Research Article

Mechanical and tribological investigation of jute fiber reinforcement in organic automotive brake pads and water repellency gain in natural fiber reinforced pads

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Abstract: Many studies have shown that the materials used in the composition of brake pads are directly related to friction, wear mechanisms and tribological behavior. Heat-resistant jute fiber is an organic fiber that can be added to the composition of brake pads that can achieve good braking. In the study, pads were produced by adding different amounts of jute fiber using phenolic resin with powder metallurgy method and the results were compared with pads without jute fiber. It was observed that adding 5% jute fiber in addition to aramid fibers used in brake pad manufacturing had positive effects on friction coefficients, friction fluctuations and wear mechanism. At the same time, water absorption values of natural fibers were reduced by 30% and water repellency was provided to the pads. According to sample A, the density of JF3 decreased by 25.4% and shear strength by 39%. The hardness value is at the standard value used in the market.

Keywords: Pad, Friction, Wear, Jute Fiber, Silane, Water repellency

1. Introduction

The rapid advances of technological developments nowadays especially in the automotive field and consideration of an environmentalist approach in the existing situation have led to usage of some environment-friendly materials in the compositions of automobile brake pads [1–5]. This way, brake pads started to be produced by using organic fibers and without using asbestos. The friction materials that are used in brake systems should satisfy the following conditions: high and stable friction coefficient, low fading, better reclamation and low wear in highly variable working conditions [6–8].

Brake pad formulations are made up of five categories as: binders, fillers, fibers, abrasives and lubricants [6,9]. Generally, phenolic resins are used as binders, while barium sulphate, calcium carbonate and clays are used as filler. Fibers such as: organic (Kevlar, jute), inorganic (lapinus), metallic (copper), ceramic (glass), natural (cellulose) and their various combinations are used in brake pads. Metal oxides and carbides are used as abrasives, whereas graphite and metal sulfides are commonly used as lubricants [10–12].

Nowadays, research is carried out on the effects of natural fibers as an alternative to synthetic fibers. Natural fibers have become a reason for choice instead of synthetic fibers as that have low density, low cost, high impact strength, easy production and no harm on the environment [9,13–15]. Likewise, with their biodegradability, they have found a prevalent usage area in several fields including the automotive, aviation and transportation industries. Components of natural fibers include cellulose, hemicelluloses, lignin, pectin, waxes and water-soluble substances [16,17].

Brake pads serve the function of reducing the velocity of or stopping a vehicle by converting the kinetic energy in the vehicle into heat energy [18–20]. For this reason, the properties and quantities of structures that will affect the friction performance of pads are a significant factor that will change the braking performance. Manufacturers have tried several different compositions to achieve properties such as increasing the high-temperature performance of the material under different

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braking conditions, increasing material strength and wear strength and achieving a stable friction coefficient [21–23].

It is known that usage of asbestos fiber leads to cancer, lung tumors and other medical problems. As usage of asbestos fiber has been prohibited, it has been considered to utilize different friction materials such as Kevlar, fiberglass and graphite to be used in brake pads. Pads manufactured by using natural fibers have high water absorbency.

This feature is a problem for all composite material production. This problem limits the use of natural fibers. Studies should be carried out to give the fibers water-repellent properties [24]. Different types of chemicals can be used to add water repellency.

In this study, jute fiber, which is a natural fiber, was used in different ratios to produce brake pads and subjected to various tests. The goal was determined as that the produced pads would have a structure not harmful to human health, their tribological properties and metallographic structures were then examined and compared to those of commercial pads. At the same time, water repellency has been provided to the pads by using Silane Agent.

2. Materials and Method

2.1. Materials and type of manufacturing

Composite materials for brake pads included Novolak-type phenolic resin (Polikem), barite and mica (Başer Madencilik), aramid fiber (DuPont Türkiye), alumina (Esan-Eczacıbaşı), quartz and magnesite (Kale Maden), iron powder (Sintek Toz Metalurji), copper powder (Mayda Toz Metal), graphite (Karabacak Madencilik), power rubber (ÜN-SAL Danışmanlık Gıda Tekstil) and jute fiber (Aksa Tekstil). The pads were manufactured by using different ratios of jute fiber, binders, filling materials, reinforcement materials and friction regulators (abrasives and lubricants). The ratios of the components were kept constant by mass and jute fiber was added as filling material by reducing the ratio of barite. The powders used in the manufacture of brake pads generally have high densities. On the contrary, the density of natural fibers is very very low. In studies, natural fibers are generally used at a maximum of 10% [2,25]. The reason for this is that the natural fibers used more are too much for the mixture. In addition, it is difficult to ensure product integrity in brake pads produced with excess natural fibers. For this reason, in the study, brake pads were produced in 4 different compositions by adding 1%, 3%, 5% and 7% jute fiber to the brake pad composition by mass (Table 1). In addition, brake pads without jute fiber were also produced and comparisons were made with brake pads with jute fiber additives.

The pads that were reinforced with jute fiber were named as JF, while those they were produced only by aramid fiber without adding extra fiber reinforcement were called A.

Table 1. Ratios of Brake Pad Components by Weight						
	A (%)	JF1 (%)	JF2 (%)	JF3 (%)	JF4 (%)	JF3-Silane (%)
Phenolic Resin	25	25	25	25	25	25
Powder Rubber	3	3	3	3	З	3
Barite	31	30	28	26	24	26
Mica	3	3	3	3	3	3
Aramid Fiber	5	5	5	5	5	5
Jute Fiber	-	1	3	5	7	5-S
Iron Powder	5	5	5	5	5	5
Copper Powder	7	7	7	7	7	7
Alumina	7	7	7	7	7	7
Quartz	3	3	3	3	3	3
Magnesite	1	1	1	1	1	1
Graphite	10	10	10	10	10	10

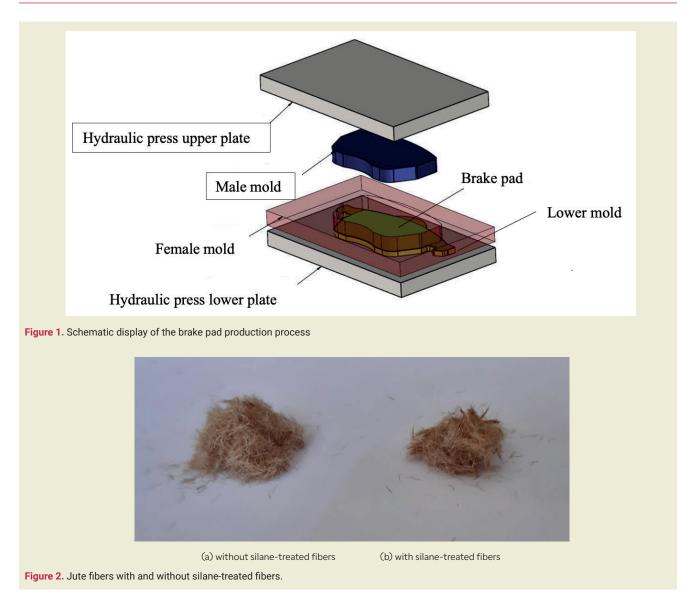
By using a KERN ACJ220-4M brand precision scale, the material components were weighted in the amounts specified in ▶Table 1 and made ready for mixing. The materials were mixed for 40 minutes using an M-TOPE-MS3040D brand laboratory mixer until a homogeneous structure was formed. The mixer was operated at 2000 rpm. The mixture was made ready for compression. Three pieces of each sample were produced, so that the experiments would have 3 replications.

A Hidrokar brand hot press with a 20 ton capacity was used for compression. As seen in **▶Figure 1**, the compression process was carried out using 192x120 mm molds at 180°C temperature and 21.5 MPa pressure [26]. The lower plate of the hydraulic press was mobile and heated, while the upper plate was fixed. The mold included a back plate below it to be compressed with the pad material. A female plate was placed above it. The pad material was laid out by hand in batches inside the mold. After each process of laying out, 10 min of compression was applied. The purpose of the gradual form of the process was to aerate the mold for each batch, release the gasses inside and prevent the gaps that could be formed inside the pad material. The brake pad sample was produced with the help of the mold shown in **Figure 1**, with dimensions of 106.34x62.5 mm.

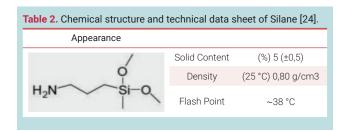
PROTHERM-PLF 120/10 brand industrial type furnace was used for sintering, which is the last stage of production. The samples were sintered at 180°C for 18 hours, allowed to cool at room temperature and the pad production process was completed [26].

2.2. Silane treatment of fibers

Karotect C1 type silane, used as a water-based and hy-



drophobic agent, was obtained from Varkim Industrial Chemicals company. The technical properties of silane are shown in **►Table 2**. When water-based silane is applied, it not only adds water repellency to the material, but is also environmentally friendly.

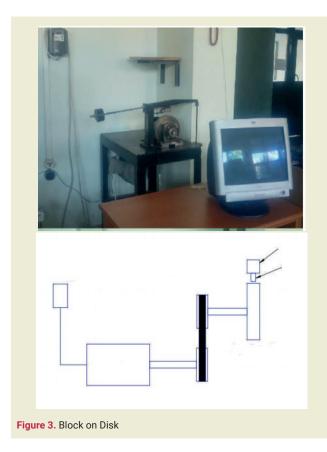


After the jute fibers were immersed in silane, they were kept at room temperature for 24 hours, ensuring that the silane was absorbed by the fibers as much as possible. They were then removed and a stripping process was performed to remove excess silane from the fibers. The drying process of the jute fibers soaked in silane was carried out by keeping them in an oven at 80°C for 24 hours. With this process, the fibers gained water repellency instead of water absorption. A new brake pad powder mixture was prepared using silane-coated jute fibers and changes in water absorption values, which is one of the biggest problems of brake pads produced with natural fibers, were observed. JF3-Silane has the same mixture percentages as JF3, the only difference being that the fibers in the mixture are coated with silane.

2.3. Tribological properties

A block-on-disk device was used for wear test (\triangleright Figure 3). In the experimental setup, the sample with dimensions of $10 \times 10 \times 30$ mm³ was placed onto a housing on the arm in a way that it would be over the disk rotating at a certain speed. The pressure between the disk and the sample was provided by the load connected to the arm. When the brake disk rotated, the sample was pressed onto the disk with the help of the load connected to the arm and wear was achieved with the movement of the disk. The block-on-disk device had an abrasive disk made out of 4140 steel with the diameter of 210 mm and hardness value of 54-56 HRc. To rotate this disk,

an electric motor with the properties of 1000 rpm and 2.2 kW was used. An inverter was used to set the rotation speed of the electric motor.



The experiment was applied in a way that 50 minutes of friction test would be applied on each sample satisfying the TS 555 standards under a speed of 3.14m/s and at a sliding distance of 9420 m. The pad sample was placed into the housing on the load arm with a connected weight. Afterwards, when the arm was released, contact was achieved between the pad sample and the disk. The weight placed onto the load arm was set to provide a pressure of 3MPa. This way, friction was achieved between the pad and the disk as soon as the disk started rotating. To obtain wear rate values, the experiments were carried out with 3 repetitions and the final friction coefficients were reached by taking the arithmetic averages of the results.

Additionally, the wear rate amounts in the samples were determined. For these amounts, the masses of the samples were measured by a precision scale before and after the experiments and the specific wear rate amounts were determined with Equation 1 below.

$$Ws = \frac{\Delta m}{L.\rho.Fn} \tag{1}$$

In the formula, Ws (mm3/Nm) is the specific wear rate amount, Δm (gr) is the sample weight loss, L(m) is the total distance covered, ρ (gr/mm3) is the density of the sample and Fn (N) is the load of the sample (7). The force applied on the pad is 300N.

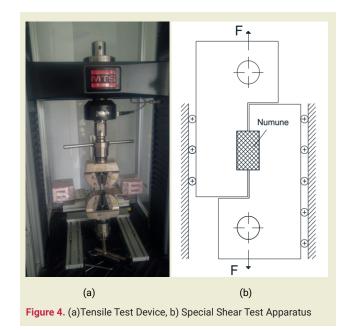
2.4. Mechanical, Physical and Chemical properties

Hardness tests were conducted at room temperature. Measurements were made from 5 different points on each sample. By taking the arithmetic average of the measured values, the hardness value for each sample was determined. The densities of the samples were determined by the Archimedes principle.

Water absorption experiments were conducted by using the ASTM D570-98 standards. The samples were kept in distilled water for 24h at 24°C. 3 pieces of each sample were freed of humidity by drying in an oven at 80°C for 24h. The dried samples were weighed by using a precision scale and then dipped in water. After 24h, the samples were taken out of the water, wiped and their dry weights were measured. The percentage water absorption ratios were calculated with equation 2 where m_{dry} is the weight dried in oven and m_{wet} is the weight kept in distilled water for 24h.

$$M(\%) = (m_{wet} - m_{drv}) / m_{drv} \times 100$$
(2)

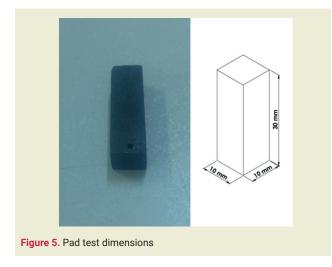
The shear tests of the samples were conducted with an MTS Systems Corporation-FXSA105A brand wedge device. An apparatus specifically prepared for the shear strength test was utilized (**>Figure 4**).



The samples were cut to 25x25x10 mm dimensions using a Yılmaz Brand Metal Cutting Machine (**Figure 5**). During shearing, increased load was applied on the samples in parallel to the stress direction at a rate of 5 mm/min. The load was increased until the material broke. Using the measured shear forces of the samples, shear strength was calculated with the formula τ =F/A.

3. Results and Discussion

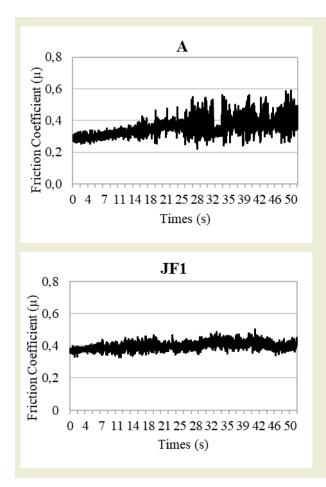
This section presents the data obtained from the wear

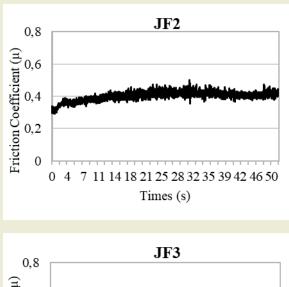


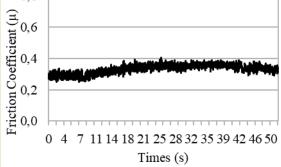
test and the specific wear rates calculated with these data. In addition to these, hardness, shear test results and water absorption are also interpreted in this section.

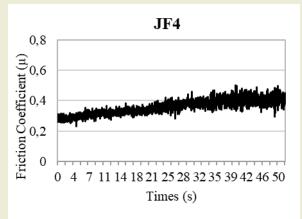
3.1. Tribological Test Result

In the wear tests, the block-disc device broke the record. The graphical representation of the obtained test data is presented in \triangleright Figure 6. Considering the friction-time graphs, it is reported that all the pads except the non-fiber reinforced pad (A) have a constant amount of sta-









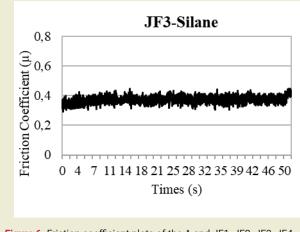


Figure 6. Friction coefficient plots of the A and JF1, JF2, JF3, JF4, JF4-Silane brake pads

bility. All other samples have changes in their stability. This is due to the increase in the brake pad components, which are stable at high speeds [27]. The lowest average change values were obtained as 0.37 in the JF3 sample and 0.386 in the JF3-Silane treated sample. It was concluded that the pads produced using natural fibers increased their friction coefficients similar to the study by Zhen-Yu et al [28] and Rajan et al [29].

The wear images of the pads are shown in \triangleright Figure 7. The specific wear values of each brake pad were calculated by using the shear test data and the specific wear ratios and average friction coefficient values are shown together in \triangleright Figure 8.

The average wear ratio values were in the range of 13.48-20.1x10⁻⁶mm³/Nm. The JF1 sample had the highest specific wear resistance. Increased ratios of jute fiber reduced the wear resistance and the JF3 sample had the minimum value.

The average friction coefficients were in the range of 0.368-0.487. The JF1 sample had the highest friction coefficient, as in the case of specific wear resistance, increased jute fiber ratios positively affected specific wear resistance and the lowest value was found in the JF3 sample. The reason for the increase in the friction coef-

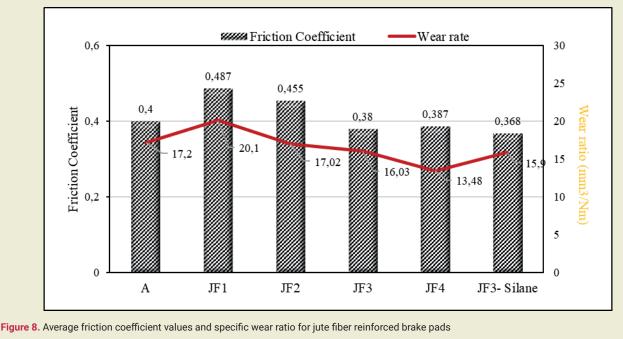
ficient up to JF3 was thought to be that the insufficient jute fibers could not resist the temperature and friction that occurred with contact with the disk which had high hardness. It is considered that, after the jute ratio of 5%, the jute reduced the friction coefficient due to its heat-resistant property and this way, it could resist friction. It is seen that the friction coefficients of JF3-silane pad and JF3 pad are close to each other. It is seen that the friction coefficient values of JF3-silane pad and JF3 pad are close to each other. When silane was applied to the fibers of the pad sample with JF3 composition, it was observed that there was not much difference in the specific wear ratios and average friction coefficient values.

3.2. Physical, Chemical and Mechanical Test Result

► Figure 9 shows the density and water absorption results of the samples graphically. The average density of the samples is between 1.818 and 1.283 g/ml. The density decreased as the jute fiber ratio increased. The hardness values in the brake pad samples reinforced with jute fiber decreased by 13-17% compared to sample A. Silane increased the density of the fibers and thus increased the pad density. The hardness values were measured on the Rockwell R scale and it was seen that the material hardness was not directly proportional to the fiber ratio, but different rates of fiber reinforcement af-



Figure 7. Pad Wear images (Respectively A, JF1, JF2, JF3, JF4, JF3-Silane)





fected the hardness values at different rates (▶Figure 9). The desired hardness value is 30-75 HRR. Lower pads cause rapid wear and higher hardness causes the disc to be damaged in a short time. All pads except A and JF4 pads comply with the standards. JF4 pad is 80.9HRR. It can be said that the reason for the high hardness of JF4 pad, which is expected to have lower hardness, is due to the fibers. The fiber ratio is high and there may not have been a homogeneous and complete mixture and the areas where the measurements were made may have misled us.

Considering the commercial brake pad values taken as reference, the shear stress of the pads should be higher than 2.3 MPa. Based on these values, considering the results listed in \triangleright Figure 6, all pads were produced with

appropriate shear strength values higher than this reference value (**▶Figure 10**). In addition, the shear stress values of samples A and JF1 are high, while FJ2 is the lowest. The silane treatment of sample JF3 caused a 30% increase in shear strength.

3.3. Water absorption

Natural fibers can be preferred in pad manufacturing as they increase mechanical properties and are environmentally friendly. In recent years, in the production of composite materials used in different fields, the use of natural materials has been emphasized. This issue is very important in terms of the environment and the benefit of future generations. Especially composites containing natural fibers have attracted great interest

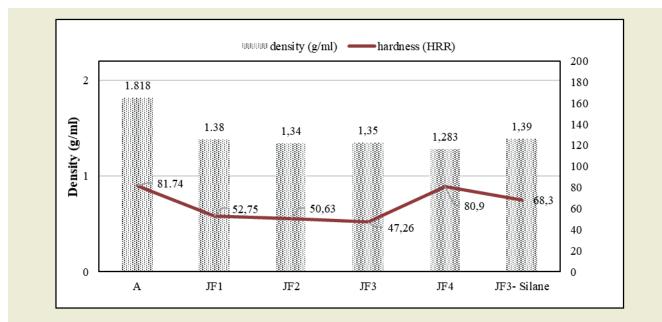
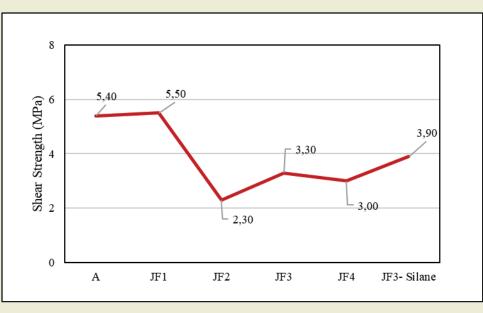
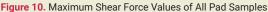


Figure 9. Density and hardness chart





in the world. These materials are completely biodegradable and do not cause emissions of carbon-based organic compounds such as petrochemicals. However, the hydrophilic nature of the fibers can negatively affect both their mechanical and physical properties [24,30,31].

Hydrophilic fibers used in axe production can be made hydrophobic, i.e. water repellent, to provide water repellency. For this reason, Silane was applied to 5% jute fibers in the JF3 mixture, which has the best performance in terms of all mechanical and tribological properties. JF3-Silane brake pad samples were produced.

Figure 11 shows the graph of water absorption rates

of brake pads kept in water for 1, 2 and 24 hours. As the jute fiber percentage increased, the water absorption rate of the brake pads also increased. While the water absorption percentages of the brake pads were very close to each other in the first 1st hour, they increased rapidly towards the 2nd hour. Brake pads without jute fiber have low water absorbency. It is seen that the FJ3-silane sample applied with silane prevents water absorption. It is very close to the values of the brake pad without jute fiber. Silane can provide water repellency to brake pads.

It is seen that the water absorption rate of the pads manufactured with jute fiber increases as the fiber ra-

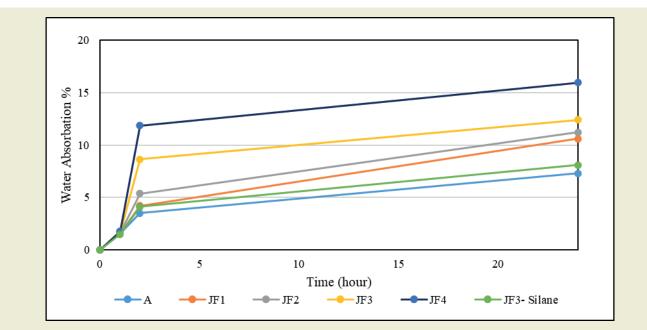
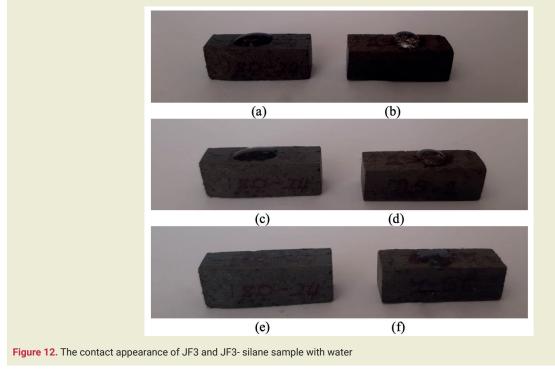
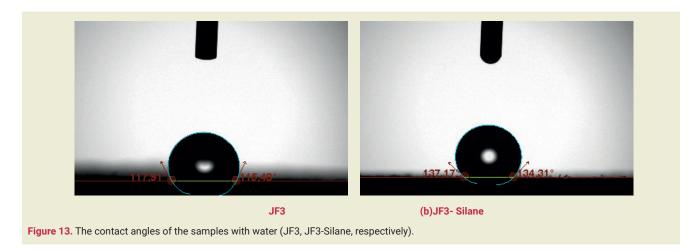


Figure 11. The graph of water absorption ratios in water for the all pads.





tio increases (**Figure 12**). At the same time, it is understood that the water absorption value of the sample with silane applied to the fibers is close to the values of the pad samples without fiber. The water repellency of the JF3- Silane sample increased by 10.32% compared to the JF3 sample.

Water drop moment (a) JF3 (b) JF3-Silane 20 seconds after adding water to the sample (c) JF3 (d) JF3-Silane 1 hour after adding water to the sample (e) JF3 (f) JF3-Silane

Figure 13 shows the contact angles of JF3 and JF3-Silane samples with water. It is seen from the study that the water repellency of the silane applied sample increased. As a result, it has been understood once again that agricultural fibers can be used in the production of composite pads. The fibers have been transformed into samples in a more hydrophobic form with the Silane agent and with this application, the water repellency of the samples can be increased and natural fibers can be used in the linings.

3.4. Surface Morphology

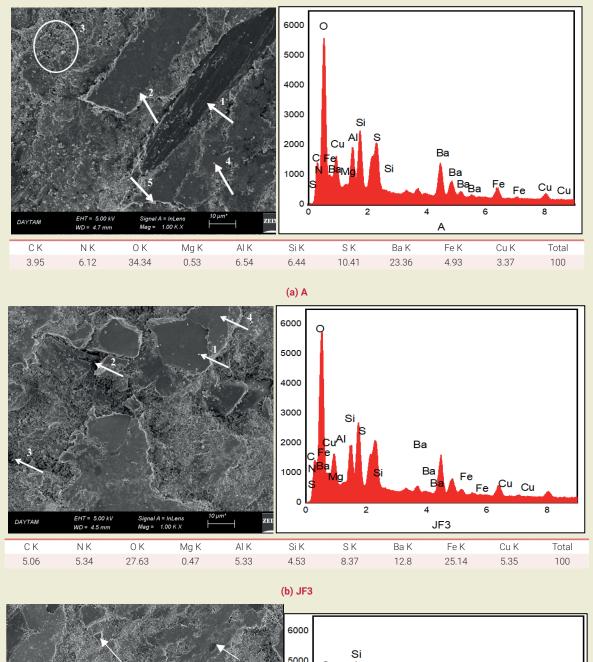
After the wear test of the pad samples, a Zeiss-brand SEM device was used to obtain and interpret their graphics for examining their surfaces. Looking at the SEM image of the A sample shown in **Figure 14**, the formation that occurred in the zone number 1 in the form of a scratch was considered to have occurred as a result of abrasive wear. In the zone number 2, this may have happened by that the pieces that were separated due to friction gathered on the surface again and led to adhesive wear. There was flocculation in the zone number 3. It is believed that the reason for this was barite and flocculation created border zones during wear. In the zone number 4, metal particles that actively participated in friction could be observed. There were white formations in the zone number 5 increases. Looking at the SEM image of the JF3 sample shown in **▶Figure** 6, the white particles seen in the zone number 1 were metal particles that took part in wear. It is known that metal particles increase wear. In the zones number 2 and 3, losses of pieces due to friction led to macro and micro gaps. In the zone number 4 with a pasting-like abrasion, adhesive wear took place. It is thought that formation of multiple pasting zones increased wear by reducing the friction coefficient. Generally speaking, it was concluded that secondary plateau zones formed in many areas on the material's surface and this was caused by the abundance of real contact zones.

The components of Batala surfaces are seen from EDX graphs. According to EDX spectra, the surfaces mainly contain sulfur (S), nitrogen (N), carbon (C), oxygen O), iron (Fe), barium (Ba), copper (Cu), aluminum (Al), silicon (Si). When the difference between A and JF3 is examined, it is seen that the Fe ratio increases. The intensity of O, S, Si, Ba, Cu peaks increases with the addition of JF. The atomic composition of JF3 treated with silane shows that C, Si increase and the atomic composition of oxygen decreases. In a similar study by Khalili et al. and Helenka et al., when EDX analyses were compared with the pure fiber, it was observed that the silane substance in oil palm fiber had higher carbon and lower oxygen content [32,33]. Higher carbon content shows the positive effect of modification. Similarly, all these elements peaked when jute fibers were treated with silane.

4. Conclusions

In this study, it was aimed to produce organic fiber-reinforced brake pads by minimizing the usage of metal-reinforced materials. Additionally, an environmentalist approach was displayed by avoiding usage of asbestos. The mechanical and tribological properties of the jute fiber-reinforced brake pads that were produced were studied and the following results were reached:

• Jute fiber reinforcement up to 5% reduced friction coefficients. From the friction coefficient time graphs, JF3 is the pad that showed the best and most stable change. Jute fiber addition at all rates positively affected the specific wear values. JF7 has



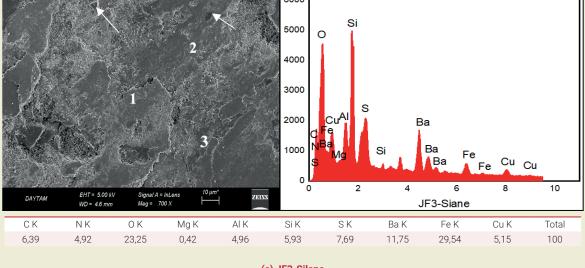


Figure 14. SEM-EDX Images of the Wear Surfaces

(c) JF3-Silane

the best wear rate but did not give a stable friction time graph.

- Adding Jute fiber to the pad mixture reduced the sample density. All samples are lower than the density value of commercial brake pads (2.0-2.3 gr/ cm3).
- The shear stress and density values of all produced pads are within the reference values.
- The hardness values of JF1, JF2 and JF3 pads are within the desired values. The hardness values of other pads are outside the standards.
- Water absorption values increased as the jute fiber ratios increased. When all the results are considered, the pad sample coded JF3 has mechanical, physical and tribological properties close to the standard pad values and the pads used in the market. For this reason, silane was applied to the jute fibers of the FJ3 brake pad and the brake pads were given water-repellent properties. With the silane treatment, 30% of the brake pad's water absorption was prevented.
- When the SEM graphics of the JF3 sample were examined, it was thought that adhesive wear occurred in many areas and that the metal particles in the areas close to the surface actively participated in the wear. As a result of adhesive wear, patch areas were formed on the surface and if these areas increased, a friction film was formed.
- It is thought that this situation will reduce mass losses. It is thought that the increase in the amount of oxygen found is not only due to the oxygen found in the components, but also to the oxygen coming from the oxidation on the surface.
- Considering the test results, it can be concluded that jute fiber reinforcement has positive effects on the material.
- · Natural fibers are now preferred in composite man-

ufacturing and are used in different applications in the industry. The jute fiber reinforced brake pads produced in the study comply with the standards of the brake pads used in the market. When considered from this perspective, the brake pads produced show their commercial usability. Jute fiber reinforced brake pads have good environmental performance with a green identity that can compete with commercial brake pads.

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Research ethics

Not applicable.

Author contributions

Conceptualization: [B. Hamamcı], Methodology: [All 2 authors], Formal Analysis: [All 2 authors], Investigation: [All2 authors], Resources: [All 2 authors], Data Curation: [All 2 authors], Writing - Original Draft Preparation: [All 2 authors], Writing - Review & Editing: [All 2 authors], Visualization: [All 2 authors], Supervision: [All 2 authors], Project Administration: [All 2 authors],

Competing interests

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