

# Mechanical and Morphological characterization of Walnut Shell reinforced epoxy composite

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**Abstract.** The present study emphasizes on the development of composite material by reinforcing Walnut Shells (WNS) in Epoxy matrix. Composite material was developed using stir casting process at different weight percentages of Walnut Shell from 0-20. Result from tensile test reveal an improvement of tensile and compression modulus by 20 and 52 % respectively as compared to neat epoxy. However there was a decrease in the tensile and compressive strength. SEM micrograph illustrates that lower filler content result in uniform particle distribution and lesser void formation in the composite which increases with increasing WNS content. However, 1.84 % of water absorption was observed at 5% content which confirm strong encapsulation of filler material by the resin at lower filler wt. content.

## 1. Introduction

Over the past decades natural filler reinforced composite are gaining interest in researchers and scientist due to its intrinsic properties such as low cost, environment friendly, availability, low density and biodegradability [1]. The shells of Argan, Rubber, Groundnut, have already been reported in the literatures for the development of composite materials. The advantage of using natural filler as reinforcement in polymer matrix is that they don't affect the processing equipment as glass and carbon [2]. The major drawback of the natural filler is their hydrophilic nature which is due to the presence of cellulose, lignin, pectin, and hemicelluloses containing hydroxyl groups with high reactive functionality [3]. It results in poor interlocking of the biofiller with the hydrophobic matrix [4]. Moreover hydroxyl group present in the natural filler may react with the atmospheric water molecule and form hydrogen bonds which negatively affect the interfacial bonding of the composite [5]. Another problem encountered for the natural filler is the presence of wax/oil, hemicelluloses, pectin etc which serves as a major restraint in binding with the matrix. A number of strategies have been considered to improve the compatibility between the matrix and the natural filler which includes chemical modification of natural fillers using alkali treatment, saline treatment, benzylation and acetylation or through grafting and copolymerization of the matrix.

The intent of the current study is to synthesize composite material from Walnut Shell (WNS) and epoxy resin after pretreatment of the shells using solvent extraction and alkali treatment. The WNS particles are rigid in structure which when reinforced in the matrix improve its properties. The study further signifies the effect of the dual pretreatment on the mechanical and morphological properties of



WNS composite. The reason for incorporating WNS as reinforcement is to increase the performance, sustainability and functionality of the natural fillers and to improve the properties of the matrix.

## 2. Materials and methods

### 2.1. Materials

Walnut Shells were collected from the north-eastern region of India. The shells were grinded in the form of fine powder nearly 100 micron. Epoxy resin 4,4'-Isopropylidenediphenol, oligomeric reaction products with 1-chloro-2,3-epoxypropane (Lapox B-11) was used as matrix having viscosity at 25°C equal to 11000 – 15000 mPas with maximum moisture content of 0.10%. Hardener N, N'-Bis (2-aminoethyl) ethane-1, 2-diamine (Lapox K6) was used for curing of the resin with refractive index from 1.4940 - 1.5000 and maximum moisture content of 1%.

### 2.2. Methods

*2.2.1. Pretreatment of Walnut Shell.* The Shells were collected through manual extraction of Walnut Seeds. The shells were crushed into small pieces through ramming and subjected to solvent extraction. Methanol was used as a solvent. The process removes wax or oil content from the seed shells making them porous in structure. The extracted seed shells were then subjected to alkali treatment. The shells were dipped in 5% NaOH solution for a period of 3 h at 90°C and then thoroughly washed with water and 5% acetic acid solution to remove excess NaOH. The process removes pectin, hemicellulose and part of cellulose from the seed shells and makes their surface rough which improves adhesion between matrix and the seed shells.

*2.2.2. Fabrication of composite.* Composite was developed using stir casting process. The powdered seed shells were reinforced in the resin at weight percentages of 5, 10, 15 and 20. The solution was stirred for 1 h at 90°C to have uniform dispersion of particles in the matrix. Hardener was added in solution in the ratio of 1:10 with the resin. The solution was poured in the requisite moulds and dried in the oven at 90°C for 1 h.

## 3. Results and Discussion

### 3.1. Scanning Electron Microscopy

High resolution Scanning electron microscope of FEI make and Quanta 200F model was used to investigate the surface morphology of the walnut shell (WNS) composite as shown in figure 1. The samples were frozen in liquid nitrogen and cryofractured before being coated by a thin conductive layer of gold using gold spatter machine. A Cressington-108 auto (Model No.7006-8) sputtering machine was used to gold coat the samples at 230 V, 50 Hz using a high speed, direct drive 2-stage vacuum pump. The gold coated samples was analysed at 20 kV with a magnification range of 150-2000X. The composite at 5% wt. of WNS content as shown in figure 1(a) shows uniform dispersion of WNS particle in epoxy resin. This is because at lower filler content the particles are completely wetted by the resin. Thus smooth surfaces are observed at 5% wt. WNS. However at higher filler content around 20% wt. WNS as shown in figure 1(b) the particles are not properly wetted by the resin resulting in rough surfaces, void formations, cracks and pores in the composite.

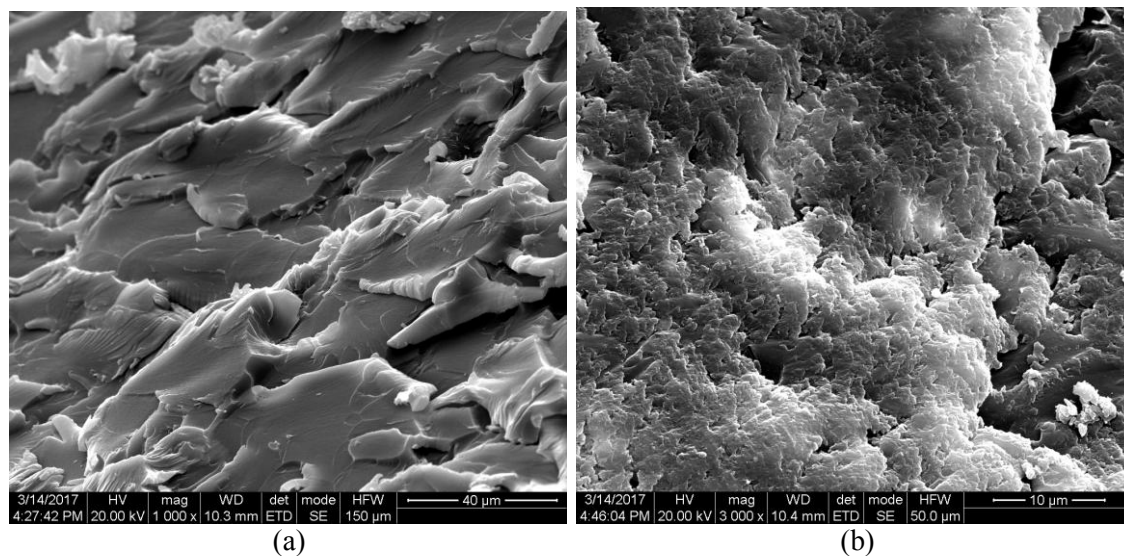


Figure 1. SEM micrographs of surface morphology at (a) 5% wt. WNS composite (b) 20% wt. WNS composite.

### 3.2. Mechanical testing

Mechanical testing was performed using both tensile and compression testing. Tensile test was performed using Instron 5569. Specimens were prepared following ASTM D-638 norms as shown in figure 2. The cross-head movement was 1 mm/min with 5 KN load cell. Figure 3 shows the tensile strength and Young's modulus of composite at different wt. percentages of WNS. The highest tensile strength of around 36 MPa for the composite was observed at 5% wt. WNS content. It is due to the lower amount of WNS filler which results in uniform mixing with the resin. The Young's modulus of the composite was observed to be highest of 1236.29 MPa at 5% wt. WNS. The incorporation of filler reduces the mobility of the polymer chains thus increasing the modulus of the composite. However the composite show highest elongation at break of 22.28 at 15% wt. WNS which further decreases with increasing filler content.

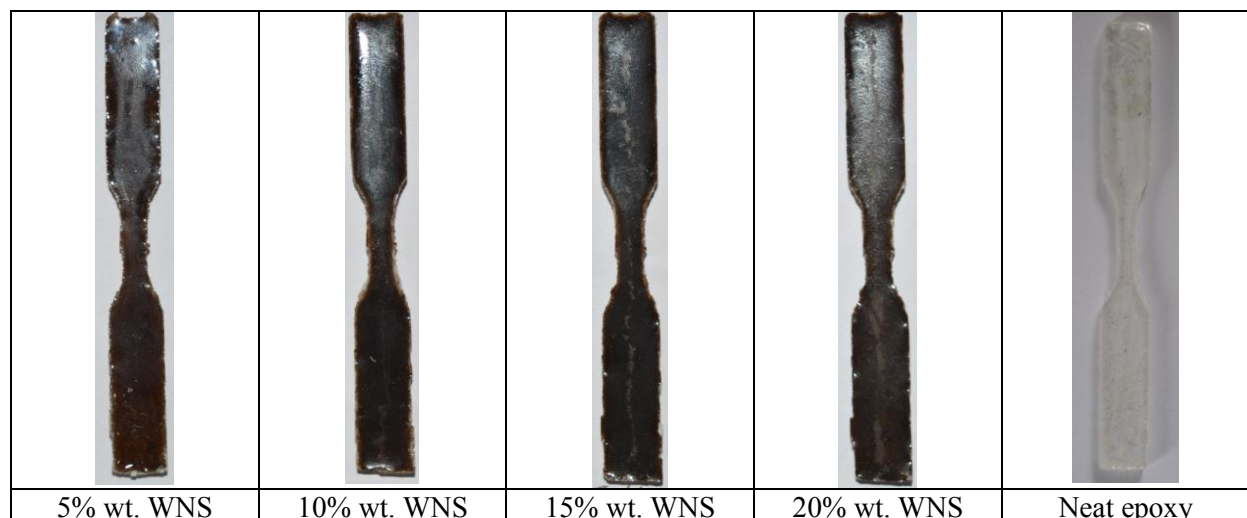


Figure 2. Tensile test specimens

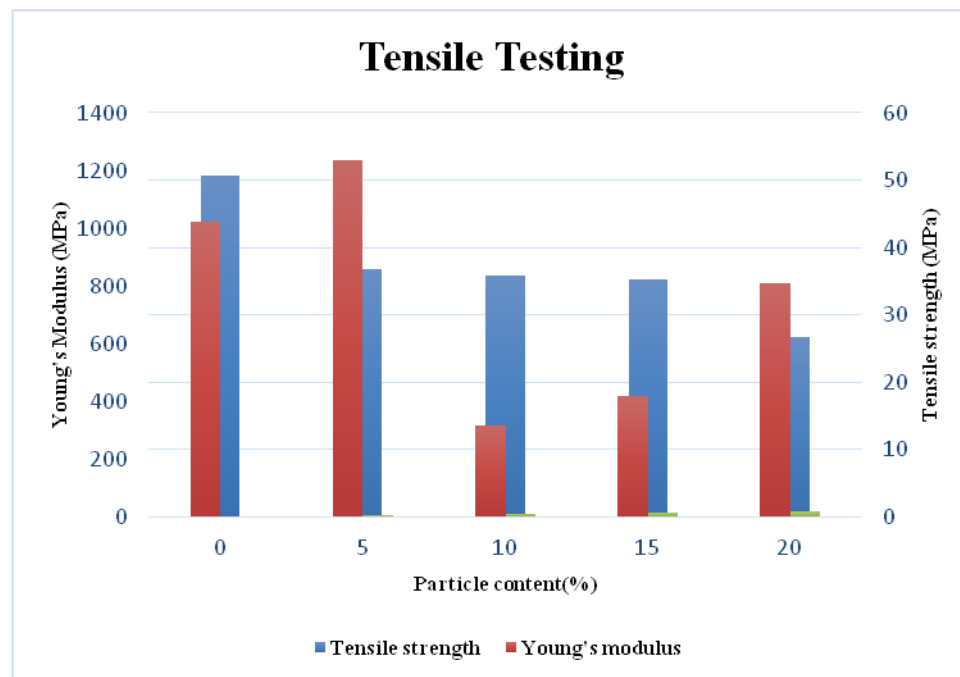


Figure 3. Tensile test at different weight percentages of WNS

Compression test was also performed using Instron 5569. Specimens were prepared following ASTM D-695 norms as shown in figure 4. The cross head movement was 1 mm/min. The composite shows highest compressive strength of 130.13 MPa and modulus of 1950 MPa at 10% wt. RSS content as shown in figure 5. It is due to the adequate mixing of the WNS filler in epoxy resin and introduction of rigid WNS particle which reduces the mobility of the polymeric chain thus improving the properties of the composite.

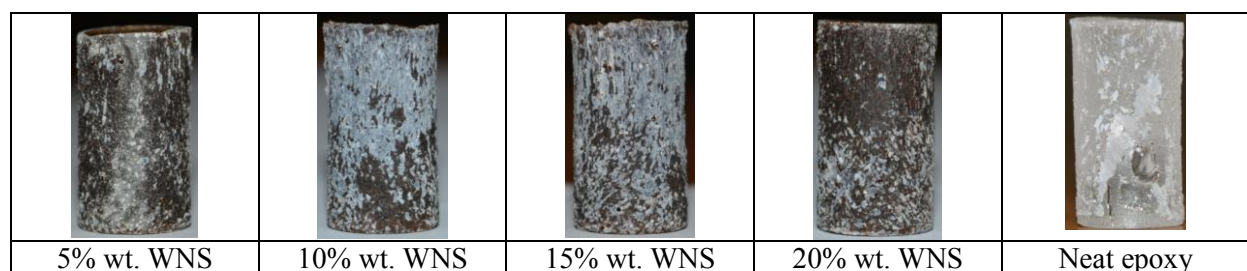


Figure 4. Compression test specimens

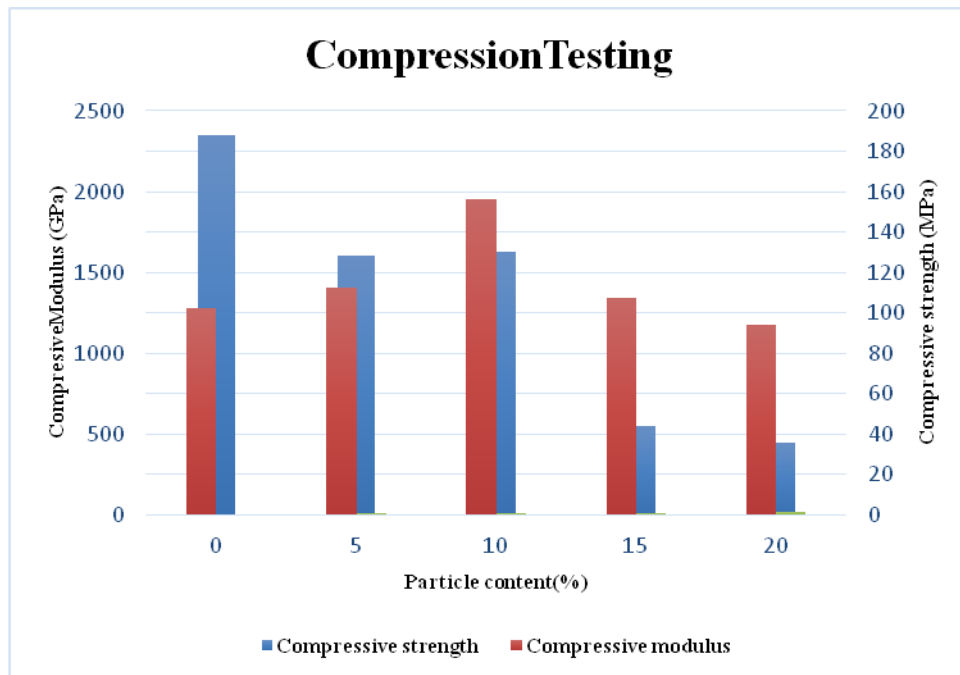


Figure 5. Compression test of samples at different weight percentages

### 3.3. Water absorption test

Water absorption test samples were prepared following ASTM D570-1995 standards having 20 mm length and 4.5 mm thickness as shown in figure 6. The samples were weighed to find the weight of dry samples. The samples were then immersed in distilled water for a period of 7 days. Thereafter the samples were taken out, dried using a tissue paper or piece of cloth and weighed. The percentage of water absorption was calculated as shown in equation 1.

$$\text{Water Absorption (\%)} = \left[ \frac{M_f - M_i}{M_i} \right] \times 100 \quad (1)$$

The result of water absorption test is shown in figure 7. The composite with 5 wt.% WNS content shows lowest water absorption of 1.84 % as compared to other composite samples. The percentage of water absorption is quite low which indicates good encapsulation of WNS particles by the resin reducing the over all hydrophilic nature of the composite. However the value increases with increasing filler content.

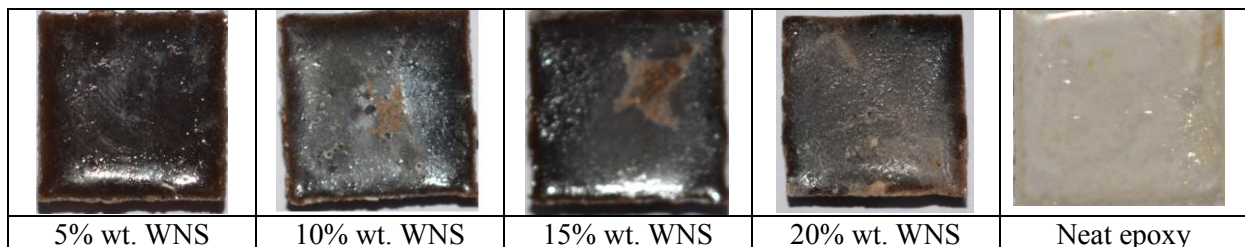


Figure 6. Water absorption test specimens



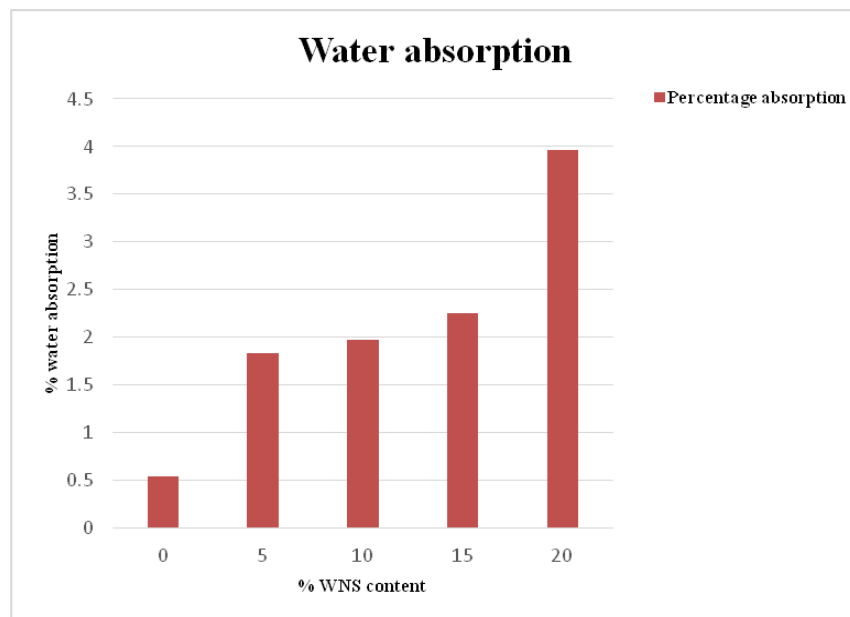


Figure 7. Water absorption test

#### 4. Conclusions

Walnut shell serves as a suitable reinforcement in the epoxy matrix. The maximum tensile strength of 36 MPa for the composite was observed at 5% wt. WNS content which further decreases with increasing filler content. It is due to the lower WNS content which results in uniform particle distribution in the matrix. The highest value of compressive strength of 130.13 MPa was observed at 10% wt. of filler content. However an increase of Young's modulus of the composite by 20% and compression modulus by 52 % from neat epoxy was observed with 10% wt. of filler content. SEM images confirm uniform particle distribution at lower filler content with no agglomeration of particles. But formation of voids and cracks are observed at higher WNS content of around 20%. The water absorption was found to be lowest for 5% wt. of WNS composite which increases with increasing WNS content. The developed composite may find possible applications in the replacement of conventional wood products and plies.

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