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MORPHOLOGY AND THERMAL STUDIES OF MAGNESIUM CARBONATE NANOPARTICLES

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ABSTRACT

Magnesium carbonate nanoparticles were synthesized via chemical co-precipitation method from magnesium sulfate 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 magnesium chloride 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: DSC, MORPHOLOGY, SAED, TEM, TGA-DTA.

1. Introduction

Magnesium carbonate occurs in nature in several minerals as hydrated, basic and double salts, The two principal minerals are magnesite and dolomite, a double salt. Both minerals are used as source materials in the production of magnesium metal. Also, they are calcined to produce basic refractory bricks. Other applications of magnesium carbonate are in flooring, fireproofing and fire-extinguishing compositions; as a filler material and smoke

suppressant in plastics; as a reinforcing agent in neoprene rubber; as a drying agent and for color retention in foods; in cosmetics; in dusting powder; a laxative to loosen the bowels; color retention in foods and in toothpaste. The high purity magnesium carbonate is used as an antacid in medicine; and as an additive to table salt. Another important application of magnesium carbonate is as a starting material in producing a number of magnesium compounds [1]. Magnesium carbonate, most often referred to as 'chalk', is used as a drying agent for hands in rock climbing, gymnastics and weight lifting. Magnesium carbonate is also used in taxidermy for whitening skulls. It can be mixed with hydrogen peroxide to create a paste, which is then spread on the skull to give it a white finish. In this work magnesium carbonate nanoparticles are synthesized by chemical co-precipitation method and their morphology and thermal properties are studied.

2. Experimental Details

Nanoparticles of nickel carbonate were prepared by chemical co-precipitation method by adding magnesium sulfate 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 magnesium carbonate nanoparticles. The TEM images show the spherical morphology of magnesium carbonate nanoparticles. The size distribution histogram of fig.1c is shown in fig.2. The size distribution histogram for nickel carbonate nanoparticles shows the average particle size is 9.65±36.4nm. The standard deviation is 36.4nm.

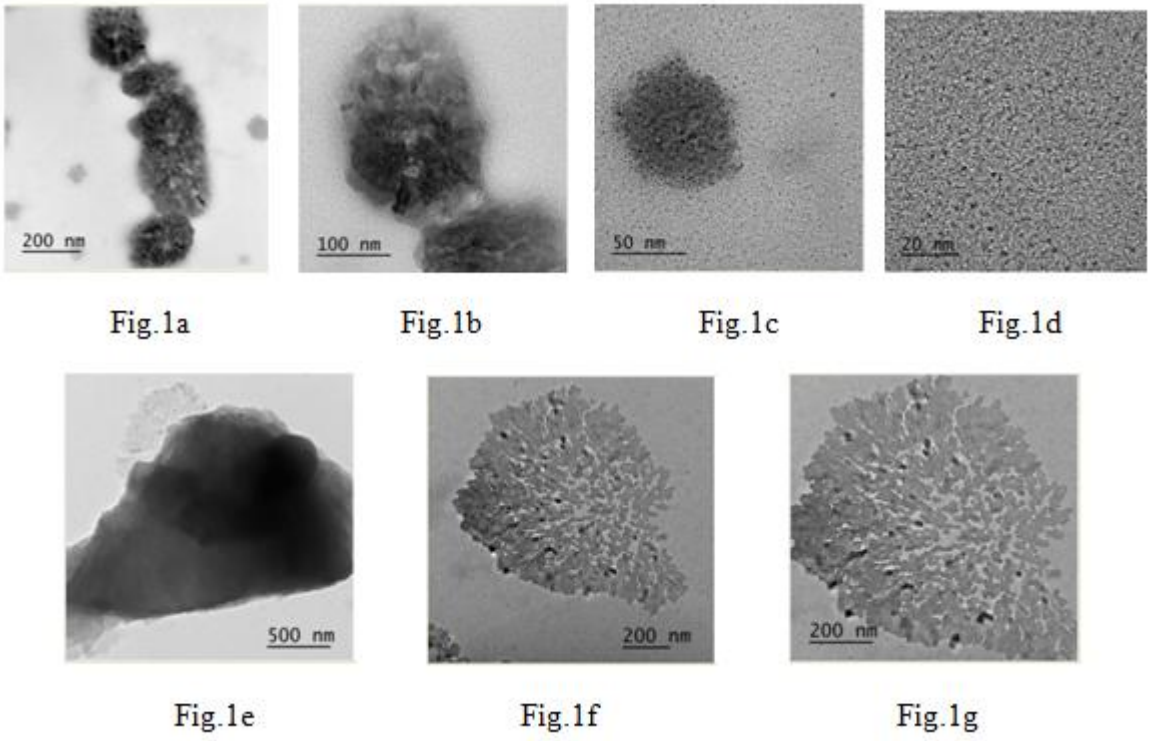

Fig.1 TEM images of magnesium carbonate nanoparticles

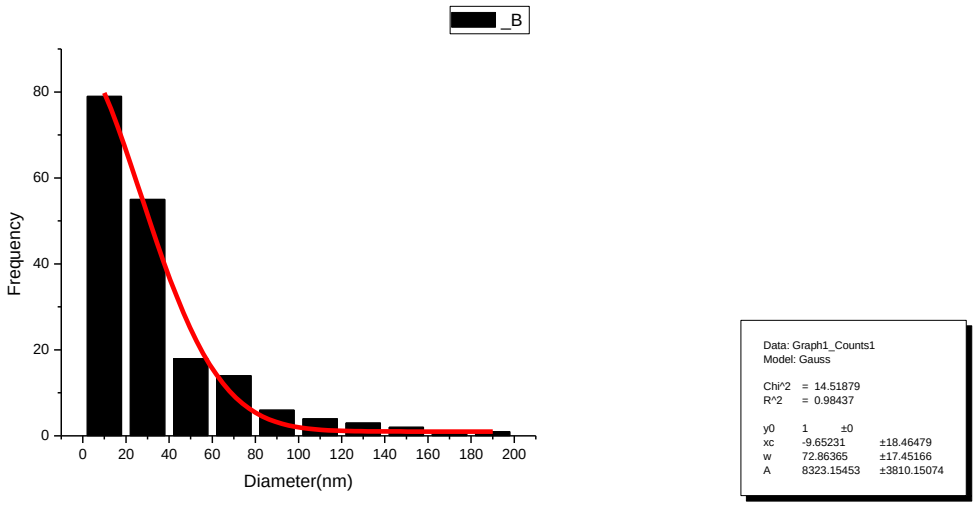

Fig.2. The size distribution histogram for magnesium carbonate nanoparticles

The SAED pattern of magnesium carbonate nanoparticles is shown in fig.3. It shows the particles are crystallized. By indexing the SAED pattern the d spacing of magnesium carbonate nanoparticles is found to be 1.7076nm and 1.1250nm which matches with the data

in JCPDS file 80-0101 which shows the diffraction rings on SAED pattern matches with the XRD pattern [2].

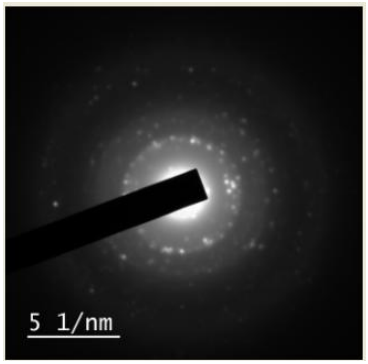

Fig.3. The SAED pattern of magnesium carbonate nanoparticles

3.2. DSC Analysis

The thermal analysis of the magnesium carbonate nanoparticles have been investigated using DSC analysis over a temperature of 30 - 300^{0}C. Fig.4 shows the DSC curve of magnesium 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. It is due to the existence of many different sizes of particles, as the particle size increased the melting point increases.

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

Sample/Properties	**Magnesium carbonate nanoparticles**				
	Low temperature endothermic peak				**High temperature endothermic peak**
Quantity	6.643mg	6.643mg	6.643mg	6.643mg	6.643mg
Heating Rate	10^{0}C/min	10^{0}C/min	10^{0}C/min	10^{0}C/min	10^{0}C/min
Onset	50.34^{0}C	79.58^{0}C	117.40^{0}C	160.52^{0}C	241.64^{0}C
Peak	61.95^{0}C	81.60^{0}C	131.57^{0}C	162.23^{0}C	265.79^{0}C
Endset	73.53^{0}C	88.81^{0}C	147.04^{0}C	175.26^{0}C	288.5^{0}C

The downward movement of the peak in DSC heating curve indicates that the peak is endothermic peak. There are four temperature endothermic peaks at 50.34^{0}C, 79.58^{0}C, 117.4^{0}C and 160.52^{0}C are due to the different sizes of the particles and a high temperature endothermic peak at 241.64^{0}C in a DSC heating curve is a melting peak. The thermal data in DSC curve of magnesium carbonate nanoparticles are shown in table.1.

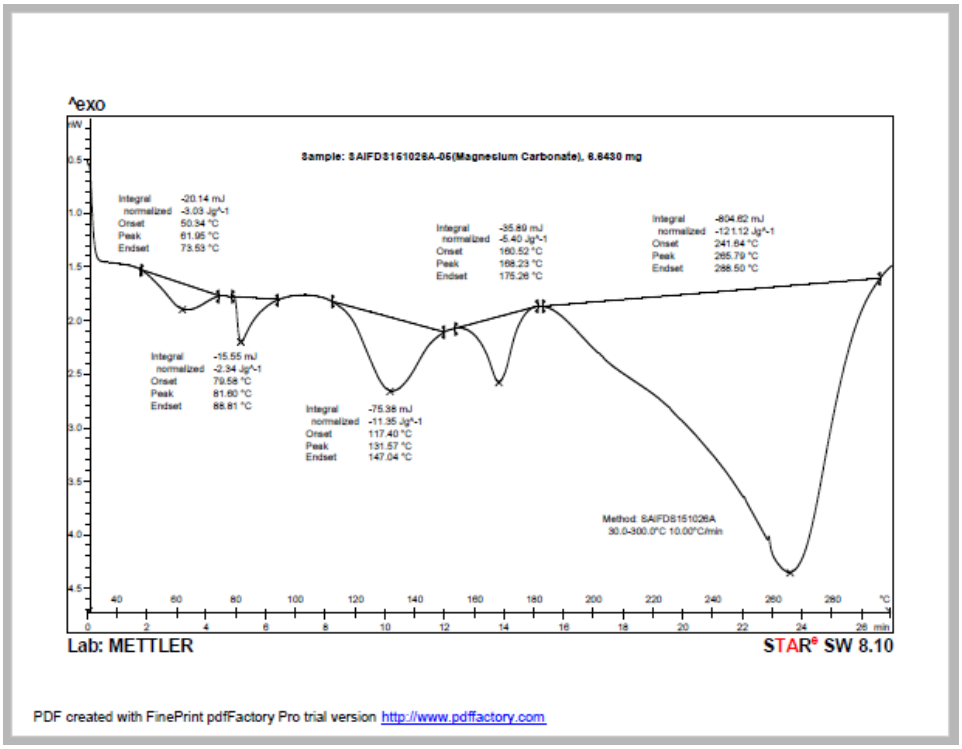

Fig.4. DSC curve of magnesium carbonate nanoparticles

3.3. TGA-DTA Analysis

Thermal analysis of magnesium carbonate nanoparticles is also carried out by thermogravymetric analysis (TGA) and differential thermal analysis. 6.984mg of magnesium carbonate nanoparticles at a temperature from 40 °C to 830 °C at 20 °C/minute is analyzed. TGA/DTA thermograms of magnesium carbonate nanoparticles are shown in fig.5.

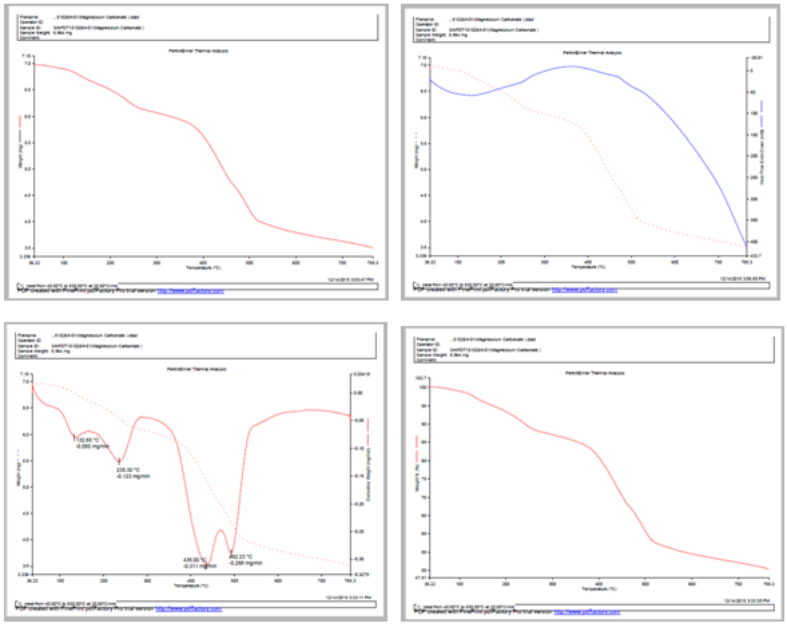

Fig.5. TGA/DTA thermograms of magnesium carbonate nanoparticles

The thermal analysis data of magnesium carbonate nanoparticles in DSC and TGA analysis are shown in table.2. The total weight loss is 13% [3]. The descending TGA thermal curve indicates a weight loss occurred. The TGA curve represents the multi stage decomposition of the sample [4]. There are four peaks in TGA curve are 132.65°C, 235.30°C, 435°C and 492.23°C indicates the decomposition of the sample in multi stages.

Table.2.The thermal analysis data of magnesium carbonate nanoparticles

Material	TGA temperature (^{0}C)	DSC peak temperature (^{0}C)	DTA weight loss (%)	Total Weight Loss(%) (40 °C to 830 °C)
Magnesium carbonate nanoparticles	132.65 235.30	131.57 162.23	6.78 6.31	13.1

4. CONCLUSIONS

The magnesium carbonate nanoparticles have been prepared by chemical co-precipitation method. TEM analysis suggests that the average particle size is 2.3±2.8nm, the

standard deviation is 2.8nm 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 multi stage.

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