PREPARATION AND CHARACTERIZATION STUDY OF BIODEGRADABLE POLYMER COMPOSITE.

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ABSTRACT: Functional polymers as biomaterials, acquired by polymerization of useful monomers and by functionalization of manufactured and characteristic polymers, keep on being an examination hotspot. The biomedical polymers display a far-going inconstancy of mechanical, physical and concoction attributes, taking into consideration the change in biocompatibility, bioactivities, boosts responsiveness, and biodegradability. The present progression of medicinal innovation results in new prerequisites for multifunctional and versatile polymeric materials. The readiness and use of different biomedical polymers, and means to cover the most energizing ongoing improvements in the field. Consequently, chitosan a modest, biodegradable and biocompatible biopolymer was blended coconut fibers. The rough fibers were pre-treated by mercerization (5% of NaOH course of action) The delayed consequences of pre-prescriptions showed that the ejection of lignin and hemicelluloses materials from the strands influenced decrease in hydrophilicity. Increase in surface smoothness, along these lines rendering the fibers suitable to be blended with chitosan. Unrefined and pre-treated strands were mixed with chitosan and cast in to films. The effects of pre-treatment of Fibers, rate structures of the blends film were evaluated by using tension test, XRD, SEM, water absorption and biodegradable test. In light of the above results chitosan coconut films organized in the present examination can be proposed as materials sensible to be created for tissue outlining applications.

Keywords – Chitosan, Coconut Fiber, XRD, SEM, Biodegradation, Water absorption.

1. INTRODUCTION
1.1 Chitosan
Chitin is a standout amongst the most plentiful normal polysaccharides on the planet and it is chiefly utilized for the creation of chitosan by a deacetylation procedure. Chitosan is a bioactive polymer with a wide assortment of uses because of its practical properties, for example, antibacterial action, non-poisonous quality, simplicity of adjustment, and biodegradability. This audit abridges the most well-known chitosan preparing strategies and features a few utilizations of chitosan in different modern and biomedical fields. At long last, ecological worries of chitosan-based films, considering the phases from crude materials extraction up to the finish of life after transfer, are likewise talked about with the point of discovering more eco-accommodating options.

1.2 Coconut Fiber
Coconut fiber is removed from the external shell of a coconut. The regular name, logical name and plant group of coconut fiber is Coir, Cocos nucifera and Arecaaceae (Palm), separately. There are two kinds of coconut filaments, dark colored fiber removed from developed coconuts and white strands extricated from youthful coconuts. Dark colored strands are thick, solid and have high scraped spot opposition. White strands are smoother and better, yet in addition weaker. Coconut filaments are business accessible in three structures, specifically bristle (long strands), sleeping pad (generally short) and decorticated (blended strands). These diverse sorts of filaments have distinctive utilizations relying on the necessity. In building, darker strands are for the most part utilized.

2. METHODOLOGY
The goal is to research the conduct of material examples under a Tensile Test, XRD, SEM, water absorption and biodegradable test. The materials to be researched on chitosan-coconut mixed film. From playing out the Tensile Test the accompanying properties will be resolved; young’s modulus, yield pressure, extreme tractable pressure, rate stretching at break, rate decrease in cross-sectional zone at crack and break pressure. This examination is utilized to decide a material’s properties, and is utilized in an extensive variety of businesses. One case of this could be to decide the Ultimate Tensile Stress of a material to be utilized for a skin embed, to check it can hold enough strain.

3. PREPARATION OF CHITOSAN-COCONUT BLENDED FILM
3.1 Mercerization of Raw coconut Fibers
- Weigh the 5 g of chopped coconut raw fiber
- Weigh out the 10 gram of NaOH flakes in a beaker.
- Prepare a 5% NaOH solution in beaker (10 gm sodium flakes).
- Treat coconut fiber with 5% NaOH (200ml) at room temperature for about 2 hours with vigorous stirring.
- The reaction mixture turned to dark brown colour.
- Mercerized fibers were filtered and thoroughly washed with water and then dried.
3.2 Preparation of coconut fiber and acetic acid solution
- Take 200 ml of water and add 4 ml of acetic acid glycial (2% acetic acid) stir the solution for 15 min
- Weigh 1 gram of coconut coconut fiber
- Chop 1 gm of mercerized coconut fiber.
- Add 1 gm of chopped coconut fiber into 2 % acetic acid (200 ml) and keep it for 5 days.

3.3 Preparation of chitosan solution
- Weigh 2 gm of chitosan powder.
- Prepare 2% acetic acid solution separately (100 ml) and
- Slowly add 2 gram of chitosan powder to acetic acid solution and stir it until it dissolve completely.

3.4 Preparation of Chitosan-coconut blended Film
- Mix 200 ml acetic acid (which contains fibers) and 100ml acidic acid (which contains chitosan powder), keep it for stirring for few hours
- Filter using strainer, pour into petri dish plate and keep it for vaporization.

4. EXPERIMENTATION
4.1 Tensile
Tensile test are performed for a couple of reasons. The outcomes of tensile tests are used as a piece of picking materials for designing applications. Tensile properties a great part of the time are fused into material points of interest to ensure quality. Tensile properties consistently are assessed in the midst of progression of new materials and techniques, with the objective that remarkable materials and systems can be examined. Finally, tensile properties as often as possible are used to predict the direct of a material under kinds of stacking other than uniaxial weight. Tensile test was conducted according to ASTM D638 for less than 1 mm thickness of films.

4.2 SEM
An ordinary SEM works at a high vacuum. The basic decide is that a light outflow is made by a suitable source, frequently a tungsten fiber or a field release weapon. The electron bar is stimulated through a high voltage (e.g.: 20 kV) and experience a plan of holes and electromagnetic central focuses to convey a thin light discharge., by then the column inspects the surface of the case by techniques for check twists (like the spot in a cathode-pillar tube "old-style" TV). Electrons are transmitted from the case by the action of the analyzing shaft and assembled by a suitably arranged identifier.
The amplifying instrument director is watching the photo on a screen. Imagine a spot on the screen looking at over the screen from left to right. At the complete of the screen, it drops down a line and breadths transversely finished yet again, the strategy being repeated down to the base of the screen.
The best approach to how the sifting electron amplifying focal point capacities (and this is the sharp piece) is that the column checking the case surface is accurately synchronized with the spot in the screen that the chairman is seeing. The electron identifier controls the splendid of the spot on the screen - as the locator "sees" more electrons from a particular segment, the screen sparkle is extended. Right when there are less electrons, the spot on the screen gets darker. These days, the screen is all things considered a propelled screen, not a glass crt, but instead the standard is the same. The enhancement of the photo is the extent of the proportion of the screen to the range of the domain kept an eye on the illustration. If the screen is 300 mm transversely finished and the separated zone on the case is 3 mm over, the enhancement is x100. To go to a higher intensification, the director channels a smaller zone; if the checked locale is 0.3 mm over, the enhancement is x 1000, soon.

4.3 X-RAY Diffraction
XRD examination relies upon important impedance of monochromatic X-bars and a crystalline case: The X-shafts are made by a cathode bar tube, filtered to convey monochromatic radiation, collimated to center, and facilitated toward the case. The collaboration of the scene bars with the case produces helpful hindrance (and a diffracted bar) when conditions satisfy Bragg's Law (nλ=2d sin θ). This law relates the wavelength of electromagnetic radiation to the diffraction point and the matrix scattering in a crystalline case.

The trademark x-shaft diffraction configuration made in a regular XRD examination gives an intriguing one of a kind finger impression of the valuable stones show in the illustration. Exactly when properly deciphered, by examination with standard reference cases and estimations, this exceptional check grants conspicuous verification of the crystalline casing.

4.4 Water Absorption
Water absorption is used to choose the proportion of water devoured under decided conditions. Components affecting water maintenance include: kind of plastic, included substances used, temperature and length of presentation. The data uncovers understanding into the execution of the materials in water or tight spots.
For the water absorption test, the cases are dried in a stove for a predefined time and temperature and after that set in desiccators to cool. Quickly in the wake of cooling the illustrations are weighed. The material is then developed in water at settled upon conditions, much of the time 23°C for 24 hours or until adjust. Examples are ousted, tapped go with a construct away free material, and weighed. Water absorption test is followed according to ASTM D5229

4.5 Biodegradable Test
Biodegradation is a kind of development of events in the midst of which materials are broken down misleadingly by microorganisms or some other characteristic ways. Most biodegradable issue includes regular materials delivered from plants, animals or fake substances which are adequately equivalent to plant and animal issue. Here are using PBS respond in due order regarding biodegradable test. Phosphate upheld saline (PBS) is a support course of action normally used as a piece of characteristic
research. The pad keeps up an unaltering pH. Generally a pH of 7.4 is kept up. The molecule centralizations of the game plan as a general rule facilitate those of the human body.

\[ \text{Weight loss} = \text{Initial weight} - \text{Final weight}. \]

5. RESULTS AND DISCUSSION

5.1 Tension test for chitosan coconut fiber.

The figure 1 (a) and (b) shows the tensile specimens of chitosan coconut fiber before and after the test. It is observed that chitosan-coconut fiber is a polymer material, and the fracture is a ductile fracture.

The plotted figure 2 shows the load versus change in length of the chitosan-coconut fiber-I specimen considered. It is observed that initially when the load is increased, the stress is directly proportional to strain. Beyond the proportional limit, the slope of the graph starts decreasing and the material attains fracture. The stress at the maximum load 18.6 N is 6.093 mm peak displacement. When the load is increased the material gets fail at a break load of 0.0 N.

![Chitosan Coconut Fiber before and after tensile test](image)

**Figure 1 (a) & (b): chitosan Coconut blended film specimen before and after tensile test**

![Graphical representation of load versus displacement for chitosan coconut blended film-I specimen](image)

**Fig 2: Graphical representation of load versus displacement for chitosan coconut blended film-I specimen**

<table>
<thead>
<tr>
<th>Sl no.</th>
<th>Test Parameters</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Peak Load</td>
<td>18.6 N</td>
</tr>
<tr>
<td>2</td>
<td>Peak displacement</td>
<td>6.093 mm</td>
</tr>
<tr>
<td>3</td>
<td>Peak Displacement Percentage</td>
<td>24.372 %</td>
</tr>
<tr>
<td>4</td>
<td>Eng. UTS</td>
<td>2.946 N/sq mm</td>
</tr>
</tbody>
</table>

The plotted figure 3 demonstrates the load versus change in length of the chitosan coconut fiber-II examples considered. It is watched that at first when the load is expanded, the pressure is straightforwardly relative to strain. Past as far as possible, the incline
of the chart begins diminishing and the material accomplishes break. The worry at the most extreme load 25.0 N is 14.214 mm top uprooting. At the point when the load is expanded the material gets fizzle at a break load of 0.0 N.

![Graphical representation of load versus displacement for chitosan coconut film-II](image)

**Fig 3: Graphical representation of load versus displacement for chitosan coconut film-II**

**Table 2: Test parameter and test results of chitosan coconut fiber-II**

<table>
<thead>
<tr>
<th>Sl no.</th>
<th>Test Parameters</th>
<th>Values</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Peak Load</td>
<td>25.0 N</td>
</tr>
<tr>
<td>2</td>
<td>Peak displacement</td>
<td>14.214 mm</td>
</tr>
<tr>
<td>3</td>
<td>Peak Displacement Percentage</td>
<td>56.856 %</td>
</tr>
<tr>
<td>4</td>
<td>Eng. UTS</td>
<td>4.411 N/sq mm</td>
</tr>
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</table>

The tensile test was led utilizing all inclusive testing machine (UTM) and plotted the chart stack versus dislodging and organized the greatest rigidity can withstand by chitosan coconut mixed film-I, the worry at the greatest load 18.6 N is 6.093 mm pinnacle relocation. At the point when the load is expanded the material gets fall flat at a break heap of 0 N. For chitosan coconut mixed film-II, the worry at the most extreme load 25.0 N is 14.214 mm crest dislodging. Tensile test shows the point when the heap is expanded the material gets come up short at a break load of 0 N.

**5.2 Morphological Study of Chitosan coconut Fiber.**

The SEM pictures of Chitosan coconut fiber are given in Figure 4(a) demonstrated the arbitrary plan of filaments and the nearness of less voids at first glance. The small strands in Figure 4(b) affirmed the expansion of chitosan demonstrates the best minimized capacity between them and observed some pores on the fiber covered with chitosan. The stacked game plan of strands and extensive thickness of the film. It was watched that unit of fiber from grid was less in chitosan-mercerized coconut fiber films. SEM may have shown of extensive interfacial bond amongst chitosan and coconut fiber.

![SEM images of Chitosan-coconut film](image)

**Fig 4 (a) and (b) SEM images of Chitosan-coconut film**

**5.3 X-Ray Diffraction Study of Chitosan coconut Fiber.**

The XRD example of Chitosan coconut fiber film is given in Figure 3. The one powerless expansive crest around 2θ= 20.6° and another solid crest around 2θ= 29.5° was watched. The pinnacle of 2θ peaks with red line which compared to calcite \([\text{Ca}(\text{CO}_3)]\) totally in the Chitosan coconut fiber film. It was noticed that the frail cooperation strands was seen because of the extraordinary unpleasantness of chitosan coconut fiber.
5.4 Water Absorption study of Chitosan-coconut fiber.

The water absorption study chitosan-coconut fiber films are given in the percentage of weight are presented. It was observed that the film lost its weight to some extent due to the presence of chitosan which is hydrophilic in nature. Change in weight was more in chitosan coconut film, due to the presence of higher amount of chitosan. The change in weight while soaking in water may due to the leaching of trapped acetic acid used for film casting process.

The gradual increase of weight in the chitosan-mercerized coconut fiber films was observed as the increase of concentration of NaOH during mercerization process. Weight loss was less for chitosan-coconut blended film due to the partial removal of hemicelluloses and lignin content. In water absorption, it can be concluded that at the water uptake behaviour is high for chitosan coconut blended film initially. After some time the rate of absorption decreases as compare to initial stage.

![Fig 5: XRD pattern Chitosan coconut fiber film](image)

5.5 Biodegradable Test study

In biodegradable test we conveyed by utilizing Phosphate supported saline (PBS). It is a cushion arrangement generally utilized as a part of natural research. The support keeps up a steady pH. Specimen is biodegradable in nature. The rate of biodegradation is slow with chitosan coconut mixed film. Initial weight of blended film specimen is 0.1896 grams.

![Fig 6: Graphical representation of water absorption (%) versus time (hours) for the Chitosan-coconut blended film specimen](image)

<table>
<thead>
<tr>
<th>No. of days</th>
<th>Weight of specimen</th>
<th>Weight loss of specimen in grams</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.1895</td>
<td>0.0001</td>
</tr>
<tr>
<td>2</td>
<td>0.1881</td>
<td>0.0015</td>
</tr>
<tr>
<td>3</td>
<td>0.187</td>
<td>0.0025</td>
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<tr>
<td>4</td>
<td>0.1864</td>
<td>0.0032</td>
</tr>
</tbody>
</table>

![Fig 7: Graphical representation of weight loss versus days for the Chitosan-coconut film specimen](image)
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REFERENCES

