delta m) of brake lining with mahogany rind carbon by 1.75 mg, while specimens containing coal fly ash material experienced enthalpy changes (delta m) by 2.50 mg.

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