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SYNTHESIS OF $Mg(OH)_2$ FROM MAGNESIUM SALTS AND NH_4OH BY DIRECT FUNCTIONALISATION WITH POLY(ETHYLENE GLYCOLS)

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Abstract. Synthesis of magnesium hydroxide using different magnesium salts and ammonium hydroxide is described. The reaction was performed in the presence of non-ionic compounds representing the group of poly(ethylene glycols) as modifiers. Dispersive properties of the products were characterised by polydispersity index, particle size, as well as SEM and TEM images. Adsorptive properties of the products were evaluated on the basis of their specific surface area, pore volume and diameter determination. Physicochemical characterisation of the products was extended by the degrees of their surface coverage with PEG, calculated on the basis of elemental analysis and specific surface area BET. Crystalline structures of the products were determined by the WAXS method and then using the Scherrer formula the crystallite size was calculated for selected samples. Wettability profiles in aqueous systems were measured and selected samples were subjected to TG/DTA analyses. The properties of the products were found to be significantly dependent on the molecular mass of PEG and type of magnesium salt used.

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keywords: $Mg(OH)_2$, poly(ethylene glycols), particle size distribution, surface morphology, adsorptive properties, crystal structure, elemental analysis, TG/DTA analysis

1. Introduction

Magnesium hydroxide is a very attractive compound because of a wide range of its important applications. Besides, $Mg(OH)_2$ is one of the most important precursors of magnesium oxide. It is used as a neutraliser of acidic pollutants of water and gases, filler in paper industry, antibacterial agent, and fertiliser additive (Dziubek, 1984; Giorgi, 2005; Dong, 2010). One of its interesting properties is the diversity of its microstructure. It crystallises as needles, plates, rods, tubes and flowers (Yan, 2005; Wang, 2011;Song, 2011). Its non-toxic character and high thermal stability make it an attractive additive to polymers used to restrict polymers flammability. The appearance of many papers on $Mg(OH)_2$ indicate increasing interest in this inorganic filler. The main subject of these papers are new methods of synthesis of nano-$Mg(OH)_2$ and ways

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of modification of the $Mg(OH)_2$–polymer system aimed at improvement its compatibility (Lv, 2007; Chen, 2009; Jurkowski, 2010; Gul, 2011).

Recently, much attention has been paid to the methods of synthesis of nanomolecular $Mg(OH)_2$. The methods proposed include: precipitation, solvo- and hydrothermal, microwave, electrochemical, sol-gel and the method based on watering of the commercial MgO with the use of dolomite and sea water (Ding, 2001; Du 2009; Yildirim, 2010; Bae, 2011; Pilarska, 2011). The above-mentioned methods, except precipitation, need a long time, complex and expensive equipment, which means that the process of magnesium hydroxide production becomes economically unattractive. The method based on precipitation is easy to conduct also on an industrial scale. It permits control of the particle size and is rather inexpensive. No wonder that it has been intensely studied in a number of research groups (Jiang, 2009; Meshkani, 2009; Meshkani, 2010).

According to the recent literature reports, the properties of the final product, in particular its morphology, are closely related to the type of precursor and the precipitating agent. Much interest is devoted to the use of modifiers. Modification of the reaction system, understood as direct participation of an additional organic or inorganic chemical substance in the process of synthesis, is undertaken to modify the original properties of the product, including its surface properties. Modifications of this type are most often applied upon precipitation of magnesium hydroxide (Wang, 2007; Huang, 2008; Wang, 2009; Pilarska, 2011). Functionalisation of the surface of fillers is aimed first of all at control of the shape and size of particles and at making the product more hydrophobic to permit binding of inorganic fillers with organic polymer matrices (Rothon, 2003).

This paper reports the synthesis of $Mg(OH)_2$ of plate morphology realised by the method of precipitation with the use of magnesium salts, ammonium hydroxide and non-ionic compounds from the group of poly(ethylene glycols) as modifiers. The influence of molecular weight and type of magnesium precursor on the dispersive-morphological and physicochemical properties of the products has been mainly analysed.

2. Experimental

The substrates used for the synthesis of magnesium hydroxide were: hydrated magnesium sulfate ($MgSO_4 \cdot 7H_2O$, analytical grade), magnesium chloride ($MgCl_2 \cdot 6H_2O$), magnesium nitrate ($Mg(NO_3)_2 \cdot 6H_2O$, analytical grade), ammonium hydroxide (NH_4OH, analytical grade) as a precipitation agent (all made by POCh SA.) and non-ionic compounds from the group of poly(ethylene glycols): PEG 200, PEG 400, PEG 8000 and PEG 20000 (purchased from Sigma-Aldrich), as solutions of appropriate concentration. Magnesium hydroxide was obtained from a 5 % solution of a given magnesium salt and a solution of ammonium hydroxide prepared to maintain the stoichiometric amount of reagents. Precipitation was carried out at 60°C. A technological scheme of the process is shown in Fig. 1.

Portions of 200 cm^3 of a given magnesium salt solution and 200 cm^3 of ammonium hydroxide were simultaneously dosed into 50 cm^3 of water with a modifier, placed in a reactor of 500 cm^3 in capacity, equipped with a high speed stirrer (1800 rpm) (Eurostar Digital, IKA Werke). The reagents were vigorously stirred. The reagents were dosed by a peristaltic pomp of Ismatec ISM833A type, at a constant rate of 2.3 cm^3/min. The reactor was placed in a thermostat (MP14, Julabo), to control and keep a constant temperature. The precipitate was washed out, filtered and subjected to conventional drying at 105ºC for about 8 h.

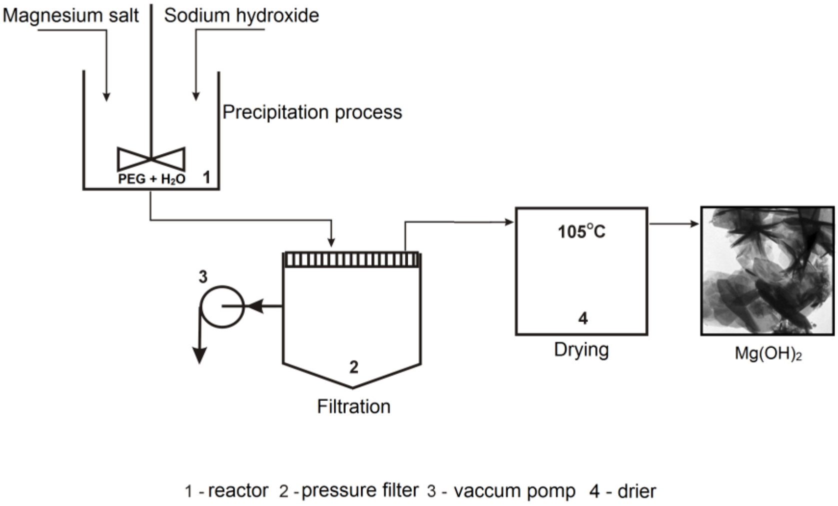


Fig. 1. Technological scheme of $Mg(OH)_2$ synthesis from a magnesium salt and ammonium hydroxide with a modifier

The particle size of the precipitate was measured by Zetasizer Nano ZS (0.6–6000 nm) and Mastersizer 2000 (0.2–2000 μm) made by Malvern Instruments Ltd., operating by the NIBS method and laser diffraction techniques, respectively. The Zetasizer Nano ZS instrument was also used for determination of the polydispersity index (PdI), which is a measure of the sample homogeneity. The morphology and microstructure of the products were characterised on the basis of observations by a scanning electron microscope Zeiss EVO 40 and a transmission electron microscope (Joel 1200 EX II). The adsorption activity of the products was evaluated on the basis of specific surface area BET, pore volume and pore diameter measurements performed using the ASAP 2020 instrument made by Micromeritics Instrument Co. Selected samples were subjected to elemental analyses (Elementar model Vario EL III), whose results together with the surface area BET allowed calculation of the degree of coverage with the modifier. Analysis of the products wettability in water systems was made using the K100 tensiometer with a special instrumentation made by Krüss. The crystalline structure of selected magnesium hydroxide samples was determined by

WAXS. The X-ray diffractograms were recorded by a computer-controlled horizontal diffractometer, equipped with a HZG-3 type goniometer. From the Scherrer formula the crystallite size in the samples was calculated. Selected samples of the products were also subjected to thermal analyses TG/DTA (Jupiter STA 449F3 – Netzsch).

3. Results and discussion

Table 1 presents the dispersive and adsorptive properties of magnesium hydroxide samples precipitated during a simultaneous dosing of reagents prepared in stoichiometric amounts into the aqueous system at the rate of 2.3 cm^3/min at 60°C.

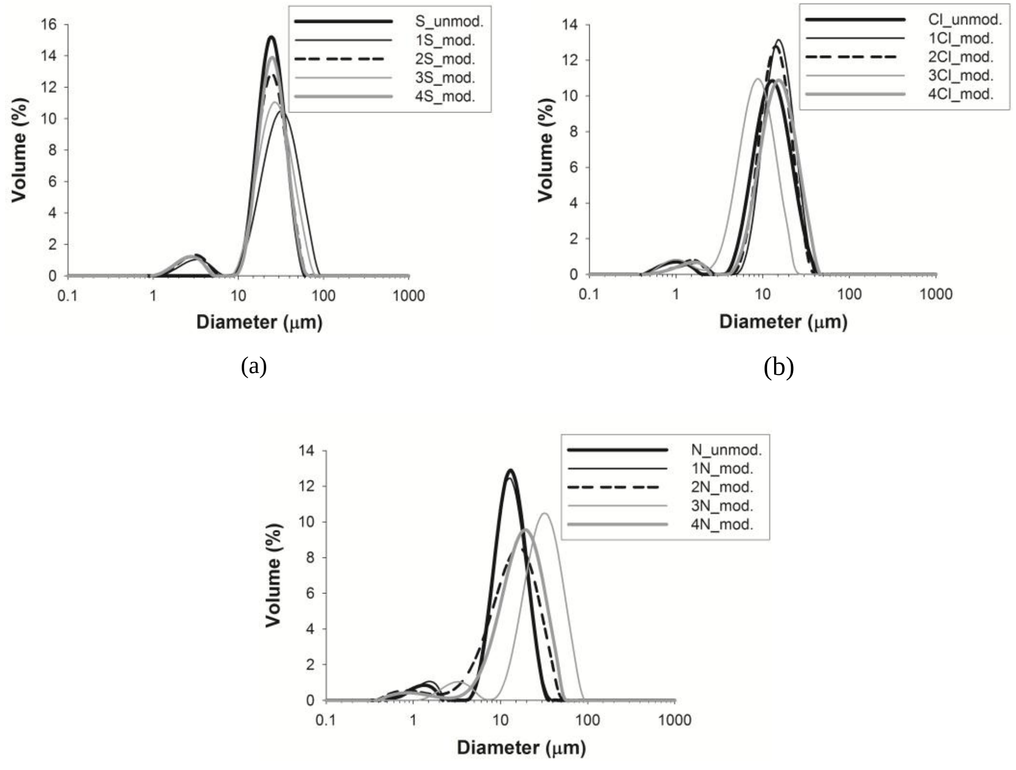
Table 1. Physicochemical properties of magnesium hydroxide precipitated with the use of 5% solution of a magnesium salts, ammonium hydroxide in stoichiometric amount and a 2% solution of one of the following non-ionic compounds PEG 200, 400, 8000, 20000

Sample No.	Precipitation process conditions		Dispersive properties		Adsorptive properties		
	Type of salt	Mean molecular weight of PEG	PdI	Particle diameter from Zetasizer Nano ZS (nm)	BET surface area (m^2/g)	Total volume of pore (cm^3/g)	Mean size of pore (nm)
S_unmod.	magnesium sulfate	–	0.225	21 – 106	21	0.01	2.7
1S_mod.		200	0.688	59 – 220	28	0.01	2.6
2S_mod.		400	0.346	59 – 142	42	0.22	28.2
3S_mod.		8000	0.435	68 – 459	30	0.01	2.6
4S_mod.		20000	0.387	79 – 255	32	0.01	2.7
Cl_unmod.	magnesium chloride	–	0.196	220 – 396	3.3	0.02	2.8
1Cl_mod.		200	0.287	51 – 459	10	0.01	3.5
2Cl_mod.		400	0.312	122 – 459	8	0.01	3.5
3Cl_mod.		8000	0.571	68 – 2670	12	0.02	3.6
4Cl_mod.		20000	0.496	59 – 342	6	0.01	3.1
N_unmod.	magnesium nitrate	–	0.365	106–255; 1110–1990	3	0.01	1.5
1N_mod.		200	0.380	33 – 396	4	0.01	2.2
2N_mod.		400	0.428	51 – 825	4.5	0.01	2.6
3N_mod.		8000	0.520	79 – 3090	7	0.01	2.5
4N_mod.		20000	0.321	68 – 190	6	0.01	2.1

The following magnesium salts, in 5% solutions, were used: magnesium sulfate (samples S_unmod., 4S_mod.), magnesium chloride (samples Cl_unmod., 4Cl_mod.) and magnesium nitrate (samples N_unmod., 4N_mod.) with an addition of a 2% solution of a modifier of different molecular mass PEG 200, 400, 8000, 20000 – for each salt. The modification was performed to obtain the products with particles of smaller size than that of unmodified sample, hydrophobic character and larger surface areas.

As follows from the above results, the particles of each modified sample were greater than those of unmodified samples or more or less of the same size. No reduction in the particle size upon modification was observed. The smallest diameters (59–459 nm) were noted for the samples precipitated from the solution of magnesium sulfate, the same samples also showed the most homogeneous and regular morphology (Fig. 2a). The samples obtained with magnesium chloride or magnesium nitrate revealed the presence of particles of diameters reaching 3090 nm (sample 3N_mod.). The increase in the particle diameters is particularly well seen in the samples obtained with magnesium nitrate (Fig. 2c). An analysis of dispersive and adsorptive properties of the products obtained with magnesium chloride and magnesium nitrate has shown a positive effect of modification with PEG 200 and PEG 20000. Dispersive properties and microstructure of the modified samples determined their specific surface area. For all the modified samples (see Table 1) the specific surface area BET is greater than in their unmodified correspondents. The best adsorptive parameters were found for the samples obtained with magnesium sulfate, S series, in particular sample 2S_mod., modified *in situ* with PEG 400, whose surface area BET was 42 m^2/g, which was twofold greater than that of sample S_unmod. Unusually, a high value of the pore volume (0.22 g/cm^3) and pore diameter (28.2 nm), indicated mesoporous structure of this material. Figure 3 presents the nitrogen adsorption/desorption isotherm for sample 2S_mod and the pore size distribution. According to the IUPAC classification it is a type IV isotherm with a hysteresis loop H3 type, which is related to the capillary condensation in mesopores between two planes. Sample 2S_mod. is the best candidate to be an adsorbent, catalysts support or a catalyst, among all samples studied in this experiment.

The inorganic salts $MgCl_2$, $MgSO_4$ or $Mg(NO_3)_2$ are commonly used as precursors of magnesium hydroxides. According to the literature, $Mg(OH)_2$ obtained in the reaction with ammonium hydroxide has particles in the form of hexagonal plates (Dong, 2010; Wang, 2011). TEM microphotographs (Fig. 4a) show the particles of magnesium hydroxide with plate-like structures (2S_mod. sample). In samples obtained with magnesium chloride (1Cl_mod., Fig. 4b) – the particles are of star-like shape. In a recent work (Wang, 2011) on magnesium hydroxide precipitated with the use of the same reagents (i.e. magnesium chloride and ammonium hydroxide) the same morphology was described as a broom shape. Wang (2011) presented in details the mechanism of formation of such unusual shapes. He claims that in these samples the process of nucleation depends considerably on the relation of surface diffusion and surface integration in the crystal growth process. In the same paper Wang (2011) analysed also the influence of a modifier (poly(ethylene glycol), PEG 12000) on the phenomenon of plate formation at different reaction temperatures (determining the way of PEG adsorption). At 60ºC and 80ºC magnesium hydroxide particles develop as plates as PEG is adsorbed exclusively on the (001) crystalline surface. Restricted growth of $Mg(OH)_2$ crystals along the (001) surface leads to formation of plates. At lower temperatures PEG adsorption leads to formation of needles. These morphological features can determine the type of application of the products obtained (catalysis, adsorption, flame retardancy).



(a) (b)

(c)

Fig. 2. Particle size distribution (Mastersizer 2000) of magnesium hydroxides obtained with the use of: (a) magnesium sulfate, (b) magnesium chloride and (c) magnesium nitrate

Physicochemical characterisation of the products was supplemented with X-ray diffraction studies. The X-ray diffraction patterns shown in Fig. 4 reveal the maxima typical of the crystalline structure of brucite $Mg(OH)_2$, identified by the program X-RAYAN (Marciniak, 1999). The X-ray patterns were almost the same for the products obtained with the same magnesium salt irrespective of the molecular mass of the PEG applied. Changes in the shape of the peaks were clearly related to the type of magnesium salt used. The structure of brucite is much better developed for samples Cl_mod. (Fig. 5b) and N_mod., than in sample S_mod, as indicated by the height and shape of relevant diffraction maxima. The differences in the crystalline structure are confirmed by the different sizes of crystallites in the samples in selected directions. For example the crystallite size calculated from the Scherrer formula for the direction perpendicular to (001) plane (maximum at $2\theta=18.5°$), for samples 1Cl_mod. and

1N_mod., reach about 35 nm, while the crystallite size in sample 1S_mod. is of about 12 nm. Similarly for the direction perpendicular to the (101) plane (maximum at $2\theta=37.9°$), the crystallite size in samples 1Cl_mod. and 1N_mod. is of an order of 40 nm (39 nm and 43 nm, respectively), while for sample 3S this size is almost twice smaller (18 nm). The above values are comparable with literature data (Ding, 2001; Klug, 2001; Wang, 2011).

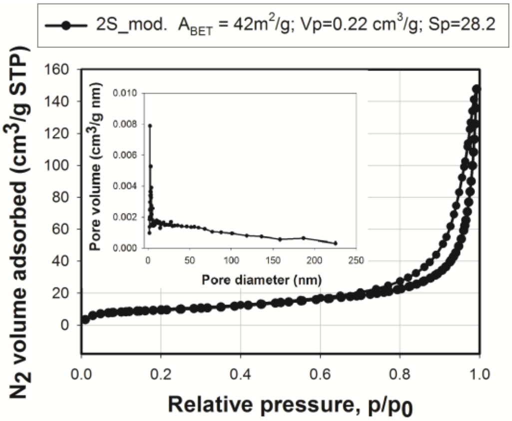
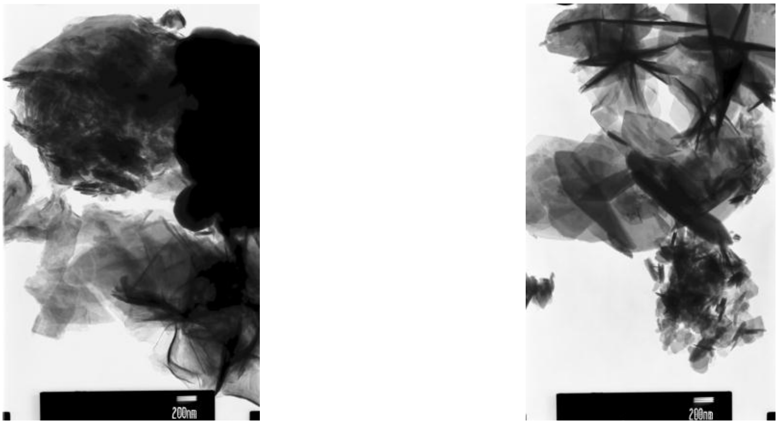
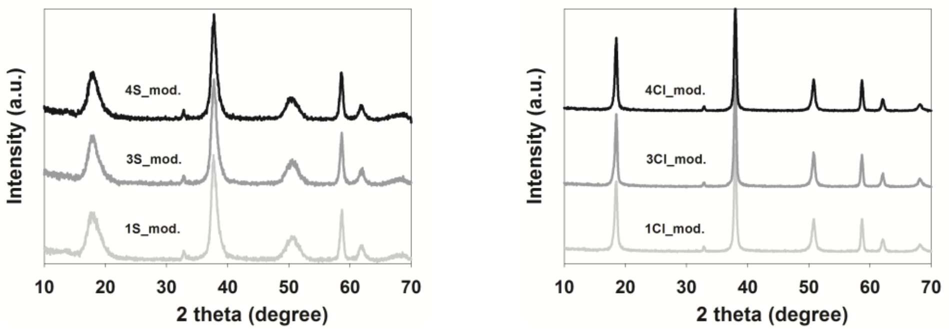


Fig. 3. Nitrogen adsorption/desorption isotherm and pore size distributions of magnesium hydroxide 2S_mod. grafted by PEG 400



(a) (b)

Fig. 4. TEM images of magnesium hydroxides (a) 2S_mod. and (b) 1Cl mod. obtained with the use magnesium sulfate and magnesium chloride

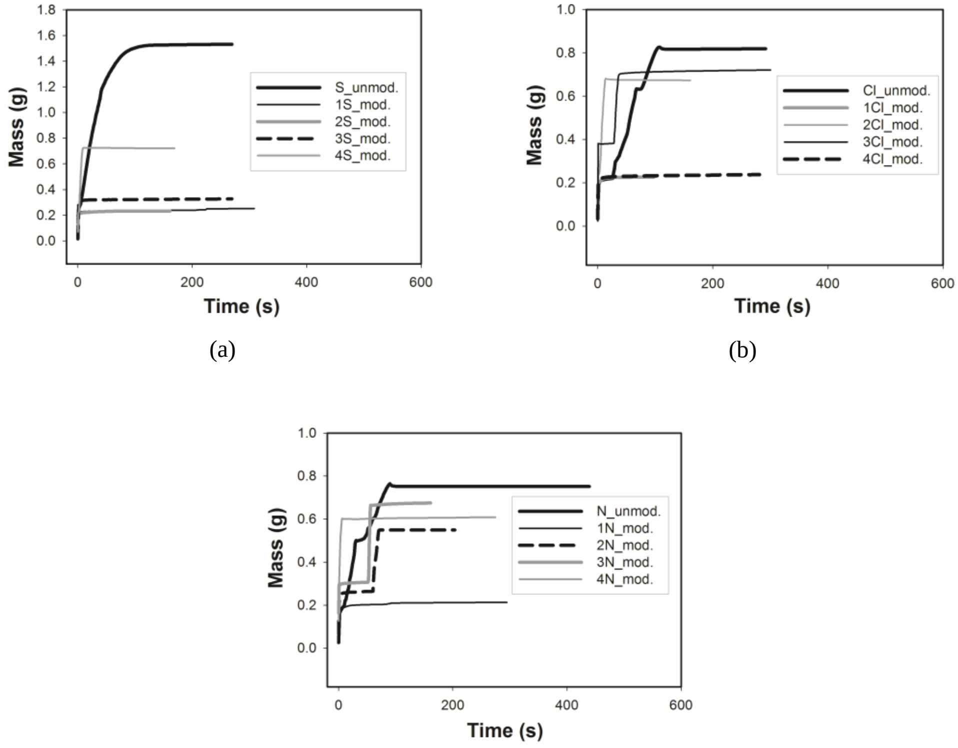


(a) (b)

Fig. 5. X-ray diffraction patterns of magnesium hydroxides obtained with the use of (a) magnesium sulfate and (b) magnesium chloride and with addition of different non-ionic compound from the group of poly(ethylene glycols)

The modifier is also applied in order to endow the samples of magnesium hydroxide with hydrophobic properties, which is essential to enhance their stability and extend their application possibilities as active fillers. The hydrophilic surface of $Mg(OH)_2$ nanoparticles is highly unfavourable for their dispersion in polymers. Poor compatibility between $Mg(OH)_2$ and polymer causes degradation in the mechanical and physical properties of the composites. Literature provides examples of facile methods to synthesize hydrophobic $Mg(OH)_2$ nanoparticles with good dispersibility and compatibility in organic phase with the use of octadecyl dihydrogen phosphate (ODP), oleic acid, poly(methyl methacrylate) (PMMA) etc. (An 2009, Yan 2009). Figures 4a, b, and c present the wettability profiles recorded for the samples precipitated with the use of the three magnesium salts studied and 2% solutions of PEG of different molecular weight. The highest tendency towards water absorption was observed for strongly hydrophilic unmodified hydroxides, while the lowest tendency towards water absorption was recorded for samples modified with PEG 200 (1S_mod., 1Cl_mod., 1N_mod.) PEG 400 (1S_mod.) and PEG 20000 (4Cl_mod.), irrespective of the salt used for precipitation. As follows from the calculated degree of coverage (see Table 3), PEG 200 undergo permanent absorption forming most probably a compact protective layer. Moreover, PEG 200 is built of shorter carbon chains and contains a lower number of hydroxyl groups. Significant hydrophobicity of samples modified with PEG 20000 (4Cl_mod., Fig. 4b) can be explained by favourable changes in magnesium hydroxide microstructure it causes (see Table 1).

The results of elemental analysis presented in Table 2 show that almost in all samples the contents of carbon and hydrogen increased in proportion to the molecular mass of the PEG applied. The differences in the contents of carbon and hydrogen in the samples modified with PEGs of the shortest and longest carbon chains are not significant. For example the content of carbon in sample 1N_mod. (PEG 200) is 0.595%, while in sample 4N_mod. (PEG 20000) it increases only to 0.735%. Much more pronounced dependence on the molecular weight of PEG is observed for the degree of coverage of magnesium hydroxide surface with the modifier, calculated on the basis of elemental analysis results and the specific surface area. The coverage degree (Table 2) takes the highest value for samples modified with PEG 200 and for those modified with PEG 20000 it takes a value by one order of magnitude lower. Such a rapid decrease in the degree of coverage with increasing length of the modifier means that the adsorption of long-chain PEGs is rather ineffective. Among the samples analysed, the highest degree of coverage was obtained for PEG 200. The results of elemental analysis are well correlated with those of wettability (Figs. 5a, b, c). A high degree of coverage of magnesium hydroxides modified with PEG 200 can explain their strongest hydrophobicity (samples 1S_mod., 1Cl_mod., 1N_mod.). The observation that adsorption of PEGs of short carbon chains is the most effective is corroborated by the dispersive, adsorptive and surface properties of the samples modified with them.



(a) (b)

(c)

Fig. 6. Wettability profiles of magnesium hydroxides obtained with the use of: (a) $MgSO_4$, (b) $MgCl_2$ (c) $Mg(NO_3)_2$

Another interesting observation was that the highest degree of coverage was calculated for the samples precipitated with the use of magnesium chloride and magnesium nitrate, which is related to small surface area BET of these samples. The degree of coverage found for the sample precipitated with magnesium sulfate having smaller surface area is considerably lower.

Table 2. Elemental content of carbon and hydrogen in modified samples of magnesium hydroxide precipitated with the use of magnesium sulfate and magnesium chloride and the calculated degree of their coverage with PEG compounds

Sample No.	Elemental content (%)		Coverage degree (μmol/m^2)
	C	H	
S_unmod.	–	–	–
1S_mod.	1.177	6.135	7.467
2S_mod.	1.276	6.335	4.091
3S_mod.	1.480	6.675	3.683
4S_mod.	1.613	6.878	0.106
Cl_unmod.	–	–	–
1Cl_mod.	0.784	2.825	31.325
2Cl_mod.	0.919	2.893	18.426
3Cl_mod.	0.821	2.924	10.194
4Cl_mod.	1.107	3.056	0.446
N_unmod.	–	–	–
1N_mod.	0.595	2.568	26.023
2N_mod.	0.645	2.825	14.124
3N_mod.	0.690	2.937	9.036
4N_mod.	0.735	2.989	0.323

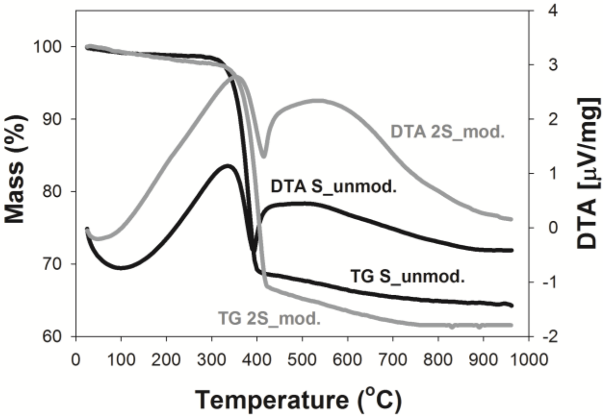


Fig. 7. TG/DTA curves of magnesium hydroxides S_unmod. and 2S_mod.

The products obtained were also subjected to TG/DTA thermal studies in order to establish the character of chemical and structural transformation induced by high temperature and to compare mass changes between the modified and unmodified samples. Figure 6 presents the TG/DTA curves recorded for the unmodified sample (S_unmod.) the sample modified by a 2% PEG 400 solution (sample 2S_mod.). The DTA curve recorded for the unmodified sample shows an exothermic effect appearing in the range 99 to 330ºC, corresponding to the loss of physically bound water. The endothermic effect appearing in the range from 420 to 900°C corresponds to decomposition of magnesium hydroxide and reorganisation of $Mg(OH)_2$ structure to that of pure crystalline form of MgO (Wang, 2007), accompanied by the loss of constitutional water. The rapid mass decrease corresponding to this endothermic effect was manifested in the TG results. The DTA curve recorded for the modified sample is slightly shifted towards higher temperatures. The corresponding TG results point to a greater, by about 4%, mass loss for the modified sample, which confirms the presence of the modifier.

4. Conclusions

Functionalisation of magnesium hydroxide (precipitated with the use of a magnesium salt and ammonium hydroxide) with non-ionic compounds from the group of poly(ethylene glycols) has not brought favourable changes in the dispersive properties of the products. The size of particles in the modified samples was greater or comparable to that in the unmodified magnesium hydroxide. The modification with PEG has resulted in increased specific surface areas BET of all samples. Relatively large specific surface area was obtained for the samples synthesized with the use of magnesium sulfate. The best dispersive properties (particle size range from 59 to 142 nm) and adsorptive properties (surface area BET of 42 m^2/g) were found for sample 2S_mod., modified with PEG 400. Introduction of the modifying PEG compounds to the reaction system *in situ* has brought beneficial changes in the character of the products' surface. The majority of modified samples were observed to show reduced ability to absorb water (to about 0.2 g). Particularly high reduction in water absorbability was found for the samples modified with G 200 (1S_mod., 1Cl_mod., 1N_mod.) and PEG 400 (2S_mod., 2Cl_mod.). The increased hydrophobicity of these samples can be related to the degree of coverage of their surface with the modifier (which reaches the highest values for PEGs of the shortest carbon chains) and with microstructural changes. Because of large BET surface area of the samples precipitated with the use of magnesium sulfate (1S_mod., 4S_mod.) the coverage of their surfaces with the modifier is smaller than that for the other samples

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