CHEMIJA. 2016. Vol. 27. No. 3. P. 170–178

Synthesis of magnesium oxide nanoparticles via sol–gel method and hydrolysis and application for paper deacidification treatment

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Pure magnesium oxide nanoparticles were prepared using the sol–gel method and calcined at various temperatures – 500, 600, 800 and 1000 °C. The average crystalline size of the smallest particles obtained at 500 °C was 7.2 nm. This powder was hydrolyzed at room temperature in the nitrogen atmosphere for 48 h with addition of 10 w.% of water. To stabilize this mixture 1 w.% of hydroxypropyl cellulose (Klucel E) was added. A suspension of pure $Mg(OH)_2$ nanoparticles (20–30 nm) in 2-propanole was directly used for paper deacidification treatment. The samples were investigated and the obtained results were compared with the samples treated with bare $Mg(OH)_2$. The antibacterial and fungicidal efficiency of obtained nanoparticles was also investigated by introducing the particles into the media containing *Saccharomyces cerevisiae* and *Escherichia coli* growth media.

Keywords: paper, deacidification, magnesium hydroxide nanoparticles, aging, sol–gel synthesis

INTRODUCTION

Magnesium and its alloys possess unique properties such as low weight and non-toxicity [1]. In a hydroxide form their nanoparticles have several possible applications. They are commonly used as flame-retardant fillers in composite materials, also can be used as functional polymer composites, reinforcing agents for hybrid materials, to treat flue gases and for neutralization of acid waste streams [2–4]. In paper manufacturing, magnesium hydroxide is used as a fertilizer additive. This alkali material has one more possible application – it can also be used for ancient cellulose paper treatment.

During cellulose degradation, two main reactions prevail – hydrolysis of glycosidic bonds and oxidation of glucopyranose rings. This leads to formation of acids all over paper sheet, which catalyze even faster paper degradation. Cellulose chains break and paper remains brittle [5]. To prevent degradation of historical paper, restorers generally use calcium hydroxide and carbonate solutions in water. Those substances neutralize acids and create protection from formation of new ones – alkali reserve. In the last decade, to prevent degradation of not only paper cultural heritage but also stone, wall paintings and wood, it was suggested to use calcium hydroxide nanoparticles in different forms (nanosols, alcohol dispersions, powders) [6–11]. Smaller particles have a higher surface area, they cover paper with greater efficiency and can penetrate into the cellulose fibers net in all thickness of paper, there neutralize acids and create better cellulose protection from hydrolysis. In this paper magnesium hydroxide nanoparticles were used. There are several methods to obtain this material in nanosize – precipitation of magnesium salt with an alkaline solution, electrolysis of an aqueous magnesium salt solution, electrodeposition and microwave assisted synthesis, also hydrothermal, solvothermal, preparation using a bubbling setup [12–14]. In this study, magnesium hydroxide nanoparticles were produced by hydration of magnesium oxide nanopowder, which was obtained using the sol–gel technique. The sol–gel process is a synthesis route consisting in the preparation of a sol and successive gelation and solvent removal. This is a cheap and environmentally friendly route to high purity oxide nanoparticles. The first step of chemical processes is always carried out at low temperatures and that minimizes chemical interactions between the material and

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container walls. Moreover, kinetics of various chemical reactions can be easily controlled by low processing temperatures and by often dilute conditions [15].

EXPERIMENTAL

All chemical reagents used in our experiments were of analytical grade and directly used without further treatment. For the synthesis of nanosized MgO the sol–gel method was used. Precursors were mixed by dissolving 5.00 g of $Mg(NO_3)_2 \cdot 6H_2O$ and 2.43 g of $H_2C_2O_4 \cdot 2H_2O$ each in 20 ml of ethanol. The obtained white gel was digested for 12 h at room temperature (20 °C) and distilled at 120–125 °C temperature. The sample was completely powdered and calcined at 500, 600, 800 or 1000 °C for 2 h by a heating rate of 10 K/min from room temperature [16].

Nanosized $Mg(OH)_2$ was prepared by hydrolysis of MgO in the nitrogen atmosphere at room temperature for 48 h (mixture of 90/10 w.% 2-propanole/water, with addition of 2 w.% of MgO). When hydroxypropyl cellulose (Klucel G, M, H or E) was used, 1 w.% of this material was added before the hydrolysis. The final product as a suspension of 0.177 M in 2-propanole was used for paper treatment [17, 18]. To estimate the effect of obtained material, analogue procedures were accomplished with a solution of bulky 0.177 M $Mg(OH)_2$ (Riedel-de Haën AG), used without further purification, as restorers use, but sonicated for 20 min before the paper treatment with $Mg(OH)_2$ at room temperature.

Cotton paper samples (Rotabilo, Carl Roth; 80 g/m^2, 100% of pure cellulose fibers) were dimensioned 120 × 15 mm. The prepared $Mg(OH)_2$ suspension was sonicated for 20 minutes and then spread on paper with a flat brush (7.5 cm) on the both sides.

One set of samples was investigated immediately and compared with other set – after accelerated aging at 90 °C for 500 h (using SNDL 67/350 furnace, ±1 °C). Artificial aging tests give us relative results, which help us to compare the stability of paper samples with and without the studied deacidification treatments. The comparison between the samples before/after deacidification and before/after aging tests was made. Cotton paper with any additives was also aged and investigated.

The pH value of the cold water extract was measured according to the international standard ISO procedure (ISO/6588-1: 2005 (E)) [19], measurements were performed using a Mettler Toledo pH meter MP 220. The alkali reserve (AR) was also determined according to the standard ISO method (ISO/10716: 1994(E)) [20].

XRD analyses of the obtained materials were performed using a Benchtop XRD MiniFlex II, Rigaku. Surface morphology of the paper samples was studied using a Hitachi TM3000 (scanning electron microscopy) and that of powders was studied using a Tecnai G2 F20 X-TWIN EDAX with an 11 MPix ORIUS SC1000B (Gatan) CCD camera (transmittance electron microscopy).

The infrared spectra were obtained by a FTIR spectrophotometer, model PerkinElmer Spectrum Frontier with ATR accessories. All spectra were recorded at a resolution of 4 cm^{-1} in an interval of 600–4000 cm^{-1}, and 20 scans were accumulated before Fourier transformation.

The sorption properties of paper were evaluated using a KSV Instruments LTD model CAM 200, the drop of 8 μL of distilled water was placed on the paper surface, and 10 pictures each 0.033 s were recorded.

The antibacterial and fungicidal efficiency of obtained nanoparticles was investigated by introducing the particles into the media containing *Saccharomyces cerevisiae* and *Escherichia coli* growth media. YEPD (yeast extract–peptone dextrose) agar plates were used for cultivation. A small amount of yeast was placed on a plate and kept in a thermostat for 48 h at 27 °C. Then the culture was placed in a flask with 40 mL deionized water and shaked. The obtained suspension was carried to Petri dishes and smoothed with McFard standard No. 2 ($2.4 \cdot 10^{-5}$ M $BaSO_4$, ~ 10^6 KSV/mL). 1 mL of *Saccharomyces cerevisiae* and 1 mL of *Escherichia coli* were placed in separate Petri dishes and covered with chilled till 60 °C grown medias of Saburo CAF (dishes for yeast) and Nutrient agar (dishes for bacteria). After the setting of media, the samples of bare MgO, nano-MgO, paper decorated with bare or nano-$Mg(OH)_2$ and non-treated paper were placed on the prepared Petri dishes. Cultivation of the samples was performed for 48 h at 27 °C. The diameters of the zones of inhibition were observed. The procedure was repeated 3 times.

RESULTS AND DISCUSSION

Magnesium oxide nanoparticles were prepared using $Mg(NO_3)_2 \cdot 6H_2O$ and $H_2C_2O_4 \cdot 2H_2O$ precursors. They were mixed separately with ethanol as a solvent and stirred to obtain a clear solution, then mixed together. The obtained gel was digested and calcined at 500, 600, 800 or 1000 °C temperature (Scheme) [16].

The X-ray diffraction (XRD) patterns recorded after the decomposition of dried gel at 500, 600, 800 and 1000 °C are shown in Fig. 1. All these patterns correspond to the periclase phase of MgO (PDF: 00-045-0946). The diffraction peaks become sharper with increasing of decomposition temperature (Fig. 1a–d). This corresponds to decreasing in the average crystallite size.

The values of an average crystalline size were determined from the Scherrer formula, and smaller crystallites of 7.2 nm were obtained at 500 °C. With increasing of the surface area the bonds in MgO are directional and there exist dangling bonds with unpaired electrons because of a reduced coordination on the outer surface of crystallites. Polar H_2O adsorbs at the surface, produces tensile stress on the underlying layer and increases the inter-ionic separation [16, 21].

The thermographs of nano-MgO, obtained at 500, 600, 800 or 1000 °C temperature, are presented in Fig. 2. Oxide

$$Mg(NO_3)_2 \cdot 6H_2O + C_2H_6O \rightarrow \leftarrow H_2C_2O_4 \cdot 2H_2O + C_2H_6O$$

$$\downarrow \text{Gel formation and digestion at 20 °C for 12 h}$$

$$MgC_2O_4 \cdot 2H_2O + 2HNO_3 + 6H_2O$$

$$\downarrow \text{Distillation at 120–125 °C}$$

$$MgC_2O_4 \cdot 2H_2O$$

$$\downarrow \text{Grinding, heating at 500, 600, 800, 100 °C}$$

$$MgO$$

Scheme. Formation of Mg nanoparticles

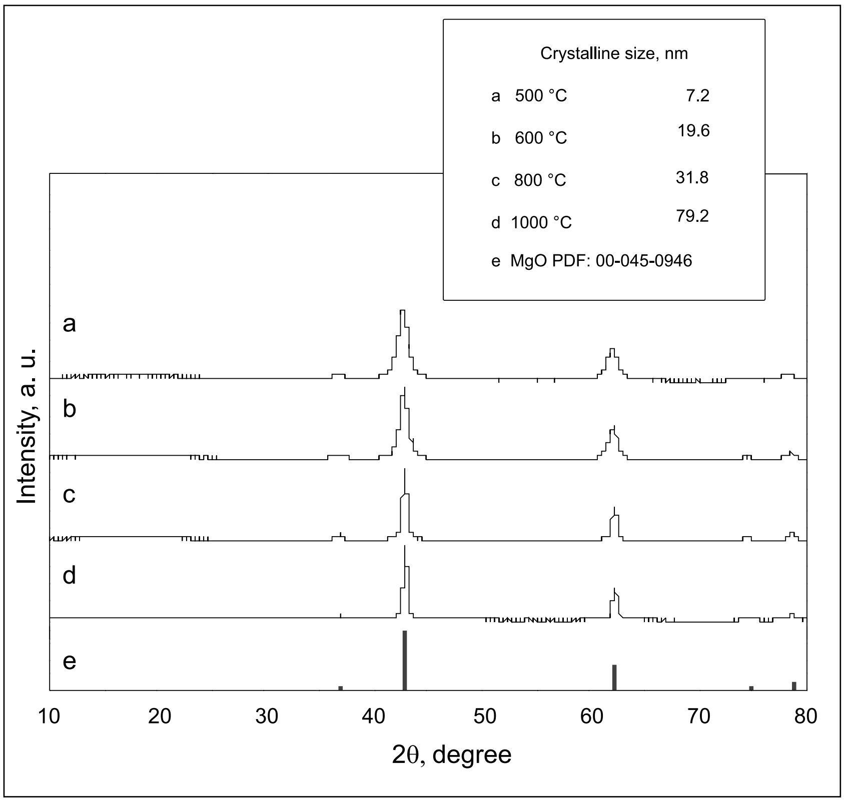

Fig. 1. The XRD pattern of MgO obtained by sol–gel method at 500 °C (a), 600 °C (b), 800 °C (c) and 1000 °C (d); PDF pattern (e)

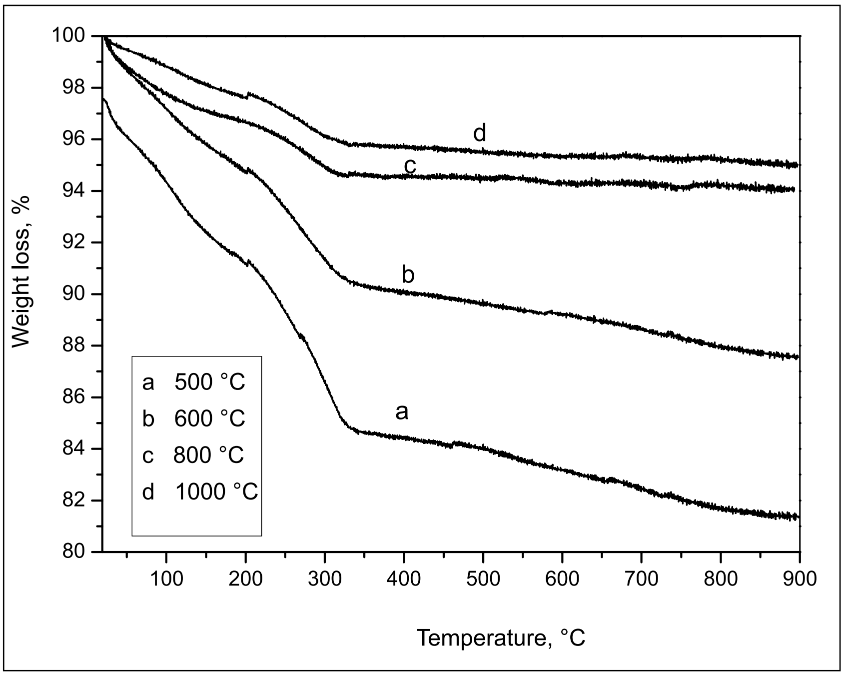

Fig. 2. TG of MgO powder obtained by sol–gel method at 500 °C (a), 600 °C (b), 800 °C (c) and 1000 °C (d)

heated at 1000 °C is the most stable – the weight loss is only 4% (Fig. 2). And the biggest weight loss of 18% was obtained after heating of material at 500 °C.

The broad absorption band in a wave number range of 3000–3600 cm^{-1} in the FTIR transmittance spectra is assigned to the –OH-stretching mode of absorbed H_2O on the surface of nano-MgO crystallites. The broadness in the spectra confirms a high degree of hydrogen bonding of water molecules among themselves and with the crystallite surface [16, 22, 23]. The amount of bonded water increased with decreasing of the particle size (Fig. 3a–d).

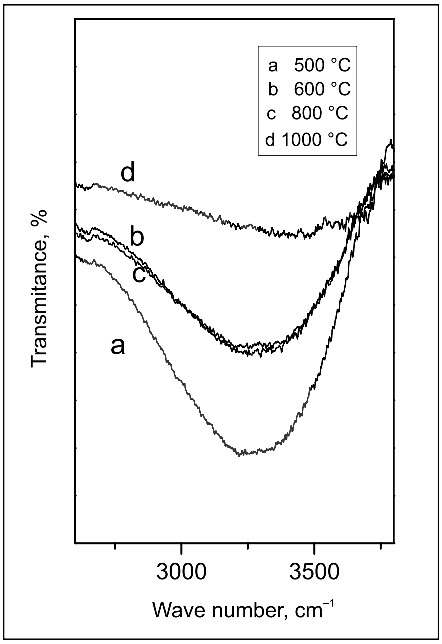

Fig. 3. FT-IR spectra of MgO powder obtained by sol–gel method at 500 °C (a), 600 °C (b), 800 °C (c) and 1000 °C (d)

The transmission electron micrographs of obtained MgO nanoparticles confirm that a higher preparation temperature leads to a bigger particle size (Fig. 4). As seen, the particles are nanosized and tend to agglomeration. The smallest particles were obtained after heating at 500 °C (Fig. 4a).

For further investigation MgO nanoparticles with a smaller particle size and a lower synthesis temperature were chosen. To decide what medium is more suitable for hydrolysis of nano-MgO, suspensions 2 w.% of MgO were prepared in 3 different media – water, ethanol and 2-propanole. Mixing was performed in the nitrogen atmosphere. After 1 h of sonication, distribution of a particle size was obtained using a Zeta sizer (Fig. 5).

It can be seen that in all cases the particles tend to agglomeration. The smallest average particle size was obtained when the suspension was prepared in 2-propanole 398 nm, so hydrolysis of MgO was decided to perform in this media. 2 w.%

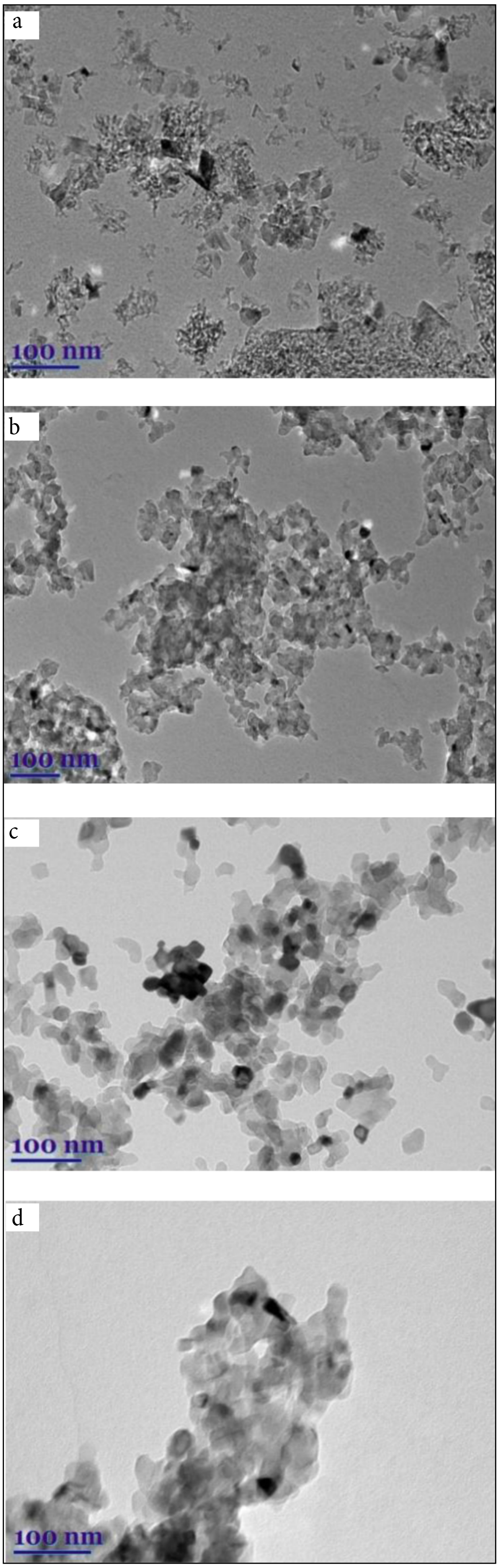

Fig. 4. The transmission electron micrograph of MgO powder obtained by sol–gel method at 500 °C (a), 600 °C (b), 800 °C (c) and 1000 °C (d)

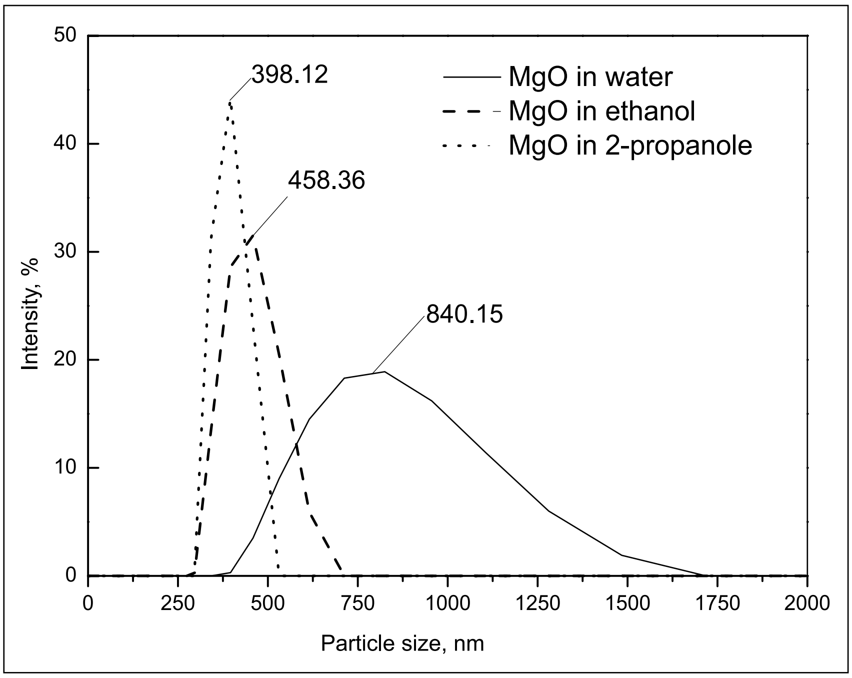

Fig. 5. The size distribution of MgO nanoparticles in water (a), ethanol (b) and 2-propanole (c)

of MgO in 2-propanole, with addition of 1–10 w.% of water, were mixed at room temperature for 48 hours in the nitrogen atmosphere and distilled in 90 °C for 24 h. The XRD pattern, recorded after the synthesis, shows that pure nano-$Mg(OH)_2$ was obtained when 10 w.% of water was added (Fig. 6). This pattern corresponds to the brucite phase of $Mg(OH)_2$ (PDF: 00-044-1482).

The transmission electron micrograph of $Mg(OH)_2$ (Fig. 7) shows that the particles tend to agglomeration and form needle-like morphology. The average particle size is 20–30 nm – the obtained $Mg(OH)_2$ is nano-sized.

In general, nanoparticles tend to stabilize themselves either by sorption of molecules from surroundings or by lowering the surface area and high surface energy through coagulation or agglomeration. In order to avoid this, nanoparticles have to be stabilized. One of the most popular stabilization mechanisms is based on the steric repulsion between molecules adsorbed on neighbouring particles. Due to geometric constraints around nanoparticles, large molecules provide a particularly effective stabilization and keep the approaching nanoparticles apart [24]. As reported, nanoparticles can be incorporated into a polymer matrix of hydroxypropyl cellulose [25–28]. This derivative of cellulose contains a fraction of the original cellulose structure, but it is less crystalline and more amorphous than cellulose. The better-known brand of this polymer is Klucel [29].

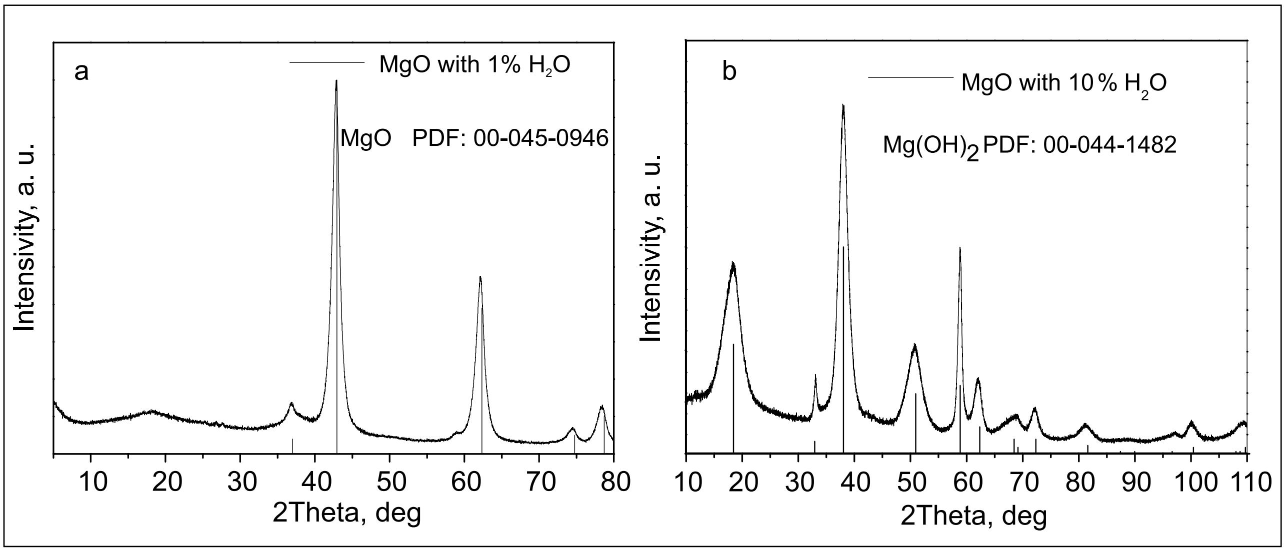

Fig. 6. The XRD pattern of nano-MgO hydrolysis products: with 1 w.% of water (a), with 10 w.% of water (b)

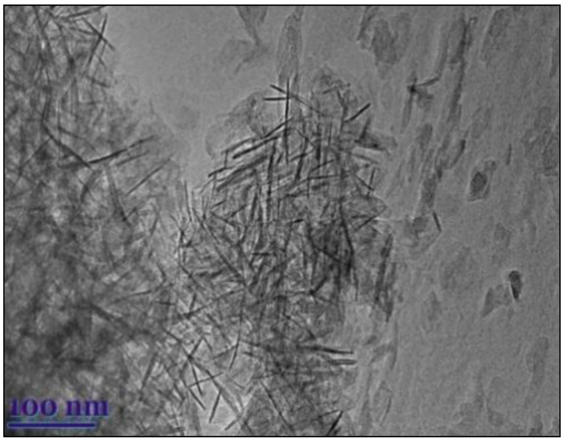

Fig. 7. The transmission electron micrograph of $Mg(OH)_2$ powder

Restorers widely use this polymer for wood and paper conservation. Depending on the molecular weight of polymer and the concentration of solution it can be used as glue, or can be applied directly to the paper surface to increase its mechanical strength. As a solvent, water, ethanol or 2-propanole can be used. There are several available Klucel types, so to determine which will be more suitable for paper treatment, a set of samples were prepared. Hydrolysis of MgO was performed with addition of 1 w.% of each type of Klucel H, M, G or E. All samples were spread on separate paper sheets and dried at room temperature for 24 h. pH of the paper cold extract was determined (Table 1).

Table 1. **pH of paper treated with $Mg(OH)_2$ nanoparticles with addition of Klucel G, M, H and E**

Klucel (commercial classification G, M, H and E) and concentration, w.%	Viscosity of Klucel, dPa·s	pH of paper
Klucel H, 1 w.%	0.88	9.27
Klucel M, 1 w.%	0.60	8.92
Klucel G, 1 w.%	0.62	9.43
Klucel E, 1 w.%	0.20	9.50

Klucel E was chosen for further paper treatment – the pH value was the highest (9.50). This polymer has the lowest molecular weight (80000 Mw^2), viscosity and with low concentration of 1 w.% will not form a glazy layer on the paper surface.

Sorption capabilities of moisture, water and dye are very important properties of paper. The molecular and intermolecular structure of fibers influences sorption properties of paper. Different additives can also affect paper surface morphology and other properties. The sorption properties of paper can be evaluated through contact angle measurements – an angle of a water droplet deposited onto its surface. If there is no swelling of water and a contact angle is higher than 90°, material can be defined as hydrophobic [30, 31].

The contact angle of a water drop, placed on paper and paper with nano-$Mg(OH)_2$ and Klucel E additive, was measured. In both cases the water drop swelled faster than in 1 s (contact angle did not comprise, $\theta = 0°$). In Fig. 8 pictures of a water drop, placed on the paper surface, can be observed. Klucel E did not change paper sorption properties.

Nanoparticles ranging in dimensions of 1–100 nm have different physio-chemical, optical and biological properties than their bulk equivalent. They are also being explored and investigated as potential antimicrobials. The small size and high surface to volume ratio enhance their interaction with the microbes. The nanoparticles can interact with bacteria's DNA, resulting in the inactivation of DNA replication or inhibition of enzyme functions. It is also possible that nanomaterial can bind with cell membranes, decreasing the osmotic stability of the cell, and damage bacteria [32].

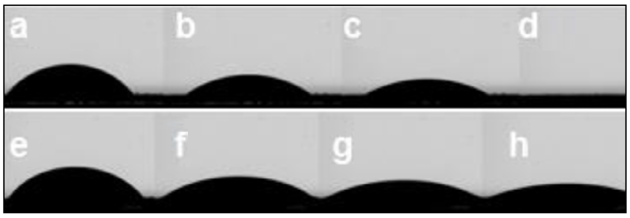

Fig. 8. Pictures, captured each 0.033 s (till 0.132 s) after a drop placed on untreated paper (a–d); paper treated with nano-$Mg(OH)_2$ with addition of Klucel E (e–h)

The antibacterial and fungicidal efficiency of MgO and $Mg(OH)_2$ nanoparticles was investigated by introducing them into the following media:

* *Escherichia coli (E. coli)* is a Gram-negative, anaerobic, rod-shaped bacterium of the genus *Escherichia* that is commonly found in the lower intestine of warm-blooded organisms. A Gram-negative bacterium membrane is more complex than that of a Gram-positive one, it consists of lipopolysaccharides, which increase the negative charge of cell membranes and are essential for structural integrity and viability of the bacteria [33].

* *Saccharomyces cerevisiae* (*S. cerevisiae)* is a single-celled eukaryote that is frequently used in scientific research. *S. cerevisiae* is a popular model organism because many yeast proteins are similar in their sequence and function to those found in other organisms [34].

Investigation was performed using YEPD agar plates for cultivation of bacteria and yeast organisms. After 48 h at 27 °C, the diameters of the zones of inhibition were measured.

In the first set of pictures (Fig. 9), the results of bare and nanosized MgO investigation are presented.

The zone of the inhibition diameter against *S. cerevisiae* was observed. The fungicidal zone was 6.0–10.0 mm for nano-MgO and 4.0–5.0 for bare MgO. The size of the zone of inhibition indicates the degree of sensitivity of bacteria to a drug. In general, a bigger area of bacteria-free media

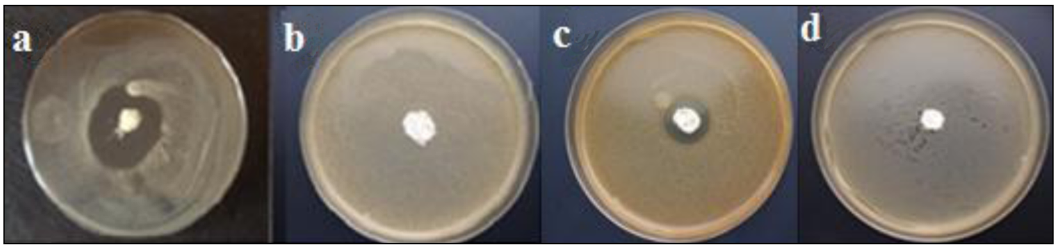

Fig. 9. Pictures of Petri dishes with MgO nanoparticles in *Saccharomyces cerevisiae* culture (a), *Escherichia coli* culture (b) and bare MgO in *Saccharomyces cerevisiae* culture (c), *Escherichia coli* culture (d)

surrounding means that the bacteria are more sensitive to the drug the disk contains. So, nanosized MgO has twice stronger fungicidal properties than bare MgO. Bactericidal properties for MgO and nano-MgO were not observed (Fig. 9b, d).

On the other hand, paper decorated with nano-$Mg(OH)_2$ has both – fungicidal and bactericidal properties (Fig. 10a, d). The inhibition zone for *S. cerevisiae* was 8.0–9.0 mm, and for *E. coli* it was 3.0–3.5 mm.

Fungicidal and bactericidal properties were observed only when magnesium hydroxide was nanosized. The inhibition zones around the non-treated paper and the paper decorated with bulky $Mg(OH)_2$ were not observed (Fig. 10b, e).

The synthesized magnesium nano-hydroxide will provide protection from biodegradation against some of bacteria and fungi (*Escherichia coli, Saccharomyces cerevisiae*). But the main causes of paper deterioration are hydrolysis of glycosidic bonds and oxidation of glucopyranose rings. So, to evaluate the effect of obtained mixtures ($Mg(OH)_2$, nano-$Mg(OH)_2$ and nano-$Mg(OH)_2$ with Klucel E), they were spread on the paper surface and the acquired samples were artificially aged for 500 h at 90 °C temperature in air.

To estimate the influence of $Mg(OH)_2$ on paper acidity, the samples were investigated before and after artificial aging. The pH and alkali reserve (AR) values of the paper samples are presented in Table 2.

In can be seen that the pH of paper decreases after aging. The source of acidity in paper is products of cellulose hydrolysis [35]. Bulky $Mg(OH)_2$ increases pH of paper to 8.15 and after accelerated aging the value is still enough high (8.01).

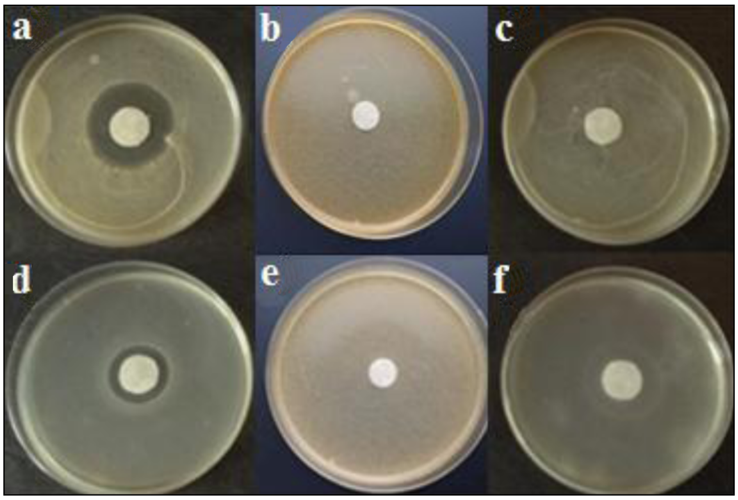

Fig. 10. Pictures of Petri dishes of *Saccharomyces cerevisiae* culture with paper decorated

Table 2. **pH and AR results of pure paper and paper treated with bulky $Mg(OH)_2$, nano-$Mg(OH)_2$, nano-$Mg(OH)_2$ with Klucel E before and after artificial aging for 500 h at 90 °C temperature in air**

Samples	pH		Alkali reserve (AR), OH– mol/kg	
	Non-aged	Artificially aged	Non-aged	Artificially aged
Paper	6.55	5.99	0.11	0.04
Paper with bulky $Mg(OH)_2$	8.15	8.01	0.38	0.25
Paper with nano-$Mg(OH)_2$	9.70	9.61	0.44	0.28
Paper with nano-$(MgOH)_2$ with Klucel E	10.01	9.99	0.52	0.38

The provided alkali reserve (0.38) slightly decreases till 0.25 relative units. The conditional dimension of alkali reserve is OH^- mol/kg and 0.2 of it corresponds to 1% of $CaCO_3$ in substance. The suitable AR value for the alkalized paper concentration is about 2–3% [20].

Nano-sized materials have a better link to the paper surface, they cover paper with a higher efficiency and can penetrate into the cellulose fibers net in all thickness of paper [9]. The pH of paper treated with nano-$Mg(OH)_2$ is 9.70, it is ~16% higher than when bare hydroxide was used, the AR value is 13.63% higher. Addition of Klucel E increases the link of alkali material to paper, so pH = 10 and AR value is 0.52, that correspond to 2.6% of alkali material, after aging it is 1.9%.

During the aging of paper not only pH and AR value changes, the surface of paper becomes damaged and cracked (Fig. 11a, b).

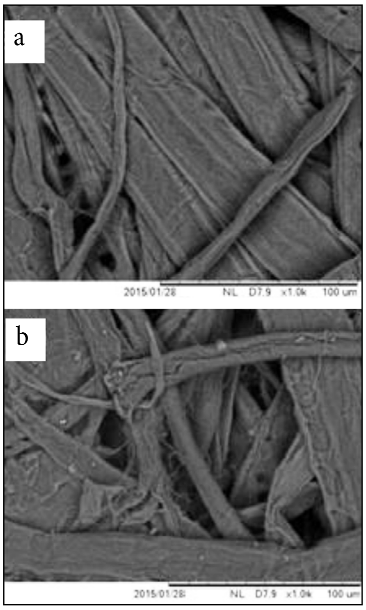

Fig. 11. The SEM pictures of paper surface: non aged (a), aged for 500 h (b)

The SEM pictures of paper treated with nano-$Mg(OH)_2$ are presented in Fig. 12. It can be seen that the nanoparticles cover the paper surface with more efficiency when Klucel E is used for nanoparticles stabilization. This confirms that nanoparticles will more likely link to the paper surface when a stabilizer is used. It can also be seen that highly alkaline material did not damage the paper surface.

CONCLUSIONS

Pure magnesium oxide nanoparticles (7.2 nm) were prepared using the sol–gel method. The decomposition temperature used was 500 °C. Magnesium oxide was hydrolyzed in the nitrogen atmosphere and pure nano-$Mg(OH)_2$ was obtained. The transmission electron micrograph of $Mg(OH)_2$ shows that the particles are nanosized (20–30 nm), tend to agglomeration and form needle-like morphology.

The obtained magnesium hydroxide is suitable for paper conservation. After the treatment of model paper samples, pH and alkali reserve values were high and with addition of Klucel E increased even more. Hidroxypropyl cellulose Klucel E not only stabilizes the suspension of nano-$Mg(OH)_2$ but also increases the link of nano particles to the paper surface. Alkali material covers paper with higher efficiency. In addition, nano-$Mg(OH)_2$ provides protection from biodegradation against some of bacteria and fungi (*Escherichia coli, Saccharomyces cerevisiae*).

ACKNOWLEDGEMENTS

The study was funded from the European Community's social foundation through the project under Grant Agreement No. VP1-.1-SMM-08-K-01-004/KS-120000-1756

Received 11 May 2016
Accepted 15 June 2016

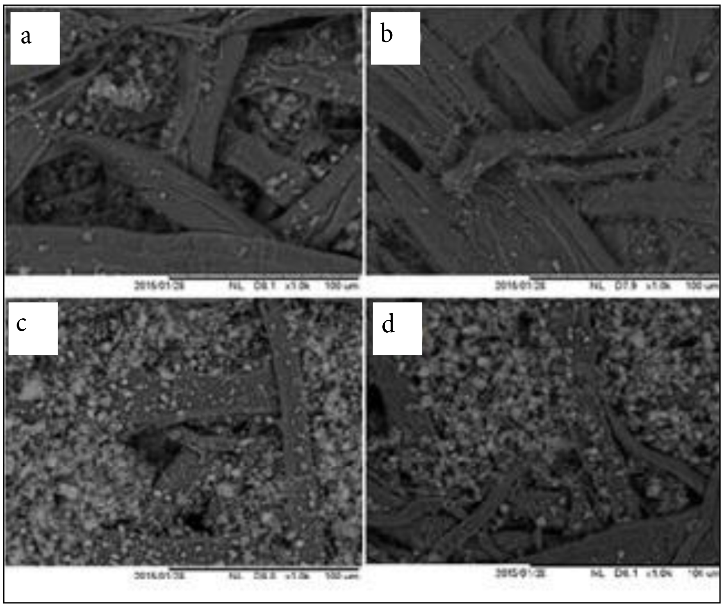

Fig. 12. SEM pictures of the paper surface with $Mg(OH)_2$ nanoparticles: non-aged (a); aged for 500 h (b); with addition of Klucel E, non-aged (c); with addition of Klucel E, aged for 500 h (d)

References

1. B. L. Mordike, K. U. Kainer, Volkswagenwerk, *Magnesium Alloys and Their Applications*, 714 p., Werkstoff-Informationsgesellschaft, Frankfurt (1998).
2. W. Jiang, X. Hua, Q. Han, et al., *Powder Technol.*, **191(3)**, 227 (2009).
3. C. Y. Tai, C. T. Tai, M. H. Chang, et al., *Ind. Eng. Chem. Res.*, **46(17)**, 5536 (2007).
4. C. Henrist, J. P. Mathieu, C. Vogels, et al., *J. Cryst. Growth*, **249(1–2)**, 321 (2003).
5. M. A. P. C. De Feber, J. B. G. A. Havermans, P. Defize, *Restaurator*, **21(4)**, 204 (2000).
6. O. Darcanova, A. Beganskienė, A. Kareiva, *Chemija*, **26(1)**, 25 (2015).
7. G. Poggi, R. Giorgi, N. Toccafondi, et al., *Langmuir*, **26(24)**, 19084 (2010).
8. C. Rodriguez-Navarro, A. Suzuki, E. Ruiz-Agudo, *Langmuir*, **29(36)**, 11457 (2013).
9. G. Poggi, N. Toccafondi, L. N. Melita, et al., *Appl. Phys. A*, **114(3)**, 685 (2014).
10. R. Giorgi, M. Ambrosi, N. Toccafondi, et al., *Chem. Eur. J.*, **16(31)**, 9374 (2010).
11. R. Giorgi, D. Chelazzi, P. Baglioni, *Langmuir*, **21(23)**, 10743 (2005).
12. G. L. Zou, W. X. Chen, R. Liu, et al., *Mater. Chem. Phys.*, **107(1)**, 85 (2008).
13. F. Meshkani, M. Rezaei, *Powder Technol.*, **196(1)**, 85 (2009).
14. A. Pilarska, M. Wysokowski, E. Markiewicz, et al. *Powder Technol.*, **235**, 148 (2013).
15. A. C. Pierre, *Introduction to Sol-Gel Processing*, The Kluwer International Series in Sol-Gel Processing, Boston: Kluwer Academic Publishers (1998).
16. A. Kumar, J. Kumar, *J. Phys. Chem. Solids*, **69(11)**, 2764 (2008).
17. M. Ghiasi, A. Malekzadeh., *Cryst. Res. Technol.*, **47(4)**, 471 (2012).
18. H. G. Shin, H. Kim, Y. N. Kim, et al., *Curr. Appl. Phys.*, **9**, S276 (2009).
19. ISO/6588-1: 2005 (E), Paper, board and pulps – Determination of pH of aqueous extracts – Part 1: Cold extraction.
20. ISO/10716: 1994 (E), Paper and board – Determination of alkali reserve.
21. A. K. Haghi, Ajesh K. Zachariah, N. Kalariakkal, *Nanomaterials: Synthesis, Characterization, and Application*, Advances in Nanoscience and Nanotechnology, Vol. 3, CRC Press Taylor & Francis Group (2013).
22. S. Bordiga, L. Regli, C. Lamberti, et al., *J. Phys. Chem. B*, **109(16)**, 7724 (2005).
23. J. Heidberg, B. Redlich, D. Wetter, *Ber. Bunsenges. Phys. Chem.*, **99(11)**, 1333 (1995).
24. T. M. Alexander Kraynov, *Applications of Liquids in Science and Technology*, **405**, 235 (2011).
25. M. R. de Moura, L. H. C. Mattoso, V. Zucolotto, *J. Food Eng.*, **109(3)**, 520 (2012).
26. E. S. Abdel-Halim, S. S. Al-Deyab, *Carbohydr. Polym.*, **86(4)**, 1615 (2011).
27. G. Cavallaro, G. Lazzara, S. Milioto, *Soft Matter*, **8(13)**, 3627 (2012).
28. S. Lu, U. Sohling, T. Krajewski, et al. *J. Mater. Sci. Lett.*, **17(24)**, 2071 (1998).
29. R. L. Feller, M. Wilt, *Evaluation of Cellulose Ethers for Conservation. Research in Conservation*, 161 p., Getty Conservation Institute, Marina del Rey, California, USA (1990).
30. Z. Persin, K. Stana-Kleinschek, T. Kreze, *Croat. Chem. Acta*, **75(1)**, 271 (2002).
31. A. G. Cunha, C. Freire, A. Silvestre, et al., *J. Colloid Interface Sci.*, **344(2)**, 588 (2010).
32. J. B. Ravishankar Rai, *Science Against Microbial Pathogens: Communicating Current Research and Technological Advances*, 197 (2011).
33. M. J. Hajipour, K. M. Fromm, A. A. Ashkarran, et al., *Trends Biotechnol.*, **30(10)**, 499 (2012).
34. M. Manikandan, H. F. Wu, *J. Nanopart. Res.*, 15(7), (2013).
35. S. Sequeira, C. Casanova, E. J. Cabrita, *J. Cult. Herit.*, **7(4)**, 264 (2006).

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MAGNIO OKSIDO NANODALELIŲ SINTEZĖ ZOLIŲ-GELIŲ METODU, HIDROLIZĖ IR PRITAIKYMAS POPIERIAUS NURŪGŠTINIMO PROCEDŪROMS

Santrauka

Magnio oksido dalelės pagamintos naudojant zolių-gelių metodą. Pirmtakai – magnio nitratas ir oksalo rūgštis etanolyje buvo sumaišyti tarpusavyje ir gautas gelis kaitintas 500, 600, 800 ir 1 000 °C temperatūroje. Mažiausios dalelės (7,2 nm), gautos 500 °C temperatūroje, buvo hidrolizuotos 2-propanolyje pridedant 10 sv. % vandens. Mišiniui stabilizuoti pridėta hidroksipropyl celiuliozė (Kliucel E). Gautos magnio hidroksido (20–30 nm) ir oksido nanodalelės buvo ištirtos, užrašyti medžiagų rentgeno difrakcijos spektrai, išanalizuotos peršviečiamosios elektronų mikroskopijos nuotraukos bei nustatytas miltelių baktericidinis ir fungicidinis poveikis prieš *E. coli* ir *S. cerevisiae*.

Magnio hidroksido nanodalelės pritaikytos popieriaus šarminimui. Gauti mėginiai termiškai sendinti ir ištirti skenuojančiu elektronų mikroskopu, nustatytas popieriaus rūgštingumas, šarminė atsarga. Rezultatai palyginti su gautais tiriant nesendintus mėginius ir mėginius, paveiktus komerciniu magnio hidroksidu. Po terminio sendinimo stebimas visų mėginių pH ir šarminės atsargos verčių sumažėjimas, tačiau naudojant nano-$Mg(OH)_2$ popierius yra labiau apsaugotas.