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EFFECT OF PH PARAMETER ON THE STRUCTURAL PROPERTIES OF THE COMPOUND MG(OH)2 AND ZNO-MG(OH)2 NANO/MICRO PARTICLES PREPARED BY A HYDROTHERMAL ROUTE AND ITS SUITABILITY FOR USE AS AN FIRE RESISTANCE

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Abstract:

The micro- and nanostructures of the product (Magnesium hydroxide -Mg(OH)2), and a composite of ZnO-Mg(OH) as a comparison have been synthesized by hydrothermal method. The structural investigations by XRd results confirmed the formation of hexagonal crystal structure of the products Mg(OH)2 with variable crystalline sizes according to PH Values of Solution. Surface morphology by SEM results are consist of a microstructure Mg(OH)2 and also nanostructure with assortments of shapes as well sizes with transformation of PH values with an aggregations, and a lowest aggregations with ZnO-Mg(OH)2 composites as comparison. FTIR results confirmed that synthesized products are crystallized and the peaks are the Mg-OH and O-H band. UV results found that the energy gap are changed by changing the particle size of product and increasing as it is decreased and adding ZnO to the hydrothermal synthesis.

Keywords: Hydrothermal method, magnesium hydroxide, PH parameter, Brucite, ZnO-MgO composite.

Introduction

The brucite structure are Mg (OH)2 which is a normal material that have a abinding structural of Magnesium and hydroxide , which a framework in a hexagonal mode. Magnesium hydroxide crystallizes in a layering construction and have a correspondingly structure as is the case in graphite or carbon mineral [1,2].

The two compounds magnesium oxide and magnesium hydroxide were studied from all aspects . Magnesium and mix up keeps an alone features , such as low weight , non poisonous [3,4].The

brucite or Magnesium hydroxide in generally are employed for flame delayng stuffing in composites materials as aconsequence of it is efficiency to afford the aheat absorption dehydration conditions in fire enviroments , acidic waste weaken in environmental protection, paper industries , fertilizer additive, recycling of elements from industrial wastewater areas, adsorption of carbon dioxide gas, Antibacterial agent, a neutralizing agent in pharmaceuticals, chemical sensors and as important precursor for magnesium oxide production [5-6,7].

Mg(OH)2 - nanocrystals can be synthesized in several forms , which are needle shape , lamellar morphology and rod-like shape. Generally its found that in the crystallization process Mg(OH)2 tends growing with the direction of (101) in acrystalline plane . So the crystals can shows more surface effectiveness and a high stress [8,9].Newly, large studies and research are concentrate to synthesizing of Mg(OH)2 nanostructures. These nanostructure cane be produced through several routes , such a solution gelatin (Sol-gel route), electrodeposition, microwave assisted synthesis, sonochemical methods, precipitation, hydrothermal, and solyothermal method [10, 11].

Different synthesizing techniques are results a different sizes, morphology, aggregations, surface charges of Mg(OH)2, then leading to promotes of optical, catalytic agent, electronic, and mechanical (such as strain) properties as decreasing the sizes of particles into nanosize scale which leads to increasing of surface area [12]. In this research , we are study to synthesizing of Mg(OH)2 compound and MgO-ZnO composite by hydrothermal process and discussing the structural properties of them.

II. Experimental Work

Hydrothermal technique is an easy route to synthesisMg(OH)2compound nano/microstructures.The magnesium chloride - hexahydrate (mgcl26H2O), zinc sulphate-heptahydrate (ZnSO4.7H2O), and (NaOH), (distilled water) were used ingenerally as take up from local sources. All chemicals in this research were of analytical ranking and were employed without any extra purification. In the first stage, Mg(OH)2 nano/micro particles were synthesized, different concentrations of it with (NaOH) have been used to prepare the solution as the ratio of molarities (MgCl2.6H2O/ NaOH) were (0.4/0.1 , 0.1/0.4 , 0.1/0.2). The second stage involved equal molarities of (1M MgCl2.6H2O , 1M ZnSO4.5H2O) to prepare a composite material with (0.2M NaOH). These concentrations are dissolved in (80ml) under constant motion in (a 100ml - glass beaker) and NaOH then added by dropwise, then solution was move out to teflon lined container inside another stainless steel container and maintained on 140 °C for 8 hours. After that, acontainer was left to cool down to room temperature. The resulting solution was filtered,then washed several times by ethanol and distilled water to eliminate the impurities and ions inform product, then dried in 60 °C for 20 minutes. Characterization of nano/micro particles was done by X-ray technique with the (USA-panalytical Xpert pro) using CuKα as radiation source having a wave length of 1.54060 Å and the FT-IR analysis was studied by (470 infrared-spectrophotometer shimadsu). To discuss and

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comprehension of particles morphology then a scanning electron microscope (EBSD-ZEISS SIGMA VP Germany) was used.Optical Properties are studied by UV-Spectroscopy.

III. The discussion of results

A. X-ray diffraction

The producting powder from hydrothermal Process has a main features. The variation in the concentrations of reaction materials that used will greatly affect in the structural installation and form Properties of Mg(OH)2 product. XRD patterns for Mg(OH)2 micro- /nano particles synthesized at 140 °C for 8h (sample 1,2,3) are illustrated in Figure1 (normalized intensity). All of the diffraction peaks are in evidence to the hexagonal structure of magnesium hydroxide (JCPDS 7-239) that have constants of lattice (a = b = 3.148 Å , c = 4.787Å)[13,14]. No peaks are observed for second phases. It indicating a high purity of the products.

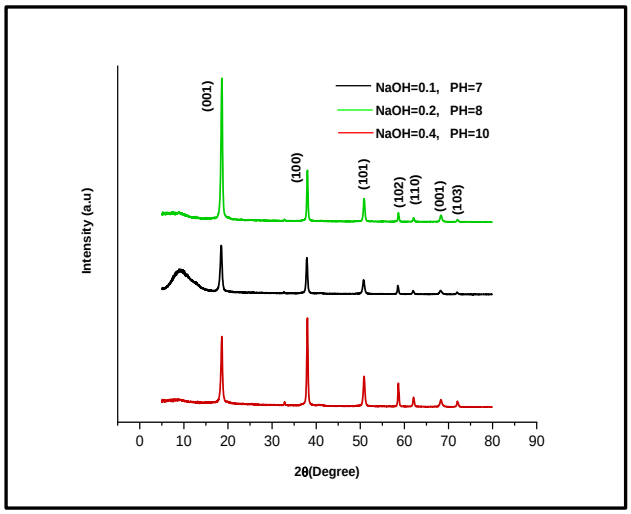

Figure 1: XRD of Mg (OH)2 with PH values.

From XRD patterns, its obvious that (0 0 1) for the sample 1 (NaOH = 0.2M) is much larger than (1 0 1), but according to the standard measurements , a peak that (1 0 1) should be the highest one instead (0 0 1) peak as in our results, which mentioned that crystal growth should be closed over the (0 0 1) direction, agree with [15]. By increasing of (NaOH = 0.4M) for the sample2, the peak intensity of (0 0 1) is smaller than the (1 0 1). On other hand, when the ratio of sodium hydroxide decreased (NaOH = 0.1M) sample3, it shows analogous intensities for the (0 0 1) and (1 0 1) peaks, as well as, the diffraction peaks intensity are a low as compared to the other samples, which indicated the reduced crystallinity which may be due a porous nature of the products material and PH value. The broadening of peaks increased slightly with sample 3 (NaOH = 0.1) compare to others, that may be indicate to the small particle size. A crystallite size was found by employing (The Debye-Sherrer equation , $D = K\lambda/\beta \cos \theta$) based on the (FWHM) full width of peak at half maximum.Where K is a constant that called shape factor which is nearly equals to unity and relates to the crystalline shape , β represents width or domain of the spotted diffraction peak at the half of highest intensity, and

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λ reprecents wavelength of the XRD device source [13,16] the estimated crystallite sizes of the samples 1,2,3 corresponding to the (0 0 1) plane are (18.60, 37.97 and 18.46 nm) respectively. XRD of a composite ZnO-Mg(OH)2 comparing to Mg(OH)2 are mentioned in Figure 2

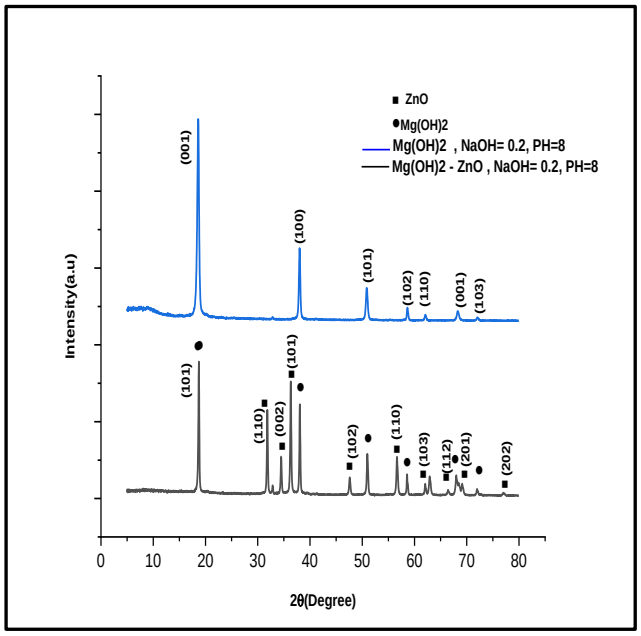

Figure 2: XRD of a products Mg (OH)2, and ZnO- Mg(OH)2 with PH = 8

All of the structures, phase conformity, and purity of nanocomposites where confirmed by XRD. As seen in figure3, a very intense peaks were clarified in XRD of Mg(OH)2/ZnO nanocomposite and denote to a dwony quality that gained by a hydrothermal teqnique. The diffraction peaks in figure agree with standardized model of the hexagonal structure for pure ZnO (JCPDS- No: 01-079-0207) [17], hexagonal brucite structure of Mg(OH)2 [13,18]. The result of XRD for samples (given in figure 3) confirmed that crystallity of products. A fine particles sizes is found in X-ray line broadening. The particle size of the composites with peak (1 0 1) found that the size of particles are 18.72nm.

B.SEM analysis

The SEM device was used to realize the morphological nature of samples and the observed distribution of micro or nanoparticles. The surface nature of the products Mg(OH)2 micro-/nanostructures is conscribed from SEM analysis . The Mg(OH)2 products that have different sizes and shapes at variable concentrations are listed in table (1)

The Mg(OH)2 particles with samples (S1, S2, S3) synthesized at the conditions, shows a vastly size ranking and distribution which are alternate from nanodisks to microdiskswith ahexagonal view, which range in (218 nm , 5μm in width and 20 – 70 nm in thickness) with more aggregations, Figure3 show it. The morphology of products (S4) (ZnO/Mg(OH)2) are vary

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from 100 nm – 600 nm in width and 50-55nm in thick, this condition that govern aggregates, this agglomeration phenomenon may caused by high surface energy of the small particles size [7]. The influence of inducing parameter (NaOH) on the crystallite size of Mg(OH)2 could illustrated by nucleation mechanism and growth [12]. The particles with these hydrothermal conditions have a high mixing quantity of micro and nanostructures with perfect particle size distribution. Also its obvious that the width of disks decreased in the composite products and no aggregations are seen.

Table 1: parameters of hydrothermal method with XRD and SEM results

Sample	Concentration(Molarity)		PH	Time(h)	T(℃)	Crystallite size (nm)	Morphology from SEM
1	$mgcl_26H_2O$/NaOH	0.1/ 0.2	8	8h	140 ℃	18.60	Disk shape
2	$mgcl_26H_2O$/NaOH	0.1/ 0.4	10	8h	140 ℃	37.97	Disk shape
3	$mgcl_26H_2O$/NaOH	0.4/ 0.1	6.4	8h	140 ℃	18.46	Disk shape
4	$mgcl_26H_2O$ – $ZnSo_47H_2O$ /NaOH	0.1/0.2	8	8h	140 ℃	18.72	Disk shape

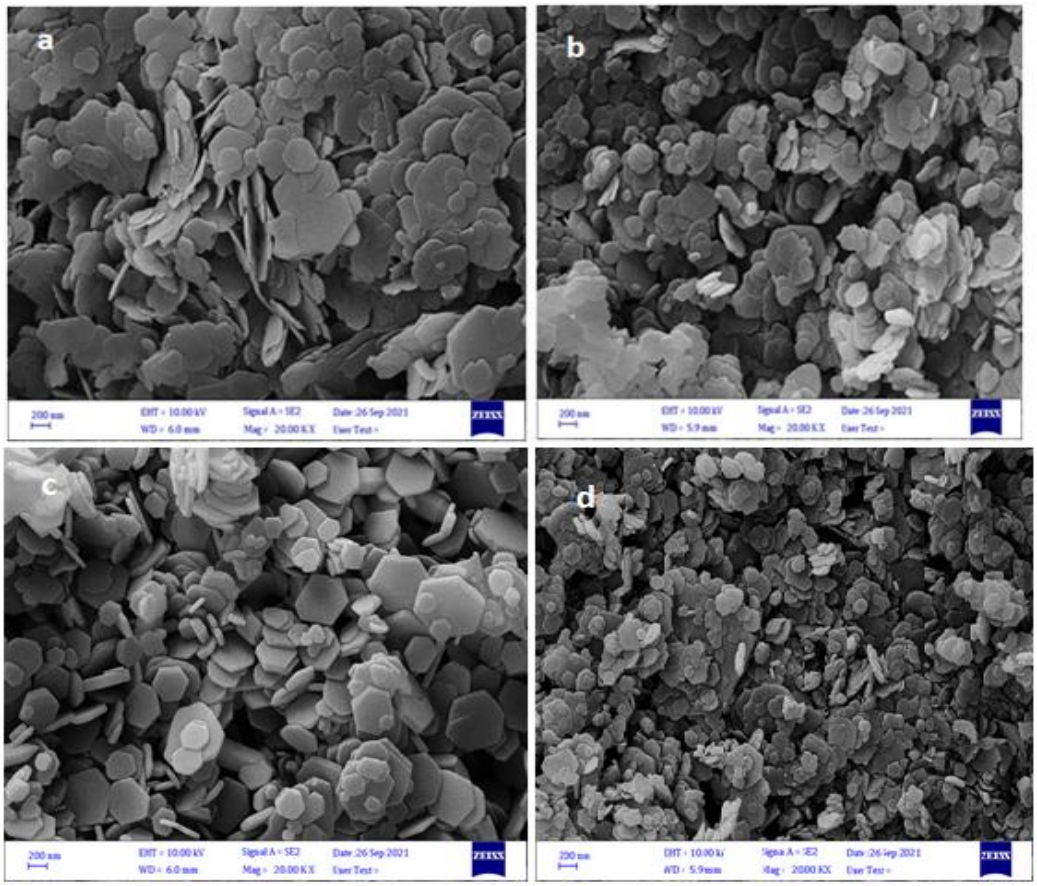

Figure 3: SEM images of (Mg(OH)2 and ZnO-Mg(OH)2 with different hydrothermal reaction concentrations (different PH values): a- Mg(OH)2 with PH = 8, b- Mg(OH)2 with PH = 10, c- Mg(OH)2 with PH=7, d- ZnO-Mg(OH)2 with PH=8.

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C.FTIR analysis

As indicated in Figure 4, the intense peak at 428 cm- 1 refers to elongate vibration of Mg – OH. Aslo, both of peaks 1417 and 1653 cm- 1 are indicate to (hydroxyl-elongating) in water. The strongest and
intense peaks located at 3697 cm- 1 and 3646 cm- l is assigned to the elongating vibration of O – H bond in the brucite crystal structure, these results are in agreement with [19,20]

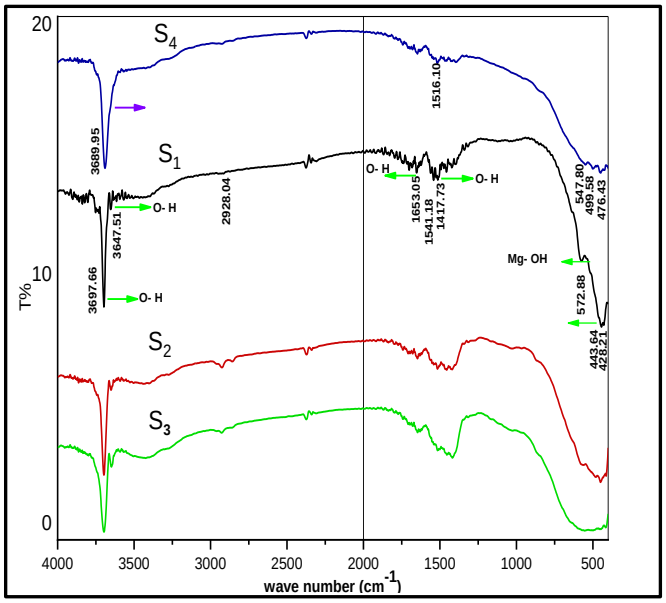


Figure 4: FTIR of Mg(OH)2 with concentration of reactants (different PH values), and ZnO-Mg(OH)2 materials.

D.UV – Visible Spectra

The UV – Visible spectrum of Mg(OH)2 and a composite of Mg(OH)2-ZnO at room temperature illustrated in Figuer 5a, and 5b respectively. Due to the nanostructure of Mg(OH)2 and an observable particle size distribution of the sample (S1), the optical features of structures were studied in detail, which is in agreement with previous paper [13]. The absorption peak of wavelength in the UV region of Mg(OH)2 and ZnO-Mg(OH)2 composite are 229nm and 237nm respectively. To find the band gap of the
particles, the direct equation is used: $Eg = hc/\lambda$
The energy gab of Mg(OH)2 were found to be 5.42 ev, and decreased go 5.24ev in a composite Mg(OH)2-ZnO. The wide energy band gap of Mg(OH)2, is concequent to the vacancies attributed by oxygen that are formed in the structure [21,22], which produce an energy levels in the band gap domain of magnesium hydroxide nanoparticles which lead to rise a band gap value comparing to the bulk Mg(OH)2. As soon as, the crystallite size of Mg(OH)2 particles are decreased in a comparing to Mg(OH)2-ZnO, so that, the absorption peak are shifted to the lower wave length region as called a blue shift, and this lead to increased of band gap, these changed are observed in previous studies [7,13], it depends on the other factors, PH values,

composite materials in solution, short range of the repulsion energy between atoms. These results provide an useful information for the optical applications. The absorption peaks in the range of (λmax = 229, and 237nm) respectively are relates to optical transitions in a band gap range, [12]. So that, the band gap of Mg(OH)2 can be controlled with adding of other materials.
Figure – 5 UV - spectrum of : a- Mg(OH)2, b- Mg(OH)2-ZnO composite

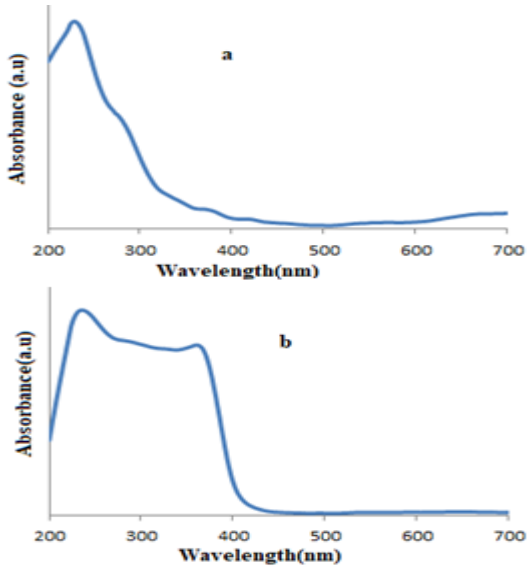
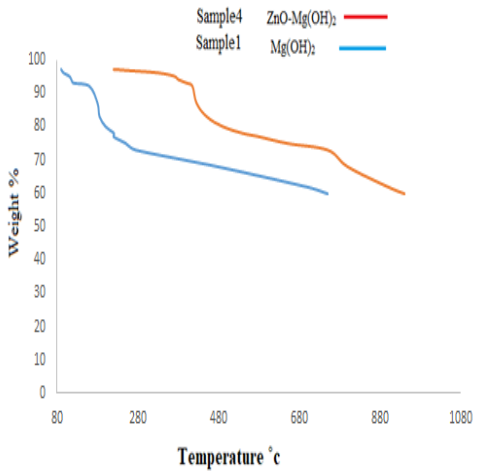


Figure5 :UV - spectrum of : a- Mg(OH)2, b- Mg(OH)2-ZnO composite

F. Thermal Analysis

The thermal analysis was performed by using (USA tool with the information of Perkin/elmer thermogravimeter. Samples of approximately 15mg were heated from (30- 900 ˚C) (Heating rate per time 10/m under nitrogen atmosphere with flow rate of 20ml/minutes
The thermal gravimetric profiles show that the samples of (Mg(OH2), ZnO/mg(OH2) behaves in the different trends as shown in Figure6.



Figuer 6:TGA of prepared materials

It is also due to the nature of the particle shape (hexagonal disc with a porous nature of Mg(OH2). Unlike the composed and containing zinc oxide, which reduces the thermal loss due to its high thermal stability and up to (900˚C).

However, there are three stages of the thermal decomposition of (mg(OH2). The first one starts at the room temperature ending at 92°C; this stage may be attributed to the surface and the hydrated water, the second step starts at 92 °C and ends at 200°C corresponding to the decomposition of Mg(OH)2 to form MgO, the third step represents the firing of MgO, converting it to a glassy state and continuing until 750 °C, and for, also the three steps for ZnO/Mg(OH)2 are (210-420°C), and (420-750°C), (750-940°C) respectively. The different between starting and ending temperatures may be due to the amount of the adsorbent water on the surface of the prepared particles and the structure and the shape of the prepared particles.

We note that the weight loss of the compound(ZnO/mg(OH)2 resists up to a temperature of 210°C, comparing to room tempertature of the compound (Mg(OH2) alone, and it is also more thermally stable for the three regions. This is due to the zinc oxide that resists the process of penetration and decomposition of the total compound. From this, it can be said that the compound composed with ZnO nano/micro particles can be used as a fire-resistant material [21,23]

E. Conclusions

The nano/micro structure of Mg(OH)2 and composite ZnO/Mg(OH)2 are prepared using hydrothermal method, and then characterized. These nano/micro powder were studied by XRD, UV, SEM and FTIR. The effect of concentration of NaOH – PH values on structure properties (morphology and particle size) are studied. XRD results shows a hexagonal system phase of Mg(OH)2, and the particle size are changing with PH value. The morphology study by SEM shows a broad domain of size allocation from hexagonal nanodisks to micro disks, and there are more aggregations that are affected by increasing of PH values, and there are not aggregations in a composite of ZnO/Mg(OH)2 materials with same shape, UV results depicts that the energy gap of Mg(OH)2 are changed by changing the particle size of product, and adding ZnO to the hydrothermal synthesis. The compound composed with ZnO nano/micro particles can be used as a fire-resistant material more than of Mg(OH)2 alone.

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