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Research article

Friction composite formulation from *Lycium ferocissimum* fibers as natural reinforcement for braking applications

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Abstract. The increase in environmental consciousness and waste-to-wealth concepts in the automobile sector has led to the use of natural fibers in desirable quantities. The current study deals with the extraction, treatment, and utilization of *Lycium ferocissimum* stem fibers for friction composite in braking applications. The fibers of *Lycium ferocissimum* were extracted through manual retting and subsequently treated with benzoyl chloride. Both the benzoyl chloride treated and untreated fibers of *Lycium ferocissimum* were employed as reinforcements in the formulation of a friction composite, following the standard practices of the industry, and the comparison was made using commercially available friction composite. The developed friction composites were tested for original equipment manufacturer quality requirements following industrial Standards. The friction composite's tribological behavior was analyzed using the Chase test following the Society of Automotive Engineers standards. The worn surface characteristics were analyzed using scanning electron microscope. The test results elucidated that benzoyl chloride-treated *Lycium ferocissimum* fibers-based friction composites showed good frictional properties with better wear resistance compared to others, having a weight loss of 5.4%.

Keywords: *Lycium ferocissimum, friction composites, chemical treatment, Chase test, brake*

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1. Introduction

Vehicles are propelled forward using gasoline or electric engines, while brakes are employed to decelerate them. The term 'brake' pertains to a mechanical apparatus that utilizes friction to transform kinetic energy into thermal energy [1]. This is achieved by exerting pressure on the friction composite, such as a pad or liner, against the counterface, which can be a disc or drum, generating friction. Friction composite should have a stable μ, improved thermal stability, decreased wear, and be environmentally friendly [2, 3]. Since no one material could ever achieve all these behaviors, the formulation will need to be an amalgamation of at least ten to fifteen different ingredients. These ingredients can be categorized into fiber, fillers, friction modifiers, and binders. The strength of composites is predominantly influenced by the amount of fiber present [4, 5]. Natural fibers are vital in advancing lightweight materials based on eco-friendly principles. The efficient utilization of these natural fibers across different applications also helps reduce the overall expenses associated with waste disposal [6]. Mineral, plant, and animal fibers are the three major groups into which natural fibers

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fall. These plant-based natural fibers may be divided into three categories: stem, leaf, and root [7]. Their extraction process, the portion of the plant they are derived from, and the growing circumstances under which the plant was mainly grown determine the strength and chemical content of plant-based natural fibers. The bonding nature with the matrix and moisture absorption nature are the critical drawbacks of natural fibers [8].

The best approach to address the problem lies in addressing the surface as the issues arise. Typically, various chemical treatments can be applied to a surface [9]. These treatments help alleviate the problems by effectively removing the amorphous substances present. Some examples of chemical treatments that can be utilized include sodium hydroxide (NaOH), hydrochloric acid (HCl), acetic acid, silane, and benzoyl chloride [10]. In a study comparing the tribological performance of friction composites made from *Eichhornia crassipes* fibers that were chemical-treated and raw, Palai and Sarangi [11] found that silane treatment improved the tribological performance of the latter. The findings showed that compared to the other two composites, silane treatment helped sustain the friction levels during braking thanks to its excellent bonding nature and increased heat resistance. Ganesh Babu [12] examined the tribological behavior of friction composites made from benzoyl chloride-treated *Cyperus pangorei* fibers using pin-on-disc testing. Excellent friction behavior without frictional undulations was observed at varying temperatures and pressures when using friction composites using fibers extracted from *Cyperus pangorei* treated with benzoyl chloride. Paramathma *et al*. [13] used the Chase test to compare the tribological performance of friction composites made from untreated/silane-treated *Citrullus lanatus* stem fibers versus synthetic fibers. The findings indicated that friction composites from silane-treated *Citrullus lanatus* stem fibers had a μ_{normal} value of 0.413. In contrast, those made from synthetic or untreated fibers had lower values. Mehta *et al*. [14] studied the effect of alkali-treated sugar cane fibers in friction composites with varying 5–20 wt% in addition to other ingredients. The results showed that 5 wt% alkali-treated sugarcane fiber-based friction composites had better frictional performance than others. Kumar *et al*. [15] studied the influence of varying alkali-treated date palm fiber loading compared with Kevlar fiber in the friction composite formulation. It was found that 5 wt% alkali-treated date palm fiber-based friction composites showed better tribological, physical, mechanical, and thermal properties. In 2021, the global market for automotive brake systems was valued at an estimated USD 45.60 billion; between 2022 and 2030, that figure is projected to rise to USD 62.40 billion, a compound annual growth rate (CAGR) of 5.4%. The global market for natural fibers is projected to grow from its current value of USD 6.4 billion in 2022 to a new peak value of USD 9.3 billion by 2026, expanding at a CAGR of 10.2% between 2022 and 2026. This increase is mainly attributable to rising environmental consciousness. Researchers have been looking at new environmentally friendly materials for brake friction composite development in response to the rising demand for brakes and natural fibers in automobiles. Although there has been much research into using natural fibers in friction composites, the current rate of productivity growth necessitates the development of even more such fibers, ideally from environmentally friendly sources and with some waste-to-wealth orientation. One such plant is *Lycium ferocissimum* (LY), often recognized as African boxthorn. *Lycium ferocissimum* is a ubiquitous weed found in various regions ranging from Africa to India. It is an annual plant that can grow to a maximum height of 15 to 60 cm. Previous studies investigating the properties of *Lycium ferocissimum* fibers have demonstrated the removal of unwanted materials from their constituents, proving their suitability for treatment, better thermal stability, and compatibility with the matrix material [16]. Thus, this research focuses on formulating and characterizing friction composites as brake pads using standard industry practices developed utilizing *Lycium ferocissimum* fibers that were untreated and treated with benzoyl chloride.

2. Materials and methods

2.1. Materials

99% pure benzoyl chloride and 99% pure sodium hydroxide solutions were obtained from Sigma-Aldrich Chemicals Private Limited dealers in Chennai, India. The other parental ingredients for the friction composite formulations were obtained as samples. Stainless steel fibers from Kasturi Metal Composite Pvt. Ltd., Amaravati, India; nitrile butadiene rubber (NBR); crumb rubber from Taprath Elastomers LLP, Mumbai, India; mechanomade from Itaprochim, Milano, Italy; cashew friction dust from

Satya Cashew and Chemicals Pvt Limited, Chennai, India, while other ingredients were provided by Steadfast Friction Products, Chennai, India, and OHM Engineering, Chennai, India.

2.2. Fiber extraction and treatment

The LY tree barks were gathered from trees grown in arid regions of Chennai, Tamil Nadu, India. These barks were immersed in water for a week, and the fibers were extracted through manual retting. Before the benzoyl chloride treatment, the fibers underwent a pretreatment step with a 5% weight/volume (wt/v) NaOH solution for 30 min at ambient temperature. Subsequently, the pretreated fibers were thoroughly rinsed with distilled water until they reached an average pH level. The pretreated fibers were then soaked in a 10% (wt/v) benzoyl chloride solution, with a ratio of 200 g of fibers per 1 l of solution. After two hours of benzoylation, the fibers were washed with acetone to eliminate any excess reagents and by-products adhering to the fiber surface. Next, the treated LY fibers were placed in a hot air oven (Lab Tech, Chennai, India) at 80 °C for 12 hours to remove residual moisture. To verify their physical and chemical characteristics, the density, cellulose, hemicellulose, lignin, wax, and moisture content of the LY fibers were compared to values reported in the existing literature. The scanning electron microscope (SEM) images of the benzoyl chloride-treated and untreated *Lycium ferocissimum* fibers are given in Figure 1. In this work, the treatment with NaOH causes the disintegration of the fiber surface, exposing a greater number of hydroxyl groups. According to the literature, benzoyl chloride also reacts with the fiber surface by breaking down hydroxyl groups, resulting in the removal of some amorphous constituents from the fiber surface. The chloride ions are removed, and the benzoyl rings bond to the oxide, forming a coating over the fiber surface. The removal of amorphous constituents during both treatments increases the cellulose ratio in comparison to the amorphous constituents. Consequently, the fiber density increases. Additionally, the bonding of benzoyl rings to the fiber surface renders the fiber hydrophobic, repelling water molecules and thereby reducing the water absorption behavior of the fibers. The possible reaction mechanism during benzoyl chloride treatment is shown in Figure 2 [17].

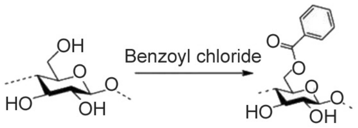

Figure 2. Mechanism of benzoyl chloride treatment.

2.3. Formulation and friction composite development

There are 19 ingredients in the friction composite, 18 of which are the same as their parent ingredients and 1 of which is different (benzoyl chloride-treated and untreated *Lycium ferocissimum* fibers). Table 1 provides the ingredients classification-based formulation. Friction composites in the form of standard

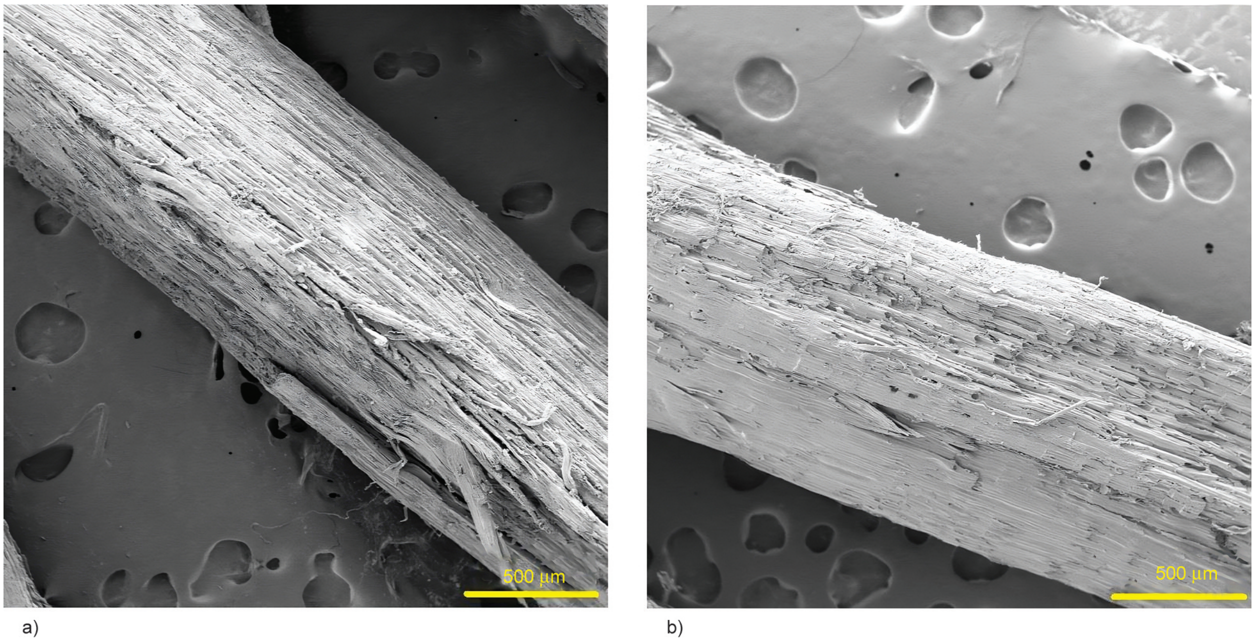

Figure 1. SEM images of LY fibers a) benzoyl chloride treated; b) untreated.

Table 1. Developed LY fibers-based friction composites formulation.

S.No.	Classification of ingredients	Ingredients	ULYBP [wt%]	TLYBP [wt%]
1	Fibers with additives	Acrylic fibers, wollastonite, *Lycium ferocissimum* (LY) fibers, stainless steel fibers, hydrated lime	22 (10 wt% untreated LY fibers)	22 (10 wt% benzoyl chloride treated LY fibers)
2	Binder with additives	Alkyl benzene modified phenolic resin, nitrile butadiene rubber (NBR), crumb rubber, magnesium oxide	21	21
3	Friction Modifiers	Zirconium silicate, synthetic graphite, molybdenum disulfide	15	15
4	Fillers	Barites, mechanomade, mica, green oxide, cashew friction dust, tin powder, vermiculate	42	42

brake pads were created using an industrial process. The specifics of the manufacturing techniques and the friction composites made after the process are shown in Figure 3. For comparison purposes, commercial brake friction composites were procured and tested. It was designed as CMLBP in this study. The friction composites with untreated *Lycium ferocissimum* fibers are designated as ULYBP, while the friction composite with benzoyl chloride-treated *Lycium ferocissimum* fibers are designated as TLYBP. The friction composites were developed using the equipment at Rajalakshmi Institute of Technology, Chennai, India, Anna University-MIT Campus, Chennai, India, and Steadfast Friction Composites, Chennai, India. The mixing of ingredients in the plough shear mixer that has 3 choppers and 1 shovel makes the mixture uniform, and that obtained powder mixture is used in composite manufacturing. This is in tandem with the literature discussed in the introduction, proving that natural fibers are used in brake pads.

2.4. Friction composite characterization

Density measurements were conducted using a digital densitometer (Model: PJS 3000; Yes Yes Technologies, Coimbatore, India) following the Archimedes principle. The hardness of the friction composites was verified through the K-scale indentation-based Rockwell hardness test (Model: 05050, T E Instruments, Chennai, India). Acetone extraction was carried out using the Soxhlet method. Loss on ignition was analyzed by taking five to ten grams of developed friction composites in a silica crucible and placing it inside a muffle furnace (Nano Tec, Chennai, India) maintained at 800 °C for 2 h. The difference in weight was measured and reported in percentage. These tests were performed according to the guidelines outlined in Indian Standards (IS 2742 Part 3: 1994) [18]. Porosity was evaluated using the method specified in Japanese Industrial Standards (JIS D 4418: 2021) [19]. To determine the adhesion between the pad and the backplate, a sample was

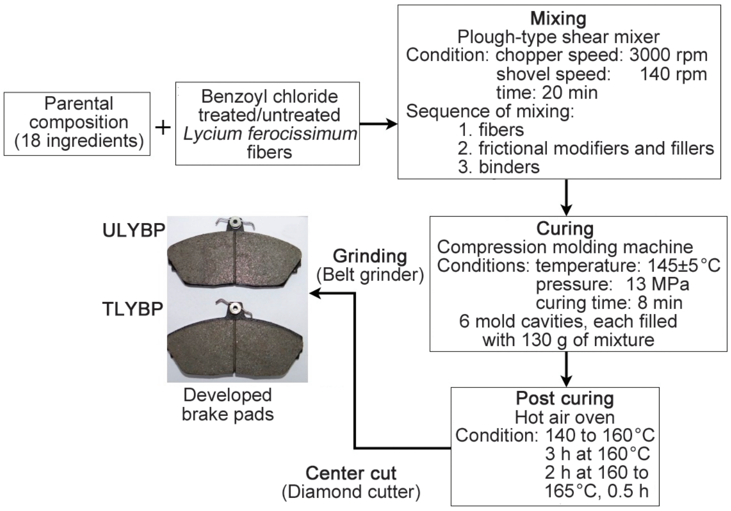

Figure 3. ULYBP and TLYBP development procedure.

inserted into the shear holder of the universal testing equipment (Model: UTES-40; Fuel Instruments & Engineers Pvt. Ltd, Maharashtra, India), and the shear strength was measured at room temperature conditions. The evaluation was conducted following the guidelines provided by the International Organization for Standardization (ISO 6312: 2010) [20]. Using a Chase test tribometer (Model: 200, Link Engineering, USA), the pad's tribological behavior was measured in accordance with SAE-J-661:2021 [21]. A test sample of 25×25 mm was used to slide against a cast-iron drum. Seven distinct cycles – initial and final baselines, wear, fade-I and II, and recovery-I and II – comprise the bulk of the testing technique. The beginning burnish cycle was completed before the test to make contact with the counterface. The application number in the test graphs refers to the engagement of brake friction composite towards the counterface. It presses the brake pad against the disc, *i.e.*, braking. Before and after testing, the sample's weight was measured using a digital weighing balance, and the sample's thickness was measured with a digital micrometer. The detailed test schedule is given below in Table 2 [22]. The allowable error per the industrial practice is 5%, and the standard deviation values are given next to the result values for the quality tests of the friction composites that are tabulated.

3. Results and discussions

3.1. Chemical and physical behavior of LY fibers

LY fibers treated with benzoyl chloride have a smaller diameter than untreated LY fibers. This decrease in diameter values is primarily because of the dissolution of hemicellulose and lignin, which causes fibers to fragment into fibrils, thereby changing the values. It is also seen that the average diameter values are given for the fibers since the fibers are not in uniform shape and size throughout the length. The findings align with the research conducted by Senthamaraikannan and Kathiresan [23]. The density of LY fibers treated with benzoyl chloride is higher due to the reduction in amorphous contents, thereby increasing the cellulose ratio due to both treatments causing such an increase in values. These observations are consistent with the findings reported by Ganesh Babu [12] in their studies on *Cyperus pangorei* fibers that were treated with benzoyl chloride. The obtained density values were lower than those of benzoylation-treated Vetiver fibers and benzoyl peroxide-treated *Cocos nucifera* L. Var typica fibers while slightly higher than those of *Cymbopogon flexuosus* stem and *Cardiospermum Halicababum* fibers [24–27]. Upon treatment with benzoyl chloride, there is a reduction in its hemicellulose and lignin content. Because of their amorphous phase and sensitivity to attack by chemical reagents, hemicellulose and lignin are eradicated during chemical treatment. Cellulose is more challenging to break down chemically because of its crystalline form and hard nature. Thus, there is a decrease in amorphous contents due to chemical treatment. The cellulose constituents after treatment are 1.1 times higher compared with untreated LY fibers, as seen in Table 3. This higher level of cellulose content aids in the thermal resistance and strength properties upon its usage in the composites that could be inferred from the following sections. Paramathma *et al.* [13] observed this behavior in scientific investigations about *Citrullus lanatus* fibers-based eco-friendly friction composites that were untreated and chemically treated. The obtained cellulose values are superior to Benzoyl peroxide-treated *Cocos nucifera* L. Var typica fibers and alkali-treated *Ficus religiosa* root fiber. However, it is less than benzoyl chloride-treated *Cyperus pangorei*

Table 2. Chase test schedule.

Cycles	Speed [rpm]	Temperature [°C]		Load [N]	On-time		Off time	No. of applications	Heater	Blower
		min	max		min	s	s			
Burnish	308	–	93	440	20	–	–	1	Off	Off
Initial baseline	411	82	104	660	–	10	–	20	Off	Off
Fade-I	411	82	289	660	10	–	–	1	On	Off
Recovery-I	411	261	93	660	–	10	–	1	Off	On
Wear	411	193	204	660	–	20	10	100	Off	Off
Fade-II	411	82	345	660	10	–	–	1	On	Off
Recovery-II	411	317	93	660	–	10	–	1	Off	On
Final baseline	411	82	104	660	–	10	20	20	Off	Off

and *Cissus quadrangularis* stem fiber [12, 25, 28, 29]. Chemical treatment disrupts the amorphous structure of the fibers, resulting in a decrease in wax content and an increase in the fiber's surface contact area. This, in turn, promotes mechanical interlocking between the fiber and the matrix, enhancing their connection and interaction. Therefore, this property strengthens the fiber's bonding nature and safeguards against debonding. It can be seen from Table 3 that the benzoyl chloride-treated LY fibers have lower wax content compared to untreated LY fibers. The obtained wax content values are superior to Benzoyl peroxide-treated *Cocos nucifera* L. Var typica, although less than Benzoylation-treated Vetiver fibers, *Muntingia calabura* [24, 25, 30]. The moisture weight gets reduced upon the chemical treatment by eliminating non-crystalline substances. It is inferred that enhanced water resistance and matrix bonding qualities resulted from the creation of interstices between a collection of microfibrils, which the linked agents subsequently blocked. This aligns with the results of Gurupranes *et al*. [31] in their studies about alkalized/raw *Ziziphus nummularia* bark fibers. The obtained moisture contents are lower than fibers of *Tamarindus indica* L., *Cymbopogon flexuosus* stem, and *Ziziphus mauritiana* [26, 32, 33].

3.2. Physical, chemical, and mechanical properties of ULYBP, CMLNP, and TLYBP friction composites

Removing undesirable components from the fiber's chemical composition increased the density of treated LY fibers, which enhanced the density of the TLYBP friction composites since they had that treated LY fiber. The density of TLYBP was 2.07 g/cm^3, which is higher than ULYBP but lower than CMLBP friction composites. This is in tandem with the literature studies of Vijay *et al.* [34] in their research on raw and silane-treated Leucas aspera-based friction composites. The acquired density values are well within the range the original equipment manufacturer (OEM) recommended. Closer packing of the treated LY fibers into the matrix accounts for the greater hardness of the TLYBP compared to the ULYBP, as shown in Table 4. Furthermore, this study supports the hypothesis that an increase in density correlates with an increase in hardness and a decrease in porosity [35]. The measured results align with the minimum range

Table 3. Comparison of LY fibers with other fiber's physical and chemical properties.

Fiber name	Cellulose [wt%]	Hemicellulose [wt%]	Lignin [wt%]	Moisture content [wt%]	Wax [wt%]	Diameter [µm]	Density [g/cm^3]	References
Benzoyl chloride-treated LY fibers	67.19	5.16	9.41	3.39	0.37	152.12	1.379	Current study
Untreated LY fibers	55.83	10.74	16.03	8.45	1.22	175.25	1.123	Current study
Untreated Vetiver fibers	42.72	33.67	18.38	–	5.23	–	1.498	[24]
Benzoylation-treated Vetiver fibers	66.32	18.90	10.76	–	4.02	–	1.598	[24]
Untreated *Cocos nucifera* L. Var typica	50.11	11.98	24.90	11.10	0.31	0.323	1.36	[25]
Benzoyl peroxide treated *Cocos nucifera* L. Var typica	57.55	8.02	23.95	8.47	0.12	0.309	1.45	[25]
Benzoyl Chloride treated *Cyperus pangorei*	72.1	9.7	6.1	0.54	3.8	270	1.243	[12]
Untreated *Cyperus pangorei*	65	15.9	9.2	1.6	7.5	390	1.107	[12]
Cissus quadrangularis stem fiber	82.73	7.96	11.27	6.6	0.18	770–870	1.22	[29]
Untreated *Ficus religiosa* root fiber	55.58	13.86	10.13	9.33	0.72	25.62	1.246	[28]
Alkali treated *Ficus religiosa* root fiber	64.35	8.85	7.64	6.65	0.43	22.54	1.272	[28]
Tamarindus indica L.	72.84	11	15.38	6.35	0.2	564–779	1.02–1.27	[32]
Cymbopogon flexuosus stem	68.13	11.56	23.9	9.17	0.53	118	1.270	[26]
Muntingia calabura	41.5	11.3	7.6	6.1	3.4	148.29	0.921	[30]
Cardiospermum Halicababum	59.42	16.75	9.3	1.9	1.3	315.4	1.141	[27]
Ziziphus mauritiana	43	10.2	5.1	7.9	–	570.2	1.132	[33]

specified by the OEM, which typically falls between 80–90. This behavior is consistent with that reported in the literature by Dharmakrishnan *et al*. [36], who investigated friction composites using fibers extracted from the stems of the *Psidium guajava* plant. A lower acetone extraction value indicates that the friction composites have been better cured since this number reflects the amount of uncured resin present. The acetone extract value of TLYBP was 1.21%, whereas that of ULYBP was 1.45%. According to the available literature, the values above are consistent with OEM requirements (values of less than 1.5%). [37]. TLYBP's shear strength is 46 kg/cm^2, which is much greater than ULYBP's shear strength of 41 kg/cm^2. The increase in the higher shear values is because of better fiber bonding with the matrix further with the backplate. This better bonding is due to a better cure, as confirmed by acetone extraction results. The developed friction composites met the OEM specifications of minimum shear strength: 40 kg/cm^2 [38]. The porosity of the TLYBP pad is less compared to the ULYBP due to better curing and better bonding with the ingredients and matrix. OEM prescribes that the porosity should be less than 10% for an ideal pad; the current study proves the same. This behavior was observed in scientific investigations by Surya Rajan *et al*. [39] in their studies on *Prosopis juliflora* fiber-based brake pads. The CMLBP friction composites exhibited higher density, hardness, and lower porosity, acetone extraction, which could be due to more dense materials in the formulation since the formulation of the commercial friction composites is not known. The exact material influence cannot be explained. The loss on ignition (LOI) values of ULYBP friction composites are greater than those in TLYBP and CMLYBP friction composites, as shown in Table 4, because they contain more amorphous than crystalline constituents, reducing thermal resistance. This is consistent with the research investigations of Dharmakrishnan *et al*. [36]. The acquired LOI values for ULYBP and TLYBP are more significant than the prescribed range (≤25%) due to the presence of natural fibers [13]. In contrast, the CMLBP friction composites fell within the specified range, which could be due to some more stable ingredients. The exact reason could not be said since the formulation is unknown for commercial pads.

3.3. Tribological evaluation of developed ULYBP, CMLBP, and TLYBP friction composites

Normal friction (μ_{normal}), hot friction (μ_{hot}), fade friction (μ_{fade}), recovery friction ($\mu_{recovery}$), and wear loss rates are some of the tribological parameters that are often evaluated for friction composites using the Chase test. The produced friction composite constituents interact with the mating surface, determining the outcomes. The term 'tribology' comes from the Greek word 'rubbing', which refers to the study of friction and wear in mechanical systems. The main requirement for friction composites is to provide adequate friction. Ideal brake friction should achieve optimal friction levels while avoiding damage to the counterface. The term 'fade' refers to the reduction in braking efficiency that occurs as friction temperatures increase. Fade has a notable influence on the overall performance of friction composites. It is primarily attributed to the pyrolysis of polymeric materials and the disintegration of the friction layer during braking, which leads to the fading effect. Recovery occurs when the friction composites subjected to a more significant temperature during braking return to their normal temperature after being released from their application. It is essential to reduce thermoelastic instability (TEI) at the dynamically mating surfaces, and this can only be done by addressing thermal concerns such as fading. This phenomenon, in which thermally induced friction changes, may be used to help explain the divergence from Amonton's law of friction [40].

Table 4. Properties of ULYBP, CMLBP, and TLYBP friction composites.

S.No.	Properties	Unit	ULYBP	TLYBP	CMLBP
1	Density	g/cm^3	2.01±0.012	2.07±0.008	2.23±0.015
2	Rockwell hardness	HRK	91±1.632	95±0.816	97±2.055
3	Acetone extraction	%	1.45±0.028	1.21±0.012	1.20±0.029
4	Shear strength	kg/cm^2	41±1.699	46±1.247	45±2.160
5	Porosity	%	6.12±0.0205	5.87±0.0124	4.71±0.0287
6	Loss on ignition	%	31±1.247	28±0.816	23±1.699

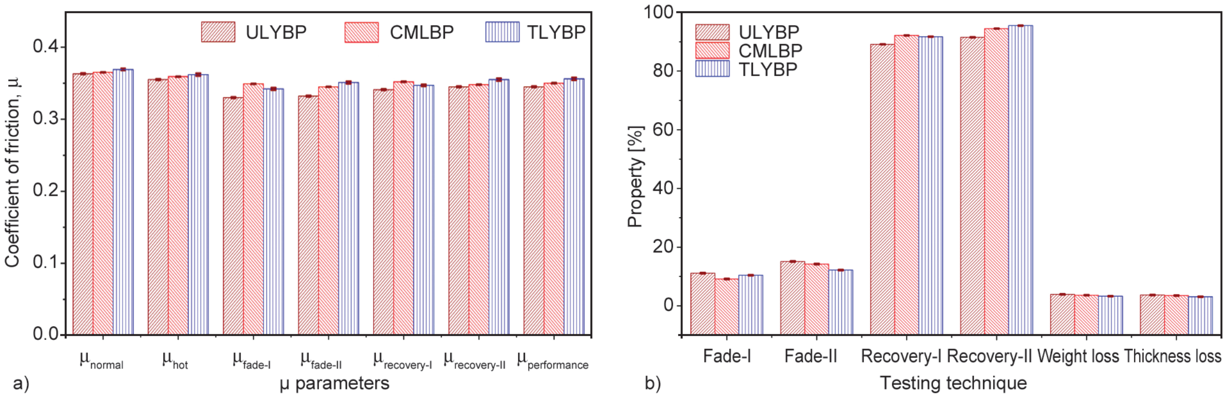

Figure 4. a) Various μ parameters and b) fade, recovery rates, and wear loss values of ULYBP and TLYBP Chase tested friction composites.

It's also crucial to remember that friction composites made from phenolic resin undergo chemical-microstructural change with localized fracture-based plastic deformation. The formulation's constituents, thus, have a significant influence in determining the brake pad's performance under varying circumstances of use. Figure 4 display the various μ parameters and wear behavior of Chase-tested ULYBP, CMLBP, and TLYBP friction composites. In contrast, Figure 5 provides the resultant Chase test graph with different cycles as per the test standards.

To get an idea about the significant difference between the groups of samples, the one-way analysis of variance (ANOVA) was performed for μ_{normal} and μ_{hot} conditions and given in Table 5.

Table 5. ANOVA for μ_{normal} and μ_{hot}.

μ_{normal}					
Source	**DF**[1]	**Adj SS**[2]	**Adj MS**[3]	**F-value**[4]	**P-value**[5]
Factor	2	0.000056	0.000028	12.07	0.037
Error	3	0.000007	0.000002		
Total	5	0.000063			
μ_{hot}					
Source	**DF**	**Adj SS**	**Adj MS**	**F-value**	**P-value**
Factor	2	0.000042	0.000021	21.17	0.017
Error	3	0.000003	0.000001		
Total	5	0.000045			

[1] DF: degree of freedom;
[2] SS: sum of square;
[3] MS: mean square;
[4] F, F-test for ANOVA;
[5] P-value is less than 0.05; hence, there is a significant statistical difference between the group of samples at the 95.0% confidence level. Jawaid *et al*. [41] reported this behavior in their studies about oil palm-woven jute fibers-based epoxy composites.

3.3.1. Fade and recovery performance of ULYBP, CMLBP, and TLYBP friction composites

Upon braking, there is more dissipation of heat at the interface; the μ_{normal} is always more significant than the μ_{hot} in an ideal brake pad (*i.e.*, the μ value determined is the combination of the recovery and fade process); thus, the cooling nature helps in the performance revival. Values of 0.355 and 0.362 were recorded for the ULYBP, 0.363, and 0.369 for TLYBP friction composites, while 0.359 and 0.365 for CMLBP, representing μ_{hot} and μ_{normal} values, respectively. These improved μ_{normal} values are analogous to the improved pad recovery following fading. The results show that the obtained values fall within the typical industrial range of 0.3 to 0.55 [22]. The performance values of a good friction composite should fall within the OEM criterion since this helps to keep the pad in good working conditions while providing the driver with satisfactory braking comfort. It is seen that treated LY fiber-based friction composite has greater performance values than untreated LY fiber-based friction composite, which is due to the enhanced thermal stability of the treated LY fibers, which could be found from the previous work contributes to improved bonding with the matrix and facilitates the development of a well-established contact plateau, as observed in the forthcoming sections through SEM studies. This finding aligns with the research conducted by Dharmakrishnan *et al*. [36] on friction composites fabricated using *Psidium guajava* stem fibers that were untreated and treated with chemicals, where the test results were consistent with the observations above.

ULYBP friction composite reduces friction value at fading circumstances due to untreated LY fibers having an increased proportion of amorphous substances, such as wax, lignin, hemicellulose, *etc*., as

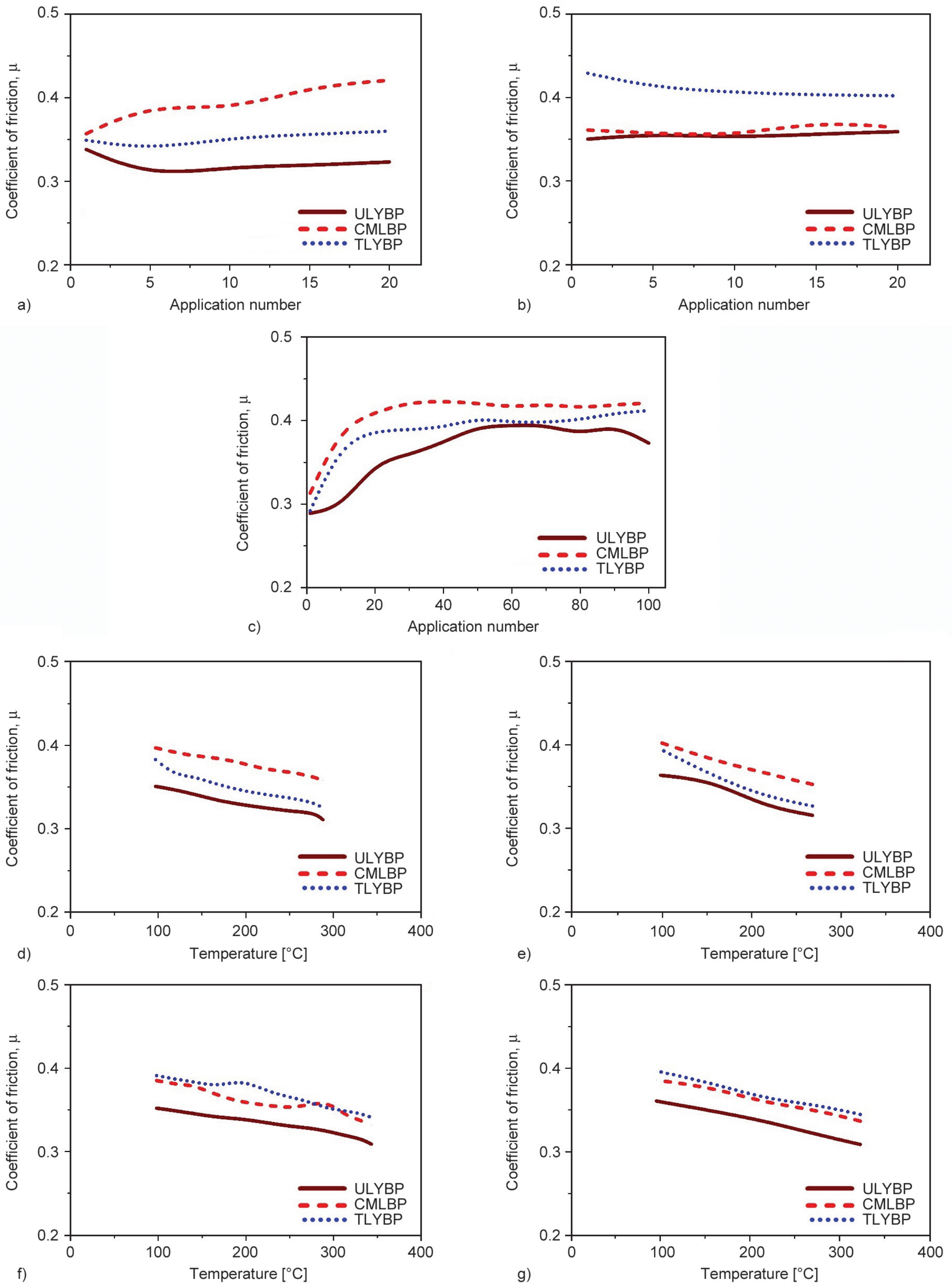

Figure 5. Resultant Chase test graph for ULYBP, TLYBP, and CMLBP friction composites showing different test cycles a) initial baseline; b) final baseline; c) wear; d) fade-I; e) recovery-I; f) fade-II; g) recovery-II.

inferred from Table 3. This is consistent with the investigations of Palai and Sarangi [11] about brake pads made up of *Eichhornia crassipes* fibers. Due to this poor performance, additional contact plateaus have been rebuilt and destroyed, contributing to increased undulations. In the case of the TLYBP friction composite, the treating of LY fibers with chemicals at optimal concentration and duration aided in the better removal of undesired amorphous contents, hence boosting the cellulose contents, which assists in stability. The friction composite must perform pristine for its entire service life (*i.e.*, high temperature and load conditions) [35]. TLYBP friction composite also lacked value variation over the working range, demonstrating their superior performance.

Figure 4 and Figure 5 indicate that the TLYBP friction composite performed better throughout fade and recovery cycles. This may be because LY fibers treated with benzoyl chloride were better able to withstand heat at the tribo interface, thus halting the rapid decomposition of the phenolic resin. There is a temperature-dependent declination in the µ values measured by the fade test, with the minimum value being reached at the highest temperature. Due to the cooling of the contact, the values recover substantially throughout the restoration process. Due to the glass transition and subsequent breakdown of the phenol-formaldehyde resin, the friction composite shear strength is reduced when the temperature is raised. This is because the smooth fiber surface facilitates further weakening of fiber-matrix adhesion at higher temperatures. Film formation tests conducted by Blau and McLaughlin [42] show a similar pattern of behavior, where a rise in the interface temperature has a negative effect on the values. The untreated LY fibers have a higher concentration of amorphous elements than treated ones. This affects the characteristics of the fiber's surface roughness. The roughness of the fiber surface is increased by the benzoyl chloride treatment in LY fibers, which also helps improve the resin-fiber interlocks. The research of Vijay *et al.* [43] on polymer matrix composites made from *Impomea pes-caprae* stem fibers that were benzoyl chloride treated and untreated provides support for this inference. The detailed study of the surface roughness of LY fibers is the near future scope of this study. The braking at harsh conditions caused more changes in the surface and some melting at higher temperatures. The friction composites should have more primary plateaus and fewer back-transfer patches. Back transfer patches are produced by degrading polymer ingredients that attach to the surface should be less. This would improve the contact area between the mating surfaces, leading to faster recovery times. Figure 5c shows a continuous application cycle graph (wear cycle) for the ULYBP friction composite, which displays mild undulations in the µ values due to the formation of third bodies during braking and the debonding of untreated LY fibers. These third bodies, *i.e.*, wear debris, can be confirmed from the SEM studies in the forthcoming sections. A similar behavior was seen in the tribological studies of brake pads made up of untreated *Eichhornia crassipes* fibers comparable to pads made from treated fibers (with silane and NaOH, respectively). Ganesh Babu [12]. It could be seen in the CMLBP friction composite that it had a higher µ during the first fade. At the same time, it got reduced in the forthcoming cycles, which could be due to the exposure of fiber/complex particles in the initial stages that caused a further increase in temperature at the interface, leading to rapidly degrading polymeric ingredients. This behavior causes more back-transfer patches that could be confirmed by the SEM studies, as shown in Figure 6c.

According to the scientific investigations of Singaravelu *et al.* [37] about brake pads filled with different friction dust, it is usually advised to have a low fade rate with a more significant recovery rate that significantly boosts the friction composite performance. The ULYBP fade-I and II rates were 11.12 and 15.15%, whereas the TLYBP was 10.42 and 12.18%, and the CMLBP was 9.13 and 14.26%. Furthermore, as shown in Figure 4b, the recovery-I and II rates for ULYBP were 89.12 and 91.45%, whereas the rate for TLYBP was 91.71 and 95.49%, while it was 92.15 and 94.39% for CMLBP. Lignin, hemicellulose, and cellulose are the three main constituents found in the cell walls of plant fibers. Chemical or other surface treatment is often used to activate the hydroxide group in the fiber. Previous X-ray diffraction (XRD) investigations of the LY fibers suggested that this chemical treatment helped remove non-cellulosic components effectively, thereby enhancing the crystalline cellulose ratio [16]. This improves thermal stability by gluing fibers together, which keeps performance steady under pressure. Moreover, the residuals may help build a glassy covering over the tribo interface, reducing the rate at which heat and oxygen may diffuse through the contact. This is consistent with the Chase test results used to evaluate the tribological performance of brake friction composites made from recycled basalt and aramid fibers, as reported in the literature by Manoharan *et al.* [44].

Friction composite made from LY fibers treated with benzoyl chloride had improved tribo-mechanical performance. It decreased hydrophilicity while maintaining greater µ fade values than friction composite from untreated LY fibers (ULYBP). Adsorption of non-polar molecules improves lubricity, which in turn aids heat conduction and reduces the fade rate. The CMLBP friction composite showed higher μ values during the first fade and recovery cycles, resulting in a lesser fade-I rate and higher recovery-I rate. Poor braking performance was observed with ULYBP friction composites made with untreated LY fibers because fiber debonding allowed the composite surface to slip during braking, which allowed tribo film to slide and smear, disrupting braking performance [12]. The decrease in the fade values further confirms the heat deterioration and resin softening resulting from the releasing gases caused by such pyrolysis. Vijay *et al*. [45] found similar results when comparing brake pads made with antimony trisulfide to those made using premixed dual metal sulfides.

3.3.2. Wear performance of ULYBP, CMLBP, and TLYBP friction composites

Wear may be caused by many different processes, including but not limited to abrasion, oxidation, adhesion, pitting, and so on, all of which are primarily driven by heat. Conditions during braking that lead to material removal and fiber fragmentation during mating reduce and speed up the wear rate. Reduced fiber-matrix bonding in the friction composites due to the untreated LY fiber's higher amorphous constituents produces more pullout and, in turn, increased wear. Moreover, the fiber wears down quickly at higher temperatures due to the lack of thermal resistance of untreated LY fibers [46]. As friction composites rub against a counter surface, they generate heat, oxidizing to iron oxide. This abrasive compound may cause plowing and the destruction of contact plateaus after some wear and tear. Friction composites made of oxide-coated steel exhibited this behavior, according to the research of Manoharan *et al*. [47]. The CMLBP friction composites showed the wear rate between the ULYBP and TLYBP friction composites. It is seen that CMLBP friction composites also showed a higher wear rate than TLYBP friction composites, which could be due to the more abrasion by the hard materials present in the formulation, leading to the wear of low thermally stable materials. Another worth noting is that the postulate of mere higher hardness will have a lower wear rate didn't work in the case of CMLBP friction composites. This is consistent with the results of Paramathma *et al*. [13]. Due to increased resistance to indentation, the tribosurface is not further plowed while using brakes. Because of this property, composites are relatively resistant to wear. The present study confirms the hypothesis of Vijay *et al*. [38] in their research of various mesh-sized MoS_2-based brake pads, demonstrating that the TLYBP friction composites exhibited less weight and thickness loss than the ULYBP friction composites displayed in Figure 4b.

3.4. Worn surface characterization of ULYBP, CMLYBP, and TLYBP friction composites

The Chase tested ULYBP, CMLBP, and TLYBP friction composites were analyzed for worn surfaces using a scanning electron microscope (SEM). Its overview of the elements in the contact plateaus and back transfer patches was analyzed using energy-dispersive X-ray spectroscopy (EDAX) spectra, which are given in Figures 6a–6c and Figure 7, respectively. The adhering fibers that comprise the contact plateaus (primary plateaus) play a crucial role in preventing the movement of wear debris particles at the interface. During braking, an increase in pressure and temperature causes these fine wear particles, the primary constituents of polymeric materials, aramid, *etc*., to adhere to each other, producing back transfer patches (secondary plateaus). It is stated that contact plateaus have a load-bearing capacity and increase friction, whereas back transfer patches degrade similarly. Therefore, it is always preferable to have more contact plateaus and fewer back-transfer patches [48]. Figure 6a and 6b shows the worn surface of ULYBP friction composites, which have more back transfer patches with considerably fewer contact plateaus. These contact plateaus are also smeared with more back transfer particles that act as film, preventing its better frictional performance. The poor thermal resistance nature of untreated LY fibers in the formulation caused more debonding of fibers and more cracks with deep propulsion. This surface also had more pits due to removing materials from the surface. Due to more wear, there is more formation of wear debris, which gets smeared and adhered over the surface, which can be seen in Figure 6a and 6b. The contact plateaus in ULYBP friction composites had more surface pits due to the abrasive actions caused by debonded materials. This is consistent with the results of Ganesh Babu *et al*. [12]. Figure 6c and 6d shows the worn surface of TLYBP friction composites with

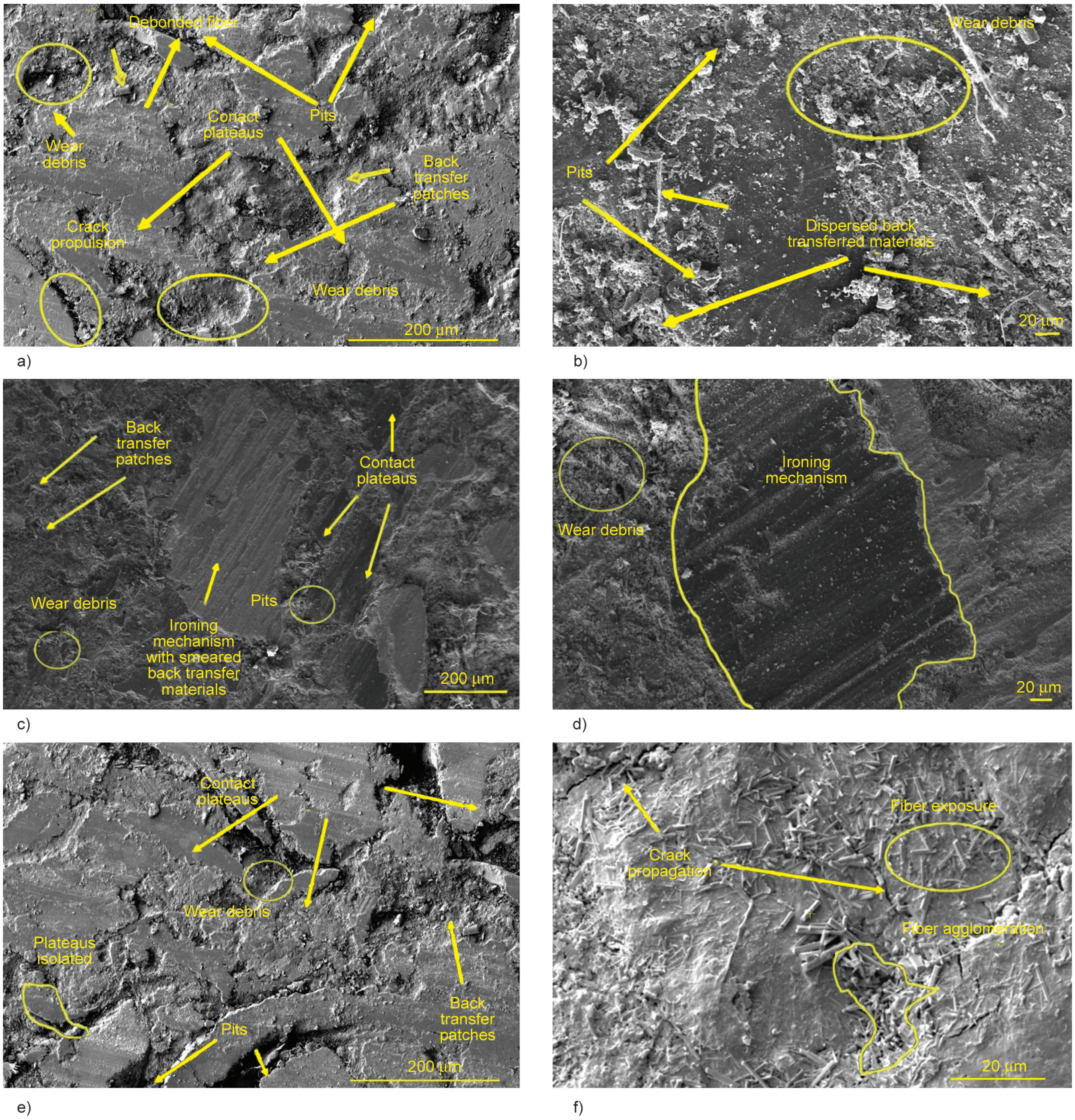

Figure 6. SEM micrographs of a) Chase-tested ULYBP, b) zoomed view of Chase-tested ULYBP, c) Chase-tested TLYBP, d) zoomed view of Chase-tested TLYBP, e) Chase-tested CMLBP, f) zoomed view of Chase-tested CMLBP.

more contact plateaus and fewer back transfer patches. The surface also has a predominant ironing mechanism, which is an indication of more wear resistance. This is consistent with the results of Manoharan *et al*. [47]. The surface also had some wear debris in the region of back transfer patches, which is expected due to the debonded materials produced by the braking. The CMLBP friction composites showed deeper cracks with pits caused by the abrasive action of the debonded hard materials; these materials cause less stable polymeric ingredients to degrade, thereby causing such unstable behavior. It also had some isolated contact plateaus produced by removing surrounding ingredients by plowing with more hard materials, leading to more pits around that region. As stated in the previous sections, since the formulation is unknown for this pad, it could be visualized from Figure 6e and 6f that some hard fibers are present over the surface. More agglomerated fibers are also seen on the surface. The ingredients present in the back transfer patches of ULYBP are given in Figure 7a. It could be seen that it had more Fe, S, and O due to the back transfer caused between the friction composites and interface [45]. Figure 7b

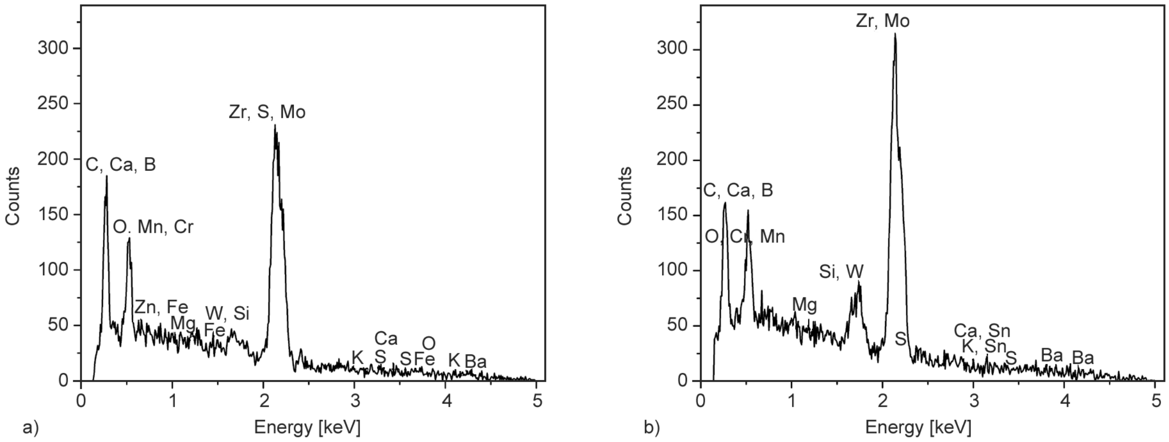

Figure 7. EDAX spectra a) back transfer region of ULYBP friction composites; b) contact plateau region of TLYBP friction composites.

shows the contact plateaus spectrum of TLYBP friction composites, where the concentration of Fe is not seen. It is a fact that the back transfer iron oxide particles from the counterface get adhered to the back transfer regions. This is consistent with the scientific investigations of Vijay *et al*. [49] about stainless steel and steel-based friction composites.

The future scope of this work is to analyze the thermal behavior of developed friction composites using Thermogravimetric analysis and analyze the real-time tribological behavior of the developed friction composites using a full-scale inertia dynamometer following the JASO-C-406 schedule.

4. Conclusions

Lycium ferocissimum fibers were extracted and treated with benzoyl chloride, following conventional industrial procedures, to develop friction composites. These composites were then compared with friction composites based on untreated *Lycium ferocissimum* fibers and commercially available friction composites regarding various performance characteristics according to original equipment manufacturer standards. Benzoyl chloride-treated *Lycium ferocissimum* fibers had cellulose of 66.85%, while it was 56.12% for untreated *Lycium ferocissimum* fibers. This was due to chemical treatment removing unwanted constituents from the chemical composition. Friction composites made from benzoyl chloride-treated *Lycium ferocissimum* fibers showed improvements in hardness, density, cold shear strength, nominal acetone extraction, and loss on ignition. However, friction composites based on untreated *Lycium ferocissimum* fibers had lower density but a higher loss on ignition, indicating a higher presence of amorphous constituents than crystalline constituents. Commercial friction composites displayed higher density, hardness, and lower porosity than the developed composites. Friction composites made from benzoyl chloride-treated *Lycium ferocissimum* fibers exhibited better recovery rates and maintained good friction values during the overall fade-recovery cycles (Fade-I and II: 10.42, 12.18%) compared to friction composites based on untreated *Lycium ferocissimum* fibers (Fade-I and II: 11.12, 15.15%). This improvement can be attributed to the thermal resistance of *Lycium ferocissimum* fibers achieved through benzoyl chloride treatment. However, the commercial friction composite showed better values in the first cycle of fade and recovery but deteriorated in subsequent cycles. Friction composites made from benzoyl chloride-treated *Lycium ferocissimum* fibers demonstrated better contact plateaus with fewer back transfer patches and more desirable features, confirming their superior performance. Overall, the findings highlight the potential of using benzoyl chloride-treated *Lycium ferocissimum* fibers as reinforcements in friction composites for braking applications.

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