Preparation of Novolac composites with improved properties for disk brake systems

Roola A.K. Abbass,

Material Science Division, Department of Applied Science, University of Technology, Baghdad, Iraq. Tel: +964 7506902189

Mahmood M. Barbooti, (Corresponding author) Applied Chemistry Division, Department of Applied Science, University of Technology, Sinaa Street, P.O. Box 35045, Baghdad, Iraq. Tel: +964 750 241 5259;

Abeer M. Abdul-Aziz

Material Science Division, Department of Applied Science, University of Technology, Sinaa Street, P.O. Box 35045, Baghdad, Iraq. Tel: +964 7505310174

Abstract

The effect of composition and the dry and wet operating conditions on the adhesive wear resistance of polymeric composite materials were studied in this work. The composite are designed to be used as pads for disc brake system of heavy vehicles. The composites were prepared with weight ratios, Ψ , in the range: 60-90% using the dry method. The composites were reinforced with a number of fillers and friction improvement agents like: silicon carbide, carbon fibers, alumina, etc at various ratios to establish diverse properties in comparison with Novolac resin. The used ceramic disc brake materials were also utilized in this preparation at certain weight ratios as one of the components of the composites. The wear resistance measurements indicated that a ratio higher than 70% will reduce the hardness of the disc material. Thus, composites with 60% Novolac weight ratio, which indicated the best performance, were prepared with 14-22% of the hardener, the HMTA. The study revealed that the abrasion rate decrease with the reinforcement of the Novolac, i.e., increase in abrasion resistance, especially when the carbon fibers ratio were increased from 1.5 to 3.0%. Meanwhile, the increase of the hardener resulted in increase in the in wear resistance. Further, it appears that the rate of abrasion changes from moderate to in accordance with operating conditions like the applied load, operating time and sliding distance. To mimic the real application conditions, the specimens were subjected to rain water by spraying for 2 min during operation. The wetting caused a lowering of the wear resistance. However, in comparison with the available disc brake materials, our specimens proved either comparable or better performance.

Keywords: Novolac composites, wear resistance, brake pads, rain water,

1. Introduction

The phenomena of wear and friction accompanied man from early stages of using the sliding, vibrating, and rolling mechanical parts of two contacting objects and result in replacement of the objects after certain service period [1, 2]. This is apparent in the brake systems which consist of steel plates on which some lining material is fixed with bolts to be used for the reduction of speed of trucks moving on roads with high kinetic energy stored as inertia. The energy is transferred then into heat due to friction to stop the vehicles and at the same time deteriorates the brake pads. The lining therefore, keeps the brake in good conditions. The brake system is considered one of the most important components of the vehicles and directly related with the safety and thus, periodical maintenance is essential part of the overall maintenance plan [3]. The brake pad may undergo:

- Mechanical deterioration: where the pad will expand and slightly separates from the moving wheel or disc and longer time will be needed to attain the required stop.
- Thermal decomposition: high temperature results in the decomposition of the pads.
- Gaseous degradation: gases and dust will result in the reduction of the friction coefficient between the two surfaces, especially with severe braking [4].

Asbestos, which has excellent friction properties, was one of the main ingredients of the brake pad until it was banned for being hazardous to health [4]. Brake pads are manufactured from mineral materials and organic materials. Organic brake pads consist of natural as well as synthetic resins that are characterized by heat resistance. For heavy vehicles organic pads are not the right choice due to the expected high temperature. However, such resins can be included in the composition of the pads up to a value of 30% [5]. Many authors studied the brake system deterioration to introduce the relevant solutions. Jang et al., 2004, [6] investigated the effect of different metallic fibers like Cu, steel, or Al upon friction and wear performance of various brakes

friction couples. The copper fibers showed a pronounced negative $\mu-\nu$ (friction coefficient versus sliding velocity) relation when the friction material was rubbed against gray cast iron disks but not against the Al-MMC counter surface. Cu fibers exhibited better fade resistance than the others elevated temperature.

The friction coefficient increases as the resin and MgO content increase and decreases in the presence of potassium titanate, copper zirconia, and rubber (Jang 2005). The use of rubber powder in the production of brake pads improves the friction properties [7].

Qiao, 2006, proved that the use rubber powder of various particle sizes is effective means for the improvement of the friction behavior of brake pads [8]. In his study on the effect of phenol on the tribological behavior of the brake material, Jang, 2008, found that the hardness, compressibility, and porosity changes with the ratio of phenol. The hardness linearly decreases with compressibility and porosity. At moderate temperatures (100° C) the friction coefficient increases with the increase of the phenol content. Meanwhile, the wear resistance increases with increase of the phenol added [9]. Sasikumar, et al., 2014, have studied the effects of micro silica on the thermal stability and ablative properties of composites of ceramic woven and phenolic resin filled with cenosphere. The result reveals that, up to 15% addition of microsilica enhanced the mechanical properties [10]. Leaibi studied the effect of reinforcement of polyesters with rice husks on the wear resistance under various operating conditions: load, sliding distance, time. He showed that the increase of such parameters result in higher wear rate (1.9 x gm.cm⁻¹) [11].

The purpose of the present is to study the use of some phenolic resin composites of Novolak type at weight ratios of 14-22% in the preparation of brake lining materials for long time and relatively high temperature performance under dry conditions [12]. The material was subjected to rain water spray to study the effect of climate conditions on the performance.

2. Materials and methods

2.1 Apparatus:

The molding of the prepared composite was carried out using Hot pressing Device, model MMP45 supplied from Laryee Technology under a pressure of 50 KN and temperatures between 140 and 150° C for 15 min.

The wear tests were carried out on a homemade instrument designed and tested following the ASTM G99 specification to furnish contact between the specimens and the rotating disc under specific load. Fig. 1 shows the wear test instrument. The instrument consists of fixed speed motor (740 rpm), and arm fixed vertically on the disc plane. The arm holds the load on its upper end and fixes the specimen on the disc at the lowest end. The instrument was designed to allow the wear testing under dry and wet conditions.

2.2. Materials:

Novolac resin was supplied by That-Assawari state company and used after grinding and mixing with the hardener, Hexamethylene - tetramine, HMTA. The reinforcement materials included fillers such as bentonite, alumina and iron turning. The technical properties of the materials are given in Table 3.1. Other components were short (diameter, 7-8 μ) carbon fibers after cutting into small pieces, 4-6 mm. Other ingredients like ZnO, zinc stearate, graphite , alumina were general purpose materials. Iron turnings were analyzed at the State institute for Engineering Examinations and found to have the composition shown in Table 1.

The composition of bentonite can be seen in Table 2. The rain water employed was obtained from a district to the south – east of Baghdad and is characterized by pH of 7.5, Total suspended solids, 7 and total dissolved solids of 43, turbidity, 11.

2.3. Methods:

2.3.1. Preparation of composites:

The composites were prepared with the modified dry method using weight ratios, Ψ , in the range 60-90%. The weight ratios of the materials are given in Table 3.

The composites were molded under relatively high temperature and pressure. The main preparation steps are:

2.3.1.2. Base material preparation:

Novolac masses were first ground to pass 75 μ m. Similarly, the HMTA was ground mixed with the Novolac powder together with cross linking accelerator, ZnO, at a maximum content of 1%.

The base material (Novolac + HMTA + ZnO) was then mixed the other ingredients at the weight ratio specified in Table 3, using electrical mixer at high speed to aid homogeneity.

The mixing of materials was aided by forging between steel cylinders already heated to $40-45^{\circ}$ C controlled by water bath. The materials were returned to the rolls for further homogenization for 1-2 min to end up with thin flakes of the mixtures. The materials were ground again and sieved to ensure homogeneous material with particle size less than 400 µm. The ground material was then mixed with carbon fiber.

Using the above mentioned conditions of molding homogeneous disc specimens could be obtained with dimensions of 4.5 cm in diameter and 1.7 cm thickness.

For the wear tests the specimens were smoothed with silicon carbide paper grade.

3. Results and discussion:

3.1. Preparation methods evaluation:

The utilization of forging for the change of mixtures into small pieces leads to overcome the weak ability of Novolak on wetting particles surface. This may not be possible with the classical dry method of sample preparation. Thus, rolling is an essential pre-maturation step to help the establishment of strong binding forces between Novolak and the fillers.

3.2. Wear rate measurements under dry operation conditions:

3.2.1: Effects of friction materials

The wear rate was estimated and plotted versus the percentage contents of the various compounds. Fig. 2 shows a decrease in the weight loss from the composite body with the increase of the reinforcement materials content. Besides, very limited deformation could be observed on the surface of the material. This can be attributed to the limited movement of the sliding contact with the cast iron disc after reinforcement with the friction modifiers.

The decrease in the weight loss refers to the increase in wear resistance of the Novolac composite in comparison with wear rates in the range of 892.5×10^{-9} g/cm for the non-reinforced Novolac resin accompanied with an increase in the plastic deformation and break-ups in the Subsurface layers [13]. The fibrous and particle reinforcement materials that are randomly distributed will prevent the deformation of the material during the loading due to their high relative hardness. The hardness of the reinforcement material will improve the resistance of the material and the coefficient of thermal expansion [14].

3.1.2. Effect of the hardener content:

The hardener usually supplies the nitrogen necessary for the cross-linking and consequently generates ammonia at an extent of 95% and the formation of intermediate compounds. The higher the hardener content the more will be the nitrogen that accelerates the cross linking. Cross linking will result in trapping volatiles and gases within the polymer body which leads to increase in the porosity of the Novolac body. The porosity will in turn cause the material to be brittle and thus, a decreased mechanical strength [15, 16]. The behavior is clearly shown in Fig. 3, where the wear resistance decreases with the increase of the HMTA content from 14 up to 22%. The wear rate has increased by 3.88×10^{-9} g/cm for novolac and by 7.169×10^{-9} g/cm for novolac composites.

The weight ratio of the reinforcement materials refers to that fraction of material that plays such important role in the improvement of the composite mechanical properties below a content of 80%. Beyond this content, the base material will act as a binder for the other components. The wear rate of the material with 60-70% weight ratio showed dramatic increase in the wear rate from $3.58*10^{-11}$ gm/cm for the 60% ratio to $234.406*10^{-9}$ gm/cm for the 80% ratio and to $299.7*10^{-11}$ gm/cm for the 90% weight ratio as can be seen in Fig. 3. The low binding extent of the components, therefore, will result in a weak composite with large number of pores [17]. Effect of carbon fiber

Fig. 5 indicated a clear improvement in wear resistance of the composites with (HMTA=14%, ψ =60%) as the carbon fiber content increases. The increase in the carbon fiber content improved the compressibility and hence kept the contact area very limited. This makes the transvers shearing process to involve only the soft surface layer and therefore lowered the ability of the rotating disc to remove the surface material of the composite [18]. This behavior may be expected due to the good physical and mechanical properties of carbon fibers when they are combined with polymeric composites as mentioned earlier [19].

Effect of operating conditions

The severity of loading, sliding time and the sliding distance appeared to have a pronounced effect on the wear rate results because of the expected changes that may occur within the two sliding bodies. Effect of Load:

Figs. 6-8 show the wear rates of specimens with weight ratios of reinforcement materials of 80-90%, HMTA of

14-22% and carbon fibers of 1.5-3%. The wear rate increases as the load increases over the range of the used sliding distance. This is related to the variation of the kink bands. The formation of such bands during the crystal slip along the main polymer chain when they are subjected to axial compression are dangerous as they will largely control the hardness and brittleness of the composites as a result of the formation of longitudinal cracks.

When the lateral tensile forces, generated from the Poisson expansion, increase on the internal tensile force resistance. Under the applied stress on the specimen surface towards the weak places, the cracks will merge together or with wear lines will result in dismantling and removal of thin layers from the composite body, where they are easily removed with the direction of sliding to form wear debris. The wear debris is the result of surface fatigue at the surface layer of the composites during the sliding [20].

Generally, the curves shown in Figs. 6-8 indicated three distinct regions: the first covers the load range 5-10 N, the mild wear region; the second cover the range of 10-15 N and called the transition wear region; and the severe wear region for load range 15-20 N [21]. The variation of the appearance of these regions can be explained on basis of the lateral flaking mechanism. The composite surface has two types of irregularities: resinous and fibrous. At low load values (5 N), the resinous type are removed from the surface due to the ploughing taking place by the groves of the metallic surface on which the composite is sliding. With the increase in the load value to 10 N, the wear rate decreases due to the removal of fibrous irregularities that are characterized with high sheer resistance and high strength, by ploughing. At this stage the initial cracking commences at the end of the fibers and at the interfaces between base material and the particulate fillers, because such locations are considerably the weakest points within the composite body. Also such regions are, supposedly, full with microvoids. Thus, the formation of cracks and their growth will be mostly subsurface, i.e. below the contact region of the two surfaces. With the progress of the sliding process and increase in the compressive sheer loads, the wear rate will increase as a result of the connection between the cracks formed at low load values to end up with large cracks. The cracks will grow and bring about the dismantling of particle flakes. Further, the increase in the load will lead to peel -off of the matrix due to the thinning and random fracturing of the fibers. Thus, the wear debris will consist of matrix particles and broken fibers with irregular shapes. It is worthwhile that the use of filler material of short fibers and particulate in the composite will result in distribution of separate microcracks within the weak points in the composite body [22, 23].

Effect of sliding time on wear:

The effect of sliding time on the wear rates were studied for various percentages of the reinforcement material, the hardener, and the carbon fiber contents in the composites for two sliding distances (Figs. 9-11). Composites with relatively high weight ratio (80-90%) showed a wear rate of $587.3 - 736.2 \times 10^{-9}$ hm.cm⁻¹ at a sliding time of 2 mins and 38.87×10^{-9} for the material containing 22% of the hardener. This can be referred to the ploughing of the tops of the irregularities of the polymeric surface accompanied with spread of polymeric debris within the grooves of the mineral surface. The spread will reduce the tilting of the grooves that will be filled with such debris materials as the ploughing is progressing and increase of sliding time. The real contact area will increase accordingly. Consequently, the compression stress will be low at the contact points and thus, a reduction in the wear rate will occur. Such a decrease was clearly noticed at sliding time of 10 mins. For a sliding time of 15 mins, the wear debris particles were larger in size exerting a pressure on the irregularities to end up with increase wear rates. The temperature will rise at the contact surface with longer sliding time which lead to softening of surface irregularities and hence a plastic creep. This results in a decrease of the wear rates.

Other specimens showed similar behavior of the transfer wear within 2-4 minutes due to the removal of large hills of resinous, fibrous and particulate types as shown above. With the increase of sliding time the wear rate changes into the mild one [22, 23].

Effects of weight ratio of constituents:

The practical tests (Figs. 12-14) revealed the dependence of wear rates on the sliding distance which is agreement with the results of Lhymn et al. [22]. All the composite specimens show an increase of wear rate with increase in the sliding distance as a result of the increase of the work performed by the friction force by the increase of the distance and the change some of the energy into heat. It is worth mentioning that such a result contradicts with the behavior of the metals. However, the dissipation of heat through the surface irregularities at high sliding speeds is less than that at the low speeds that result in molecular contact between the protrusions of the two sliding surfaces. This will result in increase of the force necessary for cutting the points of contact. Such increase will be higher than when the bonding between metal particles themselves. Besides, the increase in sliding speed will result in the formation of oxide layer on the surface. Reference to Fig. 1, the Novolac composites with 60% weight ratio of reinforcement materials and 14% of the HMTA, when used as friction pads will be close those of passenger cars as quoted from some recent references [24]. The difference is as low as

about 7.606 – 18.806 x 10^{-9} in comparison with wear rates of small cars as carried out in this study (94.386 x 10^{-9} gm.cm⁻¹).

Some authors gave lower estimates for the heavy vehicles [25] that indicate reasonable wear resistance for the Novolac based brake pads.

Effect of water contaminants

The continuous spraying of the specimens with rain water for several times will result in lowering the wear resistance by 3.798×10^{-7} , 41.22×10^{-7} , and 269.727×10^{-7} gm.cm⁻¹ for novolac composites with 60%, 70% and 80% weight ratio of the reinforcement materials, respectively (Fig. 13). The wear rate was 380.464×10^{-7} , 639.83×10^{-7} , and $949 \times \times 10^{-7}$ gm.cm⁻¹ for composites with HMTA percent of 14%, 18%, and 22% (Fig.14). For carbon fiber reinforcement the wear rate values under rain water spray are given in Fig. 15. The brake performance was found to be lower by the saturation of the pads with rain water even with greater pressure on the pedals. The pollutants of water as well as oils will reduce the contact between the irregularities of the sliding surfaces by isolating them from each other [22]. Further, rain water may accumulate inside the composite body by diffusion and capillary action, and microcracks [26].

Conclusions

The present work is an attempt to design and prepare brake pads based on Novolac and reinforced with ceramic materials. The composition of the pads is an essential parameter in determining the performance. The HMTA content of 14% gave the highest wear resistance. Carbon fiber is an effective constituent in the brake pad performance.

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Tables:

Table 1: The composition of iron turnings

Element	Wt%	Element	Wt%	Element	Wt%	Element	Wt%	
C%	0.051	S%	0.011	Al%	0.031	W	0.017	
Si%	0.287	Cr%	0.146	Co%	0.012			
Mn%	0.653	Mo%	0.028	Cu%	0.132	Fe	Balance	
P%	0.002	Ni%	0.115	V%	0.0005			
Table 2. The composition of hertonite								

Table 2: The composition of bentonite

Component	Wt%	Component	Wt%					
SiO ₂	56.77	K ₂ O	0.6					
Al_2O_3	15.67	P_2O_3	0.65					
Fe ₂ O ₃	5.12	SO_3	0.59					
CaO	4.48	Cl	0.57					
MgO	3.42	L.O.I	0.49					
Na ₂ O	1.11	C0.6	0.56					

Table 3: The composition of the prepared specimens.

Components	Material	Sample									
		components Weight ratio (%)*									
		1	2	3	4	5	6	7	8	9	10
Binding material	Novolac	33.4	25	16.6	8.3	33.4	32	30	33.4	33.4	33.4
	HMTA	5.4	4	2.7	1.4	5.4	7	8.5	5.4	5.4	5.4
	ZnO	1.2	1	0.6	0.3	1.2	1	1.2	1.2	1.2	1.2
Internal polymer	Zinc Stearate	0.7	0.8	0.88	1	0.7	0.7	0.7	0.7	0.7	0.7
lubricant											
Reinforcement	Used Brake	4.7	5.4	6.2	7	4.7	4.7	4.7	4.7	4.7	4.7
material	Pads										
	Fe	3.34	3.9	4.4	5	3.34	3.34	3.34	3.34	3.34	3.34
	Carbon	2	2.3	2.66	3	2	2	2	2	1.7	1
	Fibers										
Fillers	Bentonite	16	18.5	21.3	24	16	16	16	16	16	16.5
Friction Improvers	Alumina	30	34	39.1	44	30	30	30	30	30	30
	Graphite	0.7	0.8	0.88	1	0.7	0.7	0.7	0.7	0.7	0.7
	SiC	3.34	3.9	4.4	5	3.34	3.34	3.34	3.34	3.34	3.34

FIGURES:



Figure 1: The wear test instrument



Figure 2: Wear rates of novolac in comparison with novolac composites: r=3.6 cm; time = 30 s; and load = 5 N.



Figure 3: Effect of hardener material on the wear rates.



Figure 4: Weight ratio effect on wear rate.



Figure 5: Effect of carbon fiber content on wear rate, Load = 5 N; Sliding Distance = 334.768 m; and time = 30



Figure 6: Dependence of wear rate on the load applied for composites with various HMTA contents under dry conditions, time = 2 mins; and sliding distance = 241.77



Figure 7: Dependence of wear rate on the load applied for composites with various weight ratios of reinforcement materials under dry conditions, time = 2 mins; and sliding distance = 241.77 m.



Figure 8: Dependence of ear rate on the load applied for composites with various carbon fiber contents under dry





Figure 9: The dependence of the wear rate of Novolac composites of various weight ratios with the sliding time under dry conditions at sliding distance of 241.77 m and a load of 10 N.



Figure 10: The dependence of the wear rate of Novolac composites of various HMTA contents with the sliding time under dry conditions at sliding distance of 241.77 m and a load of 10 N.



Figure 11: The dependence of the wear rate of Novolac composites of various carbon fiber contents with the sliding time under dry conditions at sliding distance of 241.77 m and a load of 10 N.



Figure 12: The dependence of the wear rate of Novolac composites of various HMTA contents with the sliding distance under dry conditions at sliding time of 2.0 mins and a load of 10 N.



Figure 13: The dependence of the wear rate of Novolac composites of various weight ratios of reinforcing materials with the sliding distance under dry conditions at sliding time of 2.0 mins and a load of 10 N.



Figure 14: The dependence of the wear rate of Novolac composites of various carbon fiber contents with the sliding distance under dry conditions at sliding time of 2.0 mins and a load of 10 N.



Figure 15: The dependence of the wear rate of Novolac composites of various weight ratios of reinforcing materials under dry and wet conditions at sliding time of 2.0 mins and a load of 10 N and a sliding distance of 241.77 m.



Figure 16: The dependence of the wear rate of Novolac composites of various HMTA contents under dry and wet conditions at sliding time of 2.0 mins and a load of 10 N and a sliding distance of 241.77 m.



Figure 17: The dependence of the wear rate of Novolac composites of various carbon fiber contents under dry and wet conditions at sliding time of 2.0 mins and a load of 10 N and a sliding distance of 241.77 m.

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