EURASIAN PHYSICAL TECHNICAL JOURNAL

2025, Volume 22, No. 1 (51)

https://doi.org/10.31489/2025N1/28-36

Received: 05/11/2024 Revised: 10/02/2024 Accepted: 18/03/2025 Published online: 30/03/2025

Research Article

UDC 537.622.3:620.193.4:699.15

IMPACT OF POLYETHYLENE TEREPHTHALATE ON THE MECHANICAL PROPERTIES OF POLYIMIDE FILMS

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Abstract. *This study focuses on analyzing the influence of varying concentrations of polyethylene terephthalate filler on both the tensile strength and elongation at break properties of polyimide films when subjected to uniaxial tension, aiming to determine how polyethylene terephthalate filler impacts the mechanical performance and durability of these films under stress. The mechanical behavior of polyimide films and their homogeneous composites with polyethylene terephthalate under uniaxial tension were investigated. The samples were prepared by mechanically mixing polyimide varnish solutions with polyethylene terephthalate, which serves as a reinforcing filler. The results demonstrate that, for all sample types, an initial mechanical load until induces a sharp ~3% increase in relative elongation, attributed to the rotation of globules and alignment of matrix macromolecules into extended chains along the load direction. The additive dependence of viscosity observed in the study further supports that no chemical bonds are formed between the polyimide and polyethylene terephthalate macromolecules, and that no conformational changes take place within the composite.*

Keywords: polyimide, polyethylene terephthalate, filler, uniaxial tension, IR spectroscopy, viscometry.

1. Introduction

The mechanical properties of polymers and polymer-based materials, such as chain flexibility, crystallinity, and intermolecular interactions, play a key role in determining their overall characteristics. These factors directly affect essential mechanical parameters, including deformation behavior and tensile strength [1-5]. In polyimides, the arrangement of macromolecular chains is governed by the imide groups within the aromatic backbone, contributing to a specific degree of crystallinity [6-8]. Disordered segments contribute to the amorphous phase, leading to a strong dependence between the polymer's properties and its structural organization. The mobility of chain segments, especially under external forces, also plays a significant role in determining mechanical behavior [9].

It is well known that polymers undergo chemical reactions under mechanical stress, leading to the disruption of structural order and affecting the extend, to which this order is preserved. Under mechanical impact, chemical bonds within the main polymer chain can break, resulting in mechanical degradation [10–13]. Among all polymers, polyimides exhibit the highest thermal stability, enduring temperatures up to 673 K while retaining flexibility even at liquid nitrogen temperatures [14]. This remarkable property is attributed

to the presence of lone electron pairs on the heteroatom in the ring (nitrogen) and highly electronegative atoms (oxygen in carbonyl groups) [15]. At room temperature, polyimides exhibit high tensile strength (150–180 MPa) with an elongation at break of 70–90%, along with excellent electrical insulation, chemical resistance, and fire resistance [16].

The mechanical properties of polymer composite materials are largely determined by both the type and concentration of the filler [17–20]. According to [21], when fillers are introduced into a polymer solution through uniform mixing, they become evenly distributed within the polymer matrix, forming a molecular-level dispersion. This principle was fundamental in selecting fillers for the present study.

Different fillers influence the polymer matrix in distinct ways, affecting its physicochemical properties. Both the nature and content of the fillers significantly impact the strength and flexibility of polymer composites [22–27]. Research indicates that tensile strength is closely linked to factors such as cross-link density, crystallization tendency, filler characteristics, and the initial molecular weight of the polymer [18, 28]. However, the role of fillers in shaping the physico-mechanical properties of polymer composite materials (PCMs) and the mechanical and functional behavior of newly engineered polymer-based products remains insufficiently explored. A deeper understanding of these effects is critical for enhancing the durability of technological systems and optimizing industrial processes aimed at fine-tuning material properties.

The objective of this study is to investigate the effect of polyethylene terephthalate (PT) filler concentration on the tensile strength and elongation at break of polyimide films under uniaxial tension. The results of this study highlight the practical significance of incorporating polyethylene terephthalate fillers into polyimide composites, providing valuable insights for future material development.

2. Materials and methods

Polyimide (PI) films and their homogeneous compositions with polyethylene terephthalate (PT) filler were selected as the primary objects of study. The matrix polymer used was polyimide, a well-known member of the family of cyclolinear heterocyclic polymers. Polyimides belong to the class of rigid-chain, heat-resistant polymers with a high glass transition temperature, typically ranging from 600 to 800 K. Their macromolecular structure includes periodically repeating bulky polar groups (–O–C_6H_4–), which enhance intermolecular interactions—a key characteristic of rigid-chain polymers. These polar groups are integral components of the macromolecular structural unit, as shown in Fig. 1 [29].

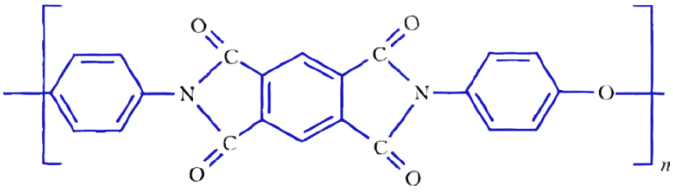


Fig.1. Structural Unit of a Polyimide Macromolecule [29]

The distinctive molecular architecture and strong intermolecular forces give polyimides exceptional thermal stability, making them highly suitable for demanding applications in high-temperature environments. Additionally, they exhibit resistance to radiation and weak acids, along with excellent electrical insulation properties [30]. Besides these advantages, polyimides maintain strong mechanical performance, retaining their strength across a wide temperature range.

2.1. Synthesis of the Polymer Composite

Composite samples were synthesized by mechanically mixing polyimide varnish with solutions containing various concentrations of fillers. The polyimide varnish used was a polyamic acid solution in dimethylformamide, produced by "Estrocom" (Russia).

A systematic approach was applied in the preparation of composite films: first, the mass of the filler corresponding to the desired concentration was measured. The mixture of polyimide varnish and polyester resin in a specific ratio was placed in a three-neck flask, where an inert gas atmosphere was maintained over the solution to prevent oxidation. The mixture was continuously stirred while gradually heating to 170°C.

The resulting viscous solution was then diluted with m-cresol to a concentration of 7%, followed by the introduction of the calculated amount of filler. Stirring continued for another two hours at 170°C to ensure homogeneity. The prepared composite mixture was poured onto a clean glass substrate, and the films were carefully spread and dried in a muffle furnace at 100°C. The resulting films were cut into parallelepiped-shaped samples, each with a working area of 50×50 mm. The thickness of pure polyimide films was 35 μm, while the thickness of composite films ranged from 40 to 140 μm.

As the reinforcing filler, polyethylene terephthalate (PT, also known as lavsan) with the chemical formula $(C_{10}H_8O_4)_n$ was selected. PT is a synthetic polymer containing a terephthalate group in its molecular structure. The structural unit of the polyethylene terephthalate macromolecule is shown in Figure 2 [31]. PT contains polar –O–CO– groups, which are evenly distributed along the polymer chain. These groups enhance intermolecular interactions, contributing to increased rigidity of the material. Compared to polyimide macromolecules, PT molecules exhibit a higher degree of structural crystallinity. The synthesized composite material contained PT fillers at concentrations of 0.25 wt.%, 0.50 wt.%, 2.00 wt.%, 5.00 wt.%, 10.00 wt.%, 15.00 wt.%, and 20.00 wt.%.

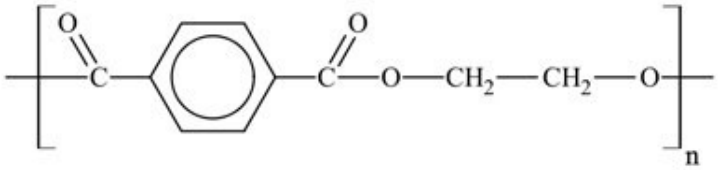


Fig.2. Structural unit of the polyethylene terephthalate (PT) macromolecule [31].

2.2 Samples Characterisation

Mechanical tests of the composite films were conducted using an Instron 5982 tensile testing machine (Instron, USA, manufactured in 2018) at a crosshead displacement speed of 0.05 mm/min [32]. The samples were secured using specialized clamps that ensured stable attachment to the machine's sliding mechanisms [32]. Mechanical loading was applied in a uniaxial tensile mode until the samples reached complete failure. All experiments were conducted under controlled conditions, with a constant load at a temperature of 20±2°C and a relative humidity of 45±5%. The measurement error for both force and film deformation was within ±0.5% of the recorded values. The study of mechanical properties followed GOST R 50583-93 (Russia) and ASTM D 3039/D 3039M - 00 (USA) standards.

The effect of the filler on changes in the polymer matrix's macromolecular structure was analyzed through FTIR spectroscopy, intrinsic viscosity measurements, and surface microphotography of the samples.

FTIR spectra of the samples were recorded using a Jasco IR-810 spectrophotometer (Japan) under standard conditions (20±2°C) within the wavenumber range of 400–4000 cm^{-1}.

Intrinsic viscosity of the polyimide solutions in m-cresol was measured using an Ubbelohde viscometer (Cannon Instrument Company, USA, model 2020), operating in a kinematic viscosity range of 6.0 to 30 m^2/s. The viscosity was determined at 20°C with a solution concentration of 0.5 dL/g.

Surface microphotographs of the samples were taken using a Jeol-ARM100F microscope (Japan) at 200x magnification. The visualization was performed on thin films cast from 7% m-cresol solutions, allowing for a detailed representation of the morphology of the synthesized composites.

3. Results and discussion

The results of experiments illustrating the change in relative elongation of the pure polyimide film without filler under various mechanical loads are presented in Fig. 3 (curve 1). Initially, in the applied load range σ = 0 to 1 MPa, there is a slight increase in relative elongation, up to about 3%. This can be explained by the development of elastic deformation, during which the globules and macromolecules inside the polymer rearrange into stretched chains in the direction of the applied load. As the load increases from 1 MPa to 40 MPa, the relative elongation (ε) of the polyimide film gradually increases. In this load range, the macromolecular chains straighten, aligning and stretching along the flow direction of the polymer matrix, forming fibrous structures. This behavior aligns with previous studies [29–31]. When the applied load exceeds 40 MPa, molecular bonds within the polyimide—particularly those between benzene rings—begin to break, resulting in structural degradation of the polymer. This process leads to a notable increase in

relative elongation, ultimately causing film rupture at a stress level of approximately σr ≈ 70 MPa, with a corresponding relative elongation of εr ≈ 45%. The addition of 2 mass.% PT filler to the polyimide matrix significantly modifies the film's plasticity and mechanical properties. Like pure polyimide, the composite material demonstrates a linear increase in relative elongation in the load range from 1 MPa to 40 MPa (Fig. 3, curve 2). However, the elongation degree of the composite is noticeably higher. At an applied load of 40 MPa, the composite reaches a relative elongation of 30%, which is 26% better compared to the pure polyimide film (Fig. 3, curve 1).

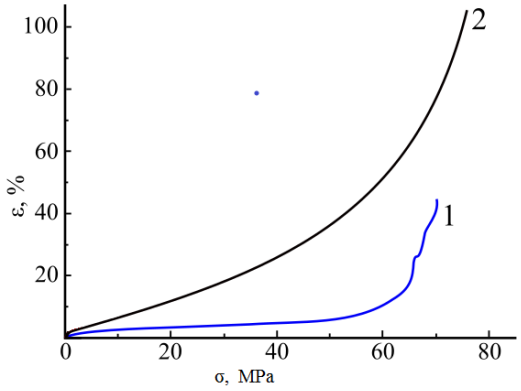


Fig. 3. The dependence of relative elongation (ε) on mechanical load (σ) for pure polyimide film (curve 1) and polyimide composites with PT filler (curve 2), measured at room temperature.

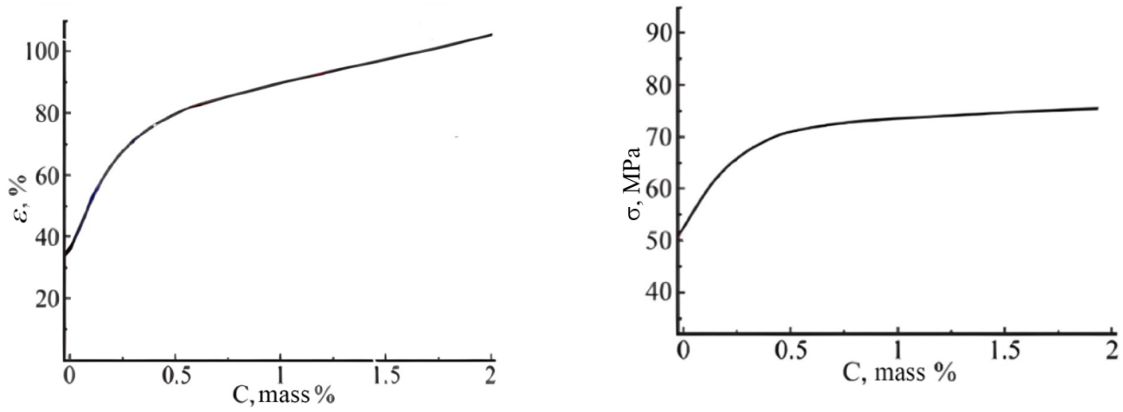
At loads exceeding 40 MPa, the relative elongation of the polyimide composite (PCM) sharply increases, ultimately leading to failure. Notably, the composite material demonstrates superior mechanical characteristics compared to pure polyimide: the strength limit of the composite reaches 80 MPa, and the ultimate elongation reaches 110% (Fig. 3, curve 2).

This improved mechanical behavior of the PCM can be explained by the molecular structure of the PT filler. PT macromolecules have regularly spaced polar groups (–O–CO–) along the polymer chain, which enhance intermolecular interactions both within the polyimide matrix and between polyimide and PT molecules. These interactions increase the rigidity and plasticity of the composite, leading to the observed improvement in both tensile strength and elongation at break.

Increasing the PT filler concentration to 5 mass.% results in a further significant increase in the plasticity of the composite material, with relative elongation (**ε**) reaching 250% without any noticeable changes in tensile strength. A distinctive feature of the ε = ε(σ) dependence in this case is that it reflects the behavior observed in pure polyimide films (see Fig. 3, curve 1). This indicates that the primary mechanisms determining the mechanical properties of this composite material remain the same as those of pure polyimide, despite the presence of the filler. However, the increased plasticity of the polyimide composite with PT filler is primarily due to the reinforcing effect of the filler, which strengthens the intermolecular interactions within the matrix and between the polyimide macromolecules and the filler.

Figure 4 shows the concentration-dependent changes in the plastic and strength properties of polyimide composite materials (PCM) containing PT filler. Changes in the physical-mechanical properties of the composite can be explained by the formation of boundary layers between the polyimide matrix and the PT filler molecules. Adding 0.5 mass.% PT to the polyimide matrix leads to a significant increase in both plasticity (Δα = 45%) and tensile strength (Δσ = 20 MPa) compared to pure polyimide. For the composite material with 2 mass.% PT filler, the elongation at break (εr) reaches approximately 78%, compared to ~37% for pure polyimide (C = 0 mass.%), with a corresponding strength limit Δσr ≈ 72 MPa (Fig.4).

Increasing the PT filler concentration to 5 mass.% leads to a noticeable improvement in the plasticity of the composite: the elongation at break (εr) reaches 250% (Fig. 4a), while the tensile strength remains stable around 75 MPa (Fig. 4b). This stabilization suggests that the reinforcing effect of PT is most pronounced in improving the plastic properties of the material without significantly altering its strength beyond a certain filler concentration.



a) b)

Fig.4. a) Effect of PT filler concentration (C) on elongation at break (ε) of polyimide composite materials at room temperature. b) Effect of PT filler concentration (C) on tensile strength (σ) of polyimide composite materials at room temperature.

The observed additive viscosity dependence on the filler concentration suggests the absence of chemical bonding between the polyimide (PI) and PT macromolecules, as well as significant conformational changes (Fig. 5). As the PT concentration increases, the polyimide composite film acquires a more porous structure.

Figures 6 (a, b) show microphotographs demonstrating the microscopic structure of polyimide composite films with PT filler concentrations of 1 wt.% (a) and 10 wt.% (b). In the composite with 10 wt.% PT (Fig. 6b), the pore sizes are noticeably larger, reaching 40 nm, compared to the composite with 1 wt.% PT (Fig. 6a). At PT concentrations above 10–12.5 wt.%, thermodynamic incompatibility between the polymers becomes evident, leading to the formation of opaque film samples.

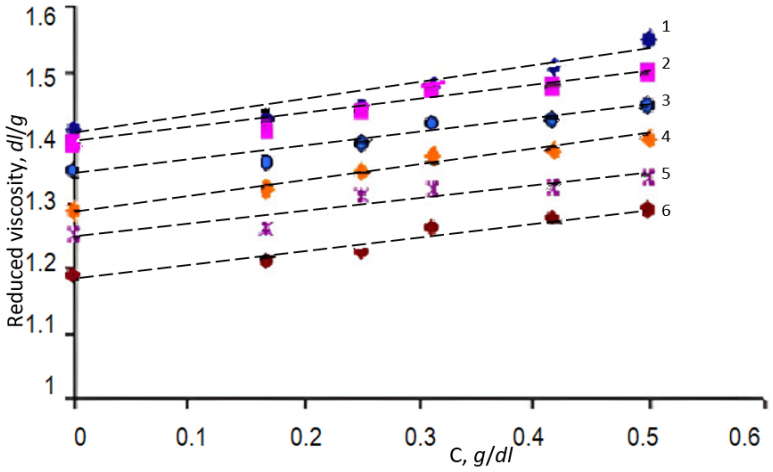
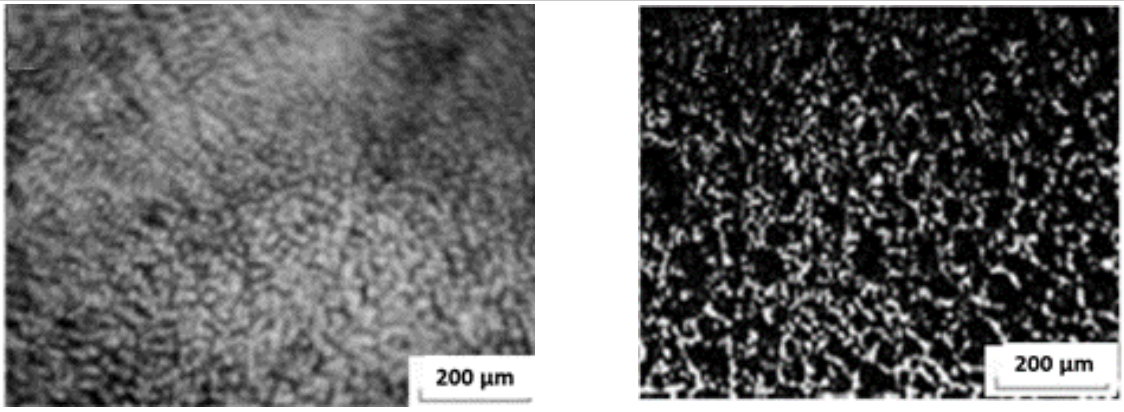


Fig.5. Change in the relative viscosity of polyimide composite material as a function of PT filler concentration (C): Line 1 – 5 wt.% PT; Line 2 – 10 wt.% PT; Line 3 – 15 wt.% PT; Line 4 – 20 wt.% PT; Line 5 – 25 wt.% PT; Line 6 – 30 wt.% PT.

To further verify the results of viscosimetric studies, infrared (IR) spectroscopic investigations were conducted on the composite materials (Figures 7 and 8). The influence of the PT filler and uniaxial mechanical load on the molecular configuration of the polyimide film after deformation was studied. The following maxima in the IR spectrum of the polyimide film after rupture were identified: 446.5; 492.5; 525.6; 605.7; 659.9; 728.3; 797.5; 835.8; 872.9; 950.3; 1008.2; 1065.5; 1107.5; 1161.7; 1252.4; 1297.5; 1378.8; 1508.2; 1626.0; 1721.6; 1771.1; 1899.0; 2028.1; 2578.8; 2950.7; 3053.0; and 3466.3 cm^{-1}.

The peak at 3466.3 cm^{-1} corresponds to the stretching vibrations of hydroxyl (OH) groups, indicating that during film rupture, atomic interactions weaken, leading to the formation of OH bonds as individual molecular bonds break. Additionally, the peak at 1297.5 cm^{-1} is associated with secondary, weak deformation vibrations of the OH group. After rupture, the intensity of this peak decreases, reflecting the weakening of these bonds as a result of material failure.



a) b)

Fig.6. Microscopic structure of polyimide composite films with PT filler content: a) 1 wt.% and b) 10 wt.%.

The presence of hydroxyl groups in the polyimide macromolecule is further confirmed by the appearance of absorption bands corresponding to C–O stretching vibrations at 950.3, 1008.2, 1161.7, and 1508.2 cm^{-1} [37]. Deformation vibrations of Car–H bonds in the aromatic ring, observed in the 600–900 cm^{-1} range, reveal structural changes after rupture: the peak at 728.3 cm^{-1} indicates bond weakening, while peaks at 797.5 and 872.9 cm^{-1} suggest enhanced bonding. The peak at 797.5 cm^{-1} corresponds to m-displaced deformation vibrations of Car–H bonds.

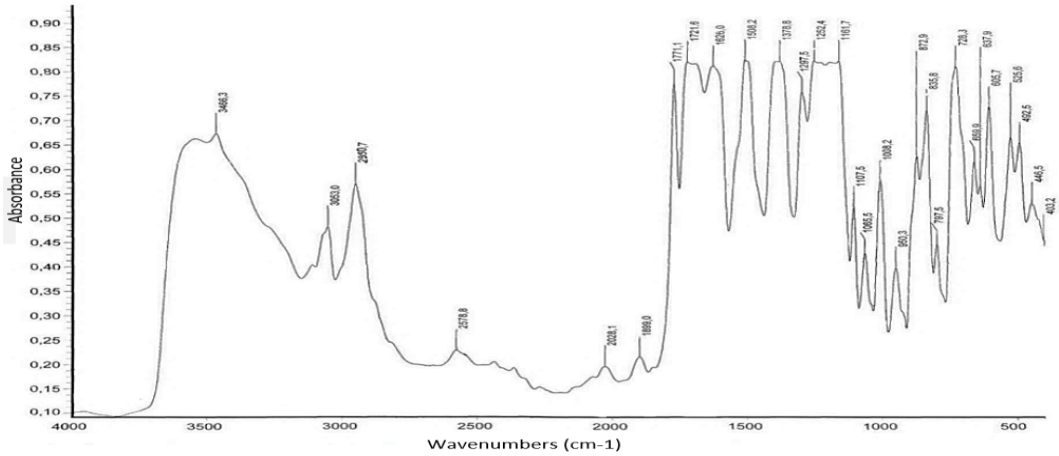


Fig.7. IR spectrum of polyimide film after film rupture

Film rupture also leads to the formation of new Car–H bonds, as evidenced by the emergence of a peak at 835.8 cm^{-1} (m-displaced deformation vibrations), as well as peaks at 3053.0 cm^{-1} and its overtone at 1899.0 cm^{-1}. The presence of these deformation vibrations in the Car–H bonds and aromatic ring after rupture suggests that some polyimide macromolecular bonds break, while others either weaken or strengthen. Film stretching weakens atomic bonds, and its rupture results in macromolecular chain shortening, leading to the formation of discrete structural units. Consequently, Car–H bonds strengthen, increasing their bond energy [31].

Fig. 8 shows the IR spectrum of polyimide containing 2 wt.% of PT filler, subjected to tensile stress until rupture. The following maxima were observed: 412.4, 439.2, 491.1, 523.3, 601.9, 636.3, 658.8, 700.0, 727.9, 797.6, 836.3, 879.1, 892.8, 920.1, 946.1, 1001.7, 1062.2, 1107.9, 1296.2, 1361.5, 1513.5, 1727.9, 1770.1, 1896.6, 2030.4, 2272.6, 2321.7, 2361.8, 2436.1, 2578.7, 2876.2, 2953.1, 3050.9, 3110.5, and 3587.0 cm^{-1}. The influence of the PT filler on the polyimide matrix manifests in the deformation processes (Figs. 4 and 5) and the resulting changes in the IR spectra of the deformed films (Figs. 7 and 8). Specifically, in the range of 400–1700 cm^{-1}, an increase in spectral intensity by up to 40% is observed, while in the range of 1700–3500 cm^{-1}, the increase is approximately 70% compared to the spectra of deformed pure polyimide films. These intensity changes occur without any noticeable shifts in the previously existing absorption bands, suggesting that the PT filler induces structural modifications within the polyimide matrix [33, 34].

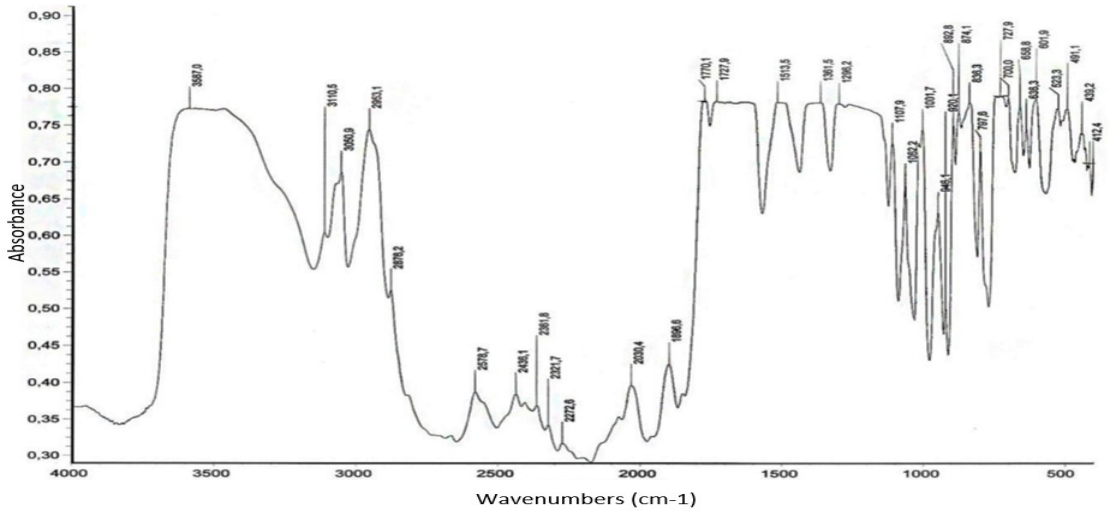


Fig.8. IR spectrum of polyimide film containing 2 wt.% PT filler, not subjected to deformation

The bands corresponding to the deformation vibrations of the C_{ar}–H bonds in the range of 600–900 cm^{-1} in the polyimide composite material (PCM) change significantly after stretching. The low-intensity peak at 727.9 cm^{-1} indicates a weak bond, while the increased intensity of the peaks at 797.5 and 874.1 cm^{-1} suggests an increase in the strength of the bond. The peak at 797.5 cm^{-1} is associated with m-disubstituted deformation vibrations of the C_{ar}–H bonds [14].

The rupture of the polyimide composite leads to the formation of new C_{ar}–H bonds. The peak at 835.3 cm^{-1} in the broken film corresponds to m-disubstituted deformation vibrations of the C_{ar}–H bonds, as well as the peak at 3053.9 cm^{-1} and its overtone at 1895.6 cm^{-1}.

4. Conclusions

The study results indicate that the addition of a polyethylene terephthalate filler significantly affects the physicochemical interactions between polyimide macromolecules, leading to noticeable changes in the composite material's mechanical properties:

– The PT filler at a concentration of 0.5 wt.% increases the plasticity of polyimide by $\Delta\varepsilon = 45\%$ and the tensile strength by $\Delta\sigma = 20$ MPa compared to the maximum changes of $\Delta\varepsilon \approx 37\%$ and $\Delta\sigma \approx 72$ MPa for pure polyimide.

– At a concentration of 2 wt.%, the PT filler enhances the tensile strength of the polyimide composite by 46% and increases its plasticity by a factor of 2.8. This concentration promotes structural organization within the polyimide matrix, resulting in a 40% increase in IR absorption intensity in the 400–1700 cm^{-1} range and a 70% increase in the 1700–3500 cm^{-1} range compared to pure polyimide. The improvement in mechanical properties is associated with the filling of the polymer matrix pores with the PT filler, which stabilizes the molecular chains and strengthens the polyimide structure with a flexible framework.

– The introduction of higher concentrations of PT (up to 10 wt.%) increases the porosity of the composite, reaching 40 nm. However, when the PT filler concentration exceeds 10–12.5 wt.%, thermodynamic incompatibility between the polymers becomes evident, leading to a decrease in compatibility. The additive dependence of viscosity on filler concentration indicates that no new bonds are formed or significant conformational changes occur between polyimide and PT macromolecules, as no new absorption bands or shifts in the IR spectra are observed.

– The observed additive viscosity dependence further confirms that no chemical bonds are formed between polyimide and PT macromolecules, and no conformational changes occur within the composite.

Conflict of interest statement

The authors confirm that they have no conflicts of interest – financial, personal, authorship-related, or any other – that could influence this research or the results presented in this paper.

CRediT author statement:

Yar-Mukhamedova G.Sh., Imanbayeva A.K.: Project Administration, Conceptualization, Methodology; **Muradov A.D., Mukashev K., Umarov F.**: Verification and Formal analysis; **Mussabek G.K., Belissarova F.**: Data Curation, Validation and Investigation. The final manuscript was read and approved by all authors.

Acknowledgements

This research was funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. AP23484310).

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