# Investigation on Tribological Properties of Horn Fibre Reinforced Epoxy Composites

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***Abstract*-- Environmental sustainability demands on the development of green composites for tribo-materials due to their low density, low cost and eco-friendly characteristics. The aim of this work is to investigate the tribological properties of composites fabricated using bio-waste horn fibre (HF) and epoxy resin. Composites were fabricated in accordance with Taguchi L9 (3x3) orthogonal array. Epoxy LY-556, hardener HY-951 mixed in the ratio of 10:1 was used as matrix and HF particles were used as bio-filler. The mixture was compression molded and cured at room temperature to produce the specimens. Properties like density, porosity, water absorption, oil absorption, hardness, coefficient of friction (COF), wear rate, surface roughness and microstructure of the specimens were investigated. Optimization of factors was done using grey relational analysis (GRA) and ANOVA. The optimum factor levels are found to be the lowest NaOH concentration ($T_1$ i.e., 0.1 N), the biggest sized particles ($S_3$ i.e., 425 µm) and the highest volume % ($V_3$ i.e., 30%). The contribution of influencing factors in decreasing order are found to be HF size with 83.7%, HF volume % with 11.3% and NaOH concentration with 1.47%. Comparison between predicted and experimental values of optimum specimen reveals that the variations are within 2.4%. Comparing the optimum specimen with untreated HF specimen, it is found that optimum specimen has lesser density and higher COF and hence can be used in frictional applications like brake pads and clutch discs.**

***Index Term*-- Horn fibre; epoxy resin; compression molding; tribological properties; optimization; grey relational analysis; ANOVA.**

## 1. INTRODUCTION

Polymeric composite materials are used in fields like automotive, marine, electrical, industrial, construction, house hold appliances and sporting goods due to their light weight, high strength, stiffness and corrosion resistance. Recently extensive research work has been carried out on the natural fibre reinforced composite materials due to their abundant availability and economically cheaper. Natural fibres like kenaf, hemp, flax, jute, banana, sisal, bamboo, etc. are obtained from plants and fibres like silk, feather, wool, etc. are obtained from animals. Horns are never branched and are found in the heads of oxen, buffalo, sheep, goat, etc. Horns are found in various shape, length and curvature. These are made of structural protein called keratin that is strong and rigid. Hard keratins have higher sulphur content and are classified into two groups, α-keratin found in mammals like hairs, horns, nails, etc. and β-keratin found in avian and reptilian tissues. Oxen horn is one of the bio-waste of the slaughter house. The central core of the horn turned into cylindrical form is used for the manufacture of useful items. Other portion of horn in the form of chips become landfill and causes environmental pollution. These chips can be used as reinforcing fibre for the manufacture of composite materials. Epoxies are cross linked thermosetting materials, which cannot be recycled. Araldite LY 556, is medium viscosity, unmodified liquid epoxy resin based on Bisphenol-A. They are stable to chemical attacks and are excellent adherents having slow shrinkage during curing and no emission of volatile gases. High bonding strength and room temperature curing makes it to be selected as matrix material. Aradur, the hardener used with the Araldite has the designation HY951. The mixing ratio between the resin and hardener is 100:10 parts by weight.

Septimus Sisson has briefed the structure of horn [1]. Marc Andre Meyers et al. have discussed the structure and mechanical properties of biological materials [2]. Tasneem Zahra Rizvi et al. have investigated the frequency and temperature dependence of dielectric constant and loss factor in cow horn keratin [3]. D. Kumar et al. have studied mechanical and thermal properties of horn fibre/PP composites and observed that 15% of horn fibre particles gave optimum results [4]. N. Venkateshwaran et al. have modified the fibre surface and found that 1% NaOH treated fibre reinforced composites behaves superior than other treated and untreated fibre composites and also found that high concentration of alkali damages the fibre and decreases the mechanical properties [5]. Shao-Yun Fu et al. have reviewed the effects of particle size, particle/matrix interface adhesion and particle loading on the stiffness, strength and toughness of particulate-polymer composites [6]. S. C. Mishra et al. have investigated the mechanical properties of alkali treated chicken feather reinforced epoxy composites and found that the interface bonding between the matrix and reinforcement is beneficial due to the formation of ester and amine groups [7]. Peter J. Blau has presented a survey of compositions, functions and testing of commercial friction brake materials and their additives [8]. Chittaranjan Deo et al. have studied on abrasive wear behavior of Lantana-Camara Fibre (LCF) reinforced epoxy composites and noticed that the optimum

wear reduction is obtained when the fibre content is 40 weight % [9]. C. Girisha et al. have investigated the effect of water absorption on mechanical properties of sisal and coconut coir reinforced epoxy composites and found that exposure to moisture caused a significant drop in the mechanical properties due to the degradation of the fibre-matrix interface [10]. O.S. Olokode et al. have studied the synergistic effects of friction materials with cow hooves dust and bagasse and noticed that friction material with 100 µm size gave better results than other sizes [11]. Mishra Antaryami have investigated the wear characteristics of teak wood dust filled epoxy composites and found that the composite with 10% wood dust may be more suitable for frictional applications [12]. Mingjiang Zhan et al. have fabricated and investigated the properties of composites with epoxy, chicken feather fibre (CFF) and E-glass fibres and found that the composites can be used as printed circuit boards (PCBs) [13]. S. C. Mishra have investigated the dielectric properties of chicken feather reinforced epoxy composites and found that the dielectric properties are dependent on operating frequency and temperature conditions [14]. V. Fiore et al. have studied on the effect of alkaline treatment on mechanical properties of kenaf fibre epoxy composites and found that the alkali treatment improves fibre-matrix interfacial adhesion, reducing the mobility of the polymer chains and enhancing stress transfer [15]. A. Shalwan et al. have studied the mechanical and tribological performance of the epoxy/date palm/graphite composites and revealed that 6% NaOH treatment of date palm enhanced the interfacial adhesion and addition of the graphite up to certain extent enhanced wear characteristics [16]. Govardhan Goud et al. have studied the effect of fibre content and alkali treatment on mechanical properties of Roystonea regia/epoxy composites and found that the alkali treatment found to be effective in improving the tensile and flexural properties while the impact strength decreased [17]. G. Sui et al. have studied cancun sand/epoxy composites and found that composite with addition of 1 weight % of sand particles showed the highest flexural properties. They have also found that high temperature storage modulus, glass transition temperature and dimensional stability of the composites increase with the addition of sand particles [18]. Mayowa Afolabi et al. have investigated PKS/cow bone particles/epoxy composite for brake pad and found that thermal, hardness and other properties were observed to be within the acceptable requirement for brake pad function [19]. A. J. Kinloch et al. have manufactured flax fibres and cellulose fibres/epoxy composites using resin infusion under flexible-tooling (RIFT) process and found significant improvements in physical and mechanical properties for the composites manufactured using the process [20].

The objective of this work is to develop a bio-waste HF reinforced epoxy composite. Surface treatment of natural fibres using NaOH (alkali) overcomes the difficulty of incompatibility between hydrophilic fibres and hydrophobic matrix. Optimum levels of parameters were obtained by fabricating the composites using Taguchi L9 (3x3) orthogonal array and optimizing using GRA and ANOVA. Apart from these nine compositions one more specimen was made with 20% untreated HF and 80% matrix. Physical, mechanical, tribological and micro-structural properties of these composites were characterized and compared with the properties of specimens made of untreated HF.

## 2. MATERIALS AND METHOD

### *2.1 Materials*

HF chips used as reinforcement were obtained from a button manufacturing industry near Ambur, Tamilnadu, India. Sodium Chloride Extrapure used to clean the HF particulates was obtained from S D Fine-Chem Limited, Mumbai, India. Diethyl Ethyl Ether (Ether Solvent) used to defat the HF was obtained from Nice Chemicals (P) Ltd., Kochi, India. Sodium Hydroxide flakes used to treat the HF surfaces was obtained from Qualikems Fine Chem Pvt. Ltd., Vadodara, India. Araldite LY 556 used as resin was obtained from Huntsman Advanced Materials India Private Limited, Mumbai, India. Aradur HY 951 used as hardener was obtained from Huntsman Advanced Materials India Private Limited, Mumbai, India.

### *2.2 HF processing*

Fig. 1. (a) shows the photograph of an ox horn. Fig. 1. (b) shows the photograph of horn chips thrown as landfill. HF chips were washed in water thoroughly, dried, ground into particulates using double bladed electrical grinder and sieved to 125 µm, 250 µm and 425 µm sieve grades using sieves. Fig. 1. (c) shows the photograph of horn fibre particles sieved to desired size. The HF particles were washed in 0.1 Normality (N) NaCl solution for 8 hours by changing the solution periodically after every two hours. It was washed in water, dried and immersed in diethyl ether for four hours to remove fat. It was washed in water thoroughly and dried. The defatted HF particles were treated with 0.1 N, 0.2 N and 0.3 N NaOH solutions for four hours, washed thoroughly in water and dried.

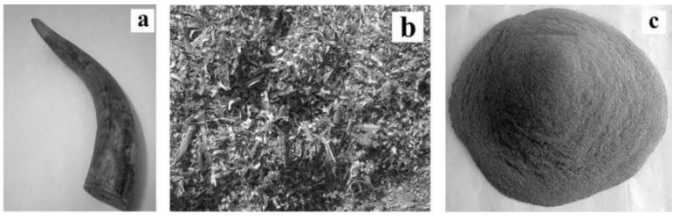


Fig. 1. Photograph of (a) an ox horn, (b) horn chips thrown as landfill and (c) particles of horn fibre sieved to a desired size.

Table I
Input factors and levels for Taguchi L9 orthogonal array.

| Factors | Level 1 | Level 2 | Level 3 | Units |
|---|---|---|---|---|
| NaOH con. | 0.1 | 0.2 | 0.3 | Normality (N) |
| HF size | 125 | 250 | 425 | µm |
| HF vol. % | 10 | 20 | 30 | % |

*2.3 Specimen preparation*

Fig. 2. (a) shows the photograph of hydraulic compression machine with dies fixed on its table and crosshead. Fig. 2. (b) shows the photograph of intermediate die with mold cavity and punch. Fig. 2. (c) shows the manufacturing procedure of specimens. Specimens of size 55 mm diameter and10 mm thickness were prepared following Taguchi L9 orthogonal array with three factors and three levels as shown in Table I and with untreated HF material. The details of various ingredients used were shown in Table II. Required amount of resin and hardener were weighed using electronic weighing balance of accuracy 0.001 g and mixed uniformly to get a homogeneous mixture of matrix. The surfaces and walls of a self-fabricated compression molding dies were applied with release agent for easy removal of the molded specimen. Required amount of HF is weighed and mixed with matrix thoroughly. The mixture was transferred to the die and pressed to a pressure of 0.5 MPa for 30 minutes. The specimens were post cured at room temperature for 24 hours in clamped condition. The specimens were allowed to sit in ambient condition for one week. Test specimens were prepared with water emery sheet of 150 grit size.

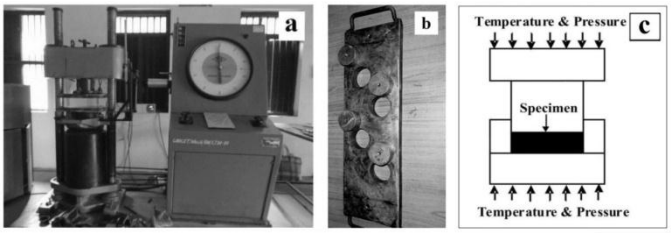


Fig. 2. (a) Photograph of hydraulic compression machine with dies fixed on its table and crosshead, (b) photograph of intermediate die with mold cavity and punch set and (c) manufacturing procedure of specimens.

Table II
Details of ingredients used.

| Trial ID | HF | | | Matrix | Total |
|---|---|---|---|---|---|
| | NaOH Con. (N) | Particle Size (µm) | Vol. % (%) | Vol. % (%) | Vol. % (%) |
| $T_1S_1V_1$ | 0.1 | 125 | 10 | 90 | 100 |
| $T_1S_2V_2$ | 0.1 | 250 | 20 | 80 | 100 |
| $T_1S_3V_3$ | 0.1 | 425 | 30 | 70 | 100 |
| $T_2S_1V_2$ | 0.2 | 125 | 20 | 80 | 100 |
| $T_2S_2V_3$ | 0.2 | 250 | 30 | 70 | 100 |
| $T_2S_3V_1$ | 0.2 | 425 | 10 | 90 | 100 |
| $T_3S_1V_3$ | 0.3 | 125 | 30 | 70 | 100 |
| $T_3S_2V_1$ | 0.3 | 250 | 10 | 90 | 100 |
| $T_3S_3V_2$ | 0.3 | 425 | 20 | 80 | 100 |
| $T_0S_2V_2$ | 0 | 250 | 20 | 80 | 100 |

## 3. SPECIMEN TESTING

Densities of the specimens were calculated by measuring the weights of the specimens in air and after immersing in 2-proponol of density 0.786 g/cc according to Archimedes principle using Mettler Toledo density measuring equipment (Model: XS 204, India) as per ASTM D792. Porosity % was determined using the weights of the specimens measured in dry condition and after immersion in water and boiled at 100°C for 8 hours. Water absorption % was determined using the weights of the specimens measured in dry condition and after immersion in water for 24 hours at room temperature, according to ASTM D570-99 standard. Oil absorption % was determined using the weights of the specimens measured in dry condition and after immersion in SAE 20/50 oil for 24 hours at room temperature, according to ASTM D570-99 standard. Specimens of size 55 mm diameter and 10 mm thickness were tested for Rockwell hardness L scale using Rockwell hardness tester (Model PRAS 4328). A steel ball indenter of ¼ inch diameter and 60 kgf force was used for the test. Rockwell Hardness number for L scale was noted directly from the dial. Wear test was conducted according to ASTM G99-05 standards using a Wear and Friction Tech computerized pin-on-disc apparatus with a spindle speed of 1450 rpm, track radius of 20 mm for a period of 27.5 min to a sliding distance of 5000 m at room temperature. The specimens of size 55 mm diameter, 10 mm thickness and 6 mm through hole at the centre were used as disc and a stainless steel pin of grade 304 with 6 mm diameter and 50 mm length was used as pin. Coefficient of friction was obtained by taking the average values of the test results using tribology data acquisition system. Wear rate was calculated using the sliding distance and the difference in weights measured before and after the test. Surface roughness on the track of wear tested specimens was measured using an inductance type surface roughness tester (Make: Metrix, Model: handySURF 10). The probe pin is made of diamond and is of 10 µm radius with 90° probe angle. Measurements were made with a probe force of 16 mN, with a cut-off length of 0.8 mm and driving speed of 0.5 mm/s. $R_a$ values were measured with an accuracy of four digits. A JEOL scanning electron microscope (Model: JSM 6390) was used for imaging the track of wear tested specimens. From the disc shaped wear tested specimen a specimen of size 10 mm x 10 mm was cut such that, complete wear track was covered. Prior to the observation, the specimens were sputter coated with Gold Palladium by electroplating process to eliminate electron charging. All specimens were examined using an accelerating voltage of 10 kV power supply. Fig. 3. (a), Fig. 3. (b) and Fig. 3. (c) shows the photographs of specimens under water absorption test, hardness tested specimen and wear tested specimen respectively.

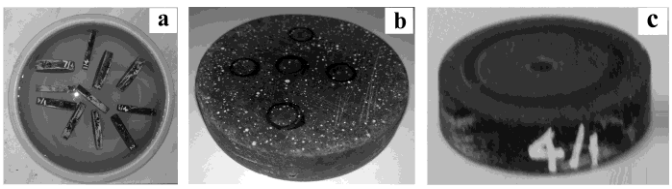


Fig. 3. Photograph of (a) specimens during water absorption test, (b) hardness tested specimen and (c) wear tested specimen.

## 4. GREY RELATIONAL ANALYSIS

In grey relational analysis, grey relational grades were obtained for analyzing the relational degree of the multiple responses. Experiments at different factor levels were conducted to obtain original response data. S/N ratios were calculated for the responses depending on the type of quality characteristics like smaller-the better and larger-the better. To avoid the effect of using different units and to reduce variability, normalized S/N ratios were calculated for the S/N ratios using appropriate formulae depending on the type of quality characteristics. Grey relational coefficients were computed for the normalized S/N ratios. Grey relational grades were determined to obtain the optimum factor levels and were used in ANOVA to find the contribution of influencing factors.

## 5. RESULTS AND DISCUSSION

### *5.1 Properties of pure horn*

Various tests to determine mechanical, thermal and surface morphological properties of horn fibre specimens were conducted. SEM with EDX test shows that the major element present in horn is Carbon with a maximum weight % of 59.03. Mechanical and thermal properties of pure horn specimens are listed in Table III. Fig. 4. (a) shows the SEM micrographs of the impact fractured surface of horn specimen. Fig. 4. (b) shows the energy dispersive X-ray spectrometry result of impact fractured surface of horn specimen with weight % and atomic % of elements present in it. Stress strain diagram of horn specimen is shown in Fig. 4. (c). From the values of elongation % at break and the stress strain diagram, it is found that horn is brittle, less dense and decomposes at higher temperature. As the properties of pure horn are better than polymers, reinforcing of HF particles with polymers will enhance the properties.

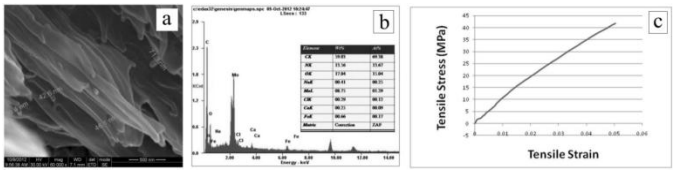


Fig. 4. (a) SEM micrographs of the impact fractured surface of horn taken at 60 k magnification and 500 nm scale, (b) energy dispersive X-ray spectrometry result of impact fractured surface of horn with weight % and atomic % of elements present in horn and (c) stress strain diagram of horn specimen.

Table III
Properties of pure horn.

| Ultimate Tensile Strength (MPa) | Tensile Modulus (MPa) | Elongation at Break (%) | Flexural Strength (MPa) | Flexural Modulus (MPa) | Impact Strength (J/m) | Compressive Strength (MPa) | Hardness (HRL) | Density (g/cc) | TGA (°C) | DTG (°C) |
|---|---|---|---|---|---|---|---|---|---|---|
| 41.86 | 806.33 | 5.04 | 122.9 | 4515.30 | 136.70 | 70.68 | 86 | 1.304 | 225 | 507 |

### *5.2 Properties of HF/epoxy composites*

Table IV shows the physical, mechanical and tribological properties (responses) of HF/epoxy composites. Table V shows the S/N ratios of physical, mechanical and tribological properties of HF/epoxy composites. Fig. 5. (a) to Fig. 5. (h) shows the main effect plots for S/N ratios of density, porosity, water absorption, oil absorption, hardness, coefficient of friction, wear rate and surface roughness respectively.

Table IV
Physical, mechanical and tribological properties (responses) of HF/epoxy composites.

| Properties | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Trial No. | Trial ID | Density (g/cc) | Porosity (%) | Water Absorption (%) | Oil Absorption (%) | Hardness (HRL) | COF | Wear Rate (g/m) | Surface Roughness $R_a$ (µm) |
| 1. | $T_1S_1V_1$ | 1.327 | 0.45 | 0.40 | 0.39 | 93.33 | 0.57 | 3.69E-05 | 0.531 |
| 2. | $T_1S_2V_2$ | 1.186 | 0.98 | 0.62 | 0.56 | 84.33 | 0.60 | 7.20E-05 | 0.956 |
| 3. | $T_1S_3V_3$ | 1.094 | 1.77 | 0.98 | 0.89 | 65.33 | 0.71 | 9.96E-05 | 1.862 |
| 4. | $T_2S_1V_2$ | 1.182 | 0.58 | 0.57 | 0.42 | 90.33 | 0.53 | 6.07E-05 | 0.559 |
| 5. | $T_2S_2V_3$ | 1.169 | 1.21 | 0.96 | 0.68 | 69.33 | 0.62 | 8.72E-05 | 1.065 |
| 6. | $T_2S_3V_1$ | 1.195 | 1.48 | 0.49 | 0.72 | 97.33 | 0.77 | 4.39E-05 | 1.254 |
| 7. | $T_3S_1V_3$ | 1.135 | 0.85 | 0.87 | 0.49 | 82.33 | 0.49 | 8.10E-05 | 0.621 |
| 8. | $T_3S_2V_1$ | 1.202 | 0.91 | 0.45 | 0.54 | 99.00 | 0.68 | 3.89E-05 | 0.726 |
| 9. | $T_3S_3V_2$ | 1.175 | 1.67 | 0.76 | 0.87 | 80.33 | 0.79 | 6.59E-05 | 1.475 |
| 10. | $T_0S_2V_2$ | 1.172 | 1.22 | 0.70 | 0.63 | 92.33 | 0.70 | 6.95E-05 | 0.980 |

Table V
S/N ratios of physical, mechanical and tribological properties of HF/epoxy composites.

| S/N ratios | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Trial No. | Trial ID | Density | Porosity | Water Absorption | Oil Absorption | Hardness | COF | Wear Rate | Surface Roughness $R_a$ |
| 1. | $T_1S_1V_1$ | -2.46 | 6.94 | 7.96 | 8.18 | 39.40 | -4.88 | 88.66 | 5.50 |
| 2. | $T_1S_2V_2$ | -1.48 | 0.18 | 4.15 | 5.04 | 38.52 | -4.44 | 82.85 | 0.39 |
| 3. | $T_1S_3V_3$ | -0.78 | -4.96 | 0.18 | 1.01 | 36.30 | -2.97 | 80.03 | -5.40 |
| 4. | $T_2S_1V_2$ | -1.45 | 4.73 | 4.88 | 7.54 | 39.12 | -5.51 | 84.34 | 5.05 |
| 5. | $T_2S_2V_3$ | -1.36 | -1.66 | 0.35 | 3.35 | 36.82 | -4.15 | 81.19 | -0.55 |
| 6. | $T_2S_3V_1$ | -1.55 | -3.41 | 6.20 | 2.85 | 39.76 | -2.27 | 87.15 | -1.97 |
| 7. | $T_3S_1V_3$ | -1.10 | 1.41 | 1.21 | 6.20 | 38.31 | -6.20 | 81.83 | 4.14 |
| 8. | $T_3S_2V_1$ | -1.60 | 0.82 | 6.94 | 5.35 | 39.91 | -3.35 | 88.20 | 2.78 |
| 9. | $T_3S_3V_2$ | -1.40 | -4.45 | 2.38 | 1.21 | 38.10 | -2.05 | 83.62 | -3.38 |

*5.2.1 Density*

From specific fuel consumption point of view low density composites are desired. Thus smaller-the better quality characteristic is used for density. From Fig. 5. (a), it is observed that increase in NaOH concentration decreases density, because NaOH treatment of HF particles makes the HF surface rough and porous. Increase in HF size also increases pores and thus decreases density. Density of HF is less than other ingredients and hence increase in HF volume % decreases density. From Fig. 5. (a), it is also observed that NaOH concentration and HF size do not influence greatly on density, whereas HF volume % influences better.

*5.2.2 Porosity*

In powder metallurgy process, porosity is one of the unavoidable characteristics. Increase in porosity decreases density but other properties are affected. High porosity generates more noise while braking. Thus smaller-the better quality characteristic is used for porosity. From Fig. 5. (b), it is observed that increase in NaOH concentration increases porosity, because NaOH treatment of HF particles makes the surface rough and porous. Increase in HF size increases interfacial area and hence increases porosity. As the sizes of all other ingredients are smaller than HF particles, increase in HF volume % increases porosity. From Fig. 5. (b), it is also observed that NaOH concentration does not influence greatly on porosity, whereas HF size influences better than HF volume %.

*5.2.3 Water absorption*

Water absorption is an undesired characteristic of composites, thus smaller-the better quality characteristic is used for water absorption. From Fig. 5. (c), it is observed that increase in NaOH concentration increases water absorption because NaOH concentration makes the HF surface rough and porous. Increase in HF size also increases pores and thus increases water absorption. Increase in HF volume % increases water absorption due to its hydrophilic nature. From Fig. 5. (c), it is also observed that NaOH concentration does not influence greatly on water absorption, whereas HF volume % influences better than HF size.

*5.2.4 Oil absorption*

Oil absorption reduces friction characteristics, thus smaller-the better quality characteristic is used for oil absorption. From Fig. 5. (d), it is observed that increase in NaOH concentration increases oil absorption because alkali treatment makes the HF surface rough and porous. Increase in HF size also increases pores and thus increases oil absorption. As the sizes of all other ingredients are smaller than HF particles, increase in HF volume % increases porosity and thus increases oil absorption. From Fig. 5. (d), it is also observed that NaOH concentration does not influence greatly on oil absorption, whereas HF size influences better than HF volume %.

*5.2.5 Hardness*

High hardness decreases wear rate, thus larger-the better quality characteristic is used for hardness. From Fig. 5. (e), it is observed that increase in NaOH concentration increases hardness because alkali treatment makes the HF surface hard and rough. Smaller the size of the HF particles, higher the surface area with the resin and hence high hardness is obtained when the size of the particles are the smallest. As the hardness of HF is less than other ingredients, increase in HF volume % decreases hardness. From Fig. 5. (e), it is also observed that NaOH concentration does not influence greatly on hardness, whereas HF volume % influences better than HF size.

*5.2.6 Coefficient of friction*

High coefficient of friction increases friction characteristics, thus larger-the better quality characteristic is used for coefficient of friction. From Fig. 5. (f), it is observed that increase in NaOH concentration increases coefficient of friction because alkali treatment makes the HF surfaces hard and rough. Increase in particle size increases surface roughness and thus increases coefficient of friction. Increase in HF volume % decreases coefficient of friction because HF is less abrasive, fills the space and makes the surface flat. HF

reacts with oxygen to help control interfacial films. This can be noticed from SEM micrographs of the wear tested specimens. From Fig. 5. (f), it is also observed that NaOH concentration does not influence greatly on coefficient of friction, whereas HF size influences better than HF volume %.

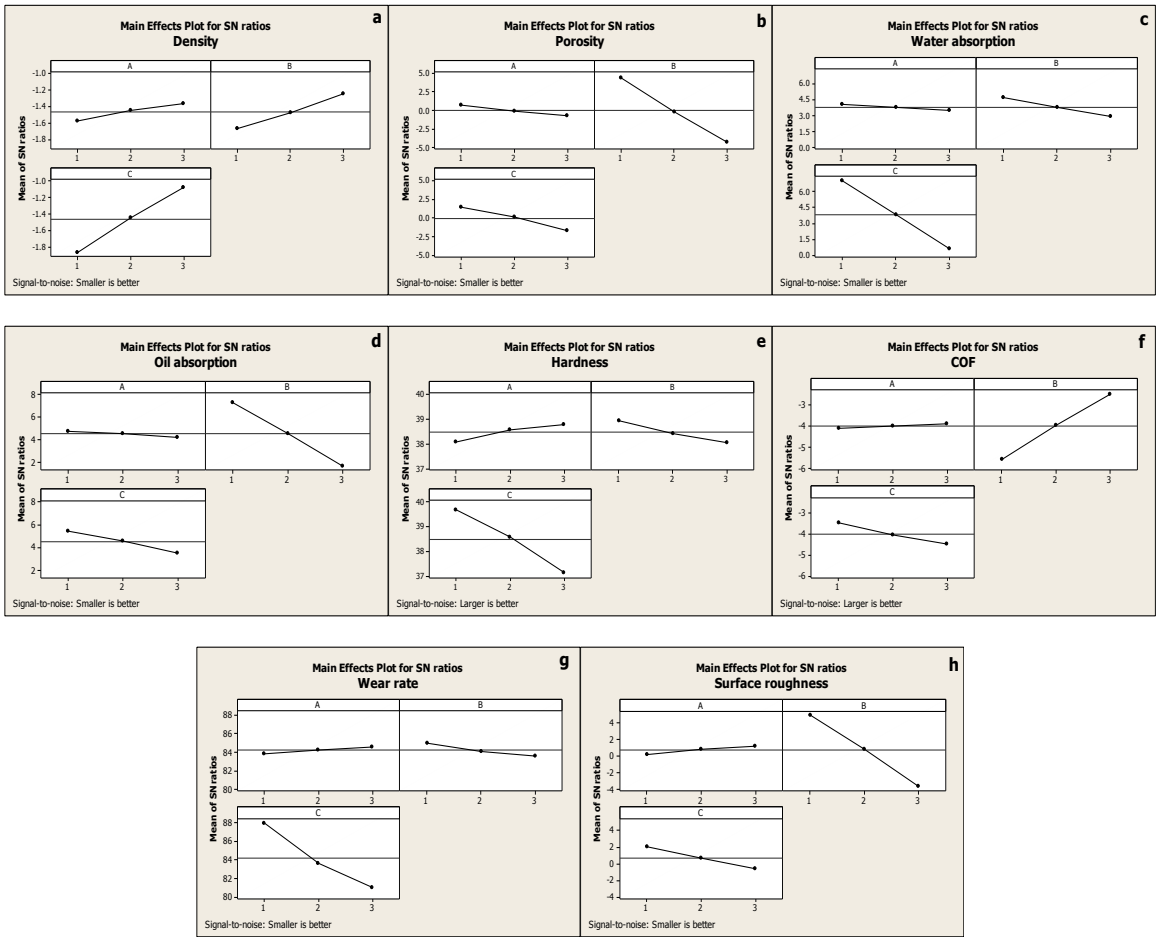


Fig. 5. Main effect plot for S/N ratios of (a) density, (b) porosity, (c) water absorption, (d) oil absorption, (e) hardness, (f) coefficient of friction, (g) wear rate and (h) surface roughness.

### *5.2.7 Wear rate*

High wear rate decreases friction characteristics, thus smaller-the better quality characteristic is used for wear rate. Wear rate increases with the increase in sliding speed, load, temperatures and particle size. From Fig. 5. (g), it is observed that increase in NaOH concentration decreases wear rate because alkali treatment makes the HF surface hard and rough. Increase in HF size increases interfacial area and decreases interfacial bonding and hence increases wear rate. When the HF volume % is increased clustering of HF takes place resulting in decreased bonding and thus increases wear rate. From Fig. 5. (g), it is also observed that NaOH concentration and HF size do not influence greatly on wear rate, whereas HF volume % influences better.

### *5.2.8 Surface roughness*

Surface roughness after the wear test is due to the micro-voids. The micro-voids are due to the removal of particles during wear test. Thus smaller-the better quality characteristic is used for surface roughness. From Fig. 5. (h), it is observed that increase in NaOH concentration increases bonding strength and hence reduces removal of particles and thus reduces surface roughness. Increase in HF particle size increases interfacial area and reduces bonding and increases surface roughness. Increase in HF volume % increases clustering of HF particles resulting in reduction of bonding strength and increase in particle removal and hence increases surface roughness. From Fig. 5. (h), it is also observed that NaOH concentration does not influence greatly on surface roughness, whereas and HF size influences better than HF volume %.

### *5.3 Scanning electron microscopy*

Fig. 6. shows the scanning electron microscopy images of wear tested surfaces of specimens taken at a magnification of 250 X, at a scale of 100 µm and at accelerating voltage of 5 kV. From Fig. 6. (c), Fig. 6. (e) and Fig. 6. (g), it can be noticed that when the HF volume % is high ($V_3$ i.e., 30%),

good compatibility between resin and HF particles occurs leading to lesser surface cracks. Fig. 6. (j) shows the SEM image of the specimen $T_0S_2V_2$, the specimen with untreated HF. Incompatibility can also be noticed on the untreated HF specimen $T_0S_2V_2$. Density and COF of the optimum specimen are better than untreated specimens which are the desired characteristics of friction.

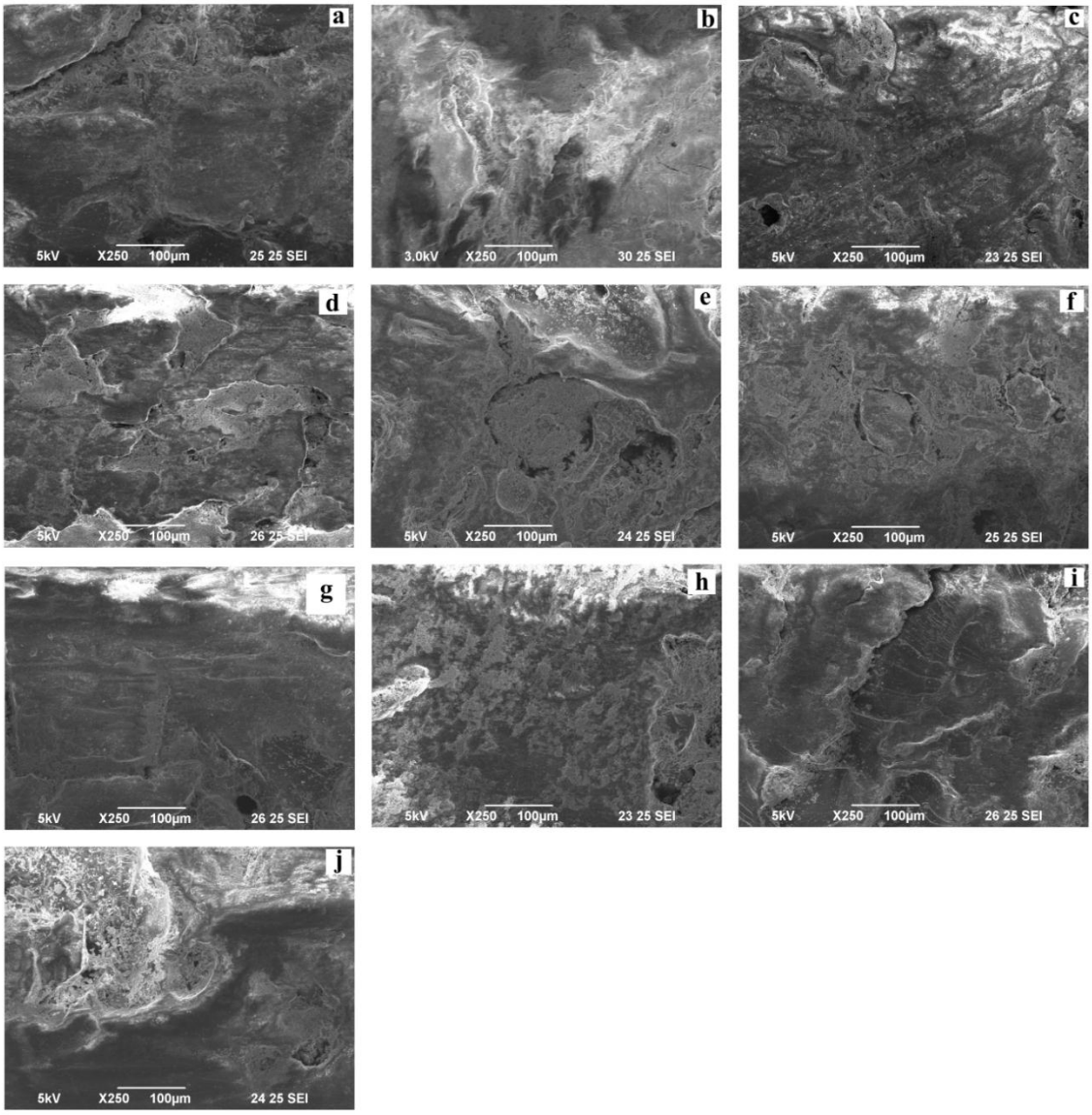


Fig. 6. SEM micrographs of worn out surfaces of specimens taken at a magnification of 250 X, at a scale of 100 µm and at an accelerating voltages of 5 kV (a) $T_1S_1V_1$, (b) $T_1S_2V_2$,(c) $T_1S_3V_3$, (d) $T_2S_1V_2$, (e) $T_2S_2V_3$, (f) $T_2S_3V_1$, (g) $T_3S_1V_3$, (h) $T_3S_2V_1$, (i) $T_3S_3V_2$ and (j) untreated HF specimen$T_0S_2V_2$.

Table VI
Mean of grey relational grades.

| Factors | Level 1 | Level 2 | Level 3 | Max-Min |
|---|---|---|---|---|
| NaOH con. | 0.60 | 0.56 | 0.58 | 0.03 |
| HF size | 0.48 | 0.55 | 0.72 | 0.24 |
| HF vol. % | 0.55 | 0.56 | 0.63 | 0.08 |

Table VI shows mean of grey relational grades. From Table VI it is found that the mean grades for the trial $T_1S_3V_3$ is found to be higher and is the optimum specimen. This is because influence of alkali treatment on particulate composites is less compared to continuous fibres. It is also noticed that NaOH treated composites have improved properties than the untreated composites because treatment makes the HF surfaces hard and rough and increases bonding strength. Therefore the lowest NaOH concentration ($T_1$ i.e., 0.1 N) is found to be optimum. Smaller sized particles are desired for mechanical properties, whereas larger sized particles are desired for coefficient of friction. Considering the entire properties, bigger sized particles ($S_3$ i.e., 425 µm) is

found to be optimum. Increase in fibre loading increases surface roughness and thus increases COF and thus when the HF volume % is the highest ($V_3$ i.e., 30%) good friction is obtained leading to optimum result. Density and COF of the optimum specimen are found to be higher than untreated specimen.

Table VII
ANOVA for the grey relational grades.

| Factors | DOF | Sum of Squares | Mean Square | F Value | % Contribution |
|---|---|---|---|---|---|
| NaOH con. | 2 | 0.002 | 0.001 | 0.42 | 1.47 |
| HF size | 2 | 0.093 | 0.047 | 23.70 | 83.70 |
| HF vol. % | 2 | 0.013 | 0.006 | 3.20 | 11.30 |
| Error | 2 | 0.004 | 0.002 | | 3.53 |
| Total | 8 | 0.111 | | | 100.00 |

Table VII shows the ANOVA table obtained for the grey relational grades used to find the significant factor and percentage contribution of influencing factors. From ANOVA table it is noticed that the major influencing factor is HF size with 83.7%, the moderate influencing factor is HF volume % with 11.3% and the least influencing factor is NaOH concentration with 1.47%. This is due to the fact that the fibre matrix interface plays an important role in deciding the properties than HF volume % and NaOH concentration. Fig. 7. shows the graphs of main effects plot for grey relational grades. From Fig. 7. it can also be noticed that $T_1S_3V_3$ is optimum specimen. Predicted values for each response of optimum specimen were calculated and compared with experimental values. Percentage variations between predicted and experimental values were found to be within 2.4%. Table VIII shows the predicted, experimental values and their % of variation for the optimum specimen $T_1S_3V_3$.

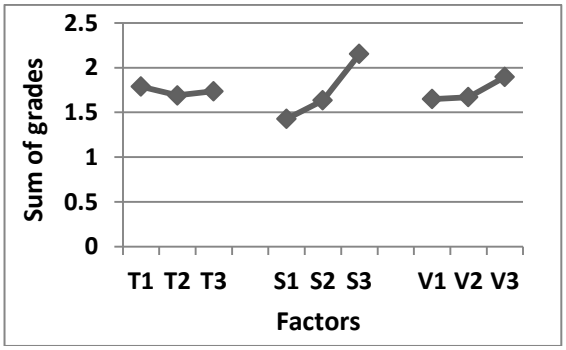


Fig. 7. Graphs of main effects plot for grey relational grades.

Table VIII
Predicted, Experimental values and their % variation for the optimum specimen $T_1S_3V_3$.

| Parameters | Density (g/cc) | Porosity (%) | Water Absorption (%) | Oil Absorption (%) | Hardness (HRL) | COF | Wear Rate (g/m) | Surface Roughness $R_a$ (µm) |
|---|---|---|---|---|---|---|---|---|
| Predicted values | 1.12 | 1.78 | 0.99 | 0.891 | 65.07 | 0.706 | 9.83E-05 | 1.818 |
| Experimental values | 1.09 | 1.77 | 0.98 | 0.89 | 65.33 | 0.7059 | 9.96E-05 | 1.862 |
| Variation % | 2.29 | 0.75 | 1.12 | 0.12 | 0.40 | 0.04 | 1.30 | 2.40 |

## 6. CONCLUSION

A new functional composite has been developed using particles of bio-waste HF and epoxy resin. Composite specimens were produced successfully following Taguchi L9 orthogonal array with three factors and three levels and with untreated HF using compression molding technique.

Physical, mechanical, tribological and morphological properties like density, porosity, water absorption, oil absorption, hardness, COF, wear rate, surface roughness and microstructure of the specimens were investigated.

Optimization of various factors was done using grey relational analysis, ANOVA and the results were compared with the untreated HF specimens. From grey relational analysis, the optimum factor levels are found to be 0.1 N NaOH concentration ($T_1$), 425 µm sized HF particles ($S_3$) and 30% of HF volume ($V_3$). From ANOVA it is noticed that the major influencing factor is HF size with 83.7%, the moderate influencing factor is HF volume % with 11.3% and the least influencing factor is NaOH concentration with 1.47% and error % is 3.53%.

From SEM images of the worn out surfaces of specimens, it can be noticed that when the HF volume % is high ($V_3$ i.e., 30%), good compatibility between resin and HF particles occurs leading to lesser surface cracks and incompatibility can be noticed on the untreated HF specimen $T_0S_2V_2$. Density and COF of the optimum specimen are better than untreated specimens which are the desired characteristics of friction.

Comparing the predicted values and experimental values of optimum specimen, % variation is found to be within 2.4%. As the properties of optimum specimen are better than the untreated specimen, optimum specimen can be used in frictional applications like brake pads and clutches.

As the fibre is derived from bio-waste, this work is innovative. Use of bio-waste fibre for composite manufacturing reduces environmental pollution and is also cost effective.

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