Influence of Pretreatment on Mechanical properties of Bio waste Eggshell Composite

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ABSTRACT

Manufacturing and evolution of complimentary operated engineering materials from unwanted substances has gained worldwide engrossment. The materials made from bio waste can able to get over the synthetic material due to their isolated properties like in expensive, non-toxic, nonabrasive, biodegradable, pollution emission free and ecofriendly. The Calcium Carbonate rich egg Shell causes serious environment hazards. Effort has been made to incorporate these bio waste egg shell materials into the matrix which form composites to boost up the mechanical properties. So the objective of this study is to make composites which should have a good compressive strength as well as low density. Most of the researchers emphasizes towards the making of light weight material which are significant to automobile, aerospace, electronics industries. Now special care has been increased gradually in the composite industries to make it greater potentiality of application in the field of medicines, chemical industries, and marine and also for household things. Keeping these requirements into mind, the present investigation is mainly focused on the mechanical properties of egg shell reinforced composites. Chicken egg shell were collected, washed with deionized water to remove the inner layer membrane, then dried and powdered. X-ray diffractometry (XRD) and Field Emission Scanning Electron Microscopy (FESEM) were used to characterize the crystalline size, constituent compounds and surface morphology respectively. The internal adhesion between the surface of reinforcement and matrix (Lapox) was enhanced by doing the chemical treatment of egg cell with NaOH solution. The composites were made by hand layout technique. Three sets of modified (NaOH treated) and unmodified (Raw) samples were prepared for the flexural tests. Result shows the enhancement of mechanical properties with increase in NaOH concentration.

Keywords: Chicken egg shell, NaOH solution, Lapox, Flexural strength

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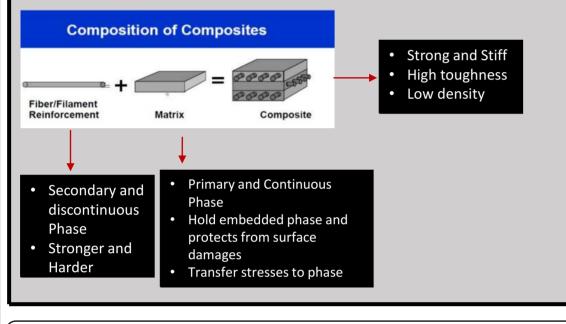


Objectives

- **Transformation of Bio-waste Eggshell into** potential use.
- **Conversion of Hydrophilic Eggshell into** Hydrophobic to make compatible with polymer.
- **Enhancement in Mechanical Properties of Eggshell Composite.**

Introduction

- Egg shell (ES) is known to be a nutritional powerhouse packed with protein, vitamins and minerals in both its yolk and white.
- Rich in Calcium and produce a purified or stable form of Calcite
- An aviculture byproduct and the worldwide consumption on industrial and domestic level leads to considerable quantity of shell residue which is considered as waste.
- Belong to fillers which play a major role in polymer industry. They are used predominantly to cheapen end products.
- Incorporation of fillers into polymer makes a potential source of light weight, high strength and stiffness biodegradable, ecofriendly composite applications.



Why Egg Shells(ES)

- It is a bio waste agricultural product.
- Calcium rich ES protects our bones.
- Alleviate joint pains. Garden fertilizer.
- **Treat skin irritation**
- Composite build out of ES has a high compressive strength.
- ES reinforced composites are biodegradable, so don't cause any environmental issues.

Experimental Procedure / Model

(1) Hen egg shells were collected from hostels.

• (2) The ES comprise with both shells and membrane. The collected ES impurities on its surface. So to remove the impurities and get purified Calcite, the membrane should be separated out of ES. For this the collected ES were dipped in normal water for 48 hours at room temperature. The floatation of membrane over water was observed with placing the heavier shells in the bottom of water. Finally the shells were collected and let them dried for 24 hours at room temperature.

Treatment of ES Powder

- · (3) For developing of good adhesion between reinforcement and matrix, the surface of ES were modified with aqueous solution of NaOH with different concentration (5%,10% AND 15%) and let them soaked for 24 hrs at room temperature. Finally the ES were rinsed with DI water various times for removal of excess amount of NaOH and let them dried for 12 hrs in Vacuum Oven at temp. of 60 degree.
- (4), (5) Treated ES were crushed mechanically with the help of mortar and pestle and sieved them to the particle size of 150µm.
- (6) The Untreated and Treated ES were collected and made them ready for further characterization.

Processing of ES out of ES wastes

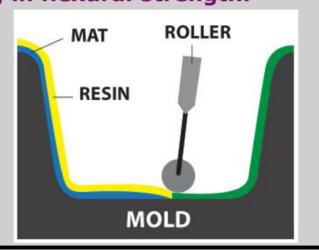


Purpose of Treatment

- · Enhance the interfacial bonding between filler-matrix.
- Decrease of hydrophilicity nature of filler material to make a better adhesion with hydrophobic polymers.

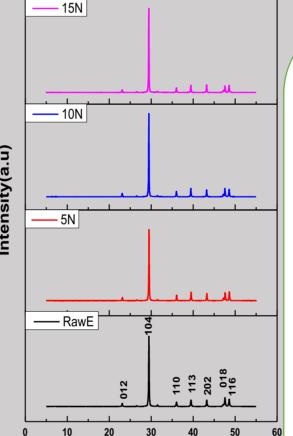
Composite Fabrication

- Hand Lay-up process was adopted.
- The thermosetting Epoxy (L-12) resin used as matrix and Hardener (K-6) used as a catalyst.
- Initially, the resin was mixed with filler and placed under ultrasonicator probe to make the solution slurry.
- Then the Hardener was mixed to accelerate the whole process.
- A mould of (100x70x5)mm3 was prepared using glass slides on a plastic sheet and a heavy silicon spray was used inside the mould for easy removal of composites.
- The solution was poured into the mould and a roller used to press roll over the surface for removal of air bubbles properly and the moulded of composite was hold for 24 hrs.
- The 4 sets of composites were cut according to ASTM standard for testing in flexural strength.



Characterization Techniques

X-Ray Diffractometer() (Specification) ⇒ • CuKα radiation (λ= 0.154 nm) • $2\theta = 5 \text{ deg to } 50 \text{ deg}$ • Scan rate = 3 deg/min •Step interval = 0.01 deg



2theta(degree)

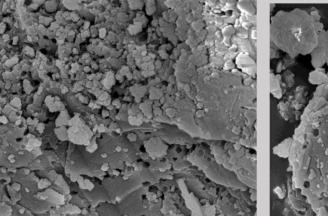
 Characteristic peaks of Raw ES particles observed at the Brgg's angle of 23.15°, 29.49°,36.07°,39.51°,43.25°,47.60°,48.60° hkl corresponding [012],[104],[110],[113],[202],[018],[116]

•Confirmed the presence of Calcite (JCPDS-72-1651) mineral, a stable form of CaCO3 and pure crystalline

- Variation of Crystalline size with treatment of different conc. of NaOH.
- D= $\frac{K*\lambda}{\beta \cos \theta}$ K= 0.94 β=Full Width Half Maxima θ=Bragg Angle
- D was more for 10% alkali treated (71.56 nm)
- followed by Raw(71.53 nm), 15%(65.03 nm) and 5%(59.61 nm). Chemical treatment didn't alter the chemical
- composition of ES particles. No remarkable change was observed in the XRD peaks when treated with NaOH.

Fourier Transform Infrared (FTIR) Spectrum (Specification) Company – PerkinElmer Spectrum •No. of Scans - 8 Resolution - 4 Determined functional groups in treated and untreated ES particles. • 3428 cm⁻¹ (broad absorption band) presence of residual water •2513cm⁻¹ and 2876cm⁻¹ - smaller vibration band for the CO₂²-•1798cm⁻¹ - weak band corresponds to C=O bonds •1427cm⁻¹ - characteristic band of C-O stretching •875cm⁻¹ ,712cm⁻¹ -out of plane and in Wavenumber(cm⁻¹ plane bending modes of CaCO3 All the bands remained same in treated FTIR data matched with XRD data that

Field Emission Scanning Electron Microscope(FESEM) (Specification)



1. (RAW ES)

Has irregular shape of

Has porosity because of semi

permeable membrane and it

allow to pass air and moisture

Somehow after cleaning,

membrane which may be

decrease the strength of

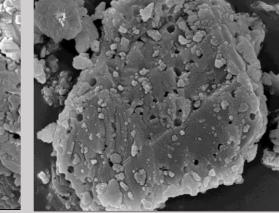
presence of

through it.

composite.

showed the complete calcite phase

present in ES particles.



2. (5% NaOH)

Treated with 5% of alkali

solution, the surface of shell

seemed to clean and smooth.

treatment may dissolved

the membrane and exposed

these pores may allowed

matrix to penetrate inside

the shells to have a better

adhesion between filler-

the pores of shell.

matrix

• Electron Energy – 10KV Magnification – 25,000x

Model No –Nova

NanoSEM 450

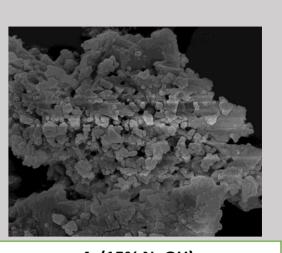
ES particles suggested that the chemical

treatment has not changed the chemical

composition of material

3. (10% NaOH) Treated with 10% alkali solution

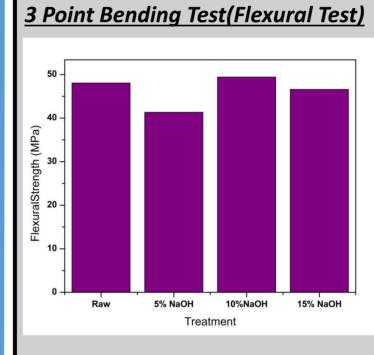
- indicated a greater washing up on the surface of ES leading to a rougher surface.
- separation of membrane from ES increased which lead to increase the surface area of the pores.
- The increased surface area of pores facilitate a better mechanical interlocking between filler-matrix for composite fabrication



4. (15% NaOH) Treated with higher conc.(15%) of alkali degraded the surface of

- •There are many pores. Increase of effective surface area of these pores lead to the swelling of particles.
- Swelling of particles have been severely damage to the surface leads to incompatibility between

filler-matrix.



•Increase in composition of NaOH during pre-treatment of eggshell makes the composite less stiff and allow it to bend more under high pressure, which satisfies our requirement

(Specification)

- INSTRON (ASTM-D-7264)
- Crosshead Speed- 5mm/min Span length – 80 mm
- Strong adhesion between filler and matrix interface can cause better stress transfer from the matrix to the filler lead to a higher flexural strength.
- Flexural strength observed to be highest for 10% NaOH treated ES filler.
- The removal of membrane from ES exposed more pores towards the penetration of chemicals into it.
- The penetration of chemicals acted as a bridge between filler and matrix and made hydrophilic filler into hydrophobic to make good bonding between with ероху.
- The more strength of 10% treated filler may also due to increase of effective surface area of pores to make available for Epoxy addition.

Future Scopes and Conclusion

 Composite were prepared by fiber treatment with varying concentrations(5%,10%,15%) of NaOH

•XRD and FTIR data confirmed the pure Calcite phase in ES filler. Significant difference between crystalline size of treated and untreated ES fillers. The 10% NaOH treated filler showed highest crystalline size.

 Incorporation of fillers into Epoxy increased flexural strength of composite and improved workability. 10% NaOH treated ES reinforced Composite showed better flexural result.

•Future trends of making composite has been shifted from traditional to natural because of biodegradability and ecofriendly nature.

 Attention has bee paid to strengthen composite to make them useful for diverse applications side such as industrial drive shafts, supporting beams of highway bridges, windmill blades.



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- Hiremath, P., Shettar, M., & Gowrishankar, M. C. (2016). Indian Journal of Science and Technology, 9(32).
- Hassan, S. B., Aigbodion, V. S., & Patrick, S. N. (2012). Tribology in Industry, 34(4), 217-225.
- Tsai, W. T., Yang, J. M., Lai, C. W., Cheng, Y. H., Lin, C. C., & Yeh, C. W. (2006). *Bio resource Technology*, *97*(3), 488-493.
- Mosaddegh, E., & Hassankhani, A. (2013). Catalysis Communications, 33, 70-75.
- Nasif, R. A. (2015). Iraqi Journal of Applied Physis, 11(1), 25-28.