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Rapid fabrication of nanostructured magnesium hydroxide and hydromagnesite via microwave-assisted technique

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ABSTRACT

Magnesium hydroxide and hydromagnesite nano and microstructures have been prepared by using microwave-assisted technique. Magnesium chloride, magnesium acetate and magnesium metal have been used as a magnesium source. Urea has been added to the solution with controlled pH = 10 while the temperature was 220 °C. The hydrolysis of urea under these hydrothermal conditions leads to the production of hydromagnesite instead of magnesium hydroxide. The as-prepared samples were investigated using scanning electron microscope (SEM), X-ray diffraction (XRD), thermographmetric analysis (TGA) and differential scanning calorimetry (DSC). SEM shows a wide distribution of pseudo-hexagonal nanodisk up to microdisk. Spherical rosette morphology has been noticed in the samples in which the urea has been used. XRD reveals the existence of $Mg(OH)_2$ with some impurities which can be attributed to the high concentrations of the initial materials and absorption of carbon dioxide from the air when urea is not employed and hydromagnesite when urea is present in the synthesis. TGA showed a weight loss within the temperature range of 360–450 °C with a total percentage of weight loss 29% which can be attributed to the ($Mg(OH)_2$). However in the case of synthesis with urea the weight loss was 57% which is the expected value for hydromagnesite. The production of pure hydromagnesite utilizing hydrothermal methods has been reported; the synthesis reported here is much simpler and faster.

1. Introduction

Magnesium hydroxide ($Mg(OH)_2$) has many applications due to its outstanding physical and chemical properties. For example due to it structural properties it can be formed in laminated form and its good compatibility with many polymer matrices, it is widely used as filler. Moreover, magnesium hydroxide ($Mg(OH)_2$) possessing an advantageous arrangement for the formation of platelet-like crystals, has high decomposition temperature and it is commonly used as a flame retardant in the composite materials [1–4].

There are many methods that can be used for the $Mg(OH)_2$ synthesis such as electrolysis from an aqueous magnesium salt solution, hydration of magnesium oxide, precipitation of a magnesium salt in alkaline solution, sol–gel technique, ammonia gas bubbling reactors and microwave assisted synthesis [5].

For example, $Mg(OH)_2$ with different morphologies such as rod tube needle or lamina have been synthesized by hydrothermal reaction using different magnesium sources such as magnesium powder, $MgSO_4$ and $Mg(NO_3)_2 \cdot 6H_2O$. The authors found that the crystallite size, shape, and structure, can be controlled well by choosing different solvents and reaction conditions [6].

Surfactants or structure directing agents have been used to control the size and shape of the metal oxides nanostructures. In the open literatures, there are a few reports about using urea in order to control the $Mg(OH)_2$ synthesis. For example, Kumari, et al. found that the addition of urea together with PEG is advantageous in controlling the nanoparticles' growth. These reports however were conducted at below 110 °C and only reported a small impurity of hydromagnesite [7].

The crystallization behavior of the $Mg(OH)_2$ also depends on the magnesium source; for example Gao et al. found that the sunflower-like structures were obtained for magnesium Chloride ($MgCl_2$) and with the column-shaped superstructures when magnesium dodecyl sulfate [$Mg(DS)_2$] was used [8].

Hydromagnesite ($Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$) is one of the only stable hydrated hydroxy carbonates at room temperature. It is a better flame retardant than $Mg(OH)_2$ since it yields more water and CO_2 per mass and has been widely used as a filler in polymer systems.

Microwave technique attracts more interest because it can generate heat internally inside the sample so the heat can be transferred homogenously and rapidly. The microwave technique requires far

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Table 1
Reactant proportions for synthesis experiments.

Experiment	Mg metal (g)	MgCl2 (g)	Mg(C2O2H4)2 (g)	NaOH (g)	Urea (g)
Reaction 1	1			16.5 in 25 ml H2O	
Reaction 2		1 in 100 ml H2O			1.8 in 25 ml H2O
Reaction 3			6.44 in 100 ml H2O		1.2 in 25 ml H2O

less energy than resistive heating and is less polluting than gas or oil fired heaters and is therefore more environmentally friendly. Moreover the rapid initial heating of the microwave can enhance the kinetics of the reaction due to the formation of high temperature throughout the sample [9].

Herein, $Mg(OH)_2$ has been synthesized via the microwave-assisted technique using Magnesium Chloride, urea and water. In addition Magnesite in high purity with fine nanostructure has been synthesized b addition of urea to the reaction mixture. The as-synthesized

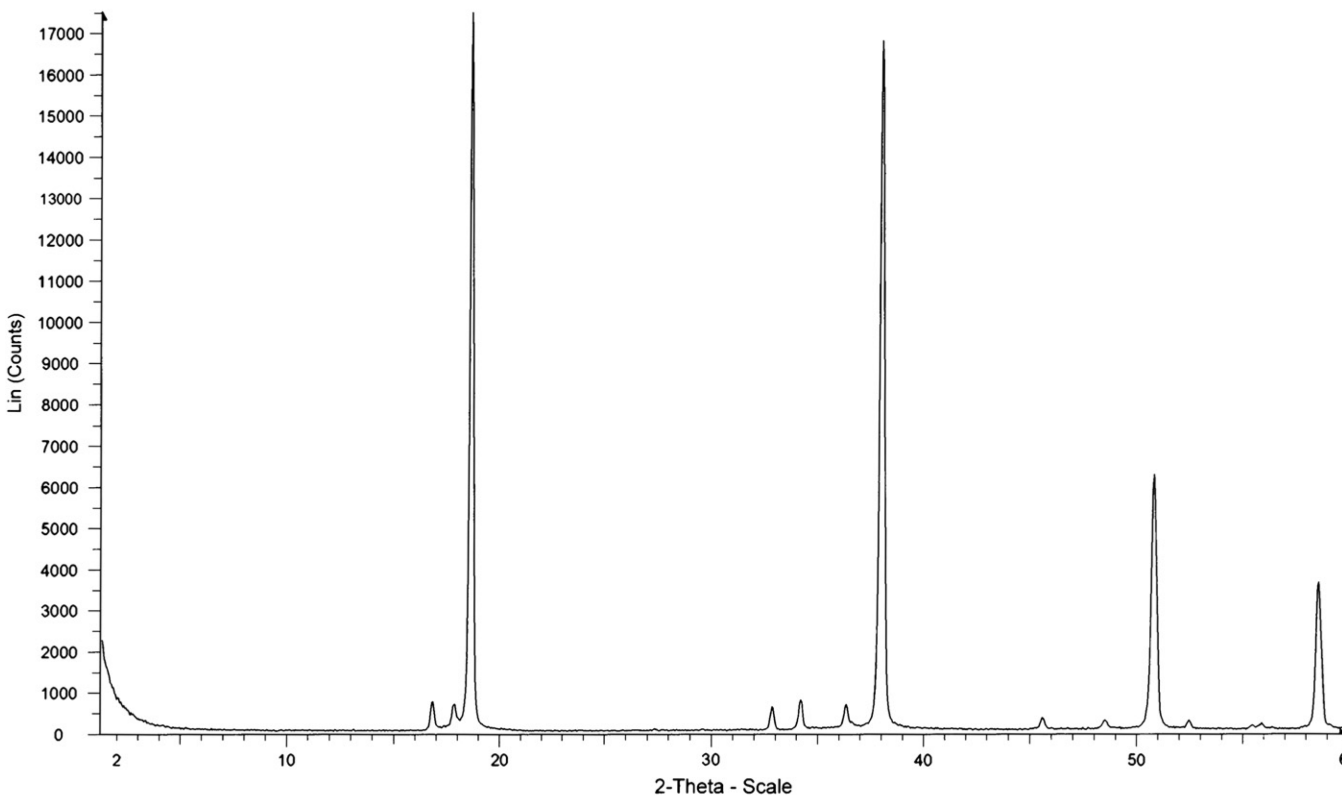

Fig. 1. Wide angle X-ray diffraction pattern of reactor sample 1.

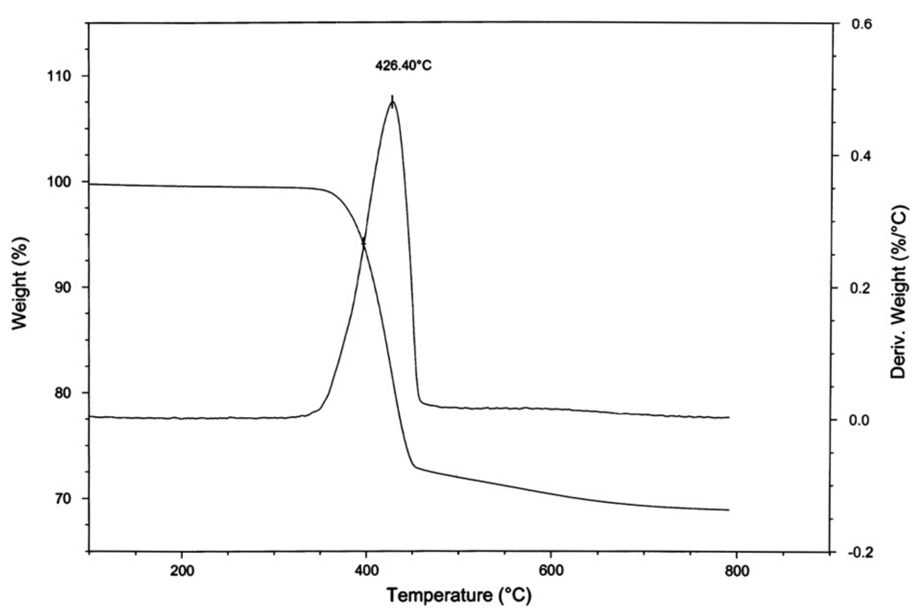

Fig. 2. TGA plot of sample 1.