Effect of Pretreatment on the Mechanical Properties of Orange Peel Particulate (Bio-Waste)-Reinforced Epoxy Composites

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ABSTRACT

Effects of chemical treatments with benzoyl chloride, acetone, and alkali on the physical and mechanical properties of orange peel particulate (OPP) reinforced epoxy composite materials have been studied. Hand lay-up technique was applied to manufacture the composites. The experimental results illustrate that chemical treatment with benzoyl chloride has a considerable impact. The properties of OPP-reinforced composite material have been enhanced by 15% (for tensile test) and 30% (in case of flexural test) due to benzoyl chloride treatment as compared to raw OPP composites. It is evident from Fourier transform infrared spectroscopy (FTIR) that non-cellulosic content was removed from the surface of the fiber due to benzoyl chloride treatment. After chemical treatment, there was good interfacial bonding between matrix and filler material as observed in SEM micrographs. From the experimental observations, it can be seen that among all fabricated composites, set of composites with 30% filler loading yields excellent mechanical properties.

KEYWORDS

Chemical Treatment, Epoxy Composite, Mechanical Properties, Orange Peel Particulate, Thermal Stability

1. INTRODUCTION

Nowadays the use of synthetic fiber reinforced composites and petroleum-based polymers is limited due to their non-bio degradable and non-recyclable nature. The use of synthetic fiber for fabrication of different material leads to a threat to the environment across the world. Hence, recent research focus is targeted towards development of green products causing little or no damage to the environment. Accordingly, consideration of a new fiber (natural) and fabrication of new type composite material is the need of hour (Ball et al., 2019). Bio-based fibers have some plus characteristics over synthetic fibers such as abundant availability, low cost, eco-friendly, recyclable, light in weight, high specific strength, and high stiffness etc. They are non-abrasive and hypoallergenic in nature and also, possess ease of manufacturing/processing (Sreenivasan et al., 2012; Sreekumar et al., 2009; Zaman et al., 2010; Herrera-Franco & Valadez-Gonzalez, 2004). The plants, animals and minerals are the source

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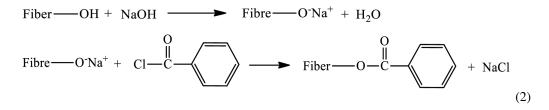
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of natural fibers. The natural fibers derived from plants generally have high cellulose content and they are widely used for the manufacture of green composite materials. Literature has explored the ability of different bio fibers as reinforcement in composite industries such as Jute (Ashraf et al, 2019; Burrola-Núñez et al., 2019), Sisal (Nagamadhu et al. 2019; Megiatto Jr et al., 2007), Kenaf (Hamidon et al., 2019), Banana (Senthilkumar et al., 2018), Bamboo (Zubaidah et al., 2018), Hemp (Sepe et al., 2018; Sunny et al., 2020), Coir (Adenivi et al., 2019), Flax (gassan et al., 1999; George & Verpoest, 1999), Aloe Vera (Chaitanya & Singh, 2016; 2018) and pine apple leaf fiber (Todkar & Patil, 2019).

However, the compatibility between natural fiber and matrix is an issue, hence, the natural fibers have limited use in the polymer industries. The properties of bio composite materials are dominated by interfacial interaction between matrix and lingo-cellulosic fibers. The existence of hemicelluloses, lignin and other materials is the reason for weak intermolecular adhesion between matrix and reinforcing material (Maldas & Kokta, 1994; Zhou et al., 2016). These issues of the natural fibers can be remedied by surface modification or through carbonization process. The surface modification may be physical, chemical or biological (Valadez-Gonzalez et al., 1999). One possible way to change the surface of the natural fiber is to reduce the nature of hydrophilicity due to chemical treatments and chemical modification. The chemicals used to treat fiber play an important role in enhancing the ability to strengthen the natural fiber. The interfacial bonds between matrix and filler materials can be increased due to coupling agents. The coupling agents are also responsible for reduction of moisture of the natural fibers. Several chemical treatment approaches are adopted for surface treatment of natural fibers such as alkaline treatment, benzoyl chloride treatment, permanganate treatment, acetylation treatment, saline treatment and peroxide treatment, etc. (Chandrasekar et al., 2017; Fiore et al., 2015; Majid et al., 2016; Kalia et al., 2019; Li et al., 2007; Kaushik et al, 2012; Liu et al., 2019). The alkaline treatment of natural fiber is carried out by using aqueous sodium hydroxide (NaOH) particularly in the case of thermosetting and thermoplastic composites and corresponding chemical reaction is given in Eq. (1). This treatment enhances the surface irregularity resulting improved mechanical anchoring leading to improved matrix fiber interpenetration (Nair et al., 1996).

$$Natural \ Fiber - OH + NaOH \rightarrow Natural \ Fiber - O^{-}Na^{+} + H_{2}O + impurities$$
(1)

Due to benzoyl chloride modification, there is a possibility of reduction of the hydrophilic nature of bio fiber and also, it enhances the interaction with the hydrophobic polymer matrix. It also increases interfacial adhesion between matrix and filler material which improves the strength of composites, reduces moisture absorption behavior and increases thermal stability (Kalia et al., 2011; Babu, 2019). The reaction between benzoyl chloride and natural fiber hydroxyl group is represented in Eq. (2).



Nonpolar organic component dissolves easily in acetone because of its non polar solvent properties. When the natural material dissolves in acetone, the lingo-cellulosic content also dissolves in acetone during acetone treatment.

The molecular weight of epoxy resins which is used as a matrix material is relatively low and these pre polymers have the capacity to process under diversity of conditions. The epoxy resin can be cured partially and its low shrinkage properties are noticed during cure and can be stored in the same state. At the same time, the viscosity and the cost of the epoxy resin are higher as compared to polyester resins. It shows unparalleled physical properties such as good moisture resistance, outstanding adhesion, mechanical properties such as high tensile strength and modulus, high thermal stability, excellent glass transition temperature and good electrical properties (De'Nève & Shanahan, 1993). Epoxy resins are generally used for protective coatings, engineering or structural adhesives in casing, construction, molding and composite applications (Jin et al., 2015). Hence, it is very much useful in manufacturing industry for commercial purposes.

Several literatures are available that consider the effects of chemical modification on mechanical properties of different natural composites. However, to the best of authors' knowledge, no study on the effect of chemical treatment of OPP composites and variation of its mechanical properties is available. Hence, in this experimental work, an attempt is made to study the feasibility of using OPP (a bio waste material) as reinforcement in epoxy-based composites fabricated using hand lay-up technique. The mechanical behavior such as tensile, impact, flexural and hardness of OPP composites have been compared with pure epoxy composites. The effect of different chemical modification/treatments such as acetone, benzoyl chloride and alkaline treatment of OPP prior to be used as reinforcement in composite has been investigated experimentally in terms of variation in physical and mechanical properties of fabricated composites.

2. EXPERIMENTAL DETAILS

2.1. Materials

Epoxy resin (Araldite LY–556) that belongs to epoxide family is used in the present study. It comprised of three-member ring structure, which contains two carbon atoms and an oxygen atom. The first product from the liquid reaction of bisphenol–A, is epoxy resin and its chemical name is Diglycidyl–Ether of Bisphenol–A (DGEBA). In comparison to other resins (thermo set or thermoplastic), epoxy resins are extensively applied in industrial sectors due to its useful properties such as low shrinkage during cure, good moisture resistance, excellent chemical resistance, good electrical and mechanical properties (Reis et al., 2015). The density and viscosity of epoxy resin are 1.2 g/cm^3 and $(11-14) \times 10^3$ MPa at ambient temperature respectively. The hardener HY-951 chemically known as 2–amineethylethane–1, 2–diamin is used to cure epoxy resin which was supplied by Ciba-Geigy India Ltd. The hardener is used to create strong cross linked molecular structures that possess good strength, toughness and virtual impermeability in the cured film.

Orange is a species of citrus tree grown around the world for their fruit. It belongs to the Rutaceae family. North eastern India, southern China and Indonesia are the countries where oranges are cultivated extensively. This is most widely consumed fruit after banana and mango in India. Consumption of orange is also very high in all over India. The outer surface of an orange is known as orange peels (OP). Previously, these were mostly used for land filling, animal feeding and composting. Later on, researchers realized the importance of the biological value of this waste and tried to make use of its medicinal properties such as for glowing skin, unclogging skin pores, treating pimples and acne, reducing inflammation etc. The chemical composition of OP particulate is shown in Table 1. The content of OP particulates was determined by proximate analysis and is shown in Table 2. These also have the ability to act as reinforcing material in the fabrication of polymer composite materials. The OP particulate is formed after putting it in the sun for two to four days to remove the moisture content from the OP and then these were grinded to the powder form as shown in **Fig. 1**. Sieve analysis was carried out to choose a particle size of $212+\mu$ m particle size.

2.2. Fabrication of Bio-Composite

The wooden mold of size 155 mm×65mm×6mm was used to fabricate the composite plate. The composite plates were fabricated with epoxy and OP particulate as matrix and reinforcement,

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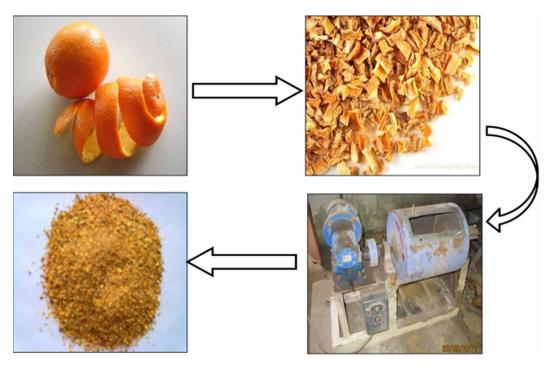
Table 1. Chemical composition of OP particulate

cellulose	38.61±0.5
Hemi cellulose	27.10±0.2
Lignin	30.10±0.3
Ash content	1.16±0.2

Table 2. Proximate analysis of OP particulates

Compositions	OPP
Fixed carbon	17.21
Moisture	8.8
Ash	0.89
Volatile	73.1

Figure 1. Procedure for OP particulate preparation



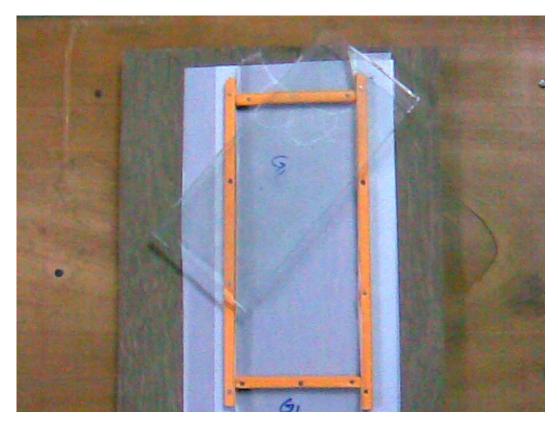
respectively. During fabrication of composite plates, the weight percentage of filler material was varied from 0 to 40%. The weight percentage (wt %) of the reinforcement material (reinforced) is estimated by using Eq. (3). Different wt % of the fabricated OPP composite is represented in Table 3.

Weight % of reinforcement = $\frac{\text{Weight of reinforcement}}{\text{Weight of reinforcement} + \text{Weight of epoxy}} \times 100$ (3)

Categories	Nature of material	O particulate composite	w filler material (%)
Group 1	Untreated	SO	(pure epoxy)
		S1	10
		S2	20
		\$3	30
		S4	40
Group 2	Alkali treated	\$3	30
	Benzoyl treated	\$3	30
	Acetone treated	\$3	30

Table 3. Types of different weight percentage of OPP reinforced epoxy composite

Figure 2. Wooden mold for fabrication of composite



In this study, hand lay-up technique was incorporated to fabricate the composite plates. The mold used for the purpose of fabrication is shown in Fig. 2. In the bed of wooden pattern, a pattern removal sheet was positioned and also pattern discharge spray was used on the inner side of pattern for speedy and effortless removal of fabricated sample.

The epoxy and hardener in the proportion of 10:1 by weight were mixed meticulously in a container at ambient conditions to manufacture composite specimens of different weight percentage. The mixture was then poured into the prepared mold. For elimination of air bubbles and uniform distribution of the mixture of the pattern a roller was rolled for two to five minutes. After that, the seamless pattern sheet was placed on the top of the pattern. A wooden plate of desired size was kept in the mold and loaded with dead weights before the reaction started and mixture hardened. The pattern was kept under observation for 48 h. During the experiment, care was taken to get uniform thickness because due to application of load, some mixture may be squeezed out from the pattern. After specific observation time, the specimens were taken out from the mold. The composite samples were cut for different tests as per ASTM standard. After that, the samples were stored in an airtight container to avoid contact with moisture.

2.3. Surface Treatment of OP Particulates

Surface treatment of the natural materials is a procedure to permanently change the behavior of fiber cell walls by inserting the polymers on the fiber surface. Although several chemical treatment methods are available, the present study considered Alkaline treatment, Benzoyl chloride treatment and Acetone treatment of OPP material for a comparative analysis.

a. Alkaline Treatment

50gm of OP powders were soaked in 5% sodium hydroxide solution for six hours in alkali treatment. During the surface modification, the ratio of weight of fiber to the sodium hydroxide solution was maintained as 15:1. Prior to the chemical modification, the OPP were washed meticulously with fresh water to eliminate the impurities that may stick to the material surface. The materials were kept in sun light for drying for four to five days to remove the moisture content. Then, the materials were soaked in alkali solution followed by cleaning with distilled water. The treated material was then rinsed with hydrochloric acid (HCl) to neutralize the alkali. The treated materials were rinsed with distilled water to maintain pH 7. The treated materials were then dried at atmospheric condition for 72 hour, followed by drying in oven at 110°C for eight hour.

b. Benzoyl Chloride Treatment

OPP (100 gm) were suspended for one hour in 10% sodium hydroxide solution (NaOH) to activate OH (hydroxyl groups) of cellulose and lignin. After that it was stirred continuously with benzoyl chloride solution for half an hour. To remove the excess amount of benzoyl chloride, the treated materials were soaked in ethanol for one hour. Finally, the treated specimens were thoroughly cleaned with distilled water and dried for six hour at 100°C in an oven.

c. Acetone Treatment

Soxhlet extractor was used to rinse the OPP powder with acetone. The time period for washing was two to three hours. All the materials were washed properly with distilled water prior to acetone treatment. The transparent color of acetone changed to light green during chemical treatment due to the existence of waxes and organic materials in bio fibers.

2.4. Characterization Techniques

a. Fourier Transform Infrared Spectroscopy

The functional group of raw and surface modified material can be found out by FTIR spectroscopy analysis. The OPP was mixed with KBr at a proportion of 1:200 by weight and pressed to prepare pallets for spectroscopic analysis. The IR-Prestige-21 spectrometer was used for FTIR analysis. During this investigation, the used resolution and scan rate were 0.5 cm⁻¹ and 40, respectively. The wave number range is 400 to 4000 cm⁻¹.

b. X-ray Diffraction Technique (XRD)

XRD is used to study the crystallographic structure (to distinguish between crystalline and amorphous material) of raw and chemically treated natural filler materials. The investigation was done by using multi-purpose X-ray diffraction, Regaku Ultima IV with the intensity CuK α , the radiation wavelength (λ) 0.154 nm and a graphite monochromator. The graphite monochromator in the range (2 θ angle range) of 5 to 45° with current and voltage are 40 mA and 40 mV, respectively was used.

c. Thermo Gravimetric Analysis (TGA)

The rising interest of using bio waste material as reinforcement in the preparation of polymeric materials has encouraged researchers to study its thermal behavior. Thermal analysis can be considered as an effective test for chemical, physical and structural assessments in a substance due to change in temperature. Basically temperature is a fundamental condition variable that affects most chemical reactions, physical properties and structural changes. The apparatus STA449C/4/MFC/G supplied by Netzsch, Germany was used for TGA analysis of both treated and untreated OP powders. The TGA of bio fiber is greatly influenced by different TGA atmosphere like inert (He and N₂) and oxidative (air and oxygen). The analysis was done at a range of 30 to 600°C with the heating rate of 10°C/min under N₂ atmosphere. The thermal stability of fabricated composite samples reinforced with natural fiber absolutely relies upon its chemical constituents which are thermally degraded at the temperature range 160° to 900° C. The thermal degradation of lignin, hemicelluloses and cellulose of natural fiber occurs at 160–900°C, 220–315°C and 315–400°C, respectively.

d. Scanning electron Microscopy (SEM)

The surface morphology of the tested specimens was investigated by using Nova NANO SEM 450 device. Fractured specimens with silver paste were stacked on the stubs. Before capturing photomicrographs, a platinum thin film is vacuum evaporated on to them to boost the conductivity of the samples.

2.5. Mechanical Behavior of Fabricated Specimens

The mechanical properties of raw and chemically treated OPP reinforced composite specimens were determined as per ASTM standard.

a. Density and void fraction

The theoretical and actual (experimental) density of the fabricated specimens determined using weight fraction and Archimedes principle are presented in Eqs. (4-6).

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$$\rho_{ct} = \frac{1}{\frac{w_f}{\rho_f} + \frac{w_m}{\rho_m}} \tag{4}$$

$$S_m = \frac{w_o}{w_o + \left(w_a - w_b\right)} \tag{5}$$

 $\rho = S_m \times \rho$ of kerosene

The voids (V_v) in terms volume fraction in the developed specimen were evaluated by using Eq. (7). The presence of voids during making of samples has a considerable effect on the mechanical properties and efficiency during use.

(6)

$$V_v = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \tag{7}$$

"w" and " ρ " are the weight fraction and density of the specimen respectively. The suffix ct and ce is used for theoretical and actual density of the specimen. The suffix "f" and "m" represent the fiber and matrix, respectively. " S_m " is the specific gravity, " w_o " is the weight of the sample; the weight of the bottle and kerosene and bottle, kerosene and sample are represented by w_a and w_b , respectively.

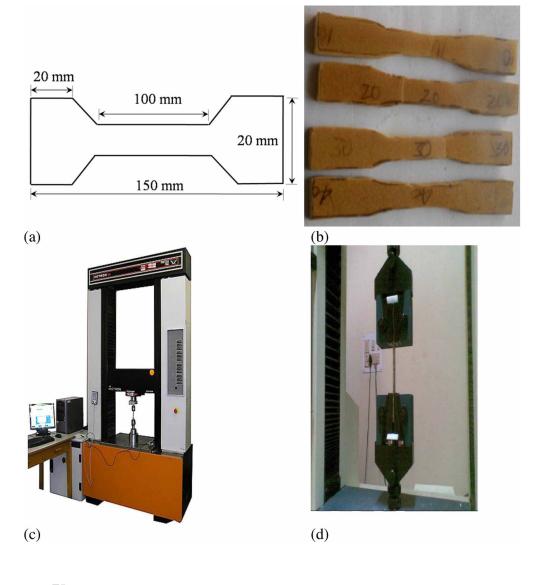
b. Tensile Strength

ASTM D 3039-76 standard was used to carry out the tensile test (tensile strength and modulus) of designed dog-bone type specimen with span length 42 mm in a Universal Testing Machine (UTM) (INSTRON H10KS). To calculate tensile modulus efficiently, an extensometer of 10kN load cell was used to measure strain at a rate of cross head speed 2 mm/min. From an accuracy point of view, five specimens for each test were measured and the average value was considered for further estimation. Fig. 3(a) and (b) present the schematic diagram of the tensile test sample and some of fabricated tensile test specimens, respectively.

c. Flexural Strength

Flexural properties such as flexural strength and modulus of the fabricated composite samples were evaluated using 3-point bend test technique by fixing the specimen (140 mm×15 mm×4 mm) under flexural loading. ASTM D790–03 standard was used to determine the flexural properties with constant span to depth ratio 16:1 and prescribed cross-head speed and load cell. Five samples were tested from each set for accuracy. Flexural test specimen and configuration of the sample is represented in Fig. 4. Flexural strength of the different specimens can be calculated using the Eq. (8).





$$\sigma = \frac{3FL}{2bt^2} \tag{8}$$

where F is breaking load (N), L, b and t are the distance between two supports, width and thickness, respectively of the specimen.

d. Impact Test

Izod impact tester was used to conduct an impact test for the development composite specimens as per ASTM D 256 standard, as shown in Fig. 5. The specimen required for this test is a V notched

 $(45^{\circ} \text{ and depth } 2.54 \text{ mm})$ rectangular bar with dimension $63.5 \text{mm} \times 12.7 \text{ mm}$. The hammer was released from an angle 150° with energy range 5.394 J for this experiment.

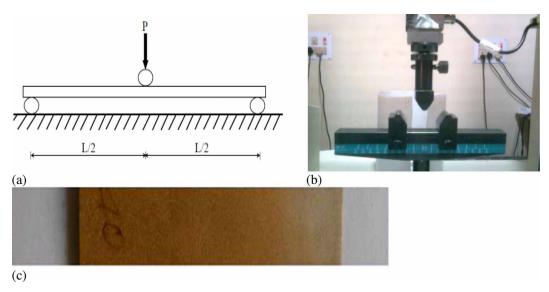
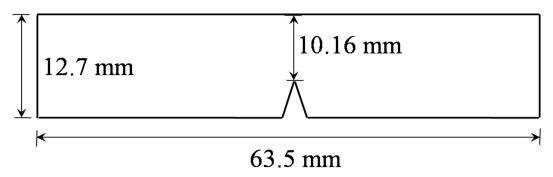


Figure 4. (a) Schematic diagram of arrangement of specimen for flexural testing, (b) Fixing the specimen in 3-point bend test technique and (c) Actual specimen for flexural testing.

Figure 5. Specimen for the impact test.



e. Micro-Hardness

The hardness of composites was evaluated by Lecco Vickers Hardness (LV 700) tester. A square base right pyramid diamond indenter was forced into the substance under load. The angle between the diamond indenter's opposite faces is 136°. Eq. (9) is used to calculate the Vickers hardness number of developed composites.

$$H_v = \frac{0.1889F}{D^2} \tag{9}$$

where F is the applied load to the specimen (kgF), $D = (D_1 + D_2) / 2$ is the mean value of D_1 and D_2 (lengths of diagonals of indentation, mm).

3. RESULTS AND DISCUSSIONS

3.1. Analysis Of Structure and Morphology Of The Composites

a. TGA Analysis

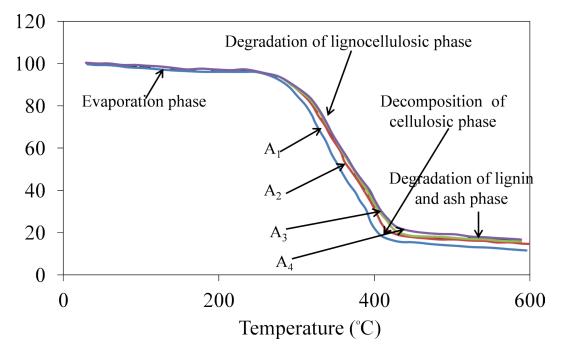
TG results are usually presented in curves of weight loss of material to temperature and called as thermo grams. The thermal stability of surface modified OPP is higher than raw OPP material which is clearly noticed from Fig. 6. Bio fibers mainly consist of cellulose, hemicelluloses and lignin which are responsible for their properties. Other stable or partially stable elements such as pectin, wax and water soluble substances may be also present in lingo cellulosic fibers. As cellulose is the main ingredient in bio fiber, it has a significant contribution towards thermal degradation. The linear chain of cellulose polymer begins to decompose at lower temperature and due to catalytic effects of natural occurring inorganic elements can form large amounts of char. Thermal decomposition of hemicellulose, which has relatively higher level of chain branching but a smaller degree of polymerization of cellulose precedes cellulose. However, the effect is limited due to proportion of fiber content. Lignin which contains both aliphatic and aromatic hydrocarbon is a complex structure. Thermal decomposition of lignin occurs in wider ranges of temperature that start earlier but extend to higher temperatures than hemicelluloses and cellulose degradation. However, the effect is also limited due to lower content in the fiber. The thermal decomposition of bio fiber is usually divided into four stages. The initial phase is the evaporation of moisture which occurs in between 30 to 100°C followed by lingo cellulosic components, cellulose components, lignin and ash at a range of 220-315°C, 315-400°C, 165-900°C and 1723°C respectively. Several authors reported that the decomposition of cellulose starts at 220°C and completed at 315°C.

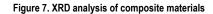
TGA graphs of treated and untreated materials are plotted as a function of percentage of weight loss versus temperature as shown in Fig. 6. From the Figure, it has been noticed that the chemically treated specimens show superior thermal stability than raw samples. At the initial stage the decomposition is due to evaporation of volatile substances between 30 to 100°C and all the treated and untreated materials showed the similar behavior of thermal degradation. Then thermal degradation of lingo cellulosic components of all samples occurred in between 330-340°C (raw 314°C, benzoyl chloride treated 340°C, acetone treated 325°C, alkali treated 333°C). In the third phase decomposition, thermal degradation of cellulosic components occurred in between 390-400°C. The decomposition of cellulosic material started after the degradation of the hemicelluloses material. The degradation of lignin, ash and cellulose occurred in this phase. The major degradation occurs in between 340 to 450°C. The figure shows that benzoyl chloride treated samples shows better thermal stability. It has been also noticed that, in case of chemically treated materials the decomposition of cellulosic material shifts towards higher temperature to some extent. Kaushik et al. (2012) reported that benzoyl peroxide treated sisal fiber shows better thermal stability than alkali treated.

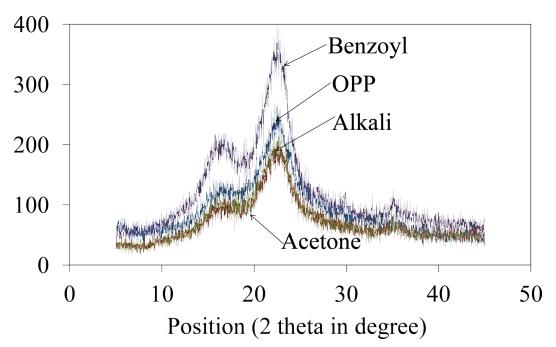
b. XRD analysis

XRD of raw and chemically treated OP powders is presented in Fig. 7. It revealed that the major crystalline peak (major) of each profile occurred between 22° to 22.75° at 2θ angle. This shows the cellulose crystallographic plane (002). The amorphous peak occurred at 2θ value ranging from 15.4° to 16.5°. There is a notable increase in the intensity of the crystallographic amorphous plane due to surface modification of material and the maximum intensity was noticed in case of benzoyl chloride

Figure 6. TGA analysis of raw and chemically treated OPP materials (A_1 stands for untreated sample, A_2 for acetone treated, A_3 for alkyl treated, A_4 stands for benzoyl chloride treated specimen).







treated OP powder. The crystallinity index I_c of raw sample and chemically treated samples were calculated using Eq. (10)

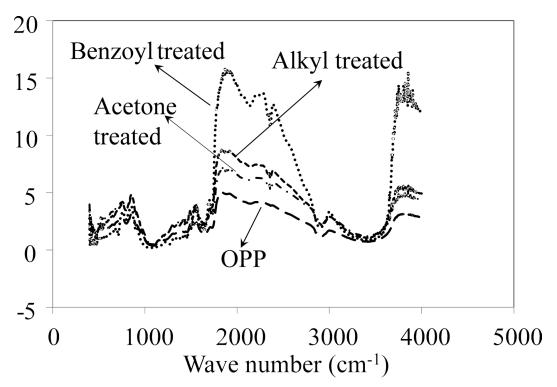
$$I_{c} = \frac{I_{002} - I_{am}}{I_{002}}$$
(10)

where I_{002} and I_{am} , respectively are the maximum intensity of diffraction of the lattice peak and diffraction of amorphous material at a 2θ angle (Roncero et al., 2005).

c. FTIR Spectroscopy

FTIR spectrum comparison graph of OP powder before and after surface modifications is presented in Fig. 8 There is a decrease in absorption spectra for the chemically treated OP powder which is clearly observed in the figure. The reason may be due to presence dissolution of non-polar covalent compounds like wax, fat, etc. The reduction of absorption spectra is more prominent in the benzyol treated OP particulate due to strong non polar solvent. It has been observed that the major functional groups of the OP particulate remain same after surface treatments. However, there is a drop for the peak intensity and shifting in many cases.

Figure 8. FTIR analysis of raw and chemically treated OPP material

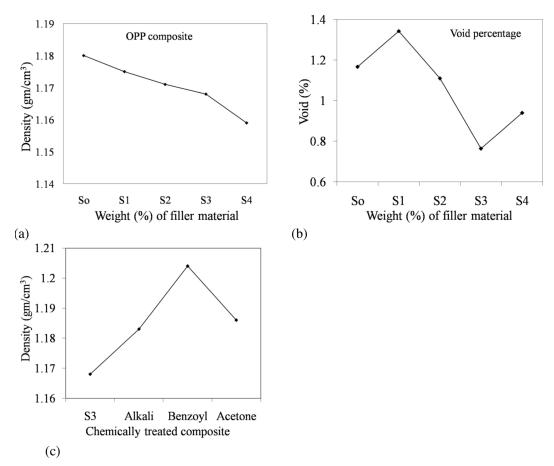


3.2. Mechanical Properties

a. Density and Void Content

Density is one of the basic parameters to establish the characteristics of the composites which depend upon the presence of the relative percentage of matrix and reinforcement in the composite. As the filler material has a low density, the density of the fabricated OP particulate composite decreases in comparison to pure epoxy composite with an increase in the filler percentage as seen in Fig. 9(a). The voids and pores are always present in the fabricated samples and its significance in the mechanical properties was marked during the calculation of experimental and theoretical density. The percentage of void present in the composite can be calculated from the experimental and theoretical values density. From Fig. 9(b), it is evident that the minimal void percentage is there in S3 composite as compared to the other weight percentage of the filler material due to good stuffing of the matrix and filler materials. It is also noticed that the void percentage of pure epoxy is higher in comparison to filler composites except S1 composite. However, on further increasing the OP particulate material (>30 wt %) the void percentage in the composite increases gradually which may be due to an imperfect combination of matrix and filler material. During fabrication of composites the formation of cavities which are filled with air are obvious (Månson et al., 2000). From Fig. 9(b), it is evident that the volume of void

Figure 9. (a) Density of OP particulate and (b) Void percentage present in different weight fraction and (c) Density of chemically treated composite.



percentage is within the limit of 2% in composites. Thus, the developed specimens are suitable for further analysis. The developed composite after chemical treatment with benzoyl chloride gives better density as compared to other chemical treatment as presented in Fig. 9(c).

b. Tensile and flexural strength

The strength and stiffness properties of the bio-waste reinforced composites depend upon the strength and weight percentage of reinforcement which is documented in several literatures. It is clear that the strength and other properties of the manufactured composite vary with different fiber loading. Fig. 10 evidently points out that both tensile strength and flexural strength increases by incorporating the OP particulates up to 30 wt %. With the further addition of OP particulates, the above mentioned properties degrade. This may have been due to reaction at the interface between the matrix and filler material, which is inefficient to transfer tensile and flexural stress between matrix and filler material along with uneven distribution of filler material within the matrix. The other cause may be due to the presence of large amounts of weak interfacial area between matrix (epoxy) and filler material (OPP). It indicates that the addition of the OP particulates up to 30% wt, the corresponding composites enhances the load bearing capacity and it develops the capacity to resist the bending of the composite. It is also noticed during the experiments that the shear strength, macroscopic properties and matrix interface bonding are improved with the addition of the voids and pores with addition of a higher wt % (<40%)



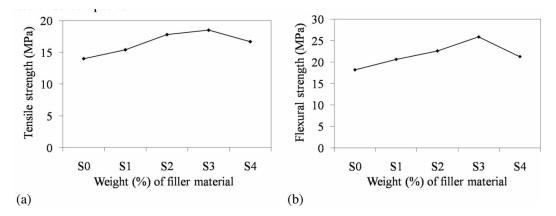
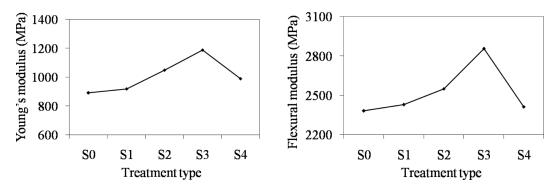


Figure 11. Variation of (a) Tensile modulus and (b) Flexural modulus with different weight fraction.



of filler. These voids are mostly found on the inter-laminar structure of manufacturing specimens which leads to reduce the tensile and flexural strength. Similar type behavior was observed by other researchers while studying jute fiber composite and coca pod husk fibers composite, respectively (Acharya et al., 2008; El-Shekeil et al., 2014).

Tensile and flexural modulus of the fabricated untreated OPP composites are shown in Fig. 11. It is clearly observed that both tensile and flexural module of the composite increases with increase in filler percentage up to 30% wt and decreases further with increase in the filler percentage that means the value of both Young's modulus and Flexural modulus is maximum in case of S3 composite.

c. Impact strength

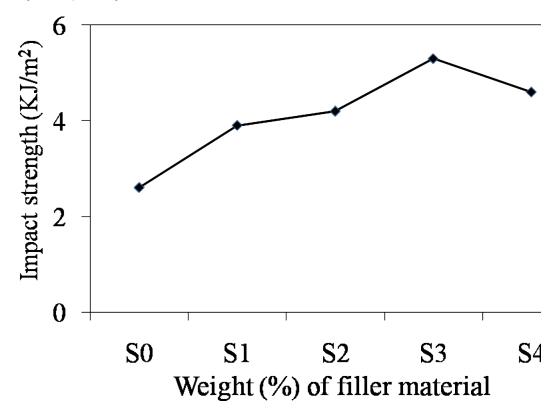


Figure 12. Impact strength at different wt % of filler material

Impact energy of the composite material depends upon the quality of the polymer, fiber and interfacial bonding of the fiber matrix (Joseph et al., 2003). The impact strength of the developed bio-waste reinforced composite material is calculated and the outcomes are reported in Fig. 12. It is observed that impact energy increases with an increase in the filler content up to 30 wt% of composites which signifies the positive contribution of the filler materials. However, impact strength decreases for higher weight percentage (40%) filler loading. This may happen due to fiber pull out from the composite materials at higher weight percentage of filler. The other reason may be the presence of

weak bond between matrix and filler material. The higher impact energy of the composite indicates the capability to absorb energy.

d. Hardness test

The property of composite material which resists scratching, indentation, penetration and plastic deformation is known as hardness. Fig. 13 presents the hardness of fabricated composites at different weight percentage of filler loading. From Fig. 13, it is noticed that the average hardness of developed composite increases with increasing the weight percentage of filler material up to the fabrication of an S3 set of composite. This may be due to the stiffness of respective composite materials. On further increasing the filler material the hardness value decreases.

The mechanical properties such as tensile, flexural and impact strength, modulus of elasticity (both tensile and bending) and hardness in every case, S3 composite gives better result as compared to other sets of composites. However, with the increasing wt % of the filler material (S4 set of composites) such properties decrease due to poor interfacial adhesion between matrix and filler material which leads to the formation of micro-crack at the internal structure of the specimen. Similar behavior is observed in jute fiber reinforced composites (Rana et al., 2003; Mohanty et al., 2001).

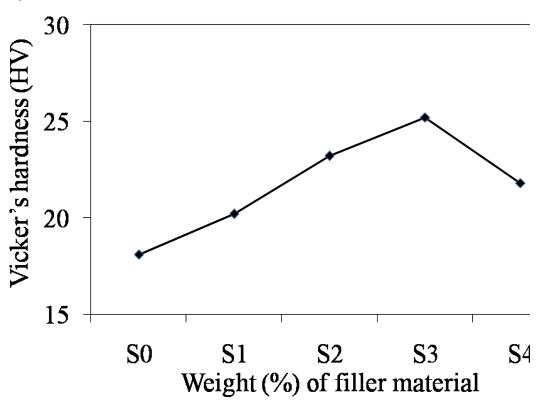
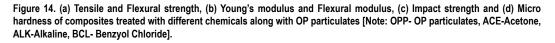
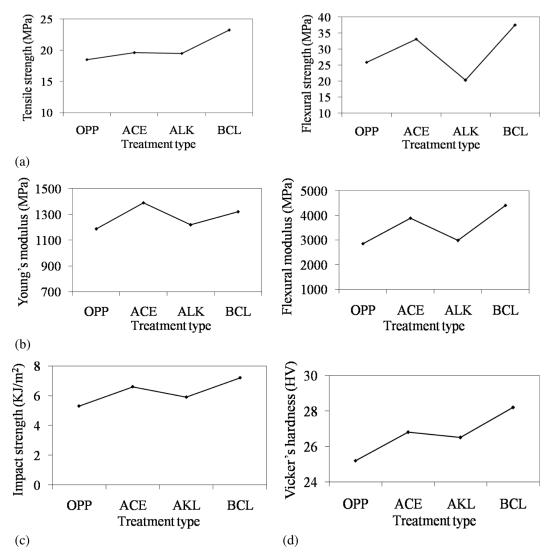


Figure 13. Micro hardness at different wt % of filler material

From the experimental analysis, it was found that the S3 set of composites has the best mechanical properties as described above. To further improve the mechanical properties, S3 composite was reinforced with OPP material pretreated with several chemicals. The improvement of mechanical





properties as shown in Fig. 14 due to the pre-treatments may be due to the change in configuration of the cellulose anhydro-glucose unit of OPP material. Due to chemical treatment the hydroxyl group of cellulose anhydroglucose units reduces and also, there is possible elimination of weak and amorphous non cellulosic content that binds the filler materials into bundles. Removal of binder materials leads to fibrillation of fiber which in turn increases the fiber aspect ratio and surface area. Due to increased surface area, there is a possibility of increase of chemical binding which leads to sites of mechanical anchoring. The mechanical anchoring causes an enhanced matrix fiber interpenetration ensuring improvement of mechanical properties. The effective load transfer between matrix and filler material is more in chemically treated fabricated composite due to availability of more surface area which ensures better wettability of the filler materials by the matrix. It is also observed that the fibrils obtained after chemical modification, are arranged themselves in the direction of applied

load. The properties enhanced significantly in the benzoyl-chloride treated composites followed by acetone treated composites and minimum enhancement occurred in alkali treated composites. Similar results are reported earlier for kenaf composites with alkali modified and benzoylated sisal fibers, respectively (Fiore et al., 2015; Nair et al., 1996).

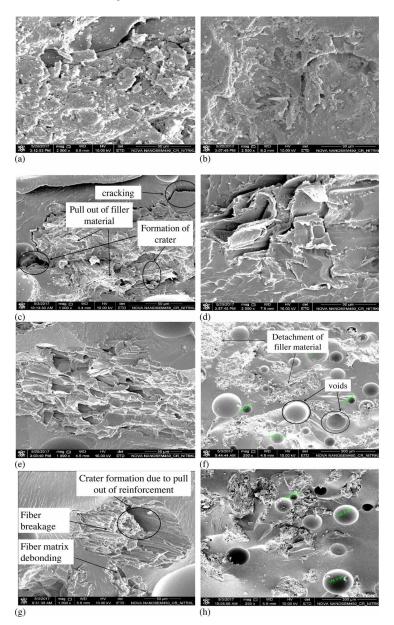
3.3 SEM Micrograph analysis

SEM micrographs of untreated and treated composite specimens after mechanical tests are presented in Fig. 15. From the SEM micrographs in Fig. 15(a) and (b), it is clearly noticed that due to tensile loading, there is no pull out of filler material. However, some cracks are clearly visible in the surface of the specimen which reveals poor quality of interfacial adhesion between matrix and filler material for the set of S3 materials. Fig. 15(c) presents that the morphology of the fracture surface of S4 composite under tensile loading where a poor inter facial bonding between matrix and filler materials is noticed which shows less tensile strength. However, the composites after benzoyl chloride/alkali treatment, it is observed in Fig. 15(d) and (e) that there is no sign of breaking or removal of filler material from the matrix. This shows the existence of enhanced adhesion at the inter phase of composites which increases the strength of the composite. The interfacial adhesion of the composites after chemical treatment gives better tensile strength.

Fig. 15(f) and (g) present the SEM micrographs of untreated S3 and S4 set composites under bending test respectively. It is observed that debonding of filler materials is observed at some places in case of S3 composite; however, most of the filler materials are intact with the matrix. Due to debonding of filler materials, less number of small voids is visible at the interface of S3 composite which do not create any problem for the characterization of the composites. On the other hand, the debonding between matrix and filler material is clearly noticeable in S4 composite. The mentioned composite has low flexural strength as small amount of the matrix material is present there. Fig. 15(h) shows the micrograph under flexural loading of S3 composite after benzoyl chloride treatment. It is noticed that there is no sign of breaking and bending of the fiber in the micrograph of the treated specimen. In addition, there are no signs of trench or grooves in the composite. The chemically treated composite is capable of withstanding flexural load without any damage to the specimen.

The present results could not be validated with previous published work in the respective field. This is due to lack of availability of any such studies (Batori et al, 2017; 2018; Li et al, 2020; Sahoo et al, 2017). Some recent studies consider OPP reinforced epoxy composites for wear characterization (Naik et al, 2021a, 20121b, 2021c) and mechanical behavior evaluation (Naik et al, 2020a, 2020b). However, to the best of the authors' knowledge, there is no such literature available that reports on chemically treated OPP reinforced epoxy composite materials attempted here. In fact, this essentially establishes the novelty of the present study. Moreover, the present study ventures into the experimental front to evaluate the effect of different chemical treatments of OPP reinforced epoxy composite materials on its mechanical behavior. It is admitted that the results are discussed here in terms of experimental observations. It would have been an added merit if the same could have been explained through some theoretical modelling or descriptions. Nevertheless, this will be attempted in future studies. Also, evaluation of tribological characteristics of such chemically treated OPP reinforced epoxy composites for such chemically treated OPP reinforced epoxy composites for evaluate the effect of the and there been an added merit if the same could have been explained through some theoretical modelling or descriptions. Nevertheless, this will be attempted in future studies. Also, evaluation of tribological characteristics of such chemically treated OPP reinforced epoxy composites defines another area of future research.

Figure 15. SEM micrographs of untreated and treated composites: (a, b) S3 composite material under tensile loading; (c) S4 composite material under tensile loading; (d) Benzoyl chloride treatment of S3 composite material; (e) Alkali treatment of S3 composite material; (f) S3 composite materials under bending test; (g) S4 composites under bending test; (h) Benzoyl chloride treated S3 composites under flexural loading.



5. CONCLUSION

In this study, the effects of chemical treatment on mechanical and physical properties of bio-waste OP particulates have been studied. Various characterization tests such as FTIR, XRD, TGA and SEM, etc. were performed to inspect the properties of OP particulates and following conclusions can be drawn:

- OP particulates are suitably bonded with epoxy resin to prepare composite material.
- Due to chemical modifications, the crystallinity index of the OP particulates increases. The effect is maximum in alkali medium and minimum in acetone medium.
- Absorption spectra of the OP bio-composites are reduced by chemical treatment (alkali, benzoyl chloride and acetone) due to dissolution of non-polar covalent compounds. With benzoyl chloride treatment, the effect is significant as it behaves as a good non polar solvent and dissolves more amount of non-polar compound.
- Strength and modulus of the composite increase with an increase in the weight percentage of filler material. S3 set of composites (30% filler) yields better result as observed from the experimental results. During SEM analysis, it is observed that the S3 set of composite shows better mechanical properties because the structure restricts the pull out of filler material from the matrix. However, there is decrease in mechanical properties for higher weight percentage filler loading (S4 composite with 40% filler) because of poor interfacial adhesion between matrix and filler material.
- Physical and mechanical properties of OP particulates composites are improved significantly after chemical modifications.
- Reinforced composite material after benzoyl chloride treatment gives better physical and mechanical properties as compared to other chemical treated filler reinforced composites. From SEM micrograph, it is clearly observed that the chemically treated composites have better interfacial adhesion between matrix and filler materials.

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