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Unleashing The Mechanical properties of Synthesized HAp (Eggshell) Particulates Reinforced in Biodegradable Polymer Composites.

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Abstract - *The purpose of the current study was to examine the morphology, physical, and mechanical characteristics of hybrid biodegradable polymer composites composed of synthetic hydroxyapatite particulate (HAp) derived from eggshells at different weights (3%, 5%, and 7%, respectively), with fixed compositions of PLA (60%) and carbon fibre (40%) using the hand lay-up method. Hence, it was determined that increasing HAp by percentage was effective in enhancing mechanical properties such maximum tensile and impact strengths by 36% and 111% respectively. Composites have a maximum hardness of 141.7VHN. This biodegradable composite material show's promising mechanical properties overall, indicating their potential for a wide variety of medical applications.*

Key Words: **Eggshell, Carbon Fibre, ISO polyester, bio medical, implants, hydroxyapatite, biodegradable.**

1.INTRODUCTION

A composite material is developed by combining two or more materials with different physical and chemical properties to produce a new desirable attribute that is appropriate for the application. Fiber, particles & sheets can be used as reinforcement. Another substance referred as the matrix, is embedded within the reinforcing material. While the fibre material is made of metallic, ceramic, or polymer, the matrix is primarily made of a polymer. The principal load-bearing part in a composite material is created by the fibre, which is stiff and stronger than the matrix. Composite materials have a new generation of materials that are hybrid composites.

Hybrid composite materials must be composed of two or more different reinforcing materials and matrix materials. This composite material offers superior mechanical properties than basic reinforced fibre composite materials because of the shearing effect of the reinforcing fibres. Aerospace interiors, naval structures, civil construction, industrial products, sporting goods, and interior and exterior automotive applications are used for hybrid composites. This paper mainly focused on specific subset of Biodegradable polymer hybrid composites, which are beneficial to medical industry.

Biodegradable composites have a wide range of potential applications in various industries. Natural fibres, synthetic fibres, nanoparticles and other materials can also be used as reinforcing materials, and they are introduced in biodegradable polymer matrix. Body implants novel application of biodegradable materials.

The particulate reinforcement used in our composite is Hydroxyapatite (HAp) which is calcium phosphate mineral that is widely used in biomedical applications such as bone grafts, dental implants, and drug delivery systems. Eggshells are potential source of calcium and have been used for the synthesis of HAp due to their abundant availability, low cost & biocompatibility and carbon Fibres are used as reinforcement. Carbon fiber is made up of thin, tightly woven strands of carbon atoms that are bonded together with a polymer resin to create a material that is both strong and lightweight. It has excellent mechanical properties, including high tensile strength, stiffness, and fatigue resistance. The above reinforcing materials are embedded with the polymer matrix.

Method used for producing the above composite is Hand – layup technique. Hand layup is a traditional and versatile composite manufacturing technique. It involves manually placing individual layers of reinforcement materials, such as fiberglass, carbon fiber, or Kevlar, into a mold or on a tool. Each layer is then infused with resin, later curing process was done by natural or artificial technique to form the required composite.

2. Materials and methods:

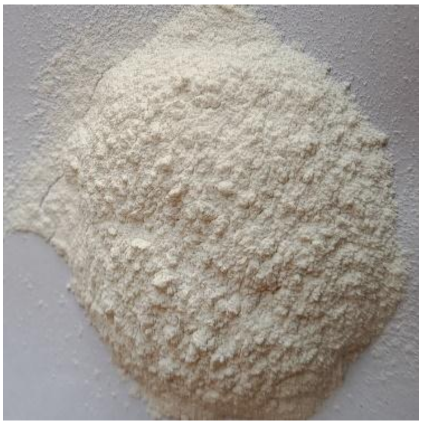


Fig 2.1 Synthesized HAp

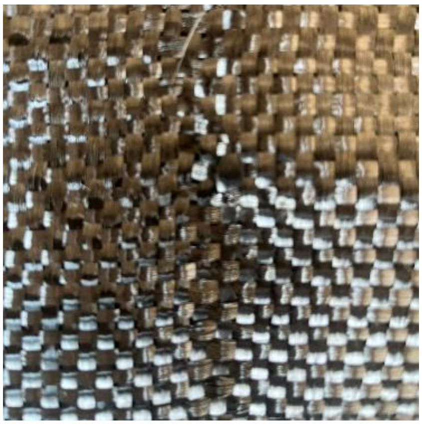


Fig 2.2 Carbon Fibre

Fig 2.3 (1, 2, 3)Resin, catlyst, Accelerator

2.1 Hydroxyapatite (HAp) is a biocompatible material that can be synthesized from eggshells. The process involves collecting and cleaning the bio waste eggshells thoroughly to remove any traces of dirt and other foreign materials. After cleaning, the eggshells are dried and ground into 64µm size fine powder. This powder is then heated at 450°C and 900°C to transform the calcium carbonate present in the eggshells into calcium oxide and Calcium phosphate (Hap) this process is known as chemical precipitation to obtain HAp powder.

2.2 Carbon fiber: It is a material that is widely used in composite manufacturing due to its high strength-to-weight ratio, stiffness, and durability. The fibers are made from a precursor material, usually polyacrylonitrile (PAN), which is chemically treated and heated to create carbonized fibers.

2.3 PLA ISOMER resin: Polyester Iso resin is a type of thermosetting polymer that is widely used in composite manufacturing. It is a low-cost resin that offers good mechanical and physical properties. Polyester Iso resin has a high resistance to chemical corrosion, UV radiation, and heat, making it suitable for outdoor applications. The resin has a low viscosity, making it easy to mix with fillers and reinforcement materials. Additionally, Polyester Iso resin is easy to process and can be cured at room temperature or with heat, depending on the application requirements.

2.3.1 MEKP catalyst: Methyl ethyl ketone peroxide catalyst is a typical initiator used in the curing of unsaturated polyester resins. In our composite, it makes up around 10% of the overall matrix weight and is often added to the resin system in tiny amounts. It is a transparent, colorless liquid that can start a quick polymerization reaction when an accelerator is present.

2.3.2 Cobalt accelerator: It is a common accelerator used in the curing of unsaturated polyester resins. It is often incorporated into resin systems in trace levels; in our composite, it makes up around 10% of the overall matrix weight, along with a peroxide initiator such as MEKP (methyl ethyl ketone peroxide). The cobalt accelerator plays a critical role in the curing process by enhancing the decomposition of the peroxide initiator, leading to a faster and more complete reaction.

Carbon fiber of 200mm × 200mm were used as a primary reinforcement. HAp powder was added as secondary reinforcement with three different weight fractions, i.e., 0%, 3%, 5%, and 7% respectively. And Polyester isoresin (80%) is mixed with the cobalt accelerator (10%) and MEKP catalyst (10%) to form the matrix required to embed the reinforcements.

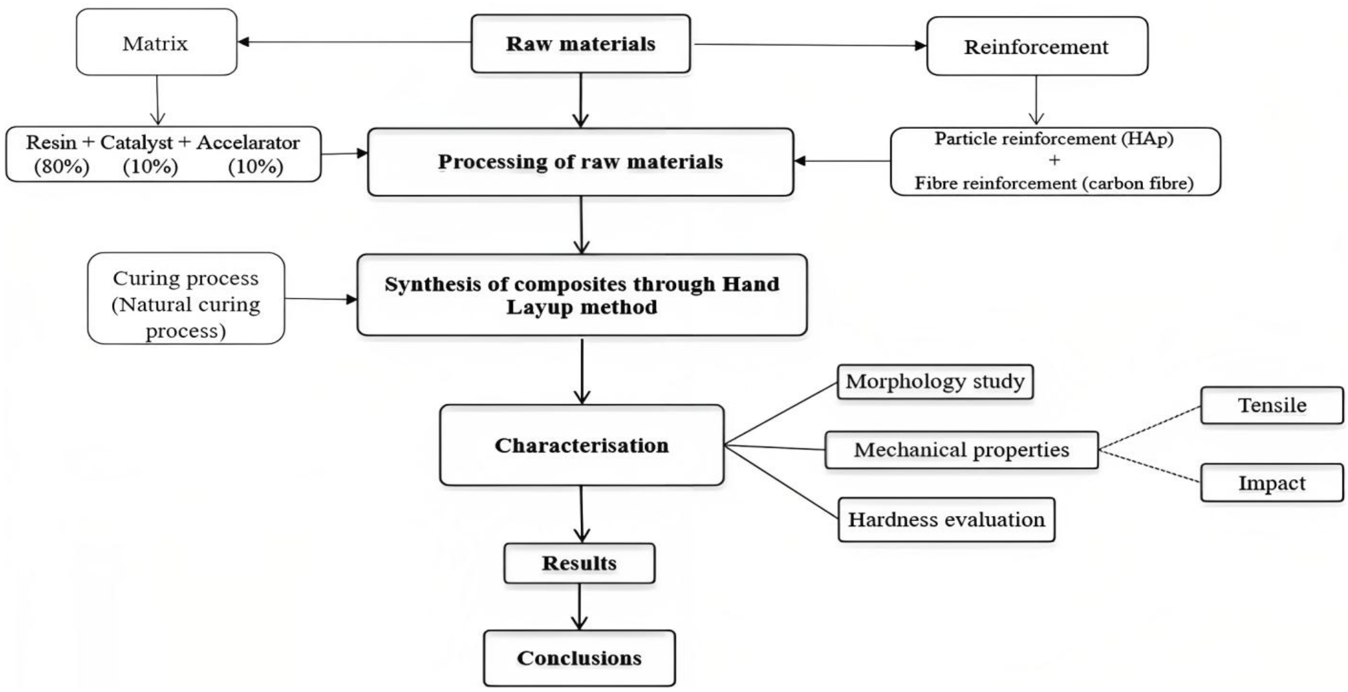
Material	Matrix wt %	Carbon Fibre wt %	EggShell powder wt %
Base	60	40	0
3% Eggshell	58.2	38.8	3
5% Eggshell	57	38	5
7% Eggshell	55.8	37.2	7

2.4 Hand Lay-Up Method: The fabrication is carried out using the hand lay-up process. Fibre and matrix constituent (PLA isomer with MEKP catalyst and Cobalt accelerator) resin and carbon fibre were taken in 1.5:1 ratio as per the reference. With the help of a suitable glass rod, PLA isomer was mixed with a catalyst in a 10:1 ratio at room temperature in a beaker until a homogeneous mixture was attained. The mixture was then thoroughly agitated to minimise the amount of air bubble that formed during mixing. To this mixture, particulate reinforcements of Egg shell of BSS- 320 mesh size are added. Besides that, three different mixtures are prepared for three different composites with 3%, 5%, and 7% weight percentage. This process involves laying a plastic sheet on a worktable, applying the predefined matrix mixture, and then overlaying it with a layer of carbon fibre. Layers of carbon fibre mats were continually applied to a PLA isomer to create a laminate. The cross-section of the carbon fibre is 200x200mm. The thickness of the carbon fibre reinforced resin composite is 5 mm. Then, to prevent defects like porosity, these laminates were compressed with a roller. This is allowed to cure for 48hrs to produce to form the required composite.

3. EXPERIMENRAL METHODOLOGY:

The experimental methodology involves using raw materials such as a matrix (resin, catalyst, accelerator) and reinforcement (particle and fiber) for the synthesis of composites through the hand layup method. The synthesized composites are then characterized through morphology study using SEM, hardness evaluation, and mechanical properties such as tensile and impact tests.

Flow Chart:



(a) Base Composite

(b) 3% Egg Shell

(c) 5% Egg Shell

(d) 7% Egg Shell

Fig 2.4 (a, b, c, d) Hardness, Impact, Samples as per ASTM Standards for the developed 4 composites.

4. Results and Discussions:

Material	Hardness (VHN)	Tensile Strength (Mpa)	Impact Strength (j)	% Rise in Hardness	% Rise in Tensile Strength	% Rise in impact Strength
Base	81.8	386.34	9	0	0	0
3% Eggshell	103.4	420.62	12	26.4	8.87	33.33
5% Eggshell	119.7	485.98	16	46.33	25.79	77.7
7% Eggshell	141.7	525.28	19	73.22	35.9	111.11

4.1.1 Hardness:

Leco vickers hardness tester machine is used to measure the hardness of base composite, 3wt% eggshell composite, 5wt% eggshell composite and 7wt% eggshell composite, with a load of 1 kg, 10 sec. for each hardness value, an average of 6 readings was taken. The results of the hardness test for alloy and composites. The comparison of hardness and %rise of hardness for base composite, 3wt% eggshell composite, 5wt% eggshell composite, 7wt% eggshell composite, as shown in the Fig 4.2. Results reveal that hardness values of 7wt% eggshell composite show superior then the base composite. Due to reinforcement particulates hardness increases as the weight% of particulates increases respectively.

4.1.2 Impact Strength:

While conducting the impact test, 3 test samples are taken for each composite. The effect of eggshell particles on the impact strength of fibre reinforced composites showed in Fig 4.3. From the result base composite (carbon fibre PLA Composite) recorded lower Impact strength value than eggshell particulate composites. The highest impact strength was recorded at 7% weight of eggshell particulates. Carbon fibres have high strength and eggshell particulates help in the improvement in toughness. This is confirmed that 7wt% of eggshell particulate composite have higher impact strength compared to remaining composites.

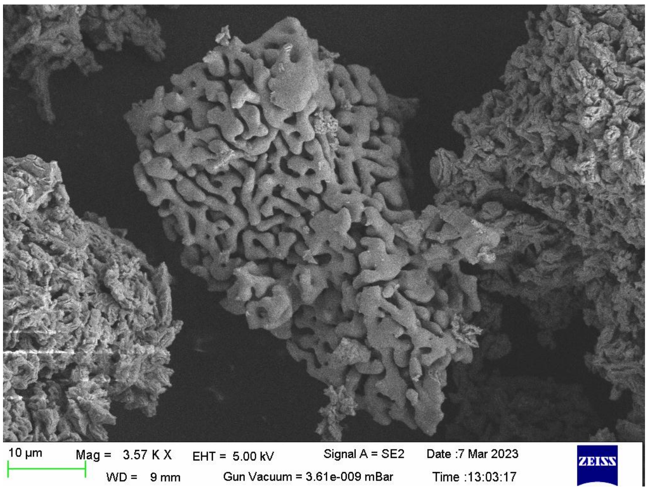


Fig 4.1 SEM Micro graph of Eggshell powder

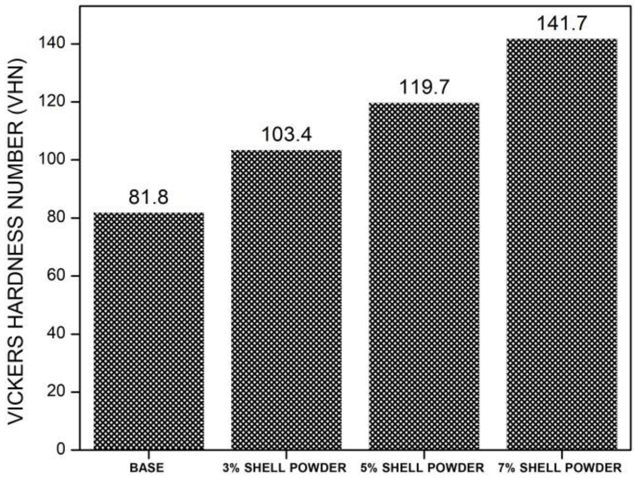


Fig 4.2 comparison of Hardness of the composites.

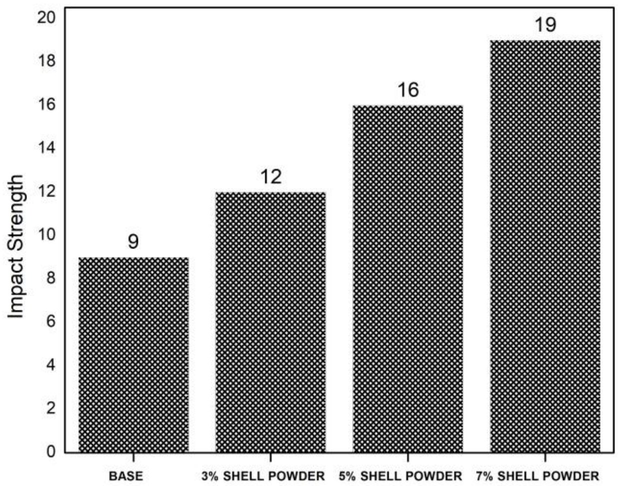


Fig 4.3 comparison of Impact Strength of the composites

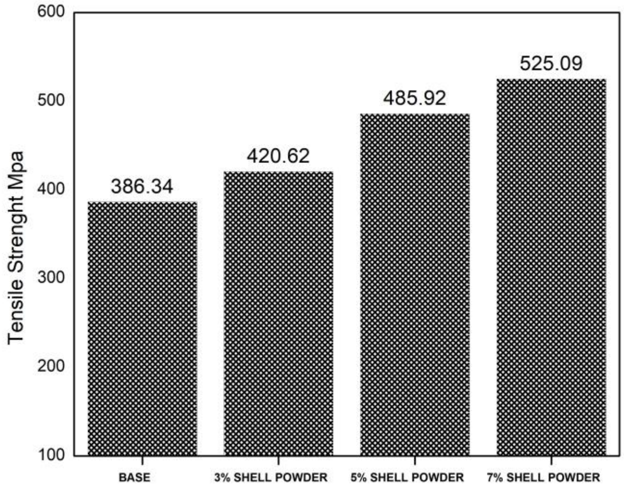


Fig 4.4 comparison of tensile Strength of the composites

4.1.3 Tensile test:

Tensile specimens of base carbon fibre composite and eggshell particulate composites were tested and clearly shows the tensile strength of different weight% shows that, among all wt%, the 7 wt% carbon fibre PLA matrix show the maximum tensile strength of 525.6Mpa and %rise in tensile strength as compared with base composite to 7 wt% carbon fibre PLA matrix is 35.9 down in the fig 4.4 & 4.8. In the stress strain curve, there is brittle behaviour shown in the Fig4.7. due to the increase of eggshell particulates.

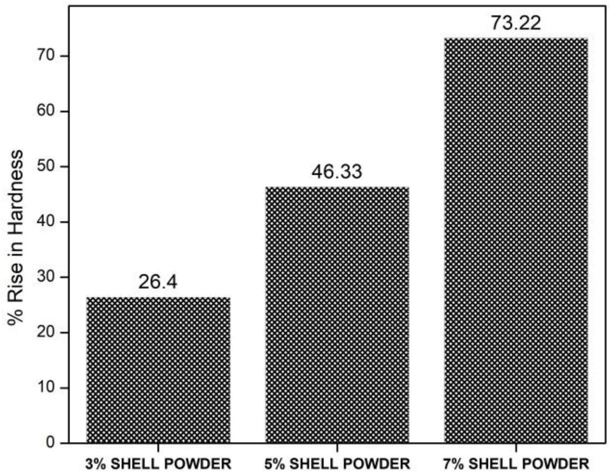


Fig 4.5 Comparison of % of the rise in Hardness

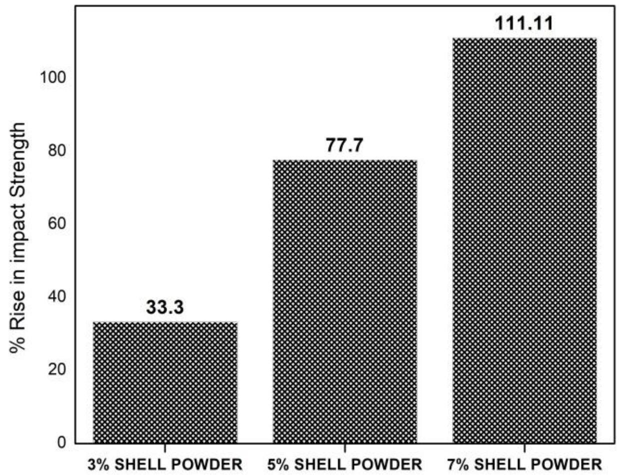


Fig 4.6 Comparison of % of the rise in Impact Strength

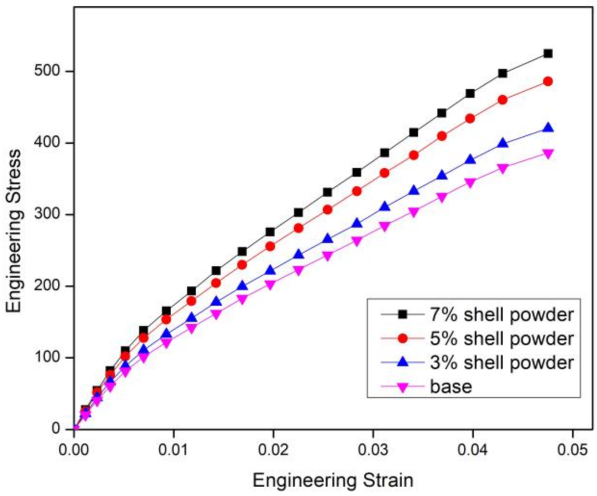


Fig 4.7 Comparison of Stress – Strain Curve

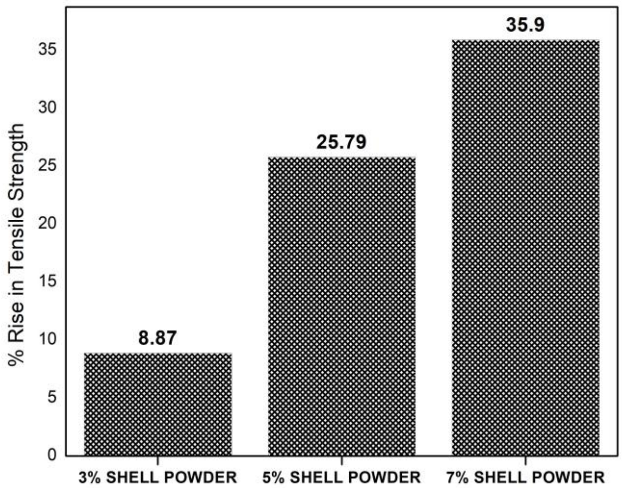
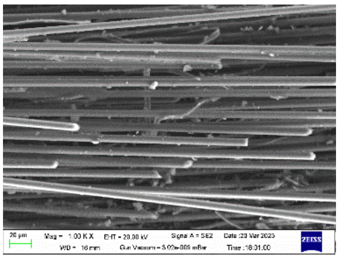


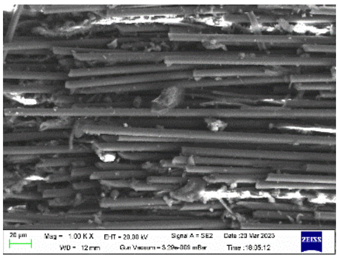
Fig 4.8 Comparison of % of the rise in Tensile Strength

4.1.4 Morphology study:

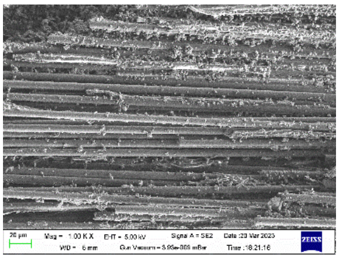
The SEM images of eggshell composites reveal that the HAp particles have been uniformly distributed along the fibre matrix. This even distribution of HAp particulates acts as a strengthening mechanism for the composites. This is due to the strong adhesion between the HAp particles and the fibres, resulting in an effective transfer of stress from the matrix to the fibres. The uniform distribution of HAp particulates also contributes to a more homogeneous microstructure, reducing the potential for defects and enhancing overall mechanical performance. Overall, the SEM images of our composite indicate that the addition of HAp particulates has significantly improved its mechanical properties, making it a promising material for various applications.



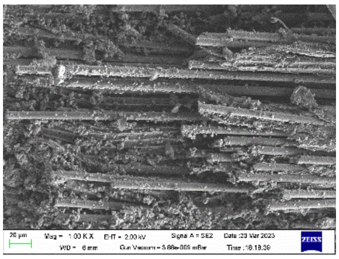
(a) Base Composite



(b) 3% Egg Shell



(c) 5% Egg Shell



(d) 7% Egg Shell

Fig 4.9 (a, b, c, d) Shows the SEM Micro Graphs of 4 Composite materials Respectively.

5 Conclusion:

The present work deals with the characterization of synthesized Eggshell particulate & Carbon fibre reinforced PLA composite. According to the shape of the particulate, the SEM images confirm that particulate has high toughness. SEM and mechanical properties of PLA resin- reinforced Carbon fibre composites with and without eggshell powder were evaluated. The SEM images revealed that the eggshell particulates are distributed uniformly throughout the PLA polymer matrix. The mechanical behaviour of the composites understudy leads to the following conclusions.

- The use of hybrid biodegradable polymer composites composed of synthetic hydroxyapatite particulate (HAp) derived from eggshells and carbon fibre reinforced in PLA matrix demonstrated improved mechanical properties such as maximum tensile, impact, by up to 35.9% & 111% respectively.
- The study found that increasing the weight percentage of HAp (up to 7%) was effective in enhancing the mechanical properties of the composites.
- The hand lay-up method was used to develop the composites, and the resulting materials showed a maximum percentage rise in hardness of 73.22 for 7 wt% of HAp composite.
- Overall, this paper suggest that the bio composites have promising mechanical properties, making them suitable for a wide range of medical applications.
- The use of HAp derived from eggshells is an innovative approach that could potentially offer a cost-effective and sustainable solution for creating biodegradable composites.

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REFERENCES

[1] Sisay Walle Mekonen,1 Sivaprakasam Palani ,1 B. Ravi ,2 Samson Mekbib Atnaw ,1 Melaku Desta ,1 and Yohanes Regassa, "Mechanical Properties of Bone Particulate and E-Glass Fiber Reinforced Hybrid Polymer composite", *Advances in Materials Science and Engineering,* Volume 2022, Article ID 5902616.

[2] Nagarajan Velmurugan ,G. Manimara , Shoba Ravi , D. Jayabalakrishnan , " Mechanical Property Of Stitched Glass Fiber, Epoxy With Bentonite Clay Composite using Hand Layup Method" , tagajournal https://www.researchgate.net/publication/337560601, , ISSN: 1748-0345

[3] Muhammad Bilal , "COMPOSITE FORMATION BY HAND-LAYUP PROCESS", https://www.researchgate.net/publication/36065486

[4] Raluca Nicoleta Darie-Nit,a 1 , Maria Râpa 2,* and Stanisław Fra̧ckowiak 3 Special Features of Polyester-Based Materials for Medical Applications Polymers 2022, 14, https://doi.org/10.3390/polym14050951

[5] J.L. Lancout, Calcium phosphate as biomaterials, in: D. Muster _Ed.., Biomaterials-Hard Tissue Repairs and Replacement, Vol. 3, Elsevier, Amsterdam, 1992.

[6] K. de Groot, C.P.A.T. Klein, J.G.C. Wloke, J.M.A. Blieck-Hogervorst, Chemistry of calcium phosphate ceramics, in: T. Yamamuro, L.L. Hench, J. Wilson _Eds.., Handbook of Bioactive Ceramics, Vol. 2, CRC Press, Boca Raton, FL, 1990.

[7] D. Bernache-Assolant, Improvements in bioceramics technology, in: D. Muster _Ed.., Biomaterials-Hard Tissue Repair and Replacement, Vol. 3, Elsevier, Amsterdam, 1992.

[8] I.J. Macha, L.S. Ozyegin, J. Chou, R. Samur, F.N. Oktar, B. Ben-Nissan, J. Aust. Ceram. Soc. 2013, 49, 122.

[9] Y. Xu, D. Wang, L. Yang, H. Tang, Mater. Charact. 2001, 47, 83.

[10] X. Zhang, K.S. Vecchio, J. Cryst. Growth. 2007, 308, 133

[11] Patra, M. Das, K. Anwar, B. Khan, K. Kamran, D.R. Jana, Investigation on Mechanical and Physical Properties of Fly Ash Reinforced Epoxy Resin Composite, Mech. Civ. Eng. 15 (2018) 64–68, https://doi.org/10.9790/16841501046468.

[12] B.R. Phanikumar, S.S. Radhey, Effect of Flyash on Engineering properties of soil, J. Geotech. Geoenviron. Eng. (2004) 764–767.

[13] S. Sunkara, R. Karthikeyan, R. Sateesh, Analysis of Mechanical Properties of Polymer Based Nano Fly Ash, 1 (2018) 45–49.

[14] G. U, M. R, V. AT, C. M, Fabrication and Testing of Composite Powder Material from Prawns Shells, Res. Rev. J. Mater. Sci. 04 (2016) 37–46.

[15] C. Casadidio, D.V. Peregrina, M.R. Gigliobianco, S. Deng, R. Censi, P. Di Martino, Chitin and chitosans: Characteristics, eco-friendly processes, and applications in cosmetic science, Mar. Drugs. 17 (2019), https://doi.org/10.3390/md17060369.

[16] R. Purohit, P. Sahu, R.S. Rana, V. Parashar, S. Sharma, Analysis of Mechanical Properties of Fiber Glass-Epoxy-Fly Ash Composites, Mater. Today Proc. 4 (2017) 3102–3109, https://doi.org/10.1016/j.matpr.2017.02.19

[17] Meng, C.J.; Zhao, J.M.; Yin, Y.X.; Luo, J.; Zhao, L.Y.; Jiang, W.B.; Feng, J.Y. Preparation and Characterization of PLA Film/3D Printing Composite Scaffold for Tissue Engineering Application. Fibers Polym. 2020, 21, 709–716. [CrossRef] Pearce, A.K.; O'Reilly, R.

[18] Han, K.S. (1983). Compressive fatigue behaviour of a glass fibre-reinforced polyester composite at 300 K and 77 K. *Composites,* **14** (2), 145-150.

[19] [7] Higashi, S., Yamamuro, T., Nakamura, T., Ikada, Y., Hyon, S.H.,Jamshidi, K. (1986). Polymer-hydroxyapatite composites for biodegradable bone fillers. *Biomaterials,* **7** (3), 183-187.

[20] Mishra, S., Mohanty, A.K., Drzal, L.T., Misra, M., Parija, S., Nayak, S.K.,Tripathy, S.S. (2003). Studies on mechanical performance of biofibre/glass reinforced polyester hybrid composites. *Composites Science and Technology,* **63** (10), 1377-1385.

BIOGRAPHIES

Project guide, assistant professor, 8 years of Research + teaching experience & 4 years of industrial experience. Sir was invited as field training instructor to CQEA(EFS) Indian Navy. Under his guidance more than 100+ students qualified and secured top ranks in competitive exams like GATE, ECET & PGECET and they got placed in reputed universities and IIT's. He is one the former students of Prof. B S Murthy Research Group.

K. Eswar Raja Varaprasad is a B.Tech undergraduate at Andhra University with a GATE rank of 424 in 2023. He has a strong foundation in composite fabrication and analysis, guided by Mr. Kumar Raja, and is highly interested in advancing his knowledge and skills in this field. He has great potential to make significant contributions to the field in the future.

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