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Morphology and Thermal studies of Lead Carbonate Nanoparticles

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Abstract— Lead carbonate nanoparticles were synthesized via chemical co-precipitation method from lead nitrate 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 lead 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.**

I. INTRODUCTION

Nanoparticles are of great scientific interest as they are effectively a bridge between bulk materials and atomic or molecular structures. A bulk material should have constant physical properties regardless of its size, but at the nano-scale size-dependent properties are often observed. Thus, the properties of materials change as their size approaches the nanoscale and as the percentage of atoms at the surface of a material becomes significant. The interesting and sometimes unexpected properties of nanoparticles are therefore largely due to the large surface area of the material, which dominates the contributions made by the small bulk of the material [1]. In this work, lead carbonate nanoparticles are synthesized by chemical co-precipitation method and their morphology and thermal properties are studied. Lead carbonate, or white lead, was one of the chief additives in lead paint. Lead carbonate is used as an additive in a variety of cements. Lead carbonate is also a part of both artistic and construction putty. There is a certain type of paper called lead carbonate paper, which is similar to more common carbon paper that is used commercially.

II. EXPERIMENTAL DETAILS

Nanoparticles of lead carbonate were prepared by chemical co precipitation method by adding lead (II) nitrate 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.

III. RESULTS AND DISCUSSION

TEM ANALYSIS

Fig.1 shows the TEM images of the synthesized lead carbonate nanoparticles. The TEM images show the spherical morphology of lead carbonate nanoparticles. The size distribution histogram of fig.1c is shown in fig.2. TEM images show that lead carbonate nanoparticles are having particle size 7.4nm. The size distribution histogram for lead carbonate nanoparticles shows the average particle size is 7.4±4nm. The standard deviation is 4nm and the polydispersity of lead carbonate nanoparticles is 54%.

The SAED pattern of lead carbonate nanoparticles is shown in fig.3. It shows the particles are crystallized. By indexing the SAED pattern the d spacing of lead carbonate nanoparticles is found to be 1.7498nm which matches with the data in JCPDS file 47-1734 which shows the diffraction rings on SAED pattern matches with the XRD pattern [2].

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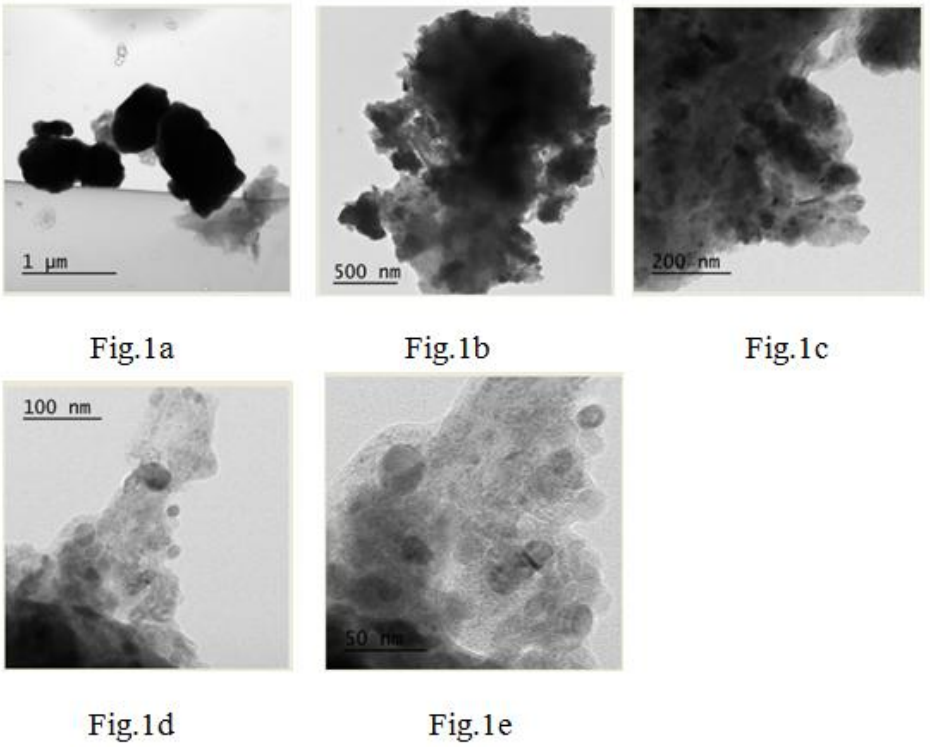

Fig.1 TEM images of lead carbonate nanoparticles

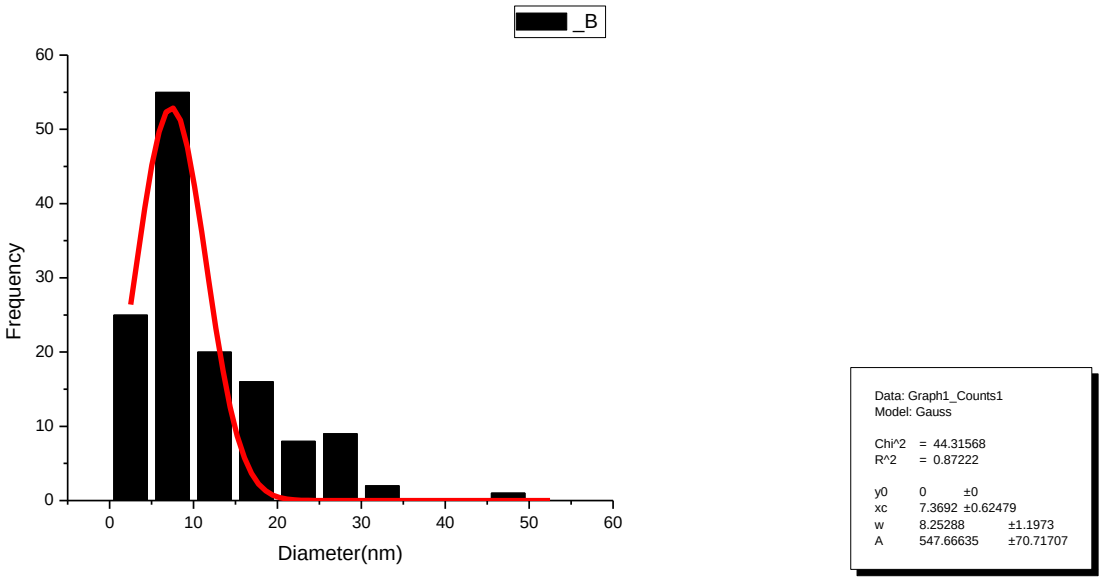

Fig.2. The size distribution histogram for lead carbonate nanoparticles

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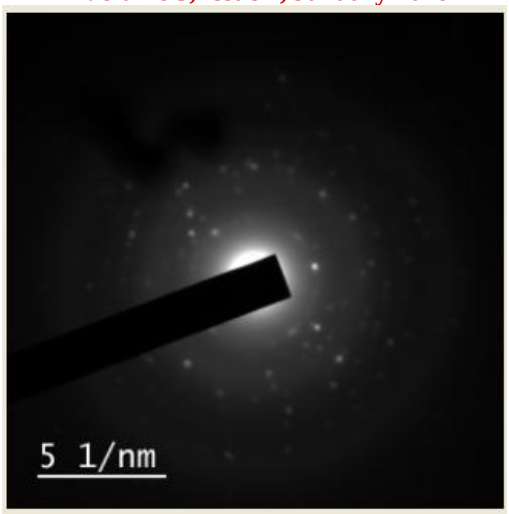

Fig.3. The SAED pattern of lead carbonate nanoparticles

DSC ANALYSIS

The thermal analysis of the lead carbonate nanoparticles have been investigated using DSC analysis over a temperature of 30 - 300^{0}C. Fig.4 shows the DSC curve of lead carbonate nanoparticles. The melting point can be determined from the melting curve with pure substances; the melting point corresponds to the onset. The downward movement of the peak in DSC heating curve indicates that the peak is endothermic peak. A large high temperature endothermic peak at 253.62^{0}C in a DSC heating curve is a melting peak. The thermal data in DSC curve of lead carbonate nanoparticles are shown in table.1.

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

Sample/Properties	**Lead carbonate nanoparticles**
	High temperature endothermic peak
Quantity	7.925mg
Heating Rate	10^{0}C/min
Onset	253.62^{0}C
Peak	275.09^{0}C
Endset	288.24^{0}C

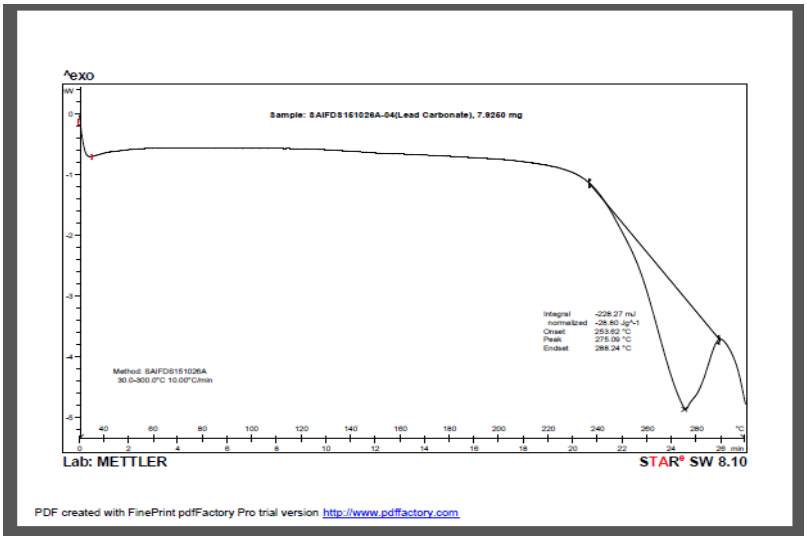

Fig.4. DSC curve of lead carbonate nanoparticles

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TGA-DTA ANALYSIS

Thermal analysis of lead carbonate nanoparticles is also carried out by thermogravymetric analysis (TGA) and differential thermal analysis. 26.331mg of lead carbonate nanoparticles at a temperature from 40 °C to 830 °C at 20 °C/minute is analyzed. TGA/DTA thermograms of lead carbonate nanoparticles are shown in fig.5.The thermal analysis data of lead carbonate nanoparticles are shown in table.2. The descending TGA thermal curve indicates a weight loss occurred. The TGA curve represents the multi stage decomposition of the sample [3]. The area in the DTA curve is 1438.071mJ and ΔH value is 54.6155J/g and the peak is 276.66°C. The three peaks in TGA curve are 291.03°C, 346.46°C and 609.29°C indicates the decomposition of the sample in three stages.

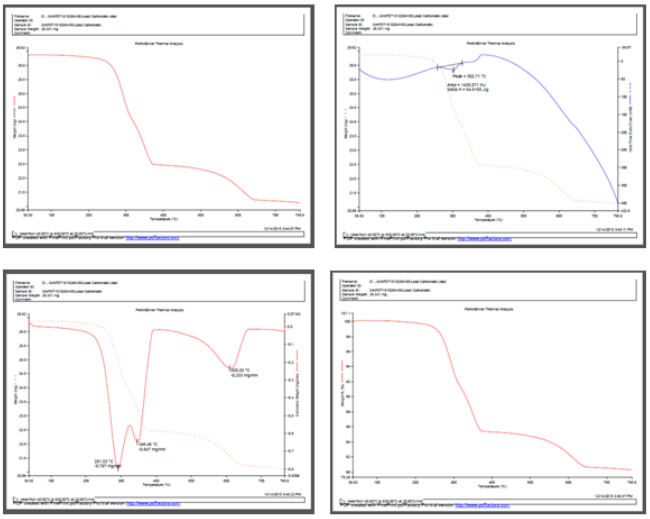

Fig.5. TGA/DTA thermograms of lead carbonate nanoparticles

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

Material	TGA temperature (^{0}C)	DSC peak temperature (^{0}C)	DTA weight loss (%)
Lead carbonate nanoparticles	291.03	275.09	24.95

IV. CONCLUSION

The lead carbonate nanoparticles have been prepared by chemical co-precipitation method. TEM analysis suggests that the average particle size is 7.4±4nm, the standard deviation is 4nm and the polydispersity of lead carbonate nanoparticles is 54% 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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[3] Tiverios C. Vaimakis, Thermo gravimetric Analysis.