

Mechanical Characteristics of Micro and Nano Silica, ZnO and Chitin Powder Filled Unsaturated Polyester Composites

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Abstract

Objectives: In this study stir processing method is used to increase the strength of polymer with the addition of reinforcements such as micro and nano silica, ZnO and chitin powder. **Methods/Statistical Analysis:** produced with this process are the production of parts having good resistance to solvents like HCl and H₂SO₄ being environmental friendly. Silica (micro and nano), chitin and ZnO were added into polyester resin in different fractions as 2%, 4%, 6% and 10% through mechanical stirrer at 1000 rpm for 1 h. Accelerator and catalyst were added and stirred using a glass rod. The influence of ZnO nano particles with the addition of silica and chitin powder on the mechanical properties like tensile strength and hardness were investigated. The comparison of different weight fractions is also studied. **Findings:** By adding ZnO nano particles with micro silica and chitin the tensile property of polyester composite was increased. The hardness increased with the addition of Silica (micro and nano) and ZnO whereas, it decreased with the addition of chitin powder. **Application/Improvements:** Investigating the effect of Silica (micro and nano) and chitin in addition to the ZnO nano particles on the mechanical and micro structure of polyester composites.

Keywords: Chitin Powder, Hardness, Polyester Composites, Resistance to Solvents, Tensile Strength

1. Introduction

The use of particulate fillers in polymers has a long history, and they continue to play a very important role today. The composites with particles as reinforcement are called particulate composites^{1,2}. If the matrix is a polymer, they are called particulate polymer composites. Polymer matrix composites are the most developed class of composite materials³ and have been accepted in a variety of aerospace and commercial applications^{4,5}. The mechanical strength, modulus etc are improved by incorporating mineral fillers into plastic resin. The mechanical properties of particulate filled polymer composites depend strongly on size, shape and distribution of filler particles in the polymer matrix⁶. In order to improve thermal, mechanical

and electrical properties of polyester⁷, particulate fillers such as alumina trihydrate, wollastonite, montmorillonite, talc, mica, silica, fly ash, kaolin etc are incorporated. To improve stiffness, modulus and to reduce costs Particulate fillers such as CaCO₃, glass fiber and carbon black are added into the polymers⁸⁻¹⁰. Fillers affect the tensile properties according to their packing characteristics, size and interfacial bonding¹¹. Polymer properties can be greatly improved by the presence of nanosized clay particles. The incorporation of nanosize particles like TiO₂¹², SiO₂¹³, ZrO₂¹⁴, SiC¹⁵, Si₃N₄^{16,17} and Al₂O₃^{18,19} to polymer matrix has lead to better enhancement in wear resistance. Particles such as mica, CaCO₃ and bamboo powders are being used as reinforcing materials for preparing particulate composites²⁰⁻²².

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2. Experimental

2.1 Materials Used

- 0) Polyester resin
- 1) Methyl Ethyl Ketone Peroxide (MEKP) as catalyst
- 2) Cobalt Octoate as accelerator
- 3) Stirrer
- 4) Mixing container
- 5) Filler materials (silica, ZnO, Chitin)

2.2 Preparation

The process of preparing the polyester composites is simple and can be done at room temperature. The curing of polyester resin is an exothermic process. The gel time and cure time are both dependent on three parameters:

- (1) the percentage of MEK-P catalyst used;
- (2) temperatures of all ingredients and the surrounding environment;
- (3) Mass concentration of the resin.

Polyester resin is most commonly used in the manufacturing of boats, surfboards, kayaks and other nautical vessels. Because of its easy application and durability, polyester resin is also applied to patch cracks and holes that need to remain watertight. Polyester resin comes in a wide range of colors and finishes to customize any project.

The experiment is done in a well ventilated area. Do not mix the resin near a heat source or under direct heat. It will cause chemical reaction. Required amount of resin is taken in a container. 1 wt% of catalyst and 1 wt% of accelerator are measured and kept aside. Various amounts of filler materials were taken. In the present work, 2, 4, 6 and 10 wt% of various filler materials were used.

Silica (micro and nano), ZnO and Chitin of definite proportions were added into the resin to produce conventionally filled composites and nano composites, respectively. It was mixed well using a mechanical stirrer at 1000 rpm for 1 h. Accelerator and catalyst were added and stirred using a glass rod. Then the slurry was casted in a mould of required shape. The curing starts in 15 minutes after the addition of catalyst and gets completed within 24 hours at room temperature.

Catalysts are added to the resin system shortly before use to initiate the polymerization reaction. The catalyst does not take part in the chemical reaction but simply activates the process. An accelerator is added to the catalyzed resin to enable the reaction to proceed at workshop

temperature and/or at a greater rate. Since accelerators have little influence on the resin in the absence of a catalyst they are sometimes added to the resin by the polyester manufacturer to create a 'pre-accelerated' resin.

Great care is needed in the preparation of the resin mix prior to molding. The resin and any additives must be carefully stirred to disperse all the components evenly before the catalyst is added. This stirring must be thorough and careful as any air introduced into the resin mix affects the quality of the final molding. This is especially so when laminating with layers of reinforcing materials as air bubbles can be formed within the resultant laminate which can weaken the structure²³. It is also important to add the accelerator and catalyst in carefully measured amounts to control the polymerization reaction to give the best material properties. Too much catalyst will cause too rapid a gelation time, whereas too little catalyst will result in under-cure. Coloring of the resin mix can be carried out with pigments. The choice of a suitable pigment material, even though only added at about 3% resin weight, must be carefully considered as it is easy to affect the curing reaction and degrade the final laminate by use of unsuitable pigments.

3. Testing Procedure

3.1 Morphology Studies

Scanning electron microscope and optical microscope images were used to study the fabricated composite specimens. The digitized images were recorded. Scanning electron and Optical microscopy were carried out to study the microstructure, particle distribution in the matrix and fracture surfaces of the composite materials.

3.2 Tensile Test

The tensile test specimens were prepared by using a standard cutting die of 80×12.5×4 mm. The tensile test was conducted on an Instron Universal Testing Machine (UTM). Crosshead speed of 50mm/min was used in tensile test. The obtained value was an average of 3 specimens.

3.3 Hardness test

The hardness test for composites was carried out by a Shore D hardness tester. A hardness tester is an instrument to quantify the sense of hardness or softness we experience by physical method

3.4 Wear test

This test is conducted in order to find the wear resistance of the composite materials. Wear pins of size 8mm diameter and 27mm height were cast in a steel mould and allowed to cure for 24h at room temperature

3.5 Solvent Resistance Test

This test was conducted by dipping the polyester sample in a solvent and calculating the weight loss after a period of time. Here concentrated HCl and H₂SO₄ were used as the liquids. The test was conducted for 24hrs. Dry the samples in an oven and weigh them prior to the test. Place the samples in a beaker containing the solvent. Samples were weighed for every ten minutes for 2hrs. The samples were cleaned with tissue paper before measuring the weight. After the completion of 2hrs, weigh the samples for every 30 minutes. Calculate the difference in the weight loss.

4. Results and Discussion

4.1 Morphology Studies

4.1.1 SEM Studies

Scanning Electron Microscope (SEM) was used to determine the fractured surfaces of the polyester based composites. The SEM images showed that the composite which is having more filler content fractured more compared to others. The interaction between matrix and filler is good at lower concentration of the filler due to that the fracture is low.

The strong interfacial adhesion between polyester and silica can be observed at low concentrations in the Figure 1. The interaction between the Si and the polyester are good. It is also showed that the silica filler in polyester matrix is properly distributed.

4.1.2 Optical Microscopy

The optical microscopy was carried out at 4X zoom. The distribution of the silica and chitin powder in polyester matrix materials can be observed in optical microscopy in Figure 2 with the increase in the amount of silica and chitin; mechanical properties of the composites were decreased due to the agglomeration of the particles observed in the Figure 3.

4.2 Tensile Test

The tensile strength was decreased by 40% with the addition of micro silica. This may be due to the inefficient

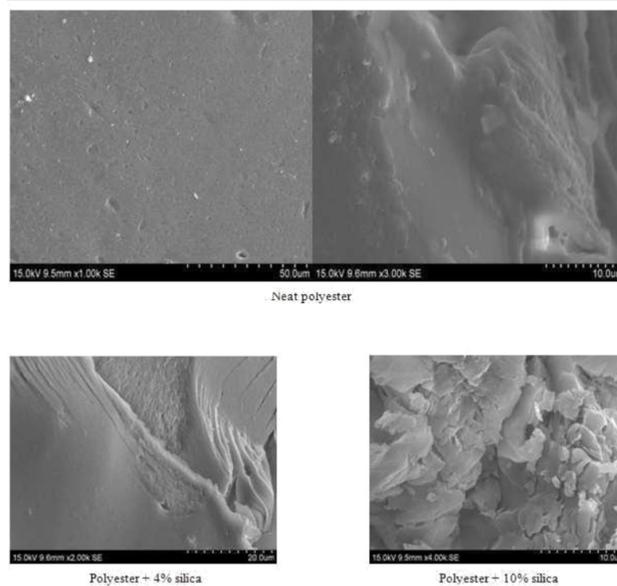


Figure 1. SEM images of micro silica filled polyester composite.

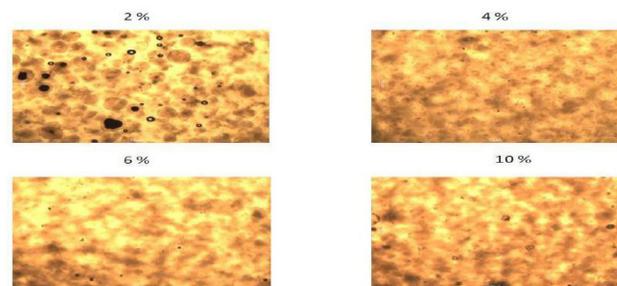


Figure 2. Optical microscope images of micro silica filled polyester.

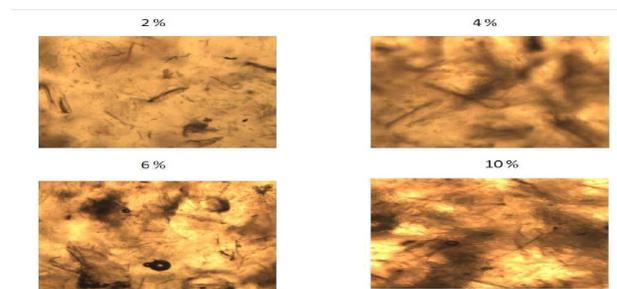


Figure 3. Optical microscope image of chitin filled polyester composite.

bonding between the silica and the matrix. The 2% of chitin shows increase in tensile strength compared to 4, 6 and 10%.this is due to the agglomeration of the chitin material at higher filler content.

The tensile strength increased by 46% for 10% of ZnO. The tensile strength of polyester and ZnO composite increased gradually with the addition of ZnO. Further investigation is to be done to find out whether the strength increases with ZnO greater than 10% observed in Figure 4.

The tensile strength was found to be increased by nearly 46% at 1 wt% of nano silica. With further addition of nano silica, the tensile strength was found to be constant at around 30 MPa. Because of agglomeration of nano silica at higher content, there is a possibility of forming pores due to increase in viscosity and hence the tensile strength decreased shown in Figure 5.

4.3 Hardness Properties

An increase in hardness value of nano composites compared to the neat polyester and the polyester filled with micro silica, ZnO and chitin clearly shown on Figure 6. The nano particles uniformly distributed in the matrix system effectively restrict the indentation. It was found that the hardness increased gradually with the addition of nano silica. The hardness was maximum at 0.6 and 1% of nano silica. In the Figure 7 observed clearly no further improvement in hardness is noticed at higher content.

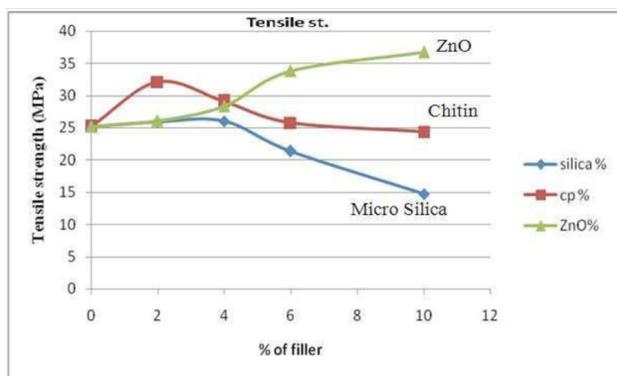


Figure 4. Variation of tensile strength with content of micro silica, ZnO and chitin.

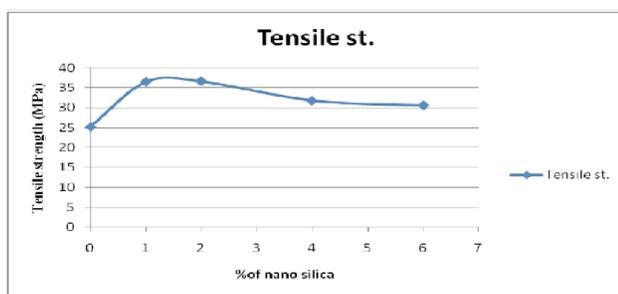


Figure 5. Variation of tensile strength with content of nano silica.

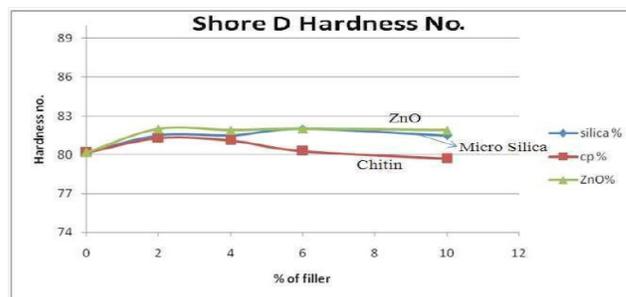


Figure 6. Variation of hardness with content of micro silica, ZnO₂ and chitin.

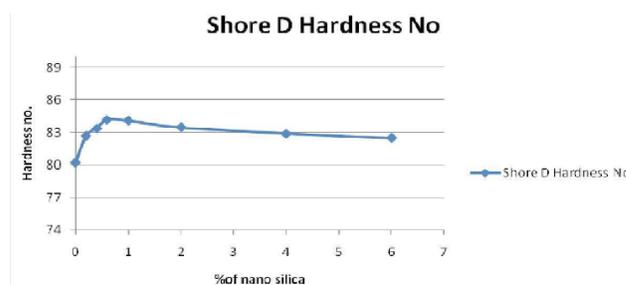


Figure 7. Variation of hardness with content of nano silica.

During processing of nano composites at high clay contents, visually it was observed that the viscosity of the resin increases significantly. So the entrapped air during mixing finds very difficult to escape out of the matrix system and remains as micro pores after curing. Another reason may be the formation of intercalated structure at high nano silica content. This may lead to the formation of nano composite structure with two phases (hard phase where intercalated nano silica is present and soft phase where the matrix alone is present). There is no significant change in the hardness of composites with other fillers used in this work. The fillers added just remain as micro tactoids and do not contribute much on the hardness improvement.

4.4 Solvent Resistance Test

From the solvent resistance test that was conducted on the fabricated composites, using concentrated HCl and H₂SO₄, it has been found that there is no weight loss of the materials. The fabricated composite materials are resistant to HCl and H₂SO₄.

5. Conclusion

A novel composite system based on the reinforcements such as micro and nano silica, ZnO and chitin powder

was prepared. The mechanical properties of polyester based composites with the addition of silica, ZnO and chitin was determined and compared. The tensile strength found to be increased with the addition of ZnO. The hardness is decreased with the addition of chitin powder and increased with the addition of Silica (micro and nano) and ZnO. The prepared composites showed good resistance to solvents like HCl and H₂SO₄. The cross linking structure of cured polyester, particle distribution in the polyester matrix and the fractured surfaces were observed by Optical Microscope and Scanning Electron Microscope.

6. References

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