

STUDY OF MECHANICAL AND THERMAL PROPERTIES

OF PLASTICIZED PVC-BASED COMPOSITE
MATERIAL FILLED WITH MODIFIED PERLITE

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Abstract

Composites filled with perlite (MP) modified with stearic acid was studied to improve the physico-mechanical, thermal and morphological properties of polyvinyl chloride (PVC)-based material. It is known that surface modification of perlite increases its interaction with PVC polymer and interfacial adhesion. The composites were prepared by thermomechanical mixing in a special laboratory extruder. The mechanical properties of these composites were studied using tensile and three-point bending tests, and the mechanical strength was determined to be up to 13.5 MPa, and the deformation was increased to 204%. Since 5%MP showed high mechanical properties, its structural properties were evaluated by Fourier-transform infrared spectroscopy (FTIR), thermal stability by thermogravimetric analysis (TGA), and morphological structure by scanning electron microscopy (SEM). FTIR analysis showed a small interaction between the polymer material and modified perlite. FTIR analysis showed a small interaction between the polymer material and the modified perlite and TGA results showed that 5% MP content increased the maximum thermal decomposition temperature of the composite to 262 °C. SEM images showed a uniform and good dispersion of the modified perlite in the PVC matrix. Overall, the addition of stearic acid modified perlite as a filler was effective in improving the structural, mechanical and thermal properties of the PVC composites.

Key word:

adhesion, extruder, dispersion, tensile, spectroscopy.

1. Introduction

Polyvinyl chloride (PVC) remains one of the most widely used and versatile thermoplastics because it is inexpensive, chemically resistant, flame retardant, and easy to process.¹ Since unmodified PVC is inherently rigid and temperature sensitive during processing, plasticizers must be added to the PVC polymer matrix to lower the glass transition temperature and provide the flexibility required for cable insulation, flooring, films, hoses, medical devices, etc.²⁻³ Typically, traditional plasticizers are phthalate esters such as di(2-ethylhexyl) phthalate (DEHP) and diisononyl phthalate (DINP), but today, due to environmental and toxicological concerns related to migration and endocrine disruption, researchers and manufacturers are actively searching for alternatives to phthalates and reformulating plasticized PVC composites, focusing more on filler development rather than just plasticizer chemistry.⁴

Adding fillers to plasticized PVC (PVC-P) is a well-known method to reduce costs, improve dimensional stability, and modify the mechanical, thermal, and processing properties of the material.⁵⁻⁶ Minerals (calcium carbonate, dolomite, marble chips, talc), wood and even biological fillers (eggshell) have been shown to alter the tensile strength, stiffness, thermal and rheological properties of PVC-P materials, while high filler content leads to reduced elongation and poor processability due to poor filler-matrix interaction.⁷⁻⁸ Thus, these results highlight the importance of surface chemistry and filler dispersion as a key criterion for filler influence on PVC-based composites and stimulate the search for lightweight and inexpensive mineral fillers with better surface properties.⁹⁻¹⁰ One of the natural mineral fillers is perlite, a hydrated amorphous volcanic aluminosilicate glass characterized by low bulk density, high porosity, thermal insulation, chemical inertness and flammability.¹¹⁻¹² When rapidly heated above 870–900 °C, perlite expands due to the release of structurally bound water

1 Kurańska, M. et al. "Walnut shell filler reinforced polyurethane composites treated with perlite, montmorillonite, and halloysite." PMC (NCBI). URL: <https://www.ncbi.nlm.nih.gov/pmc/articles/PMC8303685/>

2 «A blend of non-phthalate plasticizers for polyvinyl chloride resin compositions.» US Patent 12163007. URL: <https://image-ppubs.uspto.gov/dirsearch-public/print/downloadPdf/12163007>

3 «The Accessible: <https://www.ncbi.nlm.nih.gov/pmc/articles/PMC10214373/>

4 «Plasticizers in PVC: The safety debate between phthalates and non-phthalates.» Accessible at: <https://eureka.patsnap.com/article/plasticizers-in-pvc-phthalates-vs-non-phthalates-safety-debate>

5 «Thermal and Processing Properties of Talc- and Calcium Carbonate-Filled PVC Hybrid Composites». Accessible at: <https://www.academia.edu/26983933/>

6 «The Influence of Fillers on the Chemical, Mechanical and Dielectric Properties of PVC Composites». Accessible at: <https://www.academia.edu/80299478/>

7 «Preparation of Melt-Blended Plasticized PVC Materials Using Eggshell Powder as a Bio-filler». PMC (NCBI). URL: <https://www.ncbi.nlm.nih.gov/pmc/articles/PMC11859243/>

8 «Mechanical and Processing Properties of Plasticized PVC/Wood Composites». Polymers, 16(15), 2204 (2024). <https://www.mdpi.com/2073-4360/16/15/2204>

9 «Novel PVC/carbon fiber/carbon nanotube hybrid filler composites.» PMC (NCBI). URL: <https://www.ncbi.nlm.nih.gov/pmc/articles/PMC9415811/>

10 «Research on plant-filled polymer composites based on highly plasticized PVC.» PMC (NCBI). Accessible: <https://www.ncbi.nlm.nih.gov/pmc/articles/PMC11174432/>

11 «Perlite Concrete: A Review.» IOP Conference Series: Materials Science and Engineering, 1283, 012003. <https://iopscience.iop.org/article/10.1088/1757-899X/1283/1/012003/pdf>

12 «From volcanic popcorn to materials of the future: A critical review of the expanded applications and environmental impacts of perlite.» Sustainability, 17(4), 1454 (2025). <https://www.mdpi.com/2071-1050/17/4/1454>

and transforms into expanded perlite (EP), a low-density cellular material that can be used as a lightweight filler for polymers, cementitious composites, and insulating materials.¹ Upon heating above 870-900 °C, the perlite expands, releasing structurally bound water, and transforms into expanded perlite (EP), a low-density cellular material that can be used as a light filler for polymers, cementite composites, and insulating materials.² However, the hydrophilic nature, irregular shape, and porous structure of perlite lead to poor surface moisture and poor adhesion with the hydrophobic polymer matrix, leading to aggregation of fillers and low productivity.³ To eliminate this problem, various methods for modifying the filler surface have been developed, including processing with organosilanes, hydrophobization with silosane emulsions, polymer coatings obtained by the PECVD method, and welding with a silane binder modified with maleic anhydride. All of this is related to the surface properties of the filler and its polymer matrix. Although there is extensive literature on perlite-filled thermoset and polyolefin composites, as well as on rigid or plasticized PVC systems filled with conventional mineral fillers and modified fillers, the use of surface-modified perlite as a filler in plasticized PVC matrices has not been well studied.⁴ Due to the plasticization effect achieved when treating the perlite surface with polymers in silane and other polymer matrices, and the sensitivity of PVC-P's mechanical and thermal properties to the type, quantity, and interaction of the filler and matrix, there is a clear scientific basis for developing plasticized PVC composites modified with perlite as an inexpensive, low-density, and thermostable alternative to traditional mineral fillers.

This composite material combines the flexibility and machinability of PVC-P with low perlite density, thermal insulation, and fire resistance, expanding the application

range of PVC-based materials in construction, insulation, and lightweight structures. Therefore, the purpose of this work is to develop a modified composite material based on perlite-filled plasticized PVC and to modify the filler surface.

2. Materials and methods

2.1. Materials

Polyvinyl chloride powder with an average molecular weight (Mw) of 64,000 g/mol and a polydispersity index (PDI) of 1.2 was purchased from Navoi. Natural expanded perlite with an average particle size of 50-100 µm was used as a mineral filler and stearic acid with a molecular weight of 284.48 g/mol was purchased from local companies for filler surface modification. Dioctyl phthalate (DOP) plasticizer and calcium stearate heat stabilizer were purchased from Asir Co.

2.2. Modification of perlite with stearic acid

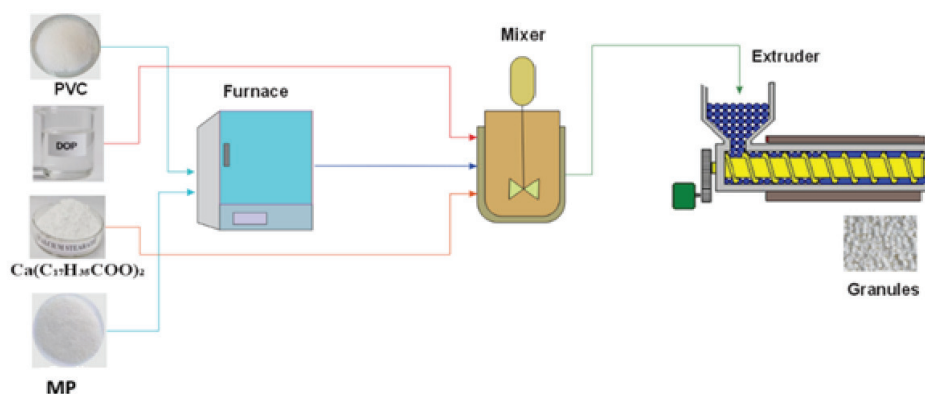
Initially, expanded perlite was dried at 105 °C until constant mass and ground to 10 in a mechanical mill. Organic modification of the perlite surface was carried out with stearic acid up to 10% by mass. Perlite was thermo-mechanically mixed with stearic acid in an extruder at 160 °C. The adsorption of stearic acid molecules on the surface of perlite particles and the coating of the surface with an organic layer occurred. Modified perlite (MP) was used in further experiments as a filler in PVC-based composites.

2.3. Preparation of composite samples

PVC-based composite samples were initially prepared by mixing PVC powder, DOP plasticizer, calcium stearate stabilizer and a specified amount of MP until a uniform mechanical homogeneity was obtained. The mixture was then

Figure 1.

Scheme of preparation of composite granules.



1 "Studies on the Chemical and Physical Properties of Perlite." International Journal of Science, Technology and Management (IJSTM). Accessible: https://www.ijstm.com/images/short_pdf/1460020555_434V.pdf

2 "Lightweight Composite Materials for Protective Pads, Cushioning Pads, Supports, etc., and Methods for their Preparation." US Patent 6756426. Accessible at: <https://image-ppubs.uspto.gov/dirsearch-public/print/downloadPdf/6756426>

3 Yan, Q., Wang, H., Luo, J., Lin, Y., Yang, Y. "Composite hydrophobic modification of expanded perlite." ACI/Concrete.org. Accessible at: <https://www.concrete.org/publications/internationalconcreteabstractsportal.aspx?m=details&i=51733148>

4 "Application of PECVD process in epoxy composites: a novel filler – polymer-coated perlite." ScienceDirect (2023). <https://www.sciencedirect.com/science/article/abs/pii/S1226086X2300802X>

Table 1.

Composition of PVC-based samples with plasticizer and additives (wt%)

№	Samples	PVC:DOP	MP	Calcium stearate
1	Sample 1	98 g (65:35)	0	2 g
2	Sample 2	93g (65:35)	5 g	2 g
3	Sample 3	88 g (65:35)	10 g	2 g

The composite components in Table 1 are prepared in various mass ratios. These compositions were selected for the optimal assessment of the composite material's properties.

melt-mixed in a two-roll laboratory extruder with 4 heating zones. During thermomechanical processing, conditions were created for uniform distribution of MP particles throughout the PVC matrix and for the formation of a good interface.

2.3. Characteristics

The structural and thermo-mechanical properties of the obtained PVC/MP composites were comprehensively studied. To evaluate the mechanical properties, tensile and flexural tests were performed on a universal testing machine [Shimadzu AG-X, Japan] at a speed of 10 mm/min in accordance with ASTM D638 and ASTM D790. The interaction of the PVC matrix with modified perlite (MP) was studied using Fourier transform infrared spectroscopy (FTIR) in the range of 4000–500 cm^{-1} with a spectral resolution of 5 cm^{-1} . The thermal stability of the composites was evaluated using thermogravimetric analysis (TGA) (SHIMADZU DTG-60 thermogravimeter) in an argon atmosphere at a heating rate of 5 $^{\circ}\text{C}/\text{min}$, in the temperature range of 30–600 $^{\circ}\text{C}$. The distribution of MP particles in the PVC matrix was studied using scanning electron microscopy (SEM), and the dispersion, agglomeration and surface area of the particles were determined.

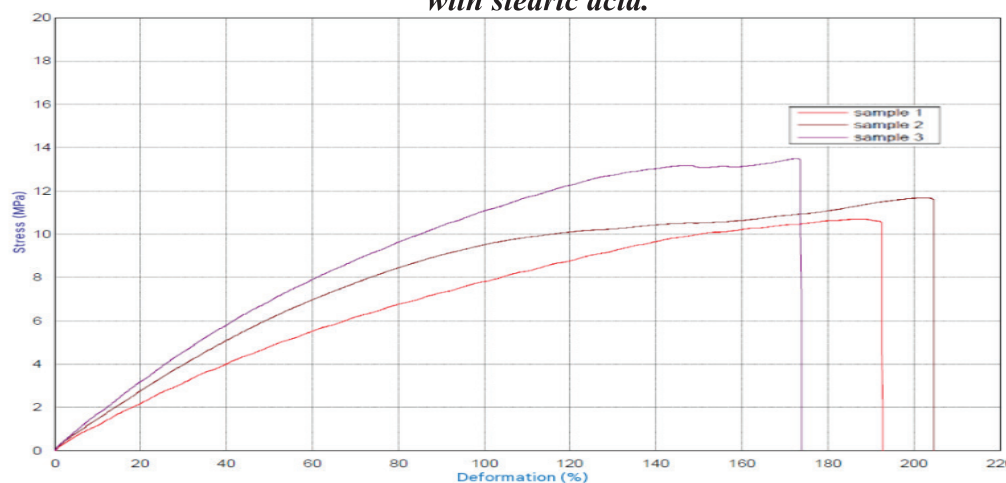
3. Results and discussion

3.1. Mechanical properties

Based on the results of the tensile testing, it can be concluded that the mechanical properties of PVC-based composites are significantly dependent on the amount and dispersion of perlite in the PVC matrix (Fig.1.). As seen from the stress-strain diagrams, a gradual increase in stresses is observed in all samples during the initial stages of deformation, but significant differences are observed in the maximum strength, final deformation, and plasticity of the deformed composites. Sample 1 is a control based on the PVC/DOP/calcium stearate system. Sample 2 is a composite with the addition of 5% perlite (MP) modified with stearic acid, and sample 3 is a composite with the addition of 10% MP. As for the control sample, it shows a fairly stable increase in stress at the initial stages of tensile tests, resulting in a maximum stress of 10.7 MPa. The maximum stress is reached at 190-195% deformation, and then it suddenly fails. This high ultimate deformation is due to the presence of the DOP plasticizer in the PVC matrix. DOP reduces the interactions between PVC macromolecules, increases segmental mobility, and facilitates polymer chain reorientation.⁵

Figure 2.

Tensile stress-strain of PVC/DOP/Ca-stearate composites containing perlite (MP) modified with stearic acid.



⁵ "Lightweight filler-filled polyurethane composites." US Patent 10030126. Accessible at: <https://image-ppubs.uspto.gov/dirsearch-public/print/downloadPdf/10030126>

The addition of MP significantly affects the mechanical properties of sample 2. For example, in sample 2 (with 5% MP), the maximum tensile strength increases to 11.7 MPa (9% compared to the control sample). In addition, the tensile strength at break increases by 204%. It can be assumed that a small amount of MP acts not only as a passive mineral filler, but also as a dispersed phase that facilitates stress transfer. In this case, surface modification with stearic acid changes the surface properties of pearlite particles, improving their wetting and surface adhesion with the PVC matrix. Due to the presence of external loads, part of the stress is transferred from the PVC matrix to the MP particles. As a result, the formation of cracks in the samples is delayed, and the strength and deformation increase. In particular, at 5% MP, the distance between the particles is large enough to prevent their agglomeration.

The composite with 10% MP (sample 3) exhibits the highest maximum strength among all samples. The maximum strength of sample 3 is 13.4–13.5 MPa, which is 25% higher than that of sample 1 and 15% higher than that of sample 2, however, this increase in strength is accompanied by a decrease in the failure stress: sample 3 fails at approximately 173–175% of the ultimate stress. Thus, the addition of 10% MP shifts the strength-strain ratio of the composite towards strength and at higher filler concentrations, the probability of contact and aggregation of pearlite particles increases. These microstructural zones act as stress concentrators during deformation and limit the ability of the polymer chains to reorient and as a result, although the composite can withstand high stresses, the area available for plastic deformation is reduced.⁵

The results of the bending test showed the effect of different amounts of perlite (MP) on the mechanical properties of PVC-based composites (Fig. 3). According to the

indicators, there was no significant difference in bending deformation for all samples, but significant changes in maximum stress were observed. Test sample 1 showed the best mechanical properties among other composite samples. The maximum stress for sample 1 was 4.3–4.4 MPa, this maximum stress value was observed in the range of 20–22% deformation. The high values indicate that DOP and Ca stearate are uniformly distributed in the material and the stress under compression is uniform. When 5% MP is added, the maximum stress decreases significantly to 2.8 MPa, which is about 35% less than for sample 1.

The relatively poor dispersion of mineral fillers from the composite disrupts the particle-matrix interface, resulting in a decrease in strength, but the flexural deformation is almost preserved even at 2% compared to sample 1, which means that the 5% MP filler powder is still uniformly distributed within the material. The maximum stress for sample 3 is very close to the value of sample 2, 2.7–2.8 MPa. This means that increasing the MP content from 5% to 10% also shows that the composite is mechanically preserved. In general, the stress-strain curves for sample 2 and sample 3 are very similar. This indicates that at 5% MP, a significant filler effect was observed, affecting the mechanical properties of the composite, while increasing the filler content to 10% also had a positive effect on the flexural deformation of the material.⁶

3.2. Fourier Transform Infrared Spectroscopy (FTIR)

Figure 4 shows the FTIR spectrum of sample 2 of the composite filled with 5 wt% stearic acid modified perlite (MP). This spectrum reflects the mineral phase, plasticizer/stabilizer components, and characteristic functional groups inherent to modified perlite (MP) in the polyvinyl

Figure 3.

Bending stress-strain of composites.

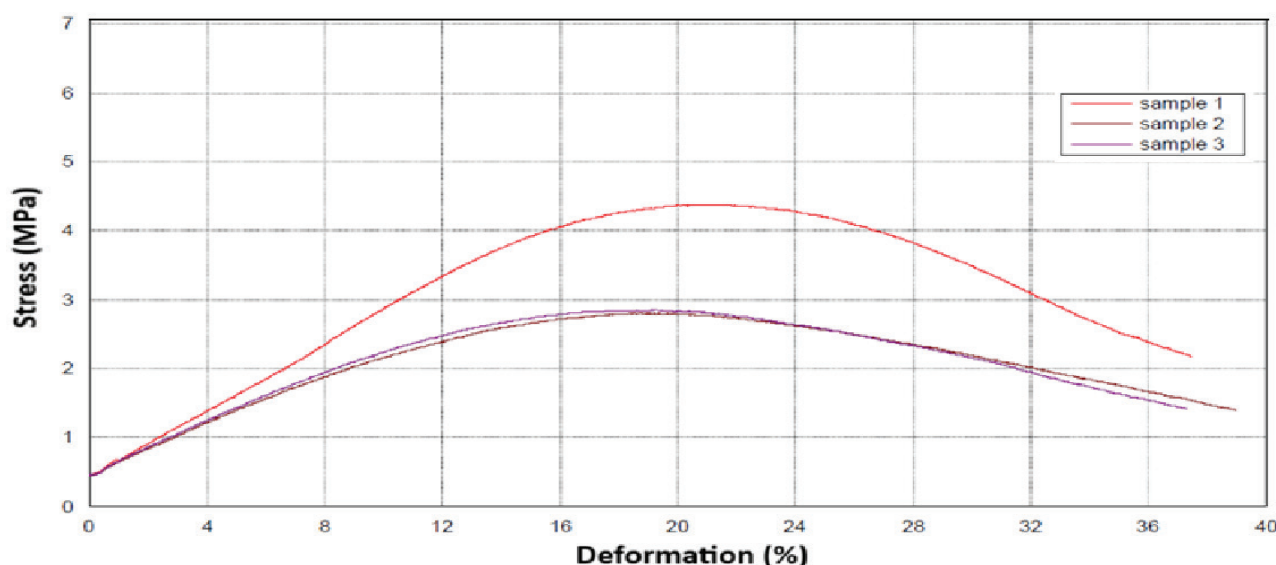
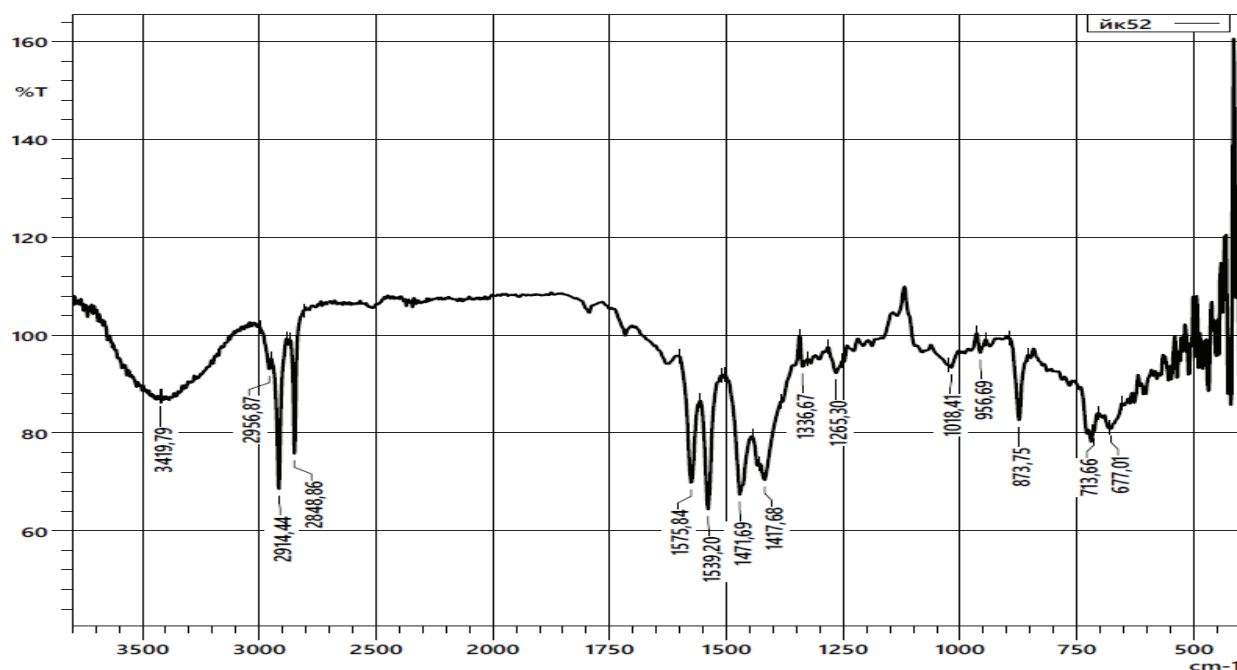


Figure 4.

FTIR spectrum of a PVC/DOP/Ca-stearate composite containing 5 wt% stearic acid-modified perlite (MP).



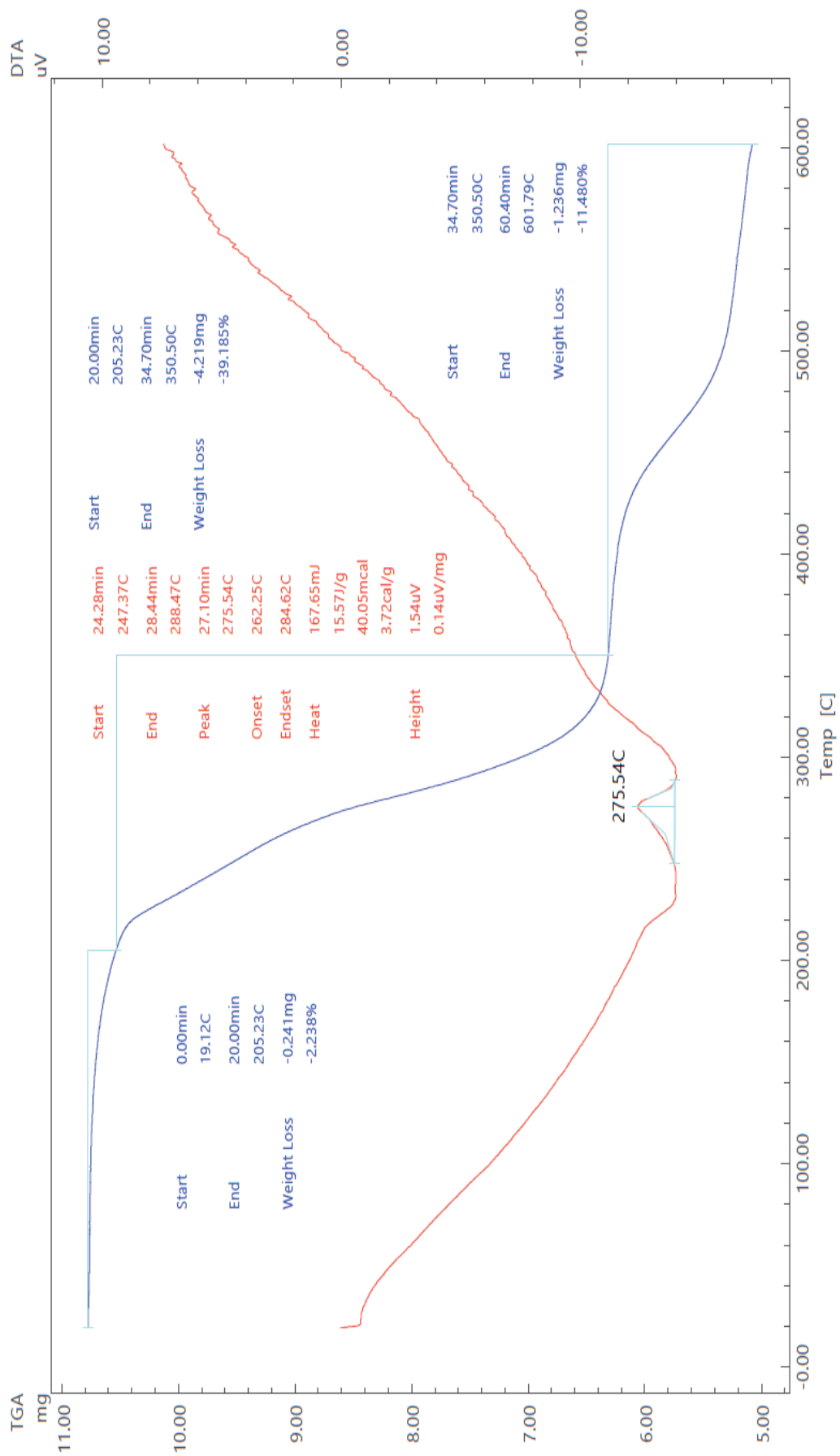
chloride (PVC) matrix. The combination of organic and inorganic absorption bands confirms the existence of a multi-phase PVC composite material and broad absorption band at 3419.79 cm^{-1} is mainly associated with the stretching vibrations of the O-H groups, which may correspond to hydroxyl groups on the surface of the mineral phase or adsorbed moisture. The presence of such a band in the perlite sample modified with stearic acid indicates that the surface modification process did not completely eliminate the polar surface features that could facilitate the interfacial interaction between the modified surface and the surrounding polymer phase.⁷

The spectrum exhibits several sharp absorption bands in the range of $3000\text{--}2800\text{ cm}^{-1}$. The absorption peak at 2956.87 cm^{-1} is associated with the asymmetric stretching vibration of the C-H group, while the absorption peaks at 2914.44 and 2848.86 cm^{-1} correspond to the asymmetric and symmetric stretching vibrations of the aliphatic CH_2 group, respectively. This type of absorption is characteristic of hydrocarbon components in PVC composites, particularly long-chain stearic acid alkyl groups. Therefore, the strong CH_2 stretching vibrations confirm the presence of an organic modifier in the MP-containing composite. The absorption bands in the fingerprint region at 1575.84 , 1539.20 , 1471.69 , and 1417.68 cm^{-1} are attributed to the deformation and skeletal vibrations of the organic components. Specifically, the absorption peak at 1417 cm^{-1} corresponds to the deformation vibration of CH_2 , while the absorption peaks in the $1500\text{--}1600\text{ cm}^{-1}$ range can be attributed to the overlap of vibrations between the polymer component and the surface-bound particles. This phenom-

non is expected given the complexity of the spectral region, as the infrared spectrum shown represents a composite material consisting of polyvinyl chloride (PVC), dioctyl phthalate (DOP), calcium stearate, stearic acid-modified perlite, and the interfacial interactions between them. The absorption peaks at 1336.67 and 1265.30 cm^{-1} correspond to the C-H deformation vibration and C-C/C-O vibration regions that may occur in the organic phase. However, the particularly strong absorption band at 1084.41 cm^{-1} is due to the Si-O-Si and Si-O vibrations of the silicate minerals in the perlite phase. This absorption band is a major spectral feature of the inorganic filler incorporated into the PVC matrix. The additional spectral lines at 956.69 and 873.75 cm^{-1} may be related to Si-O bonds. This indicates that the inorganic structure of perlite is retained after modification and composite material preparation. In the lower wavenumber range, the bands at 713.66 and 671.01 cm^{-1} are characteristic bands of the PVC/mineral composite. The absorption in the $600\text{--}700\text{ cm}^{-1}$ region is attributed to the C-Cl vibration in PVC, although some contribution from mineral lattice vibrations may also exist in this region. Therefore, the coexistence of C-Cl vibrations in PVC and silicate vibrations in perlite indicates the coexistence of polymer and mineral phases. This indicates that the inorganic structure of perlite is retained after modification and composite material preparation. In the lower wavenumber range, the absorption bands at 713.66 and 671.01 cm^{-1} are characteristic absorption bands of the PVC/mineral composite. The absorption in the $600\text{--}700\text{ cm}^{-1}$ region is attributed to the vibrations of C-Cl in PVC, although some contributions from mineral lattice vibrations may also exist in this region.

Figure 5.

TGA/DTA thermograms of a composite containing 5 wt% stearic acid-modified perlite (MP).



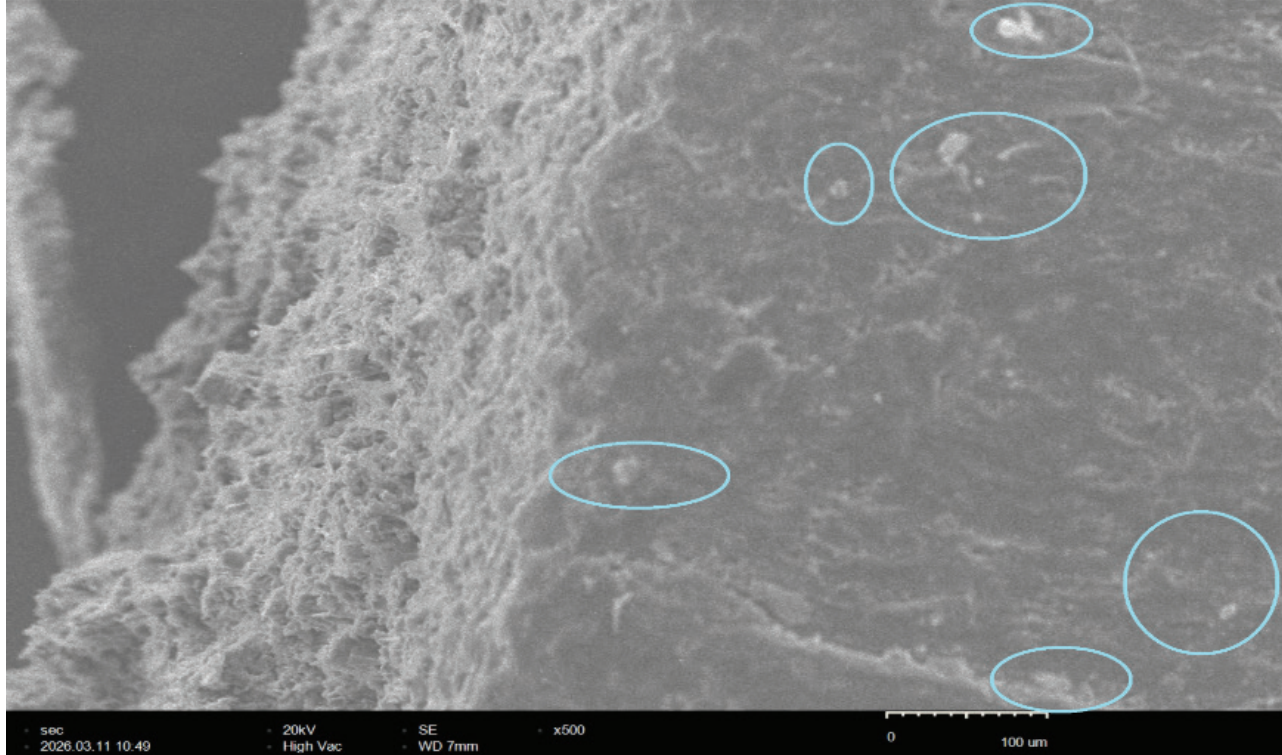


Figure 6.

SEM of a composite consisting of modified perlite (5% MP).

3.3. Thermogravimetric (TGA/DTA) results

In the initial stage, an average weight loss of 0.241 mg (2.238%) was observed in the temperature range of 19.12 to 205.23 °C. This limited weight loss can be attributed to the physical adsorption of water and the evaporation of low molecular weight volatiles; this indicates that the composite material has good thermal stability before significant decomposition occurs. Subsequently, the sample underwent the main decomposition stage in the temperature range of 247.37 to 288.47 °C, with an initial temperature of approximately 262.25 °C. In this stage, the weight decreased significantly to 4.219 mg, which is 39.185% of the initial value.⁴

These results confirmed a clear endothermic reaction with a peak at 275.54 °C due to thermal effects in the DTA curve, which is mainly associated with the early stages of PVC dehydrochlorination, where HCl is cleaved from the macromolecular chains and causes the formation of conjugated polyene sequences. Some of the thermal activity in this range may also be due to the decomposition and evaporation of the stearate and DOP components, and an additional 1236 mg (11480%) is lost between approximately 350.50 and 601.79 °C. The steady mass loss above 350 °C is due to the carbon/polyene residue remaining after PVC dehydrochlorination. It also shows progressive decomposition of other organics and this TGA data is confirmed by the DTA signal, whose calculated thermal effect is approximately 15.57 J g⁻¹ for the main event at 275.54 °C. Next comes the MP material, which introduces a phase rich in inorganic silicates. Unlike the PVC matrix, it does not follow the same decomposition pathway and is therefore part of the fraction that remains thermally stable.

3.4. Morphological analysis

The morphological structure of the PVC composite

sample with 5% MP after mechanical testing was investigated by SEM analysis at a magnification of 500 times. According to Figure 8, the variation of the 5% MP content maintained a smoother and more homogeneous surface of the composite. The presence of some particle clusters was also confirmed under the microscope. If the composite is highly cross-linked, this indicates a greater interaction of the PVC chains. The SEM image below shows a dispersion of many small particles. Another sign of this dispersion is the good adhesion of the particles with the polymer chains. 5% MP was taken as the most suitable sample, since it had good mechanical strength and deformation. Its relatively dense and compact structure was increased by the presence of perlite in its composition.¹¹

4. Conclusion

Tensile and bending tests confirmed that the MP content in the composite material improved the mechanical properties of the material. In particular, it was found that the 5% MP composite significantly improves mechanical strength and deformation; therefore, the FTIR, TGA, and SEM characteristics of the 5% MP composite were studied. The presence of stearic acid on the surface of the FTIR results confirmed a partial interaction between the perlite and the composite, which is formed on the basis of hydrogen bonds. TGA analysis revealed that the addition of MP significantly improved the thermal stability of the composites and delayed the maximum decomposition temperature. It was confirmed that SEM analysis improved the dispersion and interfacial adhesion of MP in the composite. In summary, the obtained MP can be used as a functional mineral filler in PVC-based composites, and the method can actually produce composites with improved performance using relatively inexpensive mineral resources.