

# INVESTIGATION OF MECHANICAL PROPERTIES ON VINYLESTER BASED BIO-COMPOSITE WITH GELATIN AS RANDOMLY DISTRIBUTED FILLER MATERIAL

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## Abstract

*In the present work an attempt has been made to develop Bio-composite Material using raw dupion silk fiber reinforced vinylester matrix in addition with gelatin as filler material with varying percentage using hand lay-up method. The experiments namely tensile, flexural and hardness test were conducted on the standard samples prepared. An appreciable improvement was found in the mechanical properties of the Bio-composite, which can be used for variety of applications in the human bone replacement.*

*The biocomposites were prepared in varying percentage of filler addition (0%, 10%, 20%, and 30%) and different mechanical tests (tensile, flexure and hardness) were conducted on the samples prepared as per ASTM standards. According to literature survey the human femur bone with tensile strength for female is  $30.08 \pm 7.96$  MPa. In this work it was found that tensile strength of 30% gelatin filled with Dupion silk fiber reinforced vinylester bio-composite material is 26.86 MPa.*

**Keywords:** Implantation, Bio-composite, Dupion Silk Fiber, Gelatin.

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## 1. INTRODUCTION

Engineering Materials are used in medical application to make devices to replace a part or a function of the body in safe, reliably economically, and physiologically acceptable manner. A variety of devices and materials are used in the treatment of disease or injury. Common place examples include suture needles, plates, teeth fillings, etc

Large numbers of polymeric materials alone and in combination with other materials are becoming increasingly significant in the field of biomaterials, as a result composite materials becomes an optimum choice for such applications.

The use of petroleum based products as constituents in polymer matrix composite has raised concerns regarding environmental issue and non-renewability of the resource. Hence the problems associated with it have initiated the efforts to develop Biocomposites. The development of biocomposites has attracted great interests among researchers worldwide. This is because, biocomposite have one or more constituents that are obtained from natural renewable resources also they may have partial or complete degradation and they do not emit any toxic substance during production and disposal process. By

successfully producing biocomposite, we may be able to substitute the conventional petroleum-based plastics in various applications.

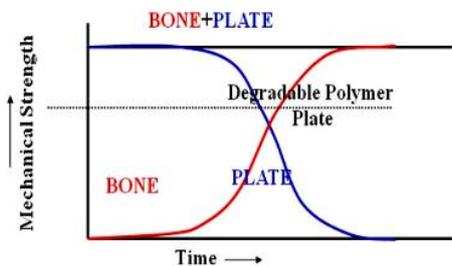
Bio-Composite are used in biomedical applications for drug/gene delivery and tissue engineering applications. They often mimic the structures of the living materials involved in the process in addition to the strengthening properties of the matrix that was used but still providing biocompatibility, e.g. in creating scaffolds in bone tissue engineering.

Metals and bioceramics for biomedical application have yielded limited successes yet substantial mismatch between their properties and bone tissue persist, thereby punctuating the need for tissue engineered products. However, metals commonly induce stress shielding and will eventually experience wear debris, ultimately leading to implant failure [1]. For example, a fractured bone, fixated with a rigid, non-biodegradable stainless steel implant, has a tendency for re-fracture upon removal of the implant. The bone does not carry sufficient load during the healing process, because the load is carried by the rigid stainless steel [2].

However an implant prepared from biodegradable polymer can be engineered to degrade at a rate that will

slowly transfer load to the healing bone [3] as shown in Graph-1. Materials used to achieve bone regeneration are diverse but not limited to metals, ceramics, synthetic polymers, naturally derived polymers, and other biocompatible substances. Success has been found by combining these materials as a strategy to eliminate the disadvantages of an individual material.

In the case of materials where starch is used as an additive to a conventional plastic matrix, the polymer in contact with the soil and/or water is attacked by the microbes. The microbes digest the starch, leaving behind a porous, sponge like structure with a high interfacial area, and low structural strength. When the starch component has been depleted, the polymer matrix begins to be degraded by an enzymatic attack. Each reaction results in the scission of a molecule, slowly reducing the weight of the matrix until the entire material has been digested [4].



Graph- 1: Mechanical Strength vs Time for Bone and Plate

The current work is an effort to utilize the natural renewable resources to develop biocomposite material for biomedical applications such as bone Plates, bone screws material for both internal, external fixations. The dupion silk fiber is a natural fiber, it is used as reinforcement in vinyl ester resin matrix and gelatin is used as fillers.

2. PREPARATION OF BIOCOMPOSITES

2.1 Materials

Unsaturated vinylester resin was selected as matrix material, Dupion silk fibers as reinforcement and gelatin as filler material.

Vinyl ester resins having density 1.05 g/cm<sup>3</sup> are becoming increasingly important for fiber reinforced composites. They combine the excellent mechanical, chemical and solvent resistance properties of epoxy resins with the properties found in the unsaturated polyester resins. The chemical structure of vinylester resin is shown in Fig.1

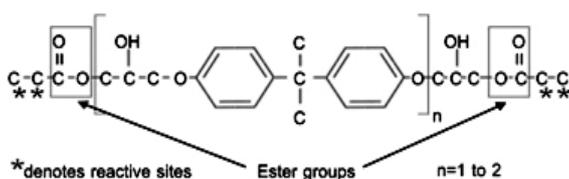


Fig 1: chemical structure of a typical vinylester resin

Silk Dupioni is a type of silk fiber created with the threads from two different silk worms. When two worms spin their cocoons close together, the fibers get tangled up; these naturally tangled fibers are then used together to make the silk thread. In spite of its delicate appearance, silk is relatively robust; the thread is rougher than regular silk as shown in Fig.2.

Gelatin is a protein obtained by boiling skin, tendons, ligaments, and/or bones with water. It is usually obtained from cows or pigs. Gelatin is used in shampoos, face masks, and other cosmetics; as a thickener for fruit gelatins and puddings (such as Jell-O); in candies, marshmallows, cakes, ice cream, and yogurts; on photographic film; and in vitamins as a coating and as capsules, and it is sometimes used to assist in “clearing” wines.

They have good Biocompatibility, Biodegradable and its degradation products are non-toxic.

Table-1: Material Procurement Table

Sl. No.	MATERIALS	SUPPLIERS
1	Unsaturated Vinylester Resin	Naphtha Resins & Chemicals (p), Bangalore, India
2	Cobalt naphthalate accelerator	
3	Methyl Ethyl Ketone peroxide catalyst	
4	Dupion Silk Fiber	Silk Exchange Office, Bangalore, India
5	Edible Gelatin	Food world, Bangalore, India

2.2 Technique

The laminates were prepared by the hand lay-up technique on the rectangular box. The rectangular box of dimension 19cm×12cm is cleaned with soft brush. A layer of wax is coated on the cleaned surface for the easy removal of the laminate after curing.

Weighed quantity of Gelatin (fillers), Methyl ethyl ketone peroxide (catalyst), Cobalt naphthalate (accelerator) are added to Vinyl ester resin taken in the bowl and stirred well for uniform distribution. The weight of constituents to be taken for each specimen is shown in Table-2 and Table-3. The catalyst and accelerator is taken at 1ml each for the 100 g of vinyl ester resin taken.

The first layer of Vinyl ester resin mixture is coated on the wax, later Dupion silk fibers are placed in the mould and the resin mixture is poured and distributed uniformly. Same procedure is repeated until the desired thickness is obtained. Alternate layers of resin and Dupion silk fiber are placed.



Fig 2: Raw dupion silk fibers

The laminate is cured under light pressure for 2 hrs, followed by curing at room temperature for 24 hours. By following the same procedure as said above bio-composite material having filler composition of 0%, 10%, 20% and 30% is prepared. Fig.3 shows the final laminate.

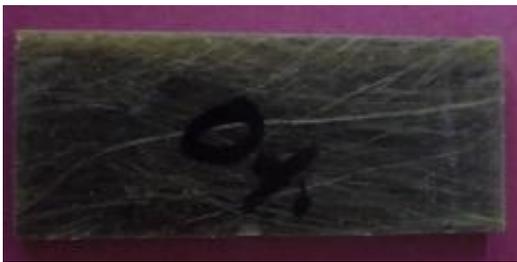


Fig-3: Prepared biocomposite laminate

The prepared Bio-Composite materials are cut into standard ASTM Dimensions using abrasive water jet machine.

**2.3 Sample Preparation Calculation**

- Density of vinyl ester resin = 1.05 g/cm<sup>3</sup>
- Density of Gelatin = 0.98 g/cm<sup>3</sup>
- Density of silk fiber = 1.33 g/cm<sup>3</sup>
- Volume of the mold, V = 190 x 120 x 3.5 = 79800 mm<sup>3</sup> = 79.8 cm<sup>3</sup> = 80 cm<sup>3</sup>

Table-2: Composition of Biocomposite

Samples	% of Resin	% of Fiber	% of Fillers
A	90	10	0
B	80	10	10
C	70	10	20
D	60	10	30

Table -3: Density Calculation of Samples

Mass of Resin (grams)	Mass of Fiber (grams)	Mass of fillers (grams)	Total mass (grams)	Density (g/cm <sup>3</sup> )
75.6	10.64	0	86.24	1.078
67.2	10.64	7.84	85.68	1.071
58.8	10.64	15.68	85.12	1.064
50.4	10.64	23.52	84.56	1.057

**3. EXPERIMENTATION**

Mechanical tests such as tensile test, flexural test and hardness tests were conducted on the specimens prepared as per the ASTM standards. The tensile test and flexural test was conducted using J J Lloyd universal testing machine and the hardness test was conducted using Rockwell hardness testing machine. All the tests were carried out at room temperature.



Fig-4: J J Lloyd UTM

**3.1 Tensile Test**

Tensile tests on composite specimens were carried out according to ASTM-D 638 standard to determine tensile strength and to observe the behavior of biocomposites under load.

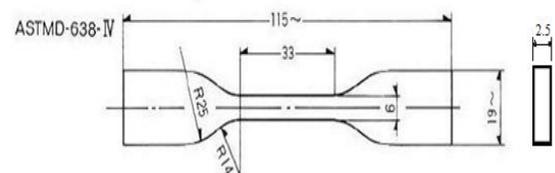


Fig -5: Standard tensile specimen dimensions

**3.2 Flexural Test**

Flexural tests on composite specimens were carried out according to ASTM D-790 standard.

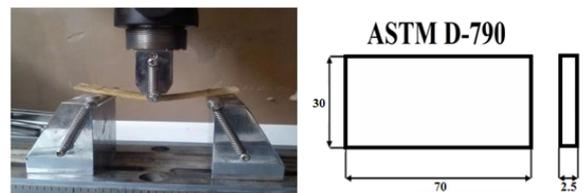


Fig -6: Standard flexural specimen dimensions

**3.3 Rockwell Hardness Test**

The Rockwell hardness is determined by the depth of the indentation in the test material resulting from application of a given force on a specific indenter.

**4. RESULT AND DISCUSSION**

In this section the results obtained from tension, Flexural and Hardness tests are tabulated and represented graphically to analyze the behavior of Biocomposites.

### 4.1 Tensile Test

The peak load and tensile strength obtained from tensile test conducted on the prepared specimens is tabulated in Table -4.

**Table 4:** Tensile Strength readings of each trail

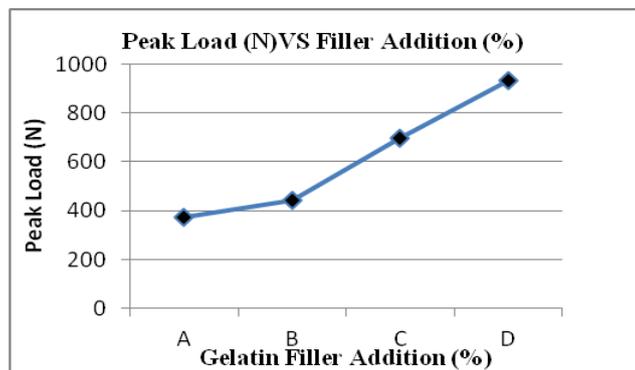
SAMPLE	TRIALS	PEAK LOAD (N)	TENSILE STRENGTH (N/mm <sup>2</sup> )
A	313	13.55	13.55
	432	17.36	17.36
B	599	23.21	33.72
	291	11.82	37.70
C	771	26.24	37.70
	621	20.71	28.60
D	892	27.05	26.12
	976	26.68	18.33

The prepared tensile test specimens before and after the tensile test experiment is shown in the figure-7



#### 4.1.1 Effect of Peak Load

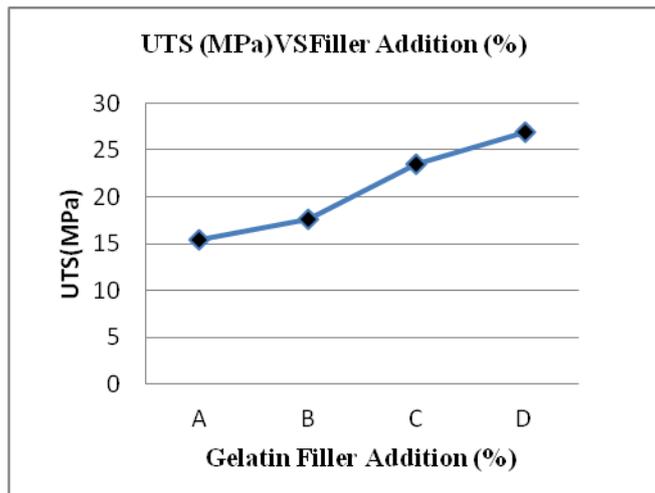
From Graph 2 it can be observed that at all filler content the highest Peak Load is for 30% filler content bio-composite with a value of 934 N.



**Graph-2:** Peak load vs Gelatin filler

#### 4.1.2 Effect of Ultimate Tensile Strength

From Graph-3 it can be observed that at all filler content the highest Ultimate Tensile Strength is for 30% filler content bio-composite with a value of 26.86Mpa.



**Graph-3:** Tensile strength vs Gelatin filler

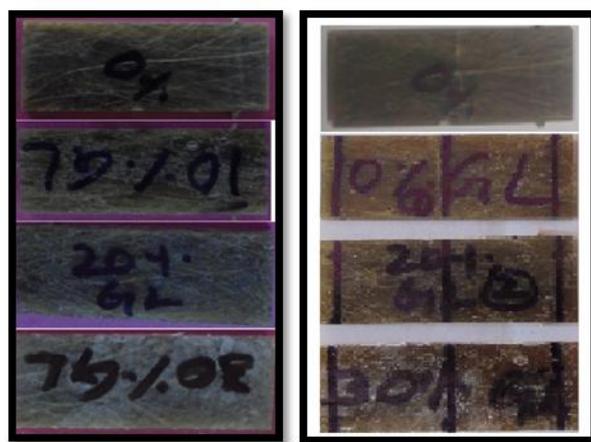
### 4.2 Flexural Test

The peak load and compression strength obtained from compression test conducted on the prepared specimens is tabulated in table-5.

**Table -5:** Flexural Strength readings of each trail

SAMPLE	TRIALS	PEAK LOAD (N)	FLEXURAL STRENGTH (N/mm <sup>2</sup> )
A	TRIAL 1	222	37.82
	TRIAL 2	217	35.13
B	TRIAL 1	118	23.33
	TRIAL 2	102	19.77
C	TRIAL 1	107	17.39
	TRIAL 2	69	11.26
D	TRIAL 1	31	6.13
	TRIAL 2	71	12.67

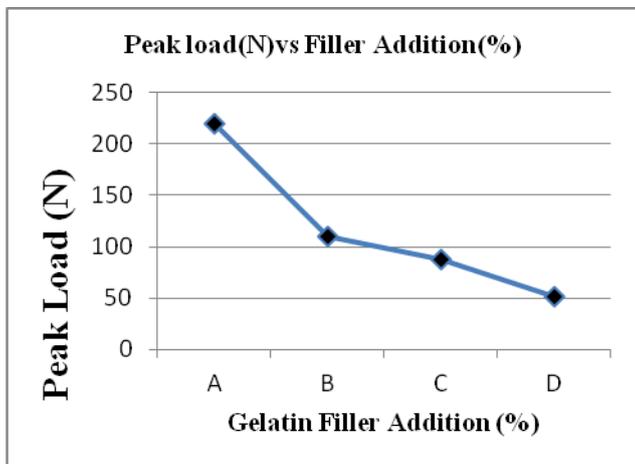
The prepared flexural test specimens before and after the flexural test experiment is shown in the figure-8



**Fig-8:** Flexural Specimen Before and After Test

### 4.2.1 Effect of Peak Load

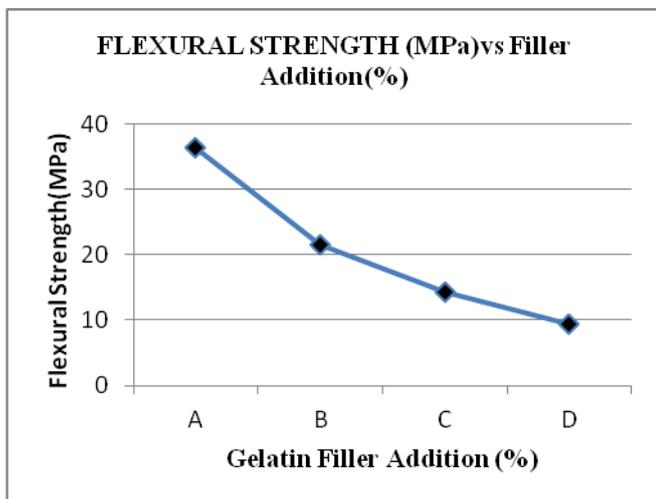
From Graph 4 it can be observed that at all filler content the highest Peak load is for 0% filler content bio-composite with a value of 219.5N



Graph-4: Peak load vs Gelatin filler

### 4.2.2 Effect of Flexural Strength

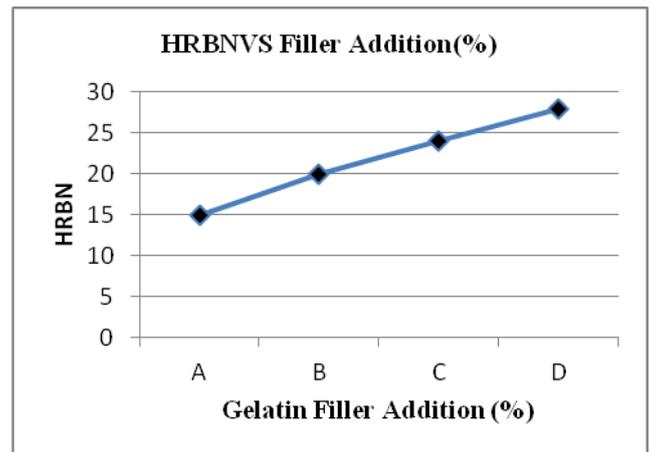
From Graph-5 it can be observed that at all filler content the highest Flexural Strength is for 0% filler content bio-composite with a value of 36.47MPa.



Graph-5: Flexural Strength vs Gelatin Filler

### 4.3 Hardness Test

Hardness is a surface property and is a measure of wear resistance on the surface of the composite. Hardness value increases from 0% filler content to 30%. The measured hardness number test results are shown in Table 6.



Graph-6: HRBN vs Gelatin filler

Table-6: Measured Hardness Number

SPECIMEN	COMPOSITION	HRBN
A	Vinylester resin + Dupion silk fiber	15
B	Vinylester resin + Dupion silk fiber + 10% Gelatin	20
C	Vinylester resin + Dupion silk fiber + 20% Gelatin	24
D	Vinylester resin + Dupion silk fiber + 30% Gelatin	28

### 4.4 Morphological Characteristics

Below figures show the micrograph of sample revealing the morphology of Gelatin Granules and silk fiber reinforcement.

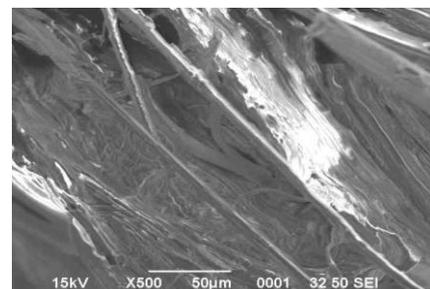


Fig-9: Gelatin Granules & Silk fibers at 500 magnification

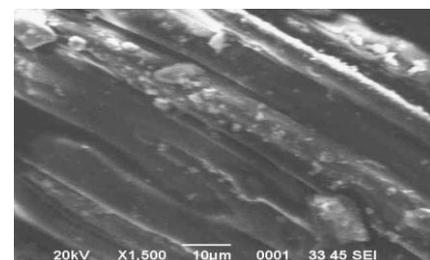


Fig-10: SEM micrograph of untreated Dupion silk fiber  
The Micrographs reveal that the bonding between reinforcement and matrix is poor and the Gelatin granules are attracted towards the fibers since both are hydrophilic in nature.

## 5. CONCLUSION

- The present work has been undertaken, with an objective to explore the potential of the natural fiber polymer Bio-composites and to study the mechanical properties of the bio-composites.
- Bio-composites can supplement and eventually replace petroleum based composite materials in several applications thus offering new agricultural, environmental, manufacturing and consumer benefits.
- In mechanical test, it was observed that 30% filler content has better tensile properties compared to the 0%,10%,20% filler content, the flexural strength is highest for 0% filler content, and it reduces for 10%,20%,30%.
- The hardness test results shows that 30% filler content Biocomposites has better Hardness number.
- In this work Tensile strength of 30% Gelatin filled Natural (Dupion silk) fiber reinforcement vinylester composite material matches the femur bone tensile strength.

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## BIOGRAPHIES



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