ULTRASOUND AND MICROWAVE ASSISTED HYDROLYSIS OF NANOSTRUCTURED MAGNESIUM OXIDE FOR IMPREGNATION OF POLYESTER FIBRE FILTERS

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Several unique properties such as photocatalytic activity, high electrical permittivity, and non-toxicity make nanostructured metal oxides exceptional materials. As a result, they find widespread applications in diverse fields such as the environment, semiconductors, optics, electronics, electricity, antiseptics, pathogens, and catalysts. This paper focused on developing and examining the morphological as well as structural properties of magnesium oxide nanoparticles for impregnation onto polyester fibre filters. To obtain these nanoparticles, the ultrasound and microwave-assisted hydrolysis method with magnesium chloride as a precursor was used. Using XRD, SEM, and ATR FT-IR, the structural and morphological properties of MgO nanoparticles have been established.

Keywords: characterization, elaboration, impregnation, magnesium oxide.

1. Introduction

Nanotechnology refers to the processing and engineering of materials at the nanoscale, typically between one and one hundred nanometers.

This field plays a vital role in advancing research across various sectors, holding immense potential for breakthroughs. Nanomaterials are constructed from components with sizes between one and one hundred nanometers. These components, known as nanoparticles, can have a structural size ranging from one to one thousand nanometers [1]. Notably, the shape of these nanoparticles can vary, including cones, spirals, and plates.

Compared to their larger counterparts, nanoparticles exhibit unique physical properties like enhanced stability and exceptional mechanical strength. These

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properties contribute to the numerous advantages and widespread applications of nanoparticles around the world [2].

Over the past decade, scientists have become increasingly fascinated by the potential of nanostructured oxides with various shapes (morphologies) [3]. Beyond the well-known nanoparticles and thin films, there is a growing interest in complex structures such as nanosheets, nanocubes, nanorods, and even nanoflowers. Research is actively exploring these unique forms in various oxide materials. This focus on morphology is not just about novelty, but also about discovering how these specific shapes offer advantages over conventional nanoparticles [2]. Due to their important physico-chemical properties, nanostructured oxides such as CuO [4], TiO_2 [5], CeO_2 [6], ZnO [7] [8], SiO_2 [9], Fe_3O_4 [10], Fe_2O_3 [11], and Al_2O_3 [12] offer a wide range of advantages for diverse areas of investigation including catalysis, energy storage, drug delivery, optoelectronics, etc.

In a similar way, nanostructured magnesium oxide has significant potential for application in environmental research, as well as numerous other applications in agriculture, catalysis of chemical reactions, dye removal and lithium batteries [13]. The unique properties of MgO nanoparticles, such as: stability, magnetizability, crystallinity, absorbability, electrical and thermal conductivity, well-defined composition, large surface area and reactivity simple and cost-effective preparation methods (even eco-friendly synthesis), contribute to their diverse applications. These remarkable characteristics place MgO nanoparticles (MgO NPs) at the forefront of environmental research.

Bottom-up procedures, including chemical techniques such as sol-gel [14], solvo-/hydrothermal [15], co-precipitation [16], and so-called green synthesis, can be used to produce MgO nanoparticles.

This paper describes the synthesis of nanostructured magnesium oxide (MgO) using ultrasound- and microwave-assisted hydrolysis. The impregnation process of polyester fiber filters is also discussed. Polyester filters are widely used in various applications due to their excellent thermal stability, mechanical strength, and chemical resistance. However, the performance of these filters can be improved by impregnation with magnesium oxide (MgO) nanoparticles. Properties of polyester filters to be enhanced by impregnation with magnesium oxide nanoparticles include filtration efficiency, antimicrobial activity, durability, and chemical resistance. Nanostructured magnesium oxide is known for its antimicrobial properties, and the impregnation of PES (polyester) fibre can reduce microbial load and prevent the growth of bacteria and fungi on the filter surface. In addition, the integration of nanostructured MgO is likely to increase the adsorption capacity for various pollutants, thus increasing the filtration efficiency of PES filters.

In the elaboration of magnesium oxide nanoparticles, magnesium chloride ($MgCl_2 \cdot 6H_2O$) was used as a precursor and sodium hydroxide (NaOH) as a precipitating agent.

The synthesis was performed using two distinct concentrations of $MgCl_2 .6H_2O$ (0.5 M and 1 M) and a constant concentration of NaOH. The MgO powders obtained to be used for impregnation of PES (polyester) filters were morphologically and structurally characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FT-IR).

2. Experimental

2.1. Reagents and materials

All the chemicals ($MgCl_2 \cdot 6H_2O$, PEG 4000, and NaOH) used are of reactive quality and are used without any supplementary purification. The precursor $MgCl_2 \cdot 6H_2O$ was used to prepare the nanostructured magnesium oxide and NaOH was added as precipitation agent. Demineralized water was used in the preparation of the solutions.

PES filters are impregnated with powders prepared by microwave-assisted hydrolytic synthesis.

2.2. Synthesis of Magnesium Oxide Nanoparticles

An ultrasound- and microwave-assisted hydrolytic procedure employing $MgCl_2 \cdot 6H_2O$ as a precursor and a sodium hydroxide solution as a precipitating agent was used to create MgO nanoparticles. To begin, two solutions of magnesium chloride with different concentrations were prepared. This was done by dissolving 10.165 grams of $MgCl_2 \cdot 6H_2O$ in 100 ml of deionized water to create a solution with a concentration of 0.5 mol/L. Similarly, 20.33 grams of $MgCl_2 \cdot 6H_2O$ were dissolved in 100 milliliters of deionized water to prepare a solution with a concentration of 1 mol/L. In order to precipitate these solutions, a 1M sodium hydroxide solution was progressively added with magnetic stirring at 500 rpm at a pH of 11. To keep the solutions stable, 0.2 g of PEG 4000 was added. Berzelius beakers containing the solutions obtained were immersed one by one in a CD-4800 ultrasonic water bath. The sonication was performed for 10 minutes at a frequency of 42 Hz and a power of 60 W to disperse the particles. The precipitates were then placed in a household oven (Samsung) for 5 minutes at 850 W and 2 minutes at 1000 W. The temperatures of the solutions reached 85°C and 95°C, respectively, after microwave irradiation. The precipitates were washed seven times with deionized water and ethyl alcohol to eliminate impurities from the synthesis. The powders were dried at 120°C for 1 hour before being calcined at 550°C for 2 hours to produce the final nanoparticles.

Table 1 shows the experimental parameters for the elaboration of magnesium oxide nanoparticles by microwave and ultrasound assisted hydrolytic synthesis.

Table1

The parameters in microwave and ultrasound assisted hydrolytic synthesis

Sample code	Precursor concentration	Hydrolysis agent/ Ph	Heating Time (min)/Power (W) using MW	Frequency (Hz) Power (W) using US	Calcination temperature (°C)/time (h)
S1	0.5M $MgCl_2.6H_2O$	1M NaOH/11	5 min /850 W	42 Hz/60 W	550°C/2h
S2			2min/1000 W		
S3	1M $MgCl_2.6H_2O$		5 min /850 W		
S4			2min/ 1000W		

2.3. Characterization of Magnesium Oxide Nanoparticles

Various characterization techniques were used to perform morpho-structural investigations on calcined magnesium oxide particles. Among these, X-ray powder diffraction in Bragg-Brentano geometry was carried out using CuK radiation (λ=0.154 nm) at 45 kV, and 40 mA and a one-dimensional D/teX Ultra detector with graphite monochromator. Diffractograms were acquired in the 2θ range from 15° to 100° using the RIGAKU ULTIMA IV diffractometer. A Tensor 27 spectrometer was also used to acquire Fourier transform infrared spectroscopy (FTIR) spectra in the range 350–4.000 cm^{-1}, with a resolution of 4 cm^{-1}. A HITACHI SU5000 scanning electron microscope (SEM) was used to examine particles morphology.

2.4. Impregnation of filters with powders S1, S2, S3, and S4

Working solutions are prepared using the prepared powders S1, S2, S3, and S4 (50 mg MgO dispersed in 50 ml deionized water). The prepared solutions are subjected to magnetic stirring for 10 minutes. Then, 0.1 g PEG (polyethylene glycol) is added to each prepared dispersion and homogenized by magnetic stirring at 500 rpm for 10 minutes. One piece of PES filter is introduced into each of the four Berzelius beakers with the working solutions and heated on an electric hotplate at ~60°C for 20 minutes. After this step, the filters are removed from the solutions and placed in an oven at 115°C for drying.

3. Results and discussion

3.1. XRD characterization

An examination was conducted on the crystalline phase and nanoparticle structure synthesized by MgO using X-ray diffraction techniques.

Fig.1 illustrates the overlay of XRD spectra of MgO nanoparticles. The two highest peaks in the XRD diffractogram are at 2θ = 42.764° and 62.016°,

corroborated by four smaller peaks at 2θ = 36.76°, 74.51°, 78.33°, 93.83°. These phase peaks correspond to MgO, having a cubic structure and a space group of 225: Fm-3m, according to ICCD (PDF4 + 2023 DB: 9006750 card).

From the overlay of spectra shown in Fig.1, the absence of peaks originating from Mg or other impurities can be observed, indicating a high purity of the produced MgO nanoparticles.

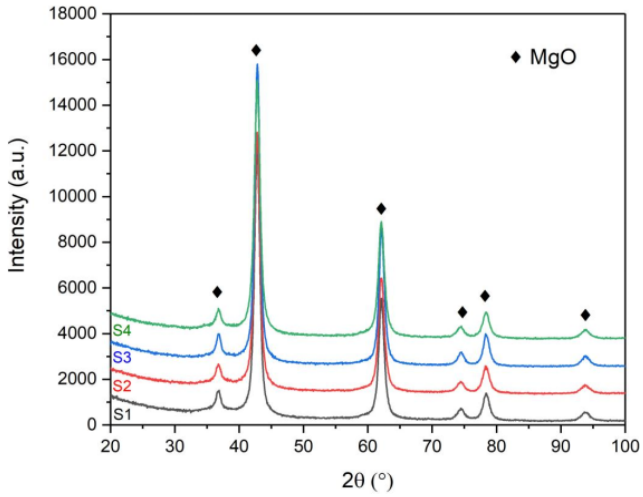

Fig. 1. X-ray diffraction (XRD) patterns of S1, S2, S3, and S4

Compared to the results obtained by C. Karthika *et all* [17] by the conventional method, which yielded crystallites with sizes of 29 nm, the ultrasound and microwave-assisted synthesis led to significantly smaller crystallite sizes. This result is attributed to the ultrasonic waves and rapid and uniform heating that limit crystal growth. The Williamson-Hall method was used to determine the mean crystallite sizes D (nm) for all the four samples. (Table 2).

Table 2

Average crystallite size by Williamson-Hall method for MgO

Sample code	Precursor concentration /Synthesis parameters (W/ min)		Crystallite size (nm)
S1	0.5 M $MgCl_2.6H_2O$	850 W/ 5 min	7.18± 0.2
S2		1000 W/ 2 min	6.83± 0.2
S3	1 M $MgCl_2.6H_2O$	850 W/ 5 min	7.33± 0.3
S4		1000 W/ 2 min	6.35± 0.3

3.2. SEM characterization

Fig. 2 shows (SEM) micrographs of MgO samples calcined for 2 hours at 550°C in air.

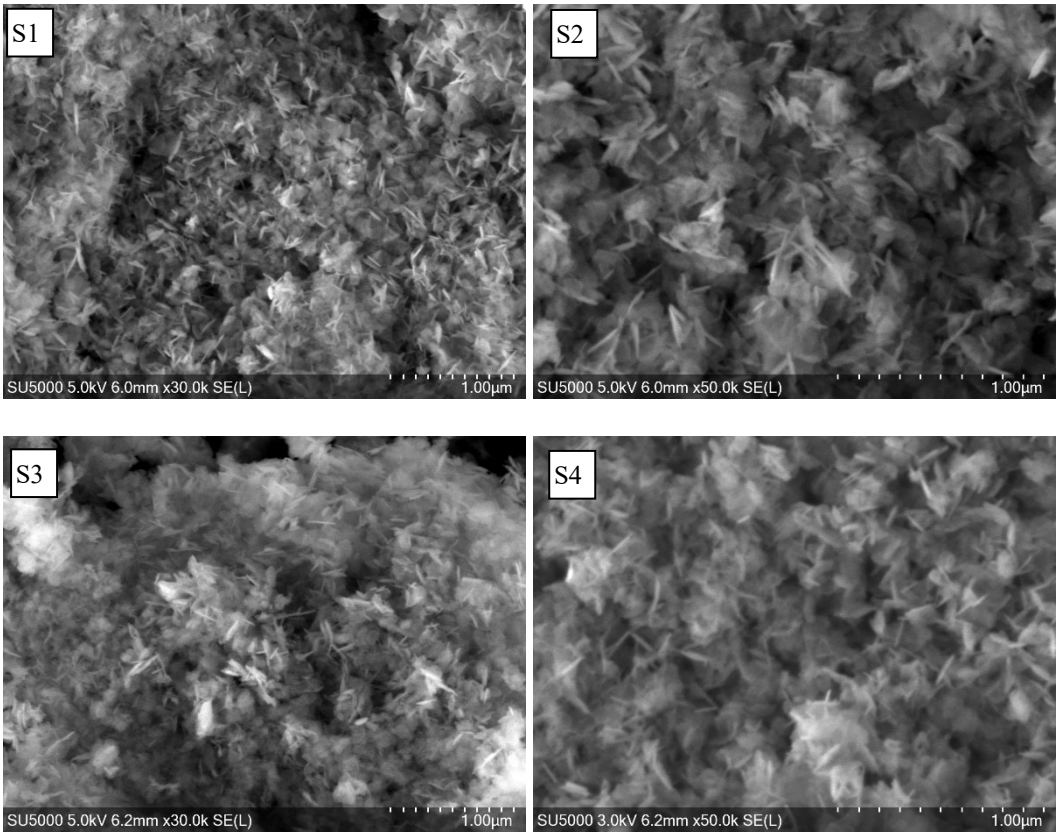

Fig. 2. SEM micrographs of MgO calcined at 550ºC/ 2h for S1, S2, S3, and S4

The scanning electron microscope, Hitachi SU5000, was used to analyze the morphology of magnesium oxide elaborated by microwave and ultrasound assisted hydrolytic synthesis. The image analysis software - ImageJ was used to determine the average particle size. From the analysis of the SEM micrographs, the MgO nanoparticles have an irregular shape (nanosheet), with sizes ranging from 127 nm to 175 for the S1 powder, with an average particle size of 175 nm. Measurements with ImageJ software were also performed for the other powders and the results are shown in the Table 3.

Table 3

Average particle shape and size for samples S1, S2, S3 and S4

Sample code	Precursor concentration	Hydrolysis agent/ Ph	Heating Time (min)/Power (W)	Calcination temperature (°C)/time (h)	Shape	Size (nm)
S1	0.5M $MgCl_2.6H_2O$	NaOH/ 11	5 min /850 W	550 °C / 2 h	nanosheet	175 ± 15.13
S2			2min/1000 W			184 ± 16.71

S3	1M $MgCl_2.6H_2O$		5 min /850 W			190 ± 45.73
S4			2min/ 1000W			197 ± 46.67

3.3. AFTIR characterization

Attenuated Total Reflectance Fourier Transform Infrared (ATR-FTIR) spectra is used to identify the chemical composition of samples.

The ATR-FTIR spectrum from fig.3 shows the intensity of infrared light absorbed by the samples elaborated at 850W and 1000W starting from 0,5M precursor solution. The x-axis of the graph represents the wavelength of the infrared light corresponding to 378 cm^{-1} for sample S1 and 369 cm^{-1} for sample S2. The spectrum typically has one peak that corresponds to a Mg-O functional group, according to the commercial standard MgO.

The ATR-FTIR spectrum from fig. 4 shows the intensity of infrared light absorbed by the samples elaborated at 850W and 1000W starting from 1 M precursor solution. The x-axis of the graph represents the wavelength of the infrared light corresponding to 372 cm^{-1} for samples S3 and S4. The spectrum has typically had one peak which corresponds to a Mg-O functional group according to etalon MgO commercial.

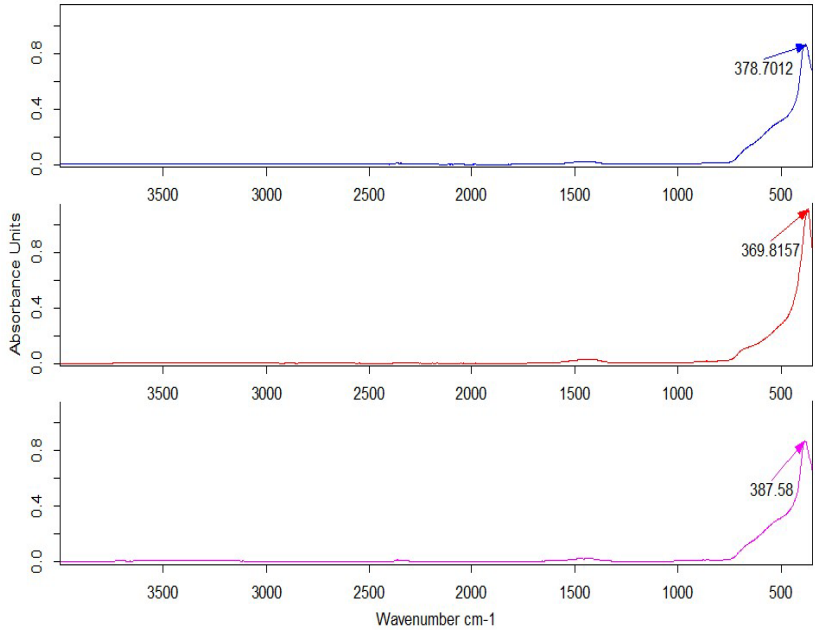

Fig. 3. ATR-FTIR spectra of S1 (blue) and S2 samples (red) and MgO etalon (magenta)

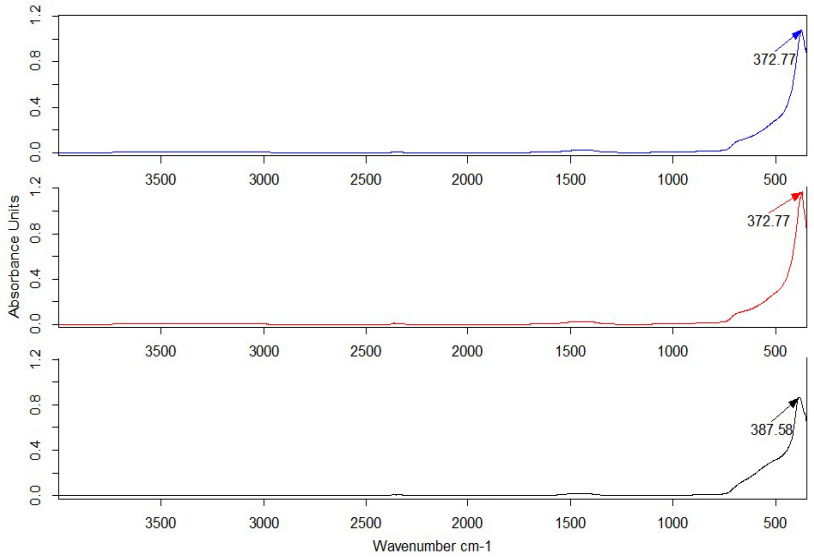

Fig. 4. ATR-FTIR spectra of S3 (blue) and S4 samples (red) and MgO etalon (black)

No contaminants are presented in the spectra. Due to size variations of MgO nanoparticles, peaks in the spectrum of all samples are broader compared to commercial MgO (etalon). Fig. 5 illustrates a representative scanning electron microscopy (SEM) micrograph of the PES filter impregnated with nanostructured magnesium oxide nanoparticles.

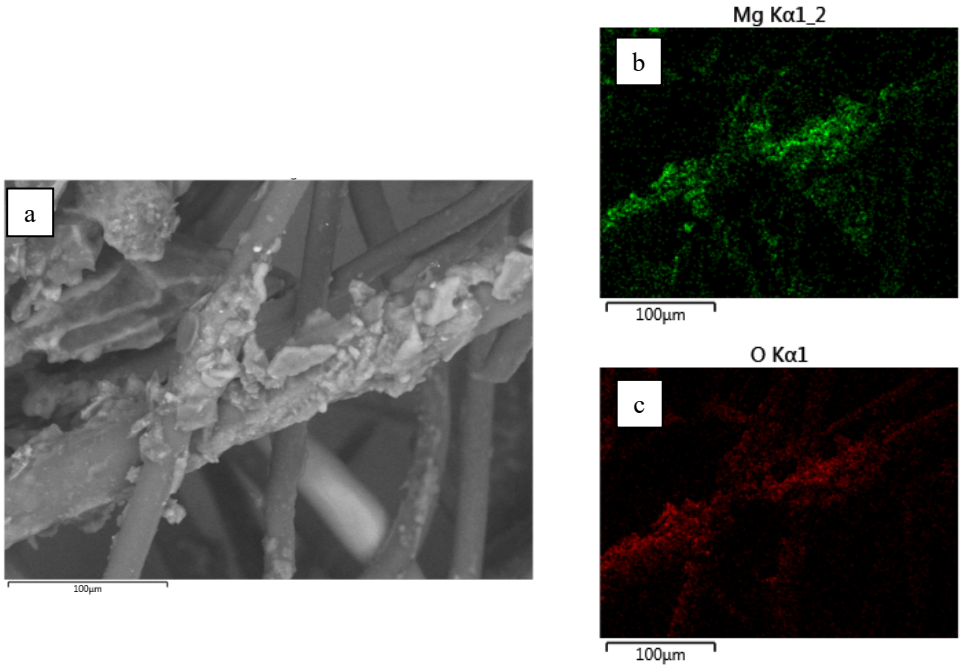

Fig. 5. a) SEM micrograph; b) and c) EDS mapping for Mg (b), and O (c)

Figures 5b and 5c show the corresponding energy-dispersive X-ray spectroscopy (EDS) results. Elemental mapping highlights the presence of Mg and O elements bond on PES fiber, thus confirming the impregnation.

3. Conclusions

This research presents the successful synthesis of magnesium oxide (MgO) nanoparticles using a microwave- and ultrasound-assisted hydrolytic procedure. X-ray diffraction (XRD) analysis confirmed the formation of pure MgO nanoparticles with a cubic crystal structure. No peaks corresponding to impurities were observed. The size of the nanoparticles ranged from 127 nm to 175 nm, with an irregular, nanosphere-like morphology as observed by scanning electron microscopy (SEM). The impregnated filters also showed the presence of Mg and O elements by SEM with energy dispersive X-ray spectroscopy (EDS) analysis, indicating successful impregnation with MgO nanoparticles.

Due to their unique properties and easy preparation methods, MgO NPs are positioned as a promising solution for a wide range of applications in environmental research and beyond. Optimizing the synthesis of MgO nanoparticles is an important step towards discovering new applications and solving significant environmental problems.

Further testing to be carried out at laboratory level to validate these properties will be antimicrobial tests (assessing the ability of the nano MgO impregnated filter to inhibit the growth of microorganisms by culture methods and inhibition zone analysis), filtration efficiency tests (measuring the ability of the filter to retain particles of different sizes using standardized methods), adsorption capacity tests (these will determine the ability of the filters to adsorb specific contaminants, highlighting the contribution of MgO nanoparticles to the overall filtration performance). Therefore, in the future we want to develop more efficient and durable nanostructured magnesium oxide impregnated PES filters capable of meeting the stringent requirements of various industrial applications such as: in ventilation and air conditioning systems to ensure clean and healthy air in commercial premises.

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