



## Precipitation of Modified Nano Molecular Magnesium Oxide from Bittern and Ammonia

Sh. El Rafie<sup>1</sup>, Marwa S. Shalaby<sup>1</sup>, Fatma M. Zaher<sup>1</sup>, Rania Ramadan<sup>1</sup>, Tarek A. Abdel-halim<sup>2</sup>

<sup>1</sup>National Research Center, Chemical Engineering and Pilot Plant Department, EL Bohouth St. Dokki- Giza-Egypt- P. O. 12622.

<sup>2</sup>Nuclear Materials Authority, Kattamia road, Maadi, Egypt, P.O. Box 530.



**P**ILLOT scale magnesium hydroxide was precipitated by ammonia solution and concentrated bittern of 58 Mg<sup>2+</sup> g/L. The effect of poly-ethylene glycol (600)PEG and ethylene glycol EG in crystalline size of Mg(OH)<sub>2</sub> 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)<sub>2</sub> 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)<sub>2</sub>, Crystal structure.

### Introduction

Mg(OH)<sub>2</sub> 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)<sub>2</sub> 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)<sub>2</sub>

[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)<sub>2</sub> [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.

\*Corresponding author e-mail: shelrafie0000@yahoo.com

Received 29/7/2019; Accepted 2/1/2020

DOI: 10.21608/ejchem.2020.14178.1933

©2019 National Information and Documentation Center (NIDOC)

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.

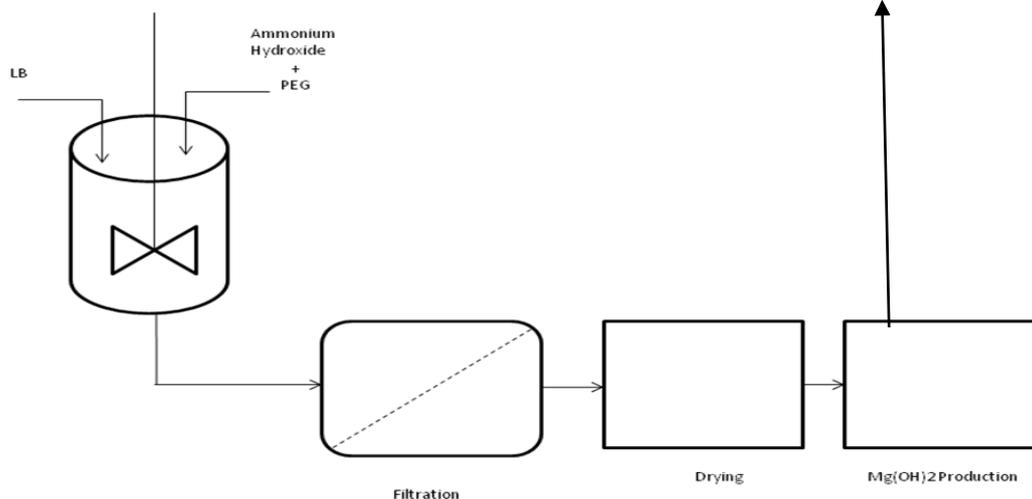
FTIR analysis of magnesium hydroxide precipitated with PEG600 or EG as modifiers are illustrated in the spectra indicating the presence of the functional groups introduced with 1%, 0.5%, 0.25% PEG and 10%, 5% and 2.5% EG respectively. Fig(2)<sub>a</sub> shows unmodified sample of  $Mg(OH)_2$ . Fig (2)<sub>b</sub>, Fig(2)<sub>c</sub>, and Fig(2)<sub>d</sub> 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 intensity than those bands corresponds to samples modified with EG as shown in Fig (3)<sub>a</sub>, Fig (3)<sub>b</sub> and Fig(3)<sub>c</sub>.

The first absorption maxima, of sharp and high intensity at 3697  $cm^{-1}$  in Fig (2)<sub>c</sub> 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

$Mg(OH)_2$  heated at 500°C 4h

Crystalline  $Mg(OH)_2$

SEM of Pure  $Mg(OH)_2$



**Fig. 1. Scheme of Magnesium hydroxide preparation on pilot scale using ammonia and Bittern**

$\text{cm}^{-1}$  corresponds to the adsorption  $-\text{OH}$  PEG groups, while the low-intensity bands at  $2163 \text{ cm}^{-1}$   $2065 \text{ cm}^{-1}$  presents the methylene groups  $-\text{CH}_2$  (stretching vibrations of PEG). The broad peak at  $1413 \text{ cm}^{-1}$  also presents  $\text{CH}_2$  groups from PEG (bending-scissoring vibrations). The bands

at  $1130 \text{ cm}^{-1}$  corresponds to the asymmetric stretching vibration of ether groups  $\text{C}-\text{O}-\text{C}$  PEG. The intense and broad maxima at  $445 \text{ cm}^{-1}$  represents the stretching vibration of  $\text{Mg}-\text{O}$ . Only a significant band at  $1637 \text{ cm}^{-1}$  in Fig(3)<sub>a</sub> > at  $2166 \text{ cm}^{-1}$  in Fig(3)<sub>b</sub> > at  $2171 \text{ cm}^{-1}$  in Fig(3)<sub>c</sub>

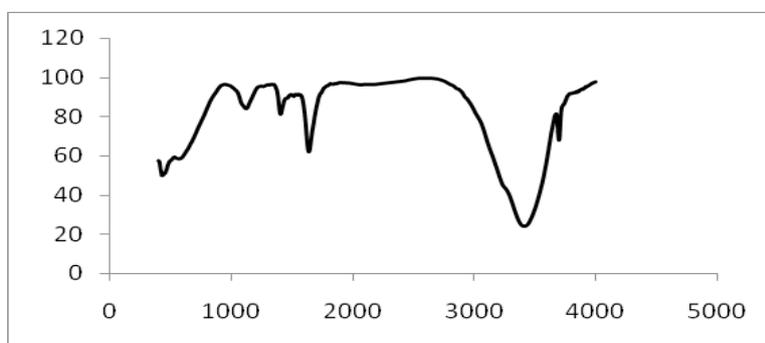


Fig.2a . FTIR spectrum of  $\text{Mg}(\text{OH})_2$  unmodified.

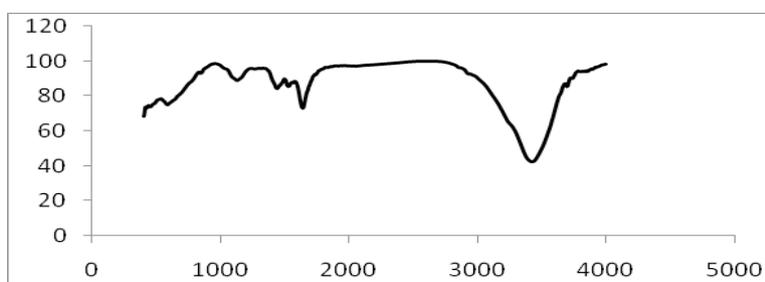


Fig. 2b. FTIR spectrum of  $\text{Mg}(\text{OH})_2$  modified With 1% PEG Peaks picking:  $3697 \text{ cm}^{-1}$ ,  $3425 \text{ cm}^{-1}$ ,  $2022 \text{ cm}^{-1}$ ,  $1638 \text{ cm}^{-1}$ ,  $1438 \text{ cm}^{-1}$ ,  $1126 \text{ cm}^{-1}$ ,  $442 \text{ cm}^{-1}$ .

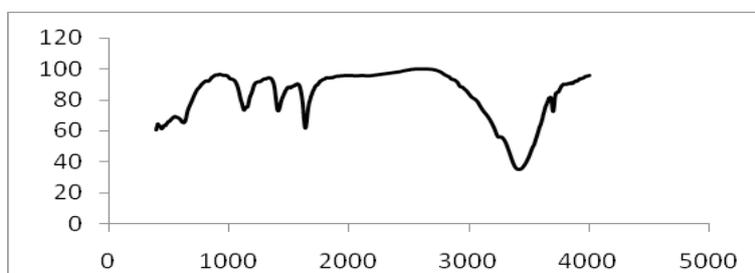


Fig. 2c. FTIR spectrum of  $\text{Mg}(\text{OH})_2$  modified With 0.5% PEG Peaks picking:  $3697 \text{ cm}^{-1}$ ,  $3414 \text{ cm}^{-1}$ ,  $2163 \text{ cm}^{-1}$ ,  $2065 \text{ cm}^{-1}$ ,  $1639 \text{ cm}^{-1}$ ,  $1413 \text{ cm}^{-1}$ ,  $1130 \text{ cm}^{-1}$ ,  $444 \text{ cm}^{-1}$

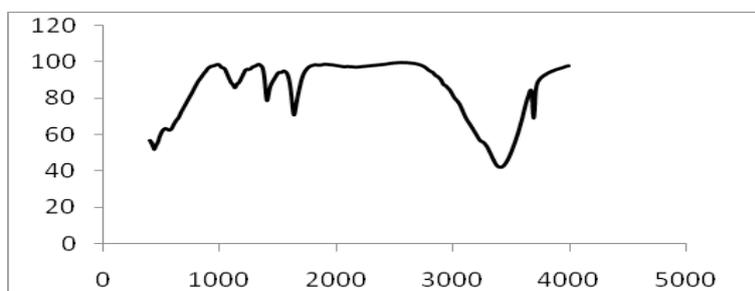


Fig. 2d. FTIR spectrum of  $\text{Mg}(\text{OH})_2$  modified With 0.25% PEG Peaks picking:  $3698 \text{ cm}^{-1}$ ,  $3413 \text{ cm}^{-1}$ ,  $2170 \text{ cm}^{-1}$ ,  $1638 \text{ cm}^{-1}$ ,  $1406 \text{ cm}^{-1}$ ,  $1130 \text{ cm}^{-1}$ ,  $435 \text{ cm}^{-1}$

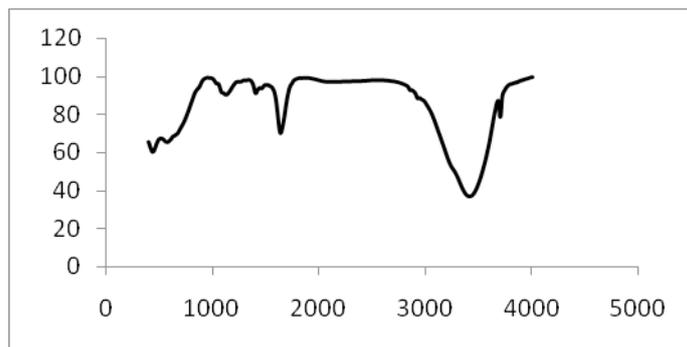


Fig. 3a. FTIR spectrum of Mg(OH)<sub>2</sub> modified with 10% EG Peaks picking: 3408 cm<sup>-1</sup>, 1637 cm<sup>-1</sup>, 1404 cm<sup>-1</sup>, 1128 cm<sup>-1</sup>, 437 cm<sup>-1</sup>.

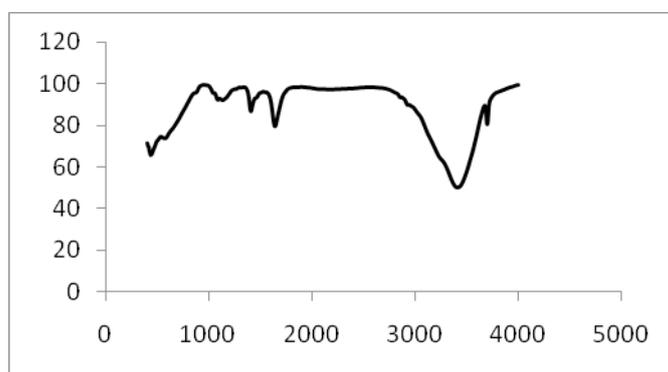


Fig. 3b. FTIR spectrum of Mg(OH)<sub>2</sub> modified with 5% EG Peaks picking: 3409 cm<sup>-1</sup>, 2166 cm<sup>-1</sup>, 1405 cm<sup>-1</sup>, 1131 cm<sup>-1</sup>, 432 cm<sup>-1</sup>.

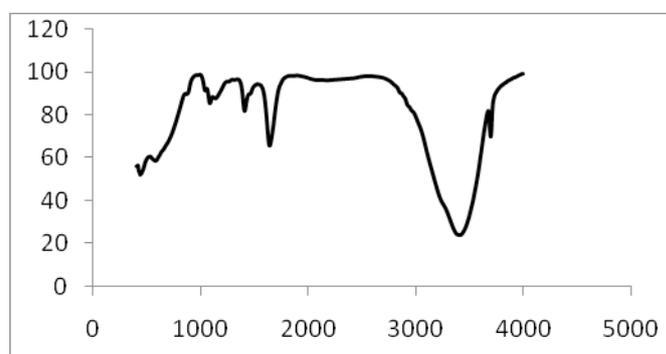


Fig. 3c. FTIR spectrum of Mg(OH)<sub>2</sub> modified with 2.5% EG Peaks picking: 3407 cm<sup>-1</sup>, 2171 cm<sup>-1</sup>, 1083 cm<sup>-1</sup>, 433 cm<sup>-1</sup>.

corresponds to -OH of water.

The broad peaks at 3408 cm<sup>-1</sup>, 3409 cm<sup>-1</sup>, and 3407 cm<sup>-1</sup> corresponds to the adsorbed -OH of EG groups. The broad peak at 1404 cm<sup>-1</sup> and 1405 cm<sup>-1</sup> is assigned to -CH<sub>2</sub> groups from EG (bending – scissoring vibrations). While bands at 1128 cm<sup>-1</sup>, 1131 cm<sup>-1</sup> and 1083 cm<sup>-1</sup> represents the asymmetric stretching vibrations of ether groups C-O-C EG. The broad maxima at 437

cm<sup>-1</sup>, 432 cm<sup>-1</sup> and 433 cm<sup>-1</sup>, 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)<sub>2</sub>, the bands are more modified, with respect to pure Mg(OH)<sub>2</sub> bands at 1413 cm<sup>-1</sup> mainly shifted to 1126 cm<sup>-1</sup>, 1130 cm<sup>-1</sup> and 1100 cm<sup>-1</sup> (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  $2065\text{ cm}^{-1}$ ,  $2022\text{ cm}^{-1}$ ,  $2163\text{ cm}^{-1}$  belonging the C-H stretching vibration in  $-CH$  group. The bands at  $3425\text{ cm}^{-1}$ ,  $3414\text{ cm}^{-1}$  Fig (2<sub>a,b</sub>) and  $3408\text{ cm}^{-1}$ ,  $3409\text{ cm}^{-1}$ ,  $3407\text{ cm}^{-1}$  Fig (3<sub>a,b,c</sub>) 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  $420^\circ\text{C}$  and  $428^\circ\text{C}$  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  $780^\circ\text{C}$  and  $500^\circ\text{C}$  respectively; of almost 25.74% and 20.70% respectively indicates the decomposition of magnesium hydroxide to magnesium oxide and more stability of  $Mg(OH)_2$  -

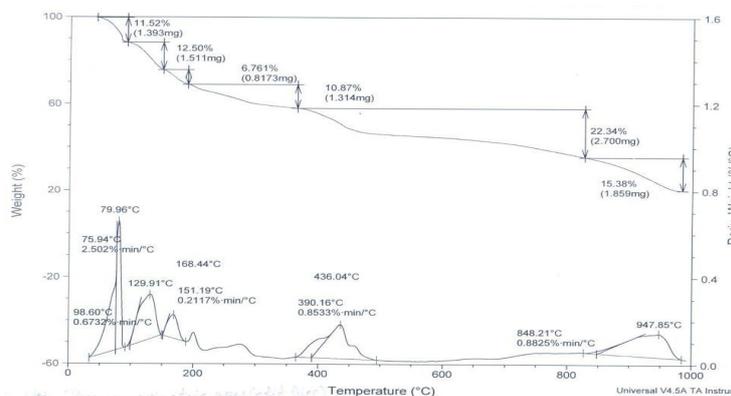


Fig. 4a. Thermogravimetric(upper solid line) and deferential scanning calorimetric (DSC) pilot peaks of the  $Mg(OH)_2$  powder with 1% PEG600.

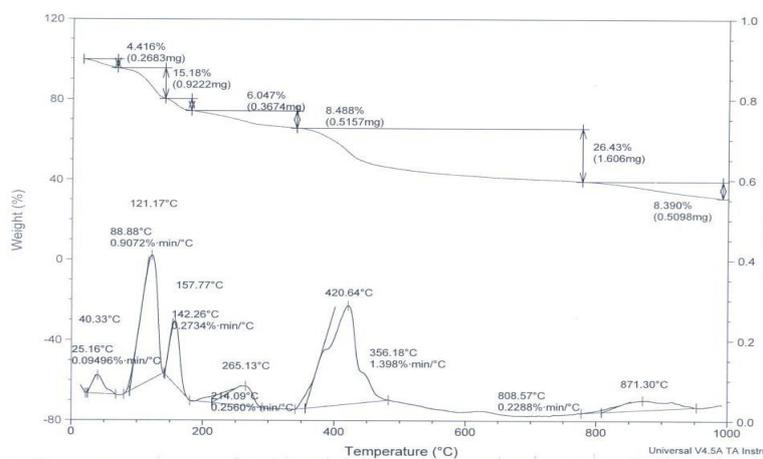
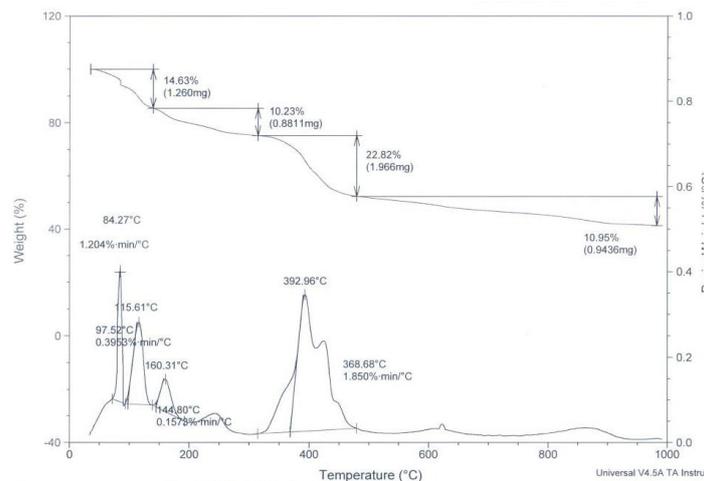
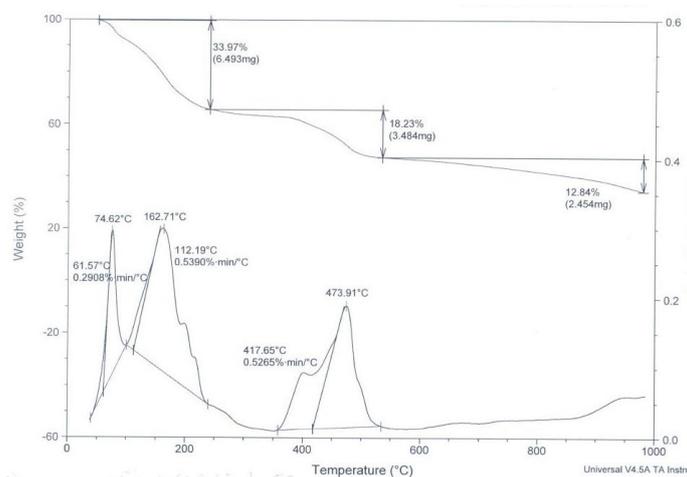


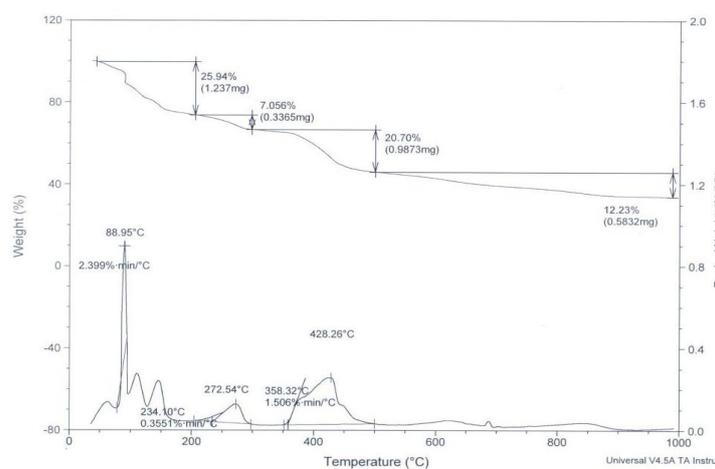
Fig. 4b. Fig (4)b Thermogravimetric(upper solid line) and deferential scanning calorimetric (DSC) pilot peaks of the  $Mg(OH)_2$  powder with 0.5% PEG600.



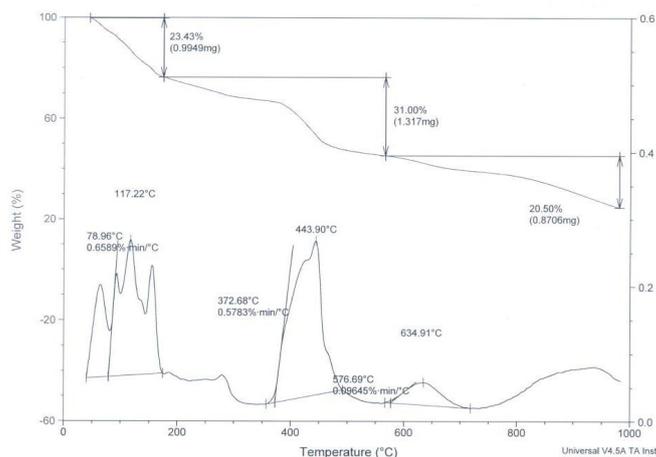
**Fig. 4c.** Thermogravimetric (upper solid line) and differential scanning calorimetric (DSC) pilot peaks of the  $\text{Mg}(\text{OH})_2$  powder with 0.25% PEG600.



**Fig. 5a.** Thermogravimetric (upper solid line) and differential scanning calorimetric (DSC) pilot peaks of the  $\text{Mg}(\text{OH})_2$  powder with 10% EG.



**Fig. 5b.** Thermogravimetric (upper solid line) and differential scanning calorimetric (DSC) pilot peaks of the  $\text{Mg}(\text{OH})_2$  powder with 5% EG.



**Fig. 5c.** Thermogravimetric(upper solid line) and deferential scanning calorimetric (DSC) pilot peaks of the  $\text{Mg}(\text{OH})_2$  powder with 2.5% EG.

**TABLE 1.** Shows the effect of PEG addition on weight loss of  $\text{Mg}(\text{OH})_2$  prepared:

Ratio of PEG	0%	1%	0.5%	0.25%
Weight loss%	10%	11%	4.4%	14.6%
OH type	2.359 mg	2.700 mg	0.515 mg	0.8811 mg
Endothermic transition at °C	432°C	436°C	420°C	392°C
Endothermic transition at weight loss %	40%	49%	33%	46%

**TABLE 2.** shows the effect of EG addition on weight loss of  $\text{Mg}(\text{OH})_2$  prepared:

Ratio of EG	0%	10%	5%	2.5%
Weight loss%	10%	33%	4.4%	14.6%
OH type	2.359 mg	3.484 mg	0.3365 mg	1.317 mg
Endothermic transition at °C	432°C	473°C	428°C	443°C
Endothermic transition at weight loss %	40%	54%	52%	54%

Morphology analysis of  $\text{Mg}(\text{OH})_2$ :

PEG than  $\text{Mg}(\text{OH})_2$ –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  $\text{Mg}(\text{OH})_2$  prepared with addition of PEG600 and EG are shown in Fig (6)<sub>a</sub>, Fig (6)<sub>b</sub>, Fig(6)<sub>c</sub> and Fig(7) shows MgO. Fig(6)<sub>a</sub> illustrates image of magnesium hydroxide without modifier addition. Fig (6)<sub>b</sub> and Fig (6)<sub>c</sub> illustrates the images of nano-crystalline structure of  $\text{Mg}(\text{OH})_2$ . While Fig(6)<sub>b</sub> shows image of smaller crystallite size due

to PEG addition during precipitation. Fig(6)<sub>c</sub> shows plate crystallite structure. It is believed that PEG600 facilitates homogeneity and dispersion of particles during synthesis with a significant effect on  $\text{Mg}(\text{OH})_2$  nano-crystals.

SEM images of pure  $\text{Mg}(\text{OH})_2$  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.

*Egypt. J. Chem.* **62**, Special Issue (Part 1) (2019)

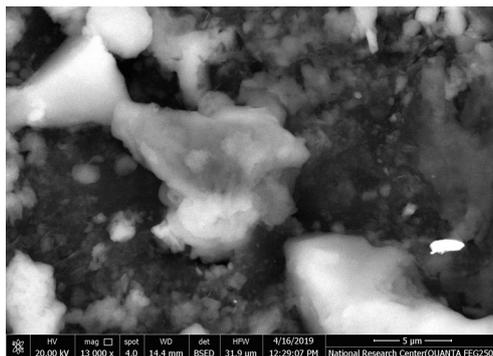


Fig. 6a. SEM image of  $\text{Mg}(\text{OH})_2$  powder precipitated without modifier

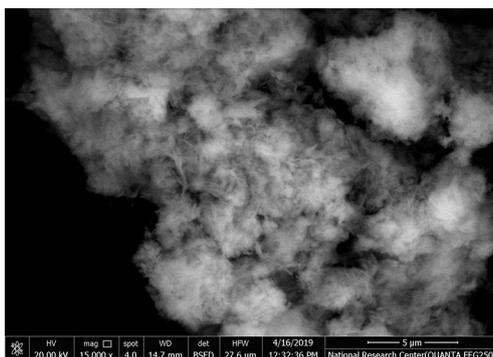


Fig. 6b. SEM image of  $\text{Mg}(\text{OH})_2$  powder precipitated with PEG600 0.5% modifier

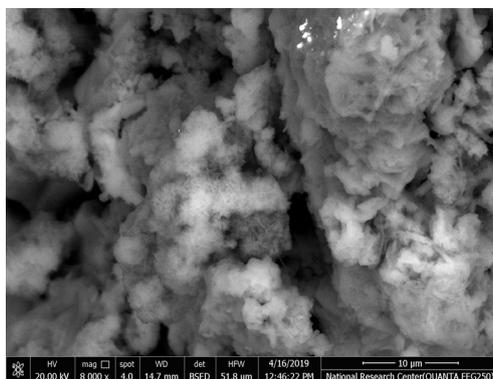


Fig. 6c. SEM image of  $\text{Mg}(\text{OH})_2$  powder precipitated with EG 5% modifier.

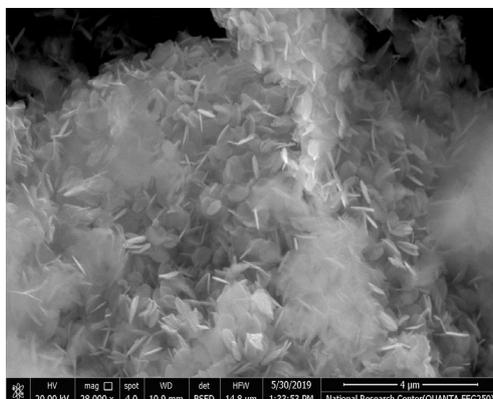


Fig. 7. SEM image of pure calcined  $\text{Mg}(\text{OH})_2$  powder precipitated with PEG 0.5% modifier

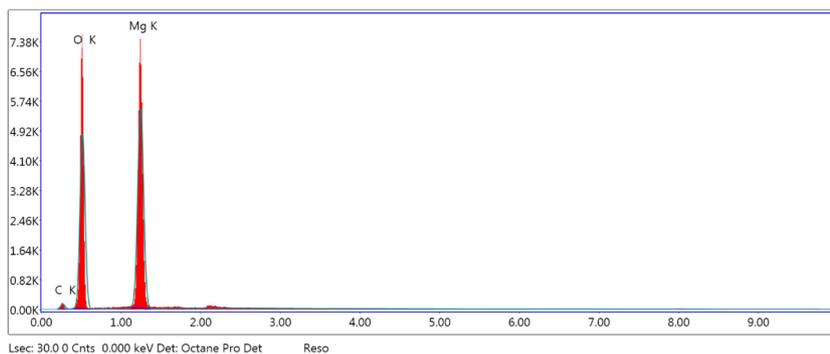


Fig. 8. EDX Spectra of pure  $\text{Mg}(\text{OH})_2$  modified with 0.5% PEG 600 showing low carbon content.

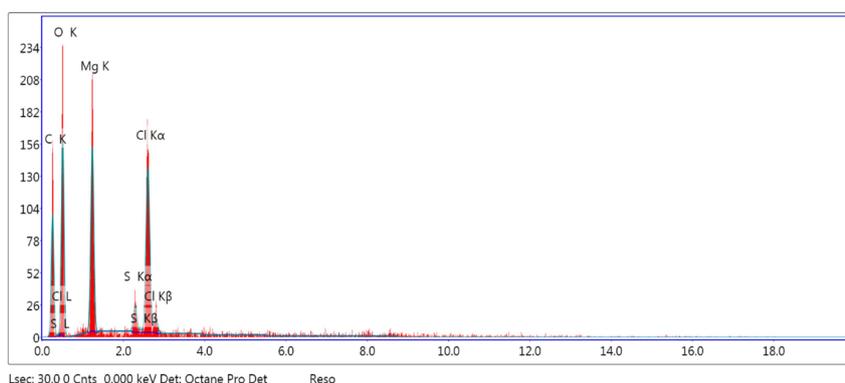


Fig. 9. EDX Spectra of pure  $\text{Mg}(\text{OH})_2$  modified with 10% EG showing higher carbon content

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

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

Table (4) shows modified  $\text{Mg}(\text{OH})_2$  – 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  $\text{Mg}(\text{OH})_2$  – PEG.

From Table (3) and Table(4) the elemental analysis(EDX) shows in nano  $\text{Mg}(\text{OH})_2$  –PEG increase in oxygen 62.12 % more than in nano  $\text{Mg}(\text{OH})_2$ – EG 37.5% , this is due to the modified surface porosity of nano  $\text{Mg}(\text{OH})_2$  –PEG is more than that of nano  $\text{Mg}(\text{OH})_2$  – 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)  $\text{Mg}(\text{OH})_2$ , (ICSD card No. 01-086-0441) as the major phase with **trace** of minor phase represented by ammonium magnesium chloride hydrate  $\text{NH}_4\text{MgCl}_3 \cdot 6\text{H}_2\text{O}$  (orthorhombic) (ICSD Card No. 00-0025-0039). The occurrence of ammonium magnesium chloride hydrate  $\text{NH}_4\text{MgCl}_3 \cdot 6\text{H}_2\text{O}$  (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.(10<sub>D</sub>).

The particle size values, peak position ( $2\theta$ ), full width at half maximum  $\beta(2\theta)$ , Miller indices (hkl),  $\cos(2\theta)$  were calculated and were listed in Table (3). The particle size was calculated for all samples using the **Scherrer** formula. The results were presented in Table (3). From the results

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  $\pm 10\%$ ).

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  $500^\circ C$  in air. The crystallite of  $Mg(OH)_2$  powders are characterized by intense peaks locations in  $2\theta^\circ$  shown in Table (3) and Table (4). The crystallite size of samples were calculated using Scherer's

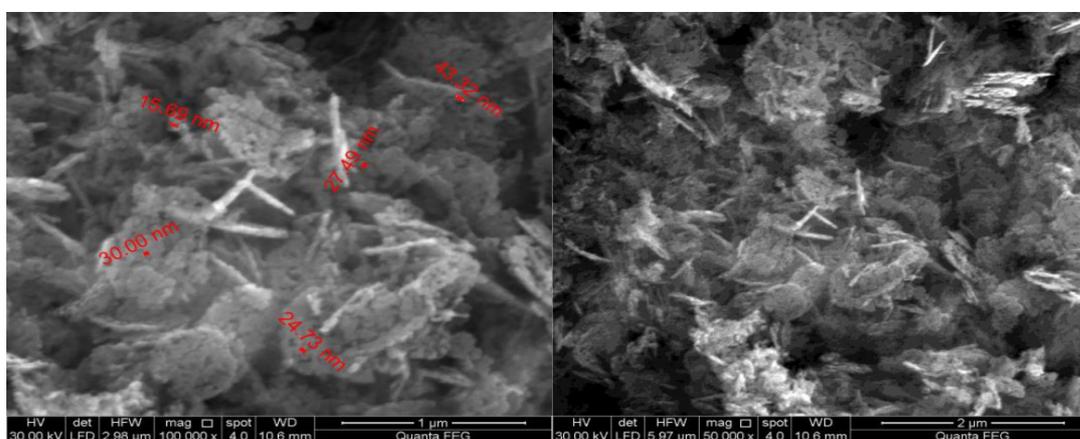


Fig. 10. SEM image of pure calcined 8h MgO powder precipitated with PEG 0.5% modifier.

TABLE 3. Showing % weight element content present in modified pure  $Mg(OH)_2$

Element	Weight %	Atomic %	Net Int.	Error %
C K	8.05	11.85	35.66	12.82
O K	56.18	62.12	1259.2	6.87
MgK	35.77	26.03	1658.16	5.78

TABLE 4. Showing % weight element content present in  $Mg(OH)_2$  modified with EG 10%

Element	Weight %	Atomic %	Net Int.	Error %
C K	42.72	53.44	23.81	13.01
O K	40.01	37.57	40.91	12.65
MgK	8.36	5.16	44.12	8.85
S K	1.04	0.49	7.07	17.97
ClK	7.88	3.34	47.98	5.39

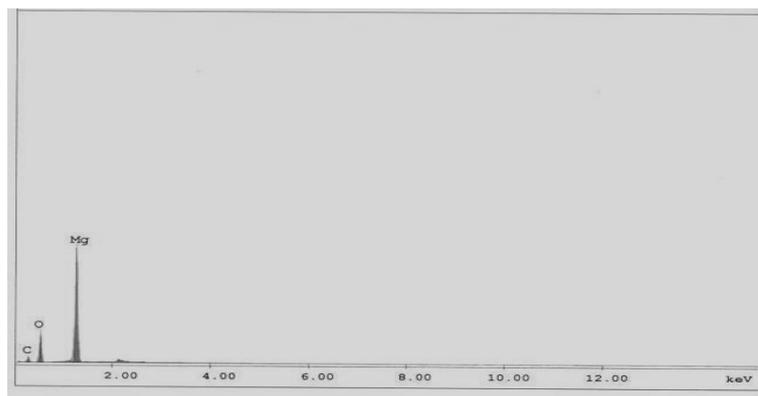


Fig. 11. EDX Spectra of pure MgO modified with 0.5% PEG showing low carbon content

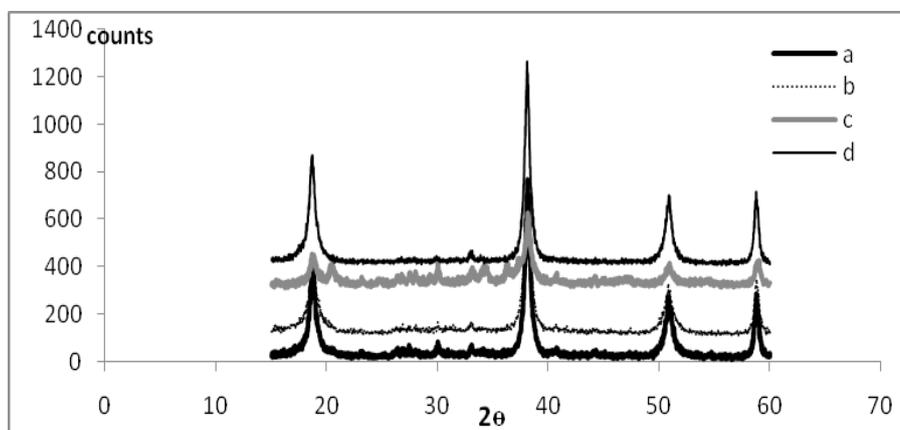


Fig. (12A,B,C,D) XRD pattern of Sample A, B, C, and D for Mg(OH)<sub>2</sub> with 1% PEG, 0.5% PEG, 10% EG, and 5% EG modifiers respectively.

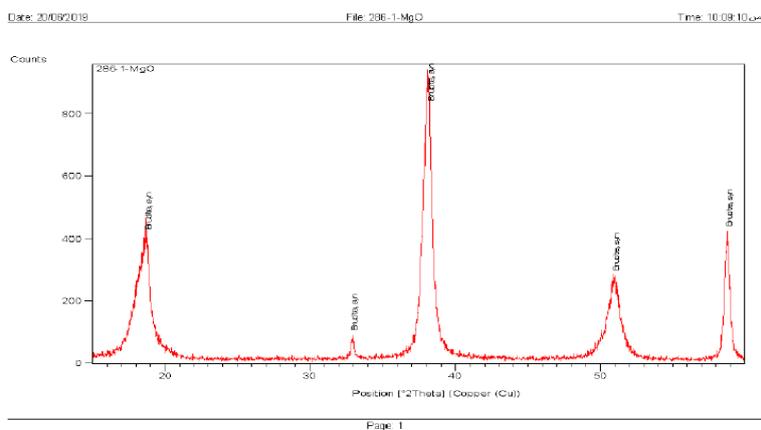


Fig. 13. XRD pattern of Pure calcined Mg(OH)<sub>2</sub> with 0.5% PEG modifier

formula [14, 15]. Results are shown in Fig. (12<sub>A</sub>), Fig(12<sub>B</sub>), Fig(12<sub>C</sub>), Fig(12<sub>D</sub>), for modified product by PEG and EG respectively. Fig(13) and Fig(14) shows the XRD pattern for pure calcined Mg(OH)<sub>2</sub> and standard nano Mg(OH)<sub>2</sub>.

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.

## Conclusions

The modification of Nano-magnesium hydroxide with addition of PEG600 or EG resulted increase in specific area of Mg(OH)<sub>2</sub> powder samples. The modifiers brought beneficial changes in the character of Mg(OH)<sub>2</sub> 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)<sub>2</sub>-PEG 62.12% and for Mg(OH)<sub>2</sub>-EG 37.5% indicating more

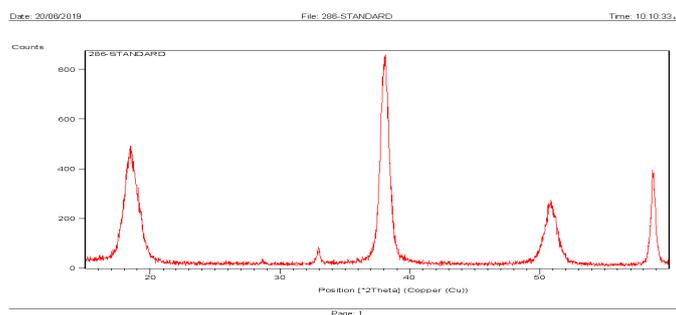


Fig. 14. XRD pattern of Standard Nano Mg(OH)<sub>2</sub>

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

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

hydrogen bonding with nano  $\text{Mg}(\text{OH})_2$ -PEG than for nano  $\text{Mg}(\text{OH})_2$ -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  $\text{Mg}(\text{OH})_2$  Nano-particles can be synthesized in pure state from liquid bittern, this was confirmed with XRD analysis with presence of single crystal peak formed.  $\text{Mg}(\text{OH})_2$  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  $\text{Mg}(\text{OH})_2$ -PEG at 780 °C than nano  $\text{Mg}(\text{OH})_2$ -EG at 500°C 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**

The authors would like to thank all colleagues for helpful assistance with XRD, FTIR, SEM-EDX, TGA-DSC studies. This work was supported and funded by National Research Centre of Egypt. From to pilot scale.

#### **References**

1. Tanısalı E., Cenikli C., Orman R., Kılıç Y., Güldem K. Ş., Timur S. I. An Alternative Process Design For Production of  $\text{Mg}(\text{OH})_2$  From Waste Dolomite. Istanbul Technical University, Faculty of Chemical and Metallurgical Engineering, Department of Metallurgical and Materials Engineering, İstanbul, Turkey *Uluslararası Metalurji ve Malzeme Kongresi | IMMC* (2018).
2. Pilanska A., Paukszta D., Ciesialezyk F., Jesionowski T., Physico-chemical and dispersive characterization of magnesium oxides precipitated from the  $\text{Mg}(\text{NO}_3)_2$  and  $\text{MgSO}_4$  solutions. *Pol. S. Chem. Technol*, **12**, 252-256 (2010).
3. Echeverry-R.M., Duquea V., Quinteroa D., Harmsenb M. C. Echeverriaa F. Considerations about sterilization of samples of pure magnesium modified by plasma electrolytic oxidation. *Surface & Coatings Technology* **363**, 106-111 (2019).
4. Song X., SWS., Zhang D., Wang J., Synthesis and characterization of magnesium hydroxide by batch reaction crystallization *Front. Chem. Sci. Eng.*, **5**, 416 – 421 (2011).
5. Fernandez S. A., Villalba G.S.L, Muñoz L., Flores G., Fort1R. & Rabanal E.M. Effect of Temperature and Reaction Time on the Synthesis of Nano Crystalline Bruchit. *International Journal of Modern Manufacturing Technologies* ISSN 2067-3604, **VI**(1), (2014).
6. Mai, K , Qiu Y, Lin Z., Mechanical Properties of  $\text{Mg}(\text{OH})_2$ /Polypropylene Composites Modified by Functionalized Polypropylene. <http://www.paper.edu.cn>
7. Dong J, Li B, Bao Q. In situ reactive zone with modified  $\text{Mg}(\text{OH})_2$  for remediation of heavy metal polluted groundwater: Immobilization and interaction of Cr(III), Pb(II) and Cd(II). Copyright © 2017 Elsevier B.V. All rights reserved.
8. Tang X.Z. and Feng L. B., MgO Nanoparticles as antibacterial agent: Preparation and activity. *Brazilian Journal of Chemical Engineering*, **31**(03), 591-601, July-September, (2014). dx.doi.org/10.1590/0104-6632.20140313s00002813.
9. Alfaro A., Leon A., Guajardo E., Reuquen P., Torres F., Mery M., Segura R., Zapata P.A., Orihuela A. P. MgO nanoparticles coated with Polyethylene Glycol as carrier for 2-Methoxyestradiol anticancer drug. bioRxiv preprint first posted online Mar. 25, (2019); doi: <http://dx.doi.org/10.1101/588939>.
10. Samadi S. N., Khairul M., Mustajab A.A, and Abdul Rahim Yacob A.R.. Activation Temperature Effect on the Basic Strength of Prepared Aerogel MgO (AP-MgO). *International Journal of Basic & Applied Sciences IJBAS-IJENS* Vol:10 No:02. 106702-5454 IJBAS-IJENS © April 2010 IJENS.
11. Yana S.S.K., Yua T., Wanga K., Fengb C., Xub L., Xiea G., Highly enhanced photoelectrochemical cathodic protection performance of the preparation of magnesium oxides modified TiO<sub>2</sub> nanotube arrays. *Journal of Electroanalytical Chemistry* **834**, 138-144 (2019).
12. Hiromoto S, Yamamoto A. Control of degradation rate of bioabsorbable magnesium by anodization and steam treatment. *Mater. Sci. Eng. C [Internet]*. Elsevier B.V.; 2010 [cited 2015Feb21];30:1085-93. Available from: <http://linkinghub.elsevier.com/retrieve/pii/S0928493110001360>.
13. Wang P., LIC., Gong H., Liu J., Morphology *Egypt. J. Chem.* **62**, Special Issue (Part 1) (2019)

- control and growth mechanism of magnesium hydroxide nanoparticles via a simple wet precipitation method, *Ceram.Int.*, **37**, 206-2066 (2011).
14. Brahama S., Choudhary R.N.P., Thakur A.K., *Physica B* **355**, 188-201 (2005).
  15. Mbarki R., Maidhi I., M'nif A., Hamzaoui A.H., Poly ethylene glycol ratio effect on magnesium oxide prepared by chemical precipitation; Impact on structure, morphology and electrical properties. *Material Science in Semiconductor Processing* **39**, 119 – 131 (2015).
  16. Pilarska A., Linda I., Wszakowski M., Paukszta D., Iesinowski T., Synthesis of Mg(OH)<sub>2</sub> from magnesium salts and NH<sub>4</sub>OH by direct functionalization with poly(ethylene glycols) physicochem. *Probl. Minert. Process*, **48**, 631-643 (2012).