

Mechanical Properties of Chitin Whisker Loaded Water Soluble Phenol Formaldehyde Nano Composites

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Abstract

Polymer based composite materials are being used extensively for the development of a wide spectrum of products. The demands for many applications need a set of properties that cannot be full fill by single polymer. One of the methods to satisfy these demands is to mix two or more polymers. The compounds so formed are generally known as polymer blends. Polymer blending has by now emerged as a well-established route to synthesize specialized polymer systems. Well known examples of blends include impact modified and toughened polymers, where polymers with different glass transition temperatures are combined. Many blends are used in the industries, where specific polar or non-polar polymers are mixed in order to increase the resistance against the transport of water vapour and gases.

In the present paper mechanical properties of the nano particle reinforced phenol formaldehyde (PF) composites are studied, for which water soluble phenol formaldehyde composites (PFC) are prepared by solvent casting technique, with varying chitin loading percentage. To optimize the chitin loading, on the bases of its mechanical properties, chitin whisker loaded samples were studied. Nano Chitin particle loaded Phenol Formaldehyde (PF) composites were prepared, varying from 0 to 1.5 % nano chitin by weight, in a variation of 0.5% chitin in the composite. The study showed

that the nano chitin loaded PF with 0.5% chitin nano particles have the highest tensile strength and the percentage elongation for the same sample were high. Hence the optimum percentage loading for the composites were decided to be for those having 0.5% chitin loading.

Keywords: *Chitin, Nano composites, Tensile Strength, Maximum force, Elongation at Break.*

INTRODUCTION

Composite materials are interesting heterogeneous systems which can be derived from polymers, metals etc. They consist of two or more elements which are combined at a macroscopic or nano level and are not miscible with each other. These are elements made of materials which maintain their identities even after the component is fully formed [1]. There are two major phases obtained in composites, the reinforcing phase and the matrix phase. The reinforcing phase in composites can be in the form of fibers, flakes, whiskers or powders and serve for giving strength while matrix phase serving as a bulk material acting as a barrier between the reinforcing particles. The two phases combine to give a material with a set of desired properties which are not available with the components alone. Composite materials allow a great versatility of design. When a specific application is contemplate, it is not only essential for the designer to know the

mechanical characteristics (as strength and stiffness) but also how the material will behave during its design life i.e. what changes are likely to happen to the composite under a given set of service conditions [2]. Particle reinforced composites have a large volume fraction of particle dispersed in the matrix and the load is shared by the particle and the matrix, but at nano level only a little amount of particle will make a huge difference in their properties.

Most commercial ceramics and many polymers are made as particle-reinforced composite. Particles are used to increase the modulus, to decrease the permeability and to increase the ductility of the reinforced composites as in the automobile tyre which has carbon black particles in a matrix of SBR (styrene butadiene rubber) [3, 4]. Another example is spherodized steel, where cementite is serving as particle, reinforced element is concrete where the aggregates (sand and gravel) are

the particles and cement is the matrix. Particle reinforced composites support higher tensile, compressive and shear stresses [5, 6].

In the present work Phenol Formaldehyde (PF) is reinforced with chitin where chitin is in the form of nano particle reinforcing phase and water soluble phenol formaldehyde is the resin serving as matrix phase. Both these elements are combined to obtain phenol formaldehyde composite (PFC). PFC composites were prepared with varying fractions of nano chitin ranging up to (0 to 1.5%) with a variation of 0.5% chitin by volume, which were prepared by acid hydrolysis and ultra-sonication process. The prepared samples were studied for their mechanical properties. The mechanical properties of sample with 0, 0.5, 1, 1.5 percent chitin volume fraction were studied and compared for their mechanical strength, elongation at break, maximum force and curing characteristics.

EXPERIMENTAL WORK

1) Preparation of Chitin Nano

Particles: Nano particles have been prepared by acid hydrolysis chemical process [7]. This includes three basic steps

a. Decalcification or Bleaching:

This process involves cleaning of particles by removing any calcium salts present (decalcification) in chitin particles obtained from the crab shells as a main source. In this the chitin particles are first boiled and stirred simultaneously in 5% KOH solution for 6hrs. The sample was subsequently kept at room temperature overnight under stirring. They were further filtered by vacuum pump and washed several times with distilled water until pH 4.

b) Deproteinization:

In this step the chitin particles are processed to make them protein free. The chitin particle suspension which was kept in 5% KOH solution for 48hrs was washed thoroughly until pH 4 to make it a neutral suspension. Chitin samples were then bleached with 17g of NaClO₂ in 1L of water containing 0.3N sodium acetate buffer, for 6 hrs at 80°C. The bleaching solution was changed for every 2 hrs following by abundant rinsing of sample with distilled water. After the sample was bleached, the suspension was kept in a 5% KOH solution for 48 hrs to remove any residual proteins.

2) Whisker Formation:

In this step the micro particles are formed into nano whiskers by acid hydrolysis method.

Protein free whisker suspension was prepared by hydrolyzing the purified chitin sample with 3N HCL at the boil for 90min under stirring. The ratio of 3N HCL to chitin was 30cm³/g. After acid hydrolysis, the suspension was diluted by transferring them to dialysis bag and dialyzed in running water for 2hrs and then overnight in distilled water until pH 4 was reached. This dispersion of whisker was completed by a further 5min ultrasonic treatment for every 30 cm³ aliquot. It was subsequently stored at 600C temperature after adding sodium azotize (to impregnate with nitrogen) to avoid bacterial growth until used.

3) Preparation of composites:

Phenol formaldehyde sheets of certain thickness can be prepared by solvent casting process. Water soluble Phenol formaldehyde has been used for the preparing thin sheets of thickness about 3mm. Phenol formaldehyde being a thermo setting polymer will cure on

heating. It has been found that the water soluble phenol formaldehyde will cure at 1300C in about 90min. In the process of heating water soluble phenol formaldehyde, it takes the shape of the mould. The water soluble phenol formaldehyde was cured in a laboratory oven which is provided with time and temperature control equipment.

The chitin whisker suspension and water soluble phenol formaldehyde resin shall be mixed in various proportions in order to obtain final suspension to obtain dry composite of varying particular volume fraction of chitin particles by thoroughly stirring in probe ultra sonicator for obtaining uniformity in particle distribution.

These samples are casted in metal dies by using solvent casting method. In this the samples were heated at a temperature of 900C for 60min. for uniform distribution of heat though out the volume of resin so that the water present in the medium is evaporated and then the temperature is increased up to 1300C to complete the chemical cross linking process.

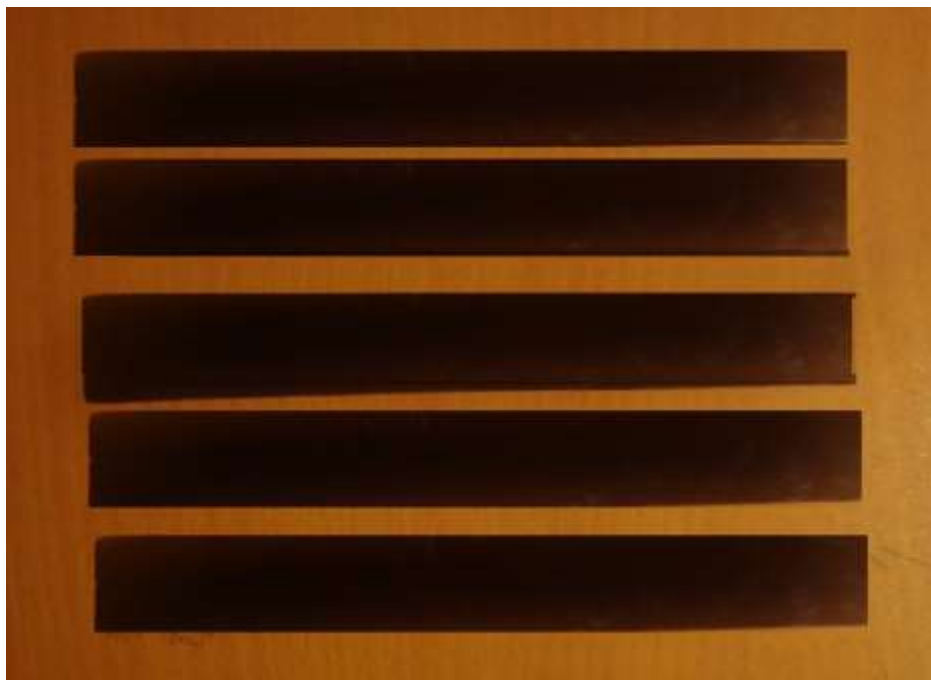


(a)



(b)

Fig1. A



(c)

RESULTS AND DISCUSSIONS

The study of the cure characteristics is essential for the successful development of a polymer composite system. This gives the information regarding the time required for curing the composite system, which in turn depends upon the interaction between the matrix and the reinforcement. The graph of optimum cure time vs. chitin particle loading for PFC system has been given in Fig. 4. The cure time has been found to be highest for 0% chitin loaded sheet then the cure time decreases with increase in chitin loading.

This clearly shows that the chitin particles accelerate the curing process of phenol formaldehyde (PF) matrix. Smaller the

cure time higher is the production rate possible in industry. Therefore, chitin reinforcement is better for PF matrix in terms of production time.

Table 1 clearly indicates that the Tensile strength, maximum force and percentage Elongation at break. The stress has been observed to increase with increase in chitin loading up to 0.5% chitin loading and further keeps decreasing. The strain is also increasing with increase in chitin loading which attributed to the increase in the Young's modulus with increase in chitin loading. Hence it can be concluded that the chitin particle loading reduces the brittleness and increases the toughness of the composites.

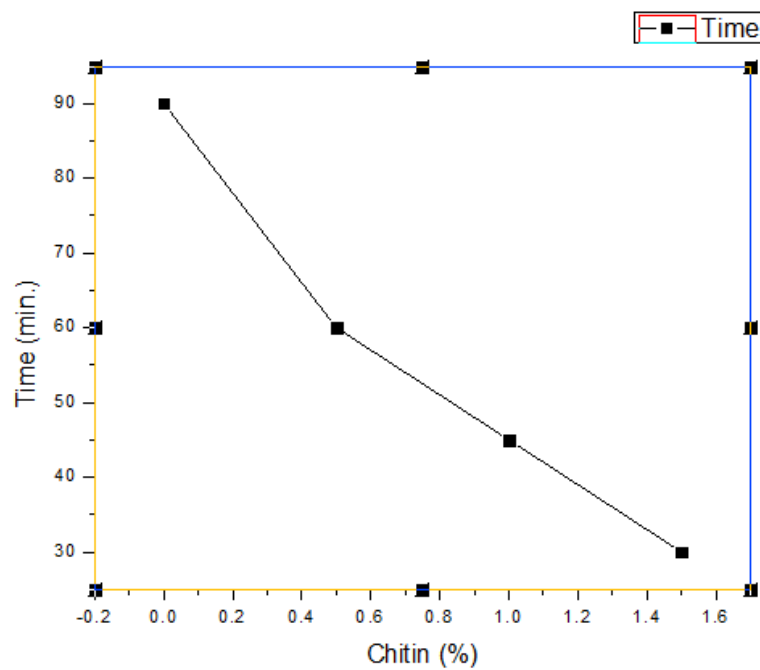


Fig 4: Optimum cure time Vs % loading of Chitin



Fig 5: Universal testing machine

CONCLUSION

Incorporation of chitin particles was successfully done with uniform distribution of particles in the water and thereby in a matrix of water soluble phenol formaldehyde resin which is immiscible with water solvents in normal conditions. The use of water soluble phenol formaldehyde has made it possible to disperse the particles in PF resin as the chitin particles were in the form of water suspension and further be cured into sheets of required sizes.

The results obtained by mechanical investigation of this sheets showed that the chitin nano particle in the composites were helpful in increasing the mechanical properties of the composite. Presence of chitin nano particles helped in improves curing time hence production rate. Further it has improved young's modulus by reducing brittleness leading to an improvement in the properties of phenol formaldehyde like material.

Out of the prepared composites the composite containing only 0.5% of chitin particles shows the highest strength which attributes to the high cross linking molecules in the phenol formaldehyde.

Further from the above results it is concluded that the chitin nano particles were helpful in improving the mechanical properties to a very little extent that is up to only 0.5% nano chitin on further increase in which did not show any improvement in its mechanical properties which attributes to the high cross linking of phenol formaldehyde polymer but it is also observed that incorporation of chitin particles will increase the ductility of a highly brittle materials like phenol formaldehyde.

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