

# SYNTHESIS AND ANALYSIS OF BIOCOMPOSITES USING BEESWAX-STARCH-ACTIVATED CARBON & HYDROXYAPATITE

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## ABSTRACT

*Biocomposites are synthesized by two or more materials having bio source. These materials have attributes that eliminate the weaknesses of individual materials while combining the strength. The present work is aimed at exploring the possible biocomposite synthesis, by using various proportions of biomaterials originating from agro source. Two types of bio composites have been synthesized, CBS comprising carbon, beeswax and starch whereas in CBSH, hydroxyapatite is also employed in addition to other materials. Based on visual observation, texture and other apparent physical properties, two samples have been selected for FTIR analysis. Based on the interpretation of which, two more samples have been synthesized for detailed analytical study that included compressive strength, SEM and EDX. Based on the interpretation of the findings of these analytical methods, it can be concluded that the present work has successfully synthesized these biocomposites. The compressive strength is obtained in the range of 7-7.7 MPa which is comparable with light weight brick. The added advantages of these biocomposites are low cost raw materials, light weight and biodegradability.*

**Keywords:** *Biocomposite, Beeswax, Starch, Activated carbon, Hydroxyapatite, Compressive strength*

## I. INTRODUCTION

Composite materials are entering in various areas of applications ranging from biomedical to aeronautics. These products are providing solutions to many unsolved problems of mankind from human body parts to heat resistant material for space shuttle. Composite materials are made from combination of various proportions of components so as to give a product that will represent the better attributes and discarding the deficiencies of individual components. The challenge to designer/researcher is thus to reach the optimal proportion of individual components to arrive at better product. This is relatively newer field and lot of research is been conducted which is leading to products with lower cost, biodegradability and better qualities.

## II. INTRODUCTION TO PRESENT WORK

Present work is aimed at the synthesis of a biodegradable composite from the combinations of activated carbon, starch, beeswax and hydroxyapatite.

Activated carbon is a solid, porous, carbonaceous material prepared by carbonizing and activating organic substances. Carbonization is possible for materials like sawdust, peat, lignite, coal, cellulose residues, coconut shells, petroleum coke, etc., at high temperature with or without addition of inorganic salts in a stream of activating gases such as steam or carbon dioxide they can be activated. Alternatively,

the mixture of carbonaceous matter treated with chemical activating agents is carbonized at elevated temperature and then by water washing chemical activating agents are removed. It can be used as adsorbent or decolorizing agent. <sup>[1]</sup>Starch is a polymer and may contain about 25% and 75% amylose and amylopectin respectively, depending upon its source for example, wheat, corn and potato etc. High-amylose starch is a very efficient film-forming material as it ameliorates mechanical strength including tensile strength and gas barrier properties. It is economically important to explore possible ways of improving functional properties of starch by blending it with some other materials such as beeswax, activated carbon powder, hydroxyapatite, etc. so that it can be used successfully in various areas of application. <sup>[2]</sup>Beeswax, with its unique biologically active characteristics, is one of Nature's oldest ingredients. It can be used as a thickening agent, emollient, emulsifier and humectant. From colorless liquid it turns to semi-solid substance on contact with the atmosphere. Beeswax is an inert material and it shows high plasticity at relatively low temperature (around 32 °C). Upon heating the physical properties of wax changes. It becomes plastic at 30-35°C and at 46-47°C the structure of a hard body is destroyed and between 60 to 70°C it starts melting. Heating to 95-105 °C leads to formation of surface foam, while at 140°C the volatile fractions start evaporating. After cooling down beeswax shrinks by about 10 % heating at 120°C for at least 30 minutes the remaining water is removed which causes an increase of hardness. <sup>[3]</sup> The utilization of Hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , HAp) as an important inorganic biomaterial has brought it under the limelight of contemporary researchers in the biomaterials field. The similarity in the chemical and structural properties with the mineral phase of bone and teeth render HAp to be widely used for hard tissues repair. Hence properties such as bioactivity, mechanical strength, solubility and sinterability are controlled by controlling its composition, morphology and particle size.

Findings of a literature survey are summarized.

Hydrophobic beeswax emulsion was incorporated to hydrophilic starch film to modify physical, mechanical and thermal properties of films. The researchers concluded that beeswax addition at the level of 40% concentration causes formation of amylose-lipid complex that leads to the sudden changes of physical and thermal properties of the films.<sup>[1]</sup>

Production of properties of spin-coated cassava starch-glycerol-beeswax films is reported. Glycerol, starch, water and lipid ethanol solution were heated in order to produce starch based polymer films containing glycerol as a plasticizer and different lipids as additives. Further heating under vacuum and drying under controlled condition resulted in stable films having water vapour permeability increased by 150-250%.<sup>[4]</sup>

Composite films are prepared by casting wheat, starch and whey-protein in various proportions and are also characterized. It is observed that by increase in the whey-protein content, the swelling index and tensile strength increased, whereas the vapor permeability decreased slightly.<sup>[5]</sup>

Water vapour permeability and tensile properties of sodium caseinate based films studied in terms of ratio of plasticizer and lipids. Sorbitol is less effective as plasticizer than glycerol in the caseinate matrices. Presence of Oleic acid, pure or mixed with beeswax leads to the increase in elasticity, flexibility and stretchability and decreases water vapour permeability with respect to sodium caseinate films.<sup>[6]</sup>

Properties of glycerol-plasticized cassava starch-carnauba wax emulsion films were studied. By increasing wax concentration elongation improves, but eventually it impairs tensile strength and elastic modulus gives plasticizing effect. Carnauba wax reduces water solubility of films and decreases their water permeability due decrease in water solubility. Carnauba wax affects starch crystallization by forming complexes with amylose or amylopectin.<sup>[7]</sup>

The deficiencies of single layer coating are overcome by preparing bilayer coated papers. Bilayer coated papers are prepared by two separate coatings procedures using various combinations of proteins or polysaccharides along with beeswax. Increase in concentration of chitosan solution decreases the water vapour transport rate using reduced beeswax coating weight.<sup>[8]</sup>

In present work trial runs have been conducted in bio composite synthesis by using various proportions of biomaterials originating from agro source. Two types of bio composites have been synthesized CBS

comprising carbon, beeswax and starch whereas in CBSH hydroxyapatite is also employed in addition to other raw materials. Based on visual observation, texture and other apparent physical properties two samples have been selected for FTIR analysis. Based on that interpretation two more samples have been synthesized for detailed analytical study that included compressive strength, SEM and EDX. The final conclusion is based on detailed interpretation of analytical observation.

The paper presents the work with brief introduction to biocomposites followed by the materials used and relevant literature review. The next section is methodology that gives the details of the procedure, observations and interpretation of the analytical findings with appropriate discussions. Paper concludes with the relevant inference drawn based on the results and discussion. The possible areas that need to be further worked upon are highlighted in future scope section.

### III. MATERIALS AND METHODS

#### 3.1. Methodology:

The methodology which is adopted in present work is depicted in figure 1.<sup>[9]</sup>

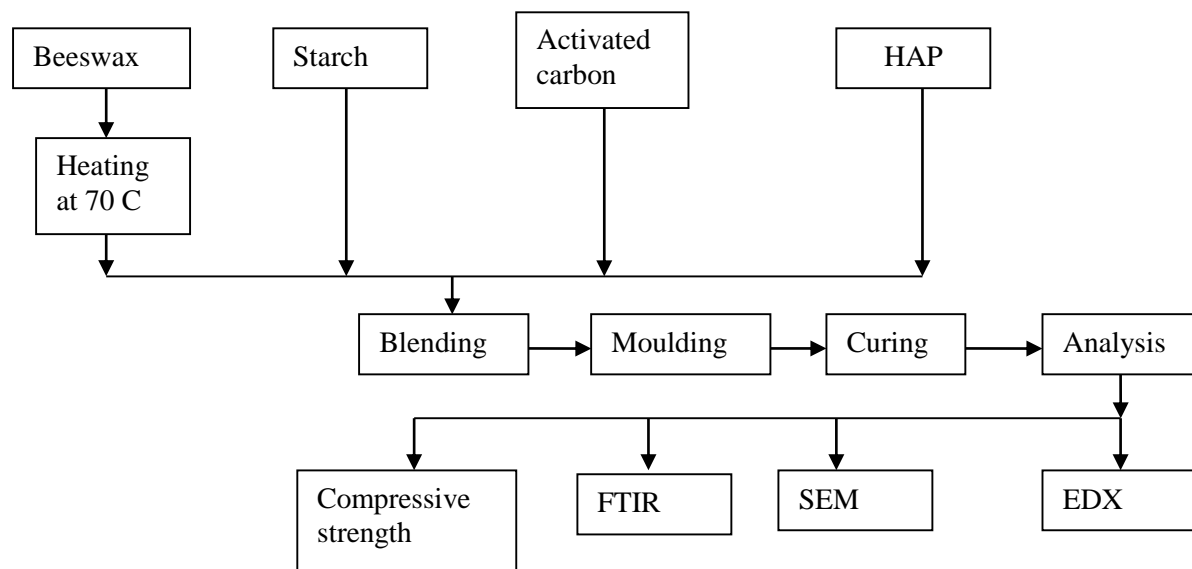


Figure 1: Flow-chart of methodology

#### 3.2. Materials:

Materials used in the preparation of the bio-composites are as follows-

- Beeswax: Procured from Agricultural College, Nagpur
- Starch: Commercial grade
- Activated carbon powder: Commercial grade, 300 mesh size.
- Hydroxyapatite: Synthesized in the lab from egg shell
- Appropriate moulds fabricated using plywood

#### 3.3. Procedure:

The method adopted for the preparation of bio composites is as follows-

- Beeswax of known quantity was taken and heated up to 60-70 °C to convert it to molten mass state.
- Known quantity of starch was added into the molten mass of beeswax.
- Known quantity of activated carbon powder was added in the slurry formed by beeswax and starch and the entire mixture was stirred.
- In case of composite CBSH, hydroxyapatite was added prior to the addition of starch powder.

- Finally the entire mixture was cured either with air or running water.
- The entire mixture was then filled into mould of required dimensions.
- Finally the solid block of composite is separated from the mould, stored and used for testing.

**3.4. Observations:**

Several trial runs have been conducted and the details of compositions as shown in table 1.

**Table 1:** Details of compositions for various biocomposites synthesized

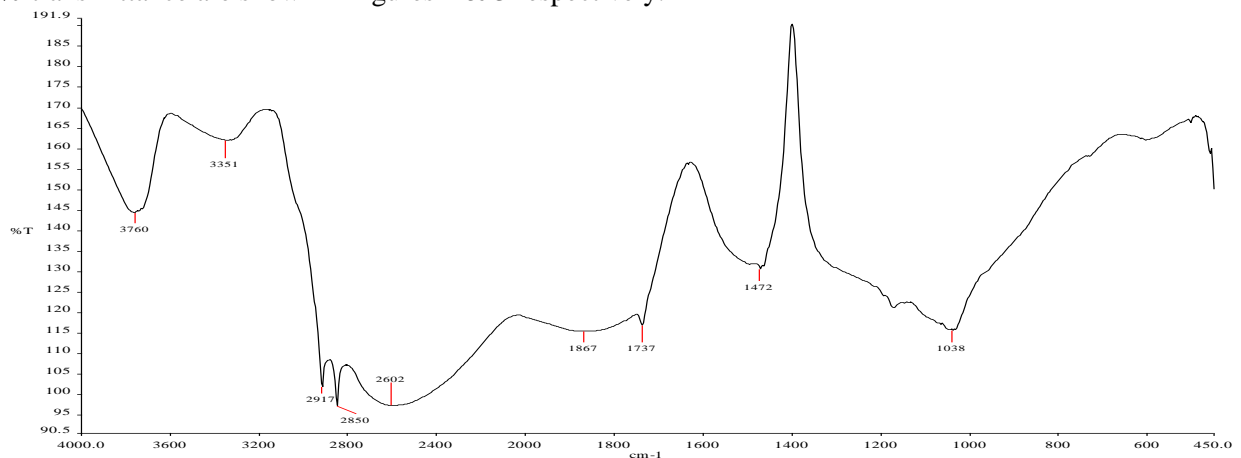
		BIOCOMPOSITE SAMPLES(WEIGHT IN GRAM)										
Sr. NO	MATERIAL	CBSH1	CBS1	CBSH2	CBS2	CBS3	CBS4	CBS5	CBS6	CBS7	CBS8	CBS 9
1	BEESWAX(B)	5	5	5	5	5.5	5.5	5.5	5	5	5	3
2	STARCH(S)	3	3	3	2	1.5	1	2	3	1	2	1
3	ACTIVATED CARBON(C)	3	3	2	3	1.5	2	1	0	2*	1*	1
4	HAP(H)	3	0	0.2	0	0	0	0	0	0	0	0

(\*graphite powder was used instead of activated carbon powder)

Based on the visual observations, texture of the surface, the apparent hardness and apparent density of two samples selected are CBSH1 and CBS1 for further analysis using FTIR.

**3.5. Analysis of samples CBSH1 and CBS1:**

The samples CBSH1 and CBS1 were analysed by FTIR and the graphs depicting wave number versus % transmittance are shown in figures 2 & 3 respectively.



**Figure 2:** FTIR of sample CBSH1

Sample CSBH1.pk

Sample~CBSH1.ASC 3551 4000 450 97 190 4 %t 8 1

REF 4000 169 2000 118 600

3760 144 3351 162 2917 101 2850 97 2602 97 1867 115 1737 116 1472 130 1038 115

End 9 peak(s)

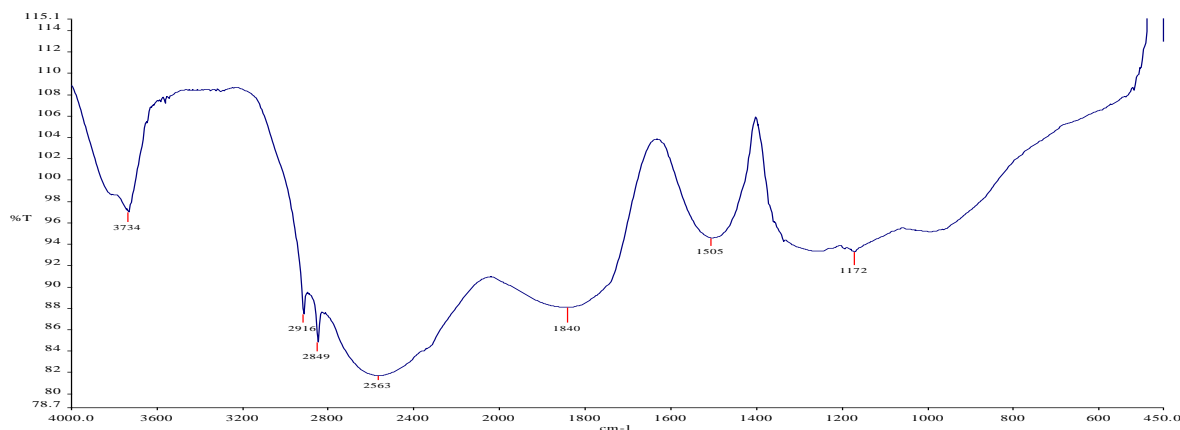


Figure 3: FTIR of sample CBS1

Sample CBS1.pk

SAMPLE~CBS1.ASC 3551 4000 450 81 121 4 %T 8 1

REF 4000 108 2000 90 600

3734 97 2916 87 2849 84 2563 81 1840 88 1505 94 1172 93 465 117

End 8 peak(s) found

The interpretation of the peaks obtained in FTIR analysis of these samples is carried out & the summary is given in table 2.<sup>[9]</sup>

Table 2: Interpretation of FTIR analysis (P-present, A- Absent)

SR.NO	WAVE NUMBER RANGE IN CM-1	WAVE NUMBER PEAKS CM-1	CBSH1	CBS1	REMARKS
1	>3700	3760,3734	P	P	O-H STRETCHING (STARCH)
2	3500-3300	3351	P	A	N-H STRETCHING (HAP)
3	2962-2853	2917, 2916	P	P	ALKANE
4	2900-2820	2850,2849	P	P	ALDEHYDES, C-H STRETCHING VIBRA IONS, TWO BANDS
5	3200-2500	2602,2563	P	P	CHELATE COMPOUNDS, ALCOHOLS AND PHENOLS
6	1870-1540	1867,1840	P	P	C=O STRETCHING BY KETONE, ALDEHYDE, ACID, ESTER, ACID HALIDES, ANHYDRIDES, AMIDES
7	1550-1500	1505	A	P	N-H BENDING VIBRATIONS SECONDARY AMIDE DILUTE SOLUTION
8	1720-1740	1737	P	A	CARBONYL STRETCHING VIBRATION (ALDEHYDE)
9	1165-1180	1172	A	P	SULPHONYL CHLORIDE
10	1485-1445	1472	P	A	ALKANE, -CH <sub>2</sub> -
11	465	465	A	P	STRONG MICROCLINE
12	1038	1038	P	A	PO <sub>4</sub>

#### IV. RESULTS AND DISCUSSION

As can be seen from the interpretation of FTIR analysis of biocomposite samples, CBSH1 & CBS1; all the functional groups which are added during synthesis have been identified in the form of peaks. This is indicative of successive binding and formation of biocomposite. It is felt necessary to conduct more rigorous tests for strengthening the claim further. For this purpose two more samples, CBSH3 & CBS10 have been synthesized having similar proportions as CBSH1 and CBS1 respectively. The details of the proportions and dimensions of CBSH3 & CBS10 are given in table 3.

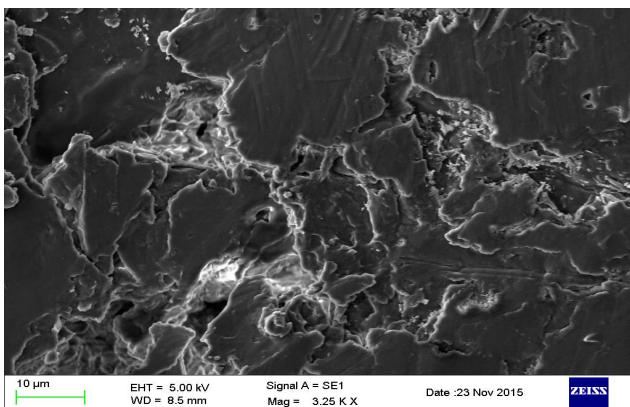
The samples CBSH3 and CBS10 are analysed further for its mechanical load bearing ability using compressive strength, spatial variance in its structure at nano-level using Scanning Electron Microscopy and elemental analysis using EDX.

**Table 3:** Details of biocomposite samples CBSH3 and CBS10

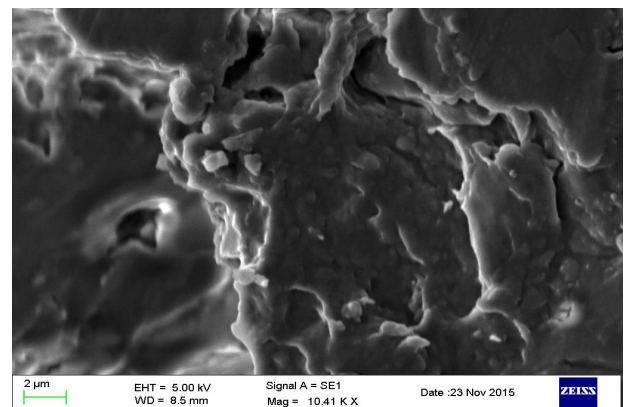
MATERIAL	BEESWAX (GRAM)	STARCH (GRAM)	ACTIVATED CARBON (GRAM)	HAP (GRAM)	DIMENSIONS L*W*H (CM)
CBSH3	6	3	3	4	2.5*2*1.2
CBS10	6	3	3	0	2.5*2*1.2

##### 4.1. SEM

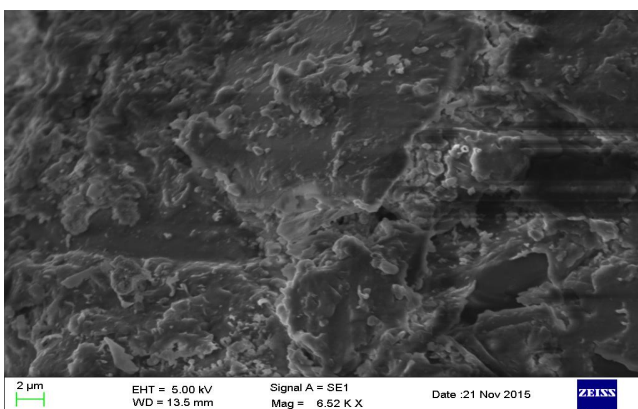
The details of the SEM photographs of samples CBSH3 and CBS10 are shown in figure 4, 5 & figure 6, 7 respectively. It can be inferred from these photographs that these samples are having fairly homogenized even surface however with interstices and voids in between. This may due to the coarse raw material particles used in synthesis and/or inadequate homogenization.



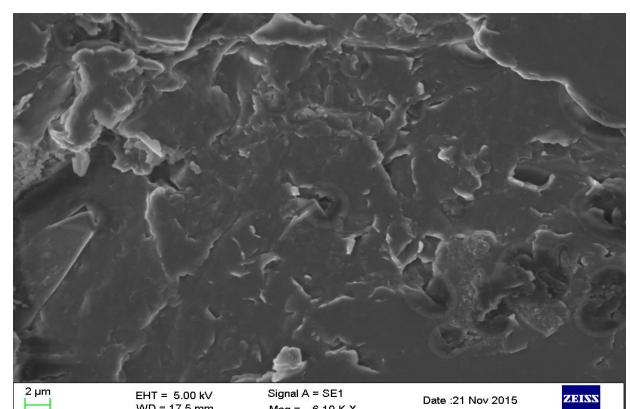
**Figure 4:** SEM of sample CBSH3



**Figure 5:** SEM of sample CBSH3



**Figure 6:** SEM of sample CBS10



**Figure 7:** SEM of sample CBS10

4.2. EDX

The details of the elemental analysis using EDX technique for samples CBSH3 and CBS10 are shown in figure 8 & figure 9 respectively. Similarly the details of elemental composition are given in table 4.

Table 4: Observations of EDX test

ELEMENTS	CBSH3 (%)	CBS10 (%)
CARBON	37	46
OXYGEN	2	1
CALCIUM	2	0
PHOSPHOROUS	1	0
NITROGEN	0	0



Figure 8: EDX of sample CBSH3



Figure 9: EDX of sample CBS10

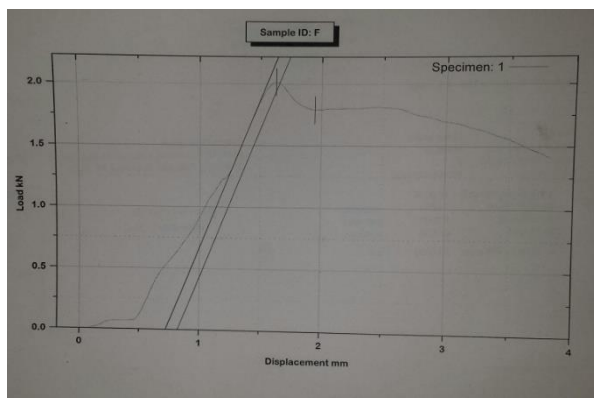
It can be seen from the EDX analysis principle elements carbon and oxygen are constituting 46% & 1% for CBS10 whereas these numbers are 37% and 2% CBSH3. Also the other constituents observed in CBSH3 are Ca-2% and Phosphorous-1%. The results are in accordance with what is expected and the presence of the Ca and P and N is due to addition of HAp.

### 4.3. Compressive strength

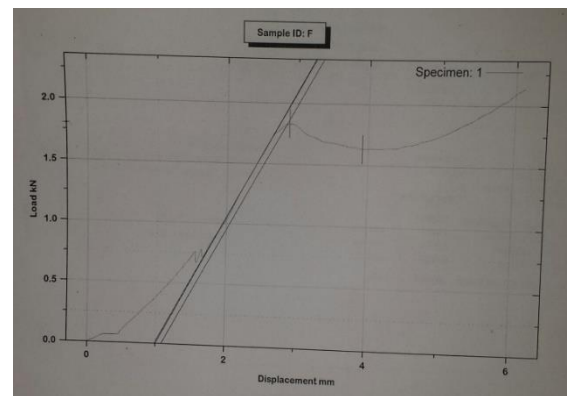
The details of the compressive strength analysis of samples CBSH3 and CBS10 are given in figure 10 & 11 and table 5. It can be inferred from this compressive strength analysis that both the samples have excellent compressive strength of 7.184 and 7.647 MPa respectively. These are indicative of excellent mechanical strength possessed by biocomposites synthesized in present work is comparable to that of light weight brick. This compressive strength also revealed the plasticity of sample and the complete breakdown of the slab did not result even at maximum applied load. The photographs of CBSH3 and CBS10 after compression are shown in figure 12 & 13 respectively. Another noticeable feature is seen CBS10 sample which has shown the increased in plasticity even after reaching its maximum yield point. The plasticity in both the samples is imparted by beeswax. The addition of HAp in CBSH3 has resulted in lower plasticity.

**Table 5:** Observations of compressive test

PARAMETERS	CBSH1	CBS1
WIDTH (MM)	20.84	19.78
HEIGHT (MM)	13.84	11.9
AREA (MM <sup>2</sup> )	288.42	235.38
LOAD AT MAXIMUM KN	2.015	1.8
COMPRESSIVE STRESS KN/MM <sup>2</sup> (MPA)	7.184	7.647



**Figure 10:** Compressive strength of CBSH3



**Figure 11:** Compressive strength of CBS10



**Figure 12:** CBSH3 after compression test



**Figure 13:** CBS10 after compression test

## V. CONCLUSION

The present work is aimed at exploration of synthesis of biocomposites comprising of biomaterials such as starch, beeswax, activated carbon and hydroxyapatite. Trial runs have been conducted and eleven samples containing two of CBSH and nine of CBS have been synthesized. Based on the visual observations and apparent physical properties one sample each of CBSH and CBS have been selected for FTIR analysis that supported the successful synthesis of biocomposites. Two additional



biocomposite samples having similar proportions of components have been synthesized and analysed using SEM, EDX, Compressive strength. Based on the interpretation of the findings of analytical methods it can be concluded that the present work has successfully synthesized biocomposites comprising beeswax, activated carbon, starch & hydroxyapatite. The visual observations and the compressive strength are indicative of the possible utilization of these biocomposites effectively. The added advantages of these biocomposites are low cost raw materials, light weight and biodegradability.

## VI. FUTURE WORK

The present work is demonstrative and it is felt necessary to conduct more number of experimental runs having different proportions of beeswax, starch, activated carbon and hydroxyapatite adequately supported with analysis. This will arrive at optimal proportion at which these are to be mixed so that the product will be imparted optimum hardness, compressive strength, lightweight & degradability at low cost. The appropriate application areas are to be identified.

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