Precipitation of Modified Nano Molecular Magnesium Oxide from Bittern and Ammonia

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Pilot scale magnesium hydroxide was precipitated by ammonia solution and concentrated bittern of 58 Mg²⁺ g/L. The effect of poly-ethylene glycol (600)PEG and ethylene glycol EG in crystalline size of Mg(OH)₂ was investigated through X-ray diffraction(XRD). To confirm the presence of functional groups introduced by modifiers the samples were subjected to Fourier transforms infrared spectroscopy (FTIR). Morphology properties of the selected samples were characterized by SEM images and EDX analysis confirmed the low carbon content and high oxygen % in product with Poly ethylene glycol (PEG). Thermal Gravimetric Analysis (TGA) and (DSC) were used to investigate PEG different ratios effect on Mg(OH)₂ crystallite and stability of product. The PEG 0.5% ratio addition shows the best sample properties in all analysis. Pure Nano- magnesium hydroxide was prepared by calcination at 500 °C producing flower plates with XRD 2θ= 38.16 °

Keywords: Bittern, Pilot chemical precipitation, PEG 600, Mg(OH)₂, Crystal structure.

Introduction

Mg(OH)₂ is one of the most important precursors of magnesium oxide, new method of synthesis of nano- magnesium hydroxide with high thermal stability when using poly ethylene glycols as modifiers. Recently, different methods were proposed for nano-Mg(OH)₂ preparation[1]. The precipitation method is only the one that is simple, non-expensive equipment that makes the process of production becomes economically attractive. These precipitation methods are easy to conduct on an industrial scale[2]. The use of modifiers as an additional organic or inorganic chemical substance in the direct precipitation are used to modify the original properties of magnesium hydroxide[3]. As to make product with more hydrophobic property with controlled shape and size of particles which crystallizes as needles, plates, rods, tubes and flowers [4]. The purpose of such modification of original properties of reaction products is to increase the activity and selectivity of catalysts based Mg(OH)₂ [5]. The modification results in changing hydrophilic character of the surface into hydrophobic, which improves mechanical properties and help in reduce the magnesium hydroxide particle size and develop their surface area[6,7]. Recently, poly ethylene glycols are cheap and have beneficial effect on the microstructure and surface character of Mg(OH)₂ [8]. The nanostructure materials have unique properties due to their interesting physico-chemical properties and wide range of application in Nano-devices [9-11] and [12].

Experimental

Materials:

All chemicals used in this work were A.R. grade, such as ethanol, ammonia solution(33 %– 35%) was used as precipitating agent, poly ethylene glycol (PEG600) and ethylene glycol (EG) were used singly with different molar ratios as modifiers and were used directly. All chemicals were ADWIC CO and NASR PHARMACEUTICAL CHEMICAL CO.
The concentrated liquid bittern 58% Mg\(^{2+}\) g/L was used as substrate for the synthesis of Nano-
magnesium hydroxide. Bittern was obtained from EL- MEX SALINES COMPANY, Alexandria, Egypt.

**Synthesis of Mg(OH)\(_2\), Nano particles on Pilot scale:**

Reaction of magnesium hydroxide precipitation on pilot scale was carried out in reactor of 100 litre capacity, equipped with a rotary stirrer 700 rpm. The process was conducted at normal temperature 25 – 30 °C. The dissolved PEG or EG in ethyl alcohol were dosed gradually into the bittern with continuous stirring. Surfactant is used to confirm homogeneity of solution, prevent agglomeration and have good dispersion during the reaction. A mixture of dissolved PEG or EG in alcohol and ammonia was added drop wise to bittern solution.

On completion of reagents introduction, the reaction system was stirred continuously for two hours. The precipitate of Mg(OH)\(_2\) was allowed to settle 48 h. The precipitate was filtered off, washed twice, and dried by solar energy for two days. Finally a portion of the dried material was calcined in air at 500 °C for 4 h to obtain pure Mg(OH)\(_2\) powder.

**Results and Discussion**

The different samples prepared were subjected to FTIR analysis to record the interaction between magnesium hydroxide and PEG600 or EG as shown in Fig. 2 and 3.

FTIR analysis of magnesium hydroxide precipitated with PEG600 or EG as modifiers are illustrated in the spectra indicating the presence of the functional groups introduced with 1%, 0.5%, 0.25% PEG and 10%, 5% and 2.5% EG respectively. Fig(2)\(_a\), Fig(2)\(_b\), Fig(2)\(_c\), and Fig(2)\(_d\) shows the Mg(OH)\(_2\) samples modified by different PEG %. The spectra of modified sample with PEG shows the bands assigned to the functional groups O-H (PEG) C-H (PEG) with significant higher intense than those bands corresponds to samples modified with EG as shown in Fig (3)\(_a\), Fig (3)\(_b\), and Fig(3)\(_c\).

The first absorption maxima, of sharp and high intensity at 3697 cm\(^{-1}\) in Fig (2) is assigned to the asymmetric stretching vibration of –OH groups from Mg(OH)\(_2\). The band at 1639 cm\(^{-1}\) is assigned to the stretching vibration of the OH groups from water. The broad peak at 3414
cm\textsuperscript{-1} corresponds to the adsorption –OH PEG groups, while the low-intensity bands at 2163 cm\textsuperscript{-1} 2065 cm\textsuperscript{-1} presents the methylene groups – CH\textsubscript{2} (stretching vibrations of PEG). The broad peak at 1413 cm\textsuperscript{-1} also presents CH\textsubscript{2} groups from PEG (bending-scissoring vibrations). The bands at 1130 cm\textsuperscript{-1} corresponds to the asymmetric stretching vibration of ether groups C-O-C PEG. The intense and broad maxima at 445 cm\textsuperscript{-1} represents the stretching vibration of Mg-O. Only a significant band at 1637 cm\textsuperscript{-1} in Fig( 3) \textsubscript{a} at 2166 cm\textsuperscript{-1} in Fig(3) \textsubscript{b} at 2171 cm\textsuperscript{-1} in Fig (3) \textsubscript{b}.

Fig. 2a. FTIR spectrum of Mg(OH)\textsubscript{2} unmodified.

Fig. 2b. FTIR spectrum of Mg(OH)\textsubscript{2} modified With 1% PEG Peaks picking: 3697 cm\textsuperscript{-1}, 3425 cm\textsuperscript{-1}, 2022 cm\textsuperscript{-1}, 1638 cm\textsuperscript{-1}, 1438 cm\textsuperscript{-1}, 1126 cm\textsuperscript{-1}, 442 cm\textsuperscript{-1}.

Fig. 2c. FTIR spectrum of Mg(OH)\textsubscript{2} modified With 0.5% PEG Peaks picking: 3697 cm\textsuperscript{-1}, 3414 cm\textsuperscript{-1}, 2163 cm\textsuperscript{-1}, 2065 cm\textsuperscript{-1}, 1639 cm\textsuperscript{-1}, 1413 cm\textsuperscript{-1}, 1130 cm\textsuperscript{-1}, 444 cm\textsuperscript{-1}.

Fig. 2d. FTIR spectrum of Mg(OH)\textsubscript{2} modified With 0.25% PEG Peaks picking: 3698 cm\textsuperscript{-1}, 3413 cm\textsuperscript{-1}, 2170 cm\textsuperscript{-1}, 1638 cm\textsuperscript{-1}, 1406 cm\textsuperscript{-1}, 1130 cm\textsuperscript{-1}, 435 cm\textsuperscript{-1}.
Fig. 3a. FTIR spectrum of Mg(OH)₂ modified with 10% EG Peaks picking: 3408 cm⁻¹, 1637 cm⁻¹, 1404 cm⁻¹, 1128 cm⁻¹, 437 cm⁻¹

Fig. 3b. FTIR spectrum of Mg(OH)₂ modified with 5% EG Peaks picking: 3409 cm⁻¹, 2166 cm⁻¹, 1405 cm⁻¹, 1131 cm⁻¹, 432 cm⁻¹.

Fig. 3c. FTIR spectrum of Mg(OH)₂ modified with 2.5% EG Peaks picking: 3407 cm⁻¹, 2171 cm⁻¹, 1083 cm⁻¹, 433 cm⁻¹

corresponds to –OH of water.

The broad peaks at 3408 cm⁻¹, 3409 cm⁻¹, and 3407 cm⁻¹ corresponds to the adsorbed –OH of EG groups. The broad peak at 1404 cm⁻¹ and 1405 cm⁻¹ is assigned to –CH₂ groups from EG (bending – scissoring vibrations). While bands at 1128 cm⁻¹, 1131 cm⁻¹ and 1083 cm⁻¹ represents the asymmetric stretching vibrations of ether groups C-O-C EG. The broad maxima at 437 cm⁻¹, 432 cm⁻¹ and 433 cm⁻¹, corresponds to stretching vibration of Mg-O.

From the above FTIR data the PEG showed more significant peaks for introduced functional groups and our results agreed with Wang P. et al. (2011) [13]. When PEG was added to nano-Mg(OH)₂, the bands are more modified, with respect to pure Mg(OH)₂, bands at 1413 cm⁻¹ mainly shifted to 1126 cm⁻¹, 1130 cm⁻¹ and 1100 cm⁻¹ (C-
O) stretching vibration due to PEG interaction with surface of nano- Mg(OH)$_2$ particles. In FTIR spectrum, we found several changes including new bands at 2065 cm$^{-1}$, 2022 cm$^{-1}$, 2163 cm$^{-1}$ belonging the C-H stretching vibration in \(-\text{CH}\) group. The bands at 3425 cm$^{-1}$, 3414 cm$^{-1}$ Fig (2, 2$\alpha$) and 3408 cm$^{-1}$, 3409 cm$^{-1}$, 3407 cm$^{-1}$ Fig (3, 3$\alpha$, 3$\beta$) is related with the stretching vibration of hydroxyl group, this band is broadened with an increased intensity with respect to PEG or EG. This suggests that the interaction of PEG or EG may occur through hydrogen bonding of the hydroxyl groups on nano Mg(OH)$_2$ surface. Our results agreed with Aline et al.[9].

**DSC-TGA analysis**

The DSC-TGA measurements were carried out to analyze thermal behavior and decomposition process of the obtained Mg(OH)$_2$ powders. A representative DSC-TGA profile is shown in Fig(4) and Fig(5).

Table 1 and Table 2 shows the variation in weight loss% and endothermic transition at weight loss of Mg(OH)$_2$ modified samples with PEG or EG as. The best ratio is assigned at 0.5% and 5% for PEG or EG addition, showing weight loss about 4.4% and 25% respectively. The DSC- TGA analysis shows endothermic transition at 420$^\circ$C and 428$^\circ$C with weight loss 33% and 52% shown in Fig 4 and Fig 5. This can be ascribed to the decomposition of Mg(OH)$_2$. The major weight loss happens for Mg(OH)$_2$–PEG at 0.5% addition and Mg(OH)$_2$–EG 5% addition at temperature 780$^\circ$C and 500$^\circ$C respectively; of almost 25.74% and 20.70% respectively indicates the decomposition of magnesium hydroxide to magnesium oxide and more stability of Mg(OH)$_2$–.
Fig. 4c. Thermogravimetric (upper solid line) and deferential scanning calorimetric (DSC) pilot peaks of the Mg(OH)$_2$ powder with 0.25% PEG600.

Fig. 5a. Thermogravimetric (upper solid line) and deferential scanning calorimetric (DSC) pilot peaks of the Mg(OH)$_2$ powder with 10% EG.

Fig. 5b. Thermogravimetric (upper solid line) and deferential scanning calorimetric (DSC) pilot peaks of the Mg(OH)$_2$ powder with 5% EG.

Fig. 5c. Thermogravimetric (upper solid line) and differential scanning calorimetric (DSC) pilot peaks of the Mg(OH)₂ powder with 2.5% EG.

TABLE 1. Shows the effect of PEG addition on weight loss of Mg(OH)₂ prepared:

<table>
<thead>
<tr>
<th>Ratio of PEG</th>
<th>0%</th>
<th>1%</th>
<th>0.5%</th>
<th>0.25%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight loss%</td>
<td>10%</td>
<td>11%</td>
<td>4.4%</td>
<td>14.6%</td>
</tr>
<tr>
<td>OH type</td>
<td>2.359 mg</td>
<td>2.700 mg</td>
<td>0.515 mg</td>
<td>0.8811 mg</td>
</tr>
<tr>
<td>Endothermic transition at °C</td>
<td>432°C</td>
<td>436°C</td>
<td>420°C</td>
<td>392°C</td>
</tr>
<tr>
<td>Endothermic transition at weight loss %</td>
<td>40%</td>
<td>49%</td>
<td>33%</td>
<td>46%</td>
</tr>
</tbody>
</table>

TABLE 2. Shows the effect of EG addition on weight loss of Mg(OH)₂ prepared:

<table>
<thead>
<tr>
<th>Ratio of EG</th>
<th>0%</th>
<th>10%</th>
<th>5%</th>
<th>2.5%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight loss%</td>
<td>10%</td>
<td>33%</td>
<td>4.4%</td>
<td>14.6%</td>
</tr>
<tr>
<td>OH type</td>
<td>2.359 mg</td>
<td>3.484 mg</td>
<td>0.3365 mg</td>
<td>1.317 mg</td>
</tr>
<tr>
<td>Endothermic transition at °C</td>
<td>432°C</td>
<td>473°C</td>
<td>428°C</td>
<td>443°C</td>
</tr>
<tr>
<td>Endothermic transition at weight loss %</td>
<td>40%</td>
<td>54%</td>
<td>52%</td>
<td>54%</td>
</tr>
</tbody>
</table>

Morphology analysis of Mg(OH)₂:

PEG than Mg(OH)₂ – EG. This major decomposition illustrated as the beginning formation of MgO from their respective hydroxide form and the water as by-product released.

SEM images of Mg(OH)₂ prepared with addition of PEG600 and EG are shown in Fig (6) a, Fig (6) b, Fig(6) c, and Fig(7) shows MgO. Fig(6) illustrates image of magnesium hydroxide without modifier addition. Fig (6) a and Fig (6) b illustrates the images of nano-crystalline structure of Mg(OH)₂. While Fig(6) c shows image of smaller crystallite size due to PEG addition during precipitation. Fig(6) a shows plate crystallite structure. It is believed that PEG600 facilitates homogeneity and dispersion of particles during synthesis with a significant effect on Mg(OH)₂ nano-crystals.

SEM images of pure Mg(OH)₂ prepared on pilot scale with PEG addition as modifier is shown in Fig(7) illustrating the flower of plates shaped in homogenous crystallite, indicating purity of product in Nano-size. This is confirmed by EDX analysis Fig(8) and Fig(9) indicating lower carbon content compared to EG modifier addition.

Fig. 6a. SEM image of Mg(OH)$_2$ powder precipitated without modifier

Fig. 6b. SEM image of Mg(OH)$_2$ powder precipitated with PEG600 0.5% modifier

Fig. 6c. SEM image of Mg(OH)$_2$ powder precipitated with EG 5% modifier.

Fig. 7. SEM image of pure calcined Mg(OH)$_2$ powder precipitated with PEG 0.5% modifier

EDX analysis as indication of elemental analysis shows lower carbon content in modified Mg(OH)$_2$ samples by 0.5% PEG600 compared to Mg(OH)$_2$ samples modified with 2.5% of EG as shown in Fig (8) and Fig(9) as follows.

Table (3) shows modified nano Mg(OH)$_2$ – PEG600 illustrating oxygen content 62.12% indicating more hydrogen bonding interacted with polymer on surface area which inhibits wettability formation.

Table (4) shows modified Mg(OH)$_2$ – EG illustrating less oxygen content 37.57% indicating hydrogen bonding formation on surface which inhibits wettability formation, but with lower extent compared to hydrogen bonding of Mg(OH)$_2$ – PEG.

From Table (3) and Table(4) the elemental analysis(EDX) shows in nano Mg(OH)$_2$ –PEG increase in oxygen 62.12 % more than in nano Mg(OH)$_2$ – EG 37.5%, this is due to the modified surface porosity of nano Mg(OH)$_2$ –PEG is more than that of nano Mg(OH)$_2$ – EG. These changes on the surface configuration (presence of more hydrogen bonding) leads to improve more resistance for water adsorption. Our results agree with Hiromot et al.[12].

**X-ray Diffraction analysis(XRD):**

The X-ray diffraction patterns for selected samples A, B, and C revealed the occurrence of brucite (hexagonal) Mg(OH)$_2$, (ICSD card No. 01-086-0441) as the major phase with trace of minor phase represented by ammonium magnesium chloride hydrate NH$_4$Mg Cl$_3$.6H$_2$O (orthorhombic) (ICSD Card No. 00-0025-0039). The occurrence of ammonium magnesium chloride hydrate NH$_4$Mg Cl$_3$.6H$_2$O (orthorhombic) varies from one sample to another with sample C being the greatest which could explain the high contrast in the scanning electron microscope image for this sample. The X-ray diffraction pattern for sample D revealed the occurrence of pure brucite phase with no trace of other phases Fig.(10D).
listed in Table (3), it can be observed that the particle size values in the direction perpendicular to the (001) plane direction are lower than the particle size values in the direction perpendicular to the (011) plane direction, which indicates that the particles have a morphology of thin plate shape with thin layers in the (001) plane direction. The ratio between particle size in the direction perpendicular to the (001) plane direction to the particle size in the direction perpendicular to the (011) plane direction is equal to 0.85 closer to unity for sample 2 while it is 0.68 for sample C. This explains the appearance of needle shaped particles in the SEM graph along with the plate shaped particles Fig. (12). The particle size values of all samples were compared to the particle size of the standard nano sample and it was found that samples A and D had values close to the standard values, while samples B and C were higher than the standard values (estimated error about ± 10%).

The identification of modified samples, with PEG600 and EG were carried out by XRD patterns showing the Mg(OH)$_2$ with different addition% of(1%, 0.5%, 0.25%) and EG was added to Mg(OH)$_2$ with(10%, 5%, 2.5%) respectively. Mg(OH)$_2$ with 0.5% PEG addition was converted into pure Mg(OH)$_2$ by thermal treatment 4h at 500°C in air. The crystallite of Mg(OH)$_2$ powders are characterized by intense peaks locations in 2Ɵ° shown in Table (3) and Table (4). The crystallite size of samples were calculated using Scherer’s

![Fig. 10. SEM image of pure calcined 8h MgO powder precipitated with PEG 0.5% modifier.](image)

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
<th>Net Int.</th>
<th>Error %</th>
</tr>
</thead>
<tbody>
<tr>
<td>C K</td>
<td>8.05</td>
<td>11.85</td>
<td>35.66</td>
<td>12.82</td>
</tr>
<tr>
<td>O K</td>
<td>56.18</td>
<td>62.12</td>
<td>1259.2</td>
<td>6.87</td>
</tr>
<tr>
<td>MgK</td>
<td>35.77</td>
<td>26.03</td>
<td>1658.16</td>
<td>5.78</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
<th>Net Int.</th>
<th>Error %</th>
</tr>
</thead>
<tbody>
<tr>
<td>C K</td>
<td>42.72</td>
<td>53.44</td>
<td>23.81</td>
<td>13.01</td>
</tr>
<tr>
<td>O K</td>
<td>40.01</td>
<td>37.57</td>
<td>40.91</td>
<td>12.65</td>
</tr>
<tr>
<td>MgK</td>
<td>8.36</td>
<td>5.16</td>
<td>44.12</td>
<td>8.85</td>
</tr>
<tr>
<td>S K</td>
<td>1.04</td>
<td>0.49</td>
<td>7.07</td>
<td>17.97</td>
</tr>
<tr>
<td>ClK</td>
<td>7.88</td>
<td>3.34</td>
<td>47.98</td>
<td>5.39</td>
</tr>
</tbody>
</table>
Fig. 11. EDX Spectra of pure MgO modified with 0.5% PEG showing low carbon content

Fig. (12A,B,C,D) XRD pattern of Sample A, B, C, and D for Mg(OH)2 with 1% PEG, 0.5% PEG, 10% EG, and 5% EG modifiers respectively.

Fig. 13. XRD pattern of Pure calcined Mg(OH)2 with 0.5% PEG modifier

formula [14, 15]. Results are shown in Fig. (12_A), Fig(12_B), Fig(12_C), Fig(12_D), for modified product by PEG and EG respectively. Fig(13) and Fig(14) shows the XRD pattern for pure calcined Mg(OH)_2 and standard nano Mg(OH)_2.

These results of XRD and Table(5) indicated different particle sizes proves that addition of PEG600 0.5% and addition of EG 5% ratio for samples A and D gave better crystal Nano sizes than samples B and C with PEG600 addition of 0.25% and 5% EG respectively.

**TABLE 5. Particle size of magnesium hydroxide samples prepared under different modifiers addition.**

<table>
<thead>
<tr>
<th>Sample No</th>
<th>Peak Pos. (2θ)</th>
<th>Miller indices (hkl)</th>
<th>FWHM(2θ)</th>
<th>Cos(2θ)</th>
<th>β(2θ)</th>
<th>Particle size(nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>18.7261</td>
<td>(001)</td>
<td>0.3542</td>
<td>0.987</td>
<td>0.0062</td>
<td>22.743</td>
</tr>
<tr>
<td></td>
<td>38.0601</td>
<td>(011)</td>
<td>0.3149</td>
<td>0.945</td>
<td>0.0055</td>
<td>26.701</td>
</tr>
<tr>
<td></td>
<td>18.6427</td>
<td>(001)</td>
<td>0.2755</td>
<td>0.987</td>
<td>0.0048</td>
<td>29.240</td>
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<tr>
<td>B</td>
<td>37.9888</td>
<td>(011)</td>
<td>0.1968</td>
<td>0.946</td>
<td>0.0034</td>
<td>42.730</td>
</tr>
<tr>
<td>C</td>
<td>18.6765</td>
<td>(001)</td>
<td>0.2755</td>
<td>0.987</td>
<td>0.0048</td>
<td>29.242</td>
</tr>
<tr>
<td></td>
<td>38.1534</td>
<td>(011)</td>
<td>0.1968</td>
<td>0.945</td>
<td>0.0034</td>
<td>42.751</td>
</tr>
<tr>
<td>D</td>
<td>18.6252</td>
<td>(001)</td>
<td>0.3149</td>
<td>0.987</td>
<td>0.0055</td>
<td>25.579</td>
</tr>
<tr>
<td></td>
<td>38.0710</td>
<td>(011)</td>
<td>0.2755</td>
<td>0.945</td>
<td>0.0048</td>
<td>30.523</td>
</tr>
<tr>
<td>Std</td>
<td>18.4356</td>
<td>(001)</td>
<td>0.3149</td>
<td>0.987</td>
<td>0.0055</td>
<td>25.572</td>
</tr>
<tr>
<td>Pure S.</td>
<td>38.0920</td>
<td>(011)</td>
<td>0.2755</td>
<td>0.945</td>
<td>0.0048</td>
<td>30.525</td>
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<tr>
<td></td>
<td>38.16</td>
<td>(011)</td>
<td>0.3542</td>
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</table>

**Conclusions**

The modification of Nano-magnesium hydroxide with addition of PEG600 or EG resulted in increase in specific area of Mg(OH)_2 powder samples. The modifiers brought beneficial changes in the character of Mg(OH)_2 surface and showed reduce in ability to absorb water related to degree of coverage of their surface with the modifier. As shown from EDX elemental analysis the oxygen content for Mg(OH)_2–PEG 62.12% and for Mg(OH)_2 – EG 37.5% indicating more...
hydrogen bonding with nano Mg(OH)₂–PEG than for nano Mg(OH)₂–EG. This phenomena was confirmed by FTIR spectroscopy with increase of broad bands of –OH group with addition of different dose of PEG and EG. Samples modified with 0.5% PEG600 and EG 5% improved the formation of smaller Nano-particle sizes. The Results of FTIR analysis confirmed the presence of functional groups coming from PEG and EG in modified samples, and others concerned to hydrogen bonding on the surface area of nanomaterials prepared. The present study shows that the crystalline Mg(OH)₂ Nano-particles can be synthesized in pure state from liquid bittern, this was confirmed with XRD analysis with presence of single crystal peak formed. Mg(OH)₂ crystals Nano-plates have specific surface area and aggregate into large spherical particles, as shown by SEM and EDX. TGA and DSC analysis showed more thermal stability of nano Mg(OH)₂–PEG at 780 ⁰C than nano Mg(OH)₂–EG at 500⁰C due to formation of more hydrogen bonding and inhibition of wettability. The simplicity of the precipitation process, low cost and availability of raw materials would favor scale-up industrial manufacturing.

Acknowledgment

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