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Kumar, Anil
G. B. Pant DSEU Okhla-I Campus

Jayant P. Supale
G. B. Pant DSEU Okhla-I Campus

Goyal, Kavita
U.I.E.T, MDU

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Fabrication and Material Characterization of Composite Fiberboard made by Walnut's Waste

Anil Kumar[1,*], Jayant P. Supale[1], Kavita Goyal[2]
[1]G. B. Pant DSEU Okhla-I Campus, New Delhi, 110020, India
[2]U.I.E.T, MDU, Rohtak, Haryana, 124001, India

*Author to whom correspondence should be addressed:
E-mail: anilapmae@gmail.com

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Abstract: In this study, a novel approach has been presented for manufacturing composite fiberboard by incorporating waste walnut shell powder particles into epoxy resins. The aim of this research is to evaluate the mechanical properties and micro-structural characteristics of the resultant composite plate. The composite fiberboard specimens were prepared and various tests were carried out to assess the composite's strength, stiffness and energy absorption capabilities. Furthermore, Scanning Electron Microscope (SEM) analysis was performed to study the microstructure of the composite and for understanding of the composite's morphology and the distribution of filler particles within the resin. The experimental outcomes demonstrated that the composite exhibited promising mechanical properties Moreover, the SEM analysis revealed some voids formation between the waste walnut shell particles and the epoxy matrix, contributing to reduced mechanical performance.

Keywords: Composite Fiberboard; Walnut Shell Powder; Mechanical Properties; SEM; Sustainable Materials

1. Introduction

Agriculture has been the backbone of human civilization, providing sustenance and livelihood for millennia. However, with the growth of modern agricultural practices and the increasing demands of a burgeoning global population, agricultural waste has emerged as a significant concern. The inefficient management and disposal of agricultural residues, by-products, and discarded materials have adverse implications for both the environment and society. The agriculture industry generates vast quantities of waste throughout its production cycle. Crop residues such as stalks, husks, and straw, along with fruit and vegetable peels, contribute to the mounting waste streams. Additionally, livestock farming produces substantial amounts of animal manure, posing challenges for proper waste disposal. Traditional methods of waste management, such as open burning or dumping, release harmful pollutants into the atmosphere, soil, and water bodies, leading to air pollution, soil degradation, and water contamination. Furthermore, the unchecked decomposition of organic waste releases greenhouse gases like methane, exacerbating climate change.

Moreover, the mismanagement of agriculture waste represents a significant loss of potential resources. Valuable nutrients and organic matter contained in agricultural residues could be recycled to enhance soil fertility and support sustainable agriculture. The potential for energy generation through waste-to-energy technologies remains largely untapped, hindering efforts to transition towards cleaner and renewable energy sources.

The use of natural fiber composites is progressively being considered as a viable alternative to synthetic composites[1,2)]. Natural fibers are becoming more widely used in the composites industry due to their cost-effectiveness, lightweight nature, excellent durability, biodegradability and favorable properties[3,4,29)]. Amidst growing environmental concerns and the growing global demand for energy, there is a growing focus on incorporating natural fibers into various industries[5)]. The engineering industries prefer natural fibers over synthetic fibers due to the negative environmental consequences associated with the production and disposal of synthetic fibers. Natural composites outperform manufactured composites in terms of their positive environmental influence[6-8)]. Natural fibers lead to reduced dependency on nonrenewable resources, decreased greenhouse gas emissions, and enhanced energy regeneration[9)]. The growing environmental benefits play a crucial role in driving the future adoption of "Natural Fiber Composites." With readily available options like Bagasse, Pineapple, Ramie, Coir, Sisal, Bamboo, Banana, and others, countries are actively investing in the development of "Natural Fiber Composites" to generate value-added employment opportunities[10]. Studying polymer composites with natural fibers is a significant and relevant area of research, as it has the potential to result in favourable mechanical properties[11-13]. Furthermore, composites made from natural fibers are environmentally friendly and can be recycled, making them highly suitable for recovery and future application[14-15].

The incorporation of fillers has been shown to significantly improve the wear resistance and mechanical properties of E-glass fiber[16]. Studies have compared different fillers like coconut shell, rice husk, and teakwood in lignocellulosic flour/epoxy composites, revealing that higher filler content enhances the stiffness of the composite, while the volume of fillers affects its elasticity[17]. Additionally, agricultural wastes such as bagasse, walnut shell powder, and chicken feathers have been utilized to develop composites using epoxy or polyester[18-20].

Globally, around 1.2 billion tons of food is wasted annually throughout the food life cycle, leading to increasing food waste with population growth. The current disposal method, primarily through landfill dumping, has created environmental hazards and contributed to climate change. To address these challenges, there is a need to reevaluate production and consumption models, including household waste management. This involves adopting innovative waste processing methods, such as utilizing walnut shells to obtain new materials and converting waste into fuel and energy using physical, chemical and biological means. Some countries, like Sweden, have achieved remarkable success in waste management, with a small fraction of household waste sent to landfills[21-24].

The production of bio-plastics from orange waste offers exciting possibilities for creating innovative materials that can replace traditional plastics. These bio-plastics serve as environmentally friendly biomaterials, providing an excellent solution to fruit waste disposal and effective utilization[25]. The incorporation of appropriate waste material particles in epoxy composites led to significant improvements in stiffness and hardness. This enhancement was primarily due to the reinforcing effect of the natural fillers in the composites[26, 27]. The exploration and advancement of composite materials derived from waste agricultural materials or fruit peels represent a promising and essential domain in the realm of sustainable materials. These materials have gained significant significance due to their various benefits, including lightweight nature and excellent corrosion resistance[28].

The effective utilization of agriculture waste presents immense opportunities for addressing both environmental and economic challenges. By transitioning from a linear model of waste disposal to a circular economy approach, agriculture waste can be regarded as a valuable resource with multiple applications.

Like other agricultural wastes, Walnut shells and walnut shell powder have various practical applications due to their unique properties. For Example: Crushed walnut shells serve as a natural and biodegradable abrasive media. They are used in industries like metal finishing, aerospace, and automotive for polishing and cleaning delicate surfaces. The hardness and particle size of walnut shell powder make it suitable for gentle but effective exfoliation in personal care products like facial scrubs and body scrubs. Walnut shell powder can be processed into granules and used as a filtering media for water treatment and air filtration applications. It has excellent adsorption properties and can remove impurities and contaminants from liquids and gases. Walnut shells can be used as biomass for energy generation through combustion or gasification. This process can produce heat or electricity, offering a renewable energy source.

The versatility of walnut shells and their powder as a sustainable and renewable resource opens up a wide range of possibilities for their utilization across various industries, contributing to waste reduction and environmental conservation. This research deals with fabrication of composite fiberboard using a waste material which will leads to a greener environment and helps in reducing the consumption of wood. Also very less data is available on the mechanical properties of composite fiberboard made up of walnut shell powder, which gives us an opportunity to make a research on it leading it to a novel research work. So the various objectives of this research are-

1. To fabricate a composite fiberboard using waste walnut shell powder and epoxy resin.
2. To perform various tests to identify the mechanical properties of the fabricated composite fiberboard.
3. To make SEM analysis to analysis the composite's morphology.

For attaining the aforesaid objectives, the composite fiberboard specimens will be prepared using waste walnut shell powder and epoxy resins and compressive, tensile, flexural, hardness and impact tests will be carried out to assess the composite's strength, stiffness and energy absorption capabilities. Furthermore, Scanning Electron Microscope (SEM) analysis will also be performed for analysis of the composite's morphology and the distribution of filler particles within the resin.

2. Materials and Methods

2.1 Fabrication of composite

The following materials and tools are required for the fabrication of composite fiberboard:

- Walnut powder: To prepare walnut shell powder from waste walnut shells, walnut shells are collected. Then, the shells are dried by preheating in an oven or kiln to 150-200 °C. After this, the dried walnut shells are cooled at room temperature. For the grinding process a grinder is used to crush the shells into a fine powder, making adjustments for the desired particle size. Multiple passes through the grinder may be necessary for fine powder consistency. Following grinding, a sieve was used to separate larger or coarser particles from the walnut shell powder and then the finer powder is collected in a container. Then this fine powder is stored is an airtight container so that it may be prevented from moisture absorption, ensuring it to remain dry for future use.
- Epoxy resin (Novolac): A two-part epoxy resin, consisting of the resin and hardener.
- Fiber reinforcement: Walnut shell powder. Mold: A mold in the desired shape and size for the fiberboard.
- Mixing containers: Containers for mixing epoxy and fibers.
- Stirring stick: A stick for stirring the epoxy and fibers mixture.
- Release medium: A release medium to avoid the sticking of fiberboard to the mold.
- Clamps: Clamps to hold the mold closed during the curing process.
- Safety equipment: Gloves, safety goggles, and a well-ventilated workspace.

Fig. 1: Walnut Shells and their Powder

Walnut shell powder as shown in Figure 1 was converted into a composite fiberboard as shown in Figure 2 using the casting method.

Novolac resin epoxy was chosen for its excellent resistance to chemicals and high-temperature stability. The required amount of epoxy resin and hardener has been measured according to the manufacturer's instructions and epoxy resin has been poured into a mixing container. After that hardener has been added to the resin (10:1 ratio of Epoxy and Hardener) and both are mixed thoroughly using a stirring stick.

Then gradually walnut powder has been added to the epoxy mixture with continuous stirring to ensure even dispersion of fibers. The amount of walnut powder can be taken up to 40% to 70% by weight of the composite. Using a substantial quantity of filler led to lower strength values compared to high-performance composites. However, by decreasing the filler content from 70% to 60%, the mechanical properties improved. The composite panel with a ratio of 60% groundnut shell particles to 40% epoxy resin demonstrated the highest tensile, bending, and impact strengths [26]. Here it has been taken as 60% by weight. Then release agent (mold release wax) has been applied to the inside of the mold to prevent the composite from sticking to it during the curing process. Then the mixed epoxy, walnut powder and fiber mixture has been poured into the prepared mold and a spatula or similar tool has been employed to evenly spread the mixture and to eliminate any air bubbles. Then the mould has been closed using clamps to maintain pressure during the curing process. After, that the mold has been shifted to a warm and dry environment and it has been left for 24 hours for curing.

After completion of the curing time clamps have been removed, mold has been opened and the composite fiberboard has been gently demolded. Then excess material and uneven edges have been trimmed from the fiberboard using appropriate tools. Then the surface of the composite fiberboard has been rubbed using sand to achieve a smooth finish. After that the finished composite fiberboard has been checked to inspect any defects or imperfections.

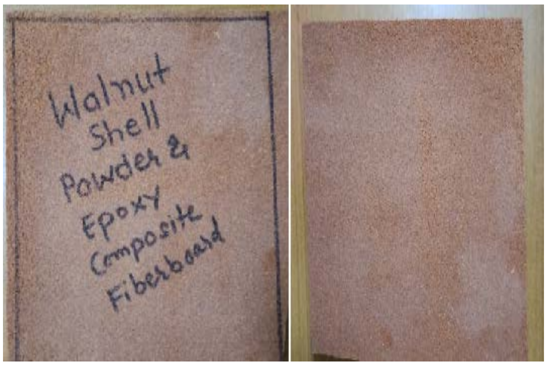

Fig 2: Finally Fabricated Composite Panel

3. Results and Discussion

3.1 Mechanical Characterization

a. Tensile Strength

The tensile test was conducted using a universal testing machine as shown in Figure 3, in accordance with the ISO 527-2-2012 standard. The Width of the parallel section is 4 mm, Thickness of the parallel section is 1 mm, Gauge length is 25 mm and the Length of reduced section is 10 mm. Three specimens for the tensile test were prepared from a composite fiberboard. The test was performed at a constant crosshead speed, as specified in the standard, to apply a uniaxial tensile force to the specimen.

The tensile test results revealed that the walnut shell powder, epoxy composite fiberboard had an average tensile strength of 12.4 MPa. This value represents the maximum tensile stress the composite could withstand before experiencing failure due to the applied load.

Fig 3: Tensile Test Specimen and Test Setup

The figure 4 shows the behavior of specimen under tensile test. However, the failure analysis indicated that the improper mixing of larger size walnut shell particles and epoxy resin contributed to the premature failure of the composite during the test. Upon failure, it was observed that the composite specimen exhibited improper mixing of higher content walnut shell particles with the epoxy resin. This non-uniform dispersion led to stress concentration points and weak bonding interfaces between the particles and the matrix, resulting in failure during the tensile test.

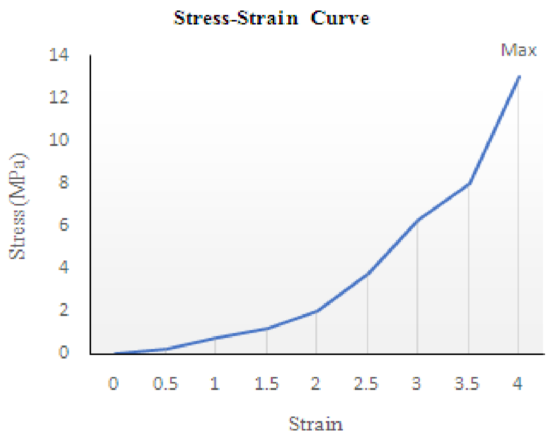

Fig 4: Stress Stain curve (Tensile test)

b. Flexural Strength

The flexural strength test was performed using a universal testing machine following the ASTM D-790-2015 standard for flexural properties of unreinforced and reinforced plastics. Three Rectangular bar-shaped specimens having Width 12.7 mm, Thickness 3.18 mm and Span Length of 50.8 mm were cut and placed on the supports one by one to perform the tests on the universal testing machine, with the loading nose centered on the midpoint of the specimen's length, as shown in Figure 5.

The test was conducted at a specified loading rate as per the ASTM standard, applying a three-point bending force to the specimen. The flexural strength test results showed that the composite fiberboard had an average flexural strength of 19.1 MPa. This value represents the maximum stress the specimen could withstand before failing under the three-point bending load.

Fig 5: Flexural Test Specimen and Test Set-up

The figure 6 shows the behavior of specimen under Three Point Bend Test test. Upon failure, it was observed that the presence of voids in the fiberboard contributed to reduced mechanical integrity and increased susceptibility to failure. The voids resulted in inadequate load distribution and decreased overall flexural strength. However, the failure analysis indicated that the presence of voids in the fiberboard contributed to its failure under the three-point bending load. To improve the flexural strength and mechanical performance of the composite, it is recommended to reduce voids and enhance the bonding between the walnut shell particles and the polymer matrix.

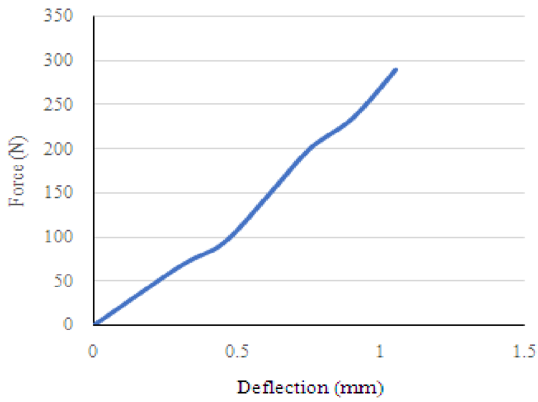

Fig 6: Flexural Strength Test Curve

c. Compressive Strength

The compressive strength test was conducted using a universal testing machine, following the ISO: 604-2002 standard for determination of compressive properties of the composite fiberboard specimen was carefully cut and shaped and positioned vertically in the universal testing machine, ensuring proper alignment and support during the test as shown in Figure 7. The test was performed at a specified loading rate as per the ISO standard, subjecting the specimen to a compressive force. The compressive strength test yielded a compressive strength value of 46.4 MPa.

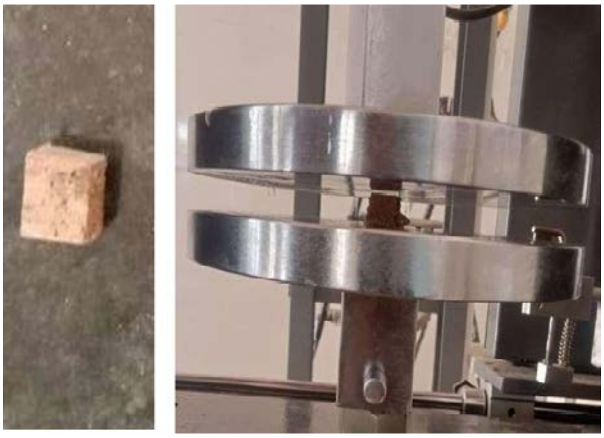

Fig 7: Compressive Strength Test specimen and Setup

The figure 8 shows the behavior of specimen under compressive Strength test. Upon failure, it was observed that the inadequate bonding between the walnut shell particles and epoxy resin contributed to reduced load transfer capacity, ultimately leading to failure during the compressive strength test. However, the failure analysis indicated that the improper mixing of walnut shell particles was the main reasons behind the failure of the specimen under compression. To enhance the compressive strength and structural performance of the composite, it is crucial to improve the mixing process of the constituent material.

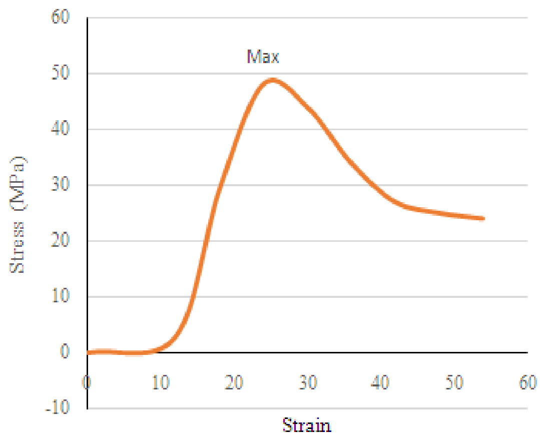

Fig 8: Compressive Strength Test Curve

d. Impact Strength

The Charpy Impact Strength Test was performed using a Charpy Impact Testing Machine, following the ISO: 179-1-2010 standard for determining the impact strength of plastics. Three composite fiberboard specimens were carefully cut, each having length 80 mm and width 10 mm and a V-shaped notch was precisely made at the centre of the each specimen, using a specified notching tool and then the notched specimen were securely placed on the supports of the Charpy Impact Testing Machine one by one, ensuring proper alignment with the pendulum hammer, as shown in Figure 9.

The pendulum hammer was released, striking the notched specimen, causing it to break. The machine recorded the energy absorbed by the specimen during the impact. The Charpy Impact Strength Test revealed average impact strength of 1.32 KJ/m^2 (Notched) for the composite fiberboard. This value represents the energy absorbed by the specimen during the impact test. Upon failure, it was observed that the voids formed during the fabrication process, affects the structural integrity and impact resistance of the composite.

Fig 9: Charpy Impact Test Specimen and Test Setup

However, the failure analysis revealed that the larger walnut shell particles compared to epoxy resin, along with the presence of voids formed during fabrication, were the primary reasons behind the failure of the specimen during the impact test. To improve the impact strength, it is essential to optimize the particle distribution and minimize void formation during the fabrication process.

e. Hardness Test

The Rockwell hardness test was performed on the composite fiberboard according to ASTM D785 on the specimen. The test yielded a hardness value of 90R, which was then converted to Shore D for comparison and analysis. The composite fiberboard exhibited a normal range of hardness. Upon further investigation and analysis, it was observed that the reason for the low hardness of the composite fiberboard is its poor surface finish and the formation of voids.

It's important to note that hardness is just one mechanical property of a material and does not necessarily define its overall quality or suitability for a particular application. The desired hardness level depends on the intended use and requirements of the composite fiberboard. The composite fiberboard demonstrated a typical level of hardness within the normal range. Upon closer examination, it was discovered that the composite fiberboard's low hardness can be attributed to its inadequate surface finish and the presence of voids.

The different mechanical properties observed after testing are shown below in table 1–

Table 1- Results for different tests

S. N.	Test	Observations
1	Tensile Strength	12.4 MPa
2	Flexural Strength	19.1 MPa
3	Compressive Strength	46.4 MPa
4	Impact Strength	1.32 KJ/m^2
5	Hardness	90R

3.2 SEM ANALYSIS

The surface morphology of the composite was examined using a high-resolution Scanning Electron Microscope (SEM) of FEI make, specifically the Quanta 200F model, as depicted in Figure 10. Prior to analysis, the samples were cryofractured by freezing them in liquid nitrogen. A thin conductive layer of gold was then applied to the cryofractured samples using a gold spatter machine. The gold-coated samples were subsequently analyzed at 20 kV with a magnification range of 400X.

The SEM image (Figure 7) reveals critical information about the composite. In the composite containing 60% wt. of walnut shell powder particles, it was observed that the particles were not adequately wetted by the resin. This suggests that the epoxy resin did not completely encapsulate or adhere to the walnut shell particles. The inadequate wetting of the walnut shell particles by the resin resulted in the presence of rough surfaces in the composite. These rough surfaces indicate that there was a lack of a uniform resin coating, leading to an uneven and textured appearance.

The SEM analysis showed the presence of void formations and pores within the composite. These voids are undesirable in composite materials as they can weaken the structure and reduce its mechanical properties. In this case, the voids were primarily distributed within the polymeric matrix, indicating that they were located within the resin.

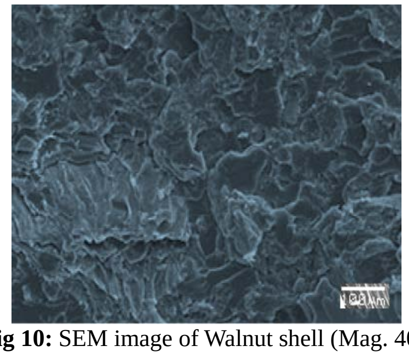

Fig 10: SEM image of Walnut shell (Mag. 400X)

4. Conclusion

This study presents the method for manufacturing composite fiberboard by incorporating waste walnut shell powder particles into epoxy resins, aiming to assess mechanical properties and micro-structural characteristics. Comprehensive testing, including tensile, flexural, compressive, hardness and impact tests, revealed promising results: a tensile strength of 12.4 MPa, flexural strength of 19.1 MPa, compressive strength of 46.4 MPa, Charpy impact strength of 1.32 KJ/m^2 (notched), and a hardness value of 90R (converted to Shore D). However, it was observed that the composite with 60% wt. of walnut shell powder particles exhibited issues related to inadequate resin-wetting, resulting in rough surfaces and void formations primarily within the polymeric matrix, likely due to hindered degassing during manufacturing. Overall, the research underscores the potential of walnut shell powder in creating composite fiberboard while efficiently utilizing agricultural waste in an environmentally friendly manner.

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Nomenclature

SEM	Scanning Electron Microscope
MPa	Mega Pascal

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