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Investigation on thermal stability and adhesion property of chitosan based biodegradable composite

Developing a biodegradable polymer composite by using chitosan as matrix and natural fiber as reinforcement plays a vital role in order to increase mechanical properties. Chitosan has been exploited for its mucoadhesvie property which has tremendous biomedical applications in order to explore the mechanical and thermal capability. This study initialized with manufacturing of chitosan-based composite with three different natural fibers namely banana, coir, and sisal. The results of the incorporation of natural fibers with chitosan on the strength of composite blends were investigated. Further the prepared sample undergone Thermo gravimetric analysis and peel test to evaluate their thermal stability and adhesive property. The results indicate that the chitosan improves the thermal property of composites, whereas the increasing percentage of chitosan in peel test sample gets saturated hence the adhesive property gets deteriorate.

Keywords: *Chitosan, TGA, peel test, biodegradable polymer, natural fibers*

1.0 Introduction

Chitosan is a natural sources polymer generated from chitin deacetylation process. Chitin, which is present in the exoskeletons of invertebrates, is abundant in nature and easily gathered as waste and recycled for a variety of industrial applications as well as biological applications. In the domains of materials science and bioengineering, the creation of multi-functional bio-composites is a high priority issue [1, 2]. Thermal properties of chitosan increased by adding carbon nano tubes as reinforcement in chitosan composite [3]. Prashanth et al., [4] carried out an extensive work on biodegradable chitosan with sisal fiber and recommended using of natural fiber as reinforcement in the primary studies. The study reported that addition of more chitosan to the composite increase the carbon residue which gives self-extinguish property of fiber [5]. Modified chitosan with nanoparticle loading enhances the thermal and mechanical properties significantly are claimed in the study [6]. Usage of natural fibers in fiber reinforced composites influences over mechanical and thermal stability of polymer composite is discussed in many literatures [7, 8]. Venkatesha B K et al. [11] studied the effect of cenosphere as particulate filler on mechanical behaviour of woven bamboo-glass hybrid composites. It was found that the mechanical properties are significantly influenced by addition of waste ceramic filler cenosphere up to 2 wt.% and increases the mechanical properties in comparison with unfilled composites. Based on the literatures the study on adhesion property and thermal stability of fiber reinforced composites are in limited number [9]. In this study natural fiber reinforced chitosan composites are developed and following analysis are made to peel bond test and thermo gravimetric analysis.

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2.0 Materials

The composition and properties of the constituents of the biodegradable composites in this study is described below:

2.1 Chitosan

Initially weigh chitosan powder and prepare 2% acetic acid solution separately in a 250 ml beaker. Slowly adds weighed chitosan powder to acetic acid solution and stirs it until it dissolves completely. Pour the solution into Petri dish plate and keeps it for vaporization process. Specimens were prepared based on different weight ratios of 2gm, 2.5gm and 3gm. Hence it is noted as 2C, 2.5C and 3C respectively [10, 12].

2.2 Natural fiber

Three different fibers are used in this study namely coir, sisal, and banana before reinforcement alkali treatment is needed to carry out to remove moisture and to enhance the surface roughness for better bonding [14]. The steps are followed to do alkali treatment as to weigh the adequate amount of raw natural fiber and treat with 10% NaOH for 2 hours at room temperature. Further it is washed with distilled water, waited till complete drain out and kept in hot oven furnace for 48 hours at 80°C [13, 14]. The same procedure followed for all three samples.

3.0 Experimentation

3.1 Peel test

The peel test is carried out as per ASTM D903 in CIPET laboratory, Hyderabad. A set of 5 specimens with a width of 20 mm and a length of 200 mm is usually needed. These can either be cut directly from the product or manufactured in the laboratory. One of the two bonded parts should be flexible enough to bend 90° degrees without breaking.

3.2 Thermo Gravimetric Analysis

The withstand temperature of developed composite material assessed by thermo-gravimetric analysis and it was carried out using Seiko thermo-analyzer model SII7200 TG/DTA instrument, in argon flow. The rate of temperature increase was about 10°C/min, from 40°C to 800°C.

4.0 Result and discussion

4.1 Peel Test for Chitosan - Sisal

Fig.1 depicts the peel test results of chitosan - sisal composite with the chitosan percentage of 2, 2.5 and 3% of matrix content. From the plotted result it clearly implies that addition of matrix enhances the adhesion property also when the percentage of chitosan increase beyond 2.5% adhesion property deteriorates. This is mainly due to the young's modulus and brittleness of composite increases by more addition of chitosan (average Lumen size of sisal fiber ranges from 10 to 11 mm which makes impact on young's modulus) especially beyond 2.5% which leads to drop of forces by average of 55N to 32N. Fig.2 represents the comparison of peel force for chitosan - sisal composites in which 2.5% chitosan sisal sample attains maximum peel force of 58.7055N.

4.2 Peel Test for Chitosan - Banana

Fig.3 depicts the peel test results of chitosan - banana composite with the chitosan percentage of 2, 2.5 and 3% of matrix content. From the plotted result it clearly implies that addition of matrix enhances the adhesion property also the results indicate as a percentage of chitosan increases, leads to diminishes the adhesion property, but it is comparatively lower than the sisal composites. The main cause for this is the lumen size of banana fiber is 5mm which half from the sisal fiber. The max force exhibited by sisal sample 2.5 chitosan as average of 40N, further adding of fiber chitosan beyond 2.5% drop of peel force to average 37N. Fig.4 represents the

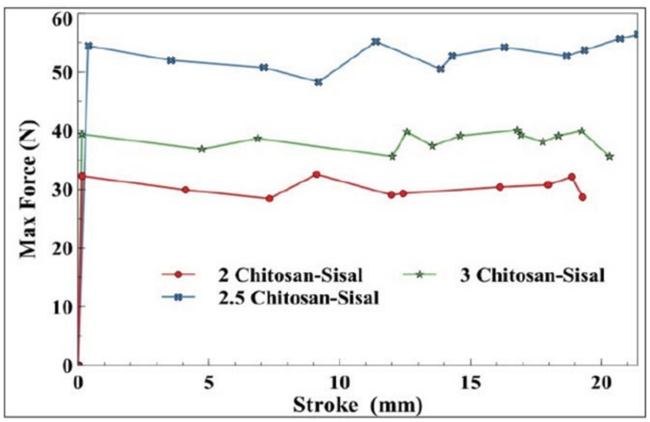

Fig.1: Peel test plots of chitosan - sisal composites

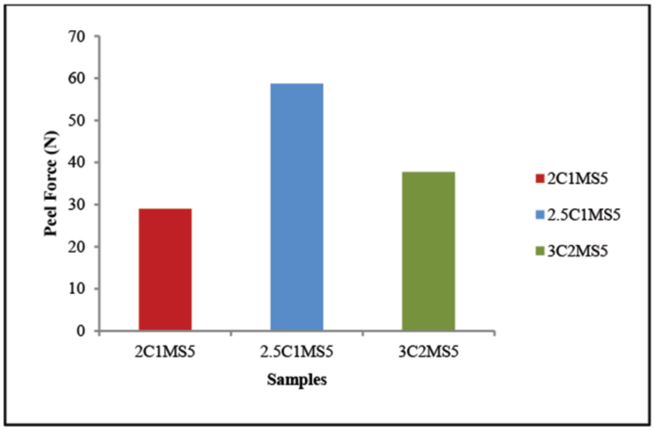

Fig.2: Comparison of peel force for chitosan - sisal composites

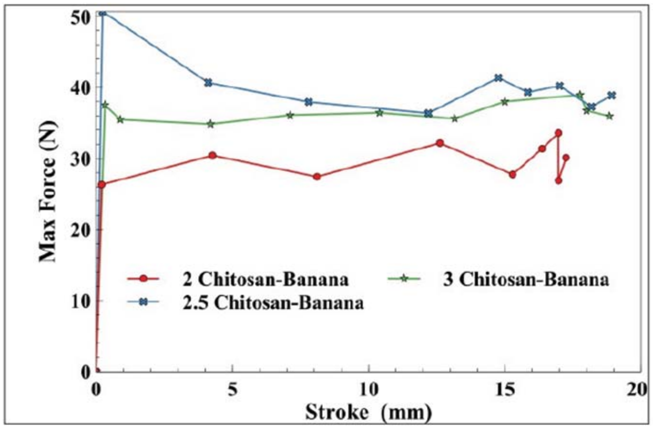

Fig.3: Peel test plots of chitosan - banana composites

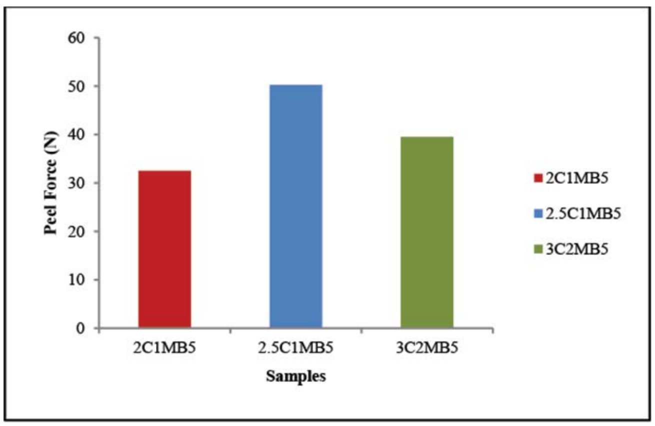

Fig.4: Comparison of peel force for chitosan - banana composites

comparison of peel force for chitosan - banana composites in which 2.5% chitosan sisal sample attains maximum peel force of 50.2673N.

4.3 Peel Test for Chitosan - Coir

Fig.5 depicts the peel test results of chitosan - coir composite with the chitosan percentage of 2, 2.5 and 3% of matrix content. From the plotted result it clearly implies that addition of matrix enhances the adhesion property; also the results indicate as a percentage of fiber increases leads to diminishes the adhesion property, but it is comparatively lower than the sisal composites. The main cause for this coir fiber is the thickest one; also it has property of moisture retention. The max force exhibited by coir sample 2.5 chitosan as average of 40N, further adding of fiber chitosan beyond 2.5% drop of peel force to average of 38N. Fig.6 represents the comparison of peel force for chitosan - coir composites in which 2.5% chitosan sisal sample attains maximum peel force of 41.153 N.

4.4 Peel Test for Chitosan

Though we have studied the composite nature of chitosan with fiber reinforcement, but it is necessary to characterize the raw chitosan property with different percentage. In such way without fibers chitosan samples are prepared and tested. Fig.7 depicts the peel test result of chitosan from the result similar trend has been noticed as like composite samples, but it is worth to note that the trend is fluctuated in nature which means that the stiffness is not adequate to the raw sample. From the result it clears the incorporation fiber which leads to the enhancement adhesion property at some extent (2.5%) after that the property get worsened. Fig.8 represents the comparison of peel force for chitosan - sisal composites in which 2.5% chitosan sample attains maximum peel force of 54.0836 N.

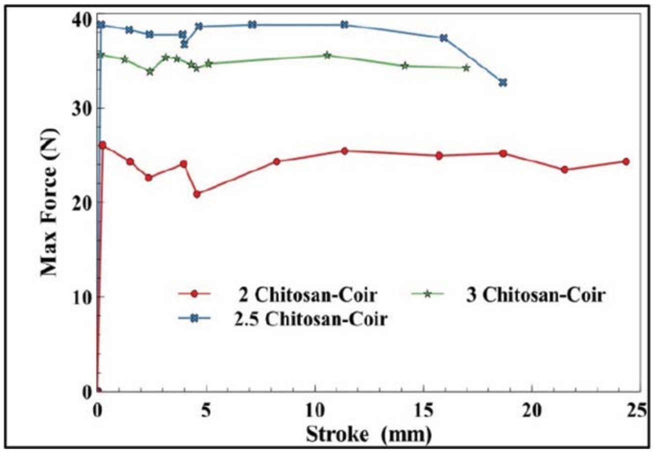

Fig.5: Peel test plots of chitosan - coir composites

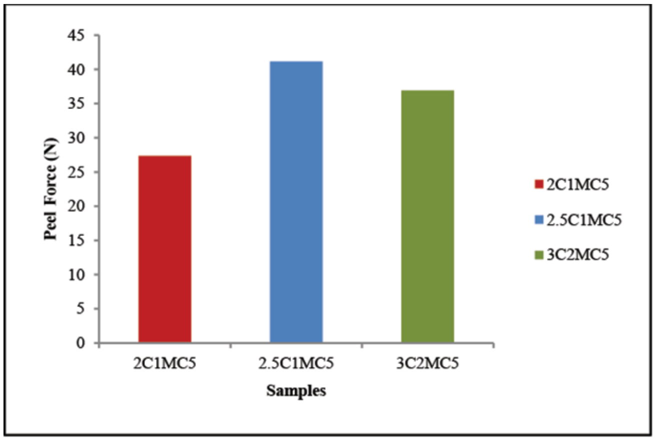

Fig.6: Comparison of Peel force for chitosan - coir composites

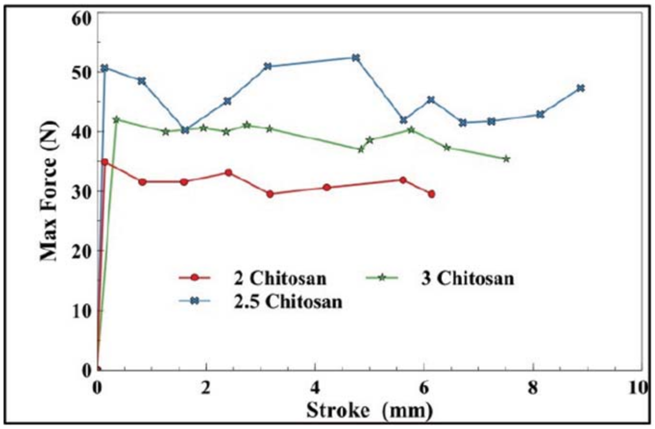

Fig.7: Peel test plots of chitosan sample

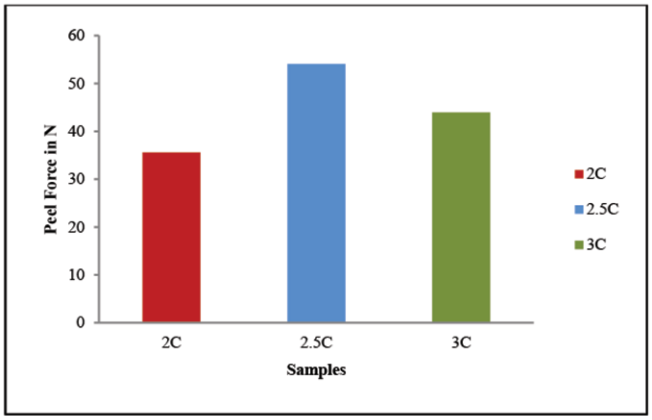

Fig.8: Comparison of peel force for chitosan sample

4.5 Peel Test for 2.5% Composites

From the overall result shown in Fig.9 the 2.5% chitosan sample possess good adhesion property out of which sisal composite exhibits average of 55N which is the maximum of all samples. Also, the fiber reinforced composite having better adhesion rather than raw sample of same percentage of matrix.

4.6 Thermo Gravimetric Analysis

Thermo gravimetric analysis (TGA) is a method of thermal analysis in which is used to analyse the changes in physical and chemical properties of materials at elevated temperatures.

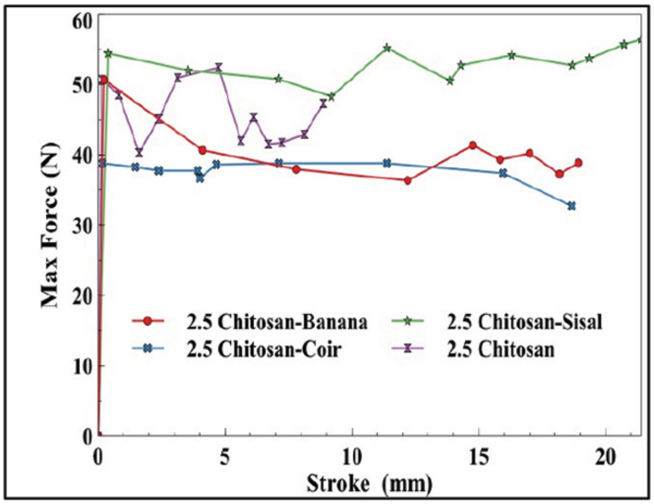

Fig.9 Peel test results of 2.5% of chitosan composite

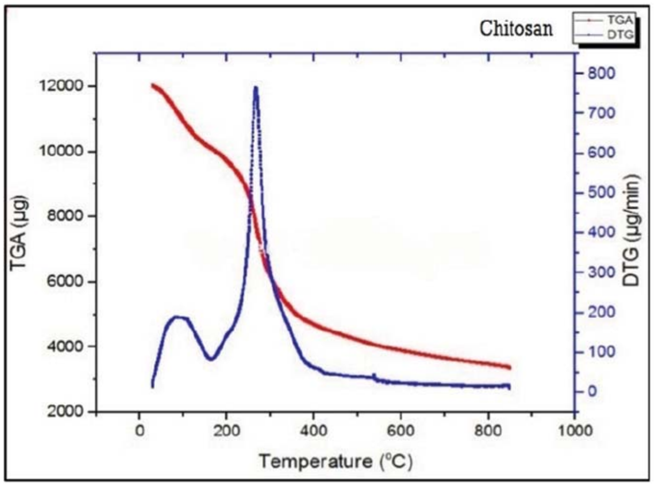

Fig.10 TGA and DTG of chitosan

The TGA curve in Figs.10 to 13 first degradation starts from 50°C and continues to above 150°C in the form of 6% loss in the weight is due to the dehydration. The prolonged weight loss of water above 100°C is due to the presence of hydrogen bonding between functional groups. The second weight loss begins at about 150°C and the corresponding 52% weight loss is attributed to the decomposition of chitosan main chains. The total weight loss of the sample at about 500°C is 58%. The remaining residue of the film which equals 42% is due to the formation of C, N and O which degrade above 800°C.

DTG is a type of thermal analysis in which the rate of material weight changes upon heating is plotted against temperature and used to simplify reading the weight versus temperature thermogram peaks which occur close together. DTG curve presented by deriv. weight (%/°C) and dashed lines, all films showed prominent pyrolysis with single step degradation and the major associated weight loss was observed from 220°C to 380°C.

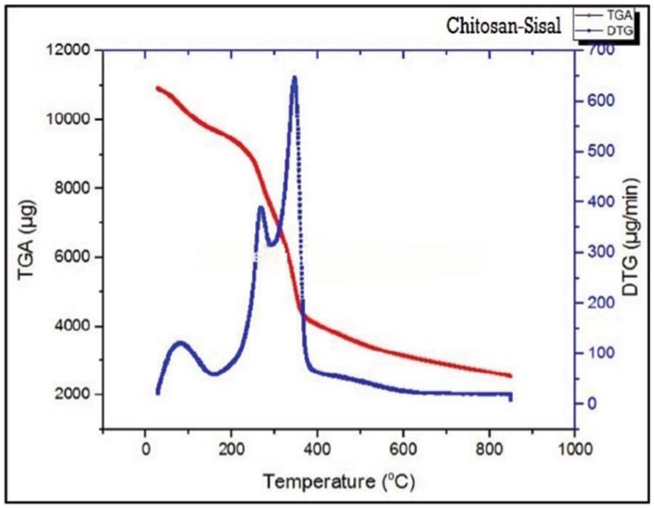

Fig.11 TGA and DTG of chitosan - sisal

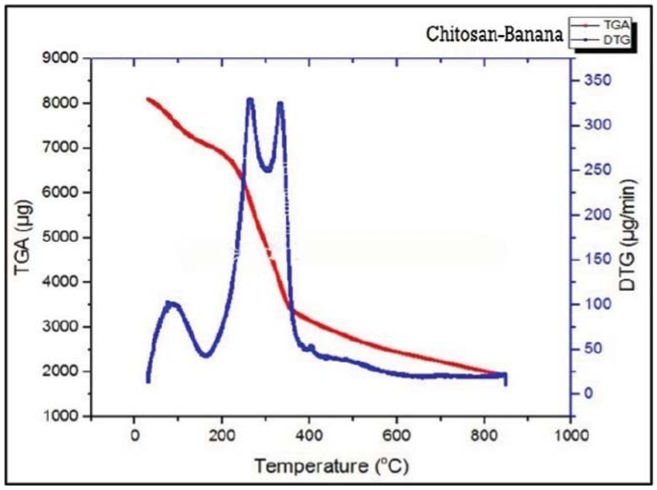

Fig.12 TGA and DTG of chitosan - banana

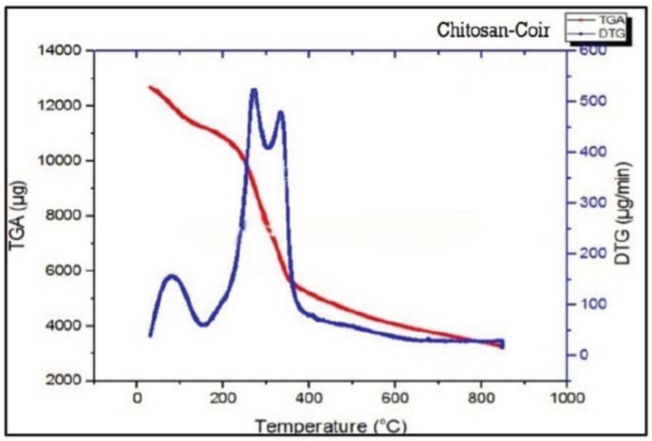

Fig.13 TGA and DTG of chitosan - coir

The TGA curve in Fig.14 depicts the weight loss (percentage) as the temperature rises. As the temperature rises, the weight percentage drops due to various reactions

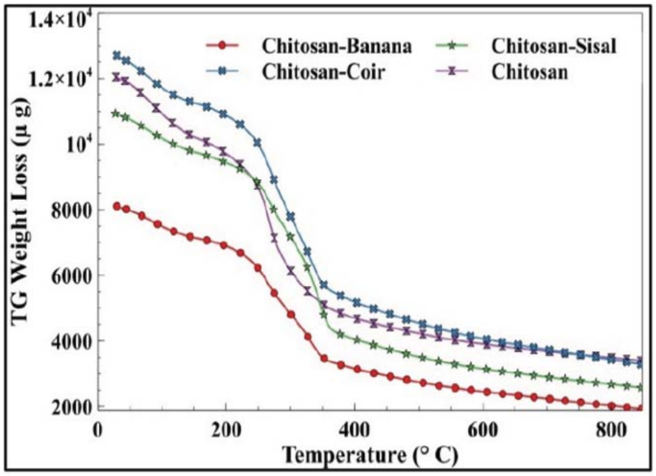

Fig.14 TGA results of 2.5% of chitosan composite

that occur as the temperature rises. The TGA curve shows that as the temperature rises, the weight per cent decreases, indicating that mass are constantly changing due to thermal treatment. In a TGA curve, breakdown begins at a lower temperature and progresses to a higher temperature when the weight percentage changes. The final segment of the curve's uniformity corresponds to the material's crystallization, and the temperature at which the material begins to crystallize varies depending on the composition. Overall, the chitosan-coir sample exhibits better thermal stability than other samples which caused by moisture retention property of coir fiber. Other sample except banana composite possess moderate performance because of banana is the light fiber compared to others.

5.0 Conclusions

The chitosan based natural composite is developed with fiber like banana, coir and sisal and the prepared sample tested to evaluate the thermal performance and adhesion capability. The concluding remarks are as follows:

- As a result of peel test 2.5 chitosan composite samples possess higher adhesion property compared to 2 and 3% chitosan composites.
- Overall result of peel test, sisal sample provides maximum peel force of 57N where the chitosan percentage is 2.5%
- Due to moisture retention property of coir fiber, chitosan - coir 2.5 % shows better thermal stability.
- As the percentage of chitosan increases leads to increase of brittleness of composite maximum of 2.5% of chitosan addition is preferred for better enhance of adhesion property.

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