

## A Study on Mechanical Properties of Vinylester Based Bio-Composite Material with Starch as a Filler Material

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### ABSTRACT

In composites a conglomeration produces material properties which are unavailable from individual constituent materials. The use of petroleum based products as constituents in polymer matrix composite has raised concerns regarding environmental issue and non-renewability of the resource. Therefore in this work an attempt has been made to develop a biocomposite material using untreated dupion silk fiber as reinforcement material and vinyl ester as matrix material with Potato Starch used as filler material by hand layup technique.

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 to the ASTM standards.

From the results of the experiments conducted on the specimen it can be concluded that the performance of 10% Starch filler content Biocomposite is satisfactory in all aspects compared to 0%, 20%, and 30% Starch filler content Biocomposites.

**Keywords** - Implantation, Biocomposite, Dupion Silk Fiber, Potato Starch

### I. INTRODUCTION

Engineering Materials are used in medical application to make devices to replace a part or a function of the body. Large numbers of polymeric materials alone and in combination with other materials are becoming increasingly significant in the field of biomaterials.

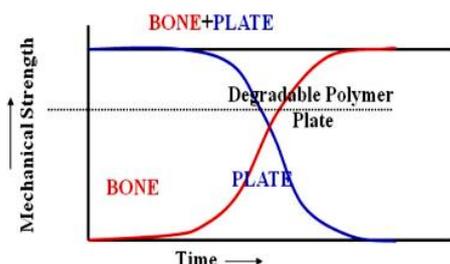
But the use of petroleum based products as constituents in polymer matrix composite has raised concerns regarding environmental issue and non-renewability of the resource. Hence the problems associated with it have initiated the efforts to develop Biocomposites. The biocomposite have one or more constituents 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.

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 (refer graph 1). 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 starch is used as fillers.

## II. PREPARATION OF BIOCOMPOSITES

### 2.1. MATERIALS

Unsaturated vinyl ester resin was selected as matrix material, Dupion silk fibers as reinforcement and Potato Starch as filler material.

Vinyl ester resins having density  $1.05 \text{ g/cm}^3$  are becoming increasingly important for fibre 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 vinyl ester resin is shown in Fig.1

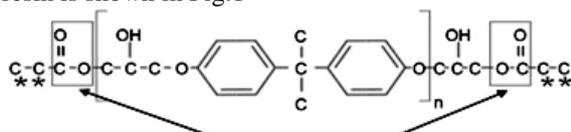


Figure 1: chemical structure of a typical vinyl ester resin

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.

Starch is the major carbohydrate reserve in plants, and represents one of the main sources of energy to sustain life. Bread, potato, rice and pasta are examples of the importance of starch in our society. Starch has also been extremely important for centuries in numerous non-food applications, for example, as glue for paper and wood, and as gum for the textile industry. Starch-based biodegradable polymers have been widely investigated in bone

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

Table 1: material procurement table

Sl. No.	MATERIALS	SUPPLIERS
1	Unsaturated Vinyl ester 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	Potato Starch	Polysales, Avenue Road Cross, Bangalore, India

### 2.2. TECHNIQUE

The laminates were prepared by the hand lay-up technique using the rectangular box. The rectangular box of dimension  $19\text{cm} \times 12\text{cm}$  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 Potato Starch (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 shown in fig. 2 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.



Figure 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.



Figure 3: prepared biocomposite laminate

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

### 2.3. SAMPLE PREPARATION CALCULATION

- Density of vinyl ester resin = 1.05 g/cm<sup>3</sup>
- Density of potato starch = 1.43 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	11.44	89.28	1.116
58.8	10.64	22.88	92.32	1.154
50.4	10.64	34.32	95.36	1.192

### III. 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.



Figure 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.

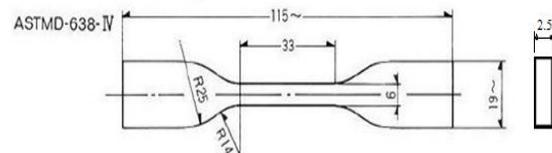


Figure 5: standard tensile specimen dimensions

### 3.2. FLEXURAL TEST

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

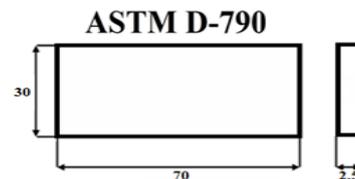


Figure 6: standard flexural specimen dimensions

### 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.

### IV. RESULT AND DISCUSSION

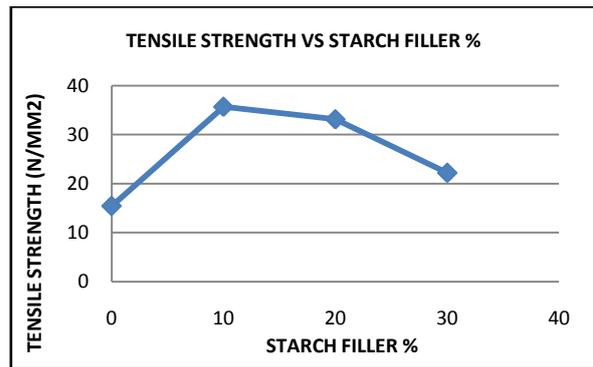
In this section the results obtained from tension, Flexural and Hardness tests are tabulated and represented graphically to analyze the behavior of Biocomposites.

### 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	TRIAL 1	313	13.55
	TRIAL 2	432	17.36
B	TRIAL 1	809	33.72
	TRIAL 2	882	37.70
C	TRIAL 1	792	37.70
	TRIAL 2	669	28.60
D	TRIAL 1	658	26.12
	TRIAL 2	451	18.33



Graph 3: tensile strength vs starch filler

The prepared tensile test specimens before and after the tensile test experiment is shown in the Fig.7

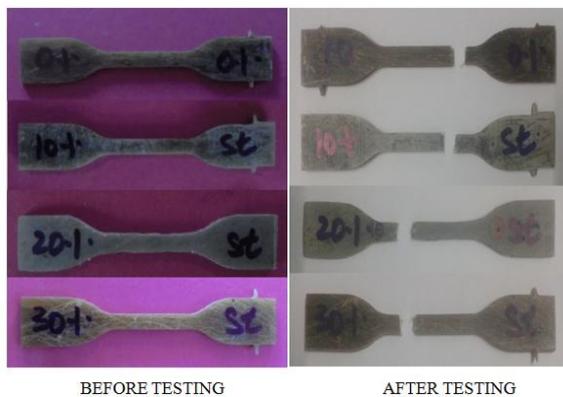


Figure 7: tensile specimen before and after test

#### 4.2. FLEXURAL TEST

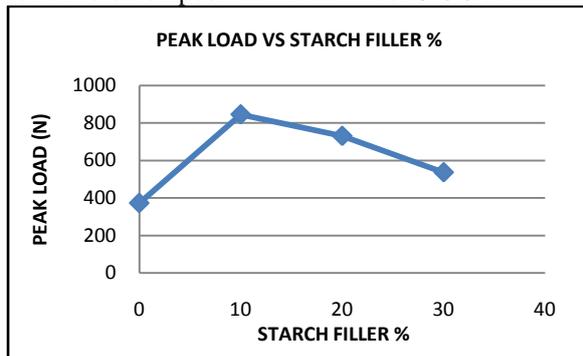
The peak load and flexural strength obtained from flexural test conducted on the prepared specimens is tabulated in table 5.

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	102	19.77
	TRIAL 2	93	18.37
C	TRIAL 1	141	30.94
	TRIAL 2	71	12.67
D	TRIAL 1	107	17.39
	TRIAL 2	108	17.60

##### 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 10% filler content bio-composite with a value of 845.5 N.



Graph 2: peak load vs starch 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 10% filler content bio-composite with a value of 35.71 Mpa.

The prepared flexural test specimens before and after the flexural test experiment is shown in the fig.8

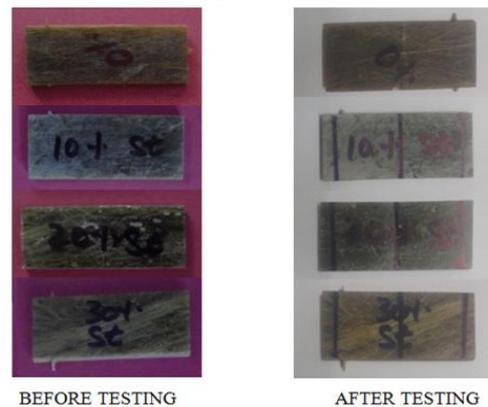
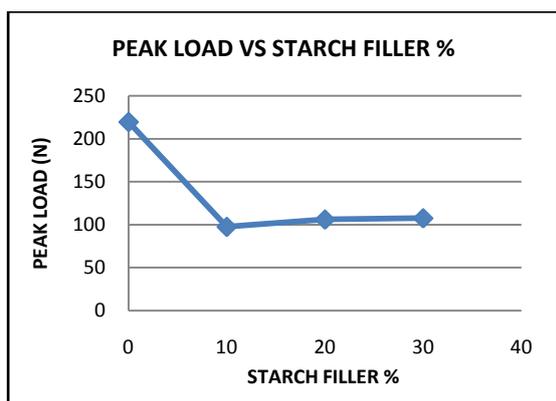


Figure 8: flexural specimen before and after test

##### 4.2.1. EFFECT OF PEAK LOAD

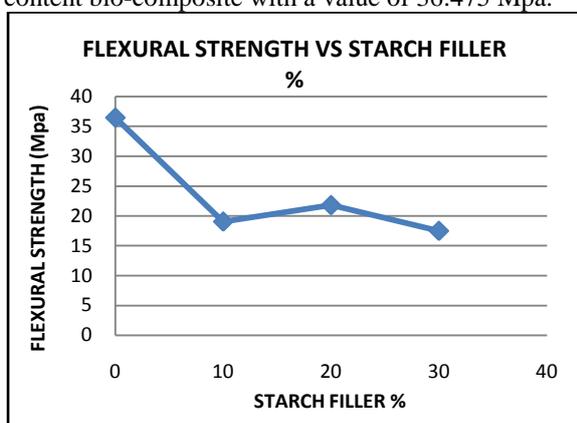
From Graph 4 it can be observed that at all filler content the highest Peak load is for 0% filler content bio-composite with a value of 219.5N. For other combinations the value of peak load is almost the same.



Graph 4: peak load vs starch filler

#### 4.2.2. EFFECT OF FLEXURAL STRENGTH

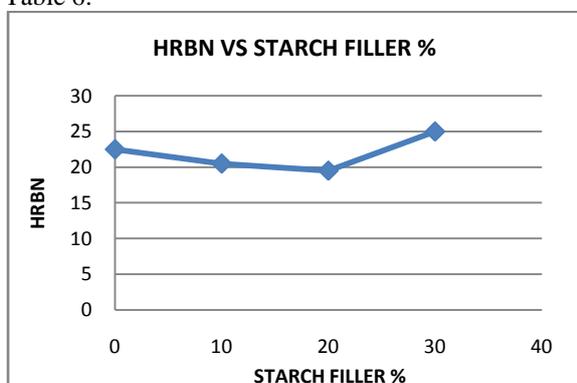
From Graph 5 it can be observed that at all filler content the highest Flexural Strength is for 0% filler content bio-composite with a value of 36.475 Mpa.



Graph 5: Flexural Strength vs Starch Filler

#### 4.3. HARDNESS TEST

Hardness is a surface property and is a measure of wear resistance on the surface of the composite. From Graph 6 it can be observed that the hardness value reduces from 0% filler content up to 20% and then increases from that point. The measured hardness number test results are shown in Table 6.



Graph 6: HRBN vs starch filler

Table 6: measured hardness number

SPECIMEN	COMPOSITION	HRBN
A	Vinylester resin + Dupion silk fiber	22.5
B	Vinylester resin + Dupion silk fiber + 10% Starch	20.5
C	Vinylester resin + Dupion silk fiber + 20% Starch	19.5
D	Vinylester resin + Dupion silk fiber + 30% Starch	25

#### 4.4. MORPHOLOGICAL CHARACTERISTICS

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

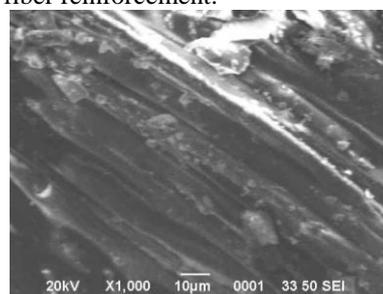


Figure 9: Potato Starch Granules & Silk fibers at 1000 magnification

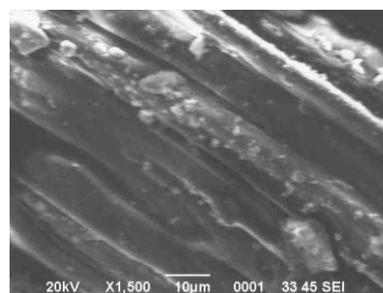


Figure 10: SEM micrograph of untreated Dupion silk fiber

The Micrographs reveal that the bonding between reinforcement and matrix is poor and the starch granules are attracted towards the fibers since both are hydrophilic in nature.

#### V. CONCLUSION

- Bio-composites can supplement and eventually replace petroleum based composite materials in several applications thus offering new agricultural, environmental, manufacturing and consumer benefits.
- MECHANICAL TEST
  - Tensile test - For all the Bio-composites tested, it is observed that the Peak Load and tensile strength increases up to 10% filler

content then it starts to reduce from that point for higher filler percentage. Hence 10% filler content has better tensile properties compared to the 0%, 20%, 30% filler content.

- Flexural test - the flexural strength is highest for 0% filler content, it reduces and remains almost constant for 10%, 20%, and 30%.
- Rockwell Hardness test - The hardness test results shows that 30% filler content Biocomposites has better Hardness number.
- Therefore from the results of experiments it can be concluded that the performance of 10% filler content Biocomposites is satisfactory in all aspects compared to all other combination.

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