



Sunflower Seed Husk Filler-Reinforced Epoxy Composites: Characterization, Water Absorption Behavior and Thermal Stability

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ABSTRACT

In the current work, the utilisation of sunflower seed husk as a filler for epoxy composites is examined. Utilising the compression moulding technique, the composites were created with filler volumes ranging from 5% to 50%. Particle size distribution evaluations, elemental analyses, physico-chemical characteristics, X-ray diffraction, Fourier transform infrared spectroscopy, water absorption, and thermal stability of the composite were among the various characterizations that were carried out. Based on the results, Sample ID "SSH-5" demonstrated optimum water absorption behavior and thermal stability.

Keywords: Sunflower seed husk, Epoxy, Water absorption, Thermo gravimetric Analyzer

DOI: 10.48047/ecb/2023.12.si8.656

1. Introduction

By adding fillers to the polymer matrices, it is possible to improve the characteristics and performance of the resulting polymer composites. Most polymers are inherently incompatible and immiscible in their initial states, in contrast to their composites, which have a variety of heterogeneous structures and morphologies as well as a wide range of attributes. A well-known and well-established method to improve the strength, hardness, stiffness, viscosity, and conductivity of polymers is the addition of fillers. The nature and types of polymers used in this approach, as well as the form, size, distribution, and attributes of the fillers used to increase the desired features[1]. Insufficient fiber content in the matrix gives rise to voids as the curing process takes place, ultimately leading to a decrease in the transfer of stress between the fiber and the matrix [2]. Non-filler composite exhibited higher water absorption attributed to its larger pore structures, while the type of filler had minimal impact;

the even filler dispersion increased impermeability, and Talcum powder and Egg shell powder's hydroxyl groups slightly reduced their water absorption [3]. Increasing the amount of filler in Date seed filler-Vinyl ester composites led to higher water absorption rates. Among different water environments, hot water had the highest absorption, while salt water had the lowest due to the filler's hydrophilic nature and the slow penetration of large salt molecules [4]. Applying chemical treatment to fiber leads to a smoother and more even surface, demonstrating reduced porosity in contrast to untreated fiber [5]. Because glass fibre (GF) has a lower moisture content than sugar palm fibre (SPF), adding it to composites of SPF and GF lowers the moisture content of the hybrid materials, increasing the durability of the composite [6]. The thermal stability of plantain fibers has been notably and substantially improved as a result of the effective application of permanganate treatments [7]. The thermal stability of the bio-composite sample was slightly improved by adding PLS filler to the vinyl ester resin[8]. For the epoxy-based hybrid composites, the lack of cellulose filler addition results in surface deformations, including fibre breakages, voids, and matrix breakages, which reduces dimensional stability and results in a less crystalline character[9]. The addition of biochar leads to improved thermal stability in the composites, evidenced by a shift in major degradation to higher temperatures, while glass fiber contributes to over 40% residue in all samples [10]. Cellulose content significantly influences the improvement of mechanical properties in fibers, and various chemical treatments result in a substantial increase in cellulose wt.% in Phoenix Pusilla fibers (PPF) compared to untreated PPFs [12]. The thermal stability of the biocomposites underwent a slight modification due to the extraction of fatty acids in the Pecan nutshell [13]. The Tamarind seed filler-Vinyl ester composite exhibits thermal stability up to 357°C, a temperature that falls within the range of the polymerization process [14]. Wood filler treated with sodium hydroxide undergoes surface modification, leading to higher thermal stability at elevated temperatures due to enhanced crystalline structure and elimination of natural polymers with lower molecular weight from the filler [16]. When compared to composites made with jute fibre, Calotropis gigantea powder produces better results in terms of heat stability[17]. Comparatively speaking to composites filled with sugarcane bagasse and pineapple chaffs, palm kernel shell-packed composites have the maximum potential to absorb water [18]. The material's overall thermal stability is decreased when particles are added to the polymer matrix and making it more susceptible to degradation at elevated temperatures [20]. Continuous addition of eucalypt particles improved the material's stiffness and mechanical strength due to enhanced Low surface

roughness, interfacial adhesion, and fewer vacant spaces [24]. This study involves creating epoxy composites by adding sunflower seed husk filler in volumes ranging from 5% to 50% compared to the unfilled composite. The samples were produced using conventional compression moulding, and various characterizations, such as particle size distribution, elemental studies, Physico-chemical properties, X-ray diffraction, Fourier transform infrared spectroscopy, water absorption and thermal stability, were conducted using the Thermo-gravimetric analyzer for both the filler and developed composites.

2 Materials and Methods

2.1 Choosing and preparing materials for composites

The materials employed in this study include sunflower seed husk (SSH) filler and epoxy resin, with their respective ratios provided in Table 1. Compression moulding was used to create the composites. For optimal curing of the composite material, a 10:1 mixture of epoxy resin and hardener was used. After adding the necessary quantity of reinforcing filler to the epoxy/hardener mixture, it was vigorously mixed with a homogenizer to ensure that the filler was well incorporated into the matrix material. The composites were cast in a mild steel mould, and the surface was treated with a mold-releasing chemical to make it simple to remove the cured composites. The completed mixture was put into the mould, where it was left to cure for 24 hours at room temperature.

2.2 Methods of Characterization

2.2.1 Physical and chemical attributes

To analyze the chemical composition of Sunflower seed husk (SSH) filler, various parameters were determined, lignin, wax, moisture, ash content, pectin, and density percentages, among other things. The following test methods were employed for the chemical composition analysis: Lignin Content (Method: SITRA/TC/FCC/02), Wax Content (Method: SITRA/TC/GT/09), Ash Content (on a dry basis) (Method: IS 199), Moisture Content (Method: IS 199), and Density (Method: SITRA/TC/FCC/03) as outlined by THE SOUTH INDIA TEXTILE RESEARCH ASSOCIATION (SITRA), Coimbatore.

2.2.2 Particle size distribution

The MALVERN PANALYTICAL analyzer and MASTERIZER 3 software were used to determine the SSH filler's particle size distribution. Laser diffraction, which involves evaluating the intensity of light scattered when a laser beam passes through a dispersed particulate sample, was used to evaluate the particle size and dispersion. The size of the particles in the filler material is then determined using the information gathered from the scattering pattern.

Table 1. Material selection proportion

Sample ID	Epoxy Resin + Hardener (vol. %)	Sunflower Seed Husk Filler (vol. %)
SSH-0	100	0
SSH-5	95	5
SSH-10	90	10
SSH-15	85	15
SSH-20	80	20
SSH-25	75	25
SSH-30	70	30
SSH-35	65	35
SSH-40	60	40
SSH-45	55	45
SSH-50	50	50

2.2.3 X-ray Diffraction

A Powder X-Ray Diffractometer (PXRD) machine was used to analyse the SSH filler using X-ray diffraction (XRD). This rapid analytical technique allowed for the determination of unit cell dimensions and identification of crystalline material phases. The analysis involved a way of continuous scanning the powder samples, with 2θ data collected from 5° to 90° utilising a single colour Cu-K α wavelength of radiation 1.54060 \AA .

2.2.4 Fourier transform infrared spectroscopy

The molecular structure of the SSH filler was analyzed using Fourier transform infrared spectroscopy (FTIR) with a PERKIN ELMER instrument equipped with IR software. The spectra were recorded over a wavenumber range from 400 to 4000 cm^{-1} .

2.2.5 Energy Dispersive X-Ray Analysis and Scanning Electron Microscopy

The morphology of the SSH filler was thoroughly examined using a TESCAN OXFORD instrument equipped with VEGA 3 INCA software. Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray investigation were used in this sophisticated investigation (EDAX). The SEM allowed for high-resolution imaging of the filler's surface structure, while the EDAX provided useful details on the elements that make up the filler substance. This comprehensive characterization technique offered insights into the microstructure and elemental constituents of the SSH filler, contributing to a better understanding of its properties and potential applications.

2.2.6 Behaviour in absorbing water

According to ASTM D570-99 standard, test specimens were created by cutting them from the manufactured composite plates. For sample preparation, the specimens were dried in an oven at 105°C for 24 hours [14]. After the drying process, the specimens were immersed in bore water (TDS Range: 684PPM (fair), Hardness: 440PPM (Very hard) and pH: 6.85) at room temperature to facilitate further testing and analysis. All tests were performed with three replications, and the test results are presented as the average values obtained from these repetitions.

2.2.7 Analysis using thermo gravimetry

Using the METTLER TOLLEDO TGA 2 thermal analyzer, the filler, matrix, and manufactured composite were evaluated for their thermal stability. This device monitors the rate of weight change and material behaviour in a controlled environment as a function of time or temperature. The samples were put on an alumina crucible and heated gradually at a rate of 10°C/min from 30.0 to 600.0°C. A constant supply of pure nitrogen gas was pumped through the furnace at a rate of 60 ml/min to maintain a regulated sample environment..

3. Results and discussion

3.1 Physical and chemical attributes

The substance is light due to its low density of 0.43 g/cm³. This property holds considerable significance, particularly in industries where weight reduction is a desirable factor, such as automotive applications. The findings of the SSH filler's chemical analysis

show the following content percentages: 4.88% lignin, 0.39% wax, 8.36% moisture, 3.64% ash, and 2.01% pectin. It is clear that the sunflower seed husk filler has a lower lignin content compared to Indian mallow 6.14% [2], coir 41-45% [5], Polyalthia Longifolia Seed Filler 19.72% [8] and walnut shell 50-55% [15]. The lower lignin content in sunflower seed husk filler might impact its mechanical and bonding properties within composite materials. Lower wax content may influence the filler's interaction with other materials and can be beneficial in scenarios where waxes could hinder desired interactions or bonding. It is evident that sunflower seed husk has a relatively moderate moisture content compared to the other materials. While coir 10% [5] has the highest moisture content, SSH falls within a similar range as Polyalthia Longifolia Seed Filler 8.32% [8] and Indian Mallow 9.6% [2]. Walnut Shell 6-7% [15], on the other hand, it has the lowest moisture content among these materials. The differences in moisture content can impact the suitability of these materials for various applications, particularly those sensitive to moisture absorption or degradation. Compare to Indian Mallow 5.85% [2], Polyalthia Longifolia Seed Filler 3.68% [8], SSH filler has a lower ash content tends to promote greater flexibility and impact resistance. Pectin's hydrophilic nature could influence the composite's water absorption behavior.

3.2 Particle size distribution

The Sunflower seed husk (SSH) filler's specific surface area and particle size distribution are crucial factors that affect how it behaves in composite materials. Insights into the average particle size based on volume and mass, respectively, are provided by the volume-weighted mean particle diameter ($D(3,2)$) of 83.0 m and the mass-weighted mean particle diameter ($D(4,3)$) of 259.0 m. Additionally, the volume median diameter values ($D_v(10)$, $D_v(50)$, and $D_v(90)$) of 79.8 m, 244.0 m, and 463.0 m reflect the distribution of particle sizes in the filler material by indicating the particle diameters below which 10%, 50%, and 90% of the volume of particles, respectively, reside. The specific surface area of 68.87 m²/kg highlights the increased surface area available for interactions with other materials, influencing the composite's mechanical, thermal, and physical properties.

3.3 X-ray diffraction

The XRD pattern obtained from the sunflower seed husk (SSH) particles exhibited distinctive peaks at 2θ angles of 21.50°, 34.59°, 44.62°, 72.62°, 75.66°, and 88.51°. These peaks correspond to the crystallographic planes of the constituents present in the husks. The

incident and diffracted X-rays' scattering angles are shown by the 2θ . The presence and intensity of each peak provide information about the arrangement, size, and orientation of the crystalline phases within the husks.

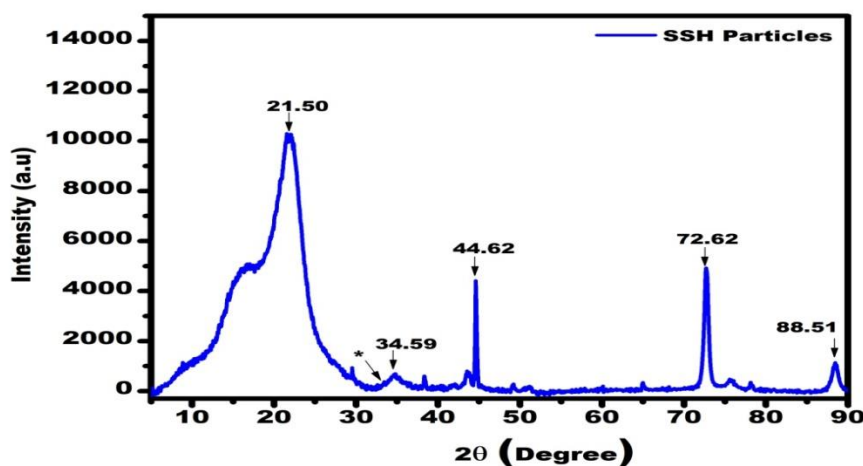


Figure 1. XRD pattern of SSH particles

From Figure 1, The average crystalline size of the SSH is 41nm evaluated using Scherer's Formula. The peak at 21.50° suggests the presence of cellulose, a major component of sunflower seed husk. The peaks at 34.59° , 44.62° , and 72.62° are likely related to the presence of lignin and hemicellulose. There is no double peak at 34.59° , which indicates that the crystals of sunflower seed hull cellulose. The type has not changed, and it belongs to the typical cellulose type-I crystal structure [21]. These compounds contribute to the structural integrity and mechanical strength of the husks. The peaks at 75.66° and 88.51° may indicate the presence of inorganic minerals or impurities absorbed from the environment. The observed XRD pattern indicates that the sunflower seed husks undergo structural changes when exposed to intense sunlight for an extended period. These changes might be attributed to the degradation or modification of organic components, as well as the influence of temperature and humidity.

3.4 Fourier transform infrared spectroscopy

In Figure 2. different types of chemical bonds present in sunflower seed husk (SSH) filler, neat epoxy, and SSH-5 are displayed. The FTIR spectrum of the neat epoxy (SSH-0) and SSH-5 exhibits similarities. The existence of peaks at 3880 cm^{-1} , 3827 cm^{-1} , 3818 cm^{-1} , 3802 cm^{-1} , 3410 cm^{-1} , 3324 cm^{-1} , and 3259 cm^{-1} indicated the presence of hydroxyl groups by

showing the stretching vibrations of O-H bonds [11, 22 & 23]. The stretching vibrations of C-H bonds are linked to the wave numbers 2922 cm^{-1} , 2907 cm^{-1} , and 2921 cm^{-1} , which suggests the existence of aliphatic hydrocarbons [19 & 22]. According to SSH-5, the signal at 2552 cm^{-1} links the stretching vibrations of C-C triple bonds, indicating the existence of acetylenic chemicals. It is clear that nitriles are present because of the strength of the peak at 2366 cm^{-1} for SSH-5, which is caused by the stretching vibrations of C-N triple bonds. Peak measurements for the stretching vibrations of C=C double bonds were 2035 cm^{-1} , 2026 cm^{-1} , 1600 cm^{-1} , and 1599 cm^{-1} . The presence of carbonyl compounds (ketones) is indicated by the presence of the band at 1634 cm^{-1} , which corresponds to the stretching vibrations of C=O bonds. At 1235 cm^{-1} , 1234 cm^{-1} , and 1236 cm^{-1} , the stretching vibrations of C-N bonds that point to the existence of amines are detected. The presence of alcohols and ethers is indicated by the peak values at 1177 cm^{-1} , 1024 cm^{-1} , 1025 cm^{-1} , and 1028 cm^{-1} , which coincide with the stretching vibrations of C-O bonds [15]. The existence of chlorinated compounds is suggested by the band at 568 cm^{-1} , 565 cm^{-1} , and 572 cm^{-1} , which is related to the bending vibrations of C-Cl bonds.

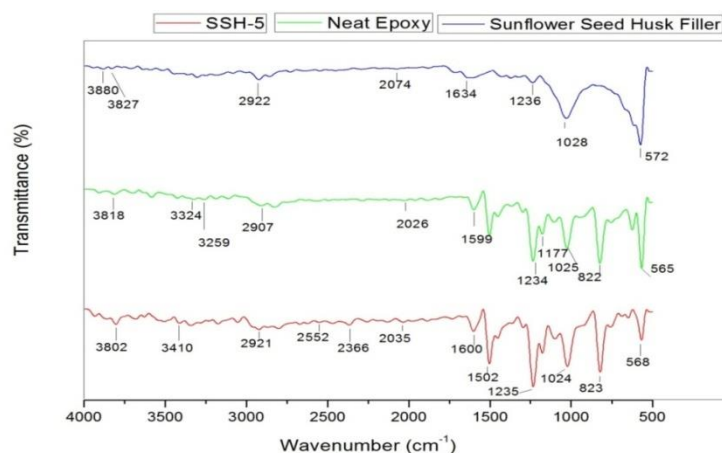


Figure 2. FTIR image of Sunflower seed husk filler, SSH-0 (neat epoxy) and SSH-5

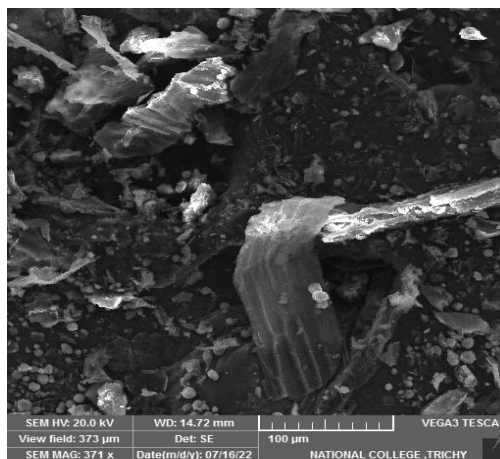


Figure 3. SEM image of Sunflower seed husk filler

3.5 Energy Dispersive X-Ray Analysis and Scanning Electron Microscopy

Utilising SEM and EDAX analysis, the elemental makeup of the Biowaste SSH filler was investigated. The SEM image of the SSH filler is presented in Figure 3. The results indicate that carbon (62.91%) and oxygen (33.93%) are the primary constituents. Correspondingly, in their studies, Nagaraj et al. [4] and Stalin et al. [8,14] noted and documented carbon and oxygen as principal elements within Date seed filler, Polyalthia longifolia seed filler, and Tamarind seed filler, respectively. With smaller contributions from magnesium (0.49%), silicon (0.14%), phosphorus (1.07%), sulfur (0.27%), chlorine (0.11%), potassium (0.73%), and calcium (0.36%). Together, these elements—Mg, Si, P, S, Cl, K, and Ca—dominate the filler material's weight proportion.

3.6 Behaviour in absorbing water

There is a distinct association between the samples' water absorption behaviour and the filler. As the amount of filler content rises, there is a corresponding elevation in water absorption due to the filler material's hydrophilic properties. Different filler/fiber reinforced composites all showed this same pattern [3,4]. The water absorption values ranged from 0.4532% for samples with 0% filler content to 2.2610% for samples with 50% filler content. This trend indicates that higher filler content allows for greater water penetration into the material, leading to increased water absorption. The presence of fillers may alter the material's porosity and surface characteristics, influencing its ability to repel or retain water. The water quality parameters, including fair TDS range and high hardness, might have

contributed to the observed water absorption behavior as well. Figure 4. displays the water absorption of the sunflower seed husk filler reinforced epoxy composite.

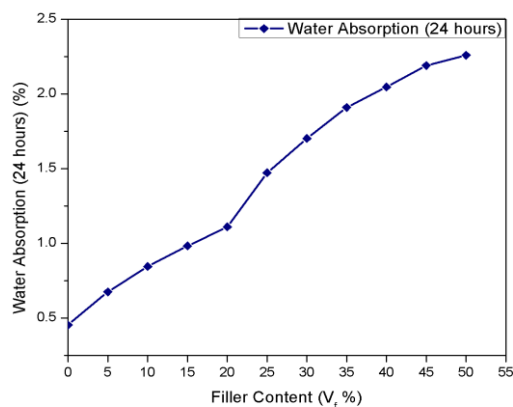


Figure 4. Water absorption vs filler percentage of SSH filler-epoxy composite.

3.7 analysis of Thermo-gravimetric

Figure 5 illustrates the thermal behaviour of SSH filler, SSH-0, and SSH-5 composites as determined by methodical temperature ramp studies. The filler composite exhibited distinct weight changes and mass losses at each stage. At Stage 1 (30-205°C), the filler composite showed a weight change of 6.7031% and a mass loss of 0.1840 mg. As the temperature increased to Stage 2 (210-450°C), the weight change significantly increased to 66.3024%, accompanied by a mass loss of 1.8200 mg. At Stage 3 (455-595°C), the weight change decreased to 4.9909%, and the mass loss reduced to 0.1370 mg. These results suggest that the SSH filler composite undergoes significant thermal decomposition at higher temperatures, leading to a substantial weight change and mass loss. In contrast, the SSH-0 composite displayed lower weight changes and mass losses compared to the SSH filler composite. At Stage 1 (30-269°C), the SSH-0 composite exhibited a weight change of 1.1064% and a mass loss of 0.0261 mg. The weight change increased to 90.7125% at Stage 2 (275-514°C), with a mass loss of 2.1390 mg. Notably, the SSH-0 composite experienced a significant weight change at Stage 2, indicating a pronounced thermal effect. At Stage 3 (520-595°C), the weight change decreased to 1.9469%, and the mass loss reduced to 0.0459 mg. These results indicate that the SSH-0 composite undergoes substantial thermal changes, leading to notable weight fluctuations and mass losses, particularly at higher temperatures. Similarly, the SSH-5 composite also demonstrated distinct thermal behavior. At Stage 1 (30-269°C), the weight change was 2.5635%, and the mass loss was 0.0571 mg. As the

temperature increased to Stage 2 (275-502°C), the weight change reached 87.8271%, accompanied by a mass loss of 1.9559 mg. At Stage 3 (508-595°C), the weight change decreased to 2.2901%, and the mass loss reduced to 0.0510 mg. These results suggest that the SSH-5 composite exhibits significant thermal sensitivity, particularly at Stage 2, where it experienced a substantial weight change and mass loss. The three composites' different thermal behaviours might be linked to variances in their chemical and micro structural compositions. The SSH filler, might have a complex thermal decomposition process, resulting in significant weight changes and mass losses. On the other hand, the SSH-0 and SSH-5 composites, being specific formulations, exhibit unique thermal responses. The observed weight changes and mass losses might be linked to phase transitions, chemical reactions, or the release of volatile components within these composites.

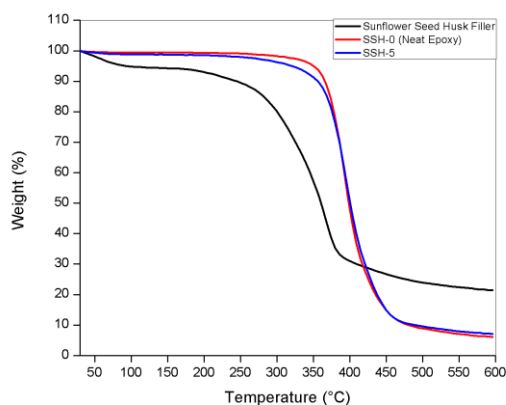


Figure 5. TGA curves of SSH filler, Neat Epoxy composite (SSH-0) and 5 v% SSH filler loadings (SSH-5)

4. Conclusions

The comprehensive analysis of sunflower seed husk (SSH) filler has yielded valuable insights into its physico-chemical properties and behavior within composite materials. Because of its low density, high lignin and pectin content, uniform particle size distribution, and high specific surface area, the filler has several possible uses. The water absorption behavior, as evidenced by a notable increase from 0.4532% to 2.2610% with higher filler content, underscores the filler's hydrophilic nature and suggests its suitability for applications where controlled water interaction is essential. Furthermore, the thermo-gravimetric analysis (TGA) results shed light on the filler's thermal stability, revealing significant weight changes

and mass losses at different stages. This thermal sensitivity could play a pivotal role in applications requiring specific temperature ranges or resistance to thermal stress. Overall, this comprehensive study enriches our understanding of SSH filler's attributes, paving the way for its utilization in diverse industrial sectors.

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