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Experimental Investigation of Date seed and Neem Powder Reinforced Natural Fiber Composites

Dinesh DHANABALAN [1] *, Rajasekar RATHANASAMY [2], Vijayakumar VEDIAPPAN [1], Nandana RAGHURAJ [1], Pavithra KATHIRESAN [1], Thirumurugan JEYAKUMAR [1], Prakashraj MUTHAIYA [1]

[1] *Bannari Amman Institute of Technology, Center for Research, Sathyamangalam, Erode-638401, Tamilnadu, India*
[2] *Kongu Engineering College, Department of Mechanical Engineering, Erode-638060, Tamilnadu, India*

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Natural fillers have received great attention among researchers due their its light weight, high strength, degradability, and eco-friendly behavior. This work is focused on developing a laminate of date palm seed powder and neem gum powder reinforced epoxy resin. Epoxy resin is an excellent thermoset polymer with anoutstanding cost to performance ratio. Date palm seeds have highly competitive properties in improving the mechanical and thermal properties of composites by their good adherence to polymer matrix. Neem gum powder is used as a binder in increasing ratios of 0 %, 10 %, 15 %, 20 %, 25 %, and 30 % to test their strength in different compositions. Date seed powder has been reinforced in decreasing weight percent as 30 %, 25 %, 20 %, 15 %, 10 %, 5 %, and 0 %. The specimens prepared through the compression moulding technique were subjected to various mechanical tests like a tensile test, flexural test, and impact test as per the ASTM standards. Fractography analysis using FESEM (Field Emission Scanning Electron Microscopy) and FT-IR(Fourier Transform Infrared spectroscopy) test have determined the surface characteristics and chemical composition of the samples. The result shows that, by adding neem gum powder in increasing quantities the mechanical properties like tensile and flexural decreases. However, it does not affect the impact strength of the material. A detailed study of their behavior is discussed in this work.

Keywords: dates seed powder, neem gum, epoxy resin, polymer filler.

1. INTRODUCTION

In recent years, researchers are focussing on replacing inorganic materials to reduce global issues like global warming, pollution, water crisis, and resources depletion. Green composites have set a trend in replacing inorganic substances by their excellent strength, degradability, and renewable characteristics [1, 2]. Industries are looking for materials that are less in weight and good in strength to improve the efficiency of their products [3, 4]. Natural materials such as polymer fillers are widely encouraged because of their high specific strength and low density. In recent decades, the usage of natural fillers in polymers has increased due to the increasing demand for greener and biodegradable materials that contribute to the satisfaction of society [5]. The need for natural fillers is rising because of their small density, high specific strength, low cost, light weight, recyclability, and biodegradability. The date palm (*Phoenix dactylifera L*) is primarily produced in the Middle East, especially in the United Arab Emirates. Date fruits (Phoenix dactylifera L.) are one of the oldest plants cultivated, from the earliest records of Predy-nastic Egypt. Cultivation of date palms in Egypt goes back thousands of years [6]. Although date seeds contain more nutritional content, it is being wasted in large quantities and are considered as a waste of biomass and they are hazardous when burnt [1]. Date seeds are economic fillers that solve the inadequacy of conventional materials. Date seeds have low density, are light in weight, and contain 73.4 % of carbohydrates, 6.3 % of protein, 13.5 % of fat, 11.5 % of fiber, and 1.8 % of ash [7]. Chemically neem gum contains mannose, glucosamine, xylose, galactose, fructose, arabinose and glucose [8]. Neem Gum is a by-product obtained as a result of certain metabolic mechanisms of neem trees. Furthermore, the neem tree (*Azadirachta indica)* plays a significant role in India because of its medicinal values and it is linked with the Indian way of life. It grows well at temperatures 0 to 49 °C [9]. The US Academy knows the neem tree and they recently declared the neem tree as the "Tree of the 21st century". Neem gum is obtained by induced or natural injury to the neem tree. Neem gum has a moisture content of 13.07 %, ash content of 3.13 %, and protein content of 32 % [10]. Neem gum is used as a binder to reinforce date seeds in the epoxy matrix. Epoxy resins are known for their high strength, good adhesion, affordability and are considered excellent thermoset polymers. They are easily available in the market as low viscosity liquids and even as powdered solids [11]. It possesses excellent mechanical properties and resistance to chemicals, and it can be modified within wide limits by using different hardeners as well as fillers. The substitution of natural fillers in epoxy resin has been shown to increase efficiency and save production costs with increasing recyclability and biodegradability [12]. This work emphasises studying the behavior of neem gum powder and date seed powder

* Corresponding author. Tel.: +91-9787771288.
E-mail: *dineshd@bitsathy.ac.in* (D.Dhanabalan)

reinforced epoxy laminates and investigating their mechanical properties, including tensile, flexural, and impact tests and their results were evaluated.

2. MATERIALS AND METHODS

2.1. Materials

The proposed composite consists of date seed powder, neem gum, and epoxy resin. They are processed upon some procedures and used to make polymer resin laminations. Dates seed powder is the natural filler, neem gum is used as a binder and AV138 Epoxy with hardener HV998 is used as a matrix in the composite.

2.2. Date seed preparation

Dates seed is subjected to various steps to use in the composite. Fresh date seeds are collected, and the surface is cleaned with distilled water. The washed seeds are dried in the air for 24 hours. Then they are heated in an electric muffle furnace at 120 °C for minutes. The dried seeds are crushed in the hammer mill and sieved up to ~ 0.5 µm which is measured using a zeta potential analyser [13].

2.3. Resin preparation

The utilized matrix is epoxy resin (Araldite LY556 catalyzed with hardener HY951). Neem gum is cooled down at atmospheric temperature. Then, the cooled resin is crushed in the powdered form and mixed with the epoxy as a binder. The particle size of neem gum is ~ 10 µm.

3. EXPERIMENTAL PROCEDURE

The compression molding technique was used to manufacture sheet materials. The materials were processed as follows for manufacture. The composite consists of Huntsman's araldite LY556 bisphenol F-type epoxy as a matrix. It is a medium-viscosity, unmodified epoxy resin-based bisphenol-A.

The preparation of laminates with various compositions of selected materials is discussed as follows. Firstly, the epoxy was taken in a common ratio of 70 % (26:1 resin:hardener ratio). Dates seed powder was added in a decreasing percentage with an increasing amount of neem gum. The filler was added as 30 %, 25 %, 20 %, 15 %, 10 %, 5 %, and 0 % to the increasing ratio of neem gum powder 0 %, 5 %, 10 %, 15 %, 20 %, 25 %, and 30 % respectively. The powdered date seed Fig. 1, neem gum Fig. 2 and the epoxy with hardener were mixed by stirring them for 3 minutes.

The mixture was poured into the mold and locked using the lid. The mold is fixed inside the compression molding machine Fig. 3 at a pressure of 21 bar for 8 hours. Then, the fixture is left for 24 hours and then removed as sheet laminates from the mold, and the specimens are prepared as per the specified ASTM standards [15].

If the reinforcement of dates seed powder goes beyond 30 %, the wettability of date seed powder in the polymer matrix decreases due to the non-availability of resin for binding. This decreases the strength of the laminates. So, we stopped the composition up to 30 % [16].

Fig. 1. Dates seed powder

Fig. 2. Neem gum powder

Fig. 3. Compression molding

Table 1. Details of material composition

Materials	S1, wt.%	S2, wt.%	S3, wt.%	S4, wt.%	S5 wt.%	S6 wt.%	S7 wt.%
Epoxy	70	70	70	70	70	70	70
Dates seed powder (DSP)	30	25	20	15	10	5	0
Neem gum powder (NG)	0	5	10	15	20	25	30

Table 1 shows the seven different matrix compositions of dates seed powder and neem gum ratios with reinforced epoxy resin.

3.1. Tensile test

The ASTM standard test method for determining the tensile properties of filler resin composites is ASTM D638 IV. The sample measures 115 mm in length and 19 mm in width. The tensile test is performed in a Universal Testing Machine model DTRX, with a force range up to 30 kN and max head travels 600mm manufactured by Poly Plast Pvt Ltd (INDIA).

3.2. FESEM analysis

ESEM is a type of electron microscopy used for the resolution of smaller objects. It helps to observe thin specimens, single polymer crystals, and the electron diffraction patterns of different surfaces. It produces images with a focused beam of electrons. It is usually carried out in temperatures below room temperature. The morphological characterization of the specimens has been determined using the fractured surfaces of tensile specimens.

3.3. FTIR Analysis

FTIR spectrum analysis was employed to characterize the raw materials and analyze the changes in the epoxy resin structure during its curing reaction, mainly to observe the introduction of new functional groups or disappearances of existing ones.

3.4. Impact test

As specified by the standards recommended in ASTM D256, the composite samples were made to evaluate the impact strength in the IZOD- CHARPY Impact machine Model D256, range of measurement upto 25 J manufactured by Poly Plast Pvt Ltd (INDIA). Before mounting on the machine, the samples were cut to length and height of 12.7 mm and 6.4 mm. The test sample is notched to a depth of 2 mm with a V-shaped hand file.

3.5. Flexural test

As specified by ASTM D790 standard the samples were cut and the test was performed on an Universal Testing Machine Model DTRX, force range up to 30 kN and max head travels 600 mm, manufactured by Poly Plast Pvt Ltd (India).

4. RESULTS AND DISCUSSION

4.1. Tensile test

From Fig. 4., it is observed that the maximum tensile strength is obtained in the case of S1 and then it starts to decrease up to S3. Usually, strength decreases as the DS powder content decreases, as the material cannot retain the applied stress to elongate. From S4 again the strength increases even when the DS powder reduces and neem gum increases, this results from good interfacial bonding of filler and matrix. But again, at S7 the strength decreases due to the non-availability of filler in the matrix. The fluctuation in the graph might be due to more formation of resin tags, voids and dimples that reduce the ductility due to the poor adhesive forces between resin and fillers. This type of fluctuation in the strength is seen in other neem constituents used in composites [17, 18].

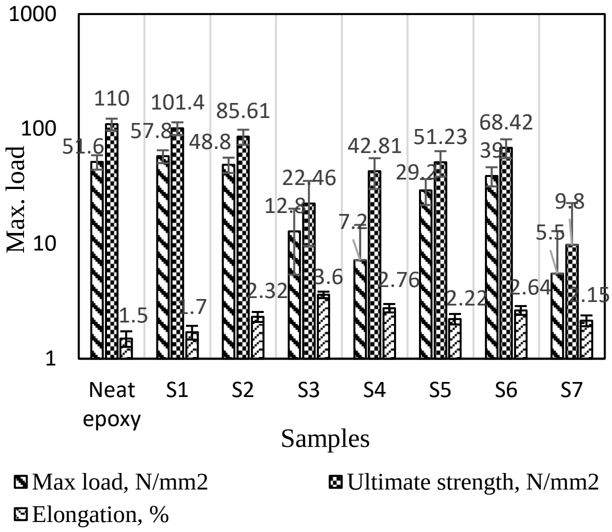
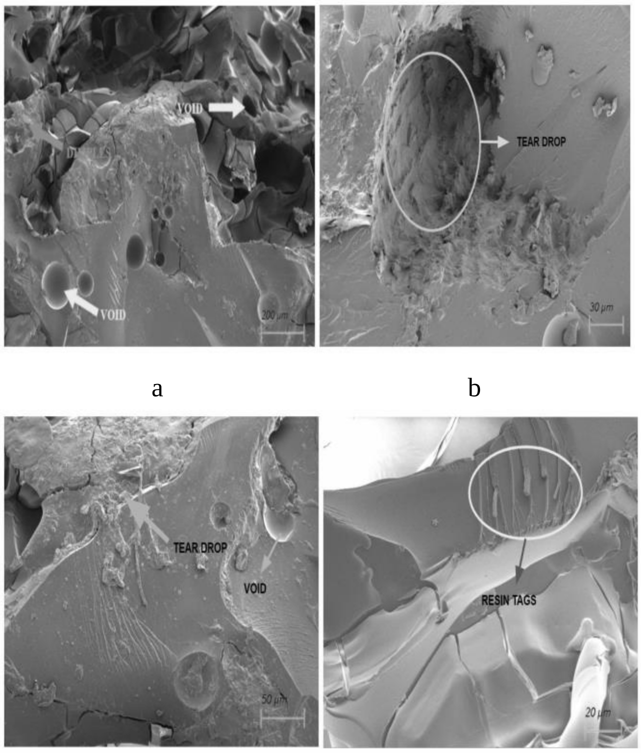


Fig. 4. Tensile test results of the various composite specimens

Neem gum binds in the pores of the material and increases the crushability by decreasing the elasticity of the material. S3 shows maximum elongation and S1 shows minimum elongation as a consequence of the decreased filler quantity of the laminate.

4.2. FESEM analysis

In Fig. 5 is the FESEM image of higher strength and lower strength tensile test samples. The fractography of the fractured tensile samples was analyzed by Scanning Electron Microscopy (SEM).



a b

c d

Fig. 5. Fractography: a, b – S1(DSP 30 % NG 0 %): c, d – S7(DSP 0 % NG 30 %)

The formation of voids and dimples developed the formation of cracks and caused the material to fracture. The river line pattern in Fig. 5 c indicates a brittle fracture of S7 and has low tensile strength. The average strength tensile of the tensile test is 35.92 n/mm^2. The strength of the laminates with an increase in dates seed powder. This is due to the good interfacial bonding between resin and filler.

4.3. FTIR analysis

The FTIR spectra for cured epoxy resin (Green line), Neem gum(Red line), Date seed powder (Blue line), neem gum-date seed powder-epoxy resin(black line) blends (0.7:0.15:0.15) are shown in Fig. 6.

In the case of FTIR of neem gum, the absorption bands were observed at 3350 cm^{-1} (due to –OH stretching vibrations in the neem gum polysaccharide), at 2931 cm^{-1} (-C-H stretching mode of -CH_3 groups in the neem gum), at 2349 cm^{-1} (due to overtones of -C-O stretching vibrations), at 1738 cm^{-1} and 1627 cm^{-1} (due to C=O stretching vibrations of carboxylic acid and amide present in the neem gum).

The absorption peaks at 1421 cm^{-1} (due to C-H deformation of methylene groups in the neem gum), and at 1248 cm^{-1} (due to C-O-C asymmetric stretching vibrations) were observed [21]. In case of FTIR of date seed powder the absorption bands were observed at 3450 cm^{-1} (due to -OH stretching vibrations in the date seed powder polysaccharide), at 2920 cm^{-1} and at 2890 cm^{-1} (-C-H asymmetric and symmetric stretching mode of -CH_3 groups in the date seed powder respectively), at 2349 cm^{-1} (due to overtones of -C-O stretching vibrations), at 1761 cm^{-1} and 1629 cm^{-1} (due to C=O stretching vibrations of carboxylic acid and amide present in the date seed powder). The absorption peaks at 1418 cm^{-1} (due to C-H deformation of methylene groups in the date seed powder), and at 1248 cm^{-1} (due to C-O-C asymmetric stretching vibrations) were observed.

After mixing epoxy resin with neem gum-date seed powder, the characteristic stretching vibration of the epoxy group around 915 cm^{-1} disappeared(black line) [18]. FTIR spectral analysis indicates that epoxy resin can be cured by the reinforcements and there may be a chemical bond between active groups within the neem gum, date seed powder and epoxy or hydroxyl groups during the curing process. This provided the possibility to form a good interfacial combination between neem gum-date seed powder and epoxy resin during the preparation of blends, which is beneficial to the properties of the resultant laminates.

4.4. Impact test

From Fig. 7. we can identify that the impact strength of the S1 and S2 are the same and decrease at S3 and then again S4 (DSP 15 % and NG 15 %) and show the same impact strength as 1 and 4 and finally S6 and S7 decrease. Samples S1, S2, S4 and S5 show high impact strength because of the high DSP content and good interfacial bonding of resin and filler. S6 and S7 show low strength because of the low quantity of date seed, so the material cannot withstand the sudden load and fractures. S3 shows low impact strength because of lower adhesion and increased crushability of the material.

4.5. Flexural test

The results of the flexural test are shown in Fig. 8. It is observed that the highest flexural strength is marked in the S1 to S2 decreases from S3 due to the incompatibility of the date seed powder and neem gum powder in the matrix as seen in the reinforcement with polyurethane. Moreover, the strength again increases in S5 and again starts decreasing. Due to the hydrophilicity of the reinforced filler and hydrophobicity of the added neem gum. Hydroxyl groups during the curing process.

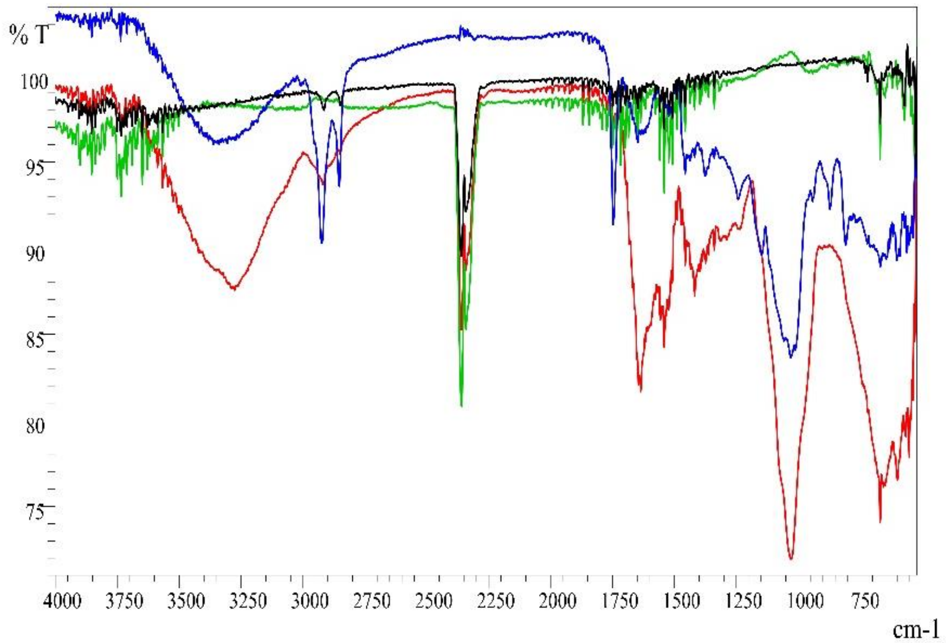


Fig. 6. FTIR spectrum

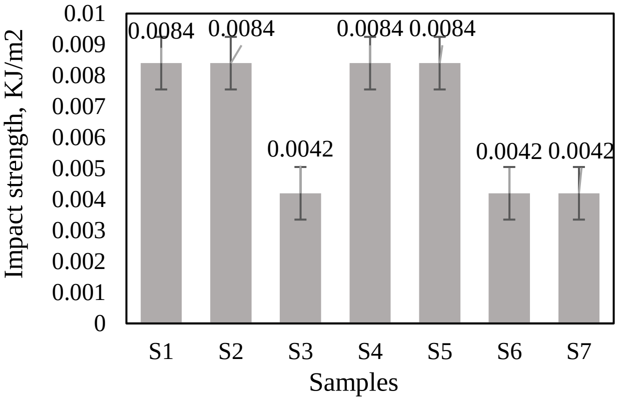


Fig. 7. Impact test results of the specimens

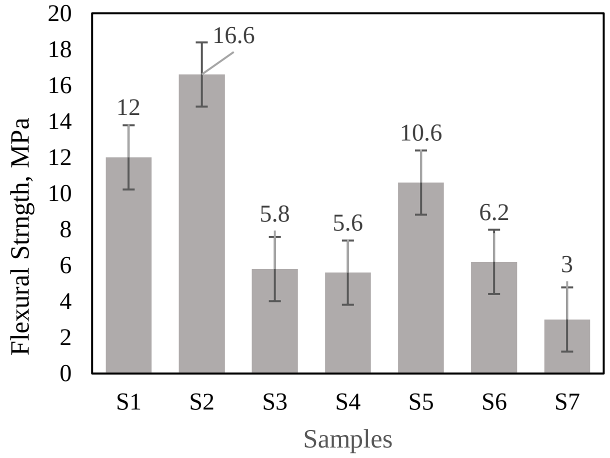


Fig. 8. Flexural strength results of varous composite laminates

5. CONCLUSIONS

A polymer composite formed by dates seed powder and neem gum powder reinforced in epoxy resin was successfully fabricated and tested with the following conclusions.

1. Epoxy matrix reinforced with Date seed powder and neem gum powder marks good tensile strength in S1 due to good binding of filler and matrix.
2. The impact strength was moreover same for all the compositions of different ratios but the highest impact strength was attained in S1, S2, S4 and S5 because of the good adherence of filler in the matrix.
3. Flexural strength increases with a decrease in neem gum and marks good strength at S2 than other samples.

Analyzing the results, S2 acquired optimal strength and better mechanical properties than the other samples such as 101.4 Kg/cm^2 ultimate tensile strength and 0.0084 kJ/m^2 impact strength. The strength of the composites decreases in the specimens that contain more than 5 % neem gum. So, 5 % NG is the optimum amount that we can add to attain good strength.

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