Influence of benzoyl chloride treatment on the tribological characteristics of *Cyperus pangorei* fibers based non-asbestos brake friction composites

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Abstract

Natural fibers are widely used in the various lightweight and medium load applications due to their vital advantages. *Cyperus pangorei* is abundantly found on the river banks. The current study deals with the usage of *Cyperus pangorei* fibers that were retted using a manual retting process in the brake pad formulation. The fibers retted were treated with benzoyl chloride to enhance adhesion characteristics. The benzoyl chloride treated and untreated *Cyperus pangorei* fibers were used in the development of brake pads with other ingredients constant as per industrial practice. The developed brake pads were analyzed for its physical, chemical, thermal and mechanical properties as per industrial standards. The tribological performance of the brake pads was analyzed using a pin-on-disc tribometer as per ASTM G-99-05. It was proved that benzoyl chloride treated *Cyperus pangorei* fibers based brake pads showed excellent performance characteristics compared to untreated one due to better bonding with matrix causing good contact plateaus formations as confirmed from Scanning Electron Microscopy.

1. Introduction

The identification of alternative fiber for an available synthetic fiber is tending to be essential for all the researchers. Even Industries are expecting to replace the synthetic fibers with natural fiber which is biodegradable and non-hazardous [1]. Three main classifications of natural fibers are present namely mineral-based, animal-based and plant-based [2]. Generally plant-based natural fibers are extracted from the stem, root, and leaves [3]. The strength of the natural fibers is also dependent on the part from which it is extracted and environmental conditions of growth which in turn design the chemical constituents and fiber dimensions [4]. Though natural fibers have some vital advantages certain disadvantages like weak bonding, higher moisture content sometimes degrade the performance properties. To overcome such disadvantages, chemical treatment is done [5]. Chemical treatment is a process of removing the surface impurities and hydrophobic constituents from the natural fibers thereby increase the interlocking nature with the matrix. This increases the performance and strength characteristics of the natural fibers when used as reinforcement in the composite materials. There are various chemical treatments involved namely NaOH, Benzoyl chloride, silane, HCl, plasma, KOH etc [6]. Benzoyl chloride treatment was chosen due to its better surface enhancements, as inferred from following literature. The acrylic acid, alkali, sodium chlorite, benzoyl chloride, and potassium permanganate treatments were done Areca Sheath fibers. It was found in the benzoyl chloride treatment that the water absorption behavior got reduced due to replacement of hydroxyl rings by benzoyl rings [7]. In another study, 10% peroxide, 10% benzoyl chloride and 5% alkali were used to treat *Vetiveria zizanioides*. It was seen from the results that benzoyl chloride treatment increased the mechanical properties by removing the unwanted constituents to enhance surface roughness of the fibers leading to better interfacial fiber and matrix bonding [8]. The benzoyl chloride treated and untreated *Impomea pes-caprae* fibers were characterized for chemical analysis, thermal stability, crystallinity index and the so developed composites were analyzed for mechanical properties namely tensile,
flexural and impact as per ASTM. It was found from the test results that benzoic chloride treatment enhanced the characteristics of both fibers and its composites [9]. There are many natural fibers used in the composite applications that too specifically for brake pad applications. Saikrishnan et al [10] developed non-asbestos eco-friendly brake pads by using varying weight percentages of areca sheath fiber. It was revealed that the five weight percentage areca fiber-based brake pads possessed better frictional properties compared to others. Suryarajan et al [11] studied the tribological performance of the silane treated, alkali-treated and untreated Prosopis juliflora fibers based brake pads using Chase test. It was found that five weight percentage silane treated Prosopis juliflora fibers based brake pads showed better friction and wear resistance. Tej Singh et al [12] investigated the tribological performance of various weight percentages of ramie fiber based brake friction composites using Chase testing machine. It was found that five weight percentage ramie fibers based brake pads showed better tribological performance compared to other composites. Naresh kumar et al [13] studied the effect of 5–20 weight % hemp fiber on the mechanical, physical and tribological characteristics of non-asbestos brake friction composites using Chase test. It was inferred from the results that 5–10 weight % hemp fibers in the brake pads increased the friction stability and good fade resistance than that of 15–20 weight % hemp-based brake pads. Zhen-Yu et al [14] investigated the influence of banana fibers with barium sulphate combination on the tribological performance of brake pads using Chase test. The brake pad that was containing 5 weight % banana fiber showed least wear while the pad containing 20 weight% banana fiber exhibited the highest recovery-. Finally the brake pads with ≤ 10 weight % banana fiber and ≥ 45 weight % barium sulphate exhibited better friction performance with least friction undulations. In the quest to find new fibers Cyperus pangorei (CP) fibers seem to be beneficial. It is an invading plant belonging to the family of Cyperaceous. It is found almost in all the Southern parts of India. These plants can grow in wetlands often found in standing waters. Around 0.6 percentage of the geographical area is covered with this species [15, 16].

In the path of finding a novel cellulose fibers Cyperus pangorei (CP) was identified as a natural cellulose fiber that was not used in brake pad formulation till now. The CP fibers were collected and retting process was done manually. The fibers were then treated with a NaOH followed by benzoic chloride solution treatment. The treated and untreated fibers were analyzed for physical and chemical constituents analysis. The primary objective of the current work is to analyze the physical, chemical, thermal, mechanical and tribological properties of the untreated and benzoyl chloride treated Cyperus pangorei (CP) fibers based brake pads that were manufactured using conventional industrial practice.

2.2. Preparation and characterization of the friction composites

The CP fibers in the form of standard brake pads were developed as per the standard procedure. In this both the Untreated and benzoic chloride treated CP fibers were utilized. The brake friction composite formulation possessed 15 parental ingredients (95 weight%) namely (fibers with additives: 13 weight%) acrylic fiber, rock wool fiber, steel fiber, hydrated lime, (binders with additives 16 weight%) phenolic resin, NBR, crumb rubber, CaCO₃, (frictional modifiers 18 weight%) graphite, silicon carbide, (filler: 48 weight%) boron-graphite modified friction dust, vermiculite, synthetic barites, remaining 5 weight% as varying ingredients in the fibers category using benzoic chloride treated CP fibers and untreated CP fibers, that were designated as BTCP and UTCP pads. The conventional methods were used to develop the brake pads, namely mixing in plough shear mixer having three chopper and one shovel rotating in the speed of 2800 rpm and 140 rpm respectively for 20 min, the sequence of mixing followed was fibers for 6 min, then fillers and frictional modifiers for 10 min and finally binders for 4 min. Followed by curing in hydraulic operated compression molding machine at 145 °C for
8 min with five intermittent breathings having pressure of 13 MPa, followed by post-curing in hot air oven for 5.5 h by following step baking process from 140 °C–160 °C for 3 h At 160 °C for 2 h; 160 °C–165 °C for 0.5 h. Finally the finishing was done to remove the unwanted resinous layer and make the pad to the required thickness using belt grinder [17, 18]. The developed brake pads are given in figure 1(a).

2.3. Characterization of developed brake pads

The benzoyl Chloride treated and untreated CP fibers based brake pads were characterized for various properties. The details of the test procedure are given in table 1.

3. Results and discussions

3.1. Physical, chemical and morphological characteristics of benzoyl chloride treated and untreated CP fibers

Untreated CP fibers had a density of 1107 kg m$^{-3}$ while it is 1243 kg m$^{-3}$ for the benzoyl chloride treated CP fibers. This increase in density is due to removal of low molecular weight substances like hemicellulose, lignin from the chemical constituents and also filling of the surface porous with graft molecules of the chemical used [24]. The diameter of the untreated CP fiber is 390 μm, which is higher than the benzoyl chloride treated CP fiber 270 μm. This reduction in diameter is due to the disintegration of the excess amorphous contents present in the fiber, thus making the fiber surface uniform [25]. This increases the aspect ratio of the fiber leading to increase in surface area of fiber in the matrix leading to better adhesion with the matrix that further enhances the strength properties of the composites so developed. There is an increase in cellulose percentage of benzoyl chloride treated CP fibers.

The cellulose percentage increases upon the benzoyl chloride treatment; this increase in value generally helps to increase the strength of the fibers and so developed composites and thermal stability [9]. It is seen from table 2 that the benzoyl chloride treated CP fibers showed reduction in wax, lignin, moisture and hemicellulose contents. This is due to fiber detachment from the bundle upon which removes the above-stated constituents and increasing the α-cellulose [26]. These values are in tandem with the literature findings of Mayandi et al[15]. The reduction in moisture and wax content of benzoyl chloride treated CP fibers is due to remove of hydrophobic contents which help to increase the strength of the composites developed by making interlocks with the matrix due to enhanced surface roughness[5]. The cellulose percentage is higher than Tridax procumbens fiber, Impomea pes-caprae fiber, while it is less than Prosopis juliflora, Flax, Sansevieria cylindrical.

Figure 1. (a) Developed Brake pads using benzoyl chloride treated and untreated CP fibers. (b) Pin on disc test samples.
Table 1. Various properties measured for BTCP and UTCP pads [19–23].

| Composite                        | Property                                      | Procedure/Standard                                                                 |
|----------------------------------|-----------------------------------------------|------------------------------------------------------------------------------------|
| Benzoyl-treated and untreated CP fibers based Brake pads | Density (Machine: Digital density measurement apparatus with accuracy 10 kg m$^{-3}$) | The test was performed as per IS 2742 Part 3 of 2014 based on Archimedes principle |
|                                  | Acetone extraction (Machine: Soxhlet extraction apparatus) | The test was performed as per IS 2742 Part 3 of 2014 was the uncured resins percentage was found |
|                                  | Hardness (Machine: Rockwell hardness tester)    | The test was performed as per IS 2742 Part 3 of 2014 was the uncured resins percentage was found |
|                                  | Shear strength (universal testing machine with shearing fixture) | Test carried out based on ISO-6312 of 2010 at room temperature conditions. In this test, the developed brake pad with the backplate was placed in the shear testing fixture. A side load was applied to the brake pad surface while another load was applied in a perpendicular direction using a rammer. The load was applied gradually until failure. |
|                                  | Friction and Wear Properties (Pin On Disc Tester) | The friction and wear properties were measured using pin-on-disc. It was carried out as per ASTM G99–05 at room temperature conditions (30 °C). A pin-type cutting was made in the form of the square cross-section having dimensions 10 × 10 mm while the length of the pin was 60 mm as shown in figure 2(b). The cast-iron disc had 191 BHN hardness with 160 mm diameter and 15 mm thickness was used. Three loads (2.4 and 6 kg) and three velocity (2.46 m s$^{-1}$) were considered for testing each of 90 min. |

The morphological surface nature of the CP fibers are shown in the SEM images figures 2(a) and (b). From the SEM images it is seen that the micro fibrils of the CP fibers are fused due to the presence of excess amorphous constituents hydrophobic constituents as shown in figure 2(a) leading to low wettability and low interlocking sites as inferred from smooth surface of CP fiber. The effect of benzoyl chloride treatment is seen in figure 2(b). The chemical treatment breaks down the OH groups present in the amorphous constituents and dissolves the excess hydrophobic constituents [28]. This phenomenon leads to considerable defibrillation of the microfibers over the fiber surface causing roughness, thus leading to good wettability. The chemical treatment forms interlock over the surface of the fiber causing better matrix adhesion leading to interfacial bonding [29]. This is because α-cellulose to the amorphous contents ratio of benzoyl chloride treated CP fiber got increased compared to the untreated CP fiber.

3.2. Physical, chemical, thermal and mechanical characteristics of the BTCP and UTCP pads

The test results of the physical, chemical, thermal and mechanical properties of the developed UTCP and BTCP pads are given in table 3. The density of the benzoyl chloride is considerably higher than that of the untreated one. This is mainly attributed due to the presence of voids and pores which got closer due the chemical treatment involved in the process. This increase in the density of the CP fiber decreased the dimensions of the CP fiber which is attributed mainly due the excess removal of lignin, hemicellulose [9]. The amount of uncured resin in the brake pad composites can be calculated with the acetone extraction test. The acetone extraction of benzoyl chloride CP fiber is marginally low than that of the untreated fiber. Both the treated and benzoyl chloride treated composites were within the industrial range of less than 1.5%. It can be concluded that the better compatibility of the CP fiber with resin is ascertained which is in tandem with the literature [30]. The higher loss of ignition is observed to the UTCP pads compared to the BTCP pads. This is mainly due to the low thermal resistance nature of CP fibers when it is untreated due to the presence of hemicellulose, lignin, wax, and moisture which causes such kind. But the values are slightly higher than industrial range of 25% which is similar to the literature findings since the natural fibers are used in the formulation [31]. The hardness of the BTCP pads is slightly higher than that of the UTCP pads. The obtained hardness values were in the industrial range between 80–90 which is in line with the literature [30, 31]. This increase in hardness is mainly due to the better curing of the composites which is caused by the excellent bonding of treated CP fiber with the matrix. The shear strength of the BTCP pads is higher than that of the UTCP pads. It is mainly due to the treated CP fibers forms better surface interlocks, which causes firm bonding with the matrix. The observed values in the shear strength are also within...
## Table 2. Chemical and physical properties of the untreated and benzoyl treated CP fibers in comparison with other natural fibers.

| Natural cellulose fibers        | Chemical properties | Physical properties |
|--------------------------------|---------------------|---------------------|
|                                | α-Cellulose (wt%)   | Hemi-cellulose (wt%)| Lignin (wt%) | Moisture (wt%) | Wax (wt%) | Diameter (μm) | Density (kg/m³) | References |
| Benzyol chloride treated CP    | 72.1               | 9.7                 | 6.1          | 3.8           | 0.54      | 270           | 1243           | Current study |
| Untreated CP                   | 65                 | 15.9                | 9.2          | 7.5           | 1.6       | 390           | 1107           | Current study |
| Benzyol chloride treated Impomea pes-caprae | 65.1             | 30.3                | 8.7          | 3.2           | 0.7       | 179.5         | 1053           | [9]            |
| Untreated Impomea pes-caprae   | 59.4               | 16                  | 12.45        | 8.5           | 1.9       | 203.1         | 987            | [9]            |
| Untreated Tridax procumbens    | 32                 | 6.8                 | 3            | 11.2          | 0.71      | 233.1         | 1348           | [4]            |
| Treated Tridax procumbens      | 45                 | 3.6                 | 2.1          | 9.6           | 0.49      | 169.7         | 1160           | [4]            |
| Prosopis juliflora             | 61.65              | 16.14               | 17.11        | 9.48          | 0.61      | 20            | 580            | [24]           |
| Benzyol Peroxide Treated Prosopis juliflora | 82.09            | 2.6                 | 9.12         | 4.67          | 0.07      | —             | 921            | [24]           |
| Sansevieria cylindrica         | 79.7               | 10.13               | 3.8          | 3.08          | 0.09      | 230–280       | 880            | [27]           |
| Flax                           | 81                 | 14                  | 3            | 10            | —         | 50–300        | 1500           | [25]           |
the industrial range as per the literature [32]. Based on the results it can be concluded that the benzoyl chloride treated CP fibers have better bonding with the matrix and the resin leading to increase the mechanical strength of the brake pad composites [9].

3.3. Tribological characteristics of the BTCP and UTCP pads

3.3.1. Frictional performance of BTCP and UTCP pads

3.3.1.1. Influence of load on the friction performance of brake pads

There is a reduction in $\mu$ with an increase in the load applied [33]. Here in the current study three loads were used to test the $\mu$ performance of BTCP and UTCP pads. The $\mu$ graphs of BTCP and UTCP at 2 kg load are given in figures 3 (a) and (b). The average $\mu$ for 2 kg loading for BTCP pads is 0.47, while it is 0.42 in the case of UTCP pads. As the load increases there is a reduction in $\mu$ in both the cases. For 4 kg load in BTCP pads it is 0.44 while it is 0.38 for UTCP. Finally for 6 kg, $\mu$ is 0.42 and 0.34 for BTCP and UTCP pads respectively as shown in figure 4.

The $\mu$ obtained in this study is in a range of 0.3—0.55 acceptable range as per industrial standards. It is seen in the results that $\mu$ reduced drastically in the case of UTCP while it decreased slightly in the case of BTCP pads. This reduced in $\mu$ is mainly due to the reduction in the space between pin and disc thus leading to wear debris agglomeration. Another possible reason could be due to increase in the real contact thus inducing the temperature rise at the interface causing asperities breakage by thermo-mechanical overloading. This exposes plateaus and sudden variations in the friction coefficient [34]. This temperature rise leading to the softening of the fibers with the matrix, in the case of UTCP pads where the untreated CP fibers have smooth surface, which doesn’t form more bonding with resin compared to benzoyl chloride treated CP fibers gets detached from the matrix. Thus the resin and debonded fiber with amorphous constituents form a thin film over the interface, causing more reduction in $\mu$. This is tandem with the literature findings of PJF and shell powders based brake pads [35].

As shown in the figures 3(a) and (b) the $\mu$ reaches stabilization after few cycles which is due to the establishment of real contact with the mating even though the burnish cycle was performed before the testing. This behavior is tandem with the literature [36].

3.3.1.2. Influence of speed on the friction performance of brake pads

The $\mu$ of the different speeds are given in figure 5. As the sliding speed increases, the $\mu$ of the brake pads decreases mainly due to the increase in kinetic energy, which causes increase in the temperature at the interface leading to resin softening further, enabling debonding of fiber and some powdery ingredients forming third bodies [36]. As
Figure 3. (a) Time versus $\mu$ for BTCP composites at 2 kg load conditions. (b) Time versus $\mu$ for UTCP composites at 2 kg load conditions.

Figure 4. $\mu$ for three different loads of BTCP and UTCP pads.
The speed increases, the asperities and mating surface contact get reduced leading to reduction in $\mu$. The contact plateaus overloading due to increased pressure deforms the asperity and reduced $\mu$ [16]. In the current study, frictional value decreases due to increased speed. The BPCP pads showed lesser variation in $\mu$. As it is essential to maintain the least sensitivity to $\mu$ since the driver requires same level of $\mu$ throughout the braking service life [37]. The better composite needs to maintain least sensitivity toward friction as the driver needs the same level of friction throughout the braking period irrespective of conditions. At lower speed both the pads showed higher value of $\mu$ compared to higher speeds. This is due to less formation of pyrolysis layer at lower speeds preventing $\mu$ reduction [38]. In the case of UTCP pads there was more reduction in $\mu$ levels at higher speed which is mainly due to the improper bonding of the untreated CP fibers with the matrix leading to such results. Also fiber must have higher thermal stability to withstand the temperature rise; the benzoyl chloride treated CP fibers being thermally stable showed good results without degrading the $\mu$ values drastically.

3.3.2. Wear of brake pads

The wear rate of the brake pads is given in figures 6(a) and (b) for various load and speed conditions. The increase in load and speed increased the wear rate; in spite of that BTCP pads showed less wear rate compared to UTCP pads. The frictional heat evolved in the mating interface causes thermal softening and plastic deformation of matrix, leading to debonding of materials. At higher load conditions, the materials increase the area of contact leading to wear loss. In reduced load conditions there is less formation of a resinous layer over the mating surface and hence is less compared to higher load conditions [34]. At high speed, debris in the counter face increases the impact based repeated loading on tribo surface. This frictional thrust and constrained vibration are due to this impact based loading, resulting in additional debonding and surface cracking [39]. Another reason is that least thermal stability of untreated CP fibers causes more wear due to the easy thermal degradation compared to treated one. Also the higher the hardness produces more resistance to penetration that in turn helps to reduce the wear. This postulate is proven in the current study [40].

3.4. Worn surface characteristics of the pin on disc tested BTCP and UTCP pads

SEM images of high load-tested BTCP and UTCP pads are given in figures 7(a) and (b), respectively. It can be visualized that large-sized primary contact plateaus which enhance friction levels. Further the back transfer patches are also comparatively less. Certain wear debris doesn’t form patches when it is large-sized; it becomes as third bodies and increases the $\mu$ levels. A similar phenomenon is seen in the current study in the case of BTCP pads as shown in figure 7(a). There is an ironing mechanism formation in the primary plateau region which indicates mild wear. This is tandem with the literature [41, 42]. This higher contact plateau formation maintains the $\mu$ consistency level without causing much undulations.

It is seen in UTCP pads in figure 7(b), which has more nucleated primary plateaus and secondary plateaus with a higher back transfer of polymeric ingredients. It is important to note that higher the back transfer, poor the wear resistance [20]. It is more severe due to least thermal stability of untreated CP fibers that resulting in higher wear rate and additional patches of polymeric ingredients due to back transfer from the mating surface. Poor wear resistance also leads to more crack formation and propulsion as inferred from figure 7(b). Also poor resistance further enables the debonding of the plateaus due to higher increase in the interface temperature.
Overall, BTCP pads revealed improved plateau formation, less back transfer and debris resulting in improved wear resistance.

Future scope of the current study is to analyze the thermal stability of the fibers, pads developed and analyze the tribological performance using a full-scale dynamometer. Then to analyze the back transfer using elemental mapping.

4. Conclusions

Natural fibers namely *Cyperus pangorei* were treated with benzoyl chloride and utilized for brake pads development as per the standard industrial practice in comparison with untreated fiber-based one. The fibers
and developed brake pads were analyzed for various properties as per standard procedure and the following conclusions were drawn:

- Benzoyl chloride treated Cyperus pangorei fibers showed higher cellulose content with higher density with a reduction in hydrophobic contents compared to untreated fibers.

- Benzoyl chloride treated Cyperus pangorei fibers based brake pads revealed favorable characteristics like higher hardness, low loss on ignition, good shear strength, less acetone extraction compared to untreated Cyperus pangorei fibers based brake pads which showed lesser density.

- Benzoyl chloride treated Cyperus pangorei fibers based brake pads had good friction and wear resistance characteristics under different load and speed conditions due to higher thermal stability and increased hardness due to better bonding of fibers with the resin.

- Benzoyl chloride treated Cyperus pangorei fibers based brake pads showed more primary plateaus and less secondary plateaus due to interlocks on the surface of benzoyl chloride treated Cyperus pangorei fibers leading to better bonding with the resin.

Overall, based on the above results, Benzoyl chloride treated Cyperus pangorei fibers based brake pads can be used effectively for brake pad applications in automobiles.

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