Section: Research Paper

TRIBOLOGICAL PERFORMANCE OF SILANE-TREATED BASALT FIBER IN A PHENOLIC-BASED FRICTION COMPOSITE

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Abstract

The purpose of this research is to investigate the influence of silane surface treatment on the tribological performance of basalt fibre in a friction composite. Basalt fibre (12 wt%), basalt fibre coated with silane, and Rockwool fibre were used to create three different brake friction compounds. The commercially available Rockwool fiber-based friction material was developed for comparison purpose. Following the IS2742 part 4 methodology, the tribological performance of the samples was investigated using the Chase friction testing apparatus. The silane surface-treated basalt fiber-based friction material's friction and wear performance is superior to untreated and Rockwool fiber.

Keywords: friction; wear; wear mechanism; brake; silane surface modification; basalt fiber

1. INTRODUCTION

The brake Friction Material (FM) formulation typically contains 10-20 ingredients to achieve desired Performance (Surya Rajan et al. 2017)(J. Bijwe 1997). These ingredients are categorized into binders, fibers, fillers, and friction modifiers. Different phenolic resins are used as binders in friction composites (Chan and Stachowiak 2004). Fibers are further categorized into synthetic, metallic, and natural fibers. Synthetic fibers such as aramid, polyacrylonitrile (PAN), and potassium titanate fibers are frequently used in friction composition. Burning and incomplete combustion of aramid and acrylic fibers can release carbon monoxide, hydrogen cyanide, and other toxic substances (DUPONT). Metallic fibers such as mild steel, stainless steel, and copper are widely preferred in the friction composition for their ductility and thermal load-bearing capacity. The usage of copper in the formulation is reduced due to its toxicity in the water bodies.

Studies show a recent increase in the use of eco-friendly natural fibres as reinforcement in friction composites based on phenol. In epoxy-modified-phenolic friction composites, Surya Rajan et al. studied the physicomechanical and tribological performance of Prosopis juliflora bark fibres with various surface modifications (Surya Rajan et al. 2019a) (Surya Rajan et al. 2019b). He observed that the alkaline and silane treatment of the fibers removes hemicellulose, lignin, and other impurities. This purification improved the physicomechanical performance of the brake friction composites. The silane surface treatment reduces the fiber's hydrophilic nature and improves the friction composition's wear resistance and rotor-friendliness. It also recommends using the silane surface over the alkaline treatment for better tribological performance. Various natural fibres, including abaca and maize stalk fibres, with surface treatment in the creation of friction composites have been explored by Liu et al. (Liu et al. 2019a). They discovered that the brake friction composites' mechanical and tribological performance is enhanced by the NaOH surface treatment. (Liu et al. 2019a)(Liu et al. 2019b). Additionally, it is concluded that the 5 weight percent silane treatment of the corn stalk fibre may serve as a potential reinforcement in brake pads to enhance friction and wear properties of the brake composite. Ranakoti et al. (Ranakoti et al. 2022) (Singh et al. 2022) have studied the physicomechanical and wear performance of wood waste and rise husk waste in the eco-friendly bio-composite. Jeganmohan et al. (Jeganmohan et al. 2020) used palm seed powder as an eco-friendly reinforcement in the brake friction composites with 0-9 wt% in the composition. Overall performance evaluation using a multi-criteria decision-making process found that the palm seed powder can be used in the formulation up to 3 wt%.

Currently, the world is focussing on alternate material development due to the ever-changing ecosystem. This ever-changing challenge led industries and academia to research sustainable Composite materials. Materials reinforced with natural fibre has become an excellent choice for alternate material source because of their strong properties like eco-friendly, processability, less cost, and non-toxic. Basalt fibre is a natural fibre that is eco-friendly and safe. It is similar to fiberglass, having better physio-mechanical properties than fiberglass but significantly cheaper than carbon fiber. The value of Basalt fibre in terms of mechanical, chemical and thermal performances are gaining industry attention to a higher level as a reinforcing material especially compared to traditional glass fibers. Even though many literature reports were available on natural fibres, to the best of my knowledge, not much research has been conducted on surface modified basalt fibre reinforced friction material. Hence, this research primarily focuses on examining basalt fiber's physicomechanical and tribological performance with and without silane surface treatment in friction composites. Two friction composites were developed with 12 wt % of basalt fibers with and without silane surface treatment, respectively, by replacing the mineral wool (Rockwool fiber). The third friction pad was developed without basalt fiber as a blank for comparison.

MATERIALS AND METHODS

The basalt fiber was procured from Fricteck Fibers Pvt. Ltd. Chennai. The Tri-ethoxy vinyl silane was procured from Harish Lab Vellore. The other ingredients in the formulations were procured from Kovai carbon products Coimbatore. The surface morphology and EDS spectra of the basalt fiber are shown in Figure 1.

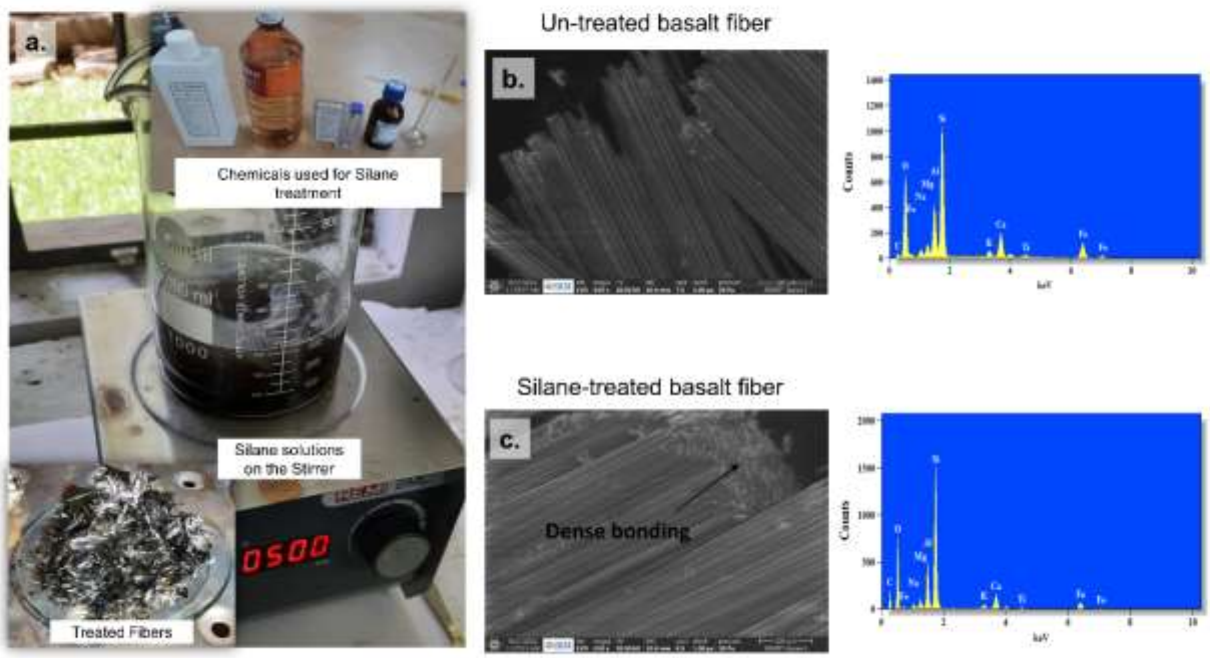

Figure-1 (a) Silanization process of basalt fiber, SEM and EDS of (b) untreated basalt fiber, and (c) silane-treated basalt fiber.

The fiber diameter and length range between 15-20 µm and 1000-1100 µm, respectively. The basalt fibers were treated with 98 % pure Tri-ethoxy-vinyl silane [CH2=CHSi-(C2H5O)3]. Initially, the 80:20 ethanol-water mixture is prepared. The 10 wt% Tri-ethoxy-vinyl silane is added to the ethanol and water solution under the stirring speed of 500 rpm for 20 minutes. The acidic acid was added to the silane solution to reduce its pH to 4. The basalt fiber of 10 g was added to the prepared solution. Finally, treated fibers were washed and dried using the hot air oven for 2 hrs at 95-100°C.

1.1. Fabrication of Friction Composites

Three brake friction materials were developed, and their composition is shown in Table 1. The primary formulation is comprised of sixteen components broken down into four distinct groups, namely fibers [Kevlar (4%) , Copper fiber (4%), Steel fiber (8%),], Friction Modifiers [Al_2O_3 (3%), Zircosil (2%), Cashew friction dust (6%), synthetic Graphite (6%), MoS_2 (5%)], Fillers [Wollastonite (8%), Vermiculite (10%), Mica (6%), Baryte (12%),], and binder [Crumb rubber (1%), NBR (1%), Phenolic Resin (12 %)]. The variation in the friction material composition is shown in Table 1.

Table 1. Formulation of the Friction Materials developed

Ingredients (wt.%)	**Brake pads**		
	FM-1	**FM-2**	**FM-3**
Basalt Fiber	12	0	0
Silane Treated Basalt Fiber	0	12	0
Mineral Wool (Rockwool)	0	0	12
Base formulation.	88	88	88

The friction material ingredients were mixed in the shear mixture model Phillips HL 7701 with the following sequence of ingredients: Fiber, Friction modifier, Filler, and Binder. Pre-weighed 120 g of the mixture is put to the mould cavity of TATA Indica (hatchback passenger car) brake pads, which are then compression-molded at 156°C and 150 kg/cm^2 for 6 minutes. During the curing process, three intermittent berating cycle is given to remove volatile from the mold cavity.

The cured brake composites were ejected from the die and post-cured for 4 hours in a hot air oven at 200 °C. The developed friction composites were surface-grounded and cut for the size 25x25x7 mm to conduct the Tribological performance evaluation using a chase friction testing rig following IS2742 part 4 standards. Physical, chemical, and mechanical properties of the friction composites were investigated in accordance with industry standards (Baskara Sethupathi & Chandradass, 2021). The Archimedean principle was used to analyze the density of the FMs. An L-scale Rockwell hardness tester with an indentor size of 6.35mm and a load of 60 kg was used to measure the hardness of the samples. The average of 5 reading is recorded along with the standard deviation. The brake pad samples were studied for acetone extraction using the soxhlet apparatus. The samples' cold and hot shear strengths were studied as per ISO 6312 of 2010.

1.2. Tribological analysis of friction composite

A chase tribometer was used to measure the tribological performance of the friction composites in accordance with IS2742 part 4 of the 1994 standard.The chase tribometer specification and the detailed test schedule are discussed in the previous article (Sathickbasha et al., 2021)(Surya Rajan et al., 2022). The FM samples' tribological performance was assessed during a variety of cycles, including baseline, fade-1, recovery-1, wear, fade-2, recovery-2, and baseline-2. The following friction and wear performance parameters were calculated based on the data acquired over the testing procedure.

1.2.1. Performance Coefficient of Friction (μ_p):

μ_p is an average Coefficient of Friction (CoF) of Fade-1&2 and Recovery-1&2 cycles above 100 °C.

1.2.2. Normal CoF (μ_n)

μ_n is an average of CoF at fade-2 cycle temperatures of 93, 121, 149, and 205 °C.

1.2.3. Hot CoF (μ_n)

μ_n is an average CoF at temperatures 205 and 149 °C recovery cycle-1, 233, 261, 289, 317, and 345 °C of the fade-2 cycle and 261, 205, and 149 °C of the recovery-2 cycle.

$$\boldsymbol{\mu_{hot}} = \frac{(\mu_{\mathbf{205}} + \mu_{\mathbf{149}})_{\boldsymbol{R_1}} + (\mu_{\mathbf{233}} + \mu_{\mathbf{261}} + \mu_{\mathbf{289}} + \mu_{\mathbf{317}} + \mu_{\mathbf{345}})_{\boldsymbol{F_2}} + (\mu_{\mathbf{261}} + \mu_{\mathbf{205}} + \mu_{\mathbf{149}})_{\boldsymbol{R_2}}}{\mathbf{10}}$$

1.2.4. % Fade

% $Fade$ is the measure of change in CoF on the downside due to the temperature. % $Fade$ is calculated as per the equation given below.

$$\% \boldsymbol{Fade} = \frac{\boldsymbol{\mu_{performance}} - \boldsymbol{\mu_{fade}}}{\boldsymbol{\mu_{performance}}} \times \mathbf{100}$$

Where, $\boldsymbol{\mu_{fade}}$ is the lowest CoF of fade cycle after 233 °C.

1.2.5. % Recovery

% $Recovery$ is the measure of how fast the CoF recovers after the brake pads undergone a thermal load cycle. % $Recovery$ is calculated as per the equation given below.

$$\% \boldsymbol{Recovery}\ \frac{\boldsymbol{\mu_{recovery}}}{\boldsymbol{\mu_{performance}}} \times \mathbf{100}$$

Where, $\boldsymbol{\mu_{recovery}}$ is the highest CoF of the recovery cycle after 233 °C.

1.2.6. *Wear % by mass*

Wear percentage in terms of mass loss is calculated based on the equation below.

$$\boldsymbol{Wear\ \%\ by\ mass} = \frac{\boldsymbol{Initial\ mass - Final\ mass}}{\boldsymbol{Initial\ mass}} \times \mathbf{100}$$

1.2.7. *Wear % by thickness*

Wear percentage in terms of thickness loss is calculated based on the equation below.

$$\boldsymbol{wear\ \%\ by\ thickness} = \frac{\boldsymbol{Initial\ thickness - Final\ thickness}}{\boldsymbol{Initial\ thickness}} \times \mathbf{100}$$

2. RESULTS AND DISCUSSIONS

2.1. Characterization of the Brake pads

Table 2. Physical, chemical, and mechanical properties of FMs.

	FM-1	FM-2	FM-3
Density (g/cm^3)	2.20 ± 0.14	2.28 ± 0.14	2.37 ± 1.09
Hardness (L-Scale)	89 ± 2.78	92 ± 2.4	93 ± 3.09
Acetone Extraction (%)	0.31	0.34	0.21
Cold shear strength (kg/cm^2)	47 ± 1.95	49 ± 3.68	44 ±1.414
Hot shear strength (kg/cm^2)	33 ±2.607	37±2.57	29+2.28

Table 2 summarises the physical, chemical, and mechanical characteristics of the brake pads. Both FM-1 and FM-2 have close values for their densities. The FM-3 holds a slightly higher density due to the higher density of the Rockwool fibers (6.76 g/cm3) compared to the basalt fibers (2.67 g/cm3). The hardness of the FM-2 is higher than the FM-1. The acetone extraction is the measure of the degree of curing. It is useful for determining the presence of uncured resin in brake pads. For brake pads, acetone extraction should be reduced. The observed results are significantly lower than the industrial standards. The cold and hot shear strength of the FM-2 sample is superior to the other samples. FM-2's improved performance metrics may be due to improved bonding of the silane surface-treated fibre with the matrix.

2.2. Friction Performance

The Friction performance of the developed FM samples was tested for friction performance and shown in Figure 2.

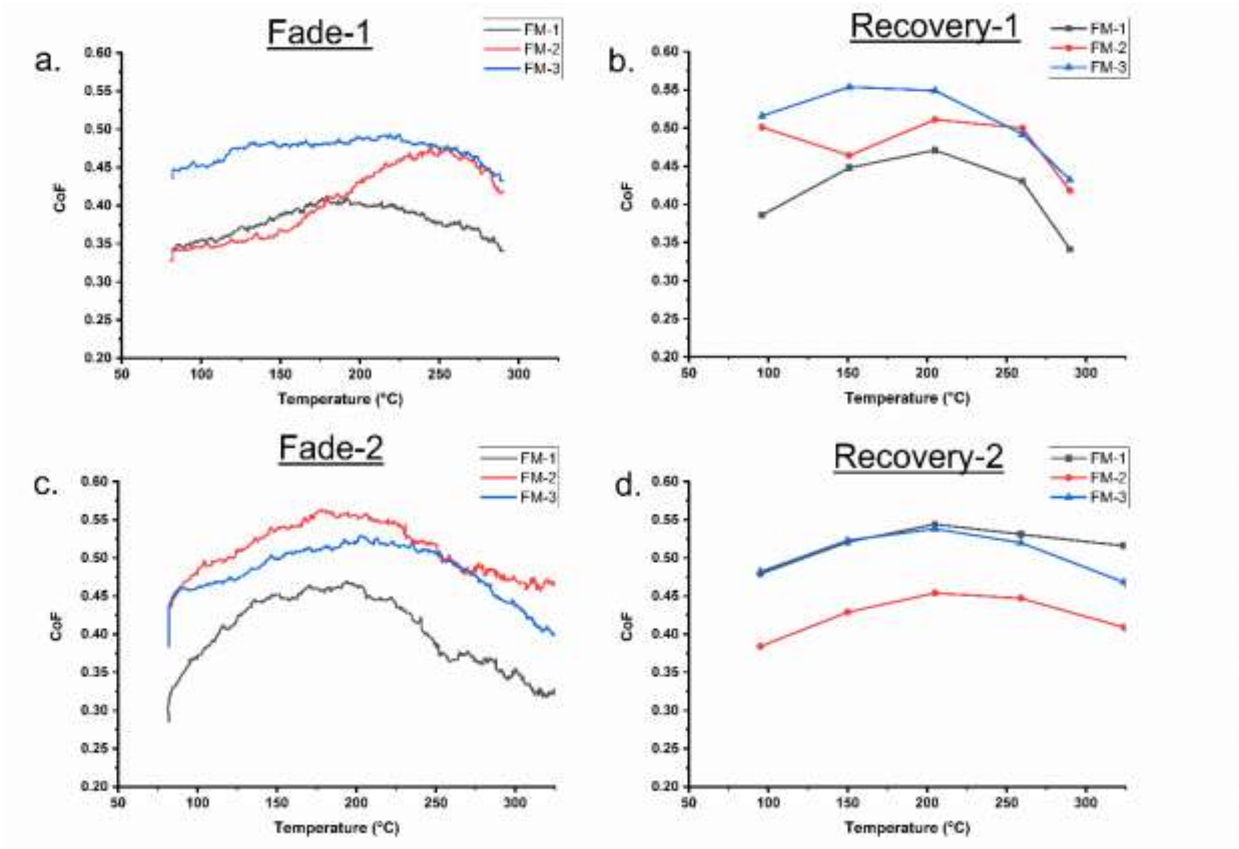

Figure 2. Friction performance of the brake samples in (a) Fade-1 (b) Recovery-1 (c) Fade-2 and (d) Recovery-2 Cycles.

The fade-1 performance of the friction materials shows that the FM samples are gaining the CoF at the initial temperature of up to 175 °C. The CoF of the FM-1 containing untreated basalt fiber reduced after 175 °C. However, in the case of FM-2 containing silane-treated fibers, the CoF is reduced after 220 °C. The blank sample (FM-3) with the Rockwool fiber showed slightly higher CoF due to its abrasive nature. Similar behavior was observed by Surya Rajan et al. (Surya Rajan et al., 2019b) in the study of *Prosopis Juliflora* bark fiber against the Rockwool fiber. In the recovery-1 cycle, all the brake pads gain their CoF after removing the thermal load. The fade-2 cycle study shows that irrespective of the composition, all the brake pads tend to drop their friction performance at higher temperatures. To properly quantify the friction performance at the higher temperature, normal and hot CoF are plotted in Figure 3. It also displays the fade and recovery percentages for the FM samples. Similar behavior was observed for the *Areva Javanica* fiber-reinforced phenolic friction composites in the previous studies (Ahmed et al., 2019)(Md et al., 2019).

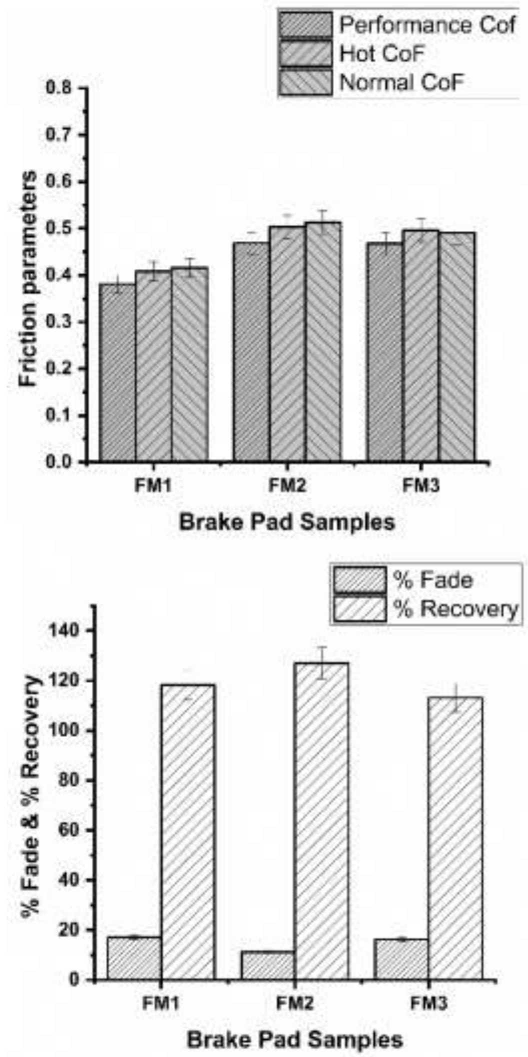

Figure 3. (a) Normal CoF and Hot CoF (b) Fade and recovery performance of the brake pads

The performance-CoF is higher for the sample with the silane-treated basalt fiber compared (FM-2) to the untreated fibers-based sample. The Rockwool fiber sample has a performance CoF value close to the FM-2. Similarly, in the case of Hot CoF, the FM-2 sample shows superior performance compared to other samples. This indicates the better performance of silane-treated basalt fiber at elevated temperatures. The silane surface treatment helps increase the bonding between the basalt fibers and the phenolic resin. It also helps increase the bonding ability between the strands of the fiber branches, as inferred in Figure 1. This improved adhesion helps deliver friction stability at harsh conditions and hence improves the Hot CoF. The friction stability of the FM-2 is also confirmed by the lowest value of % Fade, as shown in

Figure 3(b). As the %Fade measures friction stability, it should be as low as possible. The FM-2 shows this study's lowest %Fade and highest % recovery value.

2.3. Wear Performance

Figure 4 displays the wear loss percentage of the developed FMs in terms of mass loss and thickness loss. The silane-treated basalt fiber-based sample demonstrated the least wear when compared to other FMs for all categories of wear loss. The silane-modified basalt fiber's higher adherence as a reinforcement in the composite increased the wear resistance. Due to its abrasive nature, Rockwool fiber-based FM has a higher wear loss. Additionally, it is implied that Rockwool fiber-based FM takes the shortest amount of time to achieve its maximum temperature during the fade cycle.

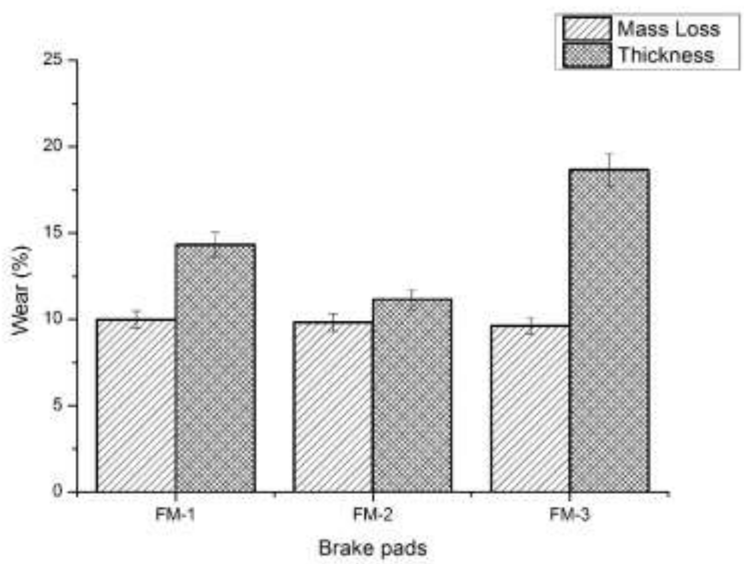

Figure 4. Wear performance of the brake samples.

2.3.1. Worn Surface analysis

The primary and secondary plateaus in Figure 4 are denoted by the letters "A" and "B," respectively.

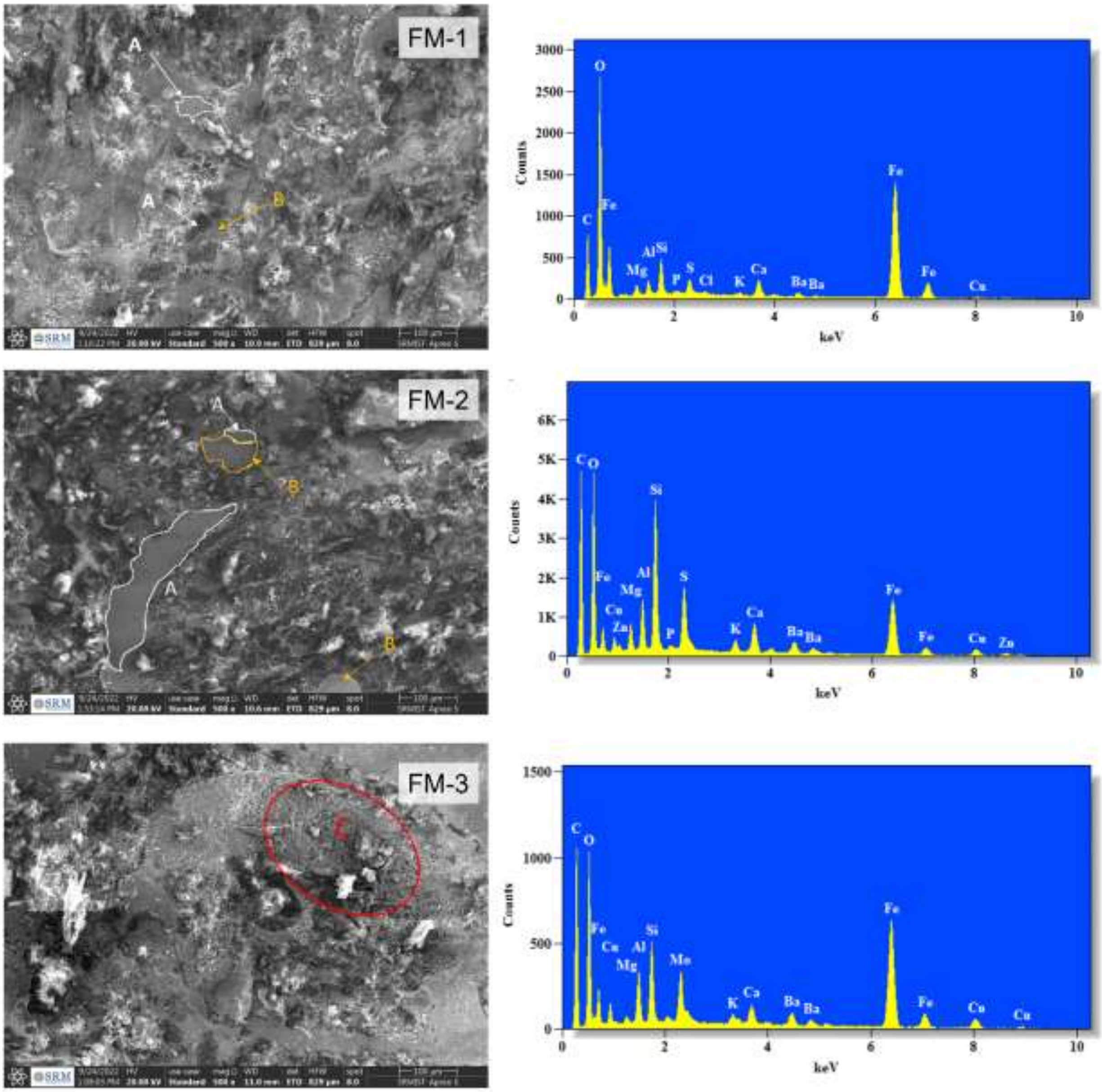

Figure 5. Worn surface morphology and EDS spectra of the friction material samples.

The hard fibers are exposed to the interface as flat plateaus, termed primary plateaus. The secondary plateaus are the extension of primary plateaus by accumulating fine wear debris near the boundaries of the primary plateaus (Eriksson & Jacobson, 2000). The formation of the primary and secondary plateau is shown in the FM-2. The tribological surface of the FM-2 is uniform, and the contact plateaus are more prominent than in other FM samples. It results in the FM-2 sample having the least wear out of all the produced samples. More wear debris labeled "C" is visible on the FM-3's deteriorated surface. The most significant wear of the sample is caused by this big debris, which functions as the third body abrasive particle. Si, the principal element of basalt fibre, was detected in the EDS examination of the worn surface (Figure 5). O, Fe and C are the other significant elements related to the oxidized products and graphite in the friction material formulation.

3. CONCLUSIONS

The tribological performance of the newly developed friction composite with the silane-treated and untreated basalt fibers was compared with the commercially used Rockwool fibers. The following conclusions were drawn from this study:

- The silane surface treated basalt fibers' overall friction and wear performance is superior to the untreated basalt fiber and the Rockwool fiber.
- The silane surface treatment on the basalt fiber improves the fade and recovery performance of the Friction composite.
- The smooth contact plateaus on the wear surface were observed on the friction composite containing silane surface-treated basalt fiber. In contrast, more loose wear debris was observed on the Rockwool-based friction composites.
- The silane surface-treated basalt fiber can be used to formulate the friction composites for better friction stability and wear performance.

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