ISSN: 2319-8753

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 3, Issue 1, January 2014

Preparation of Nanoparticle and Composite based on NanoCaco$_3$

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Abstract: Nanotechnology is an exciting technological advancement that has the potential to contribute significantly to the future of plastic. Through nanotechnology, special nano-composites can be created that will be more dent, heat and scratch resistant. Nanoparticles have a very high surface area to volume ratio. This provides tremendous driving forces for diffusion, especially at elevated temperatures and also reduces the incipient melting temperature of nanoparticles The Study of glass fiber reinforced polyester composites filled with nano $CaCO_3$ has been a less studied area. But it gives very good mechanical properties. There is a very wide scope for future scholars to explore this area of research. In future, this study can be extended to new composites using different grades of nano $CaCO_3$ and other potential fillers and the resulting experimental findings can be similarly analyzed. The visco-elastic studies such as storage modulus, loss modulus can be analyzed. The morphological studies will focus on dispersion of nano particle throught the composite.

Keywords: Composite, Resins, Reinforcement, Additives, Catalysts, Filler

I.INTRODUCTION

Nanotechnology is an exciting technological advancement that has the potential to contribute significantly to the future of plastic. Through nanotechnology, special nano-composites can be created that will be more dent, heat and scratch resistant.

Nanotechnology involves being able to understand and to control matter at the amazingly small dimensions of one to 100 nanometers, with one nanometer being equivalent to one-billionth of a meter (i. e., one millionth of a millimetres). Nano-size:- 1 nm= 10^{-9} & Nano-size range is 1 to 100 nanometers. As a point of reference, a sheet of paper is about 100,000 nanometers thick. With nanotechnology, an objective can image, measured, molded and manipulated right down to each nanometer. At this very tiny level, the chemical, physical and mechanical properties of materials are different than when in their bulk form. These new properties, therefore, can be used in different ways.

Nanoparticles have a very high surface area to volume ratio. This provides tremendous driving forces for diffusion, especially at elevated temperatures and also reduces the incipient melting temperature of nanoparticles.

Constituents of composites

- Resins, Reinforcement, Interface , Additives, Catalysts ,Promoters, Inhibitors

Chemical families of fillers for plastics

Sr. no.	Chemical family	Examples
1	Inorganic oxides	Glass (fibers, spheres, hollow spheres), MgO, SiO2 etc.
2	Hydroxides	Al(OH)3, Mg(OH)2 etc.
3	Salts	CaCO3, BaSO4, CaSO4, phosphates etc.
4	Silicates	Talc, mica, kaolin, nanoclays, asbestos etc.
5	Metal	Boron, steel etc.
6	Organic carbon, graphite	Carbon fibers, graphite fibers and f lakes, carbon nanotubes, carbon black etc.

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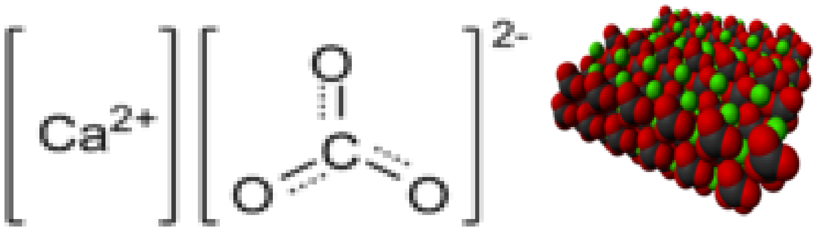
II. LITERATURE REVIEW

Calcium Carbonate

Calcium carbonate is a chemical compound with the formula $CaCO_3$. It is a common substance found in rocks in all parts of the world, and is the main component of shells of marine organisms, snails, coal balls, pearls, and eggshells. Calcium carbonate is the active ingredient in agricultural lime, and is usually the principal cause of hard water. It is commonly used medicinally as a calcium supplement or as an antacid, but excessive consumption can be hazardous.

Chemical Structure – $CaCO_3$



Sources-

- Lime stone, Chalk, Whiting, Marbles, Dolomite

Fig.9: calcium carbonate

Preparation

The vast majority of calcium carbonate used in industry is extracted by mining or quarrying. Pure calcium carbonate (e.g. for food or pharmaceutical use), can be produced from a pure quarried source (usually marble).

Alternatively, calcium carbonate is prepared by calcining crude calcium oxide. Water is added to give calcium hydroxide, and carbon dioxide is passed through this solution to precipitate the desired calcium carbonate, referred to in the industry as precipitated calcium carbonate (PCC):

$CaCO_3 \rightarrow CaO + CO_2$

$CaO + H_2O \rightarrow Ca(OH)_2$

$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2$

Properties of $CaCO_3$

Molecular weight 100.09 gm/mol
Specific Gravity 2.71
Melting point 825 °C
White colour & odor less

IJIRSET

ISSN: 2319-8753

International Journal of Innovative Research in Science, Engineering and Technology

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III.MATERIALS AND METHODS

Composite can be manufacture by various types of methods but mainly it is divided into three main categories, are

1) Open molding process

2) Close Molding process

3) Continuous Process

Open molding

Wet Lay-up/Hand Lay-up

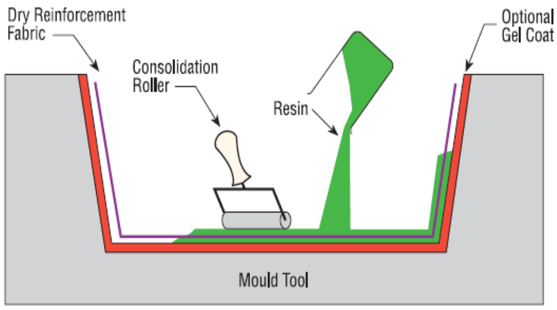

Fig.7: Hand layup technique

Description

The hand (wet) lay-up is one of the oldest and most commonly used methods for manufacture of composite parts. Hand lay-up composites are a case of continuous fibre reinforced composites. Layers of unidirectional or woven composites are combined to result in a material exhibiting desirable properties in one or more directions. Each layer is oriented to achieve the maximum utilisation of its properties. Layers of different materials (different fibres in different directions) can be combined to further enhance the overall performance of the laminated composite material. Resins are impregnated by hand into fibres, which are in the form of woven, knitted, stitched or bonded fabrics. This is usually accomplished by rollers or brushes, with an increasing use of nip-roller type impregnators for forcing resin into the fabrics by means of rotating rollers and a bath of resin. Laminates are left to cure under standard atmospheric conditions.

IV SYNTHESIS OF NANOPARTICLES

By using carbonization process we can prepared nano particles,

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)

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Equipments

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

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.

- $Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$ (Carbonation)

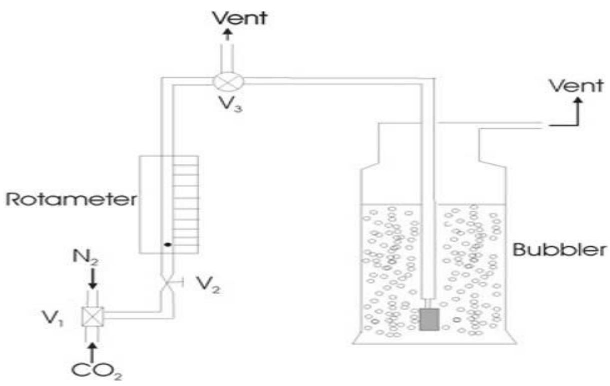

Fig. Experimental set-up for the carbonation experiments (V1, V2,V3 are manually operated valves).

V EXPERIMENTAL RESULT

Observations

- Low yield
- Less time consuming
- Fine particle size but higher than other processes.
- Chemicals- high quantity
- High equipment & chemical cost
- Less requirement of electricity

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

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ISSN: 2319-8753

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Composition of Nano $CaCO_3$ filled USP composite

Sample Code	USP(phr)	Glass Fiber (phr)	Nano $CaCO_3$ (phr)
USP	100	0	0
USP/GF	100	6	0
USP/GF/N1	100	6	1
USP/GF/N2	100	6	2
USP/GF/N3	100	6	3
USP/GF/N4	100	6	4

Table.1: Composition of NanoCaCO3 filled USP composite

Composition of Micro $CaCO_3$ filled USP composite

Sample Code	USP(phr)	Glass Fiber (phr)	Micro $CaCO_3$ (phr)
USP	100	0	0
USP/GF	100	6	0
USP/GF/M1	100	6	1
USP/GF/M2	100	6	2
USP/GF/M3	100	6	3
USP/GF/M4	100	6	4

Table.2: Composition of Micro $CaCO_3$ filled USP composite

Where,

USP = Unsaturated Polyester

GF = Glass Fiber (E-Glasss)

Phr = Parts per Hundred

100 phr = 247.23 gm of resin

6 phr = 15 gm glass fiber of resin

1 phr = 2.47 gm filler of resin

N & M = Nano & Micro

Curing system used

Initiator : MEKP= 1% on resin basis
Accelerator: cobalt-octate = 1% on resin basis
Curing time = 24 hrs,
Testing : Same testing were done for USP as describe earlier .

Testing of composite

1. Tensile Testing and Elongation(ASTM D638 ,)
2. Flexural strength and modulus (ASTM D790)
3. Izod Impact Testing (ASTM D256)

Tensile Properties

The tensile properties obtained, for the USP composite materials are as given in following. The test is carried out to bring the accurate tensile properties ie tensile strength, elongation, tensile modulus of the composite.

ISSN: 2319-8753

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The response of a composite to tensile load highly dependent on the tensile stiffness and strength properties of the reinforcement fibres and nanofillers ie nano$CaCO_3$ incorporated.

Sample Code	Glass Fibre (phr)	Nano $CaCO_3$ (phr)	Tensile strength at peak load (kg/cm²)	% Elongation at break	Young's Modulus (kg/cm²)
USP	0	0	224.628	3.42	8012.016
USP/GF	6	0	258.32	5.31	638.02
USP/GF/N1	6	1	286.66	5.oo	725.314
USP/GF/N2	6	2	250.116	6.53	6577.05
USP/GF/N3	**6**	**3**	**296.992**	**4.32**	**7866.19**
USP/GF/N4	6	4	242.1107	3.42	7214.54

Table.3: properties of USP composite with Nano $CaCO_3$.

The Table shows the behavior of tensile strength of USP composite with nano $CaCO_3$. There is gradual increase in tensile strength is observed up to 3phr loading. Result shows at 3phr loading there is 10% increase tensile strength.

The Table shows % elongation of USP composite with nano $CaCO_3$. It is observed that there is significant variation in elongation is observed. This values were not constant. 3phr loading shows max. tensile strength & good Young's modulus

Sample Code	Glass Fibre (phr)	$CaCO_3$ (phr)	Tensile strength at peak load (kg/cm²)	% Elongation at break	Young's Modulus (kg/cm²)
USP	0	0	224.628	3.42	8012.016
USP/GF	6	0	258.32	5.31	638.02
USP/GF/M1	6	1	198.70	5.oo	725.314
USP/GF/M2	6	2	208.40	4.62	1878.05
USP/GF/M3	6	3	225.52	4.32	7866.19
USP/GF/M4	6	4	205.72	3.42	7214.54

Table.4: Properties of USP composite with Micro $CaCO_3$

Flexural Properties

The flexural properties obtained of the USP composite materials are as shown in the table 8 .

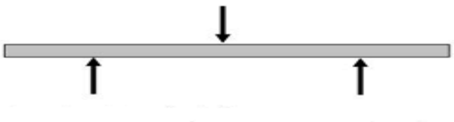

Fig.: Flexural test specimen for USP composite

Flexural loads are combination of tensile, compression and shear loads. When loaded as shown, the upper side is put into compression, the lower side into tension and the central portion of the laminate experiences shear.

Table shows the behavior of flexural strength of USP composite with Nano $CaCO_3$. The flexural strength is found to be gradually increase as the Nano $CaCO_3$ loading increased.

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Sample Code	Glass Fibre (phr)	Nano $CaCO_3$ (phr)	Flexural strength Kg/cm^2
USP	0	0	3.49
USP/GF	6	0	1.35
USP/GF/N1	6	1	1.18
USP/GF/N2	**6**	**2**	**5.42**
USP/GF/N3	6	3	4.62
USP/GF/N4	6	4	5.04

Table.5 : Flexural properties of USP composite with Nano- $CaCO_3$.

Here 2phr loading shows max. Flexural strength.

Sample Code	Glass Fibre (phr)	Micro $CaCO_3$ (phr)	Flexural strength Kg/cm^2
USP	0	0	3.49
USP/GF	6	0	1.35
USP/GF/M1	6	1	1.02
USP/GF/M2	6	2	2.38
USP/GF/M3	6	3	2.98
USP/GF/M4	6	4	3.34

Table .6 : Flexural properties of USP composite with Micro $CaCO_3$.

Impact Properties:

The impact properties of USP composite materials obtained are as shown in table 20. The test is carried out to bring the accurate impact properties of the composite.

Marginal decrease in izod impact strength is observed in USP composite as the loading of nanoclay increase as shown in figure. It can be conclude that, as the nanoclay loading increase the samples gets stiffer.

Sample Code	Glass fibre (phr)	Nano $CaCO_3$ (phr)	Impact Strength (kgm/inch) (with notch)	Impact Strength (kgm/inch) (w/o notch)
USP	10	0	1.00	1.683
USP/GF	10	0	4.96	5.22
USP/GF/N1	10	1	2.44	4.846
USP/GF/N2	10	2	2.004	3.862
USP/GF/N3	**10**	**3**	**4.6**	**6.923**
USP/GF/N4	10	4	3.11	4.33

Table.7: Impact strength of USP composite with Nano $CaCO_3$

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Sample Code	Glass fibre (phr)	Micro $CaCO_3$ (phr)	Impact Strength (kgm/inch) (with notch)	Impact Strength (kgm/inch) (w/o notch)
USP	10	0	1.00	1.683
USP/GF	10	0	4.96	5.22
USP/GF/M1	10	1	1.86	3.11
USP/GF/M2	10	2	1.91	3.862
USP/GF/M3	10	3	2.304	4.89
USP/GF/M4	10	4	2.11	4.31

Table.8: Impact strength of USP composite with micro $CaCO_3$

Comparison between micro $CaCO_3$ & nano $CaCO_3$

comparative study on the effect of micro and nano size $CaCO_3$ was studied during the project. The results found were enlisted in Table. It is observed that the incorporation of micron filler fails to improved the tensile strength, tensile modulus and elongation. As we can observed in the case of nano $CaCO_3$ that small incorporation of nano $CaCO_3$ can significantly increase the mechanical properties of composite.

Sample Code	Micro $CaCO_3$ (phr)	T.S. (kg/cm^2)	%ELON	Y.M. (kg/cm^2).	FLEX. ST. (kg/cm^2)	Impact Kg/inch
USP/GF/M1	5	263.71	4.22	4256.77	4.2	3.91
USP/GF/M2	10	272.13	3.91	5134.00	3.2	4.12
USP/GF/M3	15	293.66	4.97	6012.03	4.8	5.01

Table.9: Properties of micron size $CaCO_3$ filled USP composite

Sample Code	Nano $CaCO_3$ (phr)	Tensile strength at peak load (kg/cm²)	% Elongation at break	Young's Modulus (kg/cm²)	FLEX. ST. (kg/cm^2)	Impact Kg/inch
USP/GF/N1	1	286.66	5.00	5125.314	1.18	4.846
USP/GF/N2	2	250.116	6.53	6577.05	5.42	3.862
USP/GF/N3	**3**	**296.992**	**4.32**	**7866.19**	**4.62**	**6.923**
USP/GF/N4	4	242.1107	3.42	7214.54	5.04	4.33

Table.10:Properties of Nano $CaCO_3$ filled USP composite

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Sample Code	Micro $CaCO_3$ (phr)	T.S. (kg/cm^2)	%ELON	Y.M. (kg/cm^2).	FLEX. ST. (kg/cm^2)	Impact Kg/inch
USP/GF/M1	5	263.71	4.22	4256.77	4.2	3.91
USP/GF/M2	10	272.13	3.91	5134.00	3.2	4.12
USP/GF/M3	15	293.66	4.97	6012.03	4.8	5.01

Table.11:Comparison of Nano $CaCO_3$ with Nanoclay :

A comparative study on the effect of Nano $CaCO_3$ and nano size clay was studied during the project. The results found were enlisted in Table .We have reffered nanoclay result of last year batch (To study nanoclay filled thermoset composite)

Sample Code	Nano $CaCO_3$ (phr)	Tensile strength at peak load (kg/cm^2)	% Elongation at break	Young's Modulus (kg/cm^2)	FLEX. ST. (kg/cm^2)	Impact Kg/inch
USP/GF/N1	1	286.66	5.00	5125.314	1.18	4.846
USP/GF/N2	2	250.116	6.53	6577.05	5.42	3.862
USP/GF/N3	**3**	**296.992**	**4.32**	**7866.19**	**4.62**	**6.923**
USP/GF/N4	4	242.1107	3.42	7214.54	5.04	4.33

Table.12: Properties of Nano $CaCO_3$ filled USP composite

Sample Code	NANO CLAY (phr)	T.S. (kg/cm^2)	T.M. (kg/cm^2)	% ELON	FLEX. ST. (kg/cm^2)	FLEX. MOD (kg/cm^2)
USP/GF/N1	1	396	11638	4.8	10	2655
USP/GF/N2	2	327	9343	5.2	15	3631
USP/GF/N3	3	422	13237	5.6	13	3241
USP/GF/N4	4	330	11759	5.2	13	3455

Table13 : Properties of Nanoclay filled USP composite

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VI 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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