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THERMAL AND MECHANICAL PROPERTIES OF COMPOSITE MATERIALS BASED ON PLASTICIZED POLYVINYL CHLORIDE (PVC) FILLED WITH PERLITE

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Annotatsiya. Ushbu tadqiqotda perlit va kaltsiy stearati ($Ca(C_{17}H_{35}COO)_2$ bilan modifikatsiyalangan polivinilxlorid (PVX) kompozit materiallari bo'yicha keng qamrovli tadqiqot o'tkazildi. Materialning kimyoviy tuzilishi, issiqlik xususiyatlari va mexanik xususiyatlari tahlil qilindi. Olingan natijalar shuni ko'rsatadiki, perlit va Ca-stearat qo'shilishi PVX matritsasining strukturaviy barqarorligini sezilarli darajada yaxshilaydi. Termogravimetrik tahlil natijalariga ko'ra, kompozitlar 220–300 °C harorat oralig'ida nisbatan yuqori issiqlik barqarorligini namoyish etdi va parchalanish jarayoni kechikdi. Mexanik sinovlar materialning sinishi paytida mustahkamlikning yaxshilanganligini tasdiqladi. Eng samarali natijalar 5% perlit va 1% $Ca(C_{17}H_{35}COO)_2$ miqdorida qo'lga kiritildi, bu esa optimal muvozanatni ta'minladi. Umuman olganda, modifikatsiya PVX kompozitlarining issiqlik va mexanik xususiyatlarini sezilarli darajada yaxshilaydi.

Kalit so'zlar: perlit, ekstruziya, termogravimetriya, barqarorlik, mustahkamlik, cho'zilish.

Abstract. In this study, a comprehensive study was conducted on polyvinyl chloride (PVC) composite materials modified with perlite and calcium stearate ($Ca(C_{17}H_{35}COO)_2$). The chemical structure, thermal behavior, and mechanical properties of the material were analyzed. The results obtained show that the addition of perlite and Ca-stearate significantly improves the structural stability of the PVC matrix. According to the results of thermogravimetric analysis, the composites exhibited relatively high thermal stability in the temperature range of 220–300 °C, and the decomposition process was delayed. Mechanical tests confirmed the improvement of the strength at break of the material. The most effective results were obtained with an amount of 5% perlite and 1% $Ca(C_{17}H_{35}COO)_2$, which provided an optimal balance. In general, it was found that the modification significantly improves the thermal and mechanical properties of PVC composites.

Keywords: perlite, extrusion, thermogravimetry, stability, strength, elongation.

Аннотация. В данном исследовании было проведено всестороннее изучение композитных материалов из поливинилхлорида (ПВХ), модифицированных перлитом и стеаратом кальция ($Ca(C_{17}H_{35}COO)_2$). Были проанализированы химическая структура, термическое поведение и механические свойства материала. Полученные результаты показывают, что добавление перлита и стеарата кальция значительно улучшает структурную стабильность матрицы ПВХ. Согласно результатам термогравиметрического анализа, композиты демонстрировали относительно высокую термическую стабильность в диапазоне температур 220–300 °C, а процесс разложения замедлялся. Механические испытания подтвердили улучшение прочности материала на разрыв. Наиболее эффективные результаты были получены при количестве 5% перлита и 1% $Ca(C_{17}H_{35}COO)_2$, что обеспечило оптимальный баланс. В целом, было установлено, что модификация значительно улучшает термические и механические свойства композитов ПВХ.

Ключевые слова: перлит, экструзия, термогравиметрия, стабильность, прочность, удлинение.

1. INTRODUCTION.

In recent years, the development of environmentally friendly, energy-efficient and inexpensive building materials has become a pressing issue worldwide. Traditional synthetic polymers, in particular polyvinyl chloride (PVC), are widely used thermoplastic materials, which are widely used in various industries due to their mechanical strength, chemical stability and low cost in the production of pipes, panels, profiles and coatings. However, pure PVC has some disadvantages: it is prone to brittleness, sensitive to temperature changes and requires a lot of energy during processing. Therefore, improving the properties of PVC by modifying it with various natural or mineral fillers is one of the main directions of current research [1].

The natural mineral perlite is a promising filler for PVC. Perlite is an amorphous aluminosilicate of volcanic origin, containing many micropores, which gives it advantages such as low density, good thermal insulation and fire resistance.

The addition of perlite to the PVC matrix allows to reduce the weight of the material, increase thermal stability, improve mechanical strength and effectively use it in the construction industry. At the same time, such composites are considered environmentally friendly and economically acceptable [2;3].

The technology for obtaining PVC-perlite-based composites usually includes the following stages: drying polymer and filler particles, mixing them with a plasticizer in a certain ratio, melting with heat and molding by pressing or

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extrusion. The amount of perlite is selected in the range of 5-10%, since an excess of filler reduces the elasticity of the material. Under optimal conditions, the resulting composites provide high mechanical strength and surface smoothness [4;5].

2. MATERIAL AND METOD.

2.1 Material

Polyvinyl chloride (PVC) in the form of granules (100–200 μm) was supplied by Navoiy Azot. Dioctyl phthalate (DOP, liquid, 99 %) used as a plasticizer was also obtained from Navoiy Azot. Calcium stearate (Ca-stearate, powder, 50 μm) was purchased from local chemical suppliers. Expanded perlite (M-750, 16 mm) was supplied by Blinds, manufactured at the Tashkent factory. All materials were used without further purification or treatment.

2.2. Preparation of composites

Composite samples were prepared by mixing PVC powder, DOP plasticizer, expanded perlite and calcium stearate in different percentages, the process was carried out in several stages (Fig. 1). The PVC compound was first dried in a muffle furnace at 60–70 °C for 1 hour to remove moisture. Then, the PVC was first mixed with the stabilizer in a mixer for 2–3 minutes, and then with the plasticizer (DOP) for 3–5 minutes at 40 °C and 300 rpm. The perlite was dried in the oven, then it was also added to the mixer and the mixture was mixed for 5 minutes at a low speed of 200 rpm, which ensured an even distribution of the perlite. The extrusion process of the mixture was adjusted using a laboratory extruder in the range of 165–170 °C and the screw rotation speed was selected in the range of 20–40 rpm. These parameters ensured uniform melting of the material, stable flow and the formation of a homogeneous composition.

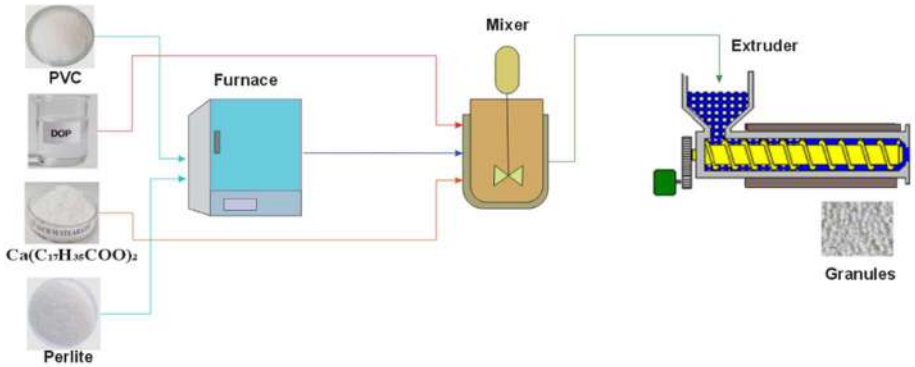


Figure 1. Scheme of preparation of perlite/Ca-stearate/DOP/PVC composite granules.

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Table 1.

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

№	Samples	PVC:DOP	Perlite	Calcium stearate
1	Sample 1	65:35 g	0	2 g
2	Sample 2	65:35 g	5 g	2 g

The composite in Table 1 was prepared with different component mass ratios. These compositions were selected to optimally evaluate the properties of the composite material.

2.3. Fourier Transform Infrared (FTIR) spectroscopy

The chemical and structural properties of the composite materials were studied using Fourier Transform Infrared (FTIR) spectroscopy on a SHIMADZU IRTracer-100. The spectra were recorded in the range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1} and 32 scan sets for each sample.

2.4. Thermogravimetric analysis (TGA/DTG)

The thermal properties of the composites were studied using a SHIMADZU DTG-60 thermogravimetric analyzer. TGA and DTA analyses were performed in the temperature range of 30–600 °C in an argon atmosphere, at a heating rate of 10 °C/min and a gas flow rate of 50 ml/min. These analyses allowed us to evaluate the thermal stability and degradation properties of PVC-based composites.

2.5. Mechanical stretching

The mechanical strength and tensile deformation of the samples were determined using AGS-X, 10N-10kN. The ratio of the thickness, width and length of the sharp test zone of the samples was 2:5:80 mm. At least three copies of each sample were evaluated using a universal testing machine (Shimadzu AGS-X series universal testing machine) at a speed of 10 mm/min.

3. RESULTS AND DISCUSSION.

3.1. FTIR measurement

Analysis of the FT-IR spectrum of pearlite (4000–450 cm^{-1}) revealed several characteristic absorption peaks. A broad and strong maximum at 3373.50 cm^{-1} is attributed to silanol (Si–OH) on the pearlite surface. In the low wavenumber region (1300–450 cm^{-1}), significant absorption peaks are observed, which are characteristic of the aluminosilicate framework of pearlite. The location of this peak around 1005 cm^{-1} is related to the aluminum content of pearlite. The two shoulder-like maxima at 1170.79 and 1136.07 cm^{-1} are probably related to

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other vibrational modes of Si–O. The relatively weak peak at 692.44 cm⁻1 is related to the deformation vibrations of the Si–O–Si or Al–O–Si bonds (Fig. 1). The spectrum does not show the sharp peaks characteristic of crystalline quartz.

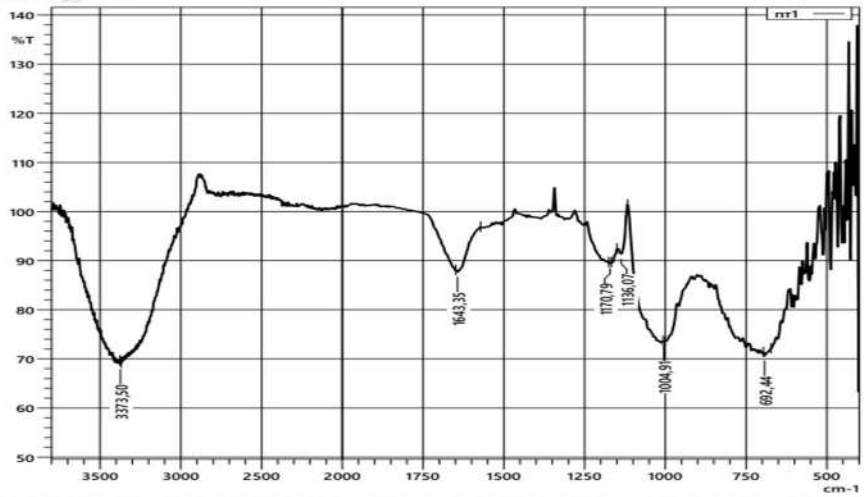


Figure 1. The chemical structures of perlite.

When analyzing the FT-IR spectrum of polyvinyl chloride (PVC) plasticized with dioctyl phthalate (DOP) plasticizer and calcium stearate ($Ca(C_{17}H_{35}COO)_2$) stabilizer, two strong peaks at 2914.44 cm^{-1} and 2848.86 cm^{-1} in the high wavenumber region are due to the asymmetric and symmetric stretching vibrations of the methylene (CH_2) groups. They originate from the PVC chain and the alkyl moieties of DOP. The broad, weak peak at 3385.07 cm⁻1 is the hydroxyl (O–H) vibration caused by the moisture adsorbed by calcium stearate. The most important diagnostic peak is the strong maximum at 1716.65 cm⁻1, which is the stretching vibration of the ether carbonyl (C=O) group in the DOP plasticizer. The weak peak at 1625.99 cm⁻1 belongs to the carboxylate group of calcium stearate. The moderately intense peaks at 1575.84 and 1539.20 cm⁻1 are C=C stretching vibrations of the aromatic ring of DOP (characteristic for phthalates). 1471.69 cm⁻1 is a CH_2 scissor vibration, and 1417.68 cm⁻1 may be a C–H deformation vibration or a symmetric vibration of the carboxylate (Figure 2).

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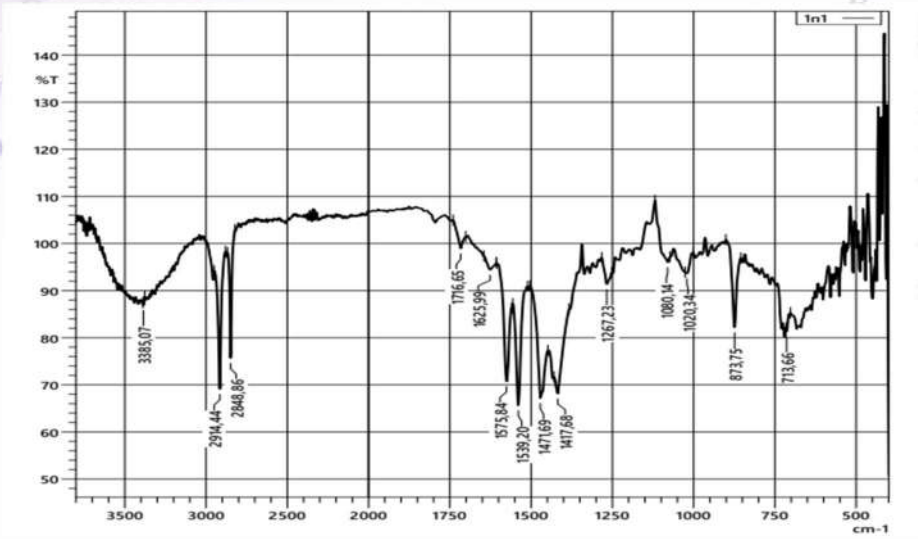


Figure 2. FT-IR spectrum of plasticized polyvinyl chloride (PVC), perlite and calcium stearate ($Ca(C_{17}H_{35}COO)_2$ composite.

The composite spectrum clearly confirms the presence of hydrogen bonding (Si–OH··O=C) between pearlite and DOP and its preservation in the component.

3.2. Thermogravimetric analysis (TGA/DTG)

This TGA–DTA plot represents the thermal stability and degradation processes of the sample (Fig. 3). The first weight loss in the TGA curve (blue) is observed in the range 23–213 °C, which is mainly due to the release of moisture and volatiles (approximately 3.3%). The second major degradation occurs in the range 213–366 °C, showing a mass loss of 37%, which corresponds to the main thermal degradation of the polymer chains. An additional 12% mass loss is observed in the range 366–601 °C, which indicates the decomposition stage of the inert residue or fillers.

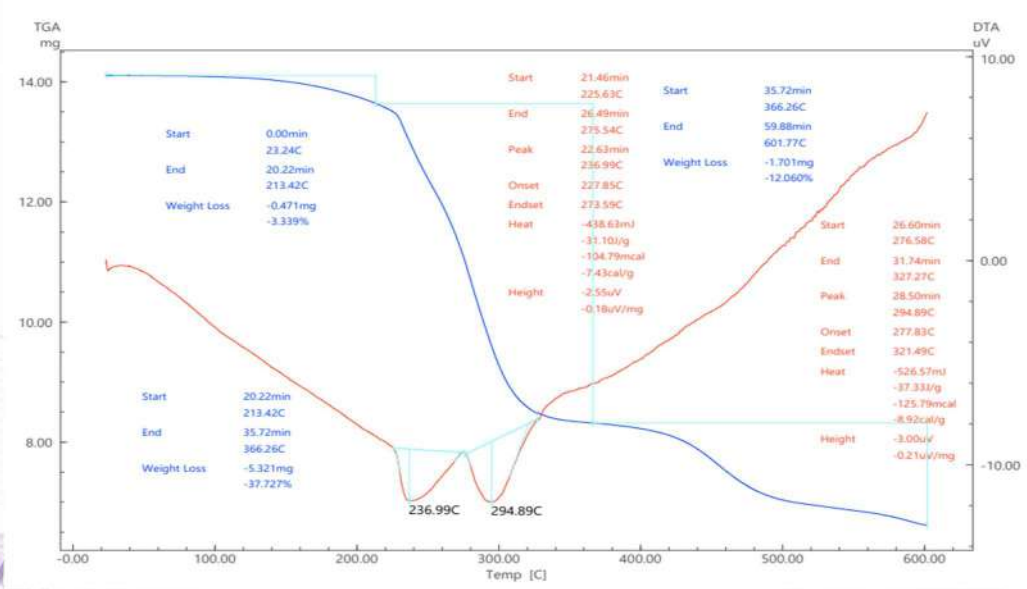


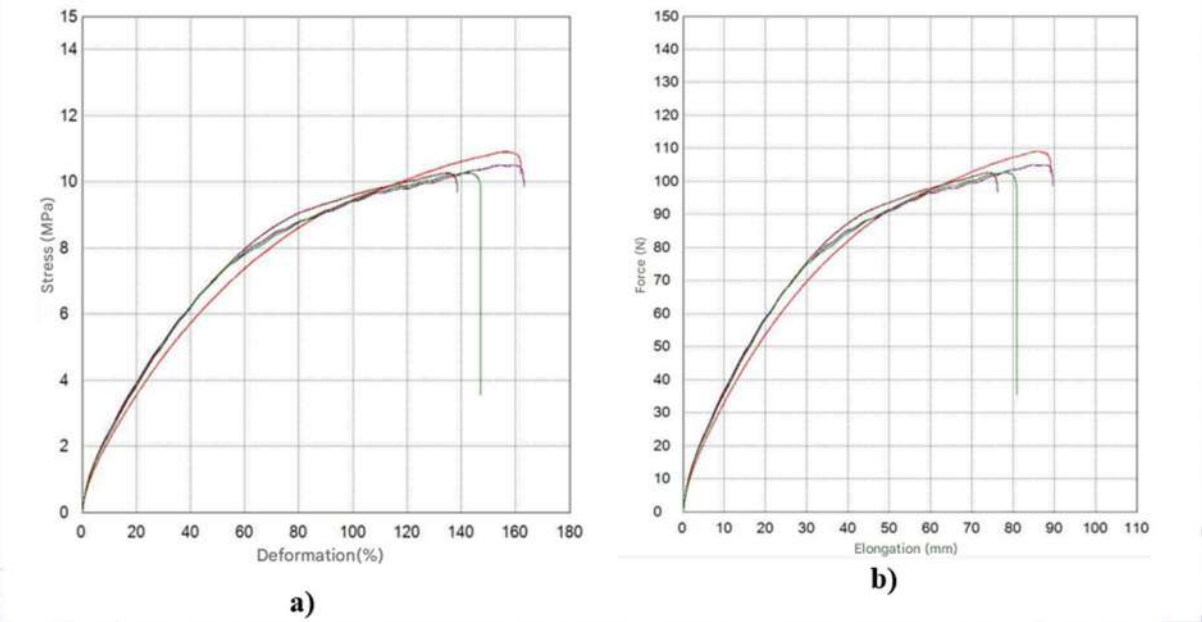
Figure 3. TGA/DTG plots of plasticized polyvinyl chloride (PVC), perlite and calcium stearate ($Ca(C_{17}H_{35}COO)_2$ material.

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The DTA plot (red line) represents the endothermic and exothermic reactions. An endothermic peak is observed around 236 °C, which indicates the softening or melting stage of the polymer. The strong exothermic peak in the range of 236–294 °C corresponds to the main degradation and energy release process. In general, the stability of the sample increased sharply above 294 °C, indicating its thermal sensitivity. Overall, the TGA–DTA results show that the material is relatively stable at low temperatures, but that the main thermal decomposition process begins in the range of 200–300 °C. In this range, intensive degradation of the polymer chains occurs, resulting in significant mass loss. At temperatures above 300 °C, the structural stability of the material decreases rapidly and additional decomposition steps continue. The results confirm the increase in overall thermal resistance.

3.3. Mechanical elongation

The total length of all samples was 80 mm, and they were fixed to the grips during the test, the test length was 55 mm, the width was 5 mm and the thickness was 2 mm, each sample was tested 2 times. The device displayed the results in graphs after the tests (Figure 3): one shows the relationship between force (N) and elongation (mm) (Figure 3a), the other shows the relationship between stress (MPa) and deformation (%) (Figure 3b). These graphs were used to determine the elongation property, mechanical strength and elastic modulus of the material.



a) b)

Figure 3. Mechanical properties of PVC-based composites: α-stress-strain and β-pressure-strain relationships.

We have visually analyzed the stress-strain properties of the materials in Figure 3a. The first sample consists of 35% plasticized PVC, and as can be seen from the graph, the initial stress is low, the strength increases gradually and increases proportionally with elongation. In this sample 1, the maximum force is

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approximately 104.5-109.4 N, and the elongation reaches 87-90 mm, which indicates that PVC is very elastic. The second sample, the maximum force reached 100.9-101.3 N, and the elongation is 75-81 mm. In this graph, this sample is almost 1.11 times less elastic than the first sample, and the stabilizer increases the resistance to tensile stress.

Pressure (MPa) and deformation (%) It is useful to study the elastic and strength properties of the materials in Figure 3b. The maximum stress of the first sample reaches approximately 10.4-11.2 MPa, and the elongation is 161-163%. This shows the basic elastic properties of PVC. The maximum tensile strength of sample 2 is reduced to 10.2-10.4 MPa and the elongation is reduced to 138-149%.

These results represent the general mechanical properties of PVC-based composites, with perlite partially reducing the mechanical strength and elasticity of the material.

4. CONCLUSION.

In conclusion, the physicochemical, thermal and mechanical properties of PVC-based composite materials modified with perlite and calcium stearate were comprehensively evaluated. The results of the study showed that the effective distribution of perlite particles in the PVC matrix and the modification with Ca-stearate led to improved interfacial adhesion. TGA analyses confirmed that the thermal stability of the composite increased and the decomposition process shifted towards higher temperatures. Mechanical tests showed a partial decrease in the strength and deformation properties of the composite. In particular, the most optimal results were observed in the sample containing 5% perlite and 1% $Ca(C_{17}H_{35}COO)_2$, and a balance was achieved between thermal stability and mechanical performance. In general, the modification based on perlite and calcium stearate was confirmed to be an effective method for improving the operational properties of PVC composites.

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