

Investigation of Tribological Properties of Brake Friction Materials Developed from Industrial Waste Products

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Abstract

Various international initiatives on environmental issues and the need to protect the environment are promoting the use of industrial waste in a variety of applications, including automotive brake pads. These studies show that the reuse of industrial waste can help to reduce the environmental impact. The development of environmentally friendly and cost-effective composites for use in a variety of engineering applications is the need of the century. The use of industrial waste in composite production is a possible solution for both problems. In this study, the potential use of talc, quartz and ceramic waste fine fire clay as a friction modifier in brake friction materials and its performance properties in accordance with industry requirements were investigated. In this study, the tribological properties of friction materials were investigated using pin-on disc. The tribological, physical and mechanical properties of the brake pads were measured, and the friction surface morphology was investigated by scanning electron microscopy. According to the results obtained, the highest specific wear rate was observed in the FM₃ sample. The FM₂ sample with the highest hardness and average friction coefficient showed the lowest wear. FM₄, FM₅ and FM₆ samples with high talc and quartz content exhibited low coefficient of friction characteristics compared to other samples.

Keywords: Industrial waste; Eco-friendly friction materials; Friction; Wear.

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1. Introduction

BFM (Brake Friction Material), which is defined as the brake friction material in vehicles, is a part of the brake system and is directly related to safety. They are subjected to different sliding speeds, brake pressure and temperature conditions. They must exhibit a stable coefficient of friction, a low wear rate, and thermal stability under braking conditions [1,2]. It is impossible to achieve these properties with a single component. Many BFMs have been produced to obtain the desired tribological properties [3]. The tribological performance of the produced friction composite is greatly influenced by the amount and type of components categorized as functional and inert filler, binder, reinforcing element, friction modifier, lubricant and abrasive [4–8]. The combination of behavior and mechanical properties, along with the formulation of

friction materials, plays an important role in achieving the best performance. Under braking conditions, this combination should provide qualities such as stable friction, good wear resistance, low noise, environmental friendliness, resistance to water and chemicals [9–12].

In addition to the economic cost, environmental issues are also a major concern when it comes to the wear of brake friction materials. Due to the intense formation of industrial and agricultural wastes, their use in the manufacturing industry becomes mandatory. Appropriate waste management and use are now essential for both industrial sustainability and the environment. Industrial ceramic wastes, which are formed in large quantities every year, are used in different industries or some of them accumulate without any benefit. This causes environmental pollution and ecological

problems to a great extent. Also, the use of various types of industrial ceramic waste in useful applications, such as fine fire clay (FFC), is not common. Recently, the use of ceramic waste has attracted attention in line with efforts to address environmental concerns [13–15]. In addition, it can contribute to cleaner production and increase industrial sustainability by reducing raw material costs while improving mechanical and tribological properties.

In previous studies, the effects of different raw materials on the tribological performance of brake pads were investigated and reported. Kristkova et al. [15] reported that the inclusion of vermiculite (natural clay mineral) improved the stability and durability properties of friction composites at elevated temperatures. Singh et al. [13] investigated the effect of wollastonite on brake friction composites and observed that powdered wollastonite reduces the density of friction composites and increases their stiffness and improves the friction coefficient. Aranganathan et al. [16] reported that aramid fiber concentration positively affects the friction and wear properties of composites, resulting in a stable coefficient of friction, fade and wear resistance. Jang et al. [17] investigated the interaction of abrasive particles with a solid lubricant. Timur et al. [18] investigated the effect of waste marble material in brake composite content and observed the use of powdered marble as a filling material instead of barite in friction composites. Tomášek et al. [19] stated that the addition of abrasive formulation (Al_2O_3) to the lining composition contributes to reducing the wear rate and improving friction performance. Bijwe et al. [20] evaluated the tribological performance of friction composites containing micro and nano abrasives (Al_2O_3 , SiC and SiO_2) and noted that nanofillers significantly improved the overall performance behavior. Sun et al. [21] investigated the effect of various abrasive silicon dioxide particles in non-commercial composite friction materials. He stated that the particle size has a significant effect on the friction mechanism with the higher contact films formed by these composites. Peng et al. [22] studied the effect of some abrasives on the friction properties of Cu-doped metallic brake pads at different braking speeds. On the other hand, Manoharan et al. [23] evaluated the effects of the red mud/iron sulfide combination on the tribological properties of brake friction composites on a Chase tester. He reported that this formulation was consistent in terms of friction with increased wear resistance. According to Dadkar et al. [24] aramid fibers and polymer matrix, Hee et al. [25] ceramics and polymers, Bahari et al. [26] rice husk dust, Qi et al. [27] walnut shell powder, Rashid et al. [28] sugar palm fiber and phenolic matrix and Aranganathan et al. [16]. Many friction materials, such as the new type of copper-free material, have been used in various ways to determine their influence on the tribological behavior and mechanical properties of brake pads. The results of the research showed that the added components were effective on the coefficient of friction, wear rate, hardness and density values. In the literature, in the evaluation of friction and wear behavior of brake friction composites, the surfaces of the pads have been examined and the microstructure properties of the friction surfaces have been evaluated [18,29–33]. The current trend in the friction materials field is to use industrial or agricultural waste as a raw material source for composite develop-

ment. In the production of composite materials from waste to create brake pads, the attractive performance-cost ratio, and the idea of exploring possible combinations of different waste materials provides motivation. FFC consists of 50 % mullite, 5 % cristobalite, 5 % corundum and 40 % amorphous phase. FFC is produced using high alumina-containing clay, which is calcined at 1400 °C and then ground to different grain sizes. FFC can be used as an alternative raw material source due to the specified characteristics [34].

In this direction, the present study aims to evaluate and explore the potential of using talc, quartz and FFC as friction modifiers in environmentally friendly brake friction materials. Six formulations of BFM were developed, in which only the type of abrasive component changed. The density and hardness values of the developed samples were measured. These specimens were evaluated by general friction performance testing through pin-on disc equipment. The friction behavior and wear rate properties of the samples were investigated. In addition, scanning electron microscopy (SEM) analysis was performed to understand the friction characteristic.

2. Experimental

2.1 Specimen Processing

Six formulations of BFM have been developed, sharing 5 main components that make up 70 % of the total composition weight. All formulations developed contain an essential ingredient to ensure a minimal tribological performance that allows for proper evaluation. The remaining 30 % was attributed to talc, quartz and FFC components to evaluate the effect of BFM on friction level. The procedure followed in this study was determined according to the parameters commonly used in the literature. [35–37]. Table 1 shows the chemical analysis results of talc, quartz and FFC. Chemical analyzes were carried out at Karabük University Materials Research and Development Center (MARGEM). Varying ingredients were used to regulate the friction coefficient of the pad to achieve smooth braking. The weight percent of the formulation ingredients produced is detailed in Table 2.

Table 1. Chemical analysis results of talc, quartz and FFC (w/%).

Element	Talc	Quartz	FFC
MgO	34.1	3.4	3.6
Al_2O_3	1.3	1.6	25.3
SiO_2	61.3	94.1	65.06
CaO	2.5	0.08	2.2

In the production of the BFM samples, the components were mixed for 40 min using a V-shape mixer. Samples with a diameter of 25.4 mm and a height of 12 mm for each mixture were prepared by pre-forming for 3 min under 10 MPa pressure. Then, it was subjected to 10 MPa pressure in the hot press and hot compression at 150 °C for 10 min. The hot pressing was interrupted 3 times to remove volatiles and moisture. The produced BFM samples were post-cured for 10 h at 150 °C, similar to the cycle used for com-

mercial brake pads, and then cooled at room temperature. The samples were then processed with sandpaper and became usable in

tribological tests. In Table 3, the production process of BFM samples is detailed.

Table 2. Identification codes and compositions of fabricated friction materials

Classification	Ingredients	Sample code / Content in w/%					
		FM ₁	FM ₂	FM ₃	FM ₄	FM ₅	FM ₆
Binder	Phenolic resin	10	10	10	10	10	10
Fiber	Glass fiber	15	15	15	15	15	15
Reinforcement	Barite	25	25	25	25	25	25
	CaCO ₃	10	10	10	10	10	10
Lubricant	Graphite	10	10	10	10	10	10
Friction modifier	Talc*	5	5	10	10	15	15
	Quartz*	10	15	5	15	5	10
	FFC*	15	10	15	5	10	5

*Varying ingredients

Table 3. Details of fabrication process

Procedure	Processing conditions
Dry-mixing	Mixer – 10 min ⁻¹
	Duration – 40 min
Pre-forming	Compression pressure – 10 MPa
	Molding temperature – Room temperature
	Molding time – 3 min
Hot compression	Compression pressure – 10 MPa
	Molding temperature – 150 °C
	Molding time – 10 min
Post curing	Curing temperature – 150 °C
	Curing time – 10 h

The generated BFMs were evaluated using Scanning Electron Microscopy SEM/EDX (Leo-1430VP). The density of BFMs was determined by the ASTM D792 method [32] by weighing on a precision balance and using the water displacement method. The hardness of the samples (Brinell scale) was measured on the TRSN-D-SPL-2018/335 digital hardness tester (Fine Manufacturing Industries) according to ASTM-E18. Hardness values were determined using a 1.59 mm diamond tipped B scale/steel ball under a 10 kgf load. Before the hardness measurement, the surfaces of the samples were prepared with SiC sandpaper. Five readings were performed at different positions of the samples.

2.2. Tribological characterization

Tribological characterization of brake friction materials was performed with a pin-on-disc (PoD) tribometer. The gray cast iron disc, common in vehicles, was used as the counter surface. Before the tests, each disc was polished with sandpaper. The test specimen was mounted on the load arm and pressed against the flat surface of the rotating disk. Each BFM was subjected to three PoD tests. Before the tests, a run-in phase was performed for the actual contact surface. PoD tests were carried out under a load of 79 N,

with a sliding speed of 7 m/s, for 30 min and the temperature was increased from 50 °C to 350 °C. These parameters were chosen to achieve mild wear conditions [29,38] typical of standard vehicle braking actions. Although the PoD tribometer does not produce real braking states, it has been stated that this test is useful for obtaining information about wear mechanisms and examining their tribological behavior [39,40]. The friction coefficient was recorded continuously during the PoD tests.

The wear of the samples was evaluated by calculating the specific wear rate and the weight loss per mm² of the sample during the tests. The mass of the sample was subtracted before and after the friction test, and it was measured with an electronic balance with an accuracy of 10⁻³ g via mass loss. Wear was assessed by calculating the specific wear rate (cm³/Nm) according to equation (1), following the following equation:

$$\text{Specific wear rate} = \frac{\Delta W}{dxFxS} \quad (1)$$

where ΔW is the mass loss (g), d is the density of the sample (g/cm³), F is the friction force and S is the sliding distance (m). Wear measurements were taken after each test.

3. Results

The physical and friction characterizations of the produced BFMs are summarized in Table 4. When the results were examined, it was determined that the brake pad with the highest hardness was FM₂ (81.1 HB), and the brake pad with the lowest hardness was FM₆ (54 HB). As the talc ratio increased, the hardness of the material decreased. When the density values were examined, it was seen that the samples with the lowest density were FM₁ and FM₃ samples. The density of the samples decreased with the increase in the FFC ratio. The mass loss and specific wear rate were highest in the FM₃ combination. Although the mass losses of FM₂ and FM₅ samples are very close to each other, the lowest specific wear rate was realized in the FM₂ sample.

Table 4. Test results of the fabricated friction materials

Sample	Density (g/cm ³)	Hardness (HB)	COF (μ)	Spec. wear rate (x10 ⁻⁵ cm ³ /Nm)	Weight loss (g)
FM ₁	2.27	75.9	0.38 ±0.031	5.60	0,019
FM ₂	2.31	81.1	0.42 ±0.038	3.70	0.013
FM ₃	2.28	58.2	0.35 ±0.016	22.40	0.077
FM ₄	2.32	66.0	0.21 ±0.026	5.14	0.018
FM ₅	2.31	65.7	0.23 ±0.018	3.93	0.013
FM ₆	2.30	54.0	0.25 ±0.009	5.67	0.019

The pin-on disc friction test results of the produced samples are shown in figure 1. When the graph was examined, it was seen that the FM₁, FM₂ and FM₃ samples exhibited a friction coefficient of more than 0.3. The common feature of these three samples is the high percentage of FFC used as a friction modifier in the structure. As the ratio of FFC in the structure decreased, a decrease was observed in the friction coefficients. The friction coefficients of the FM₄, FM₅ and FM₆ samples were found below 0.3. In the FM₄ sample, which has the lowest coefficient of friction, the least amount of FFC was added.

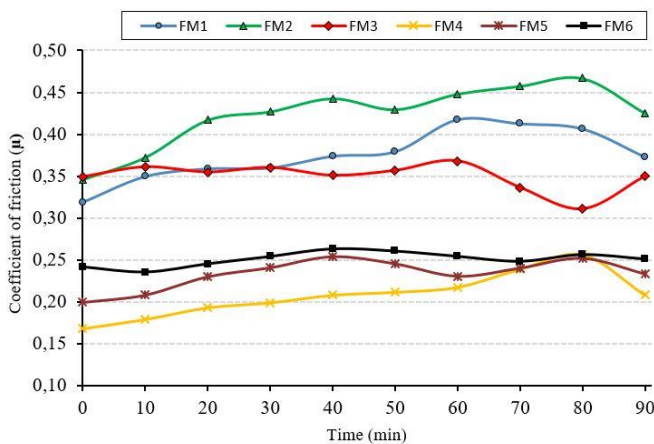


Fig. 1. Mean coefficient of friction for each formulation

In Figure 2, the specific wear rate of the samples produced as a result of the friction wear test is shown. The highest specific wear rate was clearly seen in the FM₃ sample. It can be thought that the proportions of the materials used in the FM₃ sample do not provide a suitable combination. The lowest specific wear rate was obtained in the FM₂ sample.

SEM images of the samples are shown in Figures 3. In the FM₁ sample, graphite and glass fibers are embedded in each other, but especially FFC is irregular and scattered on the surface. Since the curing temperature was 150 °C, quartz crystals did not develop, and although talc plates appear together with graphite in some areas, they do not seem to be dispersed sufficiently to form packing

in the structure. The structure actually appears to have coalesced at low temperatures due to the effect of the phenolic resin against heat, but the phases do not appear to be formed. In order for ceramic structures to show strength and have an effect on the structure, higher temperatures must be reached. The same structures are observed in FM₂, but the ratio of compacted structures and scattered parts is less. It appears to be more regular in the FM₂ sample than in the FM₁ sample. Talc and graphite structures are more on the surface, and irregular ceramic particles are more embedded in this structure. In addition, it can be seen that the glass fibers are broken within the structure, not whole. It is seen that there are crystal voids in the FM₃ sample, although not as many as in the FM₁ sample. Graphite plates were not observed and the fibers remained intact. It can be seen that the structure is slightly more regular in the FM₄ sample. The most stable structures and samples without crystal dislocations are the FM₅ and FM₆ samples.

It has been reported in the literature that an increase in hardness generally results in an improvement in the wear resistance of the brake friction material [41]. The hardness of the brake pad samples increased due to the increase in SiO₂ content. The high amount of quartz in the FM₂ sample content supported the increase in density and hardness with the formation of ceramic phases in the internal structure. The wear rate was quite low due to its medium friction coefficient. The crystal dislocations in the structure of the FM₃ sample, its very low density and low hardness of 58.2HB resulted in an increase in the wear rate.

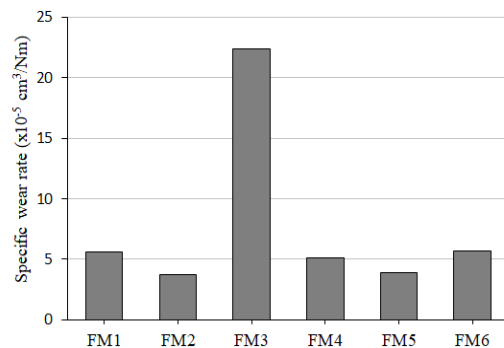


Fig. 2. Specific wear rate for each formulation

According to the findings, higher quartz content in the matrix increased wear resistance. Due to the presence of quartz and FFC, microstructural changes occur that lead to an improvement in hardness values due to increased wear resistance, resulting in improved wear behavior [42].

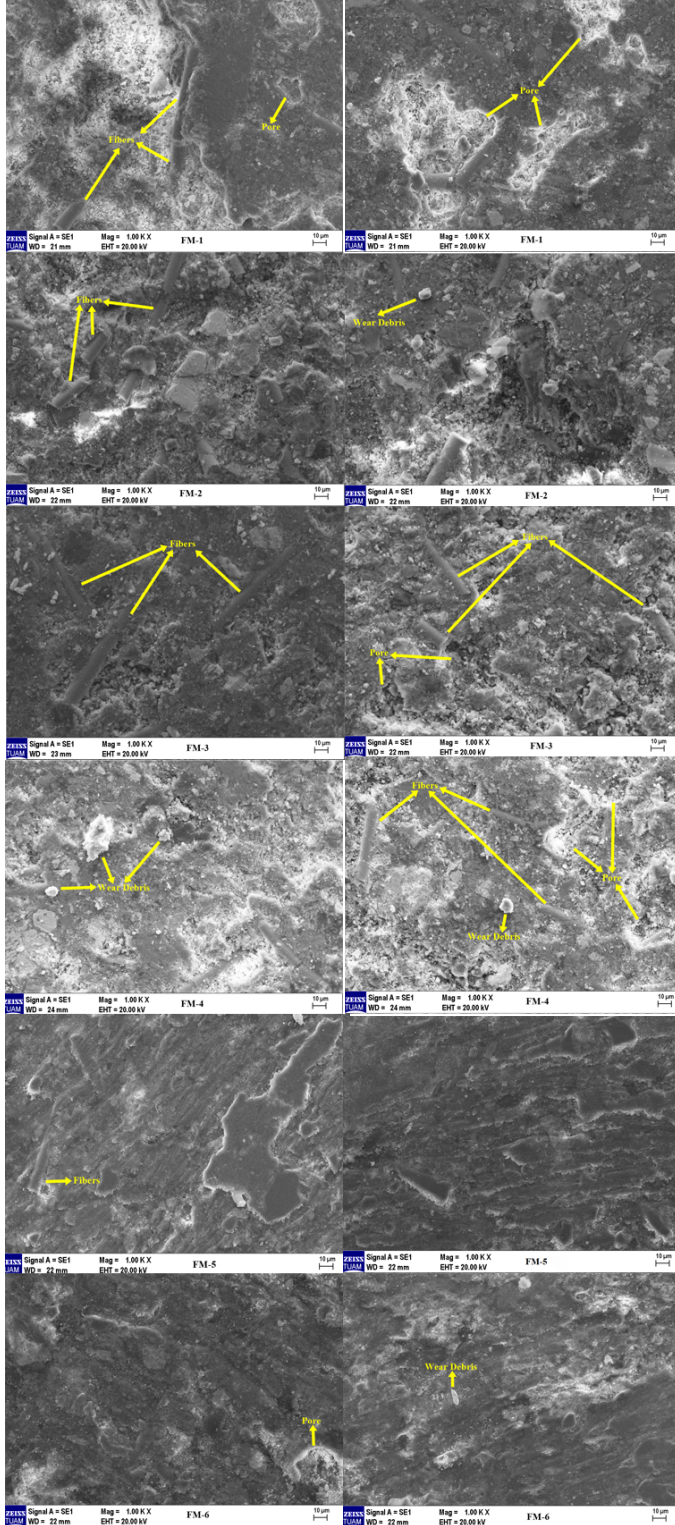


Fig. 3. SEM images of all samples

4. Conclusions

In the study, the effect of industrial waste FFC on the friction and wear performance of brake friction materials was examined experimentally using a PoD tribometer. The results were summarized as follows:

- When the friction coefficients of the samples were examined, an increase in the friction coefficient was generally observed as the FFC rate increased.
- It was observed that FM₃ and FM₆ samples, which have the lowest hardness values, had the highest specific wear rate.
- The density values of the samples were very close to each other.
- Industrial ceramic waste can be a good alternative as filler to reduce the cost of brake pads.
- The present study has shown a high potential for commercial applications of brake pads using talc, quartz and FFC as friction modifiers.
- In the first 4 samples, the FFC used as waste were observed to be scattered and unattached within the microstructure.
- When the talc content in the sample increases, the structure becomes more stable.
- It has been determined that the best rate of FFC in the structure is 10 %. When the ratio increased to 15 %, the highest specific wear rate occurred, as in the FM₃ sample, and when the ratio was reduced to 5 %, the friction coefficients dropped below 0.3.
- The present study has shown a high potential for commercial applications of brake pads using talc, quartz and FFC as friction modifiers.

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Conflict of Interest Statement

The authors declare that there is no conflict of interest in the study.

CRedit Author Statement

Furkan Akbulut: Investigation, Project administration, **Halil Kılıç:** Writing - original draft, **İbrahim Mutlu:** Supervision, **Fatma Sena Öztürk:** Investigation, Methodology, **Eray ÇAŞIN:** Investigation, Resources, **Mustafa Seyrek:** Writing - original draft, Writing - review & editing, **Abdullah Karaköse:** Investigation

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