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UV Degradation Influence on the Selected Physical Properties of Extruded PVC/Ceramic Composites

Aneta Tor-Świątek¹*, Łukasz Garbacz¹

¹ Lublin University of Technology, Faculty of Mechanical Engineering, Department of Technology and Polymer Processing, ul. Nadbystrzycka 36, 20-618 Lublin, Poland

* Corresponding author’s email: a.tor@pollub.pl

ABSTRACT
This paper presents a study of PVC-ceramic composites obtained by twin-screw extrusion. Properties such as colour, wettability, tensile strength, elongation at break and impact tensile strength were studied. Moreover, the composite samples were subjected to UV degradation process and the influence of the composite composition therefore the degradation process on the mentioned properties has been determined. The study showed the dependence of the ceramic content in the material and its granulation on the individual properties. The research showed a significant influence of degradation on the colour and wettability of samples containing ceramic filler with granulation 0.25–0.5 mm, and in the case of tests of mechanical properties, this influence is the greatest for samples with filler with grain size 0.5–1.25 mm. Additionally, the aging process significantly influenced obtained results.

Keywords: composites, polymer, ceramic, UV degradation, extrusion

INTRODUCTION

Polyvinyl chloride is one of the oldest known synthetic materials with the longest history of industrial production. PVC is one of the most worldwide popular in terms of production volume, right after polypropylene (PP) and polyethylene (PE). PVC is the first synthetic thermoplastic produced on an industrial scale [1, 20, 21, 34, 44]. It is one of the few polymers with such modern applications with the oldest origin. Mixed with additives such as stabilizers, impact modifiers, fillers, etc. are processed into finished products by various techniques [2, 4, 12, 37, 39, 40]. The main recipient of PVC is construction industry and its infrastructure. The construction sector accounts for two-thirds of the PVC market, including sewage pipes, fittings, electric cable sheaths, window profiles, roller shutters and gutters [13, 33]. The scale of its applications, especially in construction, results from the above-mentioned functional and economic features, safety in use, but also from excellent design values, including longevity and resistance to the influence of aggressive external factors [5, 24, 26, 43]. In article [1] shown a review of synthetic polymer-ceramic composites with special emphasis on the calcium phosphate group and their potential applications and their manufacture for biomedical application.

Ceramic fillers are described in industry as an excellent, ecological, lightweight aggregate that is 100% recycled. The granulate obtained as a result of a special treatment process is perfectly round, the grain diameter ranges from 0.05 to 16 mm. One of the advantages of ceramic fillers is its low weight and high compressive strength. Moreover, it is characterized by excellent thermal insulation and resistance to alkaline substances and other chemicals [3, 11, 36, 38].

Cellular glass granules are non-flammable and resistant to weather conditions, it is not a breeding ground for bacteria and mold. This type of filler is perfect for improving the properties of materials, hence it has been used in the construction industry, among others, in the production of plasters, mortars or massive wall blocks. Cellular
glass granules are also characterized by a high percentage of absorption of 20–30% by weight [10, 11, 41].

Cellular glass granules are characterized by excellent sound absorption and good thermal properties. The advantages have been appreciated by manufacturers of houses and other structures, thanks to which the walls and structural elements are light and resistant to mechanical loads [6, 9, 26, 35, 42, 43].

High exposure of the polymeric material to UV radiation can accelerate the degradation process. UV radiation affects the process of breaking polymer chains, which causes the surface of the polymer to lose its gloss and the mechanical properties of the material to deteriorate over time [7, 22, 23, 25]. Lu et al. [27, 28, 29] conducted a series of studies on the evaluation of the degradation of polymer coatings and composites with a polymer matrix, which were subjected to long exposure to UV radiation. As pointed out by Lu et al., not much research has been conducted that would focus on the analysis and development of aging models of polymeric materials subjected to UV rays. The conducted research and literature [15, 30] shows photodegradation as the main factor of damage to polymeric materials. Moreover, aging may result in the loss of products with low molecular mass, which evaporate very quickly at high temperatures [15, 32].

Most of the current research has focused on studying the degradation of polymers or composites with a polymer matrix based on one individual factor, such as UV radiation or water diffusion [15, 42]. François-Heude et al. focused on synergistic aging under multiple conditions involving UV, where they demonstrated that the combined cyclic conditions of UV radiation and humidity caused damage to the polymer matrices and that this damage was greater than in non-cyclic degradation studies [17]. T. Lu et al showed that UV radiation itself has the effect of damaging the polymer matrix by producing microparticles. The process of UV degradation accelerates when there is water on the surface of the material, at the same time it was found that water itself does not cause much degradation compared to UV rays alone [29, 31]. Yern Chee et al. in the paper [45] presented that long-term exposure to UV radiation leads to discoloration and loss of mechanical properties of polymers composites. Due to these limitations, polymeric composite materials have been developed: the incorporation of various nanoparticles, additives and fillers into polymeric materials via chemical and enzymatic treatments has improved the heat resistance properties of polymers. Nanomaterials, UV absorbers and UV stabilizers are typically added to polymer matrices to improve the UV-resistant properties of composite materials.

The conducted research includes the analysis of the mechanical properties of increased filled polymer-ceramic composites made by twin-screw extrusion and the influence of UV degradation on these properties. In addition, tests were carried out to change the colors of the composites after their premature placement in the chamber where the tested samples were exposed to UV rays.

MATERIALS

The subject of the research are polymer-ceramic composites. Their matrix was made of poly(vinyl chloride) with the trade name Al-favinyl GFM/4-TR. The properties of the polymer material used according to the manufacturer were: density 1230 kg/m³, modulus of elasticity 2600 MPa, tensile strength 21MPa, elongation at break 300%, Shore’a hardness 80°Sh. The filler was made of Poraver ceramic granules, which was recycled expanded glass. The chemical composition of the material and the percentage of individual components of the filler as quoted by the manufacturer are following: SiO₂ – 70–75%, Na₂O – 10–15%, CaO- 7–11%, Al₂O₃ – 0.5–5%, MgO – 0–5%, K₂O – 0–4%. All those ingredients were measured with XRF method.

Three gradations of filler were used for the production of composite samples. One group of samples was filled with ceramic granules, the size of granules ranged from 0.5–1.25 mm, the second with 0.25–0.5 mm, and the third also with 0.25–0.5 mm and after the second glass expansion process (PR2). The properties of the gradation materials are presented in Table 1. In order to simplify the analysis, four basic groups of samples were distinguished, the description of which is presented in Table 2.

EXPERIMENTAL

Composite samples were obtained in a conventional extrusion process carried out on an twin-screw extruder machine EHP 2x20 Sline
Figure 1), produced by Zamak Mercator (Poland). Mineral fillers were introduced into the tested PVC in the process of mechanical mixing before processing. During the extrusion process, inorganic fillers undergo the same stages as plastic working, pressing, homogenization, and transport. Organic fillers that were created in the extrusion process were introduced into PVC in the amount of 10%, 20%, 30% by weight.

Due to the size of the filler granules, an extruder die 2.3 cm wide and 0.14 cm thick was used. The rotational speed of the screw during extrusion was 45.7 rpm and the temperature of the coolant was 19.5°C. The temperature in individual zones of the system was in plasticizing system zones I – 140°C, II – 150°C, III – 152°C, IV – 160°C and in extruder head zones: I – 160 °C, II – 50°C, III – 50°C, IV – 50°C.

Laboratory studies on accelerated aging were carried out in a HD-E71 xenon test chamber in accordance with ISO 4892–2:2013, Annex B. Exposure period was 102 minutes dry and 18 minutes water spray. Wavelength (λ) were in the range 290–320 nm and the power of UV lamp was 290 W. Black standard temperature of aging was equal 65°C. The entire aging process lasted 360 h, where in the aging chamber 1 hour corresponds to 1 day in reality.

The study of mechanical properties through static stretching consisted in determining the strength parameters by subjecting the samples to static stretching. The direct research process was carried out in accordance with the applicable standard with the designation PN-EN ISO 572. Sample with type 1A were cut out of the produced composites with the ZCP 020 manual press. In order to carry out tensile strength measurements, a testing machine by Zwick Roell Z010 was used. Selected parameters of the apparatus accompanying the sample stretching process were: the test speed 50 mm/min and the distance of the handles at the starting position 110 mm.

From the group of dynamic tests, impact tensile measurements were made. Research was carried out in accordance with the PN-EN ISO 8256:2006 standard. As in the case of cutting fittings subjected to static stretching, it was carried out on ZCP 020 hand presses. Due to the method of fixing the sample, method A was chosen as the measurement method. It consists in placing the tested sample on the frame supports. One end of the sample was clamped in a clamping device and the other end in a cross yoke. When the hammer hits, the sample is stretched until it breaks. The strength parameters were measured using a Comatech Testing Machines type 639F. The hammer

| Designation | Matrix material | Filler Type and granulation | Amount of filler, % | Designation |
|-------------|-----------------|-----------------------------|---------------------|-------------|
| PVC         | poly(vinyl chloride) | -                           | 0                   | BA – before aging |
| A10         | Alfavinyl GFM/4-TR | Poraver 0.5–1.25            | 10                  | AA – after aging |
| A20         |                 |                             | 20                  |             |
| A30         |                 |                             | 30                  |             |
| B10         |                 | Poraver 0.25–0.5            | 10                  |             |
| B20         |                 |                             | 20                  |             |
| B30         |                 |                             | 30                  |             |
| C10         |                 |                             | 10                  |             |
| C20         |                 | Poraver 0.25–0.5 /PR2       | 20                  |             |
| C30         |                 |                             | 30                  |             |

| Table 1. Selected physical properties of Poraver ceramic granules (manufacturer data) |
|---------------------------------|-----------------|-----------------|
| Name of filler                  | Poraver 0.5–1.25 | Poraver 0.25–0.5 | Poraver 0.25–0.5/PR2 |
| Granular size [mm]              | 0.5–1.25        | 0.25–0.5        | 0.25–0.5               |
| Bulk density [kg/m³]            | 270 +/- 30      | 340 +/- 30      | 350–460               |
| Particle density [kg/m³]        | 500 +/- 80      | 700 +/- 80      | 650–1000              |
| Crushing resistance [N/mm²]     | >2.0            | >2.6            | 3                      |
| According to DIN EN 13055–1     |                 |                 |                        |
| Water absorption [%]            | 18              | 21              | < 35                   |

| Table 2. Selected physical properties of Poraver ceramic granules (manufacturer data) |
|---------------------------------|-----------------|-----------------|
| Designation | Matrix material | Filler Type and granulation | Amount of filler, % | Designation |
| PVC         | poly(vinyl chloride) | -                           | 0                   | BA – before aging |
| A10         | Alfavinyl GFM/4-TR | Poraver 0.5–1.25            | 10                  | AA – after aging |
| A20         |                 |                             | 20                  |             |
| A30         |                 |                             | 30                  |             |
| B10         |                 | Poraver 0.25–0.5            | 10                  |             |
| B20         |                 |                             | 20                  |             |
| B30         |                 |                             | 30                  |             |
| C10         |                 |                             | 10                  |             |
| C20         |                 | Poraver 0.25–0.5 /PR2       | 20                  |             |
| C30         |                 |                             | 30                  |             |
speed was the same for all analyzes and was 2.9 m/s, and its energy was 5 kJ.

Static and dynamic mechanical property tests were performed for 5 specimens from each series before and after the aging process.

The analysis of color changes of polymer-ceramic composites was carried out using an X-rite Ci 4200 benchtop sphere spectrophotometer connected to a desktop computer. The colour assessment was carried out with the use of CIE Lab colour space, which allows the colour to be described in Cartesian coordinates. The individual colour components were measured three times for each type of sample before and after the degradation process. Then, the average values of each measured quantity were determined. The obtained chroma parameter ($C^*$) was determined according to the equation 1.1:

$$C^* = \sqrt{(a^*)^2 + (b^*)^2}$$  \hspace{1cm} (1)

and total colour change ($DE^*$) according to the equation 1.2:

$$DE^* = \sqrt{(L^* - L_0)^2 + (a^* - a_0)^2 + (b^* - b_0)^2}$$  \hspace{1cm} (2)

where: $a^*$ – represents a standard or sample’s position on the green/red axis in CIELAB colour space, green being in the negative direction and red being in the positive direction;

$b^*$ – represents a standard or sample’s position on the blue/yellow axis in CIELAB colour space, blue being in the negative direction and yellow being in the positive direction;

$L^*$ – represents a standard or sample’s position on the lightness axis in either CIELAB or CIELCH colour space;

$L_0$, $a_0$, $b_0$ – values of the black standard.

The static wetting angle was measured using a direct method by applying a drop of liquid to a stationary substrate (the surface layer of the materials). The measurement was performed on a test stand equipped with a Kruss DSA25 goniometer integrated with a digital camera and Kruss ADVANCE software for analysing the recorded image. Tests were performed for 5 specimens from each series before and after the aging process. Measuring liquids of 0.5 µl for water and 0.75 µl for diiodomethane, respectively, were applied to the surface of the test samples using a dedicated syringe. A drop of distilled water was applied to the surface of each sample (5 drops in total) and then the contact angle was measured after a few seconds, the procedure was repeated for diiodomethane and the SFE was determined for each batch of samples. Wetting angle measurements were performed using two liquids: distilled water, characterized by a high polar component, and diiodomethane, which is a dispersion liquid. Such a choice of fluids allows to minimize the influence of errors occurring during determination of SFE components of polar and dispersion measuring fluids on the calculated SFE of the material.
Surface free energy of water $\gamma_L = 72.8 \text{ mJ/m}^2$, the dispersion component is $\gamma_{dL} = 21.8 \text{ mJ/m}^2$ and the polar component $\gamma_{pL} = 51 \text{ mJ/m}^2$. Surface free energy of diiodomethane $\gamma_L = 50.8 \text{ mJ/m}^2$, dispersion component is equal $\gamma_{dL} = 50.8 \text{ mJ/m}^2$ and the polar component $\gamma_{pL} = 0 \text{ mJ/m}^2$.

RESULTS AND DISCUSSION

Calorimetry

Examination of the colour of the polymer-ceramic composite before (BA) and after aging (AA) allowed to obtain the photographs shown in Figures 2.

Analyzing the graph of the brightness value and the filler content in the form of ceramic granules of the produced composites in the samples before and after degradation (Fig.3), it results that the significant increase is observed only for pure sample – PVC with value 16%. Before degradation we can observe gradual increase for samples A, B and C. Degradation caused significant decrease of measured parameter related to pure sample PVC AA. The decrease was 20% for samples A, 18% for samples B and 12% for samples C. Within the individual series of samples the brightness of the samples with the addition of a filler with a diameter of 0.5 mm to 1.25 mm (sample A) decreased compared to pure PVC. Samples A with a gain content of 10%, 20% and 30% show a slight change in the brightness value. As a result of degradation, their value changed 3% on average. A higher changes was observed in samples B where the filler was made of ceramic granules with a diameter of 0.25 mm to 0.5 mm. Due to degradation, their brightness values increased by 5% for sample B10 and decreased by 2% for B20 and by 5% for B30. The similar situation can be observed in the case of samples C with a diameter of ceramic beads 0.25 mm to 0.55 mm with the PR2 particle range. Samples C10 noted slight increase but samples C20 and C30 noted a decrease by 3% and 6% in brightness.

On the basis of the graph of the dependence of the a* parameter on the share of ceramics of individual samples before and after the degradation process (Fig. 4) it can be observed that under the influence of the addition of ceramics the samples from green (PVC) become red, but these changes are in a small range. Under the influence of degradation the value of a* parameter decreases for A samples, which means that they become less red, while it increases for B and C samples, that make them more red. The addition of ceramic granules of different granulation and increasing content did not significantly affect the hue and tone of the colour. The UV degradation of the samples had a significant effect on the above parameters. The samples before aging were characterised by a yellow dominant ranging from 5 to 7.5 and a brightness ranging from 65 to 73.4, indicating a darkening of the samples. As a result of UV degradation,
the yellow dominant decreased significantly to the range of 0 to 10.5 and red dominant appeared in the range of 0 to 5. In addition, the brightness of the samples decreased significantly and ranged from 77.4 to 69.

Graph of the dependence of parameter b* on the type of samples before and after the degradation process (Fig. 5) shows that all samples exhibit yellow colour. The sample closest to the blue colour is the reference PVC sample, both before and after aging. Addition of ceramics increases the value of b* parameter, i.e. yellowing of samples before aging in relation to the standard PVC sample by 30%. Further increase of ceramics content increases yellowing of samples by about 10% on average. Degradation process significantly increased yellowing of samples and this increase is greater at increasing ceramics content. Yellowing of samples after aging increases on average by 20% in samples A, by 50% in samples B and by 40% in samples C.

The graph of C* parameter dependence on the type of samples before and after degradation process (Fig. 6) shows that the chroma of colour in particular samples before degradation process does not depend on ceramics content in the matrix and type of granulation. The increase of chroma is visible between PVC master sample and composite samples and amounts about 250%. The highest and significant increase of colour chroma is observed after degradation process at content of 20% and 30% of ceramics and amounts to 106% and 148% for B samples and 154% and 111% for C samples, respectively. A significant effect of aging process, granulation and ceramic content in the matrix on the colour chroma of the composite is observed.

The total difference (DE) between colours is useful as a single value to determine the colour difference between the sample and the standard. By calculating DE from the formula, the colour difference between samples before and after aging can be clearly seen (Fig. 7). For sample A the value of
the tested parameter after aging increased by 42%. For sample B a change of 57% is observed from 13.49 for the sample with 10% filler to as much as 30.78 for the sample with 30% filler. A nearly threefold increase was recorded for C samples where the value increased from 10.86 to 31.46.

**Surface wettability**

Figures 8 and 9 shows example photographs of the PVC sample surface before and after aging with a liquid droplet deposited. The distilled water droplets deposited on the samples are shown in figures (a) and the diiodomethane droplets are shown in figures (b).

Wetting angle studies showed a direct relationship of increasing ceramic content and aging process on the wetting angle value (Fig. 10). All tested composite surfaces exhibit hydrophilic properties except for the C30 sample before aging (BA). At the limit of hydrophobicity are the benchmark PVC and B10 samples wetted with water before aging. As the ceramic content increases, the wetting angle with distilled water and diiodomethane changes slightly before degradation. The aging process has a significant effect on the wetting angle values of the liquids used. Angle values for water-wetted samples increase for the PVC reference sample and for samples from group B. For water-wetted samples from group A changes are not significant, while for samples from group B increase of angle by average 15% is observed, while for samples C gradual decrease of angle value by 22% for C10, 26% for C20 and 35% for C30 is observed. For samples wetted with diiodomethane, the angle values increase due to aging. Non-significant angle changes occur on the surface of

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**Figure 5.** Parameter b* (in color range from blue to yellow) of individual samples before and after aging

**Figure 6.** Colour chroma of individual samples before and after aging
the PVC reference sample. For composite samples the increase is intense for and averages 82% for sample A, 220% for sample B and 142% for sample C. Table 3 shows the values of surface free energy determined from contact angles according to Owens-Wendt model.
The share of the dispersion component in the total value of SFE is higher for all the studied composites both before and after the degradation process. There is an increase in SFE for the tested samples as the ceramic content in the composite increases. This increase is more intense for the samples before aging. Additionally, the degradation decreased the SFE values for composite samples by an average of 15% for A samples, 36% for B samples and 3% for C samples. The aging process did not affect the SFE value for the PVC reference sample.

Mechanical properties

The tests of the mechanical properties of the produced composite samples were carried out both before and after the degradation process in the aging chamber. In the case of samples (Fig. 11) where the filler was ceramic granules with a diameter of 0.5–1.25 mm (samples A), the tensile strength decreased with the increase in the glass fibers content in the composite, while the obtained SFE values are similar. The presented work did not include aging studies.
test results for samples filled with granules with a diameter ranging from 0.25 mm to 0.5 mm (samples B), a decrease in tensile strength was also found along with the amount of filler. This was a decrease of 13% for samples before degradation and by 40% for samples exposed to radiation, which clearly shows that the degradation of the material significantly affects its strength. For samples with the addition of 0.25–0.5 mm/PR2 granules (samples C) the values decreased by 59% for samples before degradation and by 48% for samples after degradation, which gives the highest percentage decrease in tensile strength in relation to all tested composite samples.

The results show that both the ceramics content in the material and the aging process significantly influenced the values of extrusion at break (Fig. 12). With increasing content of ceramics in PVC matrix the decrease of this property is observed. The highest decrease was observed already at the lowest content of ceramics of 10% in the material and amounted to 64% for specimen A10, 55% for specimen B10 and 52% for specimen C10. Further increase of the ceramic content increased the decrease of elongation. The smallest decrease was observed for sample B, characterized by a smaller ceramic granulation. The aging process also resulted in a decrease in elongation for all types of samples. However, the biggest decrease is observed in specimens: standard – pure PVC and amounts to 21%, and in specimens B10, B20, B30 and amounts to 16%, 33%, 42% respectively in relation to specimens of the same kind before aging process. The lowest decrease of elongation due to aging is observed in samples A20 and A30 and amounts to 2%, which could be influenced by bigger granulation of ceramics and its increased content in the material. The character of changes in the properties studied is similar to that presented in [6], but the values obtained are lower in case of tensile strength and higher in relation to elongation at break, which results from the composite matrix material used.

![Figure 11. Tensile strength of individual specimens before and after aging](image1.png)

**Figure 11.** Tensile strength of individual specimens before and after aging

![Figure 12. Elongation at break of individual samples before and after aging](image2.png)

**Figure 12.** Elongation at break of individual samples before and after aging
Tensile impact strength

The tests of the performed samples clearly showed that the pure PVC samples had the highest Tensile impact strength – their value was 337.03 kJ/m². The influence of UV radiation and gradation significantly influenced the mechanical strength of the extruded samples. After placing the samples in the chamber for 360 hours, their impact value decreased by over 94% and amounted to 20.71 kJ/m². All results of tensile impact strength are shown in the Figure 13.

The addition of ceramic granules to the polymer and the degradation significantly reduced the force needed to destroy the samples during the endurance tests. Polymer-ceramic composites to which a Poraver with a diameter of 0.5–1.25 mm was added (samples A) had much lower tensile impact strength than samples without filler. Analyzing the obtained results for the samples strengthened in the amount of 30%, a decrease 60% was observed in relation to the samples where the amount of filler was 10%. Additionally, the impact of degradation lowered their values even more. The sample strengthened in the amount of 30% after the degradation process had an impact tensile strength value of 11.11 kJ/m², which is a 77% decrease in relation to the same sample without degradation. It can be clearly stated that the size of the ceramic beads and the percentage of their content affect the strength of the composite.

Samples B reinforced with ceramic granules with a diameter of 0.25 mm to 0.5 mm showed a better tensile impact strength than samples A. However, also in this case, the measured values decreased linearly with the increase in the amount of filler. A 39% decrease was measured for the 20% gain samples from those where the amount of gain was 10%. Composite samples with the addition of 30% of filler achieved a decrease in impact tensile strength of 52% compared to those with the lowest amount of filler. After the degradation process, the samples experienced a significant decrease in strength. Taking into account the B30 sample, the value of which after the degradation process was 12.07 kJ/m², a decrease of 93% is applied in relation to the B10 sample not subjected to the destruction process.

The produced composites containing the strengthening of the ceramic beads with a diameter of 0.25 mm to 0.5 mm/PR2 (samples C) showed the highest tested impact tensile strength values compared to the previously described samples. However, also in this case, the measured values decreased in proportion to the amount of composite filler. The samples that were not subjected to the degradation process recorded a decrease by 40% and 56%, respectively, compared to the sample with 10% strengthening. The obtained values for the samples after the degradation process were in the range of 22.08 kJ/m² for those strengthened in the amount of 10% to 14.01 kJ/m² for the samples where the filler addition amounted to 30%. This shows a decrease in tensile impact strength at the level of 37%. No similar dynamic study of polymer-ceramic composites was found in the available literature.

CONCLUSIONS

The aim of the research was to analyze the influence of UV degradation of polymer composites reinforced with ceramic granules on their mechanical properties colorimetry and wettability.
Conducted investigations showed the influence of PVC modification with ceramics on particular properties depending on its type – granulation and content in matrix. An additional significant influence on all the properties studied was exerted by the aging process.

At the same time it should be kept in mind that the aging studies conducted and the resulting changes under the influence of external factors simulate a biological environment, thus the results obtained should be treated as approximate. Available literature concerning the analysis of static and dynamic mechanical properties, colorimetry and wettability of polymer-ceramic composites is limited and insufficient. This creates a field for research conducted by other research centers and for discussions on the interpretation of the results.

The obtained results suggest an extension of the conducted research by an analysis of the influence of the performed modification on the Fourier transform infrared spectroscopy (FTIR), morphology SEM and geometric structure, which will be the subject of another study.

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