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Development of Collagen Fibers Reinforced Epoxy Matrix Composites and its Degradation Study

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Abstract: In recent years, it has been discovered that composites are the most discerning and promising material for structural applications. The resilience of biomaterials to deterioration or corrosion by bodily fluids is a crucial prerequisite for their use as orthopedic implants. In the current study, collagen type I fibres and epoxy matrix are combined to create polymer matrix composites with varied fibre volume fractions, including 10%, 20%, 30%, and 40%. The breakdown rate for the collagen fibre reinforced polymer matrix composites was assessed using test specimens made from the manufactured composite and put through an immersion test in order to ascertain the material's suitability as a bio-implant. By performing an immersion test in which the specimen's surface area is exposed to a test media that simulates bodily fluids, the degradation behavior of collagen fibres reinforced epoxy matrix composites is studied. The material's mass loss is monitored throughout the test, and the degradation rate is estimated using the mass loss to determine whether the material is suitable for use as a bio-implant. Here, their potential use in orthopedic bone surgery as an implantable material was thought about and researched. Among the orthopedics uses for these composite materials are bone fixation plates, hip joint replacement, bone cement, and bone transplant.

Keywords: PMC, Collagen fibers type I, Epoxy, Degradation.

I. INTRODUCTION

Degradation is the breakdown of a designed substance into its individual atoms as a result of chemical interactions with its environment [1]. Plastics' environmental degradation mechanisms can be categorised as either (i) physically degrading, which refers to changes in the material's bulk structure like cracking, embrittlement, and flaking, or (ii) chemically degrading, which refers to molecular changes like bond cleavage or oxidation of lengthy polymer chains [2,3].

Clinical Importance of the Process of Degradation

- Degradation can significantly reduce a material's fatigue life and ultimate strength, resulting in the mechanical failure of an implant. Degradation-related fracture of the implant occurs with a low but finite frequency.
- The release of degradation products may elicit an adverse biological reaction in the host.
- Degradation products have been implicated in causing local pain and swelling in the region of the implant, in the absence of infection [4].
- The presence of degradation products in the implant's surrounding tissue may ultimately set off a chain of events that result in periprosthetic bone loss.

II. MATERIALS AND METHOD

A. Composite Preparation

In the current experiment, collagen type I fibres are employed as reinforcement to create the collagen fibre reinforced composite, and epoxy is taken into account as the matrix material together with hardener [5]. To transfer load from the matrix to the reinforcement at the interface, there must be good collagen fiber-matrix adhesion. Poor adhesion will cause debonding as load is passed from the matrix to the fibre, which will result in poor mechanical properties [6]. Alkaline NaOH treatment was used to change the surface chemistry and promote collagen-matrix adhesion [7].

To create aqueous solution of NaOH, sodium hydroxide pellets are dissolved in distilled water. These concentrations were chosen to protect the protein content of the fibre. With a solution-to-fiber ratio of 10 ml to 1 g, collagen strands were immersed in 10% aqueous NaOH solution at room temperature for various amounts of time (5 h, 10 h, and 20 h). To make sure that no NaOH remained after the immersion, the fibres were first washed in lab water and then in distilled water. The fibres were then dried for 24 hours at 60 °C.

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A 400 x 250 x 12 mm Per-pex sheet mould was used to cast the composite sheet. Different volume fractions by fibre weight, such as 10%, 20%, 30%, and 40%, are used to create the polymer matrix composite. Hand layup preparation was used to create the sample. To reduce air entrapment for different fibre volume fractions, a calculated amount of epoxy resin and hardener (ratio of 10:1 by weight) was thoroughly mixed with moderate stirring. For rapid and simple removal of the composite material, a mould release sheet is placed over the glass plate and mould release agent is applied to the inside surface of the mould. The mould was kept in place on the glass sheet, and then a thin layer of the mixture—about 2 mm thick—was poured on top. The required amount of fibre was then included throughout the mixture. The remaining slurry was then poured into the mould. Air bubble formation was prevented at all costs. The mould was then subjected to pressure from the top and allowed to cure for 72 hours at room temperature. By applying pressure, a minute amount of the epoxy and hardener mixture was forced out. Special consideration was used when creating composite sheets to account for this loss. After 72 hours, the samples were taken out of the mould, sliced into different sizes, and kept for later analysis in an airtight container. [8,9].

B. Immersion test

Basically, small portions of the candidate material are exposed to the test medium during an immersion test for degradation study, and the material's weight loss is monitored over time. Immersion testing is still the greatest way for identifying materials that shouldn't be taken into account for particular applications and removing them from further consideration. There is no easy method to extend the results from these straightforward tests to the forecast of system life, even though they are the quickest and most cost-effective way to provide a preliminary selection of the best materials. [10].

Different stages of immersion corrosion test are

- Specimen preparation
- Apparatus set up
- Test conditions
- Methods of cleaning specimens
- Calculation and Evaluation of results

1. Specimen preparation;

Specimen: A circular specimens of about 10mm diameter with a 30mm length is selected for the test according to the ASTM standards G31.

Surface Area: The total surface area of a circular specimen is given by: $A = 3.14[(1/2)D^2+DL]$

Surface Preparation

- The variation in the surface of the specimen is eliminated by grinding with a coarse abrasive paper or cloth such as no 50.
- The specimens are stamped with an appropriate identifying mark.
- The specimens are finally degreased by scrubbing with bleach free scouring powder, followed by through rinsing in water and in a suitable solvent.
- The dried specimens are weighed in electronic digital weighing machine.

2. Apparatus set up;

Fig. 1 show the apparatus required for conducting immersion test for 10%, 20%, 30%, and 40% collagen fibers reinforced polymer matrix composite specimens. The apparatus consists of a kettle of 1000ml capacity in which a reflux condenser with atmospheric seal is placed, provision is also made to place a thermowell or a temperature regulating device to measure the temperature of the solution and a heating mantle is used to maintain the constant temperature of the hanks solution throughout the experiment. A specimen support system is placed inside the kettle on which specimens are hanged throughout the test period.

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Fig. 1 Set up of Immersion corrosion test

3. Test Conditions;

The limits of controlling factors for immersion test of biomaterials are:

Temperature	- 37°C
pH	- 7
Composition	- Hanks solution
Rate of air flow	- 1lpm
Test duration	- 48hr

The composition of the solution used for the test must replace the body fluids in the human body. The common testing solution relating to simulated body fluid is Hanks solution. The composition of the solution is given below:

Solution A: 160g NaCl + 8g KCl + 4g $MgSO_4 7H_2O$ in 800ml H_2O
Solution B: 2.8g $CaCl_2$ in 100ml H_2O
Solution C: A + B + 100ml H_2O + 2ml $CHCl_3$ (chloroform)
Solution D: 1.2g $Na_2HPO_4 7H_2O$ + 2.0g $KH_2PO_4 H_2O$ + 20.0g glucose + 2ml $CHCl_3$ in 800mL H_2O – diluted to 1000ml
Solution E: 1.4%$NaHCO_3$ = 7g $NaHCO_3$ in 500ml H_2O
Final solution: 50ml C + 50ml D + 24ml E + 900ml H_2O + few drops of chloroform

Volume of Test Solution: The volume of test solution should be large enough to immerse the test specimen in the kettle.

4. Method of Cleaning Specimen

All degraded products are removed by cleaning process. Chemical cleaning process is employed for removing degraded products. In the process phosphoric acid in the form of naval jelly is applied to clean the surfaces.

5. Calculation of degradation rate

After the duration, degraded specimens have been cleaned and they are reweighed. The mass loss during the test period can be used as the principal measure of degradation. Degradation rate is the degradation of the material over the period of time, which implies that all mass loss is due to general degradation.

The average degradation rate is given by: Degradation rate = (K*W) / (A*T*D) in mm/yr

Where: D - Density in g/cm^3, K - Constant ($8.76*10^4$) mm/y, T - Time of exposure in hours, A - Area in cm^2, W - Mass loss in g

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III. RESULTS AND DISCUSSION

Degradation test for the collagen fibres reinforced PMC specimens were conducted at the rated test conditions and the test results for various composition of fibres viz. 10%, 20%, 30% and 40% collagen fibres reinforced PMC composites are tabulated and presented in below table I.

TABLE 1 Degradation rate for varying % of collagen fibers reinforced PMC sample

Sample Fiber Content (%)	Diameter (cm)	Length (cm)	Surface area (cm^2)	Initial mass (g)	Final mass (g)	Mass loss (g)	Density (g/cm^3)	Degradation rate (mm/y)
10	1.014	3.007	11.1884	2.1084	2.1082	0.0002	1.213	0.0040
20	0.997	2.977	10.8803	2.0665	2.0664	0.0001	1.211	0.0038
30	1.060	3.012	11.7892	2.2344	2.2343	0.0001	1.233	0.0035
40	1.031	3.06	11.575	2.2216	2.2214	0.0002	1.215	0.0042

Table 1 provides the test parameters considered and the degradation rate calculated for various load viz. for varying composition of fibres viz. 10%, 20%, 30% and 40% collagen fibres reinforced PMC composites. From the table maximum degradation rate is observed for 40% fibre reinforced PMC having value 0.0042mm/y, the minimum rate is observed for 30% fibre reinforced PMC having value 0.0035 mm/y.

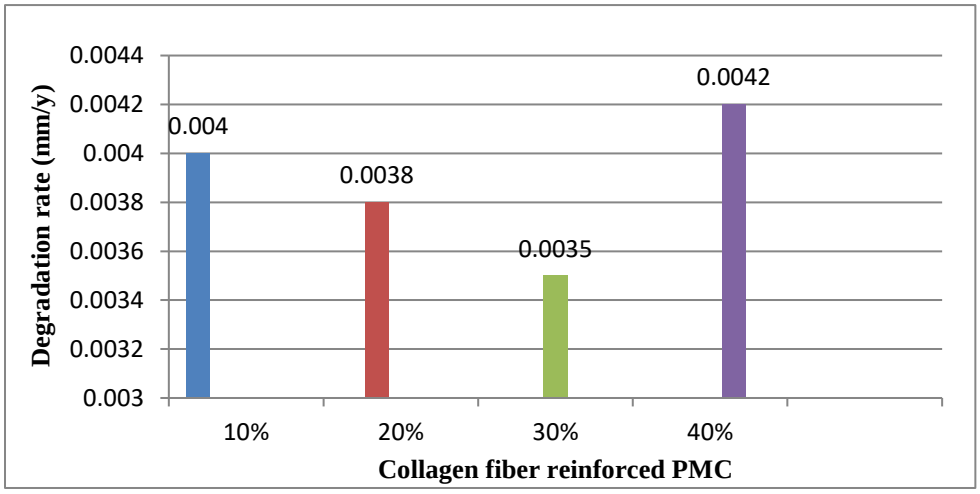

Fig. 2 Show variation of degradation rate with % fiber content

All of the collagen fibre reinforced polymer matrix composite material samples displayed superior resistance to degradation, as can be seen in fig. 2. However, due to a material feature, polymer matrix composites supplemented with 30% collagen fibres with 0.0035mm/y demonstrated high resistance to degradation. Accordingly, PMCs with 30% collagen fibres are the best material to use as implant materials in terms of their resistance to degradation. The current materials' great resistance to deterioration could be the result of improved bonding between the matrix and the reinforcement.

IV. CONCLUSION

Collagen fibres reinforced composites with different fibre contents, such as 10%, 20%, 30%, and 40% volume fractions, were created in the current study using collagen fibres type I as reinforcement and epoxy as the matrix material. Additionally, manufactured composite specimens were put through an immersion test under the prescribed test conditions. The findings were collated, and the following conclusions were drawn:

- As the percentage of collagen fibre content rises from 10% to 30%, the rate of deterioration of composites decreases. However, the rate of deterioration accelerated for collagen fibres that were 40% reinforced.

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- According to the investigation, collagen reinforced composites had a minimum wear rate for 30% composition with 0.0035mm/y.
- Adding collagen fibres to epoxy can significantly lessen degradation loss. A 30% volume fraction fibre composition was found to have good resistance to deterioration.

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