IJISET - International Journal of Innovative Science, Engineering & Technology, Vol. 3 Issue 1, January 2016.
www.ijiset.com
ISSN 2348 – 7968

Morphology and Thermal Studies of Calcium Carbonate Nanoparticles

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Abstract

Calcium carbonate nanoparticles were synthesized via chemical co-precipitation method from calcium chloride and sodium carbonate. The formed nanoparticle is characterized by transmission electron microscopy, differential scanning calorimetry technique, thermo gravimetric analysis and differential thermal analysis. The TEM image shows the synthesized calcium carbonate show well crystallized particles with spherical morphology. From the TEM image average nanoparticle size, standard deviation and polydispersity can be calculated. The d spacing can be calculated from SAED pattern. From DSC measurements the quantitative and qualitative information about physical and chemical changes that include endothermic/exothermic processes or changes in heat capacity can be studied. From TGA curve, the decomposition due to mass loss is observed.

Keywords: *TEM, DSC, TGA-DTA.*

1. Introduction

The synthesis of inorganic nano and micro materials with well-defined and controllable morphologies has stimulated considerable attention, because it is well known that the properties of the materials closely interrelate with geometrical factors such as morphology, dimensionality, and the size [1]. Particles have distinctive properties when their crystalline sizes are reduced to submicron range with respect to corresponding bulk materials, including prominent mechanical/optical properties, high specific surface area and chemical activity, and superior barrier effects especially in polymer matrix. Among the numerous nano-sized materials synthesized during the past decade, calcium carbonate remains one of the most useful and abundant [2].

Nanoparticles of calcium carbonate have much attention due to its wide applications such as marble, chalk, coral, cement, mortars, plasters, refractories, and glass as building materials, textiles, papers, paints, plastics, adhesives, sealants, and cosmetics. The characterization of the samples were carried out using Transmission Electron Microscopy (TEM), Differential Thermal Analysis (DTA), Thermo Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC). From these studies, we can determine the morphological and thermal properties.

2. Experimental Details

Nanoparticles of calcium carbonate were prepared by chemical co-precipitation method by adding calcium chloride and sodium carbonate. Precise amounts of reagents taking into account their purity were weighed and dissolved separately in distilled water into 0.1M concentration. After obtaining a homogeneous solution, the reagents were mixed using magnetic stirring. The precipitate was separated from the reaction mixture and washed several times with distilled water and ethanol. The wet precipitate was dried and thoroughly ground using agate mortar to obtain the samples in the form of fine powder.

3. Results and discussion

3.1. TEM Analysis

Fig.1 shows the TEM images of the synthesized calcium carbonate nanoparticles. The TEM images show the spherical morphology of calcium carbonate nanoparticles. The size distribution histogram of fig.1d is shown in fig.2. TEM images show that calcium carbonate nanoparticles are having particle size 5.9nm. The size distribution histogram for calcium carbonate nanoparticles shows the average particle size is 5.9 ± 4.6 nm. The standard

ISSN 2348 – 7968

deviation is 4.6nm and the polydispersity of calcium carbonate nanoparticles is 77%.

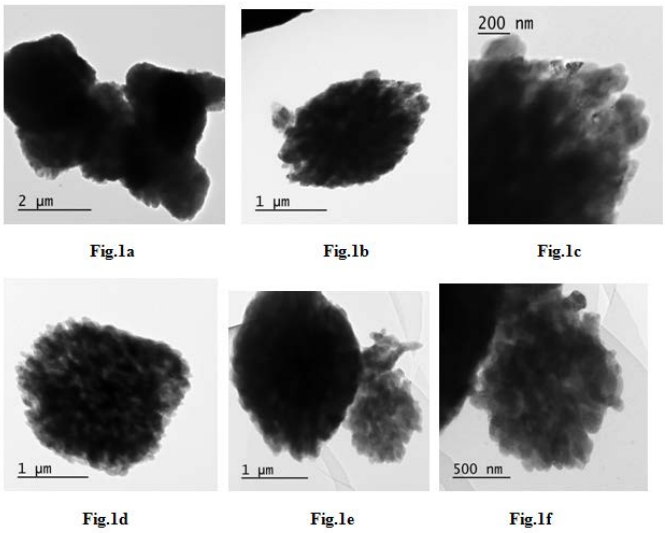

Fig.1a Fig.1b Fig.1c

Fig.1d Fig.1e Fig.1f

Fig.1 TEM images of calcium carbonate nanoparticles

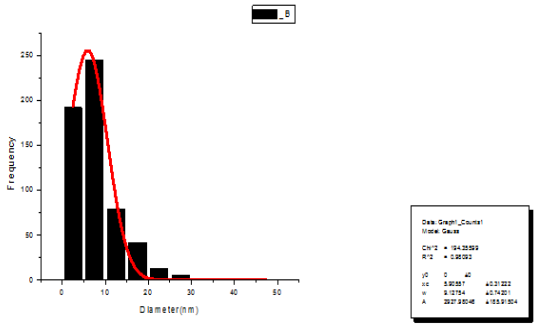

Fig.2 The size distribution histogram for calcium carbonate nanoparticles

The SAED pattern of calcium carbonate nanoparticles is shown in fig.3. It shows the particles are crystallized. By indexing the SAED pattern the d spacing of calcium carbonate nanoparticles is found to be 1.3708 nm which matches with the data in JCPDS file 85-1108 which shows the diffraction rings on SAED pattern matches with the XRD pattern [3].

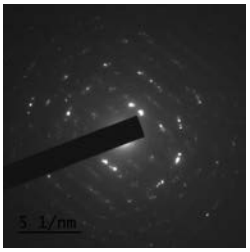

Fig.3. The SAED pattern of calcium carbonate nanoparticles

3.2. DSC Analysis

The thermal analysis of the calcium carbonate nanoparticles have been investigated using DSC analysis over a temperature of 30 - 300^{0}C. Fig.4 shows the DSC curve of calcium carbonate nanoparticles. The melting point can be determined from the melting curve with pure substances; the melting point corresponds to the onset. Impure samples often show several peaks. Substances with eutectic impurities exhibit two peaks; first the eutectic peak whose size is proportional to the amount of impurity and then the main melting point. It is due to the existence of many different sizes of particles, as the particle size increased the melting point increases. The downward movement of the peak in DSC heating curve indicates that the peak is endothermic peak. A small low temperature endothermic peak at 81.12^{0}C is due to the impurity and a large high temperature endothermic peak at 153.63^0 C in a DSC heating curve is a melting peak. The thermal data in DSC curve of calcium carbonate nanoparticles are shown in table.1.

Table.1 Thermal data in DSC curve of calcium carbonate nanoparticles

Sample/Properties	**Calcium carbonate nanoparticles**	
	Low temperature endothermic peak	**High temperature endothermic peak**
Quantity	6.85mg	6.85mg
Heating Rate	10^{0}C/min	10^{0}C/min
Onset	81.12^{0}C	153.63^{0}C
Peak	83.78^{0}C	162.35^{0}C
Endset	88.86^{0}C	173.83^{0}C

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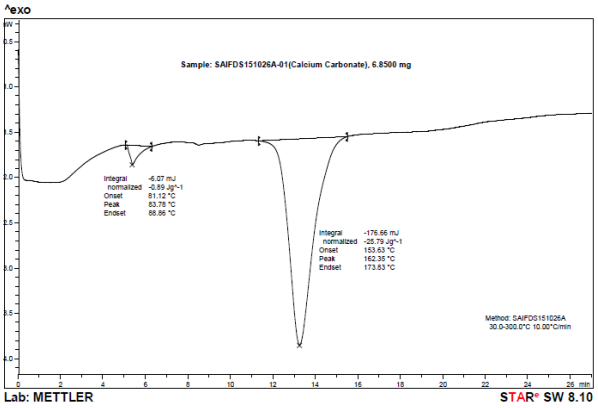

Fig.4. DSC curve of calcium carbonate nanoparticles

3.3. TGA-DTA Analysis

Thermal analysis of calcium carbonate nanoparticles is also carried out by thermogravymetric analysis (TGA) and differential thermal analysis.8.6133mg of calcium carbonate nanoparticles at a temperature from 40 °C to 830 °C at 20 °C/minute is analyzed. TGA/DTA thermograms of calcium carbonate nanoparticles are shown in fig.5.

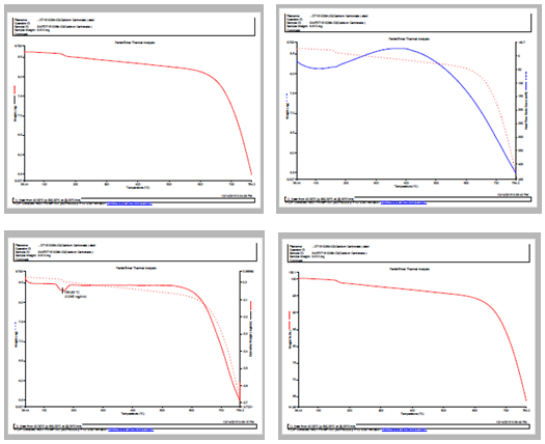

Fig.5. TGA/DTA thermograms of calcium carbonate nanoparticles

The thermal analysis data of calcium carbonate nanoparticles are shown in table.2. The descending TGA thermal curve indicates a weight loss occurred. The TGA curve represents the decomposition of the sample in single stage [4].

Table.2 Thermal analysis data of calcium carbonate nanoparticles

Material	TGA temperature (^{0}C)	DSC peak temperature (^{0}C)	DTA weight loss (%)
calcium carbonate nanoparticles	163.93	162.35	8.55

4. Conclusions

The calcium carbonate nanoparticles have been prepared by chemical co-precipitation method. TEM analysis suggests that the average particle size is 5.9 ± 4.6 nm, the standard deviation is 4.6nm and the polydispersity of calcium carbonate nanoparticles is 77% and the diffraction rings on SAED pattern matches with the XRD pattern. The DSC curve shows the melting peak. The TGA curve represents the decomposition of the sample in single stage.

5. References

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