Preparation of nano-particles of $CaCO_3$ & nano-composite based on $CaCO_3$

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ABSTRACT:

The study has been done to prepare/ Synthesis nano $CaCO_3$ and high-performance thermoset nano composite by incorporating E-glass fiber and nano $CaCO_3$ in thermoset matrix by simple hand layup technique using Unsaturated Polyester.

To characterize the mechanical and thermal properties of such thermoset resin nanoparticles E- glass fiber composite. Finally, to find out the optimal conditions of the preparing and processing to achieve best balance of properties of the composite.

Keywords*: Nanoparticles, Unsaturated polyester resin, Nano calcium carbonate, Nanocomposites.*

1. INTRODUCTION:

Nanocomposites are a new class of materials with atleast one dimension less than 100 nm. Recently, a great deal of attention has been paid to the polymer Nanocomposites, because of their unique and enhanced physical and mechanical properties, thermal and electrical properties, flame retardancy, gas barrier properties and shrinkage control behaviour.[1]

Experimental work is divided in two parts-

- Synthesis of nano $CaCO_3$
- Fabrication of nano $CaCO_3$ filled Nanocomposite & its testing.

2. EXPERIMENTAL:

Synthesis of Nanoparticles,

The chemicals and equipments of laboratory grade provided by Jyoti Comporium ,India and Chemox Enterprises, Nasik, India. by Carbonation process:

Formulation

- Solvent – Cyclohexen 100gm.
- Surfactant – Sodium lauryl sulphate 8.82gm
- Calcium Hydroxide [$Ca(OH)_2$] 1.2358gm
- Gas – CO_2 (Carbon dioxide)
- Water 83.54gm
- Molar ratio –
  - H_2O/Ca-OT = 4.64
  - $Ca(OH)_2$/Ca-OT = 0.00167
- Inert Gas – N_2 (Nitrogen Gas)

Equipments

- Reactor –Stirred bubbling tank, column rotating packed bed, Couette – Taylor
- Gas Cylinder – CO_2 & N_2
- Rubber tubes
- Methanol film
- Petry dish

3. CARBONATION PROCEDURE

A volume of 0.1 _ 1023 m3 of the reaction mixture [surfactant þ solvent, i.e. cyclohexane–heptane–decaneþ aqueous Ca(OH)2] was transferred into a cylindrical bubbler as shown in Figure 13 (capacity ¼ 0.15 _ 1023 m3). The solution was bubbled with N2 through a metal filter for 20 min to generate an inert atmosphere. The particles of $CaCO_3$ were prepared by bubbling CO2 into this reaction mixture for 3 min at 4 _ 1025 m3 min21.

The reaction was stopped by replacing the CO2 flow with N2. The amount of CO2 used was sufficient to obtain a theoretical yield of 100% of CaCO3 particles in any experiment.

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \text{ (Carbonation)}$$

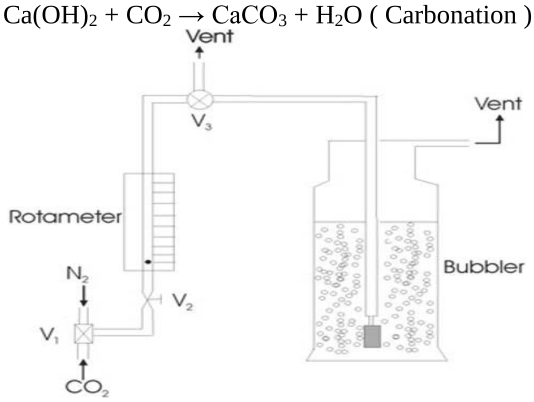


Fig: Experimental set-up for the carbonation experiments (V1, V2,V3 are manually operated valves).By In-situ deposition technique:

4. FORMULATION

- Calcium chloride (110gm + 100ml water)
- PEG (248 gm + 100 ml water)
- $CaCl_2$ + PEG 12 h.
- Molar ratio of $CaCl_2$: PEG = 1:4
- NH_4HCO_3 (79.9 gm + 100 ml water) for 12 h.

Equipments

- Beaker
- Burette
- Magnetic stirrer
- Petri dish
- Measuring cylinder

In-situ deposition process

Nanosize Calcium carbonate was synthesis by in-situ deposition technique shown in fig.14 calcium chloride (110 gm) was taken in 100 ml water. PEG (248gm) was diluted by taken 100 ml water and mildly heated for proper mixing. Complex of calcium chloride and PEG was prepared in molar ratios of 1:4 , 1:20 & 1:32. Another solution was prepared by taking ammonium bicarbonate (79.9 gm) in distilled water 100 ml. The first complex was digested for 12 hrs. then the solution of ammonium bicarbonate was added slowly and again kept for digestion for 12 hrs. The precipitate was filtered. Wash with water and dried in vacuum drier.

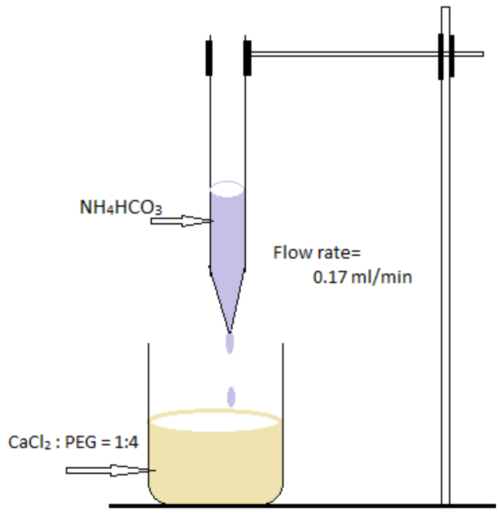


Fig.14: Experimental set-up for the In-situ deposition process

Fabrication of Nanocomposite.

The experimental work of project is carried out in stages as given below.

- Collection of raw materials
- Batch preparation and composite preparation by hand lay-up process
- Sample preparation and Testing

Raw Materials Used

Unsaturated polyester resin used in this study is having specific gravity 1.04 and viscosity 200 cps which is a general purpose commercial grade provided by Nazami Paints, Akola, India. It was used in combination with Cobalt Octate (Hardener), MEKP (Initiator) , supplied by Nazami Paints, Akola,India. The glass fiber used was E glass fibre in the form of chopped strand mat (CSM) having density 2.55 g/cc Commercial grade calcium carbonate filler supplied by Pinak Enterprises, Nashik, India.

Composite Preparation :

For preparation of thermoset composites, commercial general grade of unsaturated polyester resin is preferred. Initiator (MEKP), accelerator (Cobalt octate), are selected for thermosetting, the composites which are used to prepare test specimens are processed by hand lay-up technique by varying the phr of nano $CaCO_3$.

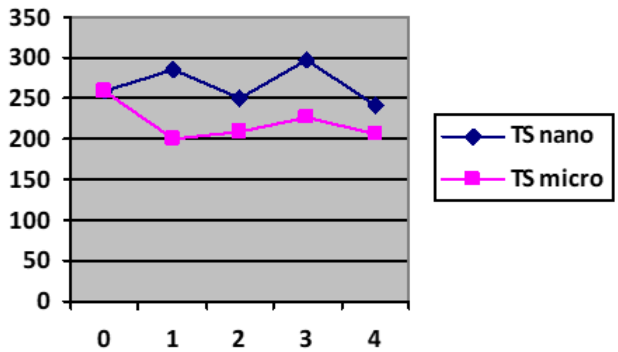
Testing's of NanoCaCO3 and Nanocomposites:

Testing of Nanoparticles: Testings of nanoparticles are made with the help of X-ray monitored gravity sedimentation principle while calculation methodused are Stokes sedimentation and Beer's law ofextinction for both processes(Carbonation and In-situ deposition).

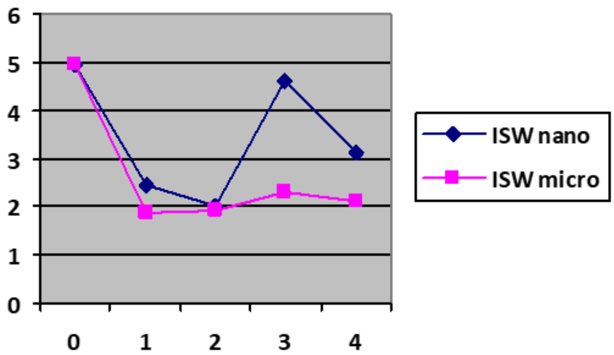
Mechanical testings of Nanocomposites: Tensile strength, elongation, Flexural strength and Impact strength are tested using Micro and Nano $CaCO_3$ in the composites for comparison.

5. RESULTS AND DISCUSSIONS

In Carbonation process, we got average particle size of nanoparticles 97 nm while in In-situ deposition technique, average particle size we got 23 nm.



Graph 1: Loading fillers in phr Vs Tensile strength in kg/cm^2



Graph 2: Loadig of filler in phr Flexural strength in kg/cm^2

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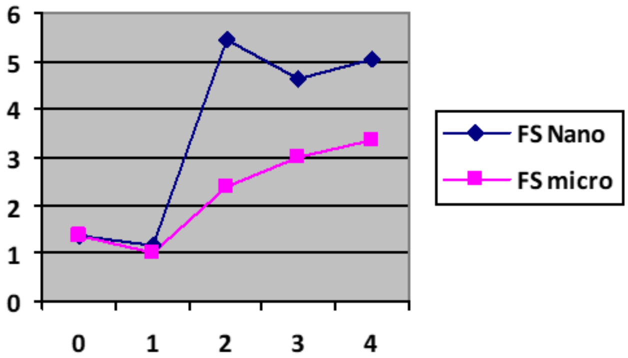
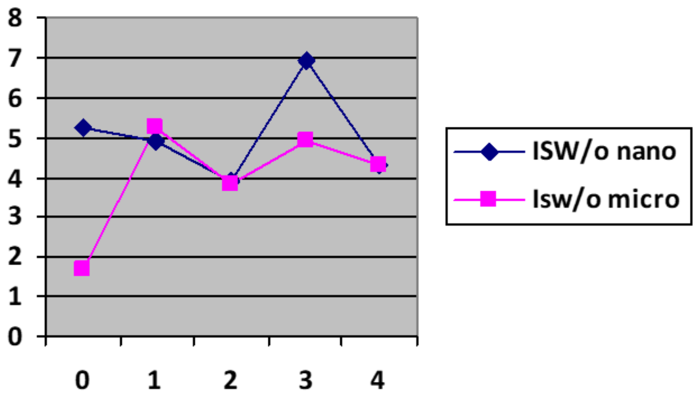


Graph 3: Loading of filler in phr Vs Impact strength with notch (kgm/inch) .



Graph 4: Loading of filler in phr Vs Impact strength without notch (kgm/inch).

From the above graphs, it is seen that the testing results of Nano CaCO3 composites are improved as compared to micro $CaCO_3$ composite. Tensile strength of nanocomposite is having better results as shown in graph 1 wherever flexural strength of nanocomposite is also increased than micro composite(showing graph 2).Izod impact with and without notch both for the nano CaCO3 composites are achieving high values than micro CaCO3 composites as seen in graphs 3 and 4.

6. CONCLUSION:

The experimental investigation into synthesis of nano-particles & behaviour based of nano $CaCO_3$ filled glass fiber reinforced USP thermosetting composite leads to the following conclusions:

- This work shows that successful synthesis of nano-particles & fabrication of a glass fiber reinforced thermosetting composites incorporated with nano $CaCO_3$ is possible by simple hand lay up technique.
- In this project work we got 90 to 100nm particle size in carbonation process & 20 to 35 nm particle size in situ deposition process which is within nano size. (0 to 100 nm).
- It is noticed that significant increase in mechanical properties like tensile strength, tensile modulus, flexural properties with nano $CaCO_3$ as filler as compaired to micro $CaCO_3$.
- The nano$CaCO_3$ gives optimum strength with minimum cost as compare to nanoclay.

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