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Original Article

Effective Tensile Energy Absorption of Chitosan-Poly Lactic Acid Composite using Injection Molding Process

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Abstract *- Significant amount of plastic being used has a negative effect on environmental sustainability, and plastic that has been buried in the ground is challenging to break down. A natural material that degrades quickly is biodegradable plastic. Polylactic Acid (PLA) is the primary component that is frequently utilized in the development of biodegradable polymers; however, PLA has drawbacks in large-scale production, particularly its extremely brittle character. It is important to add filler to PLA in order to improve the material's qualities due to its low elongation and toughness values. In this work, PLA was blended with chitosan at percentages of 100%, 98%, 96%, 94%, 92%, and 90% to enhance the mechanical capabilities of PLA. For chitosan, they are 0%, 2%, 4%, 6%, 8% and 10%. Chitosan and PLA are combined in an extrusion process that melts PLA and yields filaments through the use of the extrusion process. The outputs of the extrusion process are used for injection molding, after which they are melted and printed into the desired shape. The best results are obtained at a proportion of 98% PLA and 2% chitosan so that the energy absorption becomes bigger, according to the findings of the mechanical properties tests that have been conducted. The material injected at a temperature of 200°C has tighter holes than the material injected at a temperature of 175°C, as demonstrated by the microstructure test.*

Keywords *- PLA, Chitosan, Composite, Energy absorption, Injection molding.*

1. Introduction

A significant economic area that uses a lot of plastic materials is the packaging business [1-3]. Plastic is used for packaging the majority of necessities, including food and beverage packaging, office supplies, household goods, and electrical equipment. In comparison to other materials, polymer offers various benefits, such as being useful, rust-resistant, and lightweight. With an average annual consumption of 0.12 kg of plastic, Indonesia is the second-largest plastic-using nation after China [4].

Due to the difficulty of degrading plastic, which results in an accumulation of plastic trash that pollutes the environment, plastic's high use has a detrimental effect on environmental sustainability [5-8]. Since synthetic polymers, the primary component of plastics, take a very long time to break down—possibly even tens or hundreds of years—they are also difficult to break down when buried in the earth. Carbon emissions that damage the environment are created when plastic is burned [9-11]. Microorganisms that are part of the environment have an impact on the biodegradation process [12]. Temperature, oxygen saturation, and bacteria in the environment all affect how quickly biodegradation occurs [13]. Placing plastic in soil, sludge, active waste, and enzyme hydrolysis contributes to biodegradation.

Biodegradable plastic is plastic that can disintegrate naturally through the activity of microorganisms without leaving hazardous or damaging residue in the environment [14-16]. Environmentally friendly plastic packaging materials are still being developed by researchers and scientists. Several researchers have created biodegradable plastics technology, which allows plastics made from natural components to decompose quickly. PLA is a common constituent in the production of biodegradable polymers [17-20].

PLA has gained popularity in recent years due to its utilization of renewable resources. It is also popular because of its superior mechanical characteristics and biodegradability [21]. PLA's applications have grown to include food packaging, textiles, and composite materials. PLA has great development potential because of its high transparency qualities and ease of processing with existing technologies. Many investigations have been conducted on the interaction of PLA with biopolymers, one of which is chitosan.

PLA consumption ranks first among bioplastics worldwide in an endeavor to create technology in composite goods based on renewable natural resources, also known as biodegradable composites [3, 22]. However, PLA has drawbacks in large-scale manufacture, including its brittle nature (elongation ability of less than 10% when broken) and weak durability [23-25]. Similarly, applications that require this mechanical ability must be adjusted first. Therefore, the filler must be added to improve the material qualities of PLA. Adding chitosan to PLA to increase its mechanical characteristics is one technique to improve its qualities.

Because chitosan is a filler enhancer in PLA, including chitosan will boost the value of material qualities [26-27]. The most essential component in enhancing mechanical strength is the homogeneity of the filler size; the strength can improve with the amount of chitosan added to the polymer. The higher the surface area of the chitosan particles supplied, the better the adhesion at the interface.

Chitosan is a biopolymer that has commercial potential in various industrial fields [28-30]. Crab shell waste contains quite a lot of chemical compounds, including 30 – 40% protein, minerals 30 – 50%, and chitin 20 – 30% [31]. The production process for obtaining chitin from crab shells goes through several stages: demineralization, deproteinization, and lipid and pigment removal [32]. Then, the deacetylation process is carried out to obtain chitosan that meets industrial requirements [51].

Chitosan has several beneficial features, including biocompatibility, degradability, non-toxicity, and the ability to spontaneously form gels, which allows it to create membranes or films [34-35] easily. Because of the cationic character of chitosan, gel formation occurs at an acidic pH [36]. The inclusion of chitosan variants is known to boost the tensile strength of bioplastics. The use of chitosan as an additive in the production of plastic films improves the transparency of the resulting plastic films [37-38]. The more chitosan utilized, the greater the resulting bioplastic goods' mechanical characteristics and water resistance. Furthermore, chitosan is non-toxic, biodegradable, and polyelectrolytic in nature. In this situation, the addition of chitosan to bioplastics implies that there is an interaction between PLA and chitosan in the mixed film; the more chitosan that is added, the lower the value of the mechanical characteristics; in other words, adding chitosan to PLA results in brittle bioplastics [39].

The rate of polymer growth is relatively quick, owing to the amount of use and ease of processing. With effective and efficient outputs, processing ease is also required. The extrusion process is employed in film production since it is cost-effective and environmentally friendly [19]. The most prevalent method for producing plastic molds for food packaging is injection molding, which is a repetitive process in which the plastic is melted and injected into a mold cavity, where it is kept under pressure until it is released in a solid condition [40]. Injection molding is a technology frequently employed in producing plastic objects because it can produce difficult-to-form features.

Composite materials' capacity to absorb energy is crucial for increasing test subject safety. The performance of composite materials must be balanced, nevertheless, when it comes to structural applications. The ability of composites to increase toughness and energy absorption has been demonstrated by numerous studies [41-43]. However, the majority of research has focused on fiber composites, despite the fact that the process of collapse [44-45] and deformation for some forms of composites depend heavily on the placement of the particles. In conclusion, as far as we know, there aren't many published studies on using injection molding to create composite materials from different types of particulates. Moreover, impact testing is typically used in investigations discussing energy absorption [46-47], even though the material absorbs energy throughout the tensile testing procedure.

In light of the explanation given above, the researcher intends to explore the effects of mixing PLA and chitosan using an extrusion machine to create test specimens and an injection molding machine to create filaments. With an emphasis on energy absorption capacity, it is envisaged that the research will improve literacy levels and address issues with manufacturing plastic eating establishments.

2. Materials and Methods

2.1. Material

The PLA (NatureWorks, China) granules used are 1.75 mm and 0.05 mm in size. Chitosan (Qingdao Hibong Industrial Technology, China) comes from crab shells with a mesh size of 100. A local entrepreneur sells virgin coconut oil (VCO).

2.2. Production of Filament

PLA and chitosan were dried in an oven at 60°C for 10 hours. Furthermore, the stirring procedure utilizing the dry mixing method combines PLA and chitosan with the addition of VCO. After the PLA and chitosan have been combined, filaments with a diameter of 3 mm are formed using an extrusion machine.

2.3. The Injection Molding Procedure

Following the extrusion procedure, which produced thread rolls with a diameter of 0.3 mm, the extrusions were trimmed to a length of 3 mm. Then specimens were created using injection molding (Burket RN 350) at temperatures 175°C and 200°C and a pressure of 8 bar.

2.4. Tensile Test

A tensile testing procedure is then carried out using the Universal Testing Machine HT-2402 with a tensile speed of 2 mm/minute on the material that has been manufactured using an injection molding machine to create tensile test specimens in accordance with ASTM D 638-02A. The material is subjected to tensile testing until it breaks.

2.5. Impact Test

To calculate the value of a material's resistance to a specific shock load, impact testing (GOTECH series GT-7045-MD) was done at an impact speed of 3.46 m/sec. The amount of energy required to cause this fracture demonstrates the material's durability.

2.6. Morphology Characterization

A FESEM SEI Quanta 650 was used to investigate the morphology. The samples were loaded into the sample chamber after being fixed to a sample holder inside the SEM. The reflected BSE were gathered by the detector when the scanning electron beam swept across the specimen surface, and an image of the sample's surface morphology was shown.

3. Results and Discussion

3.1. Tensile Strength

A common method for evaluating the quality of a material is tensile strength testing; particularly, tensile strength testing can reveal if a material experiences a rise in stress or a change in strain. The kind of the material's elongation or strain—also known as the lengthening of the specimen caused by the applied force—allows one to observe the material's mechanical qualities. When conducting a static mechanical tensile test, the sample is loaded at both ends, where the tensile force is applied. The material is dragged throughout the test until it breaks.

Tensile strength tests that account for changes in the addition of chitosan to PLA reveal that the tensile strength is the maximum force that the plastic composite can withstand before breaking. Fig. 1 demonstrates that adding 0% (pure) and 2% chitosan to bioplastic composites increased their ultimate tensile strength (UTS); however, adding 4%, 6%, 8%, and 10% chitosan tended to reduce it. The results of mixing PLA with chitosan show interaction in the blended film. Chitosan can be added to PLA to boost its tensile strength, but when more chitosan is added, the material will either become brittle or lose some of its UTS.

The variation of 100% pure PLA was found to have the highest UTS at 175 °C, with a value of 12.19 MPa, and the variation of 100% pure PLA at 200°C, with a value of 10.87 MPa. The variation of 94% PLA and 6% chitosan was found to have the lowest UTS at 175 °C, with a value of 8.42 MPa, and the variation of 94% PLA and 6% chitosan.

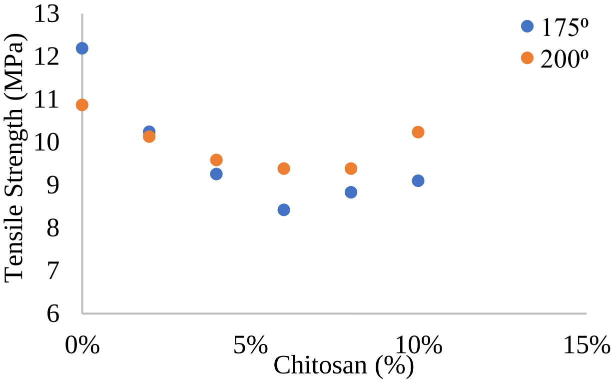

Fig. 1 Tensile strength

The results of this UTS are related to shock testing, where the amount of this value depends on the temperature of the grip and the injection pressure given. This value can be due to the fact that this tensile strength test is looking for the maximum stress that material can hold when it is stressed or pulled before finally breaking up. This process depends on the extrusion process and the injection molding process. Uneven mixing in the extrusion process causes chitosan to be poorly mixed with PLA, resulting in a UTS value that is not optimal, whereas in the injection molding process.

Due to changes in the atoms that affect the tensile strength of the polymer, an increase in temperature and pressure might result in unequal mixing of PLA and chitosan. According to the study, the UTS of PLA would decrease when chitosan is added with a filler concentration of more than 3%. To put the findings into context, additional studies using the PLA polymer and chitosan have been conducted [48]. The value of the achieved tensile strength will drop with the addition of the filler concentration. Since there is no longer any water present in the material at 200°C, chitosan cannot mix with PLA, increasing the UTS value. Hence the UTS value is higher at 200°C than it is at 175°C. This is brought on by the sample's presence of air bubbles, which causes the specimen's molecules to form unevenly bonded structures, weakening the polymer [49].

3.2. Tensile Energy Absorption

When a material experiences elastic deformation, it has the ability to absorb energy, and when the load is released, energy is also released. Resilience energy is produced in the region of the stress-strain graph before the material experiences elastic deformation. Using Eq. 1, find the large resilience as follows.

$$U = \frac{1}{2} \times \sigma \times \varepsilon \tag{1}$$

While a material attribute, the modulus of resilience (MOR) is calculated from the resilience per volume and is represented by the following equation: Eq. 2.

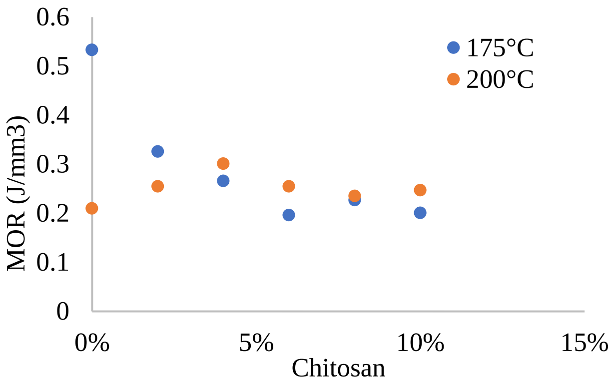

Fig. 2 Modulus of Resilience

$$MOR = \frac{Resilience}{Volume} \tag{2}$$

The ratio of energy resilience to chitosan addition can be calculated using the results mentioned above, as illustrated in Fig. 2.

According to Fig. 2, chitosan is more resilient than other materials. The highest energy resilience, measured as 0.533 J/mm^3, is found in pure PLA specimens molded at 175°C because chitosan has a higher yield strength at 0%. The specimens with the lowest resistance, 0.196 J/mm3, had a chitosan content of 6% and were molded at a temperature of 175°C. Due to chitosan's 6% lowest yield strength and 175°C injection molding temperature, this is the case.

The capacity of a substance to withstand energy until it breaks is known as energy toughness. In this toughness test, the energy absorbed per unit area is calculated as the crack propagation rate. The area under the stress-strain curve, which is calculated using equation (3), provides the toughness energy.

$$MOT = \left(\frac{1}{2}\varepsilon_n \times \sigma_n\right) \tag{3}$$

Calculated using the triangle's area, MOT on the first element's area is performed. The area up to the fracture site is calculated using MOT on the second element, and so on, by combining the areas of the triangle and square.

$$\begin{aligned} MOT = {} & \left(\frac{1}{2}\varepsilon_1 \times \sigma_1\right) \\ & + \left(\left(\frac{1}{2}(\varepsilon_2 - \varepsilon_{2-1})(\sigma_2 - \sigma_{2-1})\right)\right. \\ & \left. + \left((\sigma_{2-1})(\varepsilon_2 - \varepsilon_{2-1})\right)\right) + \cdots \\ & + \left(\left(\frac{1}{2}(\varepsilon_n - \varepsilon_{n-1})(\sigma_n - \sigma_{n-1})\right)\right. \\ & \left. + \left((\sigma_{n-1})(\varepsilon_n - \varepsilon_{n-1})\right)\right) \end{aligned} \tag{4}$$

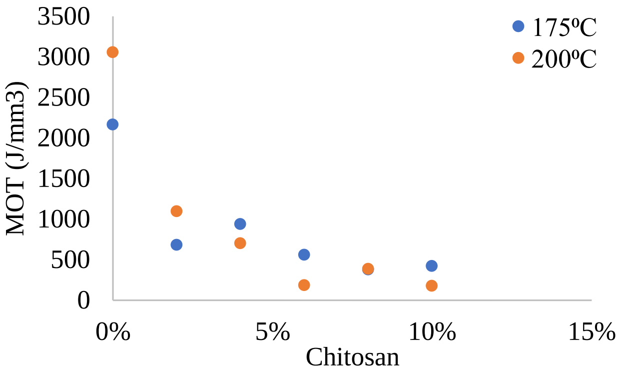

Fig. 3 Modulus of toughness

Fig. 3 shows the addition of chitosan and the quantity of energy absorbed till the material cracks.

Fig. 3 illustrates how chitosan with a proportion of 0% has stronger toughness than the others. This is due to the high amount of absorbed energy in chitosan with a 0% percentage. In this test, the PLA with the highest modulus of toughness had a chitosan content of 0% and an injection molding temperature of 200°C.

Tensile energy absorption measures a material's durability under repeated or dynamic stress or strain by measuring its capacity to absorb energy at the rate of stress-strain. Eq. 5 presents the equation for calculating TEA.

$$TEA = 1 \times 10^6 \frac{A}{LW} \tag{5}$$

The results of the test for tensile energy absorption led to the creation of the graph in Fig. 4 below, which plots tensile energy absorption against chitosan increase.

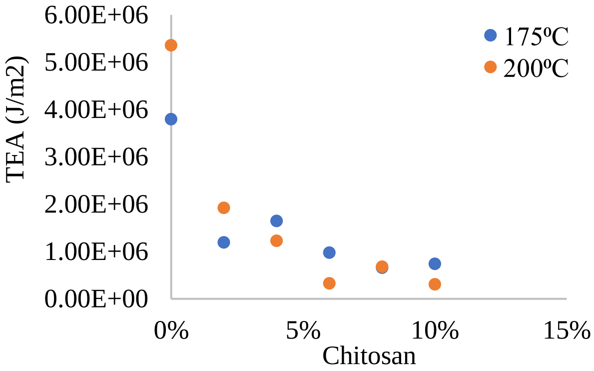

Fig. 4 Tensile energy absorption

When TEA is compared to chitosan, it can be seen that chitosan has the largest absorption energy at 0%, or an injection molding temperature of 175°C and 200°C, respectively, of 3794482.456 and 5358233.333 (J/m^2), respectively. This results from chitosan's higher underload elongation area than the others, which has a percentage of 0. Thus, in this test, the absorption energy of pure PLA without chitosan is the highest.

3.3. Impact Strength

The influence of changing the amount of chitosan in PLA on the impact price at varying temperatures between 175°C and 200°C is depicted in the graph below. The impact value can be calculated by dividing the energy needed to shatter the material by the notch's cross-sectional area. Fig. 5 depicts the chart for impact testing.

This value reflects the percentage of chitosan added, combined using an extrusion device, and the specimen is manufactured using an injection molding technique. This shock test assesses a material's capacity to withstand a sudden impact force and calculates the energy needed to break the specimen. This shock test's energy reveals how much energy the test object absorbed. Temperature influences a material's toughness; the lower the temperature, the lower the toughness value of the material, and the material will become brittle; in other words, if the low temperature causes the material's atoms to become extremely tight, there is no room for deformation, and the energy absorption will be minimal; in contrast, if the temperature increases, the material's atoms will stretch and increase the likelihood of deformation, and the energy absorption.

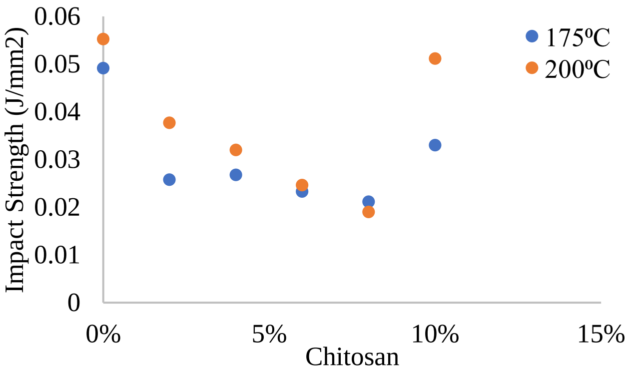

Fig. 5 Impact strength

The resistance to incoming shock loads is improved, and the softness of the material is also demonstrated by the increased shock strength value achieved on the PLA-chitosan specimen. The value of shock energy strength is influenced by the relationship between melting temperature and injection pressure. The amount of unevenly ordered atoms that are generated in the polymer will increase with increasing injection temperatures and pressures, which will lower the value of the polymer's mechanical characteristics. Since the extrusion process creates a thread-like shape that serves as the base material for the injection molding process, it has a significant impact on the shock value as well. Chitosan and polylactic acid are mixed unevenly throughout the extrusion process, which results in less-than-ideal mechanical qualities for the measured findings. This occurs because chitosan becomes stronger and harder to deform as more bonds are created when utilized [50].

3.4. Surface Morphology

The goal of surface morphology analysis is to examine the consistency of the binding between PLA and the filler, in this case, chitosan, at the microscale. The cross-sectional area was subjected to morphological investigation using a scanning electron microscope (SEM).

Fig. 6 displays the microstructure of the pure PLA used in the SEM test findings. Both demonstrate pure PLA with injection molding temperatures of 175°C and 200°C, respectively (Fig 6, *a* and Fig 6, *b*).

The characteristics of the injection molding process at a temperature of 200°C have smaller holes compared to 175°C, as can be observed from the testing results. Hence, injection molding at a temperature of 200°C has a greater energy absorption than 175°C.

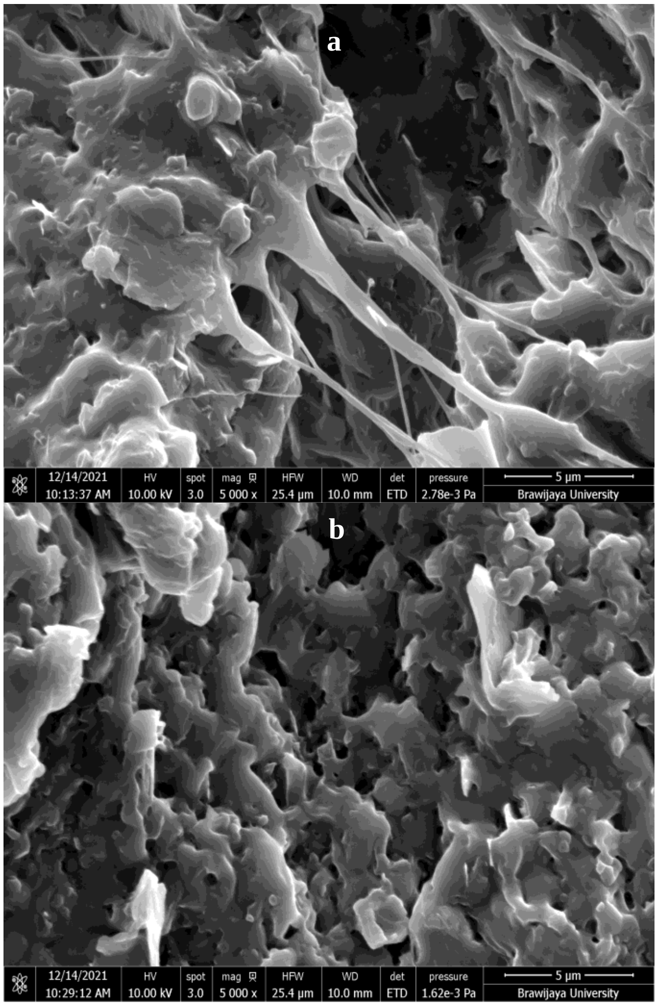

Fig. 6 Surface morphology of pure PLA (a) 175˚C (b) 200˚C

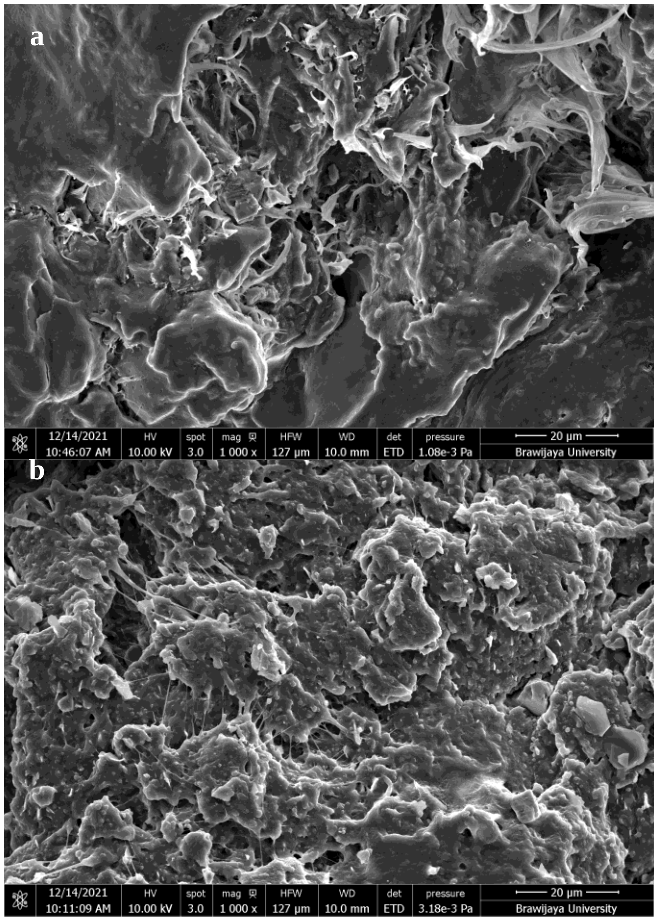

Fig. 7 Surface morphology (a) PLA 94% (b) Pure PLA

Fig. 7 depicts the surface morphology of pure PLA at a 200°C injection molding temperature without the addition of chitosan, while Fig. 7, *b* depicts a cross-section of 94% PLA at the same temperature.

The shape of the PLA and the filler, in this case chitosan, have an impact on the magnitude of the mechanical properties test value. Fig. 7 demonstrates how chitosan can join the PLA's surface, causing the PLA to become less porous. Pure PLA has a tendency to have empty holes, as illustrated in Fig. 7, *a* and Fig. 7, *b*, which can lower the value of the mechanical properties. Compared to 175°C, the pores are tighter or smaller when chitosan is added at 200°C due to their propensity to create bipolar bonds; PLA and chitosan mix because they are drawn to one of the atoms connected to one another in the bond. Due to the addition, there were no discernible boundaries in this instance. Due to the filler's addition, the addition of chitosan to PLA results in a morphology that tends not to be porous; as a result, the filler added to PLA tends to bind and mix precisely, and if more filler is added to the polymer, it can impact the mechanical properties.

4. Conclusion

It can be inferred from the findings of the study and tests that were conducted that changing the percentage of mixing chitosan with PLA has an impact on the mechanical properties of the composite, specifically based on the UTS, TEA, and impact strength values. More over 2% will reduce the value of tensile strength, TEA, and impact strength where chitosan is applied in increasing amounts.

A composition of 98% PLA and 2% chitosan yield the best result. This is caused by adding chitosan, which keeps being added and will make the substance brittle. The PLA matrix also strengthened as a result of the slow cooling caused by the increase in melting temperature during the extrusion and injection molding processes.

However, a loss in mechanical qualities will occur if chitosan is injected at an excessively high temperature since it will increase density during the injection process and prevent the material from adequately absorbing energy while testing for mechanical properties.

According to this, energy absorption increases with PLA concentration. The typical test demonstrates that, in comparison to 175°C, the pores have a tendency to be tighter or smaller when chitosan is added.

Author Contributions

Conceptualization, PHS and SW; methodology, PHS and SW.; formal analysis, PHS; investigation, FAH and DID; data curation, FAH and DID; writing—original draft preparation, PHS; writing—review and editing, PHS and DHS; visualization, FAH and PHS. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

We, the authors, declare that we have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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