



OPEN Nondestructive quantitative method for evaluation of magnesium hydroxide on surface of coated fabrics from Raman spectra

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Magnesium-based compounds, such as oxide (MgO) and hydroxide ($\text{Mg}(\text{OH})_2$), are used in various applications ranging from medicine to electrotechnics. Especially, magnesium hydroxide coatings of textile structures are frequently used as part of flame retardant compositions, antibacterial agents, and surface absorption of far infrared radiation. Numerous analytical techniques exist for evaluating these compounds on material surfaces. The main aim of this article is to propose a novel methodology for the non-destructive quantitative evaluation of $\text{Mg}(\text{OH})_2$ coatings on the surfaces of typical synthetic fibers in low concentrations by analyzing Raman spectra. A regression approach was used to determine the concentration of $\text{Mg}(\text{OH})_2$ based on the assumption of Raman spectra additivity of $\text{Mg}(\text{OH})_2$ and the synthetic fiber. A simple modification of the sensor for analyzing Raman spectra was implemented, ensuring accurate positioning along all three axes. This methodology was applied to the analysis of polyester and polyamide textiles with $\text{Mg}(\text{OH})_2$ coatings.

Keywords Magnesium oxide, Magnesium hydroxide, Raman spectra, Textile coatings, Nondestructive technique

Magnesium oxide particles and nanoparticles are used in many application areas, such as agriculture (plant protection and growth support), biomedicine (bone regeneration), medicine (antimicrobial agents), electrotechnics (sensors, batteries), wastewater treatment (removal of toxic pollutants), and as flame retardants¹. These particles possess a high melting point, low density, good mechanical properties, high thermal conductivity, self-cleaning activity (via photooxidation), high luminescence, and a high refractive index for photonic applications. They also exhibit low electrical conductivity, excellent optical transparency, and biocompatibility, among other beneficial properties^{2–5}. Additionally, they are incorporated into compounds for so-called FIR (far infrared emitted) textiles, which can reuse infrared radiation emitted by humans for heating purposes^{6–9}. The main application areas of MgO are illustrated in Fig. 1.

Magnesium oxide nanoparticles are sparingly soluble in water¹ and can easily be converted into magnesium hydroxide². Magnesium hydroxide is also used as an antibacterial agent, flame retardant, and precursor for the production of magnesium oxide. $\text{Mg}(\text{OH})_2$ acts as a neutralizer for acidic wastewater and as a fertilizer². Of particular interest are its various morphological forms, including needles, tubes, fibers, platelets, rods, and even “flower-like” shapes².

Figure 1 shows the primary application areas of $\text{Mg}(\text{OH})_2$. Numerous methods exist for synthesizing $\text{Mg}(\text{OH})_2$ and MgO , ranging from simple precipitation and calcination to chemical vapor deposition (see Fig. 2).

The use of coatings made from magnesium oxide and magnesium hydroxide and their possible combinations with ammonium polyphosphate in water dispersion for functional antibacterial finishing of textiles is described in the patent¹⁰. A simple method to fabricate multifunctional PET fabrics with excellent flame retardant and antibacterial properties based on functionalized $\text{Mg}(\text{OH})_2$ is described by Wang et al.¹¹. They used nano- $\text{Mg}(\text{OH})_2$ modified using titanate coupling agent and stearic acid to ensure better compatibility with PET fibers. Zhang et al. used $\text{Mg}(\text{OH})_2$ nanoparticles and polycarboxylic acid as cross-linkers for the surface coating of silk fabric to improve its flame retardancy and smoke suppression¹².

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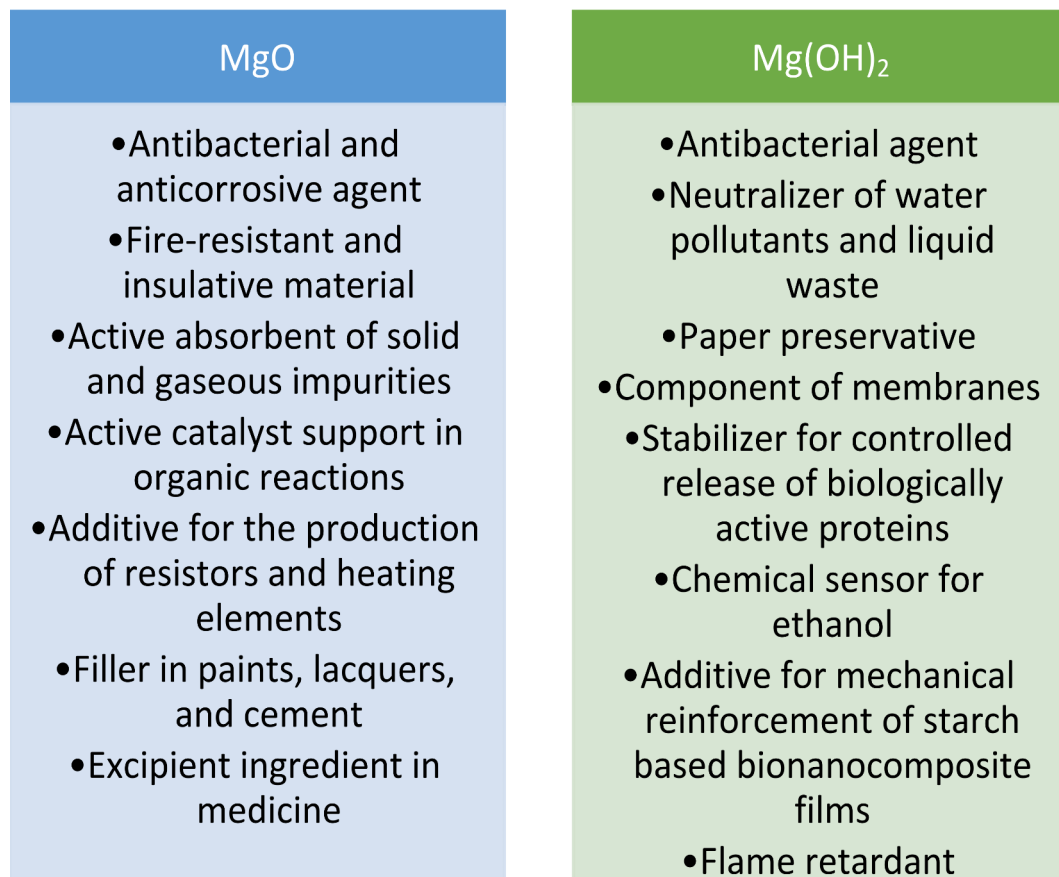


Fig. 1. Main applications areas of MgO and Mg (OH)₂.(adapted from²).

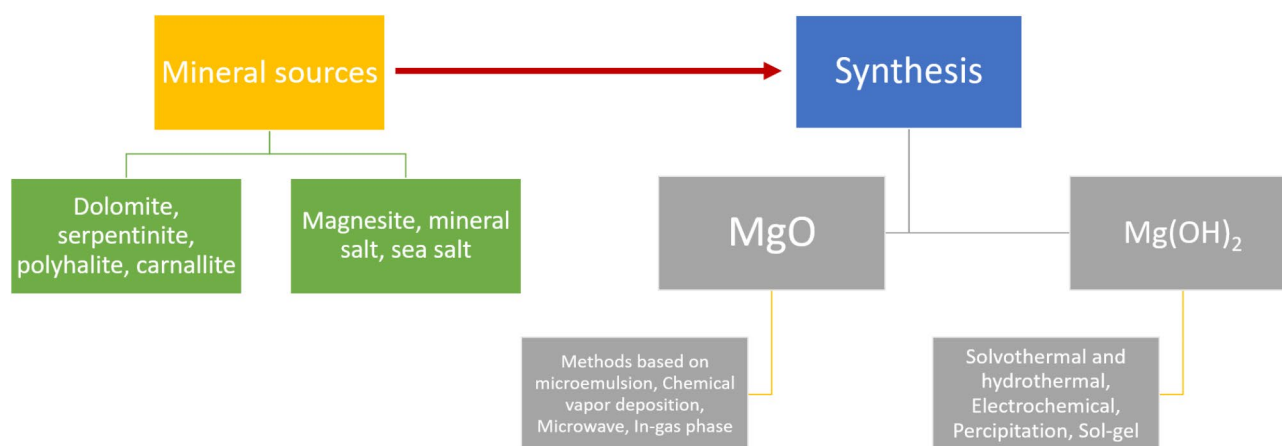


Fig. 2. Basic ways of Mg (OH)₂ and MgO preparation (adopted from² and¹³).

This report presents a methodology for the quantitative nondestructive evaluation of Raman spectra of typical synthetic fibers and Mg (OH)₂ coated on their surfaces in low concentrations. Regression analysis, based on the assumption of additivity of the spectra, was used to determine the concentration of this component. The developed methodology was applied to analyze polyester and polyamide textiles with Mg (OH)₂ surface deposits.

Evaluation of Mg (OH)₂ content

For the qualitative indication of MgO and Mg (OH)₂, spectroscopic techniques such as XRD, FTIR, and Raman spectroscopy are used^{14–16}. The quantitative analysis of Mg (OH)₂ content is commonly based on titration.

Spectroscopic methods need to create calibration curves relating the intensity of the typical peak in spectra to known concentrations of $\text{Mg}(\text{OH})_2$ powder often dissolved, e.g., in selected acids such as acetic acid. In the case of analysis of low concentrations of $\text{Mg}(\text{OH})_2$ coated on the surface of fabrics, it is necessary first to extract this compound by using suitable chemicals as acids, which lead to the destruction of the coating. In these cases, $\text{Mg}(\text{OH})_2$ particles frequently cover the fiber's surfaces only partially. To avoid the destruction of coating and laborious construction of calibration curves, it is possible to use a new simple method based on whole XRD, FTIR, or Raman spectra of the uncoated and $\text{Mg}(\text{OH})_2$ coated fabric and spectra of pure $\text{Mg}(\text{OH})_2$. The amount of $\text{Mg}(\text{OH})_2$ is calculated based on the assumption of spectra additivity. Raman spectroscopy, particularly micro-Raman spectroscopy, offers better spatial resolution, wider spectral coverage, narrower spectral bands, and lower water interference compared to XRD and FTIR spectroscopy¹⁷.

The Raman effect occurs when a sample is illuminated with monochromatic (laser) light, causing a small fraction of the scattered radiation to exhibit frequency shifts corresponding to the vibrational transitions of the material. Raman spectroscopy detects light scattered inelastically at frequencies different from the incident light, corresponding to molecular or lattice vibrations within the tested sample^{18,19}. Raman scattering is the process of re-radiation of scattered light by dipoles induced in molecules by incident light, which is modulated by the vibrations of the molecules²⁰. Therefore, the intensity of one peak in spectra is not sufficient for quantitative analysis of the sample because it corresponds to the presence of some groups only.

In normal Raman scattering, the dipoles are generated through the interaction between the electric field component (E) of the incident light and the molecules²⁰. Raman scattering thus arises from the oscillatory polarization of the molecule as a function of its vibrations rather than from the molecule's permanent dipole. Molecules in the ground state produce bands shifted to lower energies than the incident light, while higher-frequency bands result from molecules in excited vibrational states. These bands originate from the material's inelastic scattering of light. Elastic collisions, on the other hand, result in Rayleigh scattering, which appears more intense than the unshifted component of the scattered light²⁰.

Materials and methods

Fabrics

The knitted PAD 6,6 (hexamethylene adipate, fabric thickness 30 mm) and woven PET (polyethylene terephthalate, fabric thickness 32 mm) produced by Sintex Company Czechia and specified in Table 1 were used as starting material. Fiber surface roughness was evaluated by confocal laser microscope Olympus Lext 3000 (see Fig. 3). Illustrative images of both fabrics are in Fig. 3.

Before the actual coating, the surfaces of the PET fabric were activated using the following steps:

1. Hydrolytic cleaning in 5 wt% H_2SO_4 for 30 min with stirring and then rinsing and drying at 140 °C for 2 min.
2. Washing in 1 wt% NaOH for 5 min and then rinsing.
3. Drying at 120 °C for 20 min.

Activation of PAD 6.6 textiles was carried out only by alkaline hydrolysis in 2% NaOH and 0.2% decyl glycoside solution at 50 °C for 30 min, followed by rinsing and centrifugation.

Preparation of $\text{Mg}(\text{OH})_2$

A solution of 40 g of MgSO_4 (P.A., Lach-Ner) was precipitated with an equivalent amount of NaOH. After vacuum filtration at 80 °C using a Büchner funnel, the precipitate was washed with distilled water and finally with isopropanol. It was then air-dried at 20 °C. The $\text{Mg}(\text{OH})_2$ powder is shown in Fig. 4 (left).

Preparation of MgO

The dried and homogenized $\text{Mg}(\text{OH})_2$ was annealed at 900 °C for 5 min. The resulting MgO powder is shown in Fig. 4 (right).

Coating

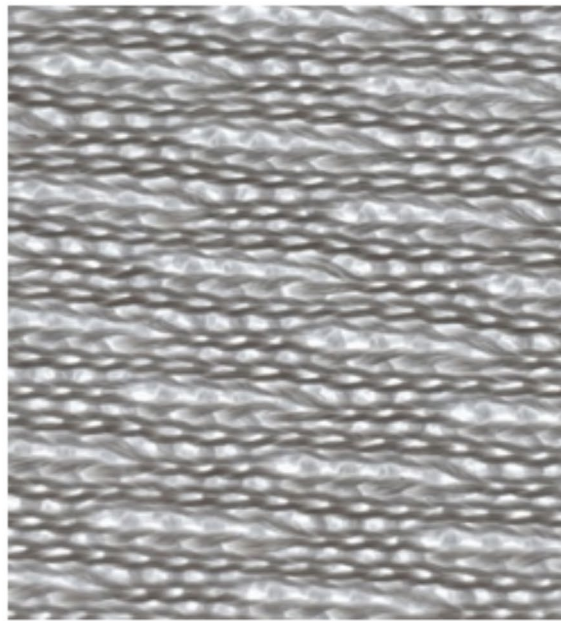
The methodology used for coating PET knitted fabric is described in the first column of Table 2. The methodology used for coating polyamide fabric is given in the second column of Table 2.

Evaluation of Raman spectra

For the evaluation of Raman spectra, the Dual Lightnovo mini Raman spectrometer was used with excitation lasers of 785 nm and 660 nm, covering the spectral ranges of 2400–400 cm^{-1} and 4000–2600 cm^{-1} . Accompanying Miraspec communication software was used to acquire the spectra. To improve focus, a stand with micrometric XY shifts and coarse Z shift adjustment was constructed by the company Trilobyte (see Fig. 5).

Type	Material composition	Pattern	Areal density [g m^{-2}]
PET	100% PES filament 82 dtex, knitted	Double face filet	143
PAD 6.6	100% PAD 6.6, filament 78 dtex, woven	Satin 5/1	105

Table 1. Material description.



PET knitted
(fiber mean surface roughness 0,22 μm)



PAD 6.6 woven
(fiber mean surface roughness 0,082 μm)

Fig. 3. Structure of starting fabrics.



Fig. 4. Powdered magnesium oxide (right) and hydroxide (left).

(PET)	(PAD 6,6)
1. Treatment of 20 × 20 cm samples in two liters of 0.02 M/l $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution for 30 min.	1. Treatment of 20 × 20 cm samples in two liters of 0.02 M/l $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution for 30 min.
2. Hydrolytic formation of $\text{Mg}(\text{OH})_2$ by gradual addition of 0.04 M/l NaOH solution for 30 min.	2. Hydrolytic formation of $\text{Mg}(\text{OH})_2$ by gradual addition of 0.04 M/l NaOH solution for 1 h.
3. Rinsing and centrifugation.	3. Rinsing and centrifugation.
4. Thermal stabilization in a thermopress at 150 °C for 30 min.	4. Thermal stabilization in a thermopress at 140 °C for 30 min.

Table 2. Textile coating procedure.

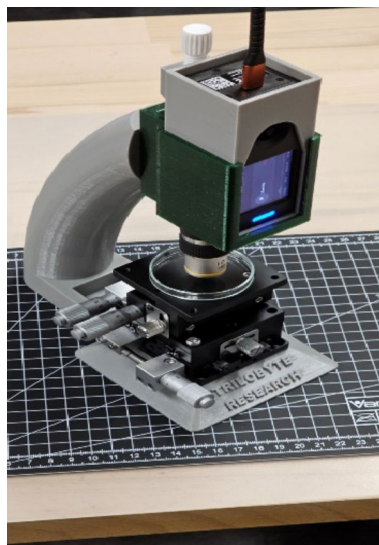


Fig. 5. LightNovo mini Raman spectrometer is on stand with the XYZ shift.

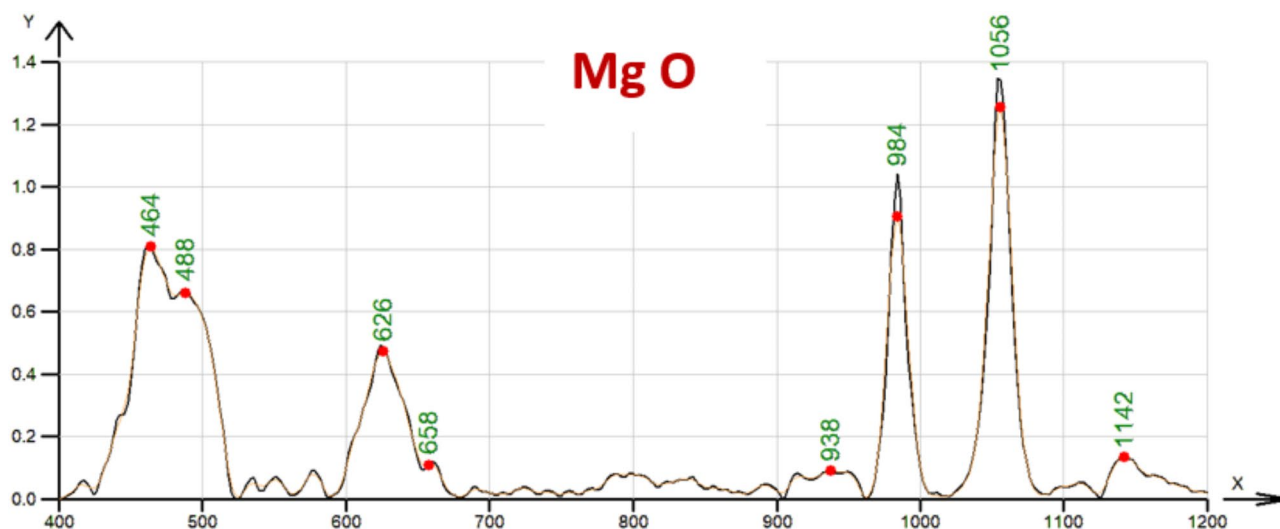


Fig. 6. Raman spectrum of MgO, excitation laser 785 nm.

Results and discussion

All Raman spectra have a y-axis intensity [-], and x axis wavenumber [cm^{-1}] as standard output from the spectrometer. Raman spectrum of MgO is shown in Fig. 6 and of $\text{Mg}(\text{OH})_2$ in Fig. 7.

The Raman spectrum clearly distinguishes magnesium oxide from magnesium hydroxide by a very strong emission band at 1056 cm^{-1} , which is present in the oxide but absent in the hydroxide. The spectrum of $\text{Mg}(\text{OH})_2$ has been utilized to detect the presence of hydroxide in coated textile fabrics.

Raman spectra in the Raman shift range of 400 to 2400 cm^{-1} for uncoated PAD 6,6 fabric are shown in Fig. 8, and for $\text{Mg}(\text{OH})_2$ – coated PAD 6,6 fabric in Fig. 9.

Raman spectra in the Raman shift range of 400 to 2400 cm^{-1} of uncoated PET fabric are shown in Fig. 10, and for $\text{Mg}(\text{OH})_2$ coated PET fabric is shown in Fig. 11.

The spectra of the uncoated and $\text{Mg}(\text{OH})_2$ coated fabrics show no marked visible differences. Table 3 shows the strongest bands of the two fabrics (PAD 6,6 and PES).

Based on the availability or experimental spectra of uncoated fabric, $\text{Mg}(\text{OH})_2$ coated fabric, and pure $\text{Mg}(\text{OH})_2$, it is possible to calculate the relative portion of uncoated fabric and magnesium hydroxide without the necessity to create calibration curves.

Assuming the additivity of Raman spectra, the spectrum of a mixture of two substances, in the absence of chemical interaction between them, can be expressed as a linear combination of the spectra of the individual substances. For the case where Raman spectra of coated fabric (R_C), uncoated fabric (R_U), and pure $\text{Mg}(\text{OH})_2$ (R_M) are at disposal (measured), the following relationship can be used:

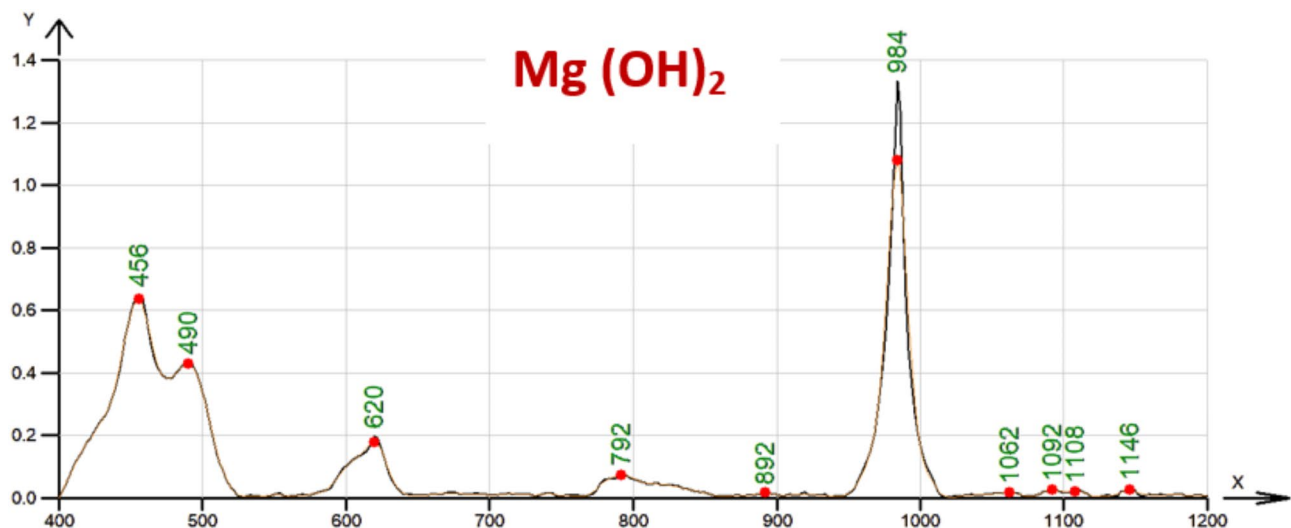


Fig. 7. Raman spectrum of $\text{Mg}(\text{OH})_2$, excitation laser 785 nm.

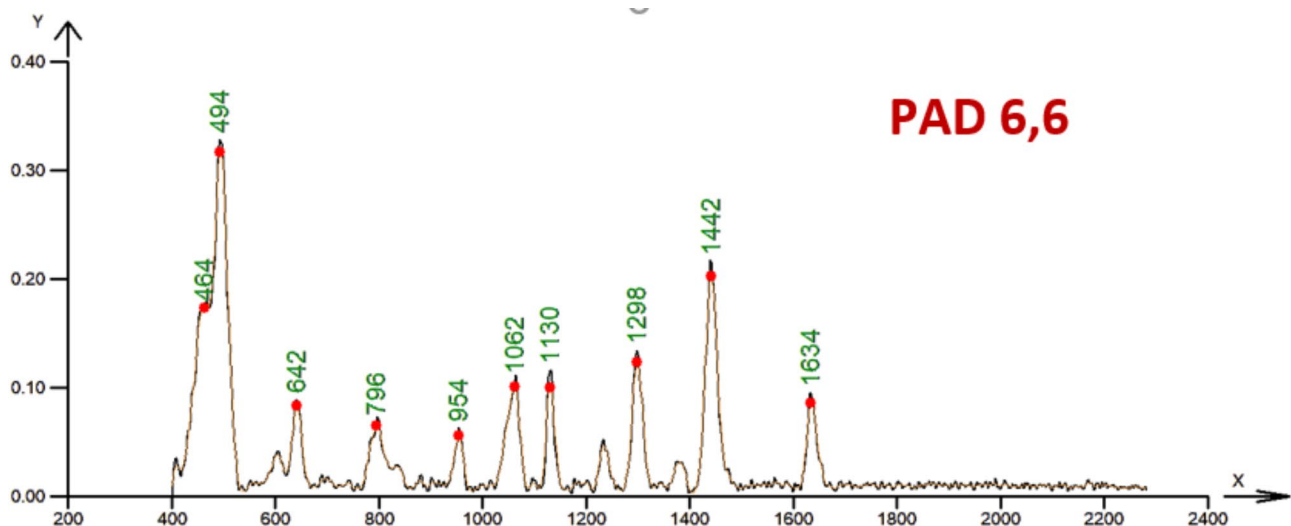


Fig. 8. Raman spectrum of uncoated PAD 6,6 fabric, excitation laser 785 nm.

$$R_C = a \cdot R_U + b \cdot R_M \quad (1)$$

Here, a represents the relative proportion of the uncoated fabric, and b represents the relative proportion of $\text{Mg}(\text{OH})_2$ (proportional to the percentages of their relative concentration). These parameters can be calculated from the whole spectrum, i.e., at all measured wavenumbers (frequencies).

It is, therefore, possible to predict the Raman spectrum of coated fabric using a linear regression model, where a and b are the regression coefficients estimated using the ordinary least squares (OLS) method (see²¹).

Table 4 summarizes the results of the OLS analysis for PAD 6,6 coated with $\text{Mg}(\text{OH})_2$. For the characterization of the significance of parameters, the P values corresponding to each parameter estimate were calculated. This value P is the smallest significance level at which the null hypothesis H_0 (estimated parameter is zero) is still rejected based on the given data. Denote that for confidence intervals, the significance level is equal to 0.05.

The results of OLS for PET coated by $\text{Mg}(\text{OH})_2$ are summarized in Table 5.

A detailed description of the calculation and interpretation of these characteristics is provided in²¹.

The coefficient of determination R^2 was selected as a characteristic of overall model quality²¹. It is well known that the predictive linear model (1) describes experimental data precisely (the sum of squared deviations between predicted values of Raman intensity and predicted values is approaching zero). In the case of coated PAD 6,6 fabric, $R^2 = 0.92$ was calculated, and for coated PET fabric, $R^2 = 0.94$ was calculated.

For the tested coated PAD 6,6 fabric, the concentration of $\text{Mg}(\text{OH})_2$ was found to be 0.802%, while for the tested PET fabric, it was determined to be 4.715%. These values closely match the results obtained through titration of extracted $\text{Mg}(\text{OH})_2$ using HCl (a destructive method) with NaOH and methyl orange as an indicator.

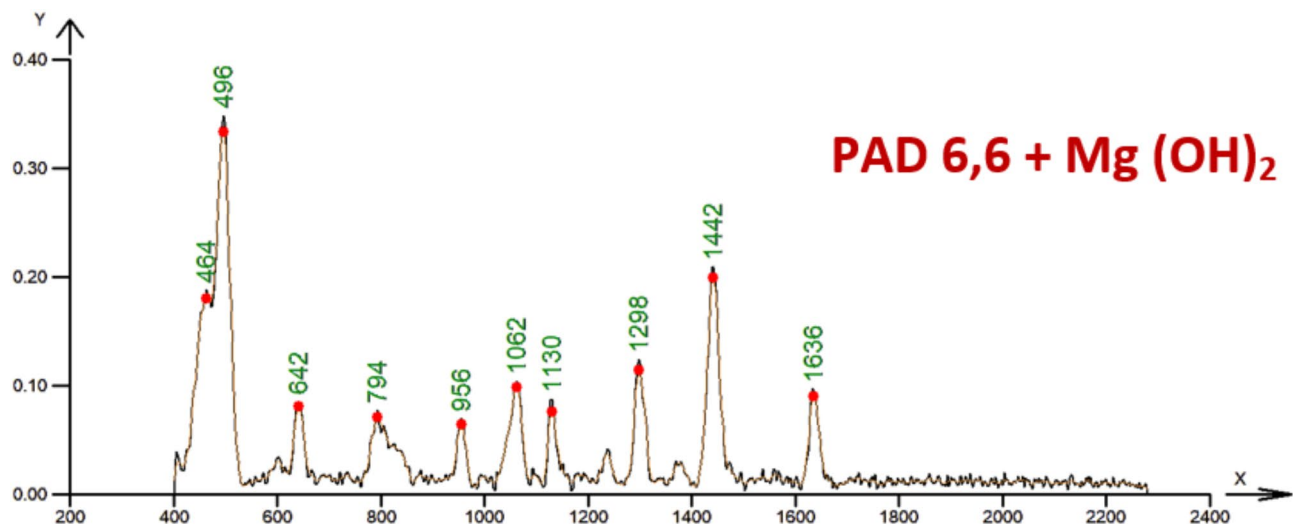


Fig. 9. Raman spectrum of PAD 6,6 fabric coated by $\text{Mg}(\text{OH})_2$, excitation laser 785 nm.

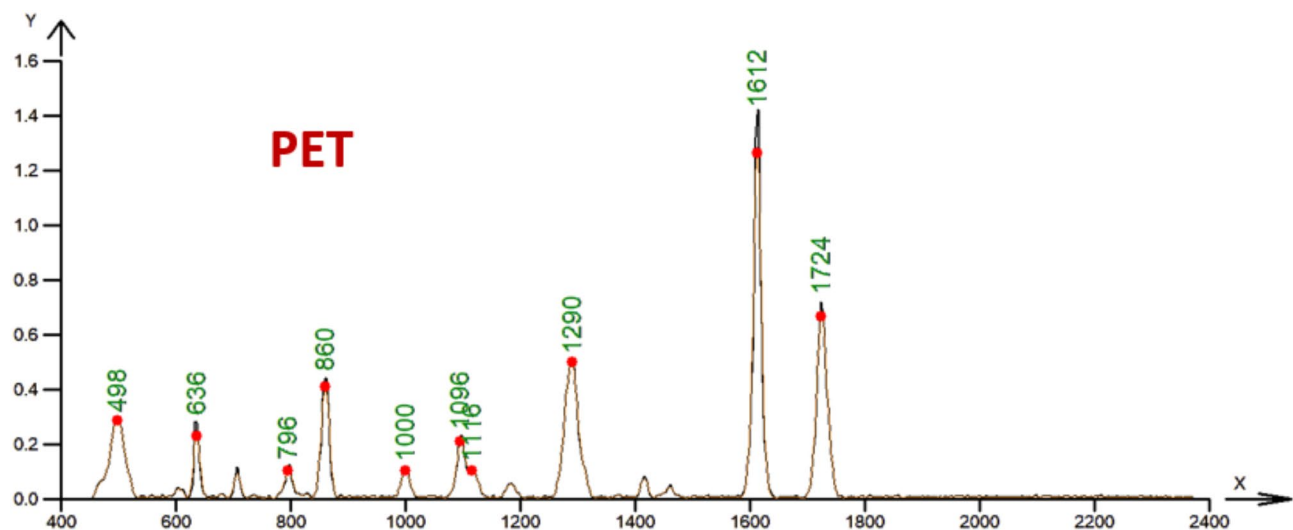


Fig. 10. Raman spectrum of uncoated PET fabric, excitation laser 785 nm.

The titration revealed an $\text{Mg}(\text{OH})_2$ concentration of 0.44% for the coated PAD 6,6 fabric and 4.30% for the coated PET fabric.

If heteroskedasticity in the noise distribution is considered—since noise may have greater variance at higher signal intensities—it may be more appropriate to use the weighted least squares method with weights inversely proportional to the signal intensity. The set of tests for the characterization of peculiarities of experimental errors of measured spectra and corresponding changes of OLS criterion are described in the book²¹.

Conclusions

Utilization of $\text{Mg}(\text{OH})_2$ is very common in plenty of applications. It ranges from thermochemical energy storage²² and shape-stabilized phase change materials²³ to CO_2 adsorption at room temperature²⁴ and hydration of $\text{Mg}(\text{OH})_2$ in reactive cement^{24–26}. In the case of using coatings as a full surface layer or partial covering surface in the case of porous materials, it is necessary not only to identify the presence of $\text{Mg}(\text{OH})_2$ but also concentration because it is directly related to changes of some properties. This task is not easy and obviously needs the construction of a calibration curve for the prediction of concentration from the measured signals. For high concentrations of $\text{Mg}(\text{OH})_2$ powder, it is often sufficient to use image analysis of SEM images. A very low (few percent) content of $\text{Mg}(\text{OH})_2$ in surface coatings cannot be easily identified visually by using SEM images. More sensitivity for the presence of $\text{Mg}(\text{OH})_2$ in the coating is used in spectral methods such as Fourier transform infrared analysis (FTIR) or Raman spectroscopy, which is less sensitive to water content and the presence of colored substances. The linear regression-based method outlined herein is direct, uncomplicated, and requires no calibrations.

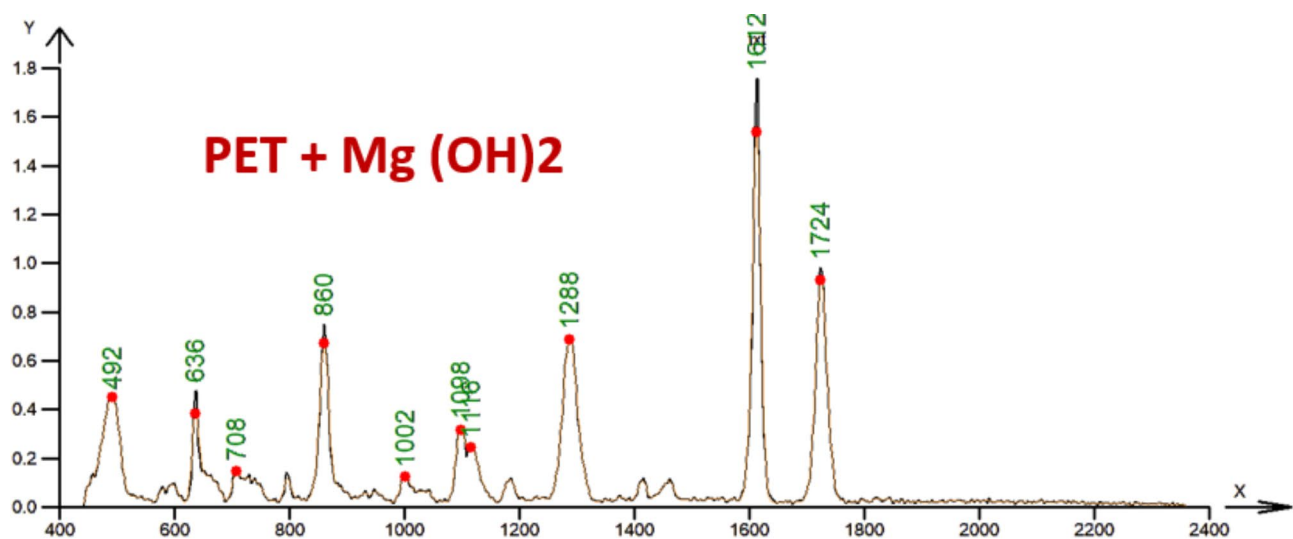


Fig. 11. Raman spectrum of PET fabric coated by Mg (OH)₂, excitation laser 785 nm.

Type	The most significant Raman bands (cm ⁻¹)
PAD 6,6	494, 1442, 464, 1298, 1062, 1130, 1634, 642, 796, 954
PET	1612, 1724, 1290, 860, 498, 636, 1096, 796, 1116, 1000

Table 3. The most significant Raman bands for PAD 6,6 and PES) fabrics.

Parameter	Estimator	Standard deviation	P value	Conclusion	Confidence interval
Intercept	0.00214	0.00025	0	Significant	(0.00165, 0.00264)
a	0.99146	0.00502	0	Significant	(0.98161, 1.00131)
b	0.00802	0.00199	0.00006	Significant	(0.00412, 0.01193)

Table 4. Parameter estimates for PAD 6,6 coated by Mg (OH)₂.

Parameter	Estimator	Standard deviation	P value	Conclusion	Confidence interval
Intercept	0.02986	0.00144	0	Significant	(0.02704, 0.03268)
a	1.29929	0.00909	0	Significant	(1.28146, 1.31712)
b	0.04715	0.01093	0.0002	Significant	(0.02569, 0.0686)

Table 5. Parameter estimates for PET coated by Mg (OH)₂.

A verified methodology for the nondestructive evaluation of coated Mg (OH)₂ on different fibrous materials, based on Raman spectra and standard regression analysis, is novel, straightforward, and can replace more complex other analytical techniques. A simple modification of the Lightnovo mini Raman spectrometer into a specialized Raman microscope can serve as a cost-effective alternative to expensive Raman spectrophotometers. This method can be forecasted to support the use of Mg (OH)₂ in many applications and will serve as a tool for the optimization of its concentration.

Data availability

All data generated or analyzed during this study are included in this published article.

Received: 5 March 2025; Accepted: 4 April 2025
Published online: 12 April 2025

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Acknowledgements

This work was supported by the project ‘Advanced Structures for Thermal Insulation in Extreme Conditions’ (reg. no. 21-32510 M) granted by the Czech Science Foundation (GACR).

Author contributions

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Funding

This work is funded by the Department of Material Engineering, Faculty of Textile Engineering, Technical University of Liberec, Czech Republic.

Declarations

Competing interests

The authors declare no competing interests.

Additional information

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