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Asbestos free brake pad using Micro cellulose fibre for automotive industry

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ABSTRACT

In this work application of Micro cellulose fibre in brake pad composites were successfully carried out. Micro cellulose fibres were modified with 3-aminopropyltriethoxy-silane before using in the brake pad. Four different brake pads were prepared using compression moulding process with the different ratio of micro cellulose fibre along with, phenolic resin, barium sulphate, aluminium oxide and graphite. Properties of prepared brake pads were evaluated in terms of density, water and oil soaking test, compressive strength, hardness, flame resistance, wear rate, X-ray diffraction, thermogravimetric analysis, the coefficient of friction and scanning electron microscope. Brake pad composition with 10% weight of micro cellulose fibre showed the promising result with respect to other attempted compositions and are comparable to commercial sample.

Keywords— *Brake pad, Micro cellulose fibres, Silane, Coefficient of friction, Wear rate*

1. INTRODUCTION

Automotive brake pad materials which date back to 117 years ago are usually made with mixtures of asbestos, metals, and ceramics. But the asbestos component has been found to release gases hazardous to human health upon the use of brake pads. Asbestos has long been known to cause diseases like asbestosis, mesothelioma, lung and other cancers. After that number of researches have been on to discover eco-friendly material replacements for asbestos. In brake pad composites, Asbestos imparts desired high friction property which used as motion stopper. Brake pads are important components of a braking system for all categories of vehicles equipped with brake discs. They are steel backing plates with friction materials bonded onto the surface facing the brake disc and are placed in the wheel assembly to continuously clamp and hold wheels to slow or stop their motion (Aigbodion et al., 2010).

There are two basic types of automobile brakes: drum brakes and disc brakes. In drum brakes, the brake shoes are located inside a drum. When the brakes are applied, the brake shoe is forced outward and presses against the drum. One of the major differences between drum brakes and disc brakes is that drum brakes tend to be enclosed where disc brakes tend to be exposed to the environment (Aigbodion et al., 2010, Bono et al., 1990).

The brake pads generally consist of asbestos fibers embedded in a polymeric matrix along with several other ingredients. Over the few years, several research works have been carried out in the area of development of asbestos-free brake pads. The use of bagasse (Aigbodion et al., 2010), palm kernel shell (Ibhadode et al., 2008) and Banana Peel (Idris et al., 2015) have been investigated in order to replace the asbestos-free brake pad material. A recent trend in the research field is the employment of industrial or agricultural wastes as a source of raw materials for composite development. This will provide more economical benefits and also environmental preservation by utilizing the waste of natural fibre and also Natural Fibres.
Asbestos has now been replaced by a mixture of alternative fibres such as agricultural waste, mineral fibres, cellulose, aramid, PAN, chopped glass, steel, and copper fibres. The current research work focuses on the application of micro cellulose fibres as filler in brake pad composites.

2. EXPERIMENTAL PROCEDURE

2.1 Materials/Equipment

The materials and equipment's used during the course of this work are: Phenolic resin (phenol formaldehyde), Micro cellulose fibre procured from Rettenmaier India Pvt Ltd, Graphite, Aluminium oxide, zinc oxide, barium sulphate, engine oil (SEA 20/50), water, hydraulic press, brake pad mould, heater, digital weighing balance, hardness tester and scanning electron microscope(SEM).

2.2 Methods

2.2.1 Treatment of micro cellulose fibres

Micro cellulose fibres were washed with 25% detergent solution at 70^0 C for 1hr followed by distilled water for 10 min and dried at 80^0C for 1 hr. Micro cellulose fibres were further treated with 5% ethanol (on the weight of fibres) at 30^0C for 2hrs followed by washing with distilled water and drying at room temperature for 24 hrs. Cellulose fibres were then treated with 5 % NaOH (on the weight of fibres) at room temperature for 4 hours. After the alkali treatment, fibres were washed multiple times to remove soluble impurities and

remaining sodium hydroxide. After washing, treated fibres were neutralized with acetic acid to get final pH 7 and dried at temperature 80°C for 6 hours. (Ray et al., 2001) This NaOH treated cellulose fibres were subsequently treated with 5% (on the weight of fibre) 3-amino propyl triethoxy silane, which was added to the ethanol: water mixture present in the ratio of 80:20. This was followed by curing at 110°C in a hot air oven for 60 minutes**.** (kim et al., 2014).

2.2.2 Preparation of brake pad composites

A metal mould of size 168mm x 98mm x 9mm was fabricated for preparation of brake pad. This mould was used to prepare brake pad composite in compression moulding technique. A polyethylene sheet (PET) was placed at the bottom of the mould. The constituents were properly mixed in a blender. Homogenous mixture was transferred to mould kept in a hot platen press at a temperature of 150^{0}C at 100 kg/cm^2 pressure for 4-5 min. The prepared brake pad samples of varied composition were cured in an oven at a temperature of 150^{0}C for 6 h. Four different brake pad composites were prepared using micro cellulosic fibres as fillers partially replacing barium sulphate in composition as shown in table 1.

Table 1: Sample formulation of brake pad from micro cellulose fibre

Material		Percentage (%) formulations			
		1	**2**	**3**	**4**
Binder	**Phenolic Resin**	30	30	30	30
Filler	**Barium Sulphate**	44	42	40	38
Abrasive	**Aluminium Oxide**	6	6	6	6
Lubricant	**Graphite**	10	10	10	10
Other ingredients	**Zinc Oxide, Chromium oxide**	4	4	4	4
Reinforcing Fibre	**Cellulose Fibre**	6	8	10	12

Fig. 1: Brake pads from micro-cellulose fibres

3. CHARACTERIZATION

3.1 Density measurement

Specific gravity measures density which depends upon the ingredient of the brake material formulation. The true densities of the specimen were determined by weighing the specimen on a digital weighing machine and measuring their volume by liquid displacement method. The specific gravity formula is (as Eq. (1)):

$$Density(\rho) = \frac{Mass}{Volume} \quad (1)$$

3.2 Water & oil absorbency

Samples were cut as per ASTM D570 standard and dried at 70^{0}C and weighted. These samples were immersed in water and oil for 24 hours and weighted. The following formula was used to calculate water and oil absorbency.

$$Water\ and\ oil\ absorbence\ (\%) = \frac{Wi-Wf}{wi} \times 100 \quad (2)$$

Where,

W_i = Initial weight of sample

W_f = Final weight of sample

3.3 Hardness testing

Rockwell Hardness is used to evaluate the bulk hardness of a material. Hardness correlates with strength, wear resistance, and other properties. The hardness testing was done according to ASTM D785 method.

3.4 Compressive strength

Compressive strength tests were carried out by Tinius Olsen universal testing machine as per ASTM D695 at a crosshead speed of 5 mm/min. To obtain a statistically significant result for each set, samples were tested three times to evaluate the mechanical properties.

3.5 Flame resistance

The flame resistance of the brake pad material was determined. Brake pad was placed on the wire gauze and burned using the blue flame of a bunsen burner. The specimens weight before and after burning were taken after 1 min on the flame.

3.6 Wear resistance

Each wear/abrasion resistance specimen measured 110mm in diameter by 3mm thickness. The test was conducted with taber abrasion tester. Each specimen was weighed and recorded as W_0 before the test. The specimen was fixed to a 100 mm diameter rotary disc of the machine with the aid of a nut in its center. Two emery wheels at the upper arm of the machine were allowed to have direct contact with the specimen fastened to the rotary disc. The machine was powered on at 220 V and disc rotated at a constant speed of 85 revs/min for the pre-set time of 1000 seconds. After completing the pre-set time, the machine was turned off automatically. The specimen was thoroughly cleaned and final weight measured and recorded as W_1.

$$Wear\ Rate = \frac{W0-W1}{S} = \frac{\Delta W}{2\pi ND \times t} \quad (3)$$

Where W_0- Initial weight; W_1- Final weight; ΔW- Weight loss; S- sliding distance; D- Disc diameter; N- radial speed (rpm) and t- time taken to expose specimen to wear.

3.7 Friction test

The brake pad samples were tested using a link chase machine. The sample was fixed against a rotating brake drum with a constant rotating speed of 417 rpm under the load of 150 psi in accordance with Society of Automotive Engineers (SAE) J- 661.

3.8 X-Ray diffraction analysis

The crystallinity of brake pad samples was measured with an X-ray diffractometer, XRD-6100 (Shimadzu Corporation, Japan) at 40 kV and 30 mA. The experiments were performed in a symmetrical reflection mode with Cu Kα radiation (λ = 1.5405 Å). The angular range (2Θ) was taken from 10° to 50°. The measurement was made with the sampling pitch of 0.02° using continuous mode of scanning, at the rate of 2°/min. Using the Segal method crystallinity index (ICr) of Brake pad samples was calculated.

3.9 Thermal gravimetric analysis

TGA of composite samples was done with Shimadzu 8400S and the degradation of the sample with respect to temperature was studied. The experiment was done from 30 to 500˚C with a heating rate of 20˚C/min.

3.10 SEM

The morphology of the brake pad composites samples was analyzed using SEM, XL 30 (Philips, The Netherlands). Prior to scanning, all samples were coated with gold for 3 to 4 minutes using sputter coater to dissipate the static charges occurring due to electron bombardment with accelerated electrons having 10 kv energy.

4. RESULT AND DISCUSSION

4.1 Density

Density measurement test has been carried out at laboratory scale to examine the density of the material using water displacement method. Density depends upon the ingredients in the brake pad composition. The average of three readings for each composition was recorded and reported as shown in figure 2. It has been observed that sample containing 6% micro cellulose fibre (sample 1) has the highest density of 1.98 g/cm^3 and a sample containing 12% micro cellulose fibre (sample 4) has a low density of 1.65g/cm^3. As the cellulose content in the brake pad composition increases the density decreases because the metallic element in the form of barium sulphate decreases in the total composition of brake pad which has a higher density than cellulose.

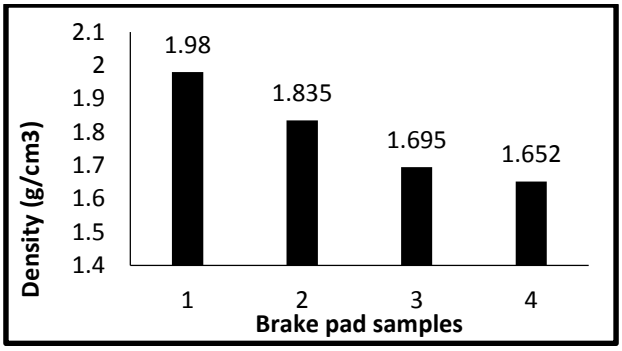

Fig. 2: Density of micro cellulose fibre brake pads

4.2 Water and oil absorption test

The water and oil uptake increased as the natural filler content increased. The trend for the water absorption is sample 4 >sample 3>sample 2>sample1. Since natural fibres are strongly hydrophilic materials due to the presence of hydroxyl groups (–OH) which form hydrogen bonds between the fillers and water thus increasing the water uptake of the composites. Figure 3 shows the average values of the water and oil absorption for 24 hours. With the increase in the immersion time, the water and oil uptake significantly increases up to 120 hrs. With further increase in the immersion time from 120 hrs to 144 hrs the rate of water uptake was found to be low which can be attributed to the saturation of the fibres. Oil is absorbed into the material by a two-step mechanism: first, oil is absorbed by capillary action into voids associated with the fibres within the cellulose; then a slow diffusion of oil into the bulk phenolic resin takes place (Bertrand 1990**).**

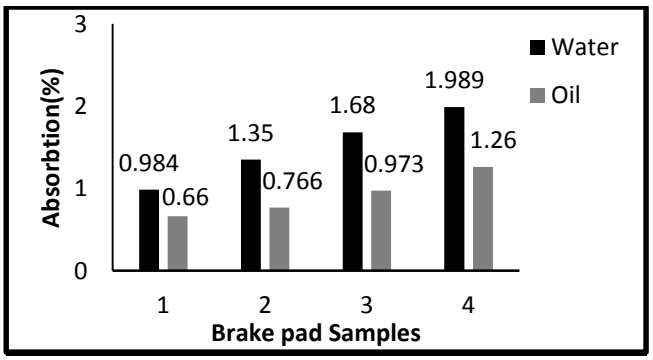

Fig. 3: Water and Oil absorption of micro cellulose fibre brake pads

4.3 Hardness testing

The hardness test performed using the Rockwell hardness test method. This was preferred because it can be used for non-metals. Indenters used during the test are small hence do not destroy the specimen. It is a high speed test and can be performed on the non-reflective surface. The hardness of sample 3 was higher as compared to sample 1, sample 2 and sample 4. Sample 3 has 99 Brinell hardness as shown in figure 4. The Hardness values increase with an increased weight percentage of micro cellulose fibre but sample 4 showed low hardness value. From figure11 it is observed that in the figure11 (a) there is more uniform distribution of the resin with the micro cellulose fibres. This is attributed to proper bonding between micro cellulose fibres and the resin and it results to closer inter-packing interface but in figure 11 (b) there is no uniform distribution and no proper bonding between micro cellulose fibres and phenolic resin because of the higher micro cellulose fibre content. Therefore there is the formation of a void also.

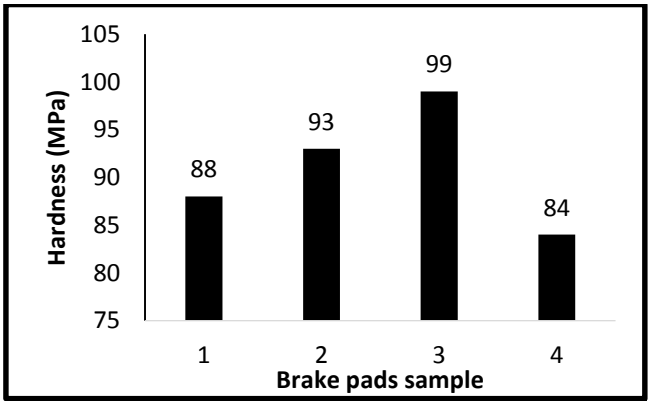

Fig. 4: Hardness of micro cellulose fibre brake pads

4.4 Compressive strength

The compressive strength test results are presented in figure 5. Sample 3 has higher compressive strength as compared to other samples except for commercial sample. The average compressive strength of sample 3 was 105.6 MPa, while in the literature the recommended compressive strength for brake materials ranges from 70– 125 MPa. (Hooton 1969). All values are within the recommended values. The results have a similar trend with that of hardness values. The compressive strength increases with increase in the wt% of micro cellulose fibres. Good distribution and dispersion of the micro cellulose fibres and resin resulting in strong-fibres-resin interaction have the influence on the compressive strength of the samples. However, in the Sample 4 interaction of the fibres and resin is weak because of excess fibre content in the composition, because of which it shows less compressive strength than other samples.

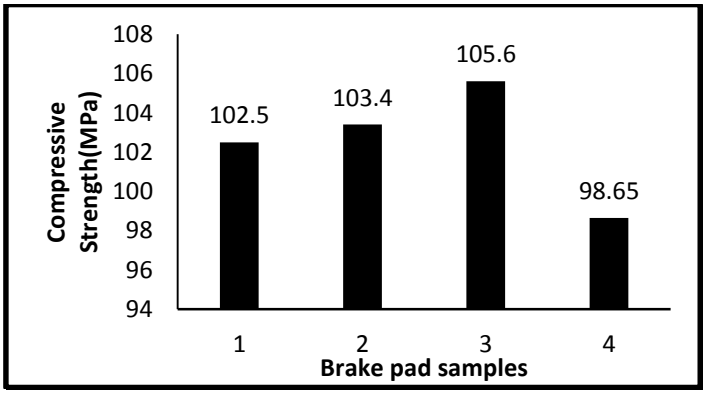

Fig. 5: Compressive Strength of micro cellulose fibre brake pads

4.5 Flame resistance

The flame resistance results are shown in Figure 6. The samples were exposed to Bunsen burner flame for 10 minutes and the charring weight of the samples after the exposure was recorded. The samples did not burn after 10 minute exposure time, however, the samples were charred. From the results, it was observed ash percentage in the charred samples were increased with the increase in weight percentage of the micro cellulose fibre in the composition. Sample 1 shows less ash (%), sample 4 shows high ash (%). The prepared samples reflected the loss of more volatile compounds because of the presence of micro cellulose fibre.

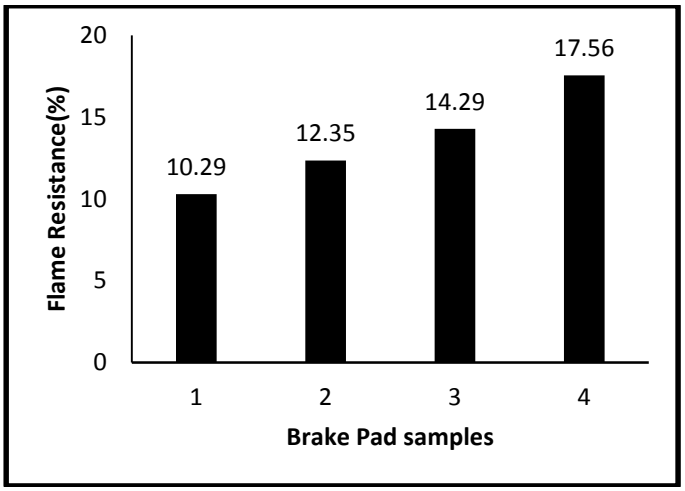

Fig. 6: Flame resistance of Micro cellulose Brake Pads

4.6 Wear rate

The wear rates were plotted against composites brake pad compositions and commercial brake pad for comparison in figure 7 which shows that wear rate of experimental brake pads decreased with increased fibre content from 6 to 10 % in the composition. The decreased wear rate of the brake pad composition can be attributed to higher load bearing capacity of composition and the better interfacial bond between the micro cellulose fibre and the phenolic resin. The fibre acted both as friction and abrasion resistant material in the composite. Sample 4 with a high weight percentage of fibre showed a higher wear rate it may be because of the weak interfacial bonding between fibre and phenolic resin. However, the wear rates compare closely with that of conventional brake pads that has wear rate of 3.80 × 10-6 g/m **(**Ibhadode et al., 2008**)**. Wear process involves adhesion, delamination, fracture, tribochemical effects and plastic flow (Anderson 1980).

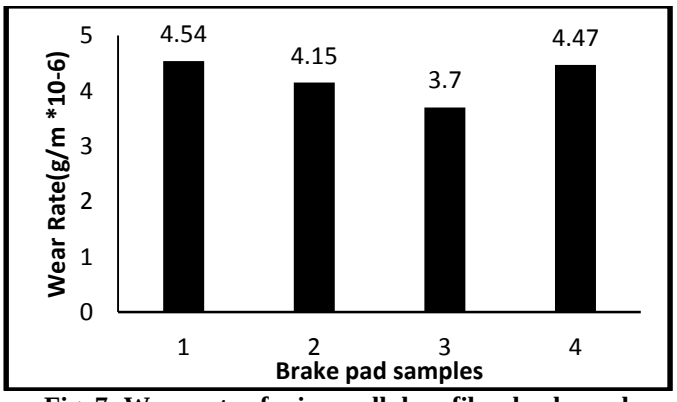

Fig. 7: Wear rate of micro cellulose fibre brake pads

4.7 Coefficient of friction

The coefficient of friction was used to plot the graph presented in figure 8 against the experimental brake pad compositions. In figure 8, the coefficient of friction decreased as the filler content of the mix decreased in each composition. The coefficient of friction of each sample composition compares favourably with that of a conventional brake pad obtained from a standard (Hooton 1969). The values of all brake pad composition with micro cellulose fibres were 0.289, 0.338, 0.357, and 0.386 respectively. The coefficient of friction of any conventional brake pad is usually in the range 0.3 - 0.4 **(**Ibhadode et al., 2008**)**.

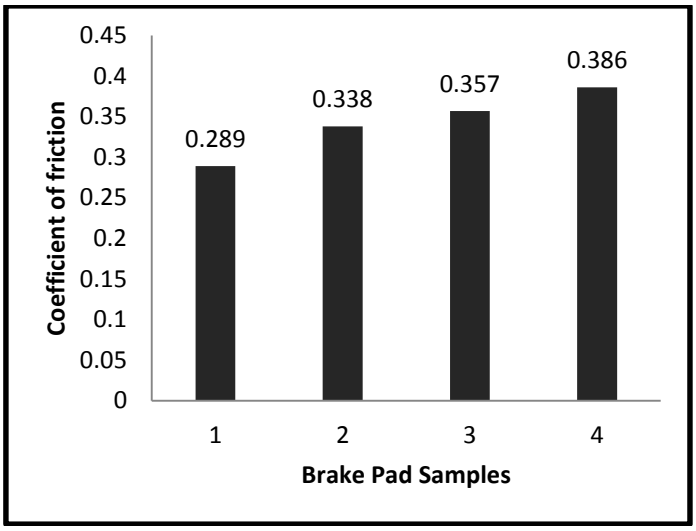

Fig. 8: Coefficient of friction of micro cellulose fibre brake pad

4.8 Thermal Gravimetric Analysis (TGA)

In a braking system, the mechanical energy is transformed into calorific energy. This energy is characterized by total heating of the disc and pads during the braking phase. The energy dissipated in the form of heat shows a rise in temperature ranging from 200 °C to 800 °C. The TGA study helps us understand the break pad wear over a wide temperature range. In figure 9, a comparison is made between the thermal stability of compositions in Sample1, sample2, sample3, and sample4. All specimen samples show thermal stability to a temperature of 300^{o}C with minimal weight loss. All samples show thermal stability up to 500^{o}C with different weight losses, Sample 1 looses 13.55%. Sample 2 looses 11.389%, Sample 3 looses 10.751% and Sample 4 looses 22.205% of its total weight respectively. Sample 3 is found to be thermally more stable than another specimen until this temperature.

In figure 9, it can be seen that small weight losses occur at 200^{o}C due to the curing behaviour of the phenolic resin that was used as a polymeric binder in the composite brake pad. Weight loss due to thermal degradation can be observed in two regions; between 200^{0}C-400^{o}C due to decomposition of binder and above 400^{o}C due to combustion of graphite. However, the weight losses in the region above 400^{o}C are larger.

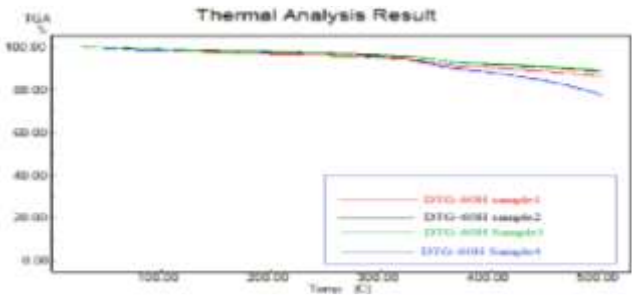

Fig. 9: Thermal analysis of micro cellulose brake pads

4.9 X-Ray diffraction analysis

Figure 10 shows the X-ray diffraction patterns of prepared brake pad samples. Sample 1 shows strong peaks at 26.1º, 29.0º, 42.8º, 43.1º, 32º, and 35.8º. Sample 2 shows strong peaks at 26.3º, 43.04º, 29.20º, 43.34º and 27.2º. Sample 3 shows strong diffraction peaks at 29°, 43.5°, 44°, 6.5°, 27°, 21°, 23°, 32°, 33°, and 36°. Sample 4 shows strong diffraction peaks at 27.3º, 43.04º, 29.20º, 42.34º and 25.2º. These peaks correspond to the various components of the composite; each component in its particular phase exhibits a particular diffraction angle. The components present in the composite material are significantly crystalline which is evident from the sharpness of the peaks.

The presence of various components was confirmed by comparing obtained XRD spectra with the Joint Committee on Powder Diffraction Standards (JCPDS). The strong peak at 29 or 30 corresponds to aluminium oxide (JCPDS card 10-173). Peaks at 43.5 and 44 confirmed presence of barium sulphate (JCPDS card 024-1035). Presence of graphite is evident from the peaks at 26.5 and 27, correlated with JCPDS card 41-1487. Prominent peaks at 32, 33 and 36 relate to Zinc oxide (JCPDS card 36-1451). The presence of micro cellulose is evident from the peaks at 21 and 23 (JCPDS card 00-056-1718).

The sample 1 and sample 3 are significantly crystalline as compared to other samples, which is evident from the sharpness of the peaks. The sample1 and sample 3 showed a crystallinity of 36.53% and 36.766% respectively. The higher crystallinity of the prepared samples can be attributed to the complex interactions of the particulate additives and their chemical reactions. These properties have a significant positive impact on the braking performance.

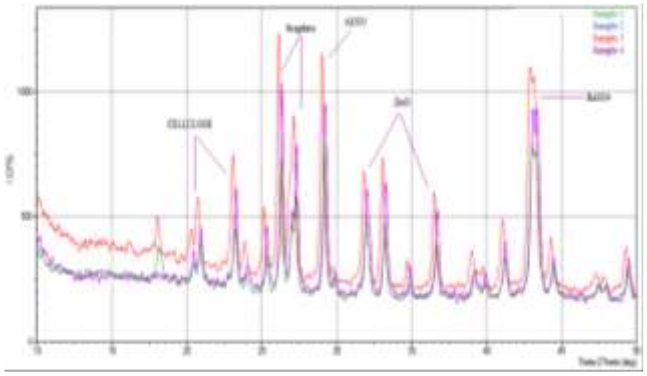

Fig. 10: XRD of micro cellulose fibre Brake Pads

4.10 SEM

SEM micrographs were taken for the sample 3 and sample 4 of brake pad composites. From Figure11 (a), there is the more uniform distribution of the phenolic resin with the micro cellulose fibres. This is attributed to proper bonding between micro cellulose fibres and the resin and it results to closer inter-packing interface. This can be noticeable if one compares Figure 11 (a) with Figure 11 (b). But in figure 11 (b) there is no uniform distribution and no proper bonding between micro cellulose fibres and phenolic resin because of the higher micro cellulose fibre content. Therefore there is the formation of void also shown in the figure with RED circles.

The microstructure reveals that there are small discontinuities and a reasonably uniform distribution of micro cellulose fibres and the resin. The micro cellulose fibres phase is shown as a white phase, while the resin phase is dark. (1)

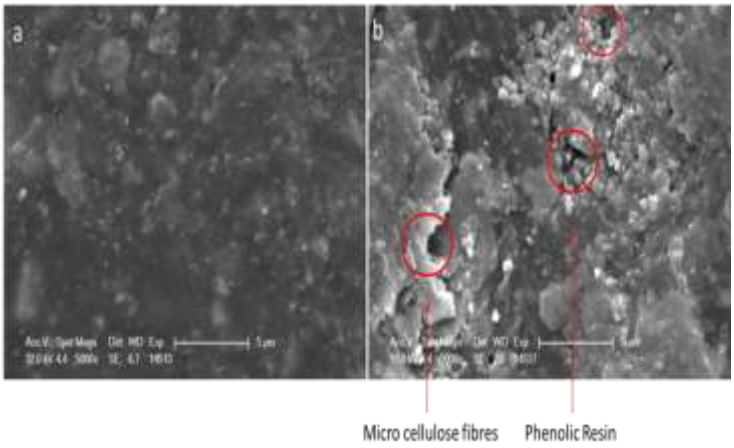

Fig. 11: SEM of micro cellulose fibres based brake pad

5. CONCLUSION

Increasing environmental awareness throughout the world has triggered a paradigm shift towards designing materials which are compatible with the environment. Asbestos free brake pads were successfully prepared using micro cellulose as a reinforcement material and properties of prepared brake pads were evaluated. The brake pad containing 10 % micro-cellulose fibre showed comparable results with the commercial brake pad. The wear rate of the prepared brake pad was found 3.7mg/m which is comparable to the commercial brake pad. The coefficient of friction was found 0.357 which is in the range of commercial brake pads. Thermal stability of prepared composite samples having different composition was very good up to 500ºC. The density hardness compressive strength and other properties were also comparable to commercial brake pad and agro fibres brake pad as shown in Table 2. These results indicate that micro cellulose fibres can be effectively used as a replacement for asbestos in the brake pad.

Table 2: Properties of different agricultural wastes fibers for brake pad composites (Mamaheswara Rao et al., 2015)

Properties	Commercial brake pad (asbestos based)	Laboratory formulation (palm kernel shell)	Laboratory formulation (Bagasse)	Laboratory formulation (Banana peels)	New Laboratory formulation (Micro cellulose fibres)
Density (g/cm^3)	1.89	1.65	1.43	1.20	1.664
Wear rate (mg/m)	3.80	4.40	4.20	4.67	3.7
Coefficient of friction	0.3-0.4	0.440	0.420	0.35	0.357
Thickness swells in water (%)	0.9	5.03	3.48	3.0	2.5
Thickness swells in engine oil (%)	0.30	0.44	1.11	1.12	0.49
Hardness Values (HRB)	101	92	100.5	71.6	88
Compressive strength (N/mm^2)	110	103.5	105.6	61.20	105.6
Flame Resistance (%)	Charred ash 9%	Charred ash 46%	Charred ash 34%	Charred ash 12%	Charred ash 14%

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