



# Synthesis and characterization of natural fibre with ZnO nanocomposites

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## Abstract

The applications of nanocomposites are not limited to a single field but are widely spread across a wide range. The application of nanocomposites embraces automotive, solar panels, sporting goods, aerospace, structural, cryogenic vessels, structural gas and oil pipelines. The polyester resin matrix-based polymer ZnO nanocomposites will be synthesized by the solution casting method. Manufacturing of nanocomposite is recommended by adding nano ZnO and nano coconut shell filler in polyesters at various weight ratios using the hand layup process. The crystalline structure of both nano-particles will be investigated using the X-ray diffraction method. Purity and base polymer composition will be determined by the Fourier transform infrared spectroscopy method. The mechanical and thermal properties of nanocomposite were evaluated. When compared to pure ZnO-polyester and polyester, the resulting CSNF-ZnO composites had better mechanical and thermal properties. The findings of this study suggest that precipitation-synthesized CSNF-ZnO nanocomposites could find potential applications in the automobile field and in the development of high-strength rubber composites materials.

**Keywords** Nanocomposites · ZnO nanocomposites · Coconut shell filler · Hand layup process · Polyesters

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## 1 Introduction

Nanocomposites are used in a variety of industries, including aerospace, manufacturing, and biological applications. These nanocomposites would be more effective in a variety of settings [1]. Nano-particle reinforcement can improve the corrosion resistance, wear resistance, mechanical strength, and damping properties of the basic matrix material [2]. The hybrid production strategy on metal nanocomposites has the potential to dramatically increase mechanical properties and age hardening response [3]. Nanocomposite films have a greater corrosion resistance than cobalt coating materials [4]. Abdiryim et al. [5] Incorporating ZnO nano-particles into composites can boost photocatalytic performance when exposed to natural sunshine, according to research. Solution-cast polyvinyl alcohol-nano zinc oxide composites are intriguing materials for prospective applications [6]. Photocatalytic ZnO nanostructures have a lot of potential for medical, sensing, and photovoltaic applications. The fundamentals of outstanding photocatalytic activity are the crystalline structure, morphologies, electrical configuration, and overall properties of ZnO nanostructures [7]. Another form of filler that is good against food-borne pathogenic bacteria is

metal oxide nano-particles. Crystallinity, shape, dimensional stability, particle size, and overall properties of ZnO are all greatly impacted by the synthesis processes, and ZnO may have its overall features considerably improved by the application of doping and coupling with other kinds of materials [8]. Although the NPs' antibacterial property has recently gotten a lot of interest, the bactericidal mechanism is still unknown. The most likely mechanism is the production of reactive oxygen species and their interaction with other activities such as electrostatic cell membrane damage, disruption of metal/metal ion homeostasis, protein/enzyme dysfunction due to amino acid side chain oxidation, and genotoxicity changes in cell membranes due to the presence of photosensitive transition metal oxides [8, 9]. One of the most extensively used techniques for determining the crystalline structure, phase nature, lattice parameters, and crystalline grain size in nano-particles is X-ray diffraction (XRD). Because it gives statistically representative, volume-averaged values, it was routinely conducted in powder form materials, usually after drying their respective colloidal solutions [10]. Structure factors like average grain size, crystallinity, strain, and crystal defects can all be determined using XRD. A monochromatic beam of X-rays scattered at precise angles from each pair of lattice planes in a sample produces XRD peaks through constructive interference. The atomic locations within the lattice planes control the peak intensity [11]. Polyester resin matrix composites have good mechanical and morphological qualities, making them appropriate for ceramic applications [12]. In the co-precipitation approach, metal nanoparticles were produced at various temperatures, yielding composites with varying characteristics [13]. Nanofillers have been shown to improve the physical properties of polyester matrices when used as additives in polyester composite manufacturing [14]. The resulting product can be used to make polyester composites with higher tensile and flexural strengths, dimensional stability, and surface quality than neat polyester in sheet and bulk moulding compounds. In a range of industries, such as aerospace, electrical, automotive, and appliance parts, nano-filled polyester has been used to replace traditional materials [15]. Yang and Pascault [16] Determined that the reduction of flexural properties at high volume fractions of fibres was due to a lack of contact between matrix and fibres, as well as present dispersion difficulties. Composites constructed of natural fibres have a higher impact strength at high weight fractions. So far, no major research on the mechanical properties of CSNF-ZnO nanocomposite materials has been reported. By separating each nanoparticle into various weight fractions, the crystalline structure basis properties and purity of each nanoparticle could be determined. The examination of mechanical qualities and the identification of mechanical parameters were carried out. The increased impact strength of composites made of natural fibres at high weight fractions [16]. There has been no major research on

the mechanical characteristics of CSNF-ZnO nanocomposite materials published too far. Simple precipitation and synthesized ZnO-CSNF nanocomposite has been developed in this study. Identification of mechanical characteristics and investigation of thermal properties were performed.

## 2 Materials and methodology

Polyester is a polymer with an ester functional group in the main chain. It most commonly refers to the material polyethylene terephthalate (PET). Polyesters are made up of both naturally occurring chemicals like plant cuticle cutin and synthetics like poly butyrate. Biodegradable polyesters include natural polyesters and a few synthetics. Polyester, depending on its chemical structure, can be thermoplastic or thermoset. Hardener-cured polyester resins are also available; however, the most common polyesters are thermoplastics. Polyester resins are unsaturated synthetic resins made by the reaction of dibasic organic acids and polyhydric alcohols. Maleic anhydride, a diacid-functional raw material, is widely used [17–22].

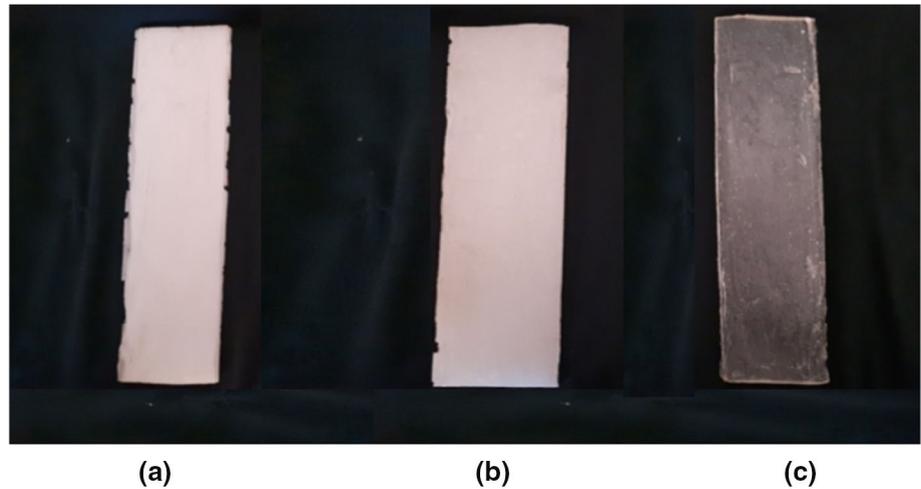
Zinc oxide nano-particles have antibacterial, anticorrosive, antifungal, and UV filtering properties that are unrivalled. Zinc oxide nano-particles are colourless hexagonal crystals or a white dusty powder. When heated, the particle turns lemon yellow and then returns to white. It weighs  $5.61 \text{ g/cm}^3$  and has a density of  $5.61 \text{ g/cm}^3$ . It evaporates at temperatures above  $1300 \text{ }^\circ\text{C}$  and sublimates at temperatures above  $1800 \text{ }^\circ\text{C}$ . As a result, there is no melt and only a direct transition from the solid to the gaseous aggregate state. Zinc oxide nano-particles have a wide range of applications, including technical products, cosmetics, and pharmaceuticals. It was synthesized using a magnetic stirrer and a direct precipitation method with zinc nitrate and sodium hydroxide as precursors in a 1:2 ratio [18]. After allowing the solution to settle for 24 h, the supernatant liquid was carefully removed. This procedure was repeated two to three times. Then it was dried and kept at  $600 \text{ }^\circ\text{C}$  in a muffle furnace for 2 h to obtain zinc oxide [23–29].

Coconut shell nano fibre is an industrial product because it is primarily used as a filler. It's used to make thermoset moulding powders like phenol formaldehyde molding powder or Bakelite, as well as synthetic resin glues. Coconut shells were free of coir pith, and the uncarbonized shells were sundried for 60 days before being broken down into smaller pieces using a hammer crusher. Crushed coconut shell pieces were ground using a two-disc grinder to obtain coconut shell powders retained in the pan below  $37 \text{ }\mu\text{m}$ , which were used as coconut shell natural nanofibres.

**Fig. 1** Various composite power used in this study



**Fig. 2** Prepared composite sheets  
**a** pure polyester sheet  
**b** ZnO/polyester nanocomposite  
**c** Pure polyester/ZnO/CSNF Composite



## 2.1 Preparation of composite sheets

Separately, 30 grammes of resin (polyester) and 0.5 grammes of hardener were placed in two beakers. For about 15 min, the resin is continuously stirred with a mechanical stirrer. The hardener (methyl ethyl ketone peroxide C<sub>8</sub>H<sub>18</sub>O<sub>6</sub>) was then added to the resin and stirred by hand. 1 drop of catalyst (liquid cobalt octoate 6%) is added and mixed against this. After that, the mixture is poured into the metal mould. It was left undisturbed for 4–5 h before being cooled at room temperature. The synthesized sheet can then be removed from the mold. Figure 2a shows the prepared polyester composite sheet [30–34].

The zinc oxide nano-particle of 1% of the weight was dispersed into 30 g of resin, and both were mixed by a high-speed mechanical stirrer for 22 h along with 0.5 g of hardener was added and mixed. Finally, a drop of catalyst is added and mixed with hand stirring. Then the mixture is then poured into the metal mold. It was kept undisturbed for about 4–5 h at room temperature. Then the synthesized composite sheet was removed from the mold, as shown in Fig. 2. with a dimension of 200 × 50 × 1 mm, respectively. The above same procedure was continued for the preparation of natural fibre composites with 1% CSNF, 1% ZnO and polyester, as shown in Fig. 2.

## 3 Characterization

### 3.1 X-ray diffraction

Crystallographic structure determination has traditionally relied on X-ray diffraction (XRD) as a means of studying the structure of crystalline materials. The crystal was treated with an X-ray beam that was collimated, and the angles at which the beam was diffracted were measured. Crystals contain regularly recurring characteristics that enable them to diffract light with a wavelength in the range of interatomic separations, which were in the range of 2–3 atomic separations at the time. When it comes to XRD tests, the CuK radiation with a wavelength of 1.5418 nm was the most regularly utilized wavelength [35–39].

The XRD estimation of crystallite size was calculated using Eq. (1) by De-Bye Scherrer formula

$$D = K\lambda/\beta\cos\theta \quad (1)$$

where  $K$  is a constant,  $\lambda$  is the wavelength used,  $\beta$  is the full width half maximum,  $\theta$  is the angle of diffraction.

XRD Results of ZnO and CSNF are illustrated in Fig. 3. Powder XRD was utilized to identify the crystallite size of the produced ZnO and CSNF nano-particles and to estimate their crystal phase composition. CuK radiation ( $\lambda = 1.54060 \text{ \AA}$ )

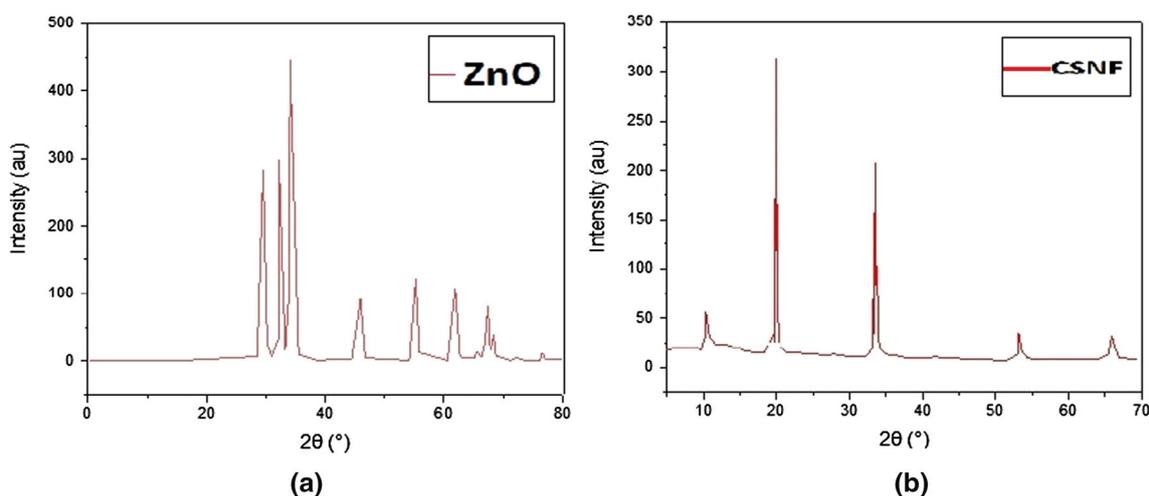


Fig. 3 XRD Results of a ZnO b CSNF

operating at 40 kV/30 mA was used in the XPERT-PRO diffractometer system (PW 3050) with automated data acquisition. The CuK radiation ( $\lambda = 1.54060 \text{ \AA}$ ) was used in the XPERT-PRO diffractometer system with automatic data acquisition. It was necessary to collect diffraction patterns in the  $2\theta$  range ( $0^\circ$ – $80^\circ$ ) with step sizes of  $0.05^\circ$  and collection times of 2.0 s per step in order to get the desired results. Every measurement began with a calibration of the diffractometer. The X-ray diffracts grams of CSNF have higher intensity  $2\theta = 22.34^\circ$ ,  $24.81^\circ$  and ZnO  $2\theta = 18.12^\circ$  and  $19.2^\circ$ . Synthesized ZnO and CSNF crystallite sizes were observed to be 49.02 nm and 53.2 nm, respectively, confirming the produced ZnO particle's nanoscale status. The X-ray diffraction spectra confirmed that the pure ZnO and CSNF nanopowder was crystalline in nature.

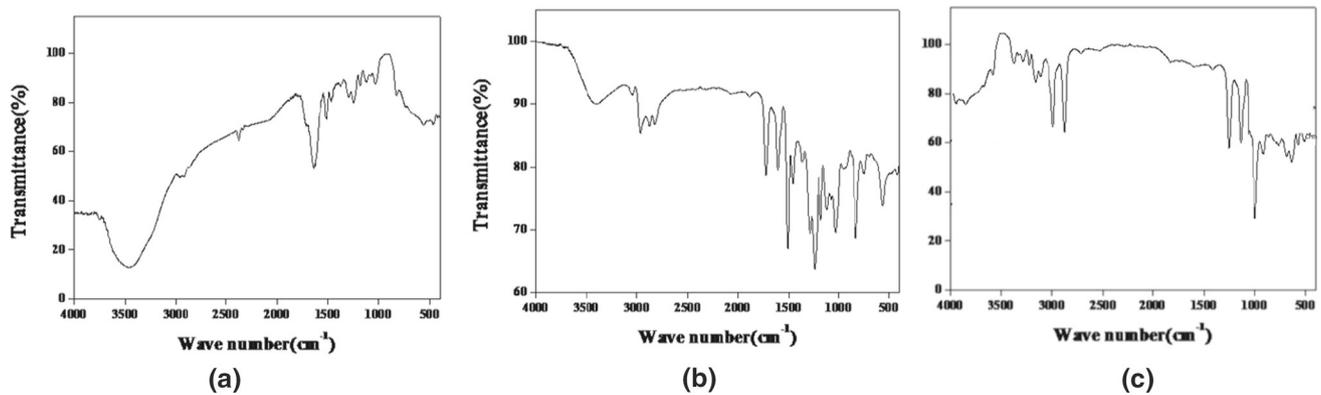
### 3.2 Fourier transform infra-red spectroscopy

The analytical method used to determine the chemical composition of organic, polymeric, and inorganic materials uses Fourier Transform Infrared Spectroscopy (FTIR Spectroscopy). The FTIR technique employs infrared light to examine the chemical characteristics of test materials. In order to determine the purity of the polymer, FTIR Spectroscopy was used to detect the basic polymer composition, as well as additions and organic contaminants. FTI-spectroscopy was utilized to examine the nanocomposite samples made from polyester, ZnO, and CSNF. It was done using a SHIMADZU IR Affinity-1 in the  $4000$ – $400 \text{ cm}^{-1}$  range for FTIR measurements. Transmission is the FTIR characterization mode. There is a clear depiction of the sample in the FTIR spectrum and assignments displayed in Fig. 4a–c.

FTIR analysis was carried out in order to identify the chemical structure of ZnO and CSNF. The resulting spectra are shown in Fig. 3a–c. The hydroxyl ( $-\text{OH}$ ) group is responsible for the large peak in the CSNF spectra that can be seen at  $3218 \text{ cm}^{-1}$ . In addition, the peak at  $1748 \text{ cm}^{-1}$  was identified as the carboxyl ( $\text{C}=\text{O}$ ) group of the acetyl group from the hemicellulose, and the peak at  $671$  was connected to the C-H vibration in the lignin. ZnO has additional bands at  $1242 \text{ cm}^{-1}$  ( $-\text{CO}$  of carbonate),  $1086$  ( $-\text{O}-\text{C}=\text{O}$ ), and  $856$  ( $\text{C}-\text{C}$  methyl group), in addition to the characteristic peak that was seen at  $3317$  for the stretching vibration of ( $-\text{OH}$ ). There are also additional peaks at  $2941$  ( $-\text{CH}_3$  vibration) and  $1723$  ( $\text{C}=\text{O}$  stretching). ZnO and CSNF absorb at  $1723$  and  $1748$ , respectively, the typical peaks. The filler and ZnO peaks are the only new peaks in the biofilm spectrum, indicating that no chemical by-products were formed during composite production between the filler and ZnO. PVA and CSNF both have OH and  $\text{C}=\text{O}$  groups, which might potentially create hydrogen bonds with ZnO and CSNF. In ZnO and CSNF, a prominent and spiky absorption band at  $1723$  was ascribed to the carbonyl ( $\text{C}=\text{O}$ ) stretching vibration. Due to the presence of a strong  $\text{Zn}-\text{O}-\text{Zn}$  band in this range, the natural fibre group was more absorbent than in  $\text{ZnO}$ -polyester or pure polyester. Due to the presence of a strong  $\text{Zn}-\text{O}-\text{Zn}$  band in this range, the natural fibre group may be more absorbent than in  $\text{ZnO}$ -polyester or pure polyester.

### 3.3 Thermal properties

The thermal properties were analyzed using differential scanning calorimetry and thermogravimetric analysis for the three specimens. The composites sheets heat flow and change of weight is monitored from the range of  $20$ – $800 \text{ }^\circ\text{C}$  at a scan speed of  $10 \text{ }^\circ\text{C}/\text{min}$ ,  $35,000 \text{ mg}$  and for DSC  $5000 \text{ } \mu\text{V}$



**Fig. 4** a FTIR spectrum of pure Polyester b FTIR spectrum of Polyester + 1% wt ZnO b FTIR spectrum of Polyester + 1% wt ZnO + 1% wt CSNF

**Table 1** TGA data of Polyester, ZnO and CSNF added nano-composites

Samples	Polyester	Polyester + 1wt% ZnO	Polyester + 1 wt% ZnO + 1 wt% CSNF
Temperature °C	420	480	520

by using Differential Scanning Calorimetry and thermogravimetry. Following the evaluation, it was exposed that the thermal stability of the ZnO nano-filler and CSNF added nanocomposites is somewhat increased when compared to the thermal stability of the pure polyester and ZnO nanofiller composite sheet after the testing. A possible explanation is that natural nano-particles interfere with the creation of a high cross-linked molecular structure in polyester or that larger free volume fractions in polymer nanocomposites arise from their spatial blockage. The thermal behaviour of the generated samples has been investigated, and withstand data is shown in Table 1.

### 3.4 Mechanical properties

The mechanical characteristics of polymer nanocomposites include strength, toughness, ductility, and elongation. Polymer nanocomposites have the following qualities: When a polymer composite is stretched while under tension, it produces strain as a result. It is determined by applying a load to the material under test that the tensile stress–strain connection exists. The ultimate strength of the material might be calculated using the greatest tensile strain peak. The break-point is the point at which the tensile stress–strain curve comes to a stop, and this point may be identified as the tensile strength value of material in certain cases. Elongation is a kind of material deformation that is often expressed as a percentage of the original length of the material. In order to

**Table 2** Tensile strength of Polyester, ZnO and CSNF added nano-composites

Samples	Pure polyester	Polyester + 1 wt% ZnO	Polyester + 1 wt% ZnO + 1 wt% CSNF
Elongation at break (%)	1.7622	2.9997	3.844
Maximum load (N)	48.4	81	128
Tensile strength (MPa)	1.936	3.24	5.12

calculate the flexural strength of polymer sheets, the maximum bend area before breaking must be determined first. Strength tests on the generated sheets are carried out using mechanical analyzers in a tensile mode in accordance with ASTM D 638 test standard and in a flexural mode in accordance with the same standard, respectively. Preliminarily, the specimen for testing is cut from the sheet with a clean razor blade and polished to generate a smooth top surface on the upper side of the cut sample for the tensile test.

### 3.5 Tensile properties

The tensile strength of the specimen was tested by a computerized universal testing machine Shimadzu- Japan, and the range of the machine was 1–100 KN. Length (L), width (d), and thickness (t) of the specimen used in the experiment were 150 mm, 25 mm, and 1 mm sheet thickness. Sheet at thickness respectively in units of Mega Pascal (MPa). The highest amount of tensile strength before breaking is evaluated by Eq. (2). Table 2. Shows the

$$Tensile\ strength(TS) = \frac{Load\ at\ breaking\ point}{Width \times Thickness}$$

**Table 3** Flexural strength of Polyester, ZnO and CSNF added nanocomposites

Samples	Pure polyester	Polyester + 1 wt% ZnO	Polyester + 1 wt% ZnO + 1 wt% CSNF
Ultimate flexural strength	95.13	78.21	65.34
Flexural modulus (GPA)	2.45587	3.2190	4.82

When CSNF with ZnO nanofiller added to polyester nanocomposites is compared to pure polyester and ZnO nanocomposite, the tensile strength rises. This suggests that agglomeration of nano-particles may occur as a result of the intense contact between the nano-particles and the resin when they are added.

### 3.6 Flexural properties

In composite materials, flexural strength refers to the capacity of the material to resist bending stresses that are applied perpendicular to its longitudinal axis. Flexural testing is carried out utilizing the 3-point bending technique in accordance with ASTM D790 standardized testing machine Shimadzu-Japan, with a range of 1–100 KN for the machine. The test specimen utilized in the experiment has the following dimensions: length (L), width (d), and thickness (t) are 1 cm, 2.5 cm, and 0.4 cm in length, width (d), and thickness (t) accordingly. When compared to pure polyester sheet and ZnO with polyester composite, the flexural strength of the CSNF and ZnO added polyester sheet rises significantly. The increase in flexural strength is proportional to the length of the chain. In addition, the flexural strength of polyester resins with CSNF and ZnO added is stronger because of the van der Waals bond, which is a weak link with a large number of atoms. Table 4 shows the values of flexural strength for pure polyester nanocomposites, polyester nanocomposites with ZnO (1 wt %) added, and polyester nanocomposites with CSNF (1 wt percent) and ZnO (1 wt %) added in combination with polyester. The Eq. (3) provides a simple method for calculating flexural strength.

$$\text{Flexural strength}(FS) = \frac{3FL}{2W \times d^2}$$

where F is the maximum test load, L is the length between load points, W is the width of the sample piece, and d is the thickness of the sample piece. Table 3. Illustrate the flexural strength of the composites.

**Table 4** Swelling percentage of Polyester, ZnO and CSNF added nanocomposites

Number of days	Weight difference for polyester	Weight difference for polyester + ZnO	Weight difference for polyester + ZnO + CSNF
1	0.019	0.001	0.023
14–15	0.030	0.002	0.039
16–30	0.044	0.004	0.14
31–45	0.055	0.005	0.18
46–60	0.064	0.007	0.25

### 3.7 Hydrophobic character

Apart from composite mechanical qualities, one of the most appealing and practical features of polyethylene is its outstanding barrier capacity, which results in a greatly decreased permeability to moisture and gases. However, this property has not been adequately studied in the past. Due to the obvious intrinsic hydrophobic nature of zinc oxide nano-particles, the introduction of zinc oxide nano-particles into the resin reduces the % water absorption in proportion to the percentage concentration. Additionally, the hydrogen bonding between zinc oxide and polyester, which promotes tight packing at the molecular level, might be a contributing factor to this. However, when CSNF is combined with ZnO and polyester, the resulting toxicity is increased. When compared to the pure polyester matrix and the CSNF with ZnO and polyester, In Table 4. It explained the ZnO doped polyester nanocomposites had greater hydrophobic characteristics than the pure polyester matrix. CSNF with ZnO and polyester has low hydrophobic qualities, but it has also been found to have good mechanical properties due to its composition.

## 4 Conclusion

The co-precipitation technique was used for producing the CSNF with ZnO nanoparticles in this research study. Two analyses were conducted, such as XRD analysis and FTIR investigation, to reveal chemical composition information and evaluation of the nanocomposites. The sharpness and intensity of the nano composites were increased by adding ZnO and CSNF. The addition of the same to the polyester system improves the mechanical and thermal properties. The composites showed a minor change in the absorption bands. Nanocomposite shows that nano-particles may agglomerate due to strong resin interactions, which is why the composite is stronger than pure polyester and ZnO with polyester composite. The incorporation of natural nano-particles in the

structure increases their mechanical properties by 15%. The Polyester/ZnO nanocomposite has a stronger hydrophobic property than CSNF with ZnO and polyester composites. The minimal environmental impact of these nanocomposites makes them ideal for a wide range of sustainable engineering applications.

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## Decalartions

**Conflict of interest** The authors declare that they have no conflict of interest.

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