*Original Article*

# Double-Condensed Phosphates of Bivalent Metals and Ammonium: Obtain NH$_4$PO$_3$ from Solutions and its Research

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*Abstract - Despite the small share of nanoparticles in the field of chemistry, the demand for modifiers is increasing every day. This is due to their high physical, mechanical, and other mechanical properties, resistance to aggressive environments, and the ability to use them in a wide temperature range. According to the results of this study, the introduction of metal phosphates into the polymer improves the physical and mechanical properties of polyolefins. The physico-mechanical properties of the obtained polymer composite materials improved by 1-3 times compared to the initial polyamide-66 samples without filling. It is more important to produce polymer materials based on metal-containing compounds and to use them in practice. The inclusion of metal phosphates among fillers increases the fire resistance of composites. IR, thermal analysis, TG and DTA, DSK, Raman spectroscopy, X-ray diffraction, burning time, and oxygen index were also determined. As a result of the introduction of fillers, polymer molecules are connected with metal binders, and the elasticity, shock, fire resistance, and heat resistance of polymers have increased up to 3 times. The burning time is shortened, and the oxygen index is increased from 17% to 24%. At the same time, ammonium polyphosphate, an inorganic salt of phosphoric acid, was used as a filler, a widely used compound for the production of paints and varnishes with fire-resistant properties. These mineral fertilizers are high-molecular fire-resistant additives, the structure of which is formed as a result of combining monomeric orthophosphates into one polymer chain.*

*Keywords - Polyamide-66, Polypropylene, Ammonium polyphosphate, Nickel oxide, Cobalt oxide, Zinc oxide, Lead oxide, Iron oxide.*

## 1. Introduction

Today, the rapid growth of the world population and production causes the demand for polymers to increase year by year. The demand for polymers is increasing day by day, especially in the industrial and manufacturing sectors. [1, 2]. In this case, high structural requirements are imposed on polymer materials in the automotive industry.

In solving this problem, due to the low cost and large production, it is of great importance that the modification of polymers and the production of polymer composite materials that can meet modern requirements based on them are widely used in various fields. [3, 4].

Modifying polymers, improving their physical and mechanical properties, adding additives without changing their composition, and obtaining composite materials that meet a number of requirements are becoming the basis of scientific research[5].

Fillers can be considered active components because their introduction into a polymer matrix leads to an improvement in its physicochemical properties. Also, the surface free energy is an important parameter since interfacial interactions depend on the ratio between the energy of the matrix and that of the filler[6].

Using fillers is most efficient for polymers with their flexible chains and mechanical strength; it increases the melting point and electrical conductivity, lowers swelling, saves the polymer material, and lowers the cost of filled materials[7,8].

With an active filler added, the interaction between active sites and macromolecules takes place, and different types of bonding are established: physical, hydrogen, and chemical bonds. As a result, the mobility of macromolecules decreases, and ordered supramolecular structures are formed. The effect is greatest when a monolayer film is formed because the orienting influence of the surface of filler particles falls rapidly with distance and barely extends beyond the first macromolecular layer[9,10].

## 2. Experimental Part

Under laboratory conditions, ammonium polyphosphate NH$_4$PO$_3$ and NiO in a 1:3 mol ratio were drawn on an electronic scale and mixed. The reaction was carried out at 250°C for 1-4 hours, and then the resulting mixture was cooled to room temperature. As a result, modifications of new polyphosphate compounds containing (NH$_4$)$_2$Ni(PO$_3$)$_4$ were created.

**Table 1. Composition of NiO-NH₄PO₃, which obtained at 250°C**

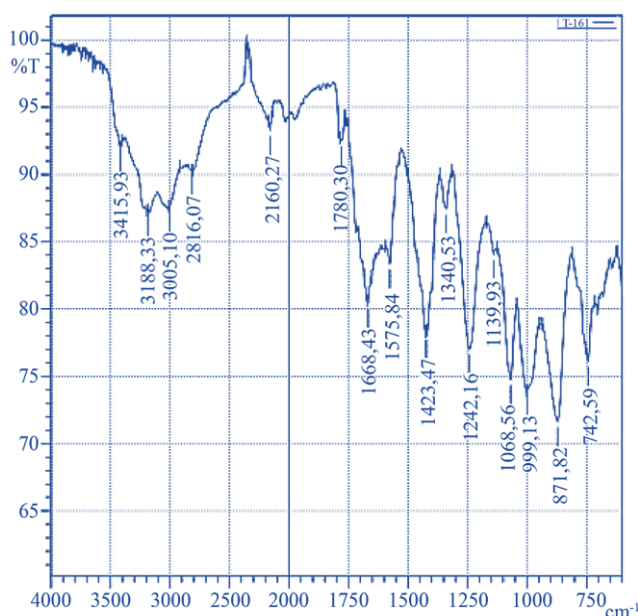| Time, h | NiO:NH₄PO₃ mole ratio | |
| --- | --- | --- |
| | **1:3** | **1:5** |
| 1 | Amorphous phase | $(NH_4)_2Ni(PO_3)_4$ |
| 2 | $(NH_4)_2Ni(PO_3)_4$ | $(NH_4)_2Ni(PO_3)_4$ |
| 3 | $NH_4Ni(PO_3)_3$ | $NH_4Ni(PO_3)_3$ |

Gray-olive crystals of this substance are in the form of thin plates collected in packages, and due to the layered structure in a humid atmosphere, the substance absorbs water with the formation of dihydrate $(NH_4)_2Ni(PO_3)_4 \cdot 2H_2O$. The results of the step-by-step study of the crystalline interaction products in the NiO-NH₄PO₃ system at 250°C at different ratios, as well as the components and reaction time, are presented in Table 1.

### 2.1. IR Spectrum of Analysis

Synthesized compounds were studied in a Fourier infrared spectrometer SHIMADZU (4000-400 cm⁻¹ range) (Figure. 1). According to the results of infrared spectroscopy analysis, the frequency of OH-bond specific valence vibration of phosphate crystallized in NiO-NH₄PO₃ system is between 3415.93 cm⁻¹ and 2816.07 cm⁻¹, 2131-2160.27 cm⁻¹ to S=O carbon corresponding stretching vibrational frequencies, 1423.47 cm⁻¹ corresponding to NH4+, 1242.16 cm⁻¹ corresponding to P=O, were observed.

Valence vibrational frequencies corresponding to nickel compounds were observed at 742-680.87 cm⁻¹. Also, in laboratory conditions, ammonium polyphosphate $(NH_4PO_3)_n$ and PbO in a ratio of 1:3 were taken and mixed. The reaction was carried out at 300 0C for 1-5 hours; then, the resulting mixture was cooled to room temperature. The obtained compound was analyzed in the Fourier infrared spectrometer of SHIMADZU (in the range of 4000-400 cm-1) (Fig. 2).

According to spectroscopy analysis, the high valence vibration frequency of N-H in the range of 3197.98 cm⁻¹, the valence vibration of -P-H at 2358.94 cm⁻¹, the valence vibration frequencies of 2131-2185.5 cm⁻¹ for S=O carbon, very intense stretching vibration of >S=O at 1699.29 cm⁻¹, a deformation stretching vibration of =C-H at 1417.68 cm⁻¹, and corresponding stretching frequencies of 1244.09cm⁻¹–P=O were observed.

In addition to PbO-NH₄PO₃ double cyclo triphosphate $(NH_4)_2PbP_4O_{12}$, polyphosphate of the composition $NH_4Pb(PO_3)_3$ In this case, the standard scheme of thermal transformations is as follows:

$$(NH_4)_2PbP_4O_{12} \longrightarrow NH_4Pb(PO_3)_3 \longrightarrow Pb(PO_3)_2.$$

Thus, ammonium polyphosphate based on mono- and disubstituted phosphates with the presence of cross-links crystallizes sequentially with decreasing NH₄:MeII ratio.

### 2.2. Differential-thermal and Thermogravimetric Analysis

The thermal stability of the synthesized complex was analyzed by differential-thermal and thermogravimetric methods on the Japanese SHIMADZU-DTG 60 device. 3.46 mg of the complex compound crystal was taken for analysis, and the process was carried out at a temperature of up to 800°C. It was studied by automatic recording of the derivatogram in the derivatograph at a speed of 10 degrees/min, T-900, TG-200, DTA - 1/10, and DTG - 1/10 galvanometer sensitivity (Fig. 3).



**Fig. 1 IR spectrum of synthesized NH4Ni(PO3)₃**
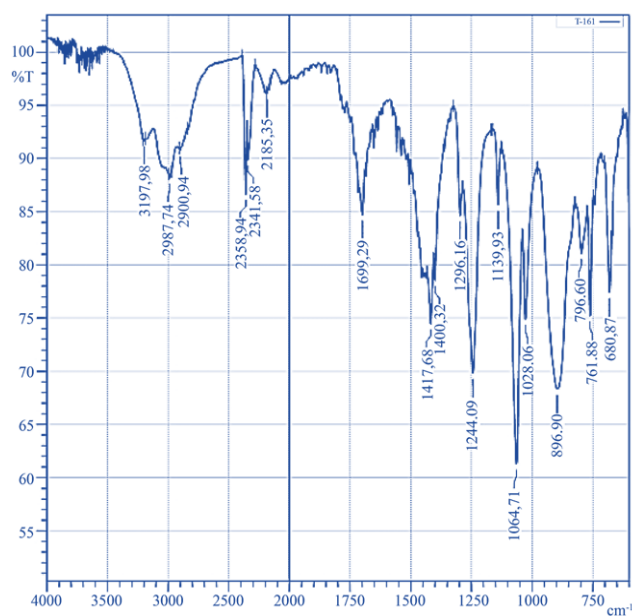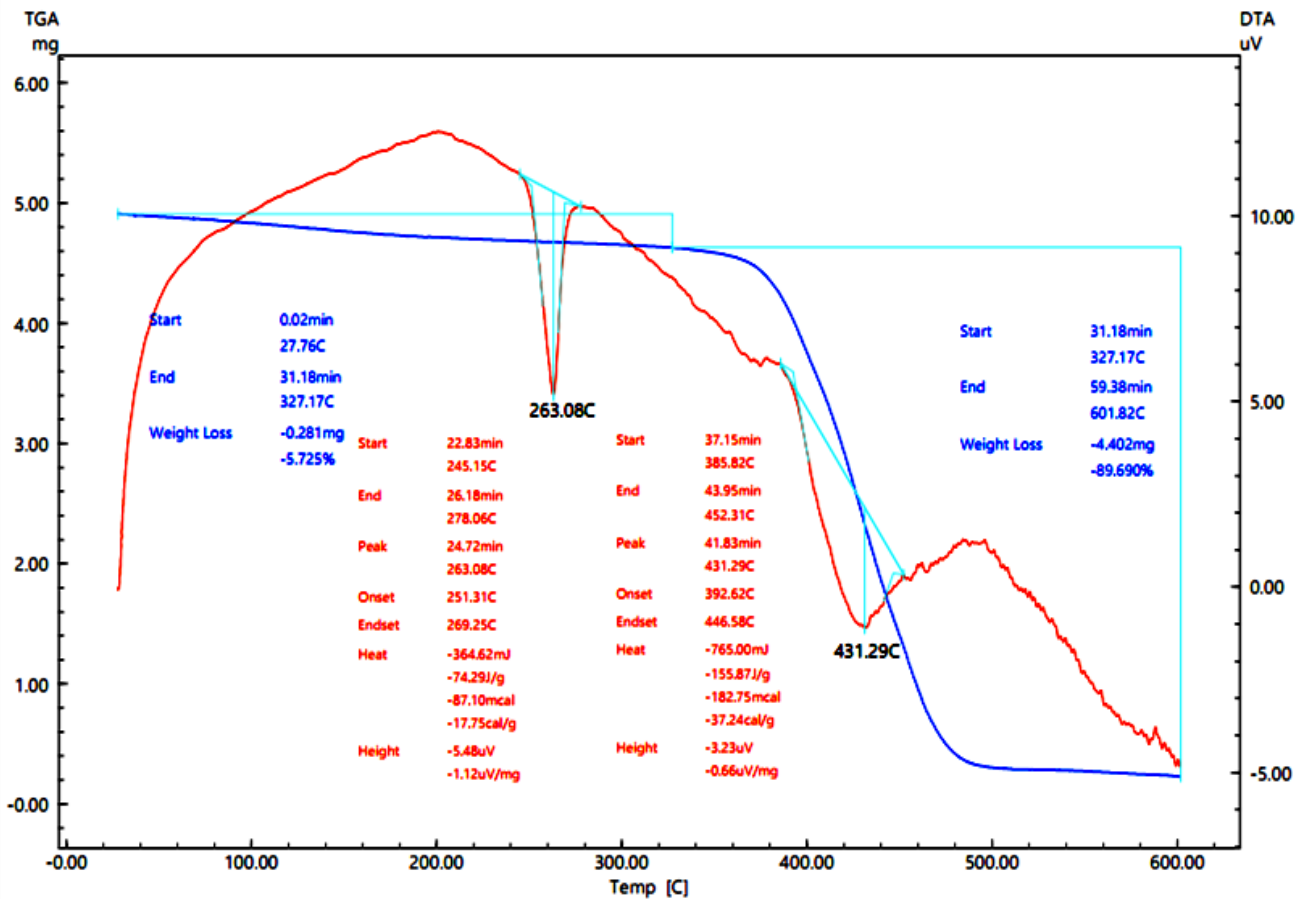


**Fig. 2 IR spectrum of synthesized NH4Pb(PO3)₃**

**Fig. 3 Thermogravimetric (TGA) and differential thermal (DTA) analysis of NH₄Pb(PO₃)₃ complex**

The analysis of the thermogravimetric curve of the synthesized complex shows that the TGA curve mainly takes place in the temperature range of 2 mass losses. The 1st mass loss range is 31.16 - 327.17 °C; the 2nd range is 327.17 - 601.87 °C; the 1st mass loss is 0.281 mg, i.e. 5.725% of water vapor and $CO_2$ gas escape, the main amount of mass loss 2 was 4,402 mg, that is, 100.265% mass reduction due to the destruction of the compound was observed. At the same time, an endothermic effect was observed at 263.08 0C and 431.29 °C in the DTA graph [11].

The obtained new compounds were processed as fillers for polymers. A mixture of PA-66 and PP in the amount of 1-5% was produced in an extruder in the form of 0.25 mm granules. PA was used in a solution state at a temperature of 220-266ºC in a muffle furnace named "DAIHAN Scientific Furnace". The tensile stress and relative elongation of the obtained composite materials were studied according to GOST 14236-81 with shovel-shaped samples of 50x6x4 mm at a deformation rate of 50 mm/min on a Universal Tensile Testing Machine (SHIMADZU SORL 34472)[12].

# 3. Results and Discussion

During the research on the physical and mechanical properties of PA-66 filled with new polyphosphates, the following table shows that the number of elasticity, impact resistance, mechanical deformation, and heat resistance have increased.

As can be seen from Table 2, after the introduction of fillers, the properties of PA include modulus of elasticity in bending (from 2900 to 6470 MPa), elongation (from 20 to 21%), bending temperature under load (from 220 °C to -250 °C) and shortening increased the tensile strength by 1-3% and the impact strength by 6 kJ to 13.38 kJ within 24 hours (from 0.85% to 13%) [13,14].

A Raman spectroscopy study was conducted to determine the binding of synthesized ammonium polyphosphate compounds with polymer molecules, the movement of molecules in the process of modification, and the vibration modes of molecules in solid bodies.
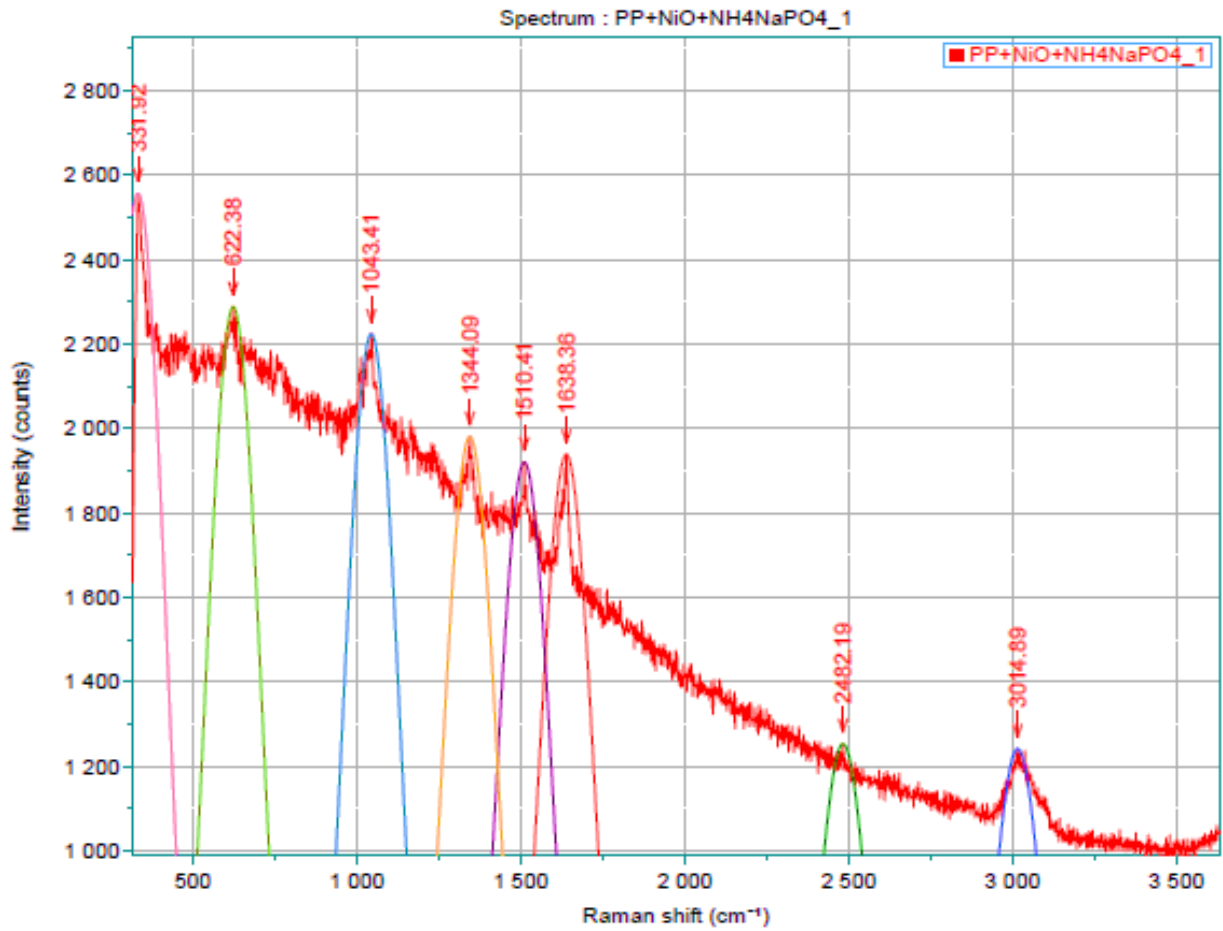
### 3.1. Raman Spectrometer Analysis

This research was carried out using the Raman spectrometer in the laboratory of Termiz State University, and the results can be seen in Figure 4.

According to the analysis of the Raman spectrum, the molecule of the vibrational modes with strong, sharp peaks of the spectra: aromatic line of C C stretching (1638, 1652, 1576, 1510, 1511, 1344-1364 cm⁻¹), C O C of bending (1312-1344 cm⁻¹) and C H (1043.1183 cm⁻¹) or the presence of metals in C H (779 cm⁻¹) and (331.92 cm⁻¹) of external bending, polymer molecules of synthesized polyphosphate ammonium compounds with metal binders indicates that it is connected.

**Table 2. Physico-mechanical properties of PA-66 filled with modified metal polyphosphates**

| Indicators | Standards | PA-66 | PA+ 5% NH$_4$Fe (PO$_3$)$_3$ | PA+ 5% NH$_4$Co (PO$_3$)$_3$ | PA+ 5% NH$_4$Pb (PO$_3$)$_3$ | PA+ NH$_4$Cu (PO$_3$)$_3$ | PA+5% NH$_4$Ni (PO$_3$)$_3$ |
|---|---|---|---|---|---|---|---|
| Modulus of elasticity in bending is MPa | ISO 178 | 2900 | 6470,8 | 6340,2 | 4750,6 | 4879,2 | 4355 |
| Elongation % | ISO 527-2 | 20 | 3,8 | 4,09 | 5,3 | 4,6 | 5,5 |
| Tensile strength, MPa | ASTM D638 | 24 | 26 | 26 | 25 | 25 | 24 |
| According to Izod | ISO 180/1A | 6 | 10,74 | 13,38 | 11,65 | 10,04 | 6,51 |
| Shock tolerance | ISO 180/1A | 6 | 7 | 7,5 | 8 | 7,8 | 6,3 |
| kDj/m2,+23°C | ISO 75-2 | 220 | 230 | 240 | 250 | 240 | 232 |
| According to Izod | ASTM D955 | 0,85 | 0,9 | 0,91 | 1,04 | 1,13 | 1,6 |
| Shock tolerance | The thickness of the sample is 1.6 mm | 10 | 7 | 8 | 7 | 8 | 7 |



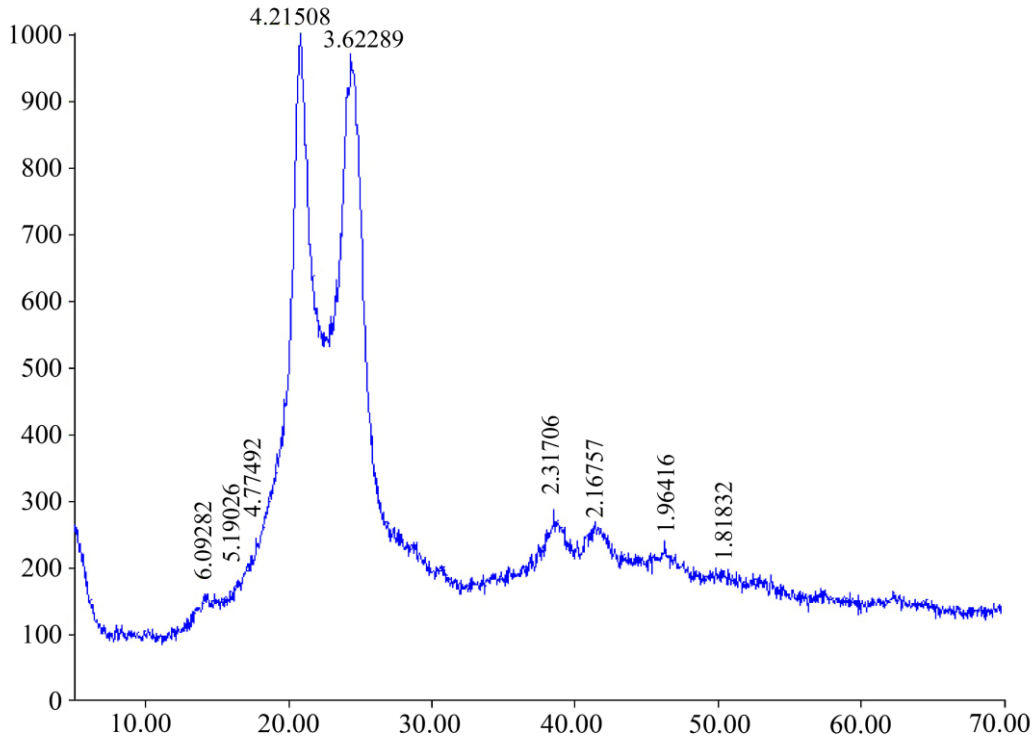| Date | 08.07.2023 1... | Acq. time (s) | 1 | Accumulations | 1 | Laser (nm) | 785 |
|---|---|---|---|---|---|---|---|
| Spectro (cm⁻') | 2181.22 | Hole | | Slit | | Grating | 685 |
| ND Filter | | Objective | | ICS correction | Off | Range (cm⁻') | |

**Fig. 4 Raman spectroscopy of PP/NH$_4$Ni(PO$_3$)$_3$**

**Fig. 5 (a) Appearance of PA + 3% NH₄Pb(PO₃)₃ in X-ray phase spectra**

### 3.2. X-Ray Diffraction Analyses

Compositions of synthesized additives with polymers were analyzed based on X-ray diffraction. Diffraction patterns were recorded on an XRD-6100 X-ray diffractometer (Shimadzu, Japan) with computer control. CuKa radiation (β-filter, Ni 1.54178; current mode and tube voltage 30 mA and 40 kV, respectively) and constant detector rotation speed (step 0.02 deg.), 4 deg/min ($\omega/2^{nd}$ scheme ) and scanning angle from 4 to 80 ºC.

Scan Range: 5.0000-70.0000 (deg) Scan Mode: Continuous Scan Speed: 2.0000 (deg/min) Sampling Interval: 0.0500 (deg) Settling Time: 1.50 (sec). X-ray diffraction patterns of the studied composites were obtained by RFA. (Figure 5)

When studying the diffraction spectra of composites that differ according to the type of filler, the greater the interlayer distance of the fillers added externally to the polyamide-based composite, the higher the degree of delamination of the layered additive.

As shown, the stratification of the layered additive depends on the time of mixing the components and the viscosity of the liquid of the mixture.

In the table, we can see the result of the PA diffraction pattern under the crystalline or amorphous structure of the polymer material obtained in the X-ray analyzer. It allows you to measure the size, shape, and internal structure of the material.

**Table 3. Results of the diffraction pattern of polyamide-66 filled with modified metal polyphosphates**

| T/r | 2theta- scanning angle | d-Interplanar distance | I-Peak Intensity | FWHM- integral width of reflexes |
|-----|------------------------|------------------------|------------------|----------------------------------|
| 1 | 14.5264 | 6.09282 | 120 | 2.10000 |
| 2 | 17.0699 | 5.19026 | 206 | 2.85000 |
| 3 | 18.5673 | 4.77492 | 459 | 2.52860 |
| 4 | 21.0598 | 4.21508 | 2269 | 2.07180 |
| 5 | 24.5519 | 3.62289 | 2345 | 2.02420 |
| 6 | 28.7949 | 3.09797 | 211 | 3.10000 |
| 7 | 30.4428 | 2.93392 | 104 | 2.22500 |
| 8 | 38.8347 | 2.31706 | 288 | 2.70000 |
| 9 | 41.6328 | 2.16757 | 271 | 3.32000 |
| 10 | 46.1801 | 1.96416 | 160 | 4.00000 |
| 11 | 50.1282 | 1.81832 | 81 | 7.30000 |

According to the results of X-ray spatial analysis, it was found that the size of particles in all composite materials is in the nanoscale. The development of modern nanotechnologies in the field of polymer materials always requires in-depth knowledge of the internal structure of micro- and nano-sized materials and their physical properties. Although there are several ways to solve these problems, a new technique - the scanning atomic force microscope - helps to achieve greater precision. By effectively using traditional methods, we can achieve excellent results. One effective way to solve these problems is atomic force microscopy.

Analysis Samples of the "Mifil" brand ultrafiltration membrane at the Institute of Polymer Physics were scanned with a SmartSPM-1000 atomic force microscope. A silicon cantilever with a probe radius of 10-20 nm was used. The size of the scanning area was 1-50 μm. Microscopy was performed in air using a semi-contact method, and changes in the cantilever needle amplitude indicate, in our opinion, fluctuations in surface topography and interfacial motion (spatial detection), local surface overlap, and changes in phase composition.

Figure 6 shows the polyamide surface filled with ammonium Pb polyphosphate (3D view). As can be seen from the above results, the surface roughness of pure polyamide-66 is 99.8 nm, and the roughness of the polyamide filled with +5% lead polyphosphate is 49.6 nm. From the 2D image, we can see that there is also a 1.16 nm spherical metal (lead) particle and small spherical particles. Each hole had approximately the same radius of 60-70 nm. The selection of this value is determined by its compatibility with the pore sizes of membranes of this class. According to the data of the studied sample, the average size of its pores is about 30 nm. It constitutes only data with radius values greater than 70 nm that were excluded from further analysis. Thus, it can be said that the modification of the polymer surface with metal particles leads to an increase in the roughness of its surface.

As shown in Figure 6 a, the change of phase movement indicates the presence of two phases on the surface of the polymer and the work in the polymer composite material where the polymer macromolecules form a mechanical mixture with the metal particles [16]. In order to study the fire resistance properties of composite materials filled with new metal-containing polyphosphate compounds, the oxygen indicator parameters of the polymer were studied using test experiments. For this, according to GOST 12.1.044-2018, a sample of 50:25 mm size was prepared and available in the laboratory of the Tashkent Institute of Chemical Technology Limited Oxygen Index Tester Model: GD-ISO 4589-1 (limited oxygen index measuring device) experiments were carried out.

To determine the oxygen index, 50:25 mm of 3 samples were taken and placed in the device. Before burning the sample, the oxygen level was checked up to 18%. At each stage of the practical experiment, the oxygen level was maintained at 1% in the process until the sample was ignited. In the process, when the oxygen index reached 20.2%, the combustion state of the sample was observed. A 50:25 mm coating sample was found to burn for 3 minutes and 86 seconds at an oxygen index of 20.2%. In the course of practical experiments, it was proved that the average value of the oxygen index of polymer composite materials filled with metal polyphosphates is equal to 41. In particular, as a result of the inclusion of filler metal polyphosphates in the composition of polymers, the burning time decreased from 240 0C to 116 0C, and the oxygen index increased from 25% to 27%, respectively, compared to the original polyamide-66. (Table 4). As a result of filling polymers with metal-containing polyphosphates, their burning time slows down by two times compared to the original polymer, and mass loss during combustion in air is relatively reduced. A positive change in flammability indicators when metal phosphates are included in the composition of all polymers confirms that the resulting composite materials are difficult to burn.
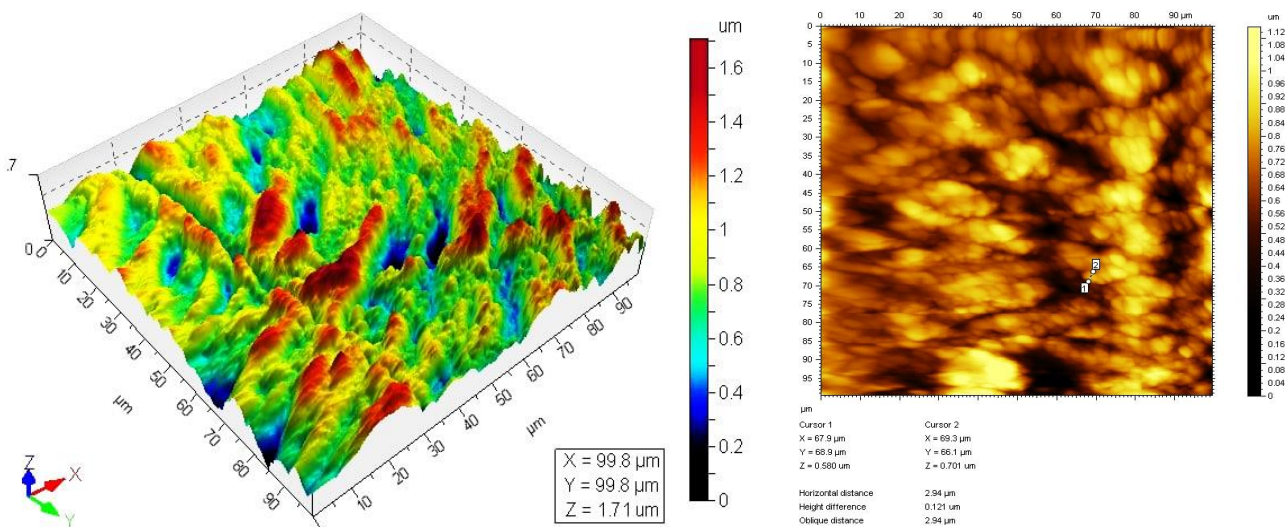


**Fig. 6 ACM image of polyamide surface filled with modified polyphosphate compounds:**
**a) - three-dimensional image, b) - two-dimensional image.**

**Table 4. Effect of composite materials filled with modified metal polyphosphates on heat resistance and flammability**

| Composite composition | Burning times | Mass loss during combustion in air, % | Oxygen index, % | Initial destruction temperature, ºC | Coke residue at 600 ºC, % | Heat resistance, according to Vica, ºC |
|---|---|---|---|---|---|---|
| PA-66 | 240 | 58 | 25 | 280 | 18 | 95 |
| PA+ 1% MeF | 113 | 28 | 26 | 282 | 32 | 100 |
| PA+ 3% MeF | 115 | 29 | 27 | 283 | 34 | 102 |
| PA + 5% MeF | 116 | 30 | 28 | 285 | 36 | 112 |

The addition of a metal-bound ammonium polyphosphate compound to the polymer increases the thermal stability of the composite and shifts the decomposition onset temperature to a higher temperature range. As can be seen from the table below, reinforcement of PA with metal polyphosphates leads to an increase in the temperature of the onset of crystallization and the temperature of the onset of liquefaction of composites. Thermoanalytical studies of the presented samples were carried out on a DSK (differential scanning calorimetry) Netzsch Simultaneous Analyzer STA 409 PG instrument (Germany), with a K-type thermocouple (Low RG Silver) and an aluminum crucible. The result of the modified DSK based on PA/5% NH4Pb(PO3)3 is recorded in Figure 7 below.

Figure 8 shows the melting (up to 270°C) and crystallization DSC thermograms (up to 371°C) of the 20 wt % metal polar polymer blends of the original PA-66. Metal polyphosphate % polar polymer Figure 8b shows DSC thermograms of melting (sections up to 273°C) and crystallization (up to 416°C of initial PA. [17].

With the introduction of fillers into the composition of polymers, it is possible to see an increase in their degree of crystallization and the temperature of liquefaction. As a result, the phase structure of polyamide can be explained by the strong adhesion between the particles of the matrix and metal compounds, which leads to the formation of new adsorption layers at the interface of the phases and junctions of the amorphous components.
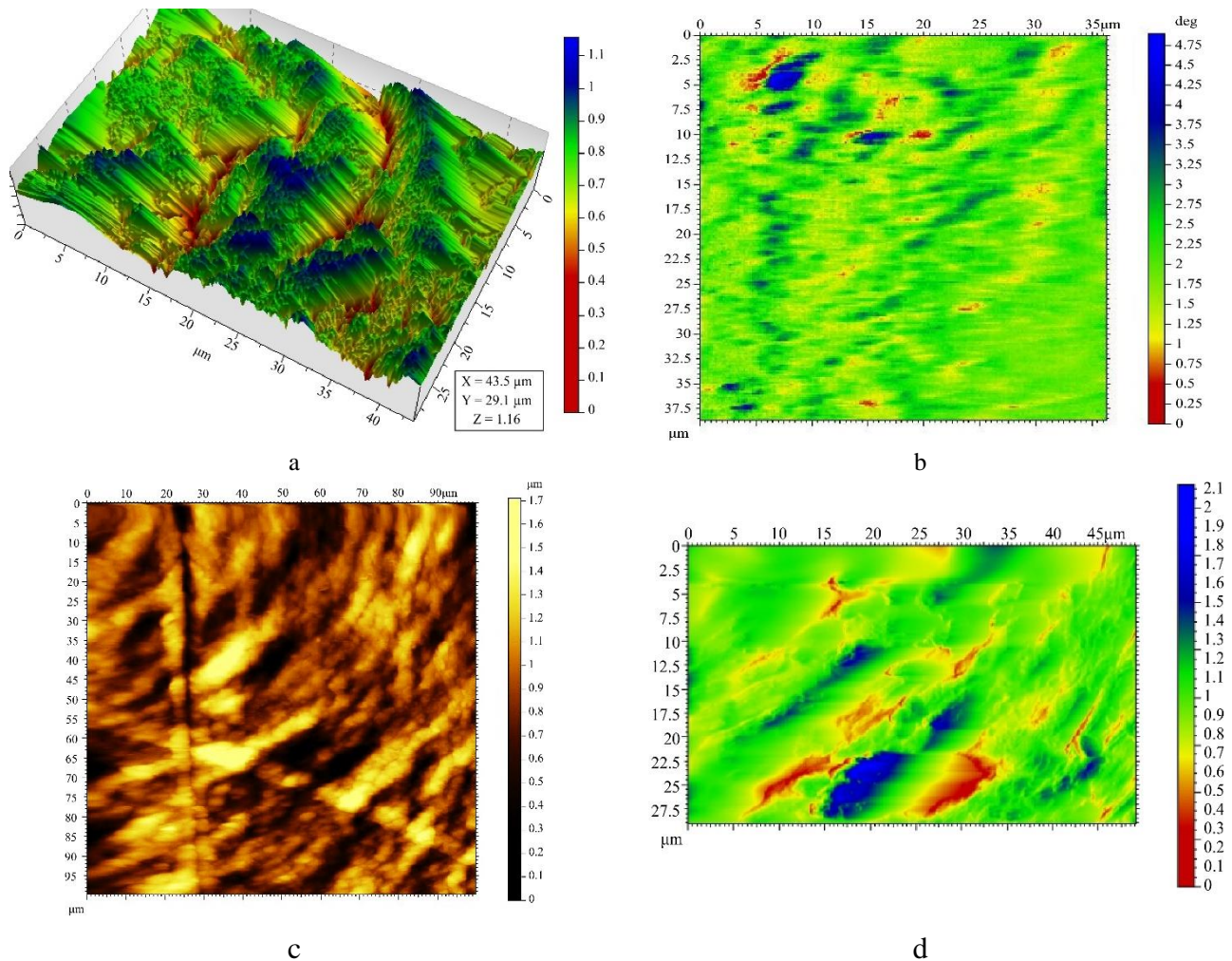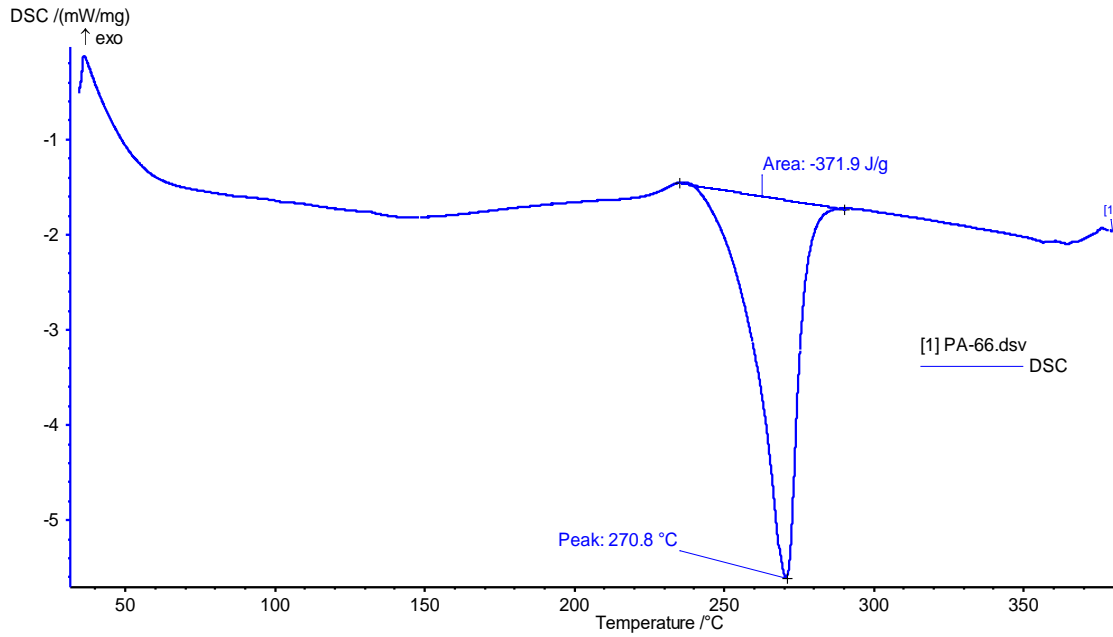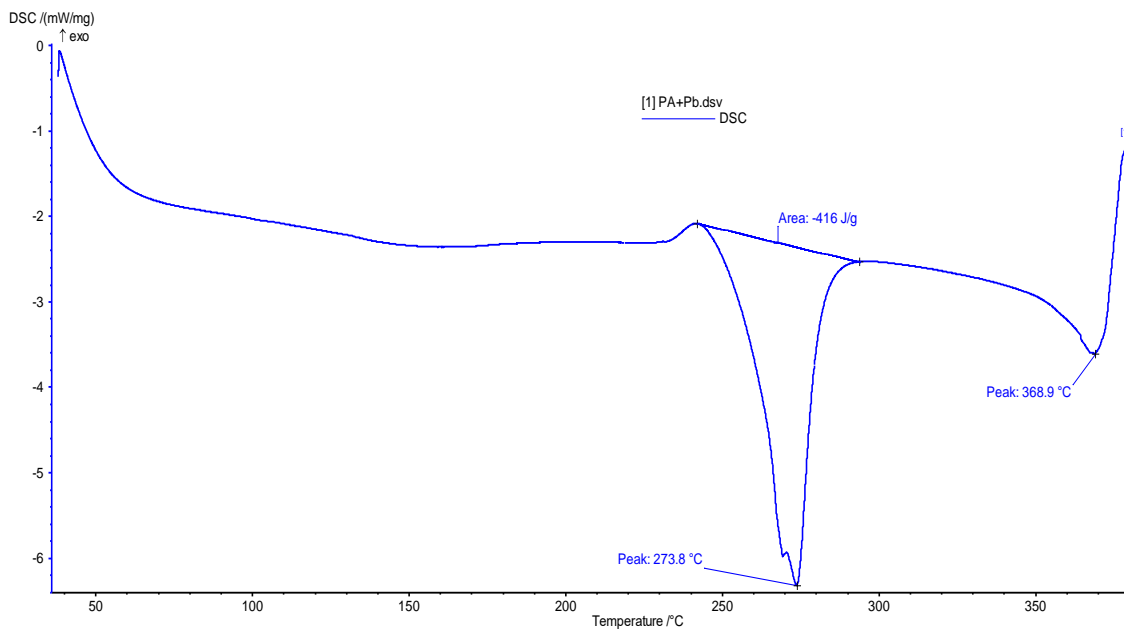


a



b



c



d

**Fig. 7 AKM image of the surface of polyamide-66 filled with Pb polyphosphate: (a) - volume image (3D view), (b) – phase, (c) - two-dimensional view (topography), (d) - o between particle size**

**(a)**



**(b)**

**Fig. 8 DSC diagrams of Polyamide-66: (a) PA, (b) PA + 3% MeF**

## 4. Conclusion

Studies of the MeO-NH₄PO₃ interaction have shown that ammonium polyphosphate is a promising phosphate-containing reagent for the preparation of divalent metals and ammonium doubly condensed phosphates in the high-temperature range. This was determined during the research with an increase in temperature or an increase in the interaction time in the solution. The presence of chemical bonds of the obtained composites is successively crystallized with a decrease in the ratio of ammonium polyphosphate based on mono- and disubstituted phosphates. The effect of the obtained new combination The study of the fire-fighting effect of the composition of the resulting compounds, taking into account the secret, is polyphosphates in a number of polymeric materials and is recommended as an effective fire-fighting filler for polymers.

After the introduction of fillers, the properties of PA-66 include modulus of elasticity in bending (from 2900 to 6470 MPa), elongation (from 20 to 21%), bending temperature under load (from 2200C to -2500C), and shrinkage in 24 hours ( 0.85% to 13%) increased the shock tolerance from 6 kJ to 13.38 kJ. Burning time in polyamide-66 from 240 s to 115 s. decreased, the oxygen index has been proven to increase from 25% to 27%.

## Acknowledgments

## Conflicts of Interest
We hereby confirm that all the Figures and Tables in the manuscript are ours.

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