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UNRAVELLING THE TRIBOLOGICAL BEHAVIOUR OF MERCERIZED COCONUT INFLORESCENCE FIBER FORTIFIED UNSATURATED POLYESTER COMPOSITES

POJASNITEV TRIBOLOŠKEGA OBNAŠANJA MERCERIZIRANIH INFLORESCENTNIH VLAKEN KOKOSOVIH OREHOV V OJAČANIH NENASIČENIH POLIESTERSKIH KOMPOZITIH

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The need for eco-friendly materials made researchers move towards lignocellulose fibres as potential fortification materials for polymer matrices. In this regard, a unique fibre known as the coconut inflorescence fiber was extricated from the coconut tree and added to unsaturated polyester resin. As the fibres are subjected to mercerization treatment, XRD and FTIR studies show that the amorphous materials present in the fibers are removed, and the tensile strength of the fibre increases. For the tribology studies of the CIF/polyester composite, the load and sliding distance were chosen as the operation parameters under dry-sliding condition. Extensive testing demonstrated that the wear rate increases as the load increases, and it is reduced as the fiber volume faction increases. Due to the hardness of the composite materials, the wear rate decreases as the sliding distance rises. The composites with fiber volume fraction 25 % exhibit the minimum wear rate of the entire experimentation. Furthermore, the friction coefficient drops as the load and sliding distance increase with the increasing volume fraction, which is due to micro-melting generated by the frictional heat at greater loads. A SEM analysis revealed fiber pull-outs in composites with fiber volume fraction 30 %, owing to a lack of fibril wetting during the manufacturing of composites.

Keywords: coconut inflorescence fibre, unsaturated polyester resin, mercerization, wear, friction coefficient, SEM analysis

Zaradi vse večje potrebe po ekološko prijaznih polimernih materialih so raziskovalci pričeli raziskave na področju materialov ojačanih z ligninsko celuloznimi vlakni. V tem smislu so edinstvena vlakna znana kot kokosova inflorescenčna vlakna (CIF; angl.: Coconut Inflorescence Fibre), ki se pridobivajo z izmotavanjem dreves kokosa in njihovim uvajanjem kot ojačitveno fazo v nenasičeno poliestersko smolo. Vlakna so obdelali s postopkom mercerizacije, ki se izvorno imenuje po iznajditelju postopka Johnu Mercerju. S kavstično sodo obdelana vlakna so analizirali z rentgensko difrakcijsko spektroskopijo (XRD) in spektroskopijo na osnovi Fourerjeve transformirane infrardeče svetlobe (FTIR), da bi potrdili odstranitev v vlaknih prisotne amorfne materiale in tako povečali njihovo natezno trdnost. Izvajanje triboloških preizkusov izdelanih CIF/poliester kompozitov je temeljilo na zasledovanju drsne razdalje pod izbrano obremenitvijo v pogojih suhe drsne obrabe. Obširni preizkusi so pokazali, da hitrost obrabe narašča z naraščajočo obremenitvijo in zmanjševanjem volumskega deleža vlaken v kompozitu. Povečevanje trdote CIF/poliester kompozita povzroča zmanjševanje hitrosti njegove obrabe oziroma podaljševanje drsne poti oz. razdalje. Od vseh izdelanih kompozitov je imel kompozit z volumskim deležem vlaken 25 % najboljšo odpornost proti obrabi. Nadalje so avtorji s tribološkimi preizkusi ugotovili, da se koeficient trenja zmanjšuje s povečevanjem obremenitve in drsne razdalje v celotnem obsegu povečevanja volumskega deleža vlaken. To pripisujejo mikrotaljenju površine materiala zaradi trenja oziroma segrevanja materiala pri višjih obremenitvah. SEM analize kompozitov so pokazale, da je prišlo do puljenja vlaken iz kompozitne matrice pri prostorninskem deležu vlaken nad 30 %. To pripisujejo pomanjkanju omakanja vlaken s polimerom med izdelavo kompozitov.

Ključne besede: kokosova inflorescenčna vlakna, smola nenasičenega poliestra, mercerizacija, obraba, koeficient trenja, analiza s pomočjo vrstičnega elektronskega mikroskopa

1 INTRODUCTION

A composite is a material that is made up of two or more substances, of which one is known as the reinforcing member (typically fibres, sheets and particulates), while the other is known as the matrix member which may be in the form of metal, polymer or ceramic. Composites in general have a fiber or particulate phase that exhibits stiffness and strength when compared to the matrix material. The fiber material or particulate serves as the principal load-bearing material and the matrix serves as the load-transmission element; to be specific, the matrix has to withstand loads that are transverse to the fibril axis. The fibre reinforcement of the matrix modifies the brittle form of the matrix into a ductile material. The matrix also protects the filler material from environmental attacks before and after the fabrication of composites. When designed and fabricated effectively, a newly developed composite exhibits better properties compared to those of the individual source materials. Composite materials find promising applications ranging from day-to-day household appliances to aeronautical applications.

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The present research is about the natural-fibre reinforcement of polymer matrices.

The reinforcement material employed in this research is extracted from renewable sources, which are available abundantly. Increasing environmental legislations force researchers to explore various natural reinforcement materials to suit a wide variety of application components. The supremacy of natural lignocellulose fibres is due to their being renewable, biodegradable, exhibiting low density and excellent physical properties. The factors that inhibit natural fibers from acting as the reinforcement material with polymer matrices are dimensional instability and a hydrophilic tendency, resulting in a lack of adhesion between the natural fibril and polymer matrix. After extraction, natural lignocellulose fibres must be subjected to surface modifications that enable them to be used as a suitable alternative to synthetically man-made fibres, reinforcing a polymer matrix. Composite materials fabricated from natural fibres as the reinforcements of polymer matrices exhibit better mechanical, thermal and tribological properties that make them a suitable alternative to composites using synthetic-fibre-reinforced polymer matrices.

The most widely used matrix materials for high-strength and low-weight applications are polymer matrix materials. The addition of natural fiber into the matrix material overcomes the drawback of the low-strength matrix, due to which the fabricated composite exhibits superior mechanical properties. The processing of a polymer matrix does not require skilled manpower as it can be processed under low pressure and does not require high-temperature conditions. Thermosetting plastics and thermoplastics are the two primary types of polymer matrix. The thermoplastics are materials with low melting points, at which they can be recycled or remoulded with the application of heat, whereas thermosetting plastics cannot even be recycled under heat once they have been cured.

Tribology is a discipline of material science that is concerned with friction, wear and lubrication. The removal of a material from the surface of a body as a result of a relative motion between two separate surfaces is well-known as the wear. Friction is the force acting parallel to the surface and proportional to the normal force that resists the motion. The wear occurs as a result of friction. The effort made to reduce the friction and wear is referred to as lubrication. Several works were reported by researchers, categorizing the wear on the basis of physical mechanism (adhesion, fatigue or oxidation type of wear), physical appearance (scuffing and scar) or the wear condition (dry or lubricated sliding). Classification is needed to understand the wear mechanism, thereby controlling the design parameters, with which the wear life can be predicted. The present research suggests the possibility of using coconut inflorescence fibre as a potential fortification material for a tribological application. Coconut inflorescence is a potential fibre with reported maximum tensile and flexural strength of 72.14 MPa and 122 MPa. There is no research work on the tribological application of coconut inflorescence fibre by any researcher, which was the motivation for the present research.

2 EXPERIMENTAL PART

Composites were prepared with the reinforcing coconut inflorescence fibre and unsaturated polyester matrix. The inflorescence fibre acts as the load-carrying element and the matrix acts as the load-transfer element. In the present study, the following materials were used for the manufacturing of composite specimens: reinforcement material – coconut inflorescence fibre (CIF) and matrix material – unsaturated polyester resin.

2.1 Coconut inflorescence fibre

The southern part of India is famous for coconut tree, which is a prominent source of lignocellulose fibrils and several natural fibers can be extracted from its different parts, namely husk,[1] coir,[2] epicarp[3] or spathe.[4] In addition, another lignocellulose fibre was identified in the coconut tree, known as inflorescence. A spadix, which can also be a double sheath, encloses the inflorescence, present in each leaf axil. The inflorescence length may vary from 200 mm to 350 mm. Inflorescence is collected from the coconut tree and placed in water for at least ten days so that the main walls of the coconut inflorescence are allowed to soften due to retting. Consequently, water penetrates into the central stalk of the inflorescence, the

Figure 1: Inflorescence of a coconut tree

Figure 2: Extracted coconut-inflorescence fibres

inner cell walls get swollen enough, the outermost layer gets softened and a decay of the primary walls of the inflorescence takes place. Then the inflorescence is beaten with a mallet to remove the primary fleshy layers so that the fibres present inside the inflorescence are extracted.

The inflorescence (a yellow membrane) of a coconut tree is depicted in **Figure 1**. A single stack includes around 20–25 inflorescences. They are placed in water so that the primary walls of the inflorescences are softened and then, with the help of a mallet, the primary walls are broken and lignocellulose fibres are extracted. **Figure 2** shows the extracted fibres. These fibres are subjected to a surface treatment with a 5 % NaOH solution for 1 h.

2.2 Unsaturated polyester resin

Unsaturated polyester resin is a condensation polymer obtained as a result of a reaction among polyhydric alcohols. Typical polyhydric alcohols used for producing polyester resin are ethylene glycol, phthalic acid and isophthalic acids. The commercial availability and good mechanical properties were the reasons for selecting unsaturated polyester resin as the polymer matrix for this research work. The unsaturated polyester matrix was combined with cobalt naphthalene (catalyst) and methyl ethyl ketone (accelerator) with a ratio of 10 : 1 : 1 to cure the resin.

2.3 Characterization technique

The inflorescence fibres were subjected to FTIR and XRD to investigate the effect of surface treatment on the improvement of their physical and mechanical qualities.

2.3.1 FTIR spectrum analysis

The effect of alkalization on the functional groups present in the inflorescence fibres was investigated using Fourier transform infrared spectroscopy. The KBr pellet method was used for the preparation of test specimens for the FTIR analysis.[5] The analysis was done at a scan rate of 42 scans per minute with a 2 cm^{-1} scan resolution in a wave length of 500–4000 cm^{-1}.

2.3.2 XRD analysis

The crystallinity of inflorescence fibres was analysed using an X-Ray diffractometer at a 2θ scale of 10° to 60° for alkali treated and untreated inflorescence fibres. The inflorescence fibre was made into powder for the preparation of the test specimen using the KBr pellet technique. The crystallinity size and index were measured at a voltage of 45 kV and a current of 30 mA using K_α and Cu radiation.

2.3.3 Single-fibril tensile test

The tensile behaviour of inflorescence fiber was tested using an Instron 550R machine at a gauge length of 100 mm, cross-head speed of 100 mm/min, operating temperature of 25 °C and humidity of 65 %. The tensile test was performed for both untreated and alkalized inflorescence fibres.

2.4 Fabrication of the composite specimens for a tribology test

The wear and friction coefficient were studied with the aid of a tribology set-up under dry sliding condition. The specimens for the dry-sliding wear test were fabricated using cylindrical test tubes. The specimens were fabricated at a varied fibre weight percentage including (10, 15, 20, 25 and 30) *w*/% with unsaturated polyester resin as the base matrix. The surface of the coconut inflorescence fibres was exposed to alkali treatment with the 5 % NaOH solution before the integration with unsaturated polyester resin. The specimens were sized according to the ASTM G-99[6] standard, which specifies a diameter of 10 mm with a length of 35 mm.

2.5 Tribology study

A pin-on-disc wear-test set-up was used to investigate the tribological behaviour of the coconut fibre reinforced unsaturated polyester composites**.** The tribology tests were conducted under five different loads of (5, 10, 15, 20 and 25) N, where the volume fraction of the composites were varied between (10, 15, 20, 25 and 30) φ/% of fibre/polyester, respectively, for sliding distances of 1000 m and 2000 m.

2.6 Scanning-electron-microscopy analysis

Using a scanning electron microscope, the surface of untreated and alkalized coconut inflorescence fibre was investigated to determine the alkalization effect on the surface of the fibre. The worn surface morphology was also investigated using the same microscope to understand the possible causes of failure.

3 RESULTS AND DISCUSSION

3.1 FTIR analysis

FTIR spectroscopy exposed the elimination of amorphous substances present in the coconut inflorescence fibers. **Figure 3** shows the IR spectra of coconut inflorescence fibers.[7] The peaks and their corresponding functional-group elimination are listed in **Table 1**. The removal of hemicellulose, lignin, pectin and wax was revealed by the disappearance of the peaks between the untreated and mercerized coconut inflorescence fibres. As a result of the fibre-surface treatment, the fiber diameter decreased from 0.51 mm to 0.42 mm, thereby the fibre surface area was improved.

Table 1: IR absorption spectra of CIF

Position of bands (cm^{-1})	Assignment
3292.33	OH stretching vibration of hydroxyl band of cellulose I and II
2852.70	C-H symmetrical stretching
1737.21	stretching vibration of carboxyl group, carboxylic acid (RCOOH)
1549	C-C=C aromatic symmetric stretching
1238	C–O stretching in acetyl group

3.2 XRD analysis

The increase in the volume of crystallinity and index was found to be a result of the removal of the amorphous substance present in the fibres. The crystallinity index increased from 57.59 to 58.36 between the untreated and mercerized fibres, showing that mercerization has a prominent effect on the surface of CIF. **Table 2** shows the crystalline and amorphous peaks of the untreated and mercerized CIF. Due to mercerization, the formation of new crystal lattices occurred, a fibril rearrangement took place and the crystallinity of the fibre increased.[7] Thus, this is a suitable fortification material for polymer matrices.

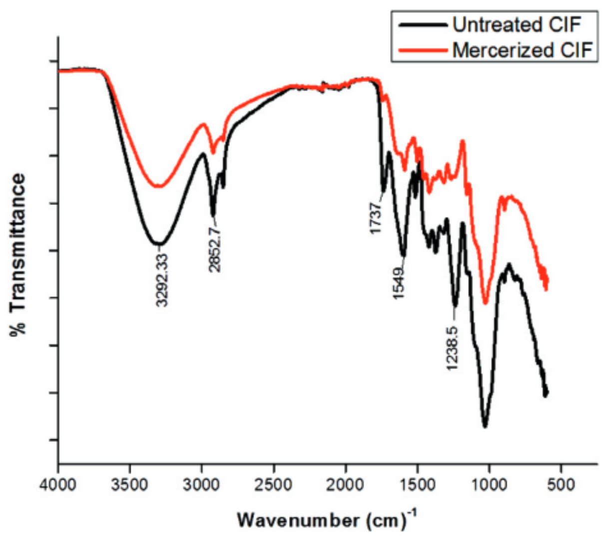

Figure 3: FTIR spectra for CIF[7]

Table 2: X-ray diffraction peaks for CIF

	2θ degree		Crystallinity (%)	Crystallinity index
	I_C	I_A		
Raw CIF	29.31	21.58	57.59	0.2637
Mercerized CIF	27.14	19.36	58.36	0.2861

3.3 Single fibre tensile test

As a result of mercerization, the tensile strength increased from 256 MPa to 331.2 MPa between the untreated and mercerized CIF. The tensile strength is determined by dividing the average load with the average area. The surface of the fibers becomes more uniform as a result of the elimination of micro-voids, hence the stress-transfer capacity between the fibre walls improves.[8] As a result, the breakdown of hydrophilic hydroxyl groups increases the moisture resistance of the fiber when increasing the mercerized-CIF tensile strength.

3.4 Influence of the load and sliding distance on the wear rate

The wear rate of the coconut inflorescence fibre reinforced polyester composite was studied under steady-state conditions. The sliding distance and load were the constraints employed for determining the wear rate of the coconut inflorescence fiber fortified unsaturated polyester composites. The wear rate of the composites was determined based on the weight loss of the composite samples. The variation in the specific wear rate of the composites with the increasing volume fraction under varying load conditions is shown in **Figure 4**. The load was changed from 5 N to 25 N to study the influence of the volume fraction and load on the wear behaviour of the composites. As the load increased, the wear rate started increasing with the increasing volume fraction of the composites. The composite with 10 $\varphi/\%$ exhibited a high wear rate under the 5 N load, which implies there were fewer fibres in the matrix; when the volume fraction increased, an incremental decrease in the wear was observed up to a certain level.[8] The composite with 25 $\varphi/\%$ exhibited a low wear rate under the 5 N load and when the volume fraction increased up to 30 $\varphi/\%$, the composite wear rate also increased.[9] However, the increase in the wear rate with the volume fraction of 30 % was due to improper wetting of the fibrils, resulting in

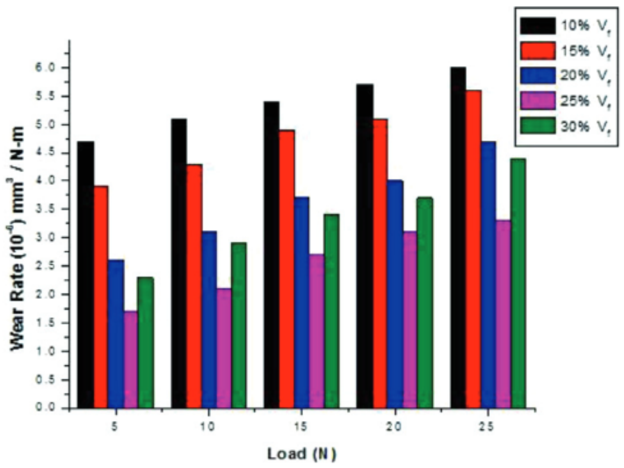

Figure 4: Load vs wear rate at the 1000 m sliding distance

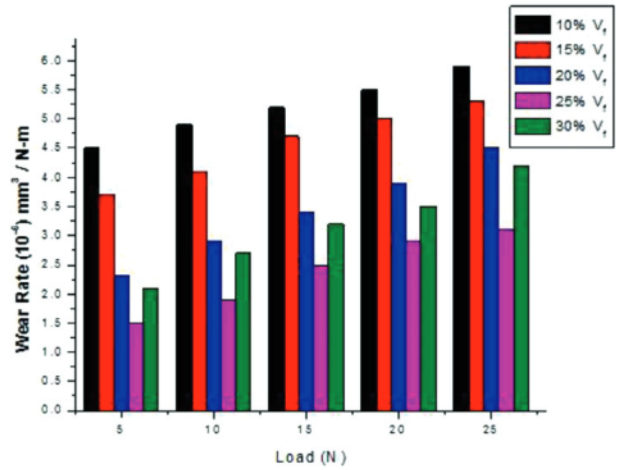

Figure 5: Load vs wear rate at 2000 m sliding distance

unfortunate bonding between the matrix and fiber.[10] Similarly, when the load increased from 5 N to 10 N, the wear rate increased up to 20 $\varphi/\%$ and then it decreased; when the fibre volume fraction was 30 %, the wear rate increased. In the entire investigation, the composites with fibre 25 $\varphi/\%$ exhibited a lower wear rate and the minimum wear rate was found to be 1.5×10^{-6} mm^3/(N·m). Ploughing and wedge structure may be the leading system for the minimum wear rate of the composites with 25 $\varphi/\%$.[11] The resistance to wear of the coconut inflorescence fiber reinforced unsaturated polyester composites was improved by adding fibers to the polyester matrix which resulted in composites with 25 $\varphi/\%$ exhibiting the minimum wear rate. The brittle nature of the unsaturated polyester matrix was transformed into a ductile nature with the addition of coconut inflorescence fibre so that the composite exhibited a higher shearing resistance due to a higher volume fraction. The presence of the fiber in the composite makes the composite exhibit a better wear resistance, thereby requiring more energy for the fibers to be detached from the matrix. The polyester matrix transfers the load to the inflorescence fibrils and then the fibrils withstand the load due to improved interfacial bonding with the matrix.

The sliding distance was taken into account in addition to the effect of the load on the wear rate of the coconut inflorescence fibre supplemented unsaturated polyester composite with the changing volume fractions. The sliding distance was altered from 1000 m to 2000 m in this case to investigate the effect of the sliding distance on the wear behaviour of the developed composites. The wear rate of the composites reduced with the increased sliding distance as shown in **Figure 5**. This is because at the 1000 m sliding distance, the rotating disc was in contact with the matrix, which had low modulus; at the sliding distance of 2000 m, the wear rate of the composite surface was decreased due to the resistance provided by the inflorescence fibers.[12] The hardness of the inflorescence fibres resulted in enhanced resistance during dry sliding so that higher energy was required for the failure of fibers. Therefore, the increment in the sliding distance contributed to the decrease in the wear rate. In both cases, that is the wear rate at 1000 m and 2000 m sliding distance, the minimal wear rate was observed for the composites with 25 $\varphi/\%$. The cause for the increase in the wear rate when the fibre volume fraction increased to 30 $\varphi/\%$ was improper wetting of the fibre as explained before, resulting in reduced bonding between the matrix and fibre.[9]

3.5 Influence of the load and sliding distance on the friction coefficient

The friction coefficient can be defined as a dimensionless number that relates the force caused by the normal force to the friction produced between two substances which are in relative motion. The value of friction coefficient is usually between 0 and 1. There is no friction between substances when the friction coefficient is 0; when the value of friction coefficient is 1, the frictional force is equivalent to the normal force. In this work, the friction coefficient of the coconut inflores-

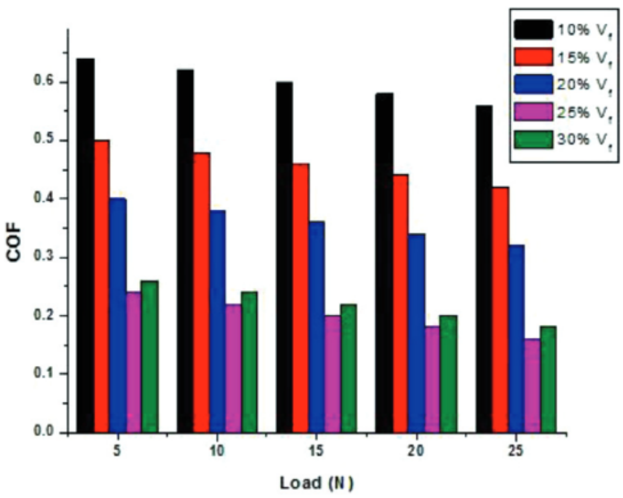

Figure 6: Load vs COF at 1000 m sliding distance

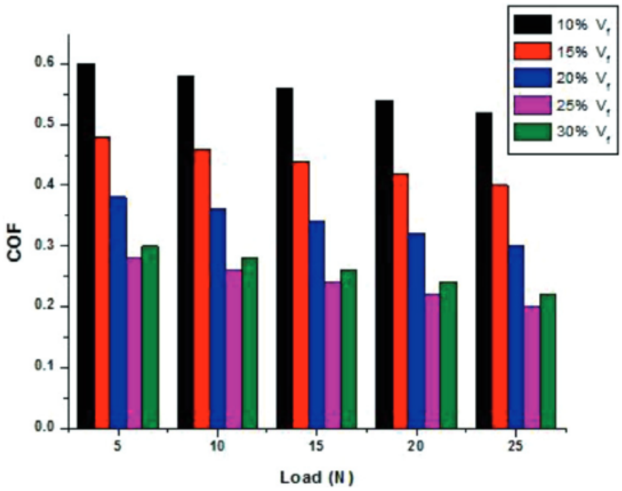

Figure 7: Load vs COF at 2000 m sliding distance

cence fiber reinforced polyester composite was a function of the fibre volume fraction, load and sliding distance.

The influence of the applied load at the varying volume fraction and 1000 m sliding distance on the friction coefficient is presented in **Figure 6**. From this figure, it can be inferred that the coefficient of friction was reduced with the increasing load for all volume fractions of the composites.[9] The increase in the load resulted in an increase in the temperature at the contact surfaces of the composite specimen and rotating disc, resulting in the heating of the disc. As a result, the coefficient of friction was decreased due to the micro-melting caused by frictional heat at higher loads. The mechanical interlocking of the asperities at the interface between the composite sample and the revolving disc is responsible for the higher frictional coefficient at lower loads.[12] The increase in the sliding distance further reduces the friction coefficient. It is evident from **Figure 7** that the coconut inflorescence fibre reinforcement of the polyester matrix reduced the friction coefficient. The film transfer onto the counter face was smooth enough, leading to reduced interlocking between the asperities on the contact surfaces; as a result, the friction coefficient was decreased when the sliding distance was increased.[13] Furthermore, the friction coefficient decreased when the volume fraction amounted to 25 % and increased when the volume of fiber fraction was increased to 30 %. This was due to the hardness of the composite specimens. Therefore, it can be understood that the 25 % volume fraction of the coconut inflorescence fibre reinforcing the polyester matrix accounted for the reduced friction coefficient, making it a potential composite for tribological applications.

3.6 Hardness of the composite specimens

Hardness is defined as the relative resistance provided by the surface of a composite to the indentation by an indenter at a particular load. From **Figure 8**, it can be inferred that the addition of coconut inflorescence fibre to the unsaturated polyester resin resulted in increased hardness with the increased volume fraction. It was discovered that the composite hardness increased significantly. The reinforcement of coconut inflorescence fiber of the unsaturated polyester resin resulted in increased hardness as a result of the fibre/matrix interaction; thereby, the brittleness of the matrix was reduced.[14] The composite with 30 $\varphi/\%$ exhibited a maximum Shore D hardness of 106 while the composites with 10 $\varphi/\%$ exhibited Shore D hardness of 96, which means that the addition of CIF resulted in an improvement of the hardness of the composites.

3.7 SEM analysis

The scanning electron microscopy (SEM) analysis performed for the composites with 30 $\varphi/\%$ revealed fibre pull-outs, indicating a reduced interfacial connection between the coconut inflorescence fiber and the polyester

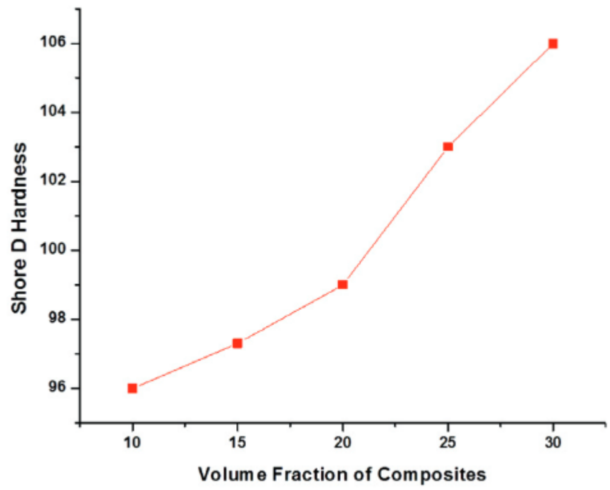

Figure 8: Shore D hardness of composite samples

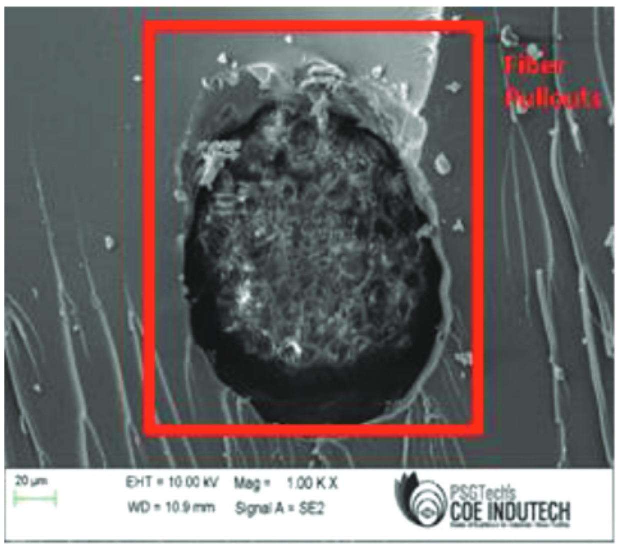

Figure 9: SEM image of a fibre pull-out in a worn specimen

matrix. The reason for poor interfacial bonding was improper wetting of the fibres during the manufacturing of the composites. **Figure 9** shows a fibre pull-out in the composite with 30 φ/%.

4 CONCLUSIONS

The research clearly shows that coconut inflorescence fiber can be a possible alternative to synthetic fibers used for reinforcing a polyester matrix, protecting it from the wear.

Extensive testing revealed that an increase in the load increases the wear rate, while an increase in the volume fraction and sliding distance decreases wear rate.

The minimum wear rate is found to be 1.4×10^{-6} $mm^3/(N \cdot m)$ for the composite with 25 φ/%. The hardness of the composite was also the influencing parameter for the decrease in the wear as the volume fraction and sliding distance increased.

The friction coefficient was also found to be minimum for the composite with 25 φ/%. The film transfer on the counter surface became sufficiently smooth, resulting in a reduced contact between the asperities, hence the friction coefficient decreased with the increase in the load and volume fraction.

The SEM analysis performed on the composites with 30 φ/% of the reinforcing fiber revealed poor interfacial bonding with the matrix due to improper inflorescence-fibril wetting.

Overall, it can be concluded that coconut inflorescence fiber can be a potential reinforcement material used with a polymer matrix to reduce its wear.

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