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The effect of modifiers and precipitation conditions on physicochemical properties of $MgCO_3$ and its calcinates

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Abstract. The effects of modifiers and process conditions on synthesis of magnesium oxide by the carbonatization method and properties of the product are studied. Magnesium carbonate obtained from magnesium hydroxide and carbon dioxide was subjected to thermal decomposition at 800ºC. The reaction of precipitation was performed checking the influence of temperature, rate and mode of reagents introduction, type and concentration of the modifier. The modifiers were the non-ionic compounds from the group of poly(ethylene glycols). The magnesium oxides produced were characterised by determination of their dispersive-morphological properties, wettability profiles, specific surface area, total volume and mean size of pores. The products were also subjected to identification by the X-ray diffraction method and TG/DTA analysis. The results revealed a significant impact of the concentration and type of modifiers on the physicochemical properties of MgO samples obtained and permitted selection of the best products for particular applications.

keywords: MgO, carbonatization, modification, thermal decomposition, particle size distribution, surface morphology

1. Introduction

One of the most important magnesium compounds is magnesium carbonate occurring in nature in the form of magnesite (anhydrous carbonate $MgCO_3$). As a result of thermal decomposition, $MgCO_3$ gives magnesium oxide and carbon dioxide (Bandi, 1976). Magnesium carbonate is described as light, white, amorphous and odourless powder capable of odour absorption. The temperature of its decomposition is in the range 230–680ºC. It is practically insoluble in water and alcohols but easily dissolves in acids (Gennaro, 1985).

There are two pharmaceutical varieties of magnesium carbonate: light and heavy. The bulk density of the light one is 2–2.5 smaller than that of the heavy variety, and each variety is obtained by different methods. In general the light variety is obtained by mixing magnesium sulphate or chloride with sodium carbonate (Gennaro 1985, Botha 2001). Precipitation in a cold solution gives a very bulky powder called *magnesia alba levis* or a light species of magnesium carbonate of the formula $3MgCO_3{\cdot}Mg(OH)_2{\cdot}3H_2O$. Precipitation in an elevated temperature gives species known as *magnesia alba ponderosa* or heavy powder of the formula $3MgCO_3{\cdot}Mg(OH)_2{\cdot}4H_2O$. The light variety can also be obtained from dolomite ($MgCO_3{\cdot}CaCO_3$) subjected to calcination, suspension in water and then saturation with carbon dioxide under pressure. The filtrate is heated to a temperature at which magnesium bicarbonate is converted into magnesium carbonate. Yildrim (2010) reported a method of obtaining magnesium carbonate and then magnesium oxide by the leach-precipitation-pyrohydrolysis process.

In our work magnesium carbonate was synthesised by the carbonatization method with an addition of a modifier. The magnesium carbonate was subjected to calcination to get magnesium oxide. Thermal analysis was made to identify the thermal effects accompanying the sample decomposition and to estimate mass loss at certain temperatures. Similar studies have been performed earlier (Khan 2001, Morozow 2001, Vagvölgyi 2008). Morozow characterised the relation between the degree of the sample decomposition and changes in the specific surface area. Khan has studied thermal analysis of magnesium carbonate in the atmosphere of nitrogen, carbon dioxide and air versus the rate of heating, he tried to explain the origin of the exothermic peak.

Methods of magnesium carbonate synthesis have not been much discussed in literature. Much more attention has been devoted to the methods of synthesis of hydromagnesite (Hollingbery 2010; Hongchang 2011). In the method proposed by us an element of novelty is the use of PEG compounds as modifiers. The modifiers were introduced to help obtain the powdered products of the possibly smallest size particles and possibly hydrophobic character (Wang 2007, Dongmin 2009, Pang 2009, Meshkani 2010).

2. Experimental

Magnesium carbonate was obtained in the reaction between the suspension of magnesium hydroxide ($Mg(OH)_2$, POCh SA) and CO_2, with addition of a non-ionic compound from the group of poly(ethylene glycols) purchased from Sigma-Aldrich of different molecular masses as a modifier. Solutions of modifiers in different concentrations were prepared by dilution in ethanol. Precipitation was performed in a reactor of 500 cm^3 in capacity, equipped with a high-speed stirrer (1800 rpm) Eurostar digital made by IKA-Werke GmbH. The substrates were introduced in two modes. In one mode carbon dioxide was introduced to the suspension with the modifier at the rate of either 1 or 7 dm^3/min, for 3 h, to get pH of 8, while in the second mode carbon dioxide was introduced in parallel with the suspension of magnesium hydroxide into a water system containing PEG. The suspension of a concentration of 5 or 10% was supplied by a peristaltic pump ISM833A, Ismatec. The process was performed at 40°C. The magnesium carbonate precipitate was dried at 105ºC, for about 8 h and calcined in a programmable furnace Controller P320 MB1, made by Nabertherm GmbH, at 300°C, 600°C or 800°C for 1.5 h to get the final product of magnesium oxide.

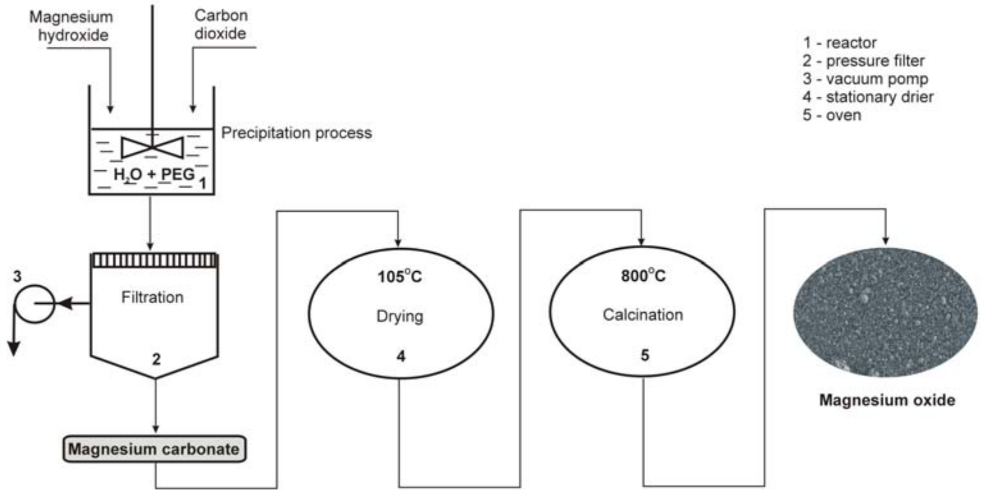


Fig. 1. A scheme of precipitation process of magnesium carbonate from a suspension of magnesium hydroxide and carbon dioxide with a modifier

The final products obtained were subjected to determination of bulk density and particle size distribution. The particle size distribution was measured by two instruments: Zetasizer Nano ZS and Mastersizer 2000 made by Malvern Instruments Ltd., allowing measurements in the range 0.6–6000 nm (NIBS method) and 0.2–2000 μm (diffraction laser scattering technique). The wettability of the final products was evaluated by using a K100 tensiometer made by Krüss. For selected MgO samples also the adsorption parameters were determined such as specific surface area (BET), total volume and mean size of pores calculated according to the BJH method. The measurements were made using an ASAP 2020 instrument made by Micromeritics Instrument Co. The crystalline structures of magnesium carbonate and magnesium oxide were characterised by the WAXS method with the use of a computer controlled horizontal diffractometer TUR M-62 with a HZG-3 type goniometer. The TG/DTA thermal analysis was made using the instrument for thermal stability measurements Jupiter STA 449 F3 made by Netzsch GmbH.

3. Results and discussion

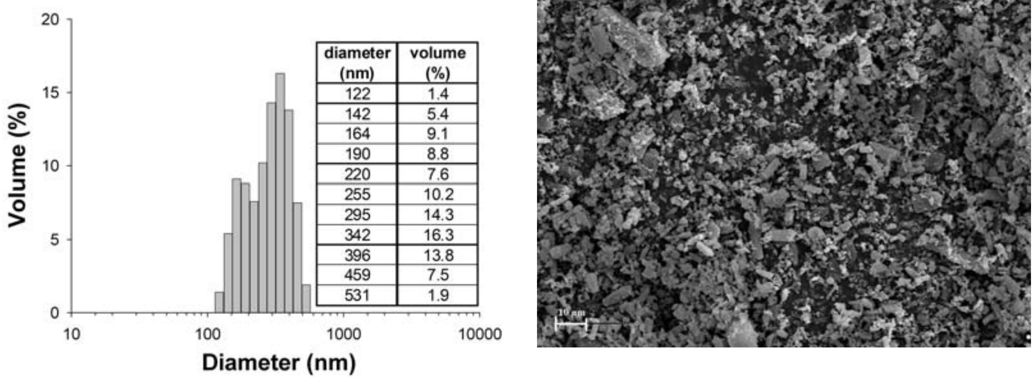
Table 1 presents the physicochemical properties of magnesium oxide samples obtained by calcination of magnesium carbonate at 800ºC, for 1.5 hour. The process of precipitation was performed at 40ºC by introducing carbon dioxide at the rate of 1 dm^3/min into a 10% suspension of magnesium hydroxide with a modifier. The modifiers used were solutions of poly(ethylene glycol) compounds: PEG 200, PEG 400, PEG 8000 and PEG 20000, of concentrations from the range 0.5–2%.

Table 1. Dispersive properties of magnesium oxide samples obtained from magnesium carbonate precipitated by introduction of carbon dioxide at the rate 1dm^3/min to a 10% suspension of magnesium hydroxide and a modifier, at 40°C

Sample No.	Precipitation process conditions		Dispersive properties				
	Mean molecular weight of PEG	PEG concentration (%)	PdI	Particle diameter from Zetasizer Nano ZS (nm)	Particle diameter from Mastersizer 2000 (µm)		
					d(0.1)	d(0.5)	d(0.9)
1	–	–	0.692	1990 – 5560	2.95	13.30	36.25
2	200	0.5	0.400	955 – 5560	2.54	11.85	32.88
3		1	0.351	1110 – 3580	2.26	8.22	30.01
4		2	0.247	122 – 531	1.74	5.05	17.37
5	400	0.5	0.620	1990 – 5560	2.87	11.09	35.65
6		1	0.580	1720 – 5560	2.85	9.04	33.35
7		2	0.243	38 – 79; 825 – 2300	8.98	8.98	26.67
8	8000	0.5	0.501	1480 – 5560	3.45	14.61	38.21
9		1	0.429	1480 – 5560	3.28	12.93	34.90
10		2	0.683	1480 – 5560	3.51	14.79	39.41
11	20000	0.5	0.679	38 – 51; 2670 – 5560	3.14	12.38	33.41
12		1	0.432	396 – 615; 955 – 5560	3.35	8.39	23.04
13		2	0.304	1990 – 5560	2.41	7.13	19.86

Comparative analysis of the dispersive-morphological properties of MgO samples synthesized with a PEG modifier and without a modifier (Sample 1) has shown a beneficial effect of the modifiers applied except for PEG 8000. The samples of magnesium oxide obtained with the use of a modifier (samples 2–7 and samples 11–13) show lower values of the polydispersity index indicating their more uniform character and smaller particle sizes. The results of measurements by Mastersizer 2000 (range up to 2000 µm) prove that the sizes of particles in the modified samples are much smaller than in the unmodified sample. Much smaller particle diameters and much lower polydispersity index values were recorded for magnesium oxide synthesised with a PEG modifier used at the highest concentration. These results illustrate the benefits following from the use of modifiers and confirm their positive effect on nucleation of the crystalline phase.

The best properties were determined for the samples of magnesium oxide synthesised with the use of PEG 200 (samples 2, 3, 4). In sample 4, 10% of the particles have diameters smaller than 1.74 µm, 50% have diameters up to 8.22 µm, and 90% of the particles have diameters smaller than 17.37 µm. A relatively low value of d(0.9) informs about a restricted tendency towards formation of large secondary agglomerations. These observations are confirmed by PdI=0.247 and SEM microphotograph presented in Fig. 2b. Figure 3a presents the particle size distribution obtained on the basis of measurements by Zetasizer Nano ZS, revealing a single band covering the particle diameters range 122–531 nm.



diameter (nm)	volume (%)
122	1.4
142	5.4
164	9.1
190	8.8
220	7.6
255	10.2
295	14.3
342	16.3
396	13.8
459	7.5
531	1.9

(a) (b)

Fig. 2. (a) Particle size distribution (Zetasier Nano ZS) and (b) SEM microphotograph of magnesium oxide (sample 4) obtained from magnesium carbonate precipitated by introducing carbon dioxide into the magnesium hydroxide suspension with 2% PEG 200

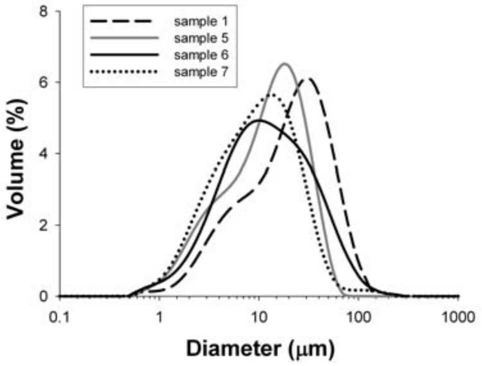


Fig. 3. Particle size distribution (Mastersizer 2000) of unmodified magnesium oxide – sample 1 and modified with PEG 400 – samples 5, 6, 7

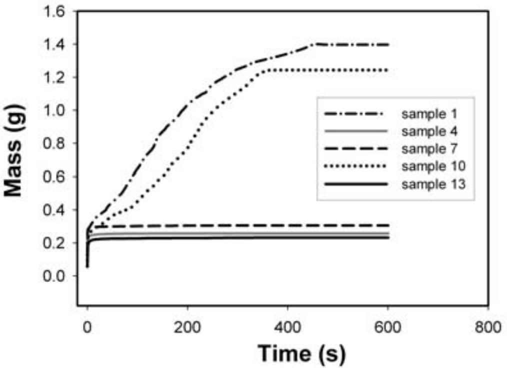


Fig. 4. Wettability profiles of unmodified magnesium oxide (sample 1) and grafted MgO (samples 4, 7, 10, 13)

The beneficial effect of the modifier on dispersive and morphological properties of MgO samples obtained by carbonatization is best manifested by a comparison of the particle size distribution curves for the unmodified sample 1 and for samples 5, 6 and 7 obtained with the use of PEG 400 of different concentrations. The shape of the band recorded for sample 1 points to polydispersity of this sample and this band is shifted towards greater diameters and dominant contribution of greater particles.

No report has been found in hitherto published literature on modification of magnesium carbonate synthesis with addition of PEG compounds. However, such a surfactant has been used in the process of precipitation of magnesium hydroxide from which magnesium oxide was obtained as a final product. Wang (2007) has provided evidence proving the influence of PEG 400 on the formation of nanosized MgO. The same author described the mechanism of PEG effect which involves adsorption of PEG on the $Mg(OH)_2$ crystal inducing changes in the surface density of Mg atoms. Generally, the face with a higher density of surface atoms is blocked by the adsorption of surfactant during the crystal growth of nanocrystals and the growth along this face is therefore considerably restricted. This mechanism can be also applied to explain the effect of PEG on magnesium carbonate.

Besides microstructural properties, another parameter determining the technological applicability of materials is their water absorption capacity. Much attention has been devoted to production of hydrophobic materials by simple and cheap methods. An exemplary modification applied to reduce water absorption is a one-step method of magnesium hydroxide precipitation with addition of octadecyl dihydrogen phosphate as a surface modifier (Dongmin 2009). This modifier has been used to control the growth of the crystal and to modify the surface properties of $Mg(OH)_2$.

Figure 4 presents the wettability profiles of magnesium oxide samples 4, 7, 10, 13 modified with PEGs 200, 400, 8000 and 20000, respectively, used in a concentration of 2% and for comparison the wettability profile of unmodified sample 1. The greatest ability to absorb water is shown by sample 1, obtained without modification and sample 10 modified by PEG 8000. Much higher hydrophobicity was shown by the samples obtained with the use of PEGs 200, 400, 20000 (0.2–0.3 g in the same time). The enhanced hydrophobic character of these samples is related to their microstructural features and partial adsorption of surfactants. An exception is the result for sample 10 obtained with the use of PEG 8000 as for this modifier no beneficial changes in the dispersive and morphological properties were noted. Most probably the relatively high ability to absorb water follows from the presence of large size agglomerates and inhomogeneous morphology of particles, similar as that observed for the unmodified sample.

At the second stage of the study magnesium carbonate was precipitated by simultaneous supply of a 5% of magnesium hydroxide and carbon dioxide (at the rate of 7 dm^3/min) into a water system without a modifier (sample 14) and with PEG modifiers of different masses and in different concentrations (samples 15–26). All samples were synthesised at 40ºC. As at the first stage, the non-ionic compounds from the group of poly(ethylene glycols) of different molecular masses and at different concentrations were used as modifiers.

Table 2. Dispersive properties of magnesium oxide samples synthesised with the use of a 5% suspension of magnesium hydroxide with an addition of a modifier, with CO_2 supplied at the rate of 7 dm^3/min and at 40°C

Sample No.	Precipitation process conditions		Dispesive properties				
	Mean molecular weight of PEG	PEG concentration (%)	PdI	Particle diameter from Zetasizer Nano ZS (nm)	Particle diameter from Mastersizer 2000 (μm)		
					d(0.1)	d(0.5)	d(0.9)
14	–	–	0.743	1280 – 3580	3.54	17.59	45.49
15	PEG 200	0.5	0.638	1720 – 4150	3.20	9.79	30.41
16		1	0.538	1480 – 5560	3.28	9.19	27.07
17		2	0.435	1480 – 5560	2.26	5.13	12.80
18	PEG 400	0.5	0.664	21 – 43; 2670 – 5560	3.08	12.23	37.89
19		1	0.599	712 – 5560	2.70	12.86	31.53
20		2	0.480	164 – 295	1.95	5.71	23.83
21	PEG 8000	0.5	0.502	1480 – 5560	2.86	9.84	36.24
22		1	0.632	2670 – 5560	3.16	13.46	46.19
23		2	0.499	3090 – 5560	2.27	11.80	42.93
24	PEG 20000	0.5	0.303	28 – 59; 955 – 1990	1.97	6.22	21.39
25		1	0.418	68 – 220; 3580 – 5560	2.19	6.11	19.92
26		2	0.373	106 – 5560	1.77	5.19	16.57

Figure 5 confirms improvement in the dispersive and morphological properties of as a result of the use of PEG 400 as a modifier. Sample 20 obtained with addition of PEG 400 in the highest concentration has particles of much smaller size than those in the unmodified sample. The best properties from the viewpoint of future technological use are shown by the samples modified with PEG 20000. These samples have the lowest tendency towards formation of large agglomerations: d(0.9) in the range 16.57–21.39 μm. The presence of nanometric particles was proved almost in all volume of sample 24 modified with a 0.5% solution of PEG 20000, which points to high effectively of this type of modifier applied in the simultaneous mode of reagents supply. The particle size distribution presented in Fig. 4a shows two bands. One is narrow, of high intensity and covers the diameters from the range 28–59 nm, with the maximum volume contribution of 39% is brought by particles of 38 nm in diameter. The second low intensity band testifies to the formation of secondary agglomerates. The results obtained for the samples synthesised in the mode of simultaneous supply of reagents confirmed the unfavourable influence of modification with PEG 8000. Samples 21–23 obtained with addition of PEG 8000 were found to be made of particles of sizes close to those of the unmodified sample 14.

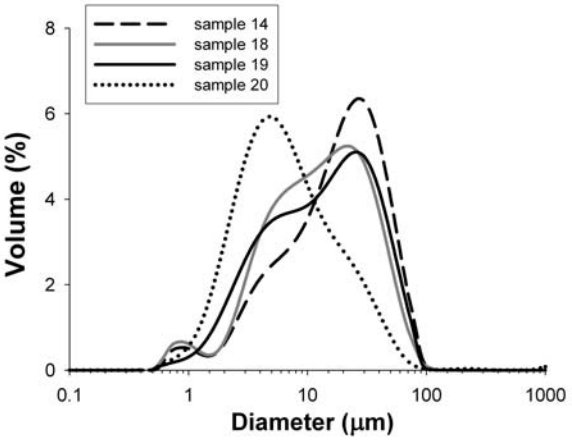
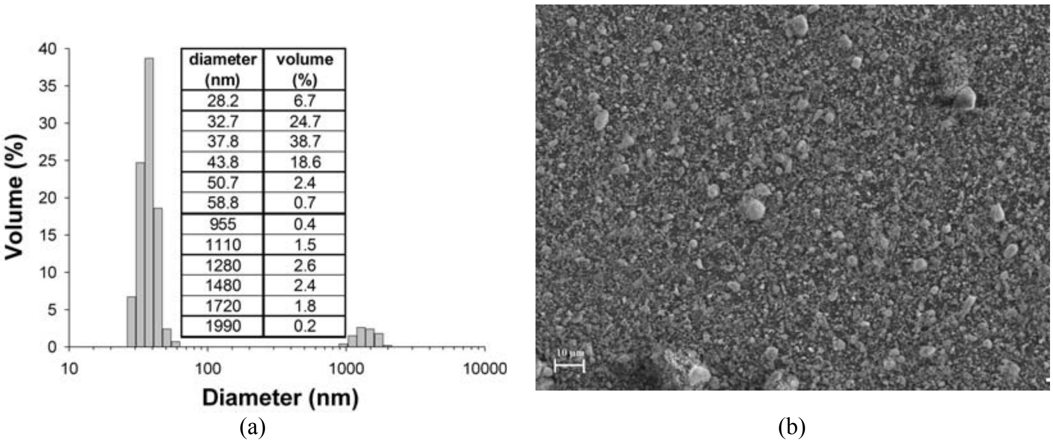


Fig. 5. Particle size distribution (Mastersizer 2000) of unmodified magnesium oxide (sample 14) obtained without a modifier and obtained with addition of PEG 400 in different concentrations (samples 18, 19, 20)



diameter (nm)	volume (%)
28.2	6.7
32.7	24.7
37.8	38.7
43.8	18.6
50.7	2.4
58.8	0.7
955	0.4
1110	1.5
1280	2.6
1480	2.4
1720	1.8
1990	0.2

(a) (b)

Fig. 6. (a) Particle size distribution (Zetasier Nano ZS) and (b) SEM microphotograph of magnesium oxide (sample 24) obtained in the mode of simultaneous supply of reagents into a water system with addition of 0.5% solution of PEG 20000

In order to evaluate the product purity and determination of the range of temperatures corresponding to significant chemical or structural transformations of magnesium carbonate, the samples were subjected to a TG/DTA study (see Fig. 6). As the results were similar for all samples, only the results obtained for the unmodified sample (sample 14) and the sample modified with 2% PEG 20000 are shown. DTA measurements permit detection of thermal effects accompanying the physical or chemical transformations. No significant changes in the courses of TG/DTA curves for the unmodified and modified samples were noted. The results obtained for modified samples revealed an exothermic effect in the range 25–370ºC related to the loss of crystallization water. The TG curve informing about the mass change upon heating shows in the corresponding point a signal indicating the mass loss of 22.47%. The second effect observed on the DTA curve was endothermic and occurred in the range 450–900°C. It was interpreted as a manifestation of decomposition of magnesium carbonate and reorganization of the $MgCO_3$ structure to the pure crystalline form of MgO, related to the loss of CO_2 recorded in the TG curve as a rapid mass loss of 43.42%.

The purposefulness of calcination of magnesium carbonate at much elevated temperatures was confirmed by the results of WAXS studies (see Fig. 7). The WAXS analysis was made for the same material but calcined at three different temperatures 300ºC, 600ºC and 800ºC. For the material calcined at 600ºC the maxima characteristic of MgO structure (cubic structure of periclase). For the material calcined at 800ºC the maxima are even more intense and have regular shapes, which proves the presence of a well-developed magnesium oxide crystals. Cloudhary (1994) has reported detection of traces of carbon in MgO samples calcined at 900ºC. He interpreted this observation by strong ability of CO_2 adsorption from the atmospheric air of MgO centres.

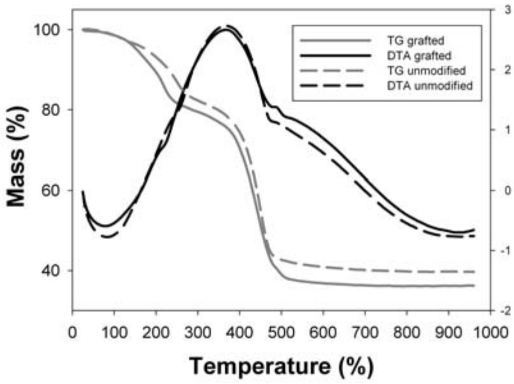


Fig. 7. TG/DTA of unmodified magnesium oxide (sample 14) and grafted MgO (sample 26)

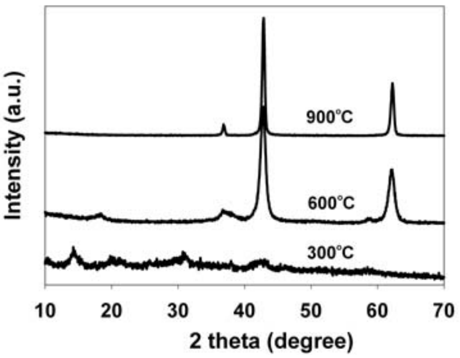


Fig. 8. X-ray diffraction patterns for a selected magnesium oxide (sample 24) for different temperatures of decomposition

Table 3 presents results of the adsorption study of some selected magnesium oxide samples unmodified (samples 1, 14) and modified obtained as a result of thermal composition of appropriate magnesium carbonates at 800ºC. The values of specific surface area were low, but a bit higher for the samples obtained with an addition of a modifier. This observation is explained, similarly as the wettability results, by a beneficial effect of PEG on surface activity of magnesium oxide.

The highest values of specific surface area were found for samples 4 and 24, which also showed the best dispersive properties (see Tables 1, 2). The samples obtained in the first mode, when CO_2 was introduced into the suspension, are definitely mesoporous as their pore diameters vary in the range 22.4–28.5 nm.

The results presented in this paper are a continuation of the previous study (Pilarska, 2011), on synthesis of magnesium carbonates and their calcinates by the method of carbonatization. The previous work concerned determination of the conditions of the process of synthesis on the basis physicochemical parameters of the final product, MgO. On the basis of the results from the previous work we could choose and apply the best parameters of the process of magnesium carbonate precipitation. The optimum reaction systems (leading to samples 1 and 14) were modified by addition of a non-ionic surfactant. The results presented in this paper prove a beneficial effect of modification with PEG compounds. Depending on the molecular mass and concentration of a modifying PEG and on the mode of reagents supply, the final materials were characterised by different dispersive-morphological and physicochemical properties.

Table 3. Adsorptive properties of selected magnesium oxide samples precipitated at different precipitation process conditions with and without a modifier

Sample No.	BET surface area (m^2/g)	Total volume of pores (cm^3/g)	Mean size of pores (nm)
1	21	0.15	28.5
4	36	0.21	22.8
7	33	0.19	22.4
14	17	0.01	2.9
20	34	0.02	2.8
24	35	0.02	2.9
26	30	0.02	2.7

4. Conclusions

As follows from the dispersive measurements, the final samples obtained with the use of a modifier contain of smaller size particles and have a more uniform morphology. In the mode of gaseous CO_2 supply to a 10% suspension of magnesium hydroxide, the sample of the most favourable properties was that obtained with the addition of PEG 200 in a concentration of 2% (particle size varied from 122 to 531 nm). In the case of simultaneous supply of the reagents into a water system, with the use of a 5% magnesium hydroxide suspension, the samples of the most attractive properties was that obtained with the addition of PEG 20 000 in a concentration of 0.5% (particles size varied from 28 to 531 nm, low tendency towards agglomerate formation). Changes in the microstructure were found to affect the sample wettability. The samples obtained with the addition of 2% PEGs 200, 400 or 20000 were almost fully hydrophobic. The MgO samples obtained with the use of a modifier show specific surface areas in the range 30–36 m^2/g, which is somewhat greater than those of the unmodified samples. All finally obtained MgO samples are characterised by a well-developed crystalline structure.

Acknowledgements

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Przedstawiono badania nad wpływem modyfikatorów i warunków procesowych na otrzymywanie tlenku magnezu metodą karbonizacji. Otrzymany z użyciem wodorotlenku magnezu i ditlenku węgla – węglan magnezu, poddano termicznemu rozkładowi w temperaturze 800ºC. Reakcję strącania przeprowadzono uwzględniając warunki eksperymentalne takie, jak: temperatura, sposób i szybkość dozowania reagentów oraz stężenie i rodzaj modyfikatora. Jako modyfikatory zastosowano związki niejonowe z grupy glikoli polietylenowych. Dla wytworzonych tlenków magnezu określono właściwości dyspersyjno–morfologiczne, profile zwilżalności oraz wyznaczono podstawowe parametry adsorpcji: powierzchnię właściwą BET, całkowitą objętość i średnią średnicę porów. Ponadto uzyskane produkty poddano identyfikacji metodą rentgenograficzną oraz analizie TG/DTA. Dowiedziono istotnego wpływu stężenia i rodzaju modyfikatorów na parametry fizykochemiczne otrzymanego MgO oraz wyłoniono produkty o najbardziej reprezentatywnych właściwościach.

słowa kluczowe: MgO, karbonizacja, modyfikacja, rozkład termiczny, wielkość cząstek, morfologia powierzchni