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NEW PIGMENTS BASED ON TEREPHTAL ACID: SYNTHESIS AND PROPERTIES

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CONTENTS

INTRODUCTION 5

CHAPTER I. MODERN METHODS OF OBTAINING ORGANIC PIGMENTS

1.1-§. Phthalocyanines and pigments based on them... 6
1.2-§. Obtaining pigments based on the modification of phthalocyanines............. 13
1.3-§. Application of modified phthalocyanine pigments as additives to lacquers and polymers.. 23
Summary of chapter I.. 29

CHAPTER II. SYNTHESIS AND RESEARCH METHODS OF NEW METAL-BEARING ORGANIC PIGMENTS BASED ON TEREPTHAL ACID

2.1-§. Research objects.. 29
2.2-§. Research methods... 31
2.3-§. Synthesis of new metal-containing organic pigments based on terephthalic acid and study of the influence of various factors on the synthesis process …….. 34
2.4-§. Research of the composition of new synthesized organic pigments by physicochemical methods 42
Conclusion on Chapter II 62

CHAPTER III. RESULTS OF RESEARCH ON THE APPLICATION OF SYNTHESIZED ORGANIC PIGMENTS

3.1-§. Preparation of alkyd enamel using synthesized organic pigments………. 64
3.2-§. Thermal analysis of lacquer products based on new organic pigments……. 65
3.3-§. SEM and elemental analysis of paints based on new organic pigments…… 72
3.4-§. Checking the compliance of loco-paint products based on new organic pigments with state standards…………………………………………………… 74
3.5-§. Study of the effect of new organic pigments added to 0320 polyethylene on polyethylene properties... 76
Conclusion on Chapter III.. 89

CHAPTER IV. TECHNOLOGICAL SCHEME OF OBTAINING ORGANIC PIGMENTS BASED ON TEREPHTAL ACID

§ 4.1. Technological scheme of obtaining new organic pigments based on terephthalic acid……………………………………………………………… 91
Conclusion ……………………………………………………………….. 92
List of used literature ………………………………………………………… 93

ABBREVIATIONS AND SYMBOLS

Pc - phthalocyanine

CuPc – copper phthalocyanine

MPc - metal phthalocyanine

IR - infrared spectroscopy

SEM – scanning electron microscopy TG – thermogravimetric analysis

DTA - differential thermal analysis

DSK - differential scanning colorimeter

DTGA - Dynamic thermogravimetric analysis

PE - polyethylene

PET - polyethylene terephthalate

EA - elemental analysis

HOMO – Highest Occupied Molecular Orbital

LUMO – Lowest Occupied Molecular Orbital

Introduction

Currently, the network of organic pigments in the world is expanding more and more. Organic pigments based on phthalic anhydride differ from their analogs by higher color intensity, better thermal stability, and resistance to solvents, as well as light absorption of wide wavelengths. In addition, the properties of pigments based on phthalic anhydride have led to the fact that the study of pigments based on other organic compounds is one of the most important research of today. Therefore, the production of organic pigments with new compositions and properties and the improvement of their synthesis mechanisms are of great importance in the world.

Today, targeted scientific research is being carried out in the world in areas that include organic pigments based on highly effective terephthalic acid. As we know, synthetic organic pigments began to be produced in the second half of the 19th century. Thousands of pigments of this type have been used in various industries such as paints, printing inks, and dyeing plastics and textiles. In this regard, special attention is paid to the development of high-quality and competitive pigments based on phthalocyanines containing nitrogen, phosphorus, and other elements, to the research of the properties of pigments, and to the development of their production technology.

Phthalocyanine pigments containing new organic compounds have been created in our republic based on new innovative technologies, certain scientific and practical results have been achieved with the help of these phthalocyanine pigments to obtain pigments with high color intensity, thermal stability, and resistance to external influences, especially effective research aimed at creating phthalocyanine pigments based on a new approach is being conducted.

CHAPTER I. MODERN METHODS OF OBTAINING ORGANIC PIGMENTS

1.1-§. Phthalocyanines and pigments based on them

In the work related to coloring pigments based on organic compounds, it was proved that individual molecules of triphenylmethane, xanthene, phthalocyanine and thiazine do not absorb light in the visible range and are not chromogenic. The individual molecules of these dyes are considered to be chromophoric particles that are necessary for coloration, but do not have sufficient intensity. It has been proven that phthalocyanine groups are colorless compounds in the monomolecular state, they absorb light in the blue range because they exist in the substance in the form of complexes, dimers, and supramolecular compounds [1]

The phthalocyanine ring is an aromatic group containing 18 delocalized electrons, and this group is distinguished by its characteristic of forming intensely colored compounds. Especially its metal complexes are widely used as pigments. Copper phthalocyanine pigments are commonly used to color modern paints, printing inks, plastics, leather, and synthetic fibers. The thermal stability of these compounds is relatively high [2].

Pigments based on phthalocyanine are divided into 4 main groups:

1) metal-free phthalocyanines;
2) metal-preserved phthalocyanines;
3) sulfonated phthalocyanines;
4) chlorine-containing phthalocyanines.

Among these groups, the most common and commercially available are copper phthalocyanine and its derivatives [3]. Synthesis of the phthalocyanine ring is essentially a cyclic tetramerization process. Phthalonitrile, phthalimide, phthalic anhydride, 1,3-diiminoisoindole, etc. are used as raw materials for this purpose (Fig. 1.1).

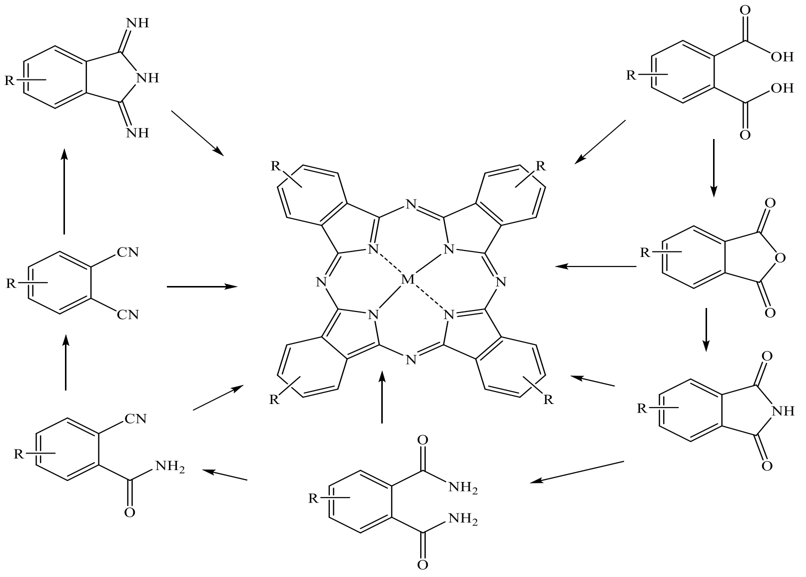


Figure 1.1. The main substances used in the production of phthalocyanines [4].

Metal-free phthalocyanine is rarely used. Although it is less toxic due to its non-metal content, it is relatively expensive to produce and has less coloring. Phthalocyanine is mainly derived from phthalonitrile. The yield of the reaction is around 70-80% and is based on the reaction of alkali metal phthalocyanines with organic bases [5].

Among the metal complexes of phthalocyanine, copper phthalocyanine has a special place, its properties are widely studied and widely used in industry. As a coloring agent, it can be used even for inert polymers such as polyethylene, polypropylene. Its color intensity was also studied by optical spectroscopic method. The amorphous phase in the composition was proved to be soluble in dioxane and heptane, and a true molecular solution of CuPc was formed. CuPc molecules have been found to be absorbed into polyethylene, polypropylene, polycaproamide and cellulose triacetate films. It has been shown that the optical spectrum of individual CuPc molecules is significantly different from that of pigment particles. It was characterized by intense vibrational lines corresponding to three p → p* transitions characteristic of aromatic structures and a series of lines characteristic of p → p* transitions involving nitrogen atoms (<29000 cm^{-1}), but in the visible spectrum (400–800 cm^{-1}) the pigment did not show absorption zones characteristic of dispersion [6].

Authors [7], the synthesis and coloring properties of copper cation phthalocyanine (CuPc cation) were highlighted. The synthesized CuPc cation was characterized by UV spectra and found to have good solubility in aqueous solution over a wide pH range. The CuPc cation has been successfully used in dyeing acrylic fiber, and dyeing was carried out using a conventional method. The effects of five important variables, such as liquid ratio, dye concentration, dyeing time, temperature, and pH, on the degree of dyeability (K/S) of dyed acrylic fiber were investigated. Maximum dyeing was found to be low liquid ratio (100:1), high dye concentration (6%), optimal dyeing time (60 min), 120°C and alkaline medium (pH=11) [7].

Au(III)-phthalocyanine (Au-Pc) and Au(III)-2(3)-tetrakis(allyloxy)phthalocyanine (Au-AoPc) were synthesized using phthalonitrile, 3-allyloxyphthalonitrile. As a starting material, 3-allyloxy phthalonitrile and gold nanoparticles were obtained. Gold nanoparticles stabilized with N,N'-dimethyl amino ethanol were prepared from HAuCl4 solution. Purple gold nanoparticles were obtained by adding N,N'-dimethyl amino to ethanol and reducing HAuCl4 solution at pH 6–7 at room temperature. Later, blue Au-Pc and green Au-AoPc paint materials were synthesized by reacting gold nanoparticles with phthalonitrile and 3-allyoxyphthalonitrile at 120°C. The resulting Au-Pc and Au-AoPc complexes were characterized by optical microscopy, transmission electron microscopy, ultraviolet, visible, IR spectroscopy and electron scattering ionization mass spectrometry. Transmission electron microscopy analysis showed that the particle sizes were mainly distributed between 30-40 nm and 10-100 nm. Using this method, the optical band gap energies (e.g., 1.66 eV for the Q-band and 3.12 eV for the B-band of Au-Pc, 1.79 eV for the Q-band and 3.32 eV for the B-band of Au-AoPc for the range) calculated as N,N'-dimethyl amino ethanol-stabilized gold nanoparticles, Au-Pc, and soluble Au-AoPc dyes with low band gap energy can be used for various technologies in electronics, painting, sensors, and solar cells [8].

Copper phthalocyanine was synthesized in a silica gel xerogel matrix using a newly developed in-situ method using a sol-gel process. Using UV and IR-spectroscopy, it was proved that copper ions present in the form of complex ions $[Cu(H_2O)_4]^{2+}$ and $[CuCl_4]^{2-}$ during the drying and wet gel stages are not present in the solution after the reaction. Copper phthalocyanine molecules were gradually synthesized in-situ during the transition from wet gel to xerogel. The phenomenon of CuPc dimerization in the composite is significantly reduced, because the CuPc molecules synthesized by the "in-situ" method are well isolated in the micropores of the xerogel matrix. By adding various additives to pigments, one of their properties can be changed. For example, copper phthalocyanine-based green G pigment has been proven to be resistant to water and abrasion when coating the surface of fabrics [9].

Phthalocyanine complexes of almost all metals have been synthesized to date, and phthalocyanine complexes of intermediate metals are stable and widely used at the industrial level. Also, phthalocyanine compounds have the property of polymorphism, for example, there are α, β and γ modifications of copper phthalocyanine. They differ from each other in terms of color and stability in organic solvents. All metal phthalocyanines mainly consist of β-modification, which is caused by the use of temperatures above 200°C in their production methods. The α-modification, which is present at low temperatures, can be obtained by driving from a sulfuric acid solution under low pressure. However, the α-modification, which is quite unstable, quickly turns back into the β-modification under the influence of organic solvents or temperature. α-modification of chlorine-protected phthalocyanines is more stable and resistant to organic solvents. γ-modification can be obtained by dissolving metal phthalocyanines in dilute sulfuric acid [10].

Spectroelectrochemical analysis of different phthalocyanines nanomorphological properties were studied. Cobalt was obtained by surface modification of graphite electrode with phthalocyanine. In this case, phthalocyanines, which are used as electrically conductive materials, are used in the analysis of the morphology of the graphite surface [11.].

Compounds with porphyrin and phthalocyanine rings are also used in photodynamic therapy as photosensitizers due to their light absorption, non-toxicity, and high photochemical efficiency [12].

Optimum conditions of the process of dyeing wool materials with trisulfonic acid of copper phthalocyanine were studied. Enzymatic treatment of wool has also been explored to improve dyeability. In this, the optimal conditions between the reduction of fiber strength and the improvement of dyeing level are indicated. In this case, fiber damage during dyeing was analyzed using SEM analysis, and the degree of elongation and its changes after dyeing were studied. The fastness of colors was studied with respect to washing and rubbing. As a result, a new method was created in which the dyeing process is carried out at 85°C instead of the current 98°C [13].

Comparison of IR spectra of copper phthalocyanine (CuPc) nanocrystals obtained by different methods revealed small but measurable changes in some vibrational frequencies of molecules on the nanocrystal surface. These changes are caused by the electrostatic interactions between the polar components of the nanocrystals and the polar components of the molecules with an external polar molecular structure. External polar molecular structures can be chemically bonded to the CuPc nanocrystal surface, or they can be located around it without forming chemical bonds with the nanocrystal. Molecular modeling (DMOL3- and Gaussian calculations) confirms the influence of selected external polar species on the vicinal CuPc molecule on CuPc molecular vibrations and the experimentally observed changes in vibrational frequencies of CuPc molecular bond configurations. Provides details of the forces involved in these interactions. The reduction of the number of external polar inclusions was carried out by centrifugation of the aqueous dispersion of nanocrystals or extraction in a Soxhlet apparatus based on an organic solvent, and their number was increased by soaking (drying) the nanocrystals in high and low pH aqueous solutions with SO3 and OH - ions. These quantitative and qualitative modifications of the amount of external polar species surrounding the CuPc nanocrystals were reflected in the corresponding change of the selected vibrational frequencies of the CuPc surface molecules, which was an effective tool for molecular species recognition. Some of these modifications can also be observed with the naked eye in the form of a noticeable color change of the CuPc nanocrystal powder. This is due to the very high specific absorption coefficient of CuPc nanocrystals, as a result of which the incident light is mainly absorbed on the surface of the nanocrystals. Changes in the electronic structure at this point, as a result of interactions with near-polar species, change the absorption spectra of the nanocrystalline powder, which changes the observed color. Preliminary data suggest that the described analytical method for the analysis of molecular polar particles near molecular nanocrystals will be of value in many fields, from molecular instrumentation to pharmaceutical materials. [14].

The possibilities of using phthalocyanines as catalysts in catalytic oxidation processes of organic compounds were studied, and it was determined that the catalytic activity of copper phthalocyanine dimer in the oxidation reaction of 2-mercaptoethanol is optimal in an environment with a pH of 11 [15].

As a new direction, various catalysts are used for photocatalytic decomposition of organic substances in waste. The working principle of such catalysts is also based on strong light absorption and its good transmission, and the possibility of using different combinations of phthalocyanines for this purpose has been studied. A three-dimensional polymer KIT-6 with interconnected pores was synthesized and used to absorb sulfated copper phthalocyanine (CuPcS). 3-(aminopropyl)-triethoxysilane was used to bind CuPcS to KIT-6 walls. The use of dimethyl sulfoxide as a solvent resulted in the uptake of high amounts of CuPcs into KIT-6. CuPcS/NH2-KIT-6 was comprehensively characterized using X-ray diffraction, nitrogen absorption-based BET and BHJ sorption, scanning electron microscopy, energy dispersive X-ray, and transmission electron microscopy. The photocatalyst was tested for the degradation of 2,4-dichlorophenol in aqueous solutions under visible and UV light irradiation. The obtained results show that a very small amount of photocatalyst can proceed the degradation of dichlorophenol slowly and effectively. The decomposition process is completed in about 0.2 hours using a dose of 3 g/l of the catalyst under visible light. The reusability of the catalyst is good, and after four cycles of repeated use, no obvious decrease in its performance was observed. The kinetics of photodegradation and intermediate products were studied, and the mechanism of the photocatalyst was determined based on the obtained results [16].

The process of synthesizing expensive dyes is a rather complex process and does not fully meet environmental requirements. Therefore, methods of synthesizing these compounds from various industrial wastes containing the phthalic group have been developed. The conditions for the synthesis of dyes from phthalic anhydride contained in coke plant waste have also been determined [17].

During the production of pigments, heavy metals are released into waste water. Extraction of metals from the composition of these waste waters by sorption method is also of great environmental importance [18].

The relative stability of phthalocyanine dyes leads to the long-term preservation of dyes in the natural environment, which have retained various heavy metals. Now the degree of decomposition of these dyes by various representatives of fungi has been studied, and high efficiency has been achieved. Eighteen fungal strains known for their ability to degrade lignocellulose or lignin derivatives were screened for their decolorization of commercially used reactive textile dyes. Three azo dyes: Reactive Orange 96, Reactive Violet 5 and Reactive Black 5, two phthalocyanine dyes: Reactive Blue 15 and Reactive Blue 38 were selected as representatives of dyes used at the industrial level. Of the 18 fungal strains tested, only Bjerkandera adusta, Trametes versicolor and Phanerochaete chrysosporium were able to decolorize all the dyes tested. During the degradation of the nickel-phthalocyanine complex, the toxicity of this dye was significantly reduced as a result of the action of Reactive Blue 38, Bjerkandera adusta, Trametes versicolor. A lot of detoxification has been achieved by exposure to Reactive Violet 5 dye Bjerkandera adusta. Trametes versicolor and Bjerkandera adusta decolorized by exoenzyme preparations Reactive Blue 38 and Reactive Violet 5 were decolorized by H_2O_2-dependent reaction. The specific reaction of exoenzyme preparations with dyes was determined and compared with the degree of oxidation by commercial peroxidase [19].

1.2. Obtaining pigments based on the modification of phthalocyanines

In addition to simple phthalocyanine compounds, its compounds modified with various organic compounds have been synthesized and used in the production of various dyes. As a result of this approach, phthalocyanines with different properties, different colors, lower toxicity, higher economic efficiency and different colors are obtained. The changes introduced as a result of such modification are divided into 4 large groups: 1) adding additional substituents to the benzene ring; 2) replacing the benzene ring with other heterocycles; 3) obtaining extracomplexes as a result of changing the central ion; 4) use of other organic compounds in the formation of basic rings [20].

In addition, in modern methods, methods of obtaining compounds with various special properties are also used only by modifying the surface with the help of nanoparticles. For example, nanocomposition of zinc phthalocyanine with titanium oxide was used as catalysts for selective oxidation of aromatic nitro compounds. The selectivity was 99% [21].

Reaction of 4-nitrophthalonitrile with 4-aminobenzoic acid in dimethylsulfoxide in the presence of potassium carbonate gave 4-[(4′-carboxy)phenylamino]phthalonitrile. He then synthesized copper tetra-4-[(4′-carboxy)phenylamino] phthalocyanine. Spectral properties of the product were studied. It has been proven that it exists in the form of complex compounds in alkaline solutions of water, and in dimethylformamide mainly in monomer form. Phthalocyanine pigments with different compositions are synthesized using different derivatives of phthalonitriles [22]. Nucleophilic substitution of bromine in 4-bromophthalonitrile with anthraquinone afforded 4-[(2-hydroxy-9, 10-dioxo-9, 10- dihydroanthracen-1-yl)oxy] phthalonitrile, which was used to obtain the corresponding cobalt phthalocyanines by the nitrile method [23].

Alkoxyphenoxyphthalonitriles were synthesized by nucleophilic substitution to obtain phenolic residues substituted with 4-nitrophthalononitrile. The stability of substituted phthalonitriles and phthalocyaninates synthesized on their basis (1:1 ratio) was studied by heating in an argon-oxygen environment. It was found that the thermal stability of the obtained compounds and the mass loss of phthalocyanines started at lower temperatures compared to the corresponding phthalonitriles. The spectroscopic properties of the synthesized complexes were studied in organic solvents and concentrated sulfuric acid. The nature of the spectroscopic curves and the position of the main absorption band of phthalocyanine complexes are determined by the effect of the metal complex and the nature of the solvent. It is noted that a shift of the linear Q maximum in absorption was observed for the studied macrocycles when passing from polar aprotic solvents to non-polar solvents. Electronic absorption spectra in sulfuric acid show a significant bathochromic shift of the absorption lines. It was found that the introduction of an alkyl substituent increases the wavelength, but hardly affects the position of the absorption maximum. A bathochromic shift of the absorption maximum is observed during the transition from complexes with Co(II) to Cu(II) [24].

Two synthetic methods for obtaining zirconium and hafnium dihydroxyphthalocyanates (i.e. (a) using zirconium and hafnium alkoxides as reactive precursors and (b) hydrolyzing zirconium and hafnium dichlorophthalocyanates) were investigated. The reactivity of zirconium and hafnium dihydroxyphthalocyanates in exchange reactions with β-diketones and decanoic acid was also studied; they have been compared with zirconium and hafnium dichlorophthalocyanates. The use of zirconium and hafnium dihydroxyphthalocyanates as precursors in their reactions with acid-sensitive ligands such as ketoesters has been found to be a more effective approach [25] (Fig. 1.2).

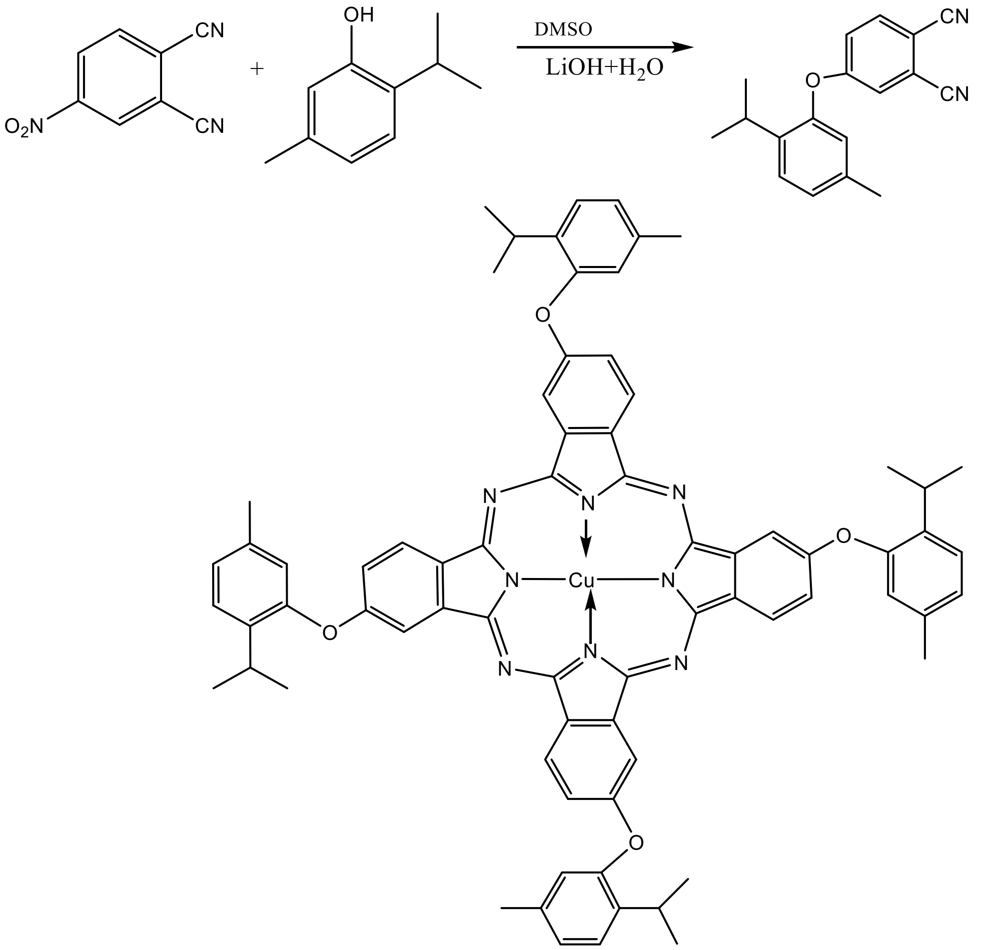


Figure 1.2. Synthesis of copper tetra(2-isopropyl-5-methylphenoxy) phthalocyanine.

A new copper phthalocyanine containing phenoxy substituents was synthesized and studied by mass spectrometry and elemental analysis methods. Its photoluminescence absorption spectra were examd. By using this copper phthalocyanine attached to 4,4′-N,N′-dicarbazole-biphenyl, organic light-emitting devices were prepared. The electrophosphorescence state at room temperature is caused by the transition from the mostly excited triplet state to the singlet state (T1–S0) of CuPc observed at about 1.1 mm [7,26] (Fig. 1.3).

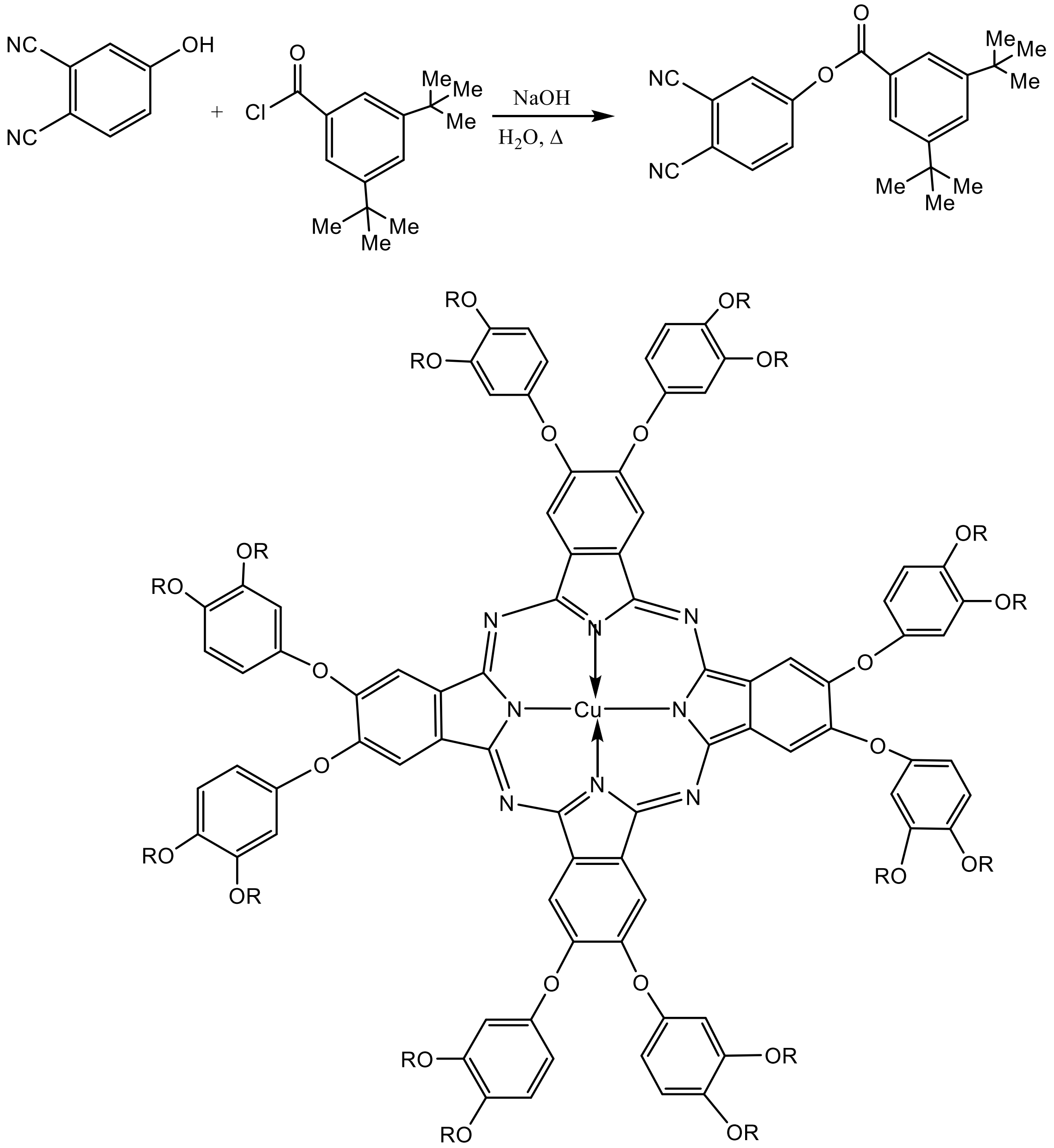


Figure 1.3. Synthesis of phthalocyanines with phenoxy derivatives.

A.S. Malyasova and co-authors synthesized 3,4-dicyanophenyl 3,5-di-tert-butylbenzoate for the first time. Copper tetrakis (3,5-di-tert-butylbenzoyloxy) phthalocyanine was obtained for the first time from this compound using cyclotetramerization. The resulting compounds were identified using UV, visible, IR, 1H and 13C NMR spectroscopic methods. Acidity constants for protonated forms of copper tetrakis (3,5-di-tert-butylbenzoyloxy) phthalocyanine, as well as copper tetra (4-tert-butyl) phthalocyanine and its ligand properties in CH_2Cl_2-100% trifluoroacetic acid system were studied [27].

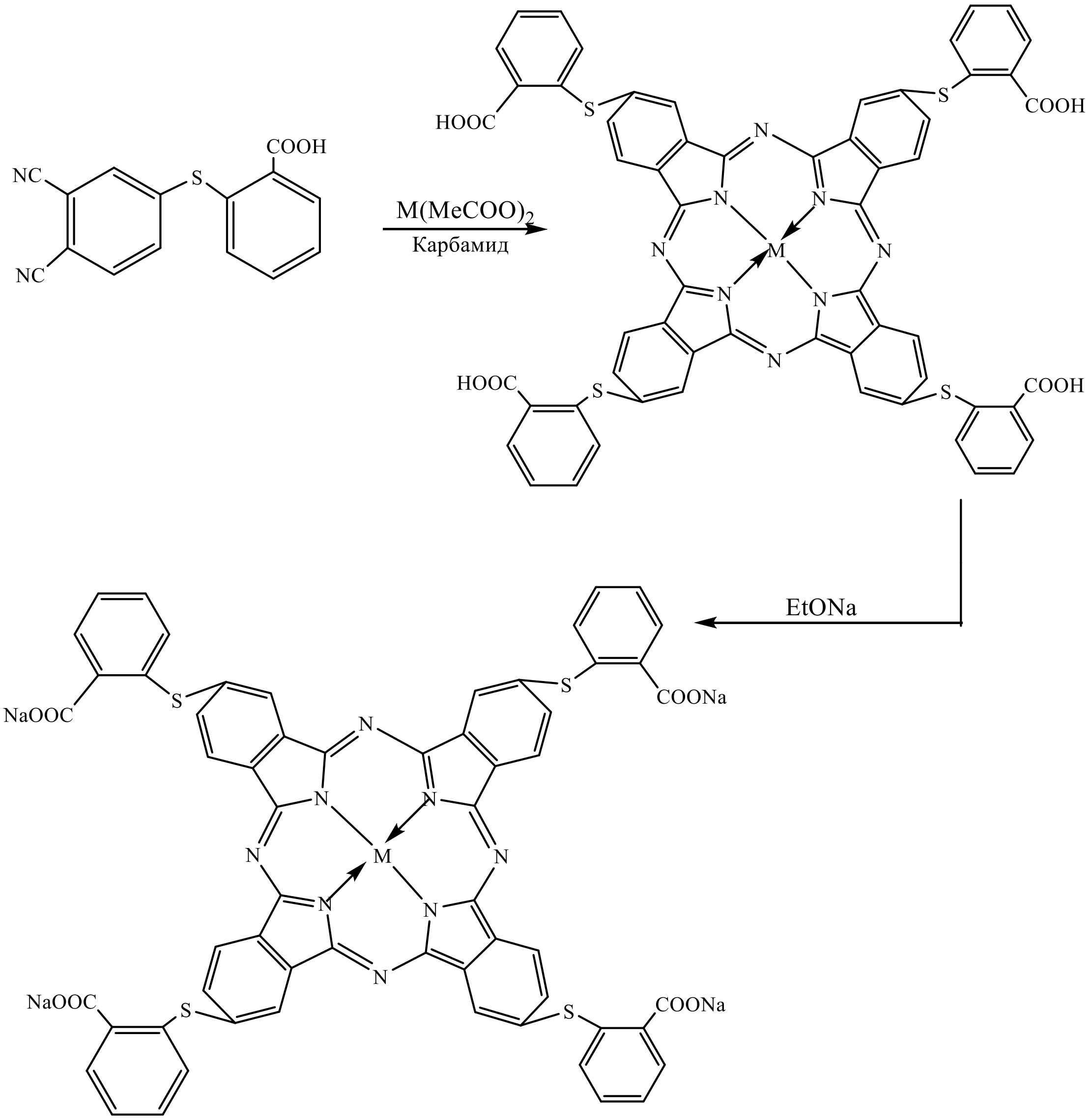


Figure 1.4. Synthesis of metal phthalocyanines retaining phenylsulfanyl groups.

Sodium salts containing phenyl sulfanyl groups containing MgII, CoII, CuII, MgII, CoII, CuII, and ZnII ions as central metal atoms of phthalocyanine derivatives of carboxylic acids were synthesized. The aggregation properties of all metal complexes in alkaline aqueous solutions have been studied. The luminescence properties of phthalocyanine derivatives with MgII and ZnII were studied. Catalytic properties were studied for macrocycles with CoII, CuII, and ZnII [28] (Fig. 1.4).

The main properties of mesogenic phthalocyanines and their derivatives were reviewed by Tsvivadze and Nosikova. Liquid crystalline materials based on self-assembled phthalocyanine molecules allow obtaining new functional materials for sensors as catalysts, optical and bioactive matrices, and semiconductor materials. Mesomorphic and electrophysical properties of materials based on liquid crystalline phthalocyanine were considered by the researchers, the possibilities of their use were discussed and the relationship between the properties of the structure was analyzed. [29].

It has been proven that 3,3′,3″,3″′-tetraaminophthalocyanines of copper (II), cobalt (II), nickel (II) and zinc can be used as antibacterial agents. A new metal (II) 3,3′,3″,3‴ was obtained by condensation of copper (II), cobalt (II), nickel (II) and zinc 3,3′,3″,3″′-tetraaminophthalocyanines with methoxybenzaldehyde. Synthesis and characterization of -tetramethoxyphenylimino substituted phthalocyanines (M-MeOPhImPcs) are described. Dark blue-green tetraimino-substituted phthalocyanine derivatives have been studied by various physicochemical methods, including elemental analysis, magnetic properties, electron, IR, X-ray diffraction, and thermogravimetric analysis (TGA). The variation of the magnetic moment as a function of the field strength indicates the presence of intermolecular interactions [14,30] (Table 1.1).

Table 1.1

Properties of metal complexes of 3,3′,3″,3″′-tetraaminophthalocyanine modified with methoxybenzaldehyde

№	The metal in the complex	Empirical formula	Color	Magnetic moment	Element analysis (calculated)	Reaction product
1	Cobalt	$C_{64}H_{44}N_{12}O_4Co$	Dark green	2.82 2.72 2.66 2.58 2.49	C: 69.63; (69.44) H: 3.99; (3.92) N: 15.23; (15.15) Co: 5.34; (5.38)	88%

2	Copper	$C_{64}H_{44}N_{12}O_4Cu$	Dark green	2.75 2.68 2.61 2.59 2.48	C: 69.63; (69.44) H: 3.99; (3.92) N: 15.23; (15.15) Co: 5.34; (5.38)	91%
3	Nickel	$C_{64}H_{44}N_{12}O_4Ni$	Dark green	-	C: 69.65; (69.66) H: 3.99; (3.79) N: 15.23; (15.12) Ni: 5.32; (5.36)	96%
4	Zinc	$C_{64}H_{44}N_{12}O_4Zn$	Dark green	-	C: 69.25; (69.05) H: 3.96; (3.91) N: 15.14; (15.10) Zn: 5.89; (5.84)	82%

Zinc phthalocyanines are mainly used in light-sensitive paints and solar cells due to their sensitizing properties. Its modifications and compositions with various compounds are used as effective hybrid materials. Synthesis of phthalocyanines containing several halogens in the benzene ring and the effect of their adsorption on polymetallic silicon on the change in the energy transport ability of dyes were studied. Phthalocyanine complexes containing 4 and 8 fluorine atoms showed optimal properties [31].

Extracomplexes of aluminum, hafnium, indium, zirconium, and gallium containing chlorine, nitro, and hydroxo groups were synthesized. The resulting complexes were identified by elemental analysis, UV and IR spectroscopic methods. It is based on the tetramerization of di-substituted phthalonitriles in the presence of metal ions. Also, because the central atom is 3-valent, extracomplexes were formed instead of flat complexes. It has been shown that almost all compounds can be used in semiconductor technology, as selective sensors in gas analyzers (Fig. 1.5).

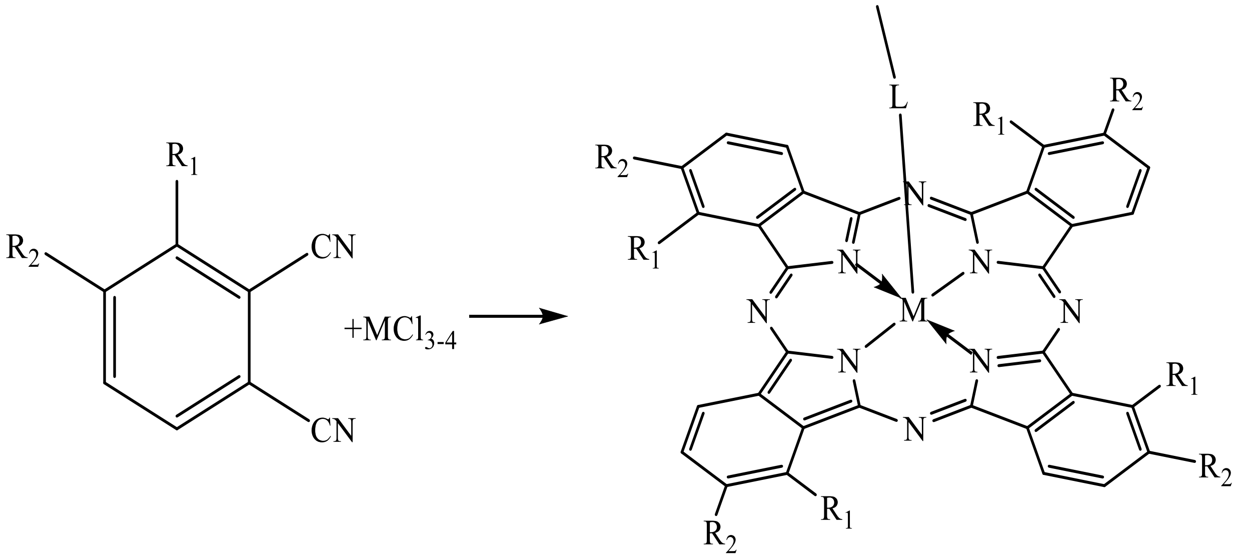


Fig. 1.5 Preparation of extracomplexes using (III) valent metals.

It is used to bind albumin protein from compounds obtained by modification of silicon phthalocyanines containing halogen with polyethylene glycol. According to the results of the effects of the compounds obtained by changing the halogen atoms in phthalocyanines on enzymes and proteins, the possibilities of their use in the pharmaceutical industry are presented [32].

Compounds with extended p-systems are obtained by replacing the benzene group in the phthalocyanine ring with a naphthol group. Naphthalocyanines also form relevant complex compounds with metals. In the synthesis of complexes of copper, magnesium, vanadium and cobalt metals, ammonium molybdate is obtained with relatively high yield at 260-270°C. Reactions carried out at 220°C and in the presence of urea have a relatively low yield. When these reactions are carried out in solvents such as dimethylsulfoxide, tetrahydrofuran, the yield does not exceed 50% [33.].

The optical and electrical properties of zinc phthalocyanines modified with 4-oxyquinoline were studied, and the dependence of the degree of modification on the change in the intensity of the green color was studied. Semiconductor properties were also investigated using voltammetric analysis methods. Their conductivity properties, changes due to expansion of conjugated systems are considered. Phthalocyanines have also been recommended by the authors for use as sensors and photoconductors [34].

Nanocomposites were prepared from polypyrrole aluminum phthalocyanine obtained by evaporation method. Ultraviolet analysis shows that it has an intense color, and the size of the nanoparticles depends on the degree of modification with polypyrrole. Obtaining such very thin composites obtained in the form of a film allows to use in the future as a method of dyeing different colors with low consumption [35].

The electrical properties of polymethyl methacrylate composite compounds with phthalocyanine and metal phthalocyanine were studied. It was determined that the electrical conductivity of polymethyl methacrylate increases due to the properties of the phthalocyanine ring. It has been found that these composites can be used to obtain light-sensitive paints with different compositions [36].

As a result of modification of the surface of graphite with thin cobalt phthalocyanine, electrodes with special properties for determining isomers of dehydroxybenzene are obtained. Electrodes have been prepared for the determination of organic compounds in food products with 95% accuracy. The good electrical conductivity of cobalt phthalocyanine allowed simultaneous detection of dehydroxybenzene derivatives with a concentration of 5*10-6 mol/l [37].

Sulfolated derivatives of copper phthalocyanine were synthesized. These compounds are also used as water-soluble pigments. Amide dyes were obtained by condensation of phthalocyanines with cyclohexylamine, acetyl-p-phenylenediamine, N,N-ethyl, hydroxyethyl-p-phenylenediamine. The resulting phthalocyanine sulfonamide dyes were studied by physicochemical methods [38].

Supramolecular compounds of perylene derivative (N , N ′-bis (2-(trimethylammonium iodide) ethylene) perylene-3, 4, 9, 10-tetracarboxydiimide) with zinc phthalocyanine were synthesized and used as pigments. A perylene derivative, N,N′-bis(2(trimethylammonium iodide)ethylene)perylene-3, 4, 9, 10-tetracarboxyldiimide (TKDI), forms nanoscale columns in water that can be used for optical absorption and emission measurements, dynamic light scattering, transmission electron characterized using microscopy (TEM) and transmission.

These conditions were compared to those in methanol. Bond formation between TKDI and zinc phthalocyanine tetrasulfogroups ($ZnPcS_4$) due to strong and ionic interactions in aqueous media has been described [39].

Table 1.2 above lists the methods for the preparation of phthalocyanines bearing various substituents.

Table 1.2

Synthesis of phthalocyanine derivatives

Compounds used for the main starting raw materials	**Central atom**	**Substitute types**	**Synthesis conditions**	**Literature**
Compounds containing the phthalocyanine ring derived from substitution of substituents on the benzene ring				
1,2-substituted phthalonitriles	Zn	fluorine	$Zn(OAc)_2$, 1-in chloronaphthalene media, 220 °C	[40;]
	Hg, Zn, Al, Ga, In, Cr	chlorine, bromine	MClx salts, fusion syntheses210-240 °C	[41].
	Hg, Zn	bromine, iodine	MCl_4, fusion 210-240 °C	[42.]
2,3-disubstituted phthalonitriles	Zn	-	$Zn(OAc)_2$, 1-chloronaphthalene in the environment,220 °C	[40]
1,2,3,4-Tetrasubstituted Phthalonitriles	Zn	fluorine	$Zn(OAc)_2$, 1-chloronaphthalene, 220 °C	[40]
	Fe		$Fe(CO)_5$, in boiling 1-methylnaphthalene medium	[43]
	Ru		$[I(NH_3)_5Ru^{III}]I_2$, 230-240 °C	[44]
	Ru	chlorine,	$[I(NH_3)_5Ru^{III}]I_2$, 230-240 °C (40%)	[44]
	In		$InCl_3$, 1-chloronaphthalene, 180 °C	[45]
5,6-disubstituted phthaloimides	$SiCl_2$	chlorine,	SiCl4, in quinoline media	[46, 47]
	Si	bromine	SiCl4, in quinoline media	
4,5,6,7-tetrasubstituted phthalic anhydride	Cu	chlorine,	CuCl, $(NH_2)_2CO$, trichlorobenzene, 170-180 °C	[48.]
	Cu, Pb	chlorine,	MCl_2, $(NH_2)_2CO$, fusion fusion, $(NH_4)_2MoO_4$	[49]
1,2,4,5-	H_2	$LiOC_3H_7$, HOC_3H_7		[4]

tetranitrilebenzene	Cu, Ni, Co, VO	Boiling metal oxide or hydroxide dimethylaminoethanol	[50]
Кенгайтирилган π-тизимли фталоцианин ҳосилалари			
naphtholonitrile	Cu, V, Mg	CuCl, $(NH_4)_2MoO_4$, термоядровий, 260- 270 °C, 5–6 с	[51]
anthracene purified phthalic anhydride	V	VCl_3, $(NH_2)_2CO$, $(NH_4)_2MoO_4$, 1-chloronaphthalene 235 °C, 4 h	[31]
	In	$InCl_3$, $(NH_4)_2MoO_4$, ДМФА, 220 °C, 2с	[51]

1.3. Application of modified phthalocyanine pigments as additives to paints and polymers

Polymer particles (colored latex particles) containing oil-soluble dyes were obtained by means of emulsion polymerization. Copper phthalocyanine and styryl dyes are used as oil-soluble dyes. The highly hydrophobic dyes themselves played the role of hydrophobes and ensure complete penetration of the dyes into the latex without additional hydrophobes. Two phthalocyanine dyes of similar color were mixed using mini-emulsion polymerization, and the resulting colored latex showed sufficient color intensity as a colorant. Colored latexes with a high dye content (more than 30% by weight for phthalocyanine dyes and 40% for styryl) and colored latexes with a particle size of less than 100 nm have been obtained [52].

It is presented that the dyes obtained by modifying the ground green pigment G in the state of fine particles in the presence of 3-aminoethyl-g-aminopropyl methyldimethoxysilane can be used in fabric dyeing. In this case, it was determined that 0.04 mm thick paint and 10% pigment in relation to monomer should be used as optimal conditions. The pigment obtained in this case showed high abrasion and washing properties [53].

The interaction between aniline cation and copper tetrasulfophthalocyanine anion was studied in aqueous sulfuric acid solution using electronic absorption spectra. When 0.15 M aniline is introduced into the solution at a dye concentration of 10-3 M, the complex (salt) between the cation of aniline and the anion of copper tetrasulfophthalocyanine begins to precipitate. The influence of the dye on the kinetics of aniline polymerization and the properties of the obtained polymer was studied. Phthalocyanine accelerates the electropolymerization of aniline and is immobilized within the polymer matrix. It was shown that the self-catalytic synthesis mechanism characteristic of polyaniline is preserved in the case of the composition of polyaniline-copper tetrasulfophthalocyanine [54].

A number of scientific researches on the synthesis and modification of phthalocyanine pigments are also being conducted in our country. Scientists of the Tashkent Research Institute of Chemical Technology have a special place in this field.

Optimum conditions for obtaining metal phthalocyanines modified with diamidophosphate have been determined. Their properties are studied by physico-chemical methods. Copper, nickel [55], cobalt [56], approximate structural formulas of phthalocyanine dyes modified with diamidophosphate containing iron and cadmium metals were determined. From the obtained pigments, the scientific and practical basis of using local raw materials in the production of nitrogen and phosphorus-retaining phthalocyanine compounds in the chemical industry was developed, the optimal conditions of the process of obtaining nitrogen- and phosphorus-retaining phthalocyanine compounds for polymer composite materials, including lacquer coatings, their structure and other important characteristics were determined. , the technology of obtaining loco-paint coatings resistant to atmospheric and aggressive external environments based on new types of phthalocyanine compounds was developed, technical and technological recommendations for obtaining loco-paint materials based on new types of phthalocyanine compounds were developed [56-62].

Phthalocyanine pigments containing several metals have also been obtained. A new composition of copper and copper-calcium-preserving phthalocyanines has been synthesized. Pigments conforming to the requirements of GOST-6220-76 were obtained by treating synthesized copper and copper-calcium-preserving phthalocyanines with 90% sulfuric acid. Anticorrosion, static and dynamic strength properties of products using synthesized copper and copper-calcium-preserving phthalocyanine pigments were determined. It has been proven that the chemical, physical and mechanical properties of polymer and varnish coatings with copper and copper-calcium preserving phthalocyanine pigments depend on phthalocyanine pigments. Based on phthalic anhydride, urea, metal salts and catalysts, an economically efficient and environmentally friendly technology for obtaining new phthalocyanine pigments has been developed [63- 70].

It is also widely used in the process of coloring polymers from phthalocyanine dyes. Amorphous copper phthalocyanine solutions in dioxane and heptane are used in the process of coloring polymers based on polyethylene, polypropylene, polycaproamide and cellulose. The process of obtaining such stable salts is directly related to their coloring level, studied by electronic and infrared spectroscopic methods [71].

The distribution of charges on the surface of polyethylene as a result of dyeing with different phthalocyanine pigments, the degree of distribution of dyes as a result of deformation processes was analyzed. In this case, it was proved that polyethylene painted with phthalocyanine pigments is 4 times better than polyethylene painted with titanium white paint during deformation processes [72; pp. 106–111].

Polyethylene compositions containing 1.0-2.0% pigment were obtained for agricultural use. Composite materials with 27-28% transparency for visible rays, 22-23% transparency for infrared radiation, and 80-82% post-ultraviolet luminescence [73; pp. 6–10].

Special composite compounds have been synthesized for coloring polymers, and their composition has been determined according to the characteristics of the materials to be dyed. One such composition is a composite compound containing 25-30% blue phthalocyanine pigment. In this case, oxyethylated alkylphenols, monoalkyl ethers of ethylene glycol were used to increase the level of dispersion [74;].

For coloring polymers and materials based on them, a mixture of blue pigment with epoxy oligoether in various proportions was obtained by plasticizing at 80-90°C for 3-4 minutes and extruding at 100-110°C [75].

Various modifications of phthalocyanine were also used for solar panel applications, and the sensitization of the obtained films was measured using a light source. The efficiency of solar panels measured over 10 days was found to be 0.055% [76].

The properties of linear low-density polyethylene products obtained by rotational molding technology were studied. The effect of pigment additives and secondary materials included in such compositions on the degree of elongation, breaking, hardness and other properties of the product was considered. It was determined that the mechanical properties of composites containing up to 5% phthalocyanine dyes do not change significantly [77].

Deformation heat resistance of polypropylene pipes at a pressure of 0.45 MPa has been found to increase from 84°C to 105°C in composites containing 0.5% pigment. The crystallization temperature has also been found to increase up to 15%. The possibility of using phthalocyanine pigments for painting polypropylene pipes was considered. Changes in the physical and mechanical properties compared to the properties of pure polypropylene pipes have been determined [78; pp. 46–51].

A three-layer composite polymer film was used to protect the skin. In this case, the outer layer is made of polyethylene or polyurethane, and the inner layer is made of polyethylene terephthalate. Changes in the mechanical properties of these layers with different compositions, paints and thickness ratios were considered [79;].

The possibility of coloring the high temperature resistant PA6 material with glass filler with blue phthalocyanine pigment was studied. In this case, it was found that it has effective dyeing capabilities even at high temperatures [80; pp. 55–58].

The effect of blue phthalocyanine pigment on the crystallization and structure of polypropylene was studied. The introduction of blue pigment causes an increase in the crystallization temperature of polypropylene. Using the method of electron microscopy, the dependence of the temperature of crystallization and the change in the size and shape of pigment particles during the dyeing process of polymers was determined [81].

Phthalocyanine compounds used as dyes are practically insoluble in water. As a result, stable sols obtained in organic solvents are often used in the preparation of paints. However, even when one sulfo group is introduced through modification, its solubility increases at the expense of the hydrophilic group and makes it possible to prepare water-miscible dyes [82]. Obtaining stable aqueous suspensions of phthalocyanines is of great practical importance. Besides the choice of solvents, surface modification methods of phthalocyanines can be used as an alternative way. For example, relatively stable suspensions were obtained by modifying the surface of copper phthalocyanine in the presence of stearic acid with various dispersants [83].

As a result of surface modification of phthalocyanines with compounds containing functional groups, technologies for obtaining stable sols that can be stored longer in various liquid systems have been developed. For example, the stability of pigments modified with 4-mercaptobenzenesulfoacids has been found to be much higher in aqueous media. In the process of creating such stable systems, methods such as sulfochlorination, hydroxymethylation, halogenomethylation are also used [84,85].

The possibility of using butyl phthalates as a waste product of the process of obtaining butyl alcohol by oxosynthesis to cellulose ethers as flame retardant plasticizers was studied [86].

Polyethylene terephthalate (PET) polymers, which are widely used today, are very slow to degrade, and many environmental problems can be solved by recycling these sustainable polymers. One way to dispose of PET waste is to chemically recycle it. Methods of obtaining hardening compounds for foam polyurethanes, foam phenoplasts, oligoethers, unsaturated polyester resins, and epoxy resins have been developed from secondary PET [87].

In 2018, more than 359 million tons of plastic were produced worldwide, with significant growth expected in the near future, leading to a global challenge of end-of-life management. The recent discovery of enzymes that break down plastics that are considered non-biodegradable opens up opportunities to shift the plastic recycling industry into the realm of biotechnology. The sequential conversion of secondary polyethylene terephthalate (PET) into two types of bioplastics has been demonstrated: medium-length polyhydroxyalkanoates and novel biobased poly(amide urethanes). PET films are hydrolyzed with thermostable polyester hydrolase, producing highly pure terephthalate and ethylene glycol. The resulting hydrolyzate is directly used as a raw material for terephthalate-degrading Pseudomonas umsongensis GO16, which has also become a raw material for the efficient conversion of ethylene glycol and the production of polyhydroxyalkanoates. The strain is then modified to secrete hydroxyalkanoyloxy-alkanoates, which are used as monomers for the chemical-catalytic synthesis of poly(amide urethane). The authors identified a new technological process value for PET recycling. Avoiding the expensive purification methods of PET monomers, it is possible to implement a new technological flexibility based on the recycling of plastics. [88].

Biodegradation of plastics is a promising way to counter the increasing pollution of our planet with man-made polymers and to develop environmentally friendly recycling strategies. Polyethylene terephthalate (PET) is a thermoplastic that has been industrially produced from fossil raw materials since the 1940s and is widely used in packaging and textiles today. Although there are established industrial processes for recycling PET, large quantities of PET still end up in the environment - much of it in the world's oceans. In 2016, the bacterium Ideonella sakaiensis has the ability to break down PET and use the degradation products as the sole carbon source for growth. Ideonella sakaiensis harbors the main enzyme responsible for the breakdown of PET into monomers. This hydrolase may have great potential for the development of PET biodegradation and recycling processes, as well as bioremediation methods for environmental plastic waste. A colony of microbial cells was created capable of producing large quantities of an engineered version of this PET-degrading enzyme and releasing the resulting products into the environment. Preliminary degradation experiments at 30°C showed activity of these enzymes against PET and copolymer polyethylene terephthalate glycol [89]

Oligomeric compounds derived from urea, phosphoric acid and metal oxides have also been used as fillers and additives for various polymer materials. In this case, the fillers caused an increase in the burning temperature of polymers [90-92].

Compounds based on terephthalic acid and metal oxides were added as additives to polymer materials. The obtained substances were studied by means of IR-spectroscopic and SEM analysis. It was found that metal terephthalates obtained in this way significantly improve the physico-chemical properties of polymers [93-95].

Conclusions on chapter I

During the analysis of the literature, the types of phthalocyanine compounds and their possibilities of use as pigments, electronic devices, sensors and catalysts were studied. Methods of obtaining compounds with special properties through their modification are given. Currently, the main methods of modification of phthalocyanine compounds are explained with examples. Information is provided on their main modifications used in the lacquer industry.

Studies on the methods of degradation of phthalocyanine compounds in the natural environment and the biological degradation of polyethylene terephthalates are presented.

CHAPTER II. SYNTHESIS AND RESEARCH METHODS OF NEW METAL-BEARING ORGANIC PIGMENTS BASED ON TEREPTHLIC ACID

2.1-§. Research objects

Terephthalic acid is a colorless white crystalline substance that is practically insoluble in water and acetic acid. Soluble in organic solvents - 6.7 g/100 ml in dimethylformamide and 20 g/100 ml in dimethylsulfoxide. The molecular mass of terephthalic acid is M=166.14 g/mol, density d204=1.51 g/cm^3, melting temperature Tq = 427 °C, boiling point Tqay = 680 °C.

Urea (urea) - white crystalline powder, chemical formula CO(NH2)2, molecular mass M=60.06 g/mol, density d204=1.32 g/cm^3, liquid temperature Liquid = 132.7°C, boiling temperature Tqay = 174°C. It is well soluble in water, soluble in some organic solvents, in particular ethyl and methyl alcohol, glycerin and ether.

Phthalic anhydride is a colorless white crystalline substance, almost insoluble in water, moderately soluble in organic solvents. The molecular mass of phthalic anhydride is M=148.12 g/mol, density д204=1.527 g/cm^3, melting point Tsuyuq = 130.85°C, boiling point Tqay = 284°C. It exhibits properties of aromatic compounds. When heated in alcohol in the presence of sulfuric acid, it forms complex mono- and diethers, polyethers with polyhydric alcohols (alkyd resins). When the benzene ring is chlorinated, tetrachlorophthalic anhydride monomer is formed to produce quenching resins. Phthalic anhydride is used as a raw material in the production of materials such as polymers, various paint pigments.

Copper (I) chloride is a white or green powder that is poorly soluble in water. Molecular mass of copper chloride M= 98.999 g/mol, density d=4.145 g/cm3, liquid temperature Tsuyuq = 426°C, boiling point Tqay = 1490°C, chemical formula CuCl.

Cobalt (II) chloride - Paramagnetic hygroscopic bright red hexagonal crystals, the color turns blue when dehydrated. Melting point 735°C, boiling point: 1049°C, molar electrical conductivity 260.7·cm^2/mol at infinite dilution at 25°C.

Relative density (water=1): 3.356, vapor pressure at 770°C is 5.33 kPa. Soluble in water, methyl and ethyl alcohol, acetone. Insoluble in pyridine and methyl acetate.

Calcium (II) chloride is a white crystalline inorganic substance. The molecular mass of calcium chloride M = 111.08 g/mol, density d = 2.15 g/cm3, liquid temperature Tyuyuq = 772°C, boiling point Tqay = 1935°C, chemical formula $CaCl_2$.

Ammonium heptamolybdate is an inorganic compound with the chemical formula $(NH_4)_6Mo7O_{24}\cdot 4H_2O$. It is used as a catalyst for the synthesis of organic phthalocyanine pigments. It is well soluble in water and forms crystalline hydrates.

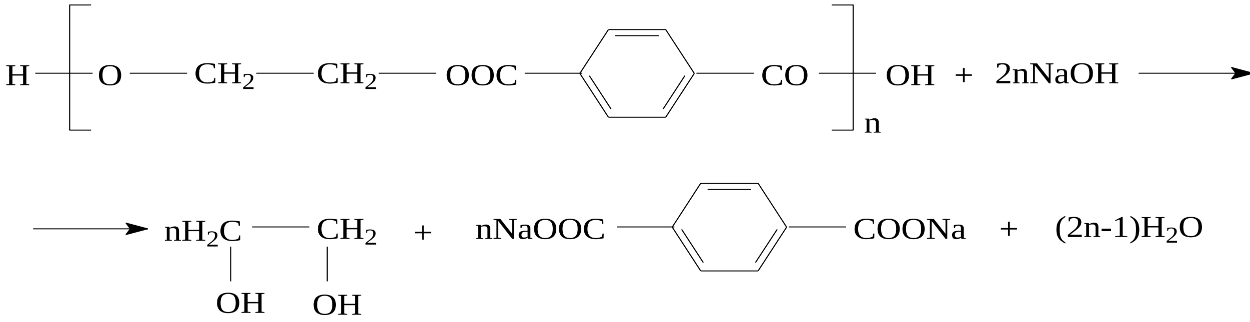
Sulfuric acid is a strong acid with H_2SO_4 2 base. Odorless, colorless, heavy oily liquid under normal conditions; density 1.83 g/cm3 (at 15°C), freezing point 10.45°C, boiling point 296.2°C. When sulfuric acid dissolves in water, it releases a lot of heat. By cooling solutions of sulfuric acid in water, its hydrates containing $H_2SO_4\cdot H_2O$, $H_2SO_4\cdot 2H_2O$, $H_2SO_4\cdot 4H_2O$ were obtained.

§ 2.2. Research methods

2.2.1. Synthesis of terephthalic acid

Alkaline hydrolysis of polyethylene terephthalate was first described by Waters in 1950 [96]. It is known that PET is very resistant to weak alkaline solutions, resistant to concentrated alkaline solutions at room temperature and begins to decompose only at the boiling point [97] This is due to the high packing density of PET not only in crystalline but also in amorphous parts. But this is an approximate qualitative indicator of polymer stability.

Only by repeated hydrolytic cleavage can the polymer be broken down into fragments corresponding to one monomer unit. The surface relief is formed for a certain period of time and does not change after that [98]. According to some researchers, the peculiarity of the hydrolysis reaction of PET with sodium hydroxide is its spontaneous acceleration, which is explained by the activity of the amorphous phase and the increase in the number of acid groups. The hydrolysis reaction of PET with sodium hydroxide is as follows:



1750-1800 rpm to obtain terephthalic acid from secondary PET. A reactor equipped with a high-speed mechanical stirrer and a thermometer was used. Hydrolysis of polyethylene terephthalate is carried out in the following sequence.

For the study, we mainly used secondary PET. The process was carried out for 2 hours at a temperature of 95-100°C using a 40% sodium hydroxide solution. An aqueous solution of sodium terephthalate was formed as a reaction product. The resulting solution was then diluted with water and filtered to remove unreacted PET. The resulting solution was neutralized with sulfuric acid. The formed terephthalic acid was separated in the form of a white powdery precipitate because it was not well soluble in water. The precipitate was filtered and then washed 3-4 times with boiling water. We dried the finished product in a drying oven at a temperature of 55-60°C until it reached a constant mass. The obtained terephthalic acid was used as a raw material for the synthesis of organic pigments, which are the main focus of our research.

2.2.2. Methods of determining the structure and properties of synthesized substances and the tools and equipment used

IR-spectroscopy-IR-spectroscopy (Fure spectrometer made in Japan. IR spectroscopic studies were carried out in the SHIMADZU infrared Fure spectrometer (range 4000-600 cm-1, dimensions 4 cm-1) in the powder method. In order to find out if there is a difference in physicochemical properties, and in other similar cases, as well as the composition of the synthesized substances, the composition of the synthesized substances was simulated using IR-spectra. - 10-3 m) by analyzing the IR-spectrum, which functional groups are present and what class the samples belong to.

Scanning Electron Microscope (SEM) - The newly synthesized semiconductor polymer and phthalocyanine-based dyes were studied with a MIRA 2 LMU scanning electron microscope equipped with an INCA Energy 350 energy dispersive microanalysis system. A scanning electron microscope shows the degree of reaction of the substances, the composition and structure of the layers near the surface from the image of the sample magnified by 100, 200, 500 and 1000 times. At the same time, it can be seen from the 100 to 500 times magnified images of semiconducting substances such as polyaniline, graphene oxide, phthalocyanine-based dyes containing silicon, copper and zinc that the porosity of the dye crystals is high and the absence of additives increases the intensity of the pigments.

Elemental analysis - the composition of organic pigments obtained using this analysis and lok - dyes prepared on their basis was analyzed based on elemental analysis, equipped with the INCA Energy 350 energy dispersive microanalysis system, based on the data of the MIRA 2 LMU scanning electron microscope. This method of elemental analysis is designed to qualitatively and quantitatively determine the elemental composition of the initial and newly synthesized substances in various aggregate states, for example: liquid, solid and gaseous substances and materials.

Thermal analysis - The thermal stability of the synthesized pigments and lacquer based on them was analyzed by differential-thermal and thermogravimetric methods in the device of the Japanese company SHIMADZU. SHIMADZU (simultaneous thermal analysis) TGA and simultaneous analysis of TGA-DTA, TGA-DSC analysis methods is an easy-to-use, reliable and high-performance thermal analysis platform. It was studied in the derivativeograph at a speed of 10 degrees/min, at the sensitivity of the T-900, TG-200, DTA-1/10, DTG-1/10 galvanometer, by automatically recording the derivativeogram on photo paper. A sample of the studied pigments with a mass of 35-46 mg was placed in a crucible made of aluminum oxide and platinum, without a cover, resistant to temperature of 1650°C, with a diameter of 10 mm. The differential mode of heating was carried out under atmospheric conditions. The pigments synthesized during the analysis were thermally analyzed up to a temperature of 20-600°C. In addition, endothermic and exothermic points of pigments were proved.

UV-spectrophotometer-UV-visible spectrophotometers are used to determine the amount of substances and components in solutions, to measure the optical properties of samples, as well as for various research purposes.

Universal testing machine - the mechanical properties of the samples, such as breaking strength and tensile strength, were studied on the AGS-X device of the Shimadzu company, manufactured in Japan. Experiments were performed at room temperature at a maximum tension of 10 kH.

2 3. § Synthesis of new metal-containing organic pigments based on terephthalic acid and study of the influence of various factors on the synthesis process

2.3.1. Synthesis of M-1 organic pigment and the effect of various factors on the synthesis process.

To carry out the experiment, 16.6 g (0.1 mol) terephthalic acid, 14.8 g (0.1 mol) phthalic anhydride, 30 g (0.5 mol) of urea, 4.95 g (0.05 mol) of copper (I) chloride and 0.31 g (0.0005 mol) of ammonium heptamolybdate as a catalyst were mixed. The resulting mixture of substances was slowly heated in the SNOL-8.2/1100 furnace until it became a liquid at 130-135°C. At this temperature, phthalic anhydride and urea liquefy, terephthalic acid dissolves in this liquefaction. After complete liquefaction, the mixture was heated in an oven at 195°C for 1 hour.

After the completion of the reaction, a porous brown substance was formed in the container. The resulting substance was cooled to room temperature and mixed with 10 ml of 92% concentrated sulfuric acid, resulting in a deep dark green solution. The solution was washed several times with boiling distilled water until a neutral medium was obtained. Initial products and intermediate products that did not enter into the reaction were dissolved. The resulting M-1 organic pigment precipitated and was filtered off using a vacuum pump in a Buchner funnel. The final product was dried in a drying oven at 60°C to constant mass. The dried organic pigment is sieved in a mortar and the finished organic pigment is weighed. The procedure was carried out based on the following reaction equation and the calculated product yield was 87.6%.

The optimal conditions, physico-chemical properties of the obtained M-1 organic pigment were studied, mainly the information on the best optimal composition of the organic pigment obtained with the highest yield was presented. Table 2.1 below shows the effects of temperature and the ratio of starting materials on the product yield in the synthesis of the highly efficient M-1 organic pigment [100

Table 2.1.

Effect of temperature and ratio of starting materials on product yield in M-1 brand organic pigment synthesis

1	**Тк:Fa:CaR:CuCl**	**T, ºC**	**ω,%**	**№**	**Тк:Fa:Car:CuCl**	**T, ºC**	**ω,%**
1	1:1:1:0,25	175	11,5	**13**	1:1:5:0,25	175	47,4
2		195	23,5	**14**		195	68,5
3		210	22,5	**15**		210	63,2
4		225	22,1	**16**		225	61,2
5	1:0,7:1:0,25	175	25,4	**17**	1:1:5:0,5	175	71,2
6		195	28,3	**18**		195	87,6
7		210	28,4	**19**		210	83,3
8		225	23,6	**20**		225	80,2
9	1:1:3:0,25	175	38,1	**21**	1:1:5:0,75	175	68,5
10		195	42,4	**22**		195	72,4
11		210	43,1	**23**		210	71,1
12		225	40,5	**24**		225	70,3

Many experiments were conducted in the synthesis of M-1 organic pigment under different conditions and proportions. As a result of the research, the pigment yield obtained was calculated as a function of temperature and the ratio of components. The optimal reaction temperature was 195°C, and the organic pigment obtained in the ratio 1:1:5:0.5 was the highest yield. A graph representing the effect of temperature and mole ratio of initial substances is shown in Figure 2.1 below.

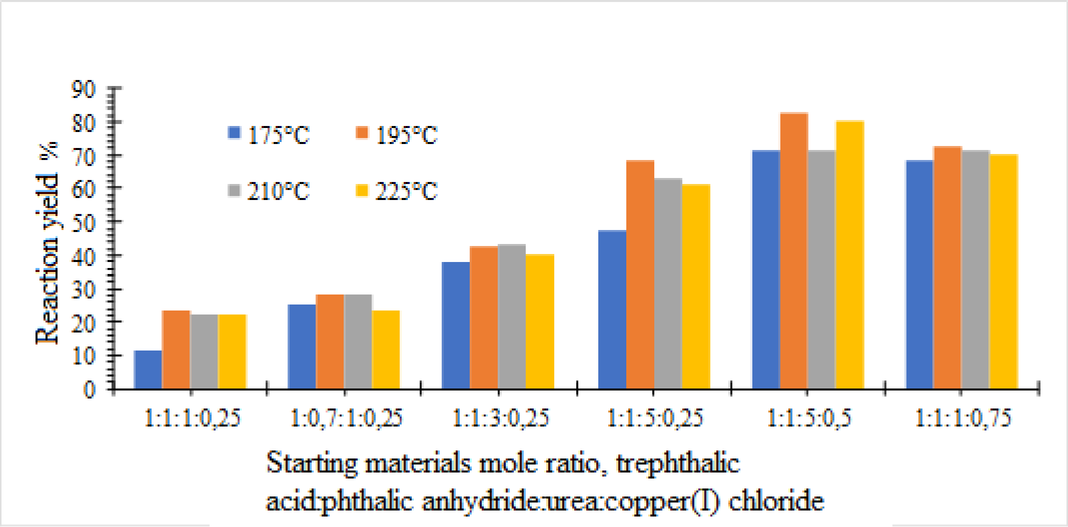


Figure 2.1. The effect of the amount of starting materials and temperature on the reaction yield of M-1 organic pigment

2.3.2. Synthesis of M-2 brand organic pigment and influence of various factors on synthesis process.

The synthesis process was carried out by heating at high temperature.

16.6 g (0.1 mol) terephthalic acid, 14.8 g (0.1 mol) phthalic anhydride, 30 g (0.5 mol) urea, 4, 95 g (0.05 mol) of copper(I) chloride, 11.1 g (0.1 mol) of calcium chloride and 0.31 g (0.005 mol) of ammonium heptamolybdate were added and stirred. The resulting mixture of substances is slowly heated in the SNOL-8.2/1100 furnace until it forms a liquid at 130-135°C. At this temperature, phthalic anhydride and urea liquefy, terephthalic acid dissolves in this liquefaction. After complete liquefaction, the mixture is heated in an oven to 185°C for 1 hour.

After the completion of the reaction, a porous brown substance was formed in the container. The resulting substance was cooled to room temperature and mixed with 10 ml of 92% concentrated sulfuric acid, resulting in a deep dark green solution. The solution is washed several times with boiling distilled water until a neutral medium is formed. Primary products and intermediate products that did not enter into the reaction are dissolved. The resulting M-2 organic pigment is precipitated and filtered using a vacuum pump in a Buchner funnel. The final product is dried in a drying oven at 60°C until it reaches the final mass. The dried organic pigment is crushed in a mortar and sieved, and the finished organic pigment is weighed on a scale. When calculating the finished product, the yield was 91.1%.

The optimal conditions, physical and chemical properties of the obtained M-2 organic pigment were studied, and information on the optimal composition of the organic pigment obtained with the highest yield was presented. Table 2.2 below shows the effects of temperature and the ratio of starting materials on the product yield in the synthesis of the highly effective M-2 organic pigment.

Table 2.2.

Effect of temperature and ratio of starting materials on product yield in M-2 brand organic pigment synthesis

№	**Ta:Fa:U:CuCl:$CaCl_2$**	**T, °C**	**ω,%**	**№**	**Ta:Fa:U:CuCl:$CaCl_2$**	**T, °C**	**ω,%**
1	1:1:1:0,25:0,25	170	15,4	**9**	1:1:5:0,5:1	170	82,1
2		185	25,4	**10**		**185**	**91,1**
3		200	23,2	**11**		200	88,2
4		225	22,1	**12**		225	85,4
5	1:1:5:0,25:0,5	170	63,5	**13**	1:1:5:0,75:0,25	175	82,4
6		185	72,4	**14**		195	87,2
7		200	68,2	**15**		210	85,2
8		225	65,4	**16**		225	84,1

Many experiments were conducted in the synthesis of M-2 brand organic pigment under different conditions and proportions. As a result of the research, the pigment yield obtained was calculated as a function of temperature and the ratio of components. The optimal reaction temperature was 185-200°C, and the organic pigment obtained in the ratio 1:1:5:0.5:1 was the highest yield. A graph representing the effect of temperature and mole ratio of initial substances is shown in Figure 2.2 below.

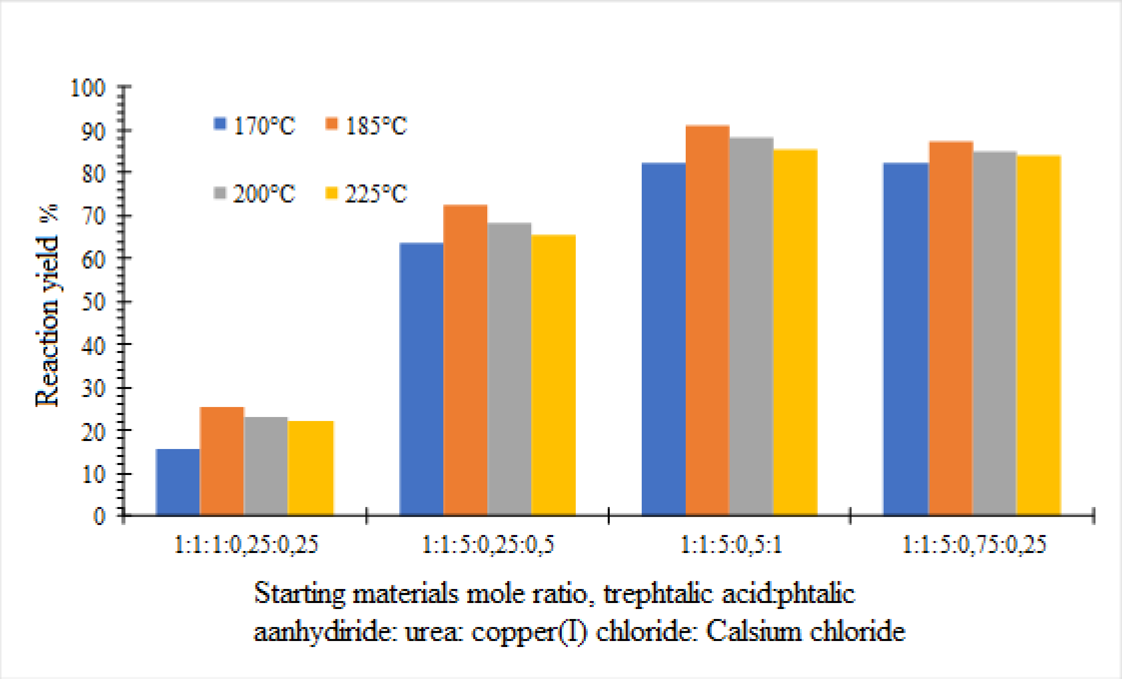


Figure 2.2. The effect of the amount of starting materials and temperature on the reaction yield of M-2 organic pigment

2.3.3. Synthesis of K-1 brand organic pigment and influence of various factors on synthesis process.

16.6 g (0.1 mol) terephthalic acid, 14.8 g (0.1 mol) phthalic anhydride, 30 g (0.5 mol) urea, 6 .5 g (0.05 mol) of cobalt (II) chloride and 0.31 g (0.005 mol) of orthoboric acid as a catalyst were mixed. The resulting mixture of substances is slowly heated in the SNOL-8.2/1100 furnace until it forms a liquid at 130-135°C. At this temperature, phthalic anhydride and urea liquefy, terephthalic acid dissolves in this liquefaction. After complete liquefaction, the mixture is heated in an oven to 195°C for 1 hour.

After the reaction, a porous brown substance is formed in the container. The resulting substance was cooled to room temperature and mixed with 10 ml of 92% concentrated sulfuric acid, resulting in a deep dark green solution. The solution is washed several times with boiling distilled water until a neutral medium is formed. Primary products and intermediate products that did not enter into the reaction are dissolved. The resulting K-1 organic pigment is precipitated and filtered using a vacuum pump in a Buechner funnel. The final product is dried in a drying oven at

60°C until it reaches a constant mass. The dried organic pigment is crushed in a mortar and sieved, and the finished organic pigment is weighed on a scale. When calculating the finished product, the yield was 84.8%.

Optimum conditions, physico-chemical properties of the obtained K-1 organic pigment were studied, and information on the composition of the organic pigment obtained with the highest yield was presented. Table 2.3 below shows the effects of temperature and the ratio of starting materials on the product yield in the synthesis of the highly effective K-1 organic pigment [101].

Table 2.3.

Effect of temperature and ratio of starting materials on product yield in K-1 brand organic pigment synthesis

№	**Ta:Fa:U:CoCl$_2$**	**T, ºC**	**ω,%**	**№**	**Ta:Fa:U:CoCl$_2$**	**T, ºC**	**ω,%**
1	1:1:1:0,25	175	13,4	**13**	1:1:5:0,25	175	47,2
2		195	25,2	**14**		195	64,9
3		210	22,2	**15**		210	63,2
4		225	20,5	**16**		225	61,1
5	1:0,7:1:0,25	175	26,3	**17**	1:1:5:0,5	175	74,4
6		195	32,1	**18**		195	84,8
7		210	27,1	**19**		210	79,4
8		225	25,2	**20**		225	78,1
9	1:1:3:0,25	175	40,1	**21**	1:1:5:0,75	175	70,2
10		195	54,2	**22**		195	75,2
11		210	50,8	**23**		210	73,3
12		225	50	**24**		225	71,4

In the synthesis of K-1 brand organic pigment, many experiments were carried out under different conditions and proportions. As a result of the research, the pigment yield obtained was calculated as a function of temperature and the ratio of components. The optimal reaction temperature was 195°C, and the organic pigment obtained in the ratio 1:1:5:0.5 was the highest yield. A graph representing the effect of temperature and mole ratio of initial substances is shown in Figure 2.3 below.

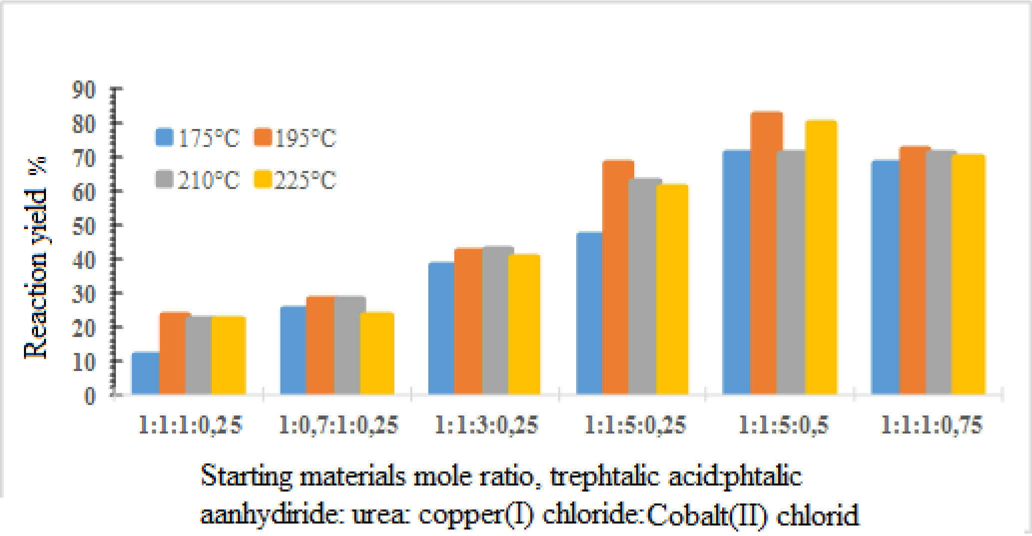


Figure 2.3. The effect of the amount of starting materials and temperature on the reaction yield of K-1 organic pigment

2.3.4. Synthesis of K-2 brand organic pigment and influence of various factors on synthesis process.

16.6 g (0.1 mol) terephthalic acid, 14.8 g (0.1 mol) phthalic anhydride, 30 g (0.5 mol) urea, 6, 5 g (0.05 mol) of cobalt(II) chloride, 11.1 g (0.1 mol) of calcium chloride and 0.31 g (0.005 mol) of orthoboric acid as a catalyst were added and stirred. The resulting mixture of substances is slowly heated in the SNOL-8.2/1100 furnace until it forms a liquid at 130-135°C. At this temperature, phthalic anhydride and urea liquefy, terephthalic acid dissolves in this liquefaction. After complete liquefaction, the mixture is heated in an oven to 185°C for 1 hour.

After the reaction, a porous brown substance is formed in the container. The resulting substance was cooled to room temperature and mixed with 10 ml of 92% concentrated sulfuric acid, resulting in a deep dark green solution. The solution is washed several times with boiling distilled water until a neutral medium is formed. Primary products and intermediate products that did not enter into the reaction are dissolved. The resulting K-2 organic pigment is precipitated and filtered using a vacuum pump in a Buechner funnel. The final product is dried in a drying oven at 60°C until it reaches the final mass. The dried organic pigment is crushed in a mortar and sieved, and the finished organic pigment is weighed on a scale. When calculating the finished product, the yield was 91.3%.

Optimum conditions, physico-chemical properties of the obtained K-2 organic pigment were studied, and information on the optimal composition of the organic pigment obtained with the highest yield was presented. Table 2.4 below shows the effects of temperature and the ratio of starting materials on product yield in the synthesis of highly effective K-2 brand organic pigment.

Table 2.4.

Effect of temperature and ratio of starting materials on product yield in K-2 brand organic pigment synthesis

№	Ta:Fa:U:$CoCl_2$:$CaCl_2$	T, °C	ω,%	№	Ta:Fa:U:$CoCl_2$:$CaCl_2$	T, °C	ω,%
1	1:1:1:0,25:0,25	170	25,2	**9**	1:1:5:0,5:1	170	88,7
2		185	29,1	**10**		185	91,3
3		200	25,1	**11**		200	89,1
4		225	24,7	**12**		225	82,1
5	1:1:5:0,25:0,5	170	58,2	**13**	1:1:5:0,75:0,25	170	74,4
6		185	65,4	**14**		185	88,1
7		200	63,2	**15**		200	82,3
8		225	62,2	**16**		225	80,1

In the synthesis of K-2 brand organic pigment, many experiments were carried out under different conditions and proportions. As a result of the research, the pigment yield obtained was calculated as a function of temperature and the ratio of components. The optimal reaction temperature was 185-200°C, and the organic pigment obtained in the ratio 1:1:5:0.5:1 was the highest yield. A graph representing the effect of temperature and the mole ratio of the initial substances is presented in Figure 2.4 below [102].

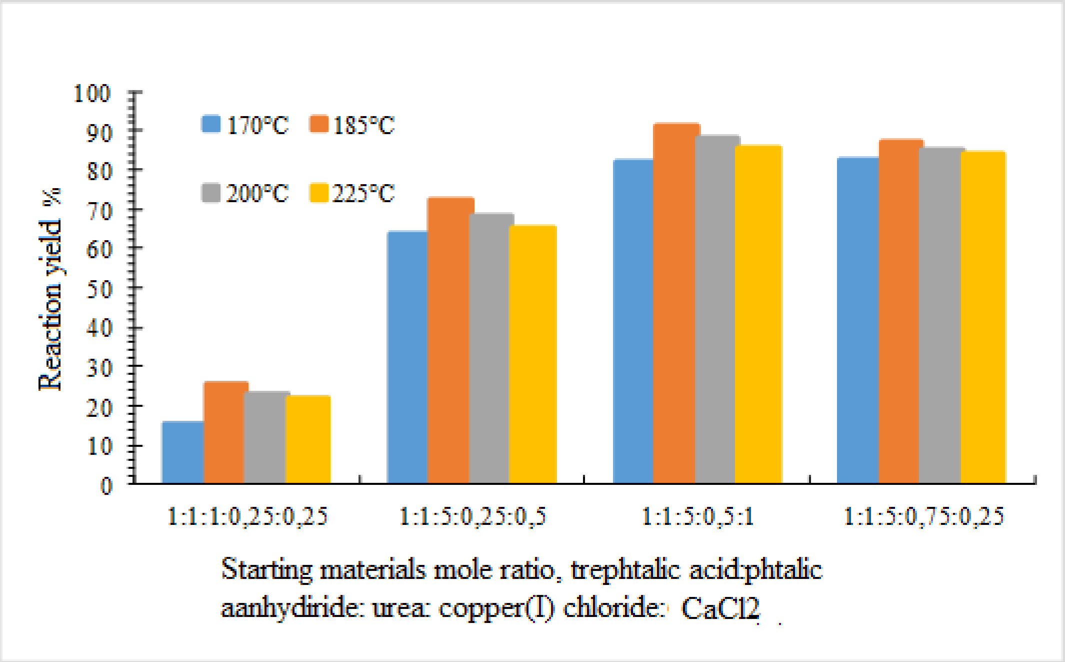


Figure 2.4. Diagram of the effect of the amount of starting materials and temperature on the reaction yield of K-2 organic pigment

§ 2.4. Research of the composition of new synthesized organic pigments by physicochemical methods

2.4.1-IR-spectroscopic analysis of newly synthesized organic pigments

The infrared spectroscopy method was used to determine the functional groups and approximate structural formula of the obtained new organic pigments. The obtained results were compared with the data presented in the literature and analyzed (Figures 2.8.-2.11).

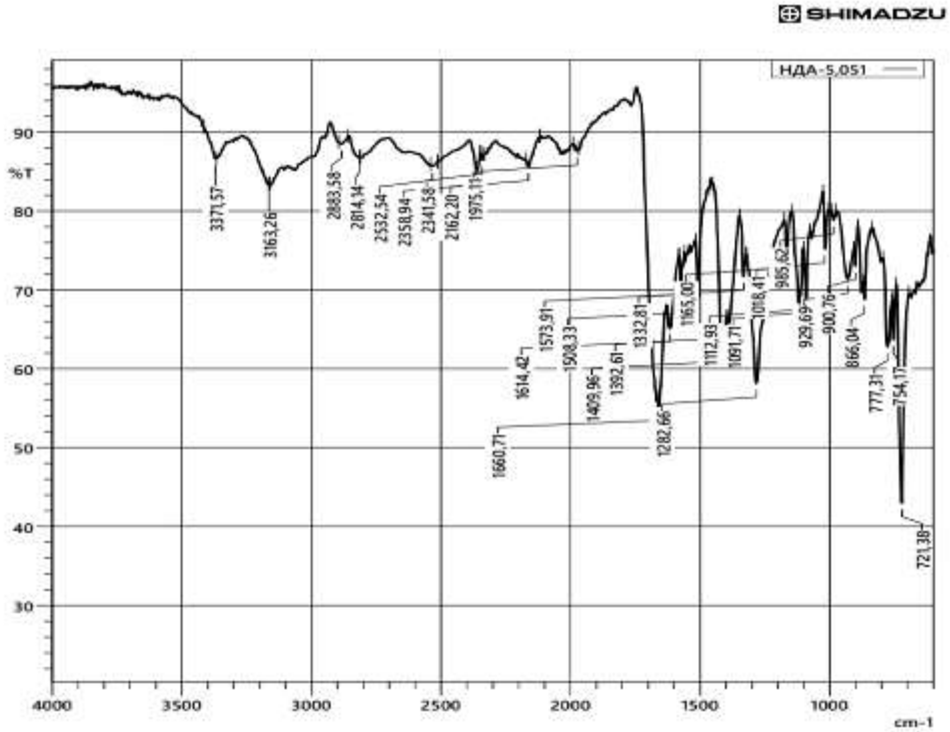


Figure 2.8. IR spectrum of new organic pigment M-1.

It can be seen from the IR-spectrum of the obtained compound that the intense absorption bands corresponding to the carboxyl groups of terephthalic acid are not visible. In the area larger than 3000 cm^{-1} there are valence absorption bands belonging to residual water molecules. It was observed that the absorption band belonging to the carbonyl group of terephal acid shifted from 1689 cm^{-1} to 1661 cm^{-1} belonging to amides. In addition, vibrations in the 1409 cm^{-1} region corresponding to the valence vibrations of the isoindole ring and in the 866 cm^{-1} region with deformation vibrations were observed. There are also vibrations of the pyrrole ring in the valence 1508 cm^{-1} and strain 1018 cm^{-1} domains.

Valence vibrations of hydrogen atoms with different positions in benzene rings are in the range of 2800-3000 cm^{-1} and deformation vibrations are in the range of 1600-1900 cm^{-1}. Also, due to the presence of phthalocyanine in the composition of the compound, the vibration of the phthalocyanine ring is observed in the region of 754 cm^{-1} [95].

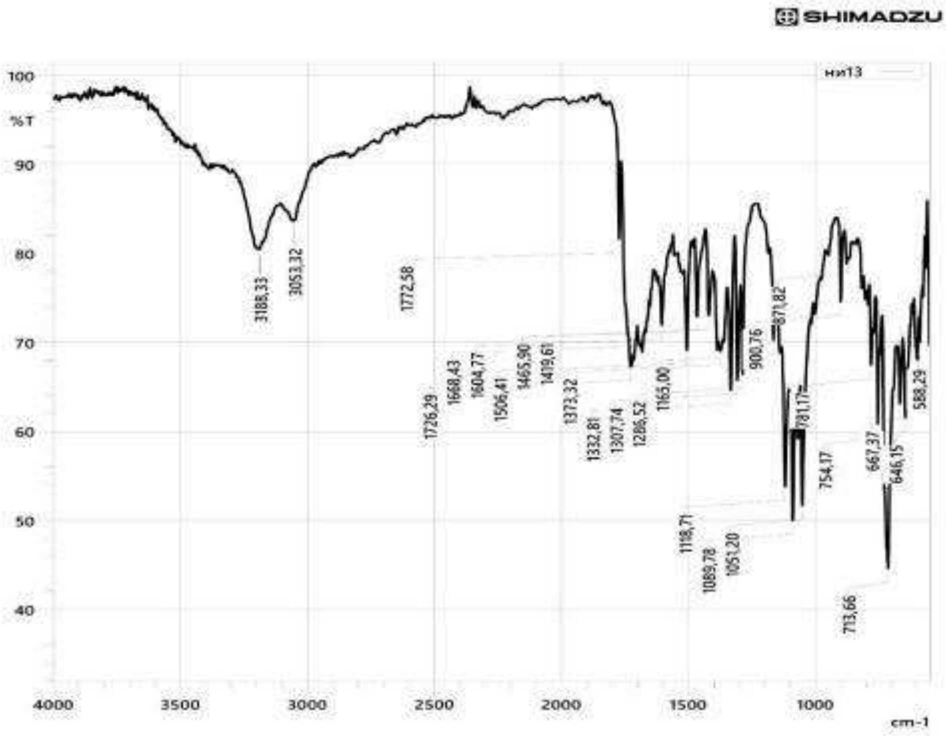


Figure 2.9. IR spectrum of new organic pigment K-1

It can be seen from the IR-spectrum of the obtained compound that the intense absorption bands corresponding to the carboxyl groups of terephthalic acid are not visible. In the area larger than 3000 cm^{-1} there are valence absorption bands belonging to residual water molecules. It was observed that the absorption band belonging to the carbonyl group of terephal acid shifted from 1689 cm^{-1} to 1668 cm^{-1} belonging to amides. In addition, vibrations in the 1419 cm^{-1} region related to the valence vibrations of the isoindole ring and in the 871 cm^{-1} region with deformation vibrations were observed. There are also vibrations of the pyrrole ring in the valence 1506 cm^{-1} and strain 1051 cm^{-1} regions [91].

Hydrogen atoms with different positions in benzene rings have valence vibrations in the region of 2800-3000 cm^{-1} and deformation vibrations of low intensity in the region of 1600-1900 cm^{-1}. Also, due to the partial presence of phthalocyanine in the composition of the compound, the vibration of the phthalocyanine ring is observed at 754 cm^{-1}.

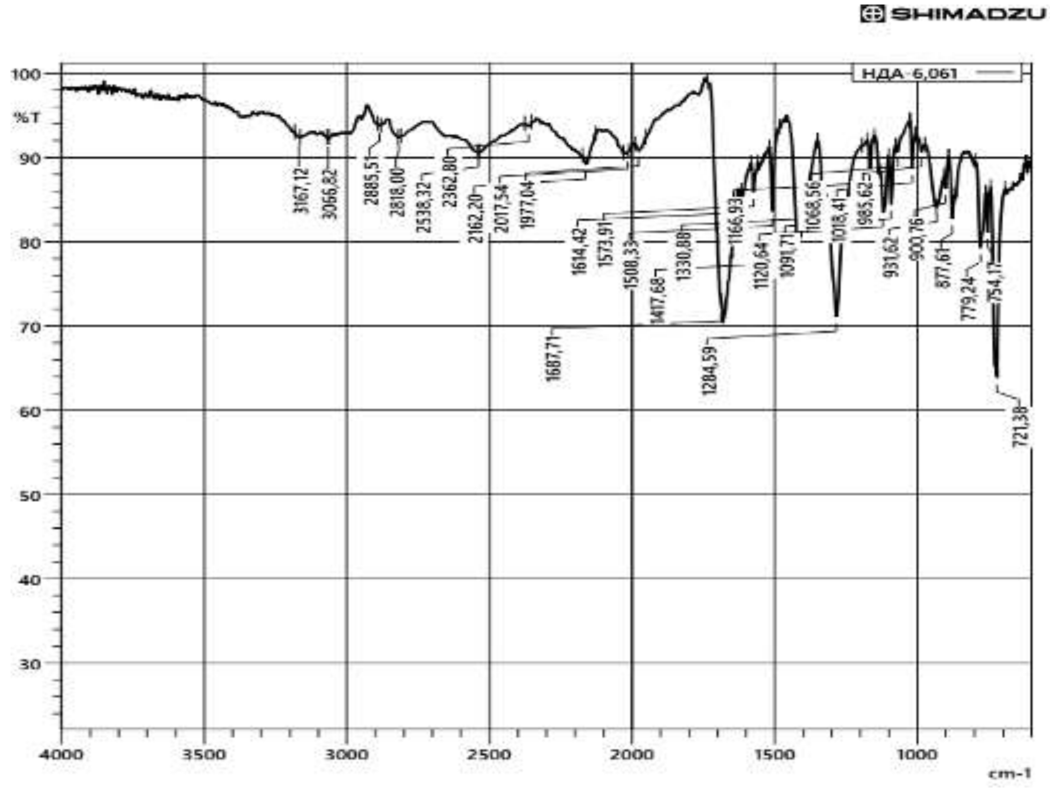


Figure 2.10. IR spectrum of M-2 organic pigment

It can be seen from the IR-spectrum of the obtained compound that the intense absorption bands corresponding to the carboxyl groups of terephthalic acid are not visible. In the area larger than 3000 cm^{-1} there are valence absorption bands belonging to residual water molecules. It was observed that the absorption band belonging to the carbonyl group of terephal acid shifted from 1689 cm^{-1} to 1666 cm^{-1} belonging to amides. In addition, vibrations in the 1417 cm^{-1} region related to the valence vibrations of the isoindole ring and in the 877 cm-1 region with deformation vibrations were observed. There are also vibrations of the pyrrole ring in the valence 1508 cm^{-1} and strain 1018 cm^{-1} domains.

Hydrogen atoms with different positions in benzene rings have valence vibrations in the region of 2800-3000 cm^{-1} and deformation vibrations of low intensity in the region of 1600-1900 cm^{-1}. Also, due to the partial presence of phthalocyanine in the composition of the compound, the vibration of the phthalocyanine ring is observed at 754 cm^{-1} [103].

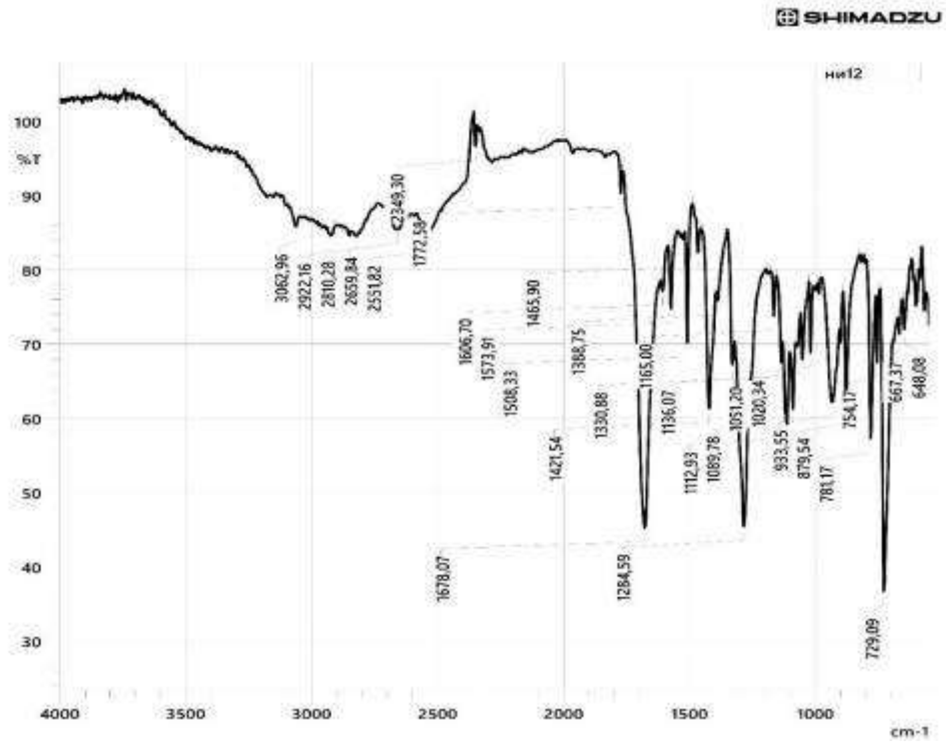


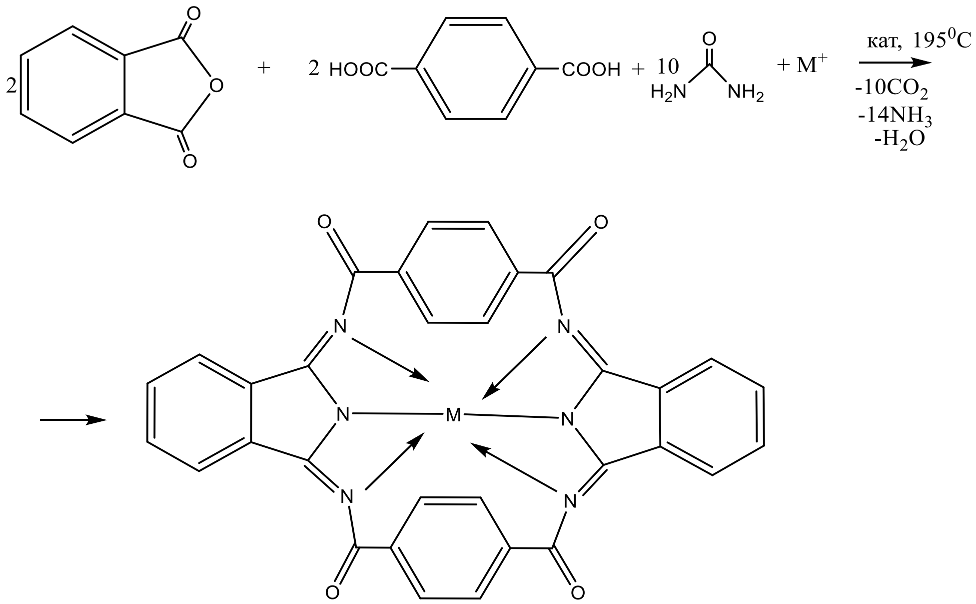
Figure 2.11. IR spectrum of K-2 organic pigment

It can be seen from the IR-spectrum of the obtained compound that the intense absorption bands corresponding to the carboxyl groups of terephthalic acid are not visible. In the area larger than 3000 cm-1 there are valence absorption bands belonging to residual water molecules. It was observed that the absorption band belonging to the carbonyl group of terephal acid shifted from 1689 cm-1 to 1678 cm-1 belonging to amides. In addition, vibrations in the 1421 cm-1 region related to the valence vibrations of the isoindole ring and in the 879 cm-1 region with deformation vibrations were observed. There are also vibrations of the pyrrole ring in the valence 1508 cm-1 and strain 1020 cm-1 domains.

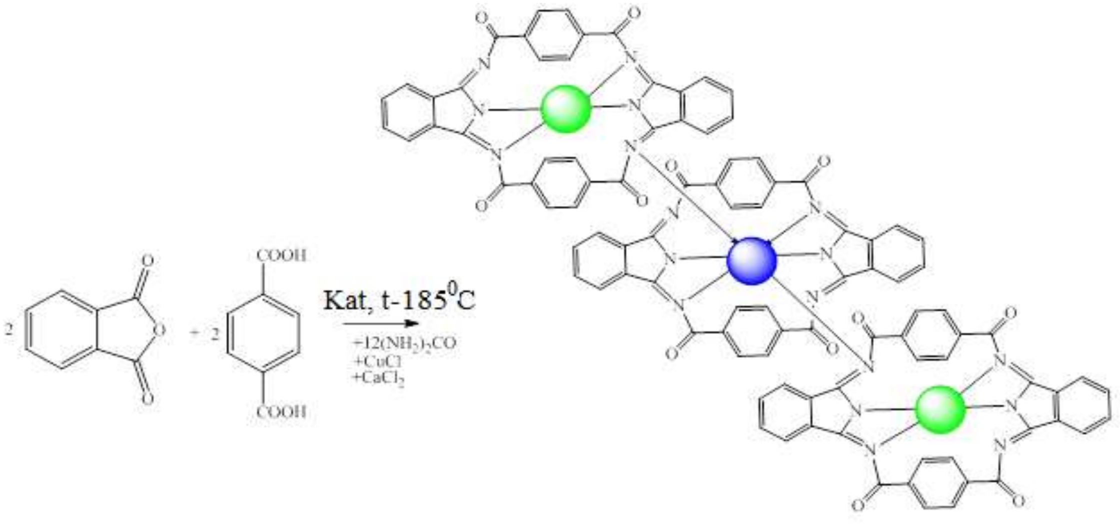
Hydrogen atoms with different positions in benzene rings have valence vibrations in the region of 2800-3000 cm-1 and deformation vibrations of low intensity in the region of 1600-1900 cm-1. Also, due to the partial presence of phthalocyanine in the composition of the compound, the vibration of the phthalocyanine ring is observed at 754 cm-1.

According to the performed IR-spectroscopic analysis, the reaction equations according to the structure and process of the new organic pigments were proposed as follows.

M-1 and K-1 organic pigments were synthesized based on the following reaction equation:



M-2 and K-2 organic pigments were synthesized based on the following reactionequation:



The fact that the synthesized organic pigments were obtained on the basis of the above-mentioned reaction equations and the proposed formulas was confirmed by the conducted studies.

2.4.2-UV-spectrophotometric analysis of newly synthesized organic pigments

UV spectra of the samples were taken in saturated solutions of organic solvent - dimethylformamide, and it was determined which wavelength corresponds to the color intensity of the samples.

Figure 2.12 shows the results of measuring M-1 brand organic pigment under the visible light spectrum of 190-1100 nm in a UV-spectrophotometer. Organic pigment M-1 was found to have a high absorbance in the visible wavelength range of 560 nm to 610 nm.

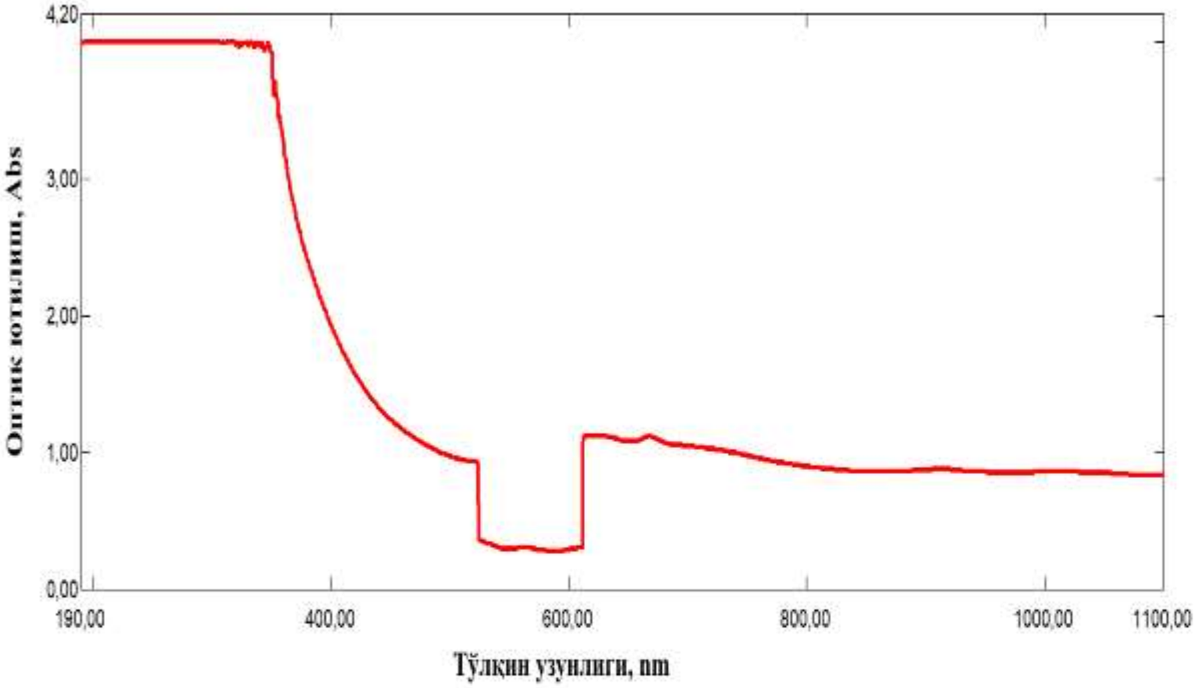


Figure 2.12. Ratio of light absorption to wavelength of M-1 brand organic pigment

Copper monophthalocyanine is often used in practical applications because it is well-studied and has porous, i.e., p-type semiconductivity. Since phthalocyanine complexes are in most cases typical p-type semiconductors, their gas sensitivity is high. The remaining positive charge is delocalized through the two phthalocyanines, resulting in a sharp decrease in resistance, which in turn reduces the time required to charge the organic semiconductor [104].

Figure 2.13 shows the results of measuring M-2 brand organic pigment under the visible light spectrum of 190-1100 nm in a UV-spectrophotometer. M-2 organic pigment was found to have high absorbance in the visible wavelength range from 430 nm to 600 nm. The highest absorption has been proven at a wavelength of 550 nm. As a result, it was found that M-2 organic pigment can also be used as a sensitive dye for solar cells obtained on the basis of sunlight-sensitive dyes, taking into account the ability to absorb photons from light. In the literature, natural pigments were used as dyes for solar cells based on sensitive dyes [104].

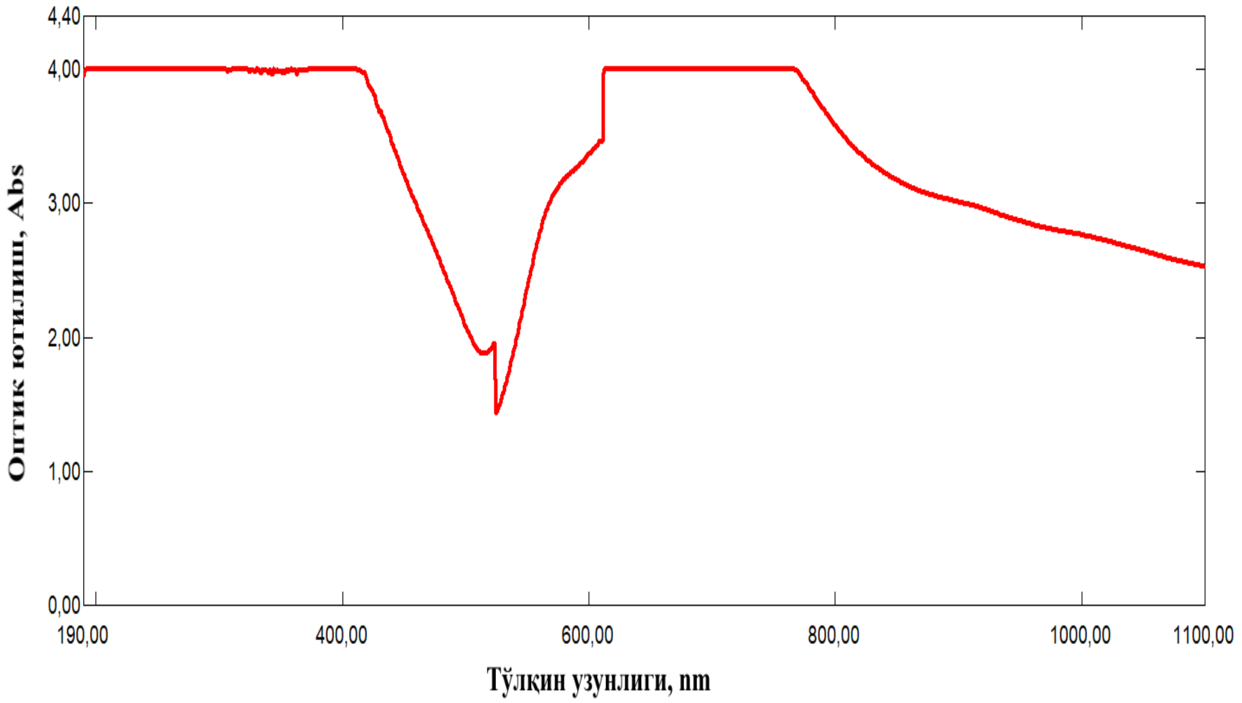


Figure 2.13. Ratio of light absorption to wavelength of M-2 brand organic pigment

Figure 2.14 shows the results of measuring K-1 organic pigment in the UV spectrophotometer under the visible light spectrum of 190-1100 nm. K-1 organic pigment was found to have high absorbance in the visible wavelength range from 430 nm to 600 nm. The highest absorption has been proven at a wavelength of 550 nm. As a result, it was found that K-1 organic pigment can also be used as a sensitive dye for solar elements obtained on the basis of sunlight-sensitive dyes, taking into account the ability to absorb photons from light. In the literature, natural pigments were used as dyes for solar cells based on sensitive dyes [104].

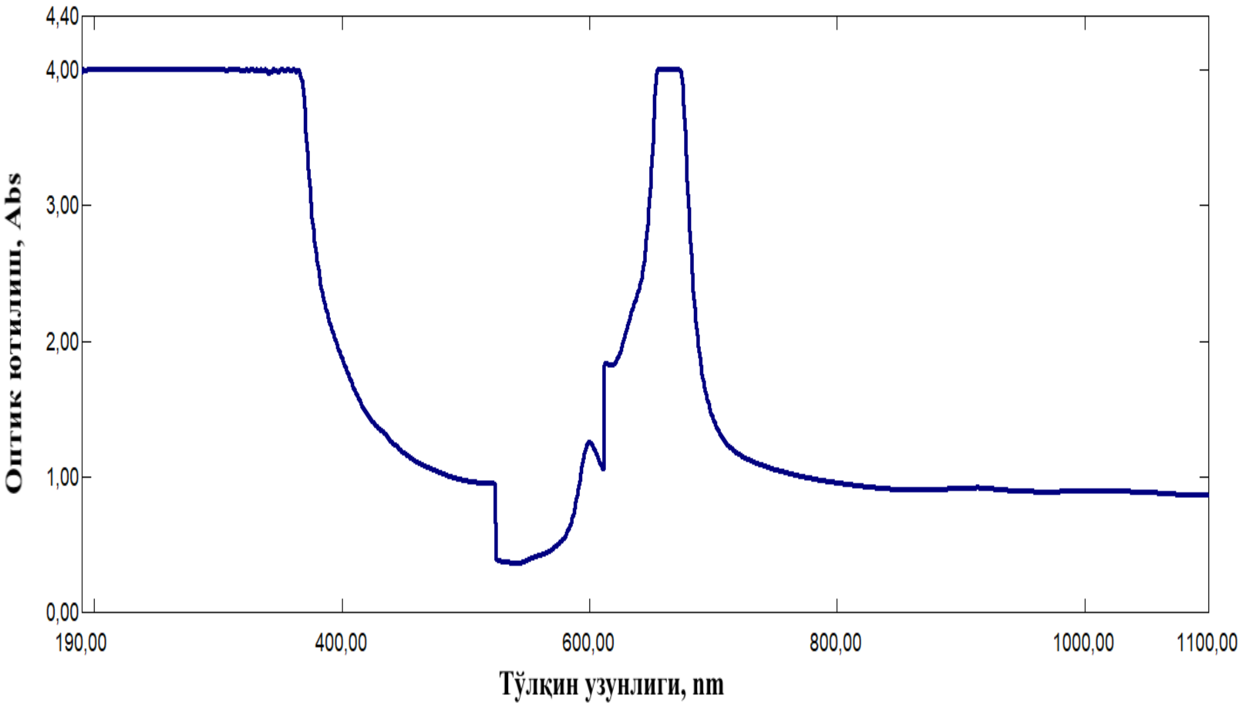


Figure 2.14. Ratio of light absorption to wavelength of K-1 brand organic pigment

The optical absorption peaks of the obtained K-1 brand organic pigment showed characteristic peaks with high absorption level in the range of 530-550 nm and low absorption level in the range of 650-700 nm. The remarkable chemical and photophysical properties of pigments based on terephthalic acid are due to their multi-electron system. A band of strong optical absorption peaks results from the transition of p-p electrons between ground state (HOMO) and excited state (LUMO) energy levels.

Figure 2.15 shows the results of measuring K-2 brand organic pigment in the UV-spectrophotometer under the visible light spectrum of 190-1100 nm. K-2 brand organic pigment was found to have high absorbance in the visible wavelength range of 400 nm to 600 nm. The highest absorption has been proven at a wavelength of 550 nm. As a result, it was found that K-2 organic pigment can also be used as a sensitive dye for solar elements obtained on the basis of sunlight-sensitive dyes, taking into account the ability to absorb photons from light. In the literature, natural pigments were used as dyes for solar cells based on sensitive dyes [104]

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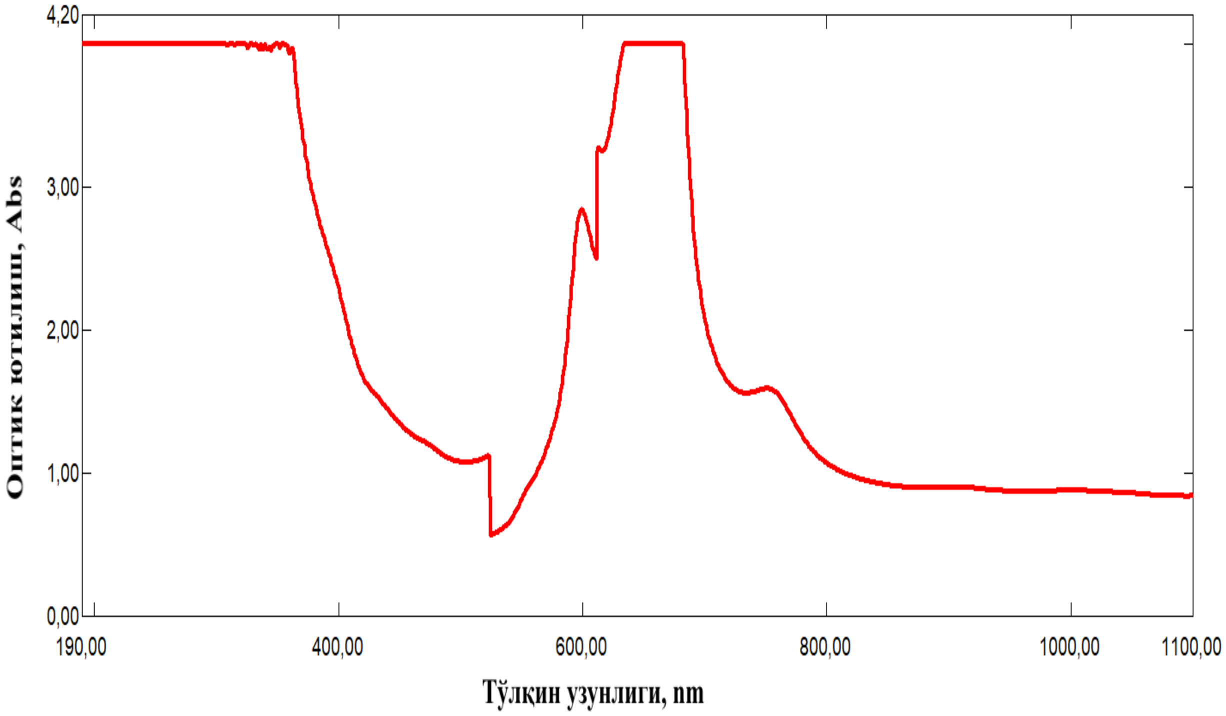


Figure 2.15. Ratio of light absorption to wavelength of K-2 brand organic pigment

According to the obtained results, the color intensity of M-2 brand organic pigments will have the highest values.

2.4.3-Thermogravimetric and differential thermal analysis of newly synthesized organic pigments

Thermal analysis of M-1 brand organic pigment. The synthesized organic pigment was obtained in the presence of terephthalic acid, phthalic anhydride, urea, copper salt and catalyst. Thermogravimetric analysis was performed to study the thermal properties of the compound. Simultaneous TGA and DTA analyzes were performed in the temperature range of 20-600°C on the SHIMADZU DTG-60 thermal analyzer (Fig. 2.16).

Thermal analysis of M-1 brand organic pigments with a new composition was carried out in the temperature range of 20-600°C. All samples of the thermal analysis of the mentioned pigments were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar. In addition, endothermic and exothermic points of organic pigment were proved.

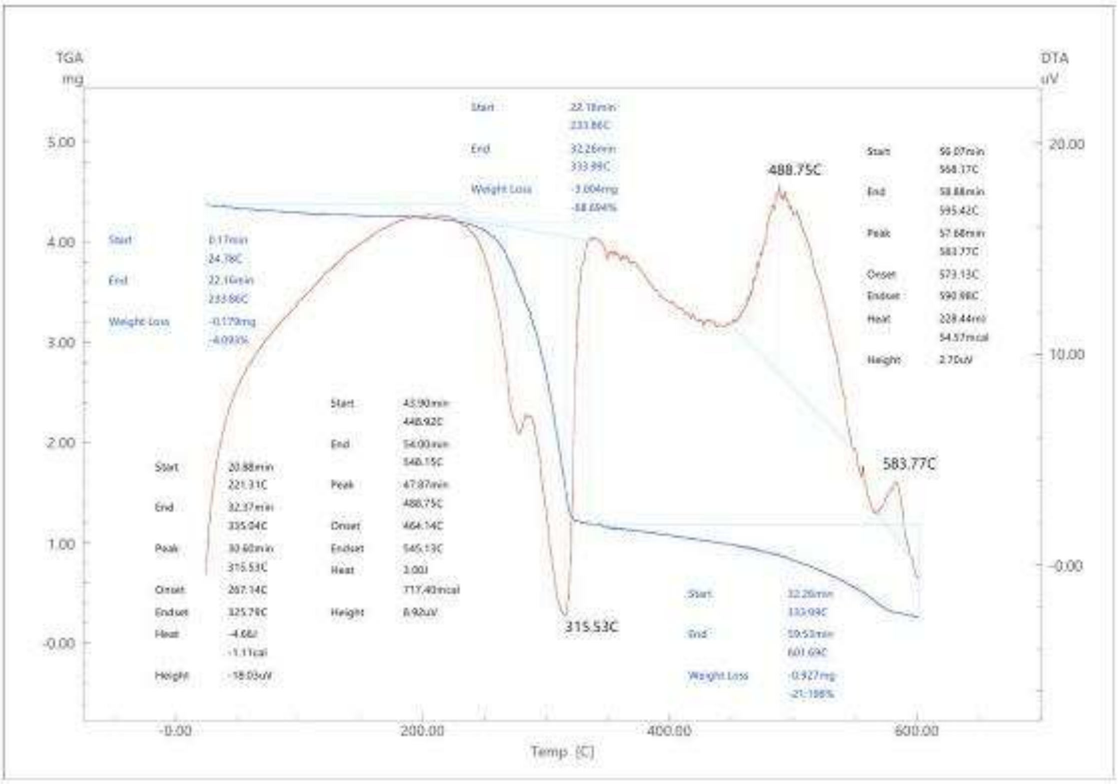


Figure 2.16. Derivatogram of M-1 brand organic pigment

The maximum temperature of 600°C was selected for the organic pigment M-1 with new content synthesized in the dry mass shown in Figure 2.16, and the results of the pigment analysis were studied according to the given thermogravimetric derivatogram (TG) and differential thermogravimetric analysis. Two exothermic effects were observed at 488.75 and 583.77°C and one endothermic effect at 315.53°C. 4.373 mg of pigment was taken in an open-mouth crucible made of M-1 organic pigment 600°C temperature-resistant aluminum, and the temperature was gradually increased starting from 20°C.

The analysis of the thermogravimetric curve of organic pigment shows that the TG curve mainly takes place in the 3 intensive mass loss temperature range. Mass loss range 1 corresponds to temperatures of 24.78 - 233.86°C, mass loss range 2 corresponds to temperatures of 233.86 - 333.99°C, and mass loss range 3 corresponds to temperatures of 333.99 - 600°C. The analysis shows that mass loss 1 is the most intensive decay, with mass loss 0.179 mg, i.e. 4.093% observed in mass loss range 1. The main amount of mass loss in this decay is 3.004 mg, i.e.

68.694%. In the 3rd mass loss interval, the mass loss is 0.927 mg, i.e. 21.198% [105].

It can be seen that the first mass loss is the loss of unbound water. In the second main stage of decomposition, in the case of lower molecular compounds such as carbon dioxide, ammonia, after the decomposition of the organic part, mainly metal carbonates, a mixture of oxides and partially coal remain. In the third stage of decomposition, carbonates are also decomposed, leaving metal oxides and coal residues.

Differential thermogravimetric analysis of an organic pigment is presented in Figure 2.18. Differential thermogravimetric analysis of organic pigment shows that energy absorption occurred in the range of 221.31 - 335.04°C. Energy release occurs between 448.92 - 548.15°C and 568.17 - 595.42°C.

The analysis of the results of the thermal decomposition of the substance at different temperatures is given in Table 2.5.

Thermal analysis of M-2 brand organic pigment.

3.8 mg was taken for thermogravimetric analysis of M-2 organic pigment and the process was studied at temperatures up to 600°C. Thermogravimetric analysis (TG) and differential thermal analysis (DTA) of an organic pigment are shown in Figure 2.18 below.

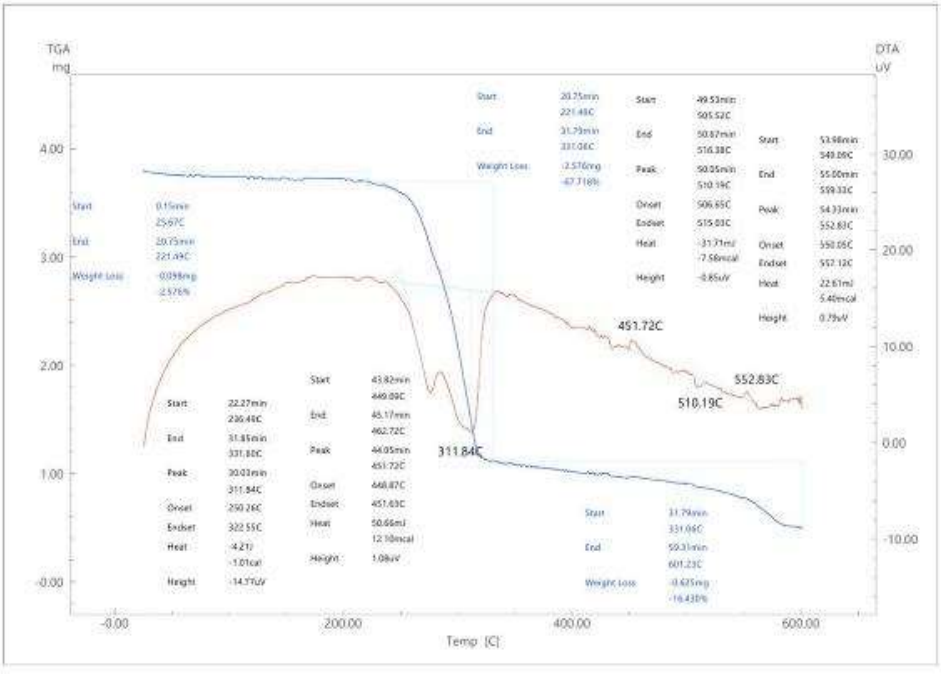


Figure 2.18. Thermogravimetric (TG) and differential thermal analysis (DTA) derivative diagram of M-2 organic pigment.

Analysis of the thermogravimetric curve of M-2 organic pigment shows that the TGA curve mainly takes place in the temperature range of 3 intensive mass losses. Mass loss range 1 corresponds to temperatures of 25.67 - 221.49°C, mass loss range 2 corresponds to temperatures of 221.49 - 331.06°C, and mass loss range 3 corresponds to temperatures of 331.06 - 600°C. The analysis shows that mass loss 1 is the most intensive decay, with mass loss 0.098 mg, i.e. 2.576% observed in mass loss range 1. The main amount of mass loss in this decay is 2.576 mg, i.e. 67.718%. In mass loss interval 3, the mass loss is 0.625 mg, which is 16.43%.

It can be seen that the first mass loss involves the loss of excess moisture and adsorbed water. In the second main stage of decomposition, in the case of lower molecular compounds such as carbon dioxide, ammonia, after the decomposition of the organic part, mainly metal carbonates, a mixture of oxides and partially coal remain. In the third stage of decomposition, carbonates are also decomposed and metal oxides and coal residues remain [106].

Differential thermal analysis of M-2 organic pigment is presented in Fig. 2.19. From the differential thermal analysis of M-2 brand organic pigment, it was revealed that two exothermic effects were observed at temperatures of 451.72°C, 552.83°C and two endothermic effects at temperatures of 311.84°C, 510.19°C.

The analysis of the results of the thermal decomposition of the substance at different temperatures is given in detail.

The synthesized organic pigment was obtained in the presence of terephthalic acid, phthalic anhydride, urea, cobalt salt and catalyst. Thermogravimetric analysis was performed to study the thermal properties of the compound. Simultaneous TG and DTA analyzes were performed in the temperature range of 20-600°C on the SHIMADZU DTG-60 thermal analyzer (Fig. 2.19).

Thermal analysis of K-1 brand organic pigment with new composition was carried out in the temperature range of 20-600°C. All samples of the thermal analysis of the mentioned pigments were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar. In addition, endothermic and exothermic points of organic pigment were proved.

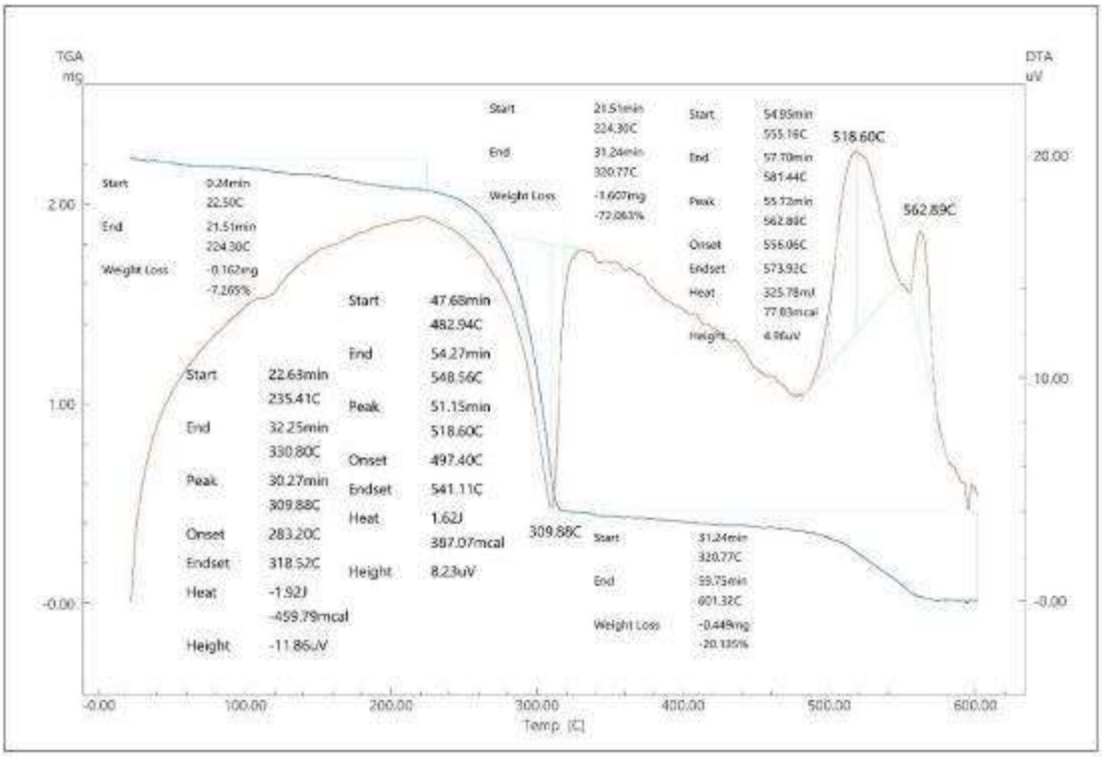


Figure 2.20. Derivatogram of K-1 brand organic pigment

The maximum temperature of 600°C was selected for the organic pigment of K-1 brand with a new content synthesized in the dry mass shown in Figure 2.20, and the results of the pigment's analysis were studied according to the given thermogravimetric derivatogram and differential thermogravimetric analysis. Two exothermic effects were observed at temperatures of 518°C, 563°C and three endothermic effects at temperatures of 116°C, 310, 478°C. 2.23 mg of pigment was taken in an open-mouth crucible made of K-1 pigment resistant to temperature of 600°C, starting from 20°C, the temperature was gradually increased (Figure 2.22).

The analysis of the thermogravimetric curve of the organic pigment shows that the TGA curve mainly takes place in the 3 intensive mass loss temperature range. Mass loss range 1 corresponds to temperature 22.5 - 224.3°C, mass loss range 2 corresponds to temperature 224.3 - 320.77°C, and mass loss range 3 corresponds to temperature 320.77 - 600°C. The analysis shows that mass loss 1 is the most intensive decay, with mass loss 0.162 mg, i.e. 7.265% observed in mass loss range 1. The main amount of mass loss in this decay is 1.607 mg, which is 72.063%. In mass loss interval 3, the mass loss is 0.449 mg, which is 20.135%.

It can be seen that the first mass loss is the loss of unbound water. In the second main stage of decomposition, in the case of lower molecular compounds such as carbon dioxide, ammonia, after the decomposition of the organic part, mainly metal carbonates, a mixture of oxides and partially coal remain. In the third stage of decomposition, carbonates are also decomposed, leaving metal oxides and coal residues.

Differential thermogravimetric analysis of organic pigment is presented in Figure 2. Differential thermogravimetric analysis of organic pigment shows that energy absorption occurred in the range of 235.4 - 330.8°C. The highest heat absorption occurs at a temperature of 309.88°C. Energy release occurs between 482.94 - 548.56°C and 555.16 - 581.44°C. The highest heat output occurs at a temperature of 518.6°C.

The synthesized organic pigment was obtained in the presence of terephthalic acid, phthalic anhydride, urea, cobalt salt, calcium salt and catalyst. Thermogravimetric analysis was performed to study the thermal properties of the compound. Simultaneous TG and DTA analyzes were performed in the temperature range of 20-600°C on the SHIMADZU DTG-60 thermal analyzer (Fig. 2.23).

Thermal analysis of K-2 brand organic pigment with new composition was performed in the temperature range of 20-600°C. All samples of the thermal analysis of the mentioned pigments were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar. In addition, endothermic and exothermic points of organic pigment were proved.

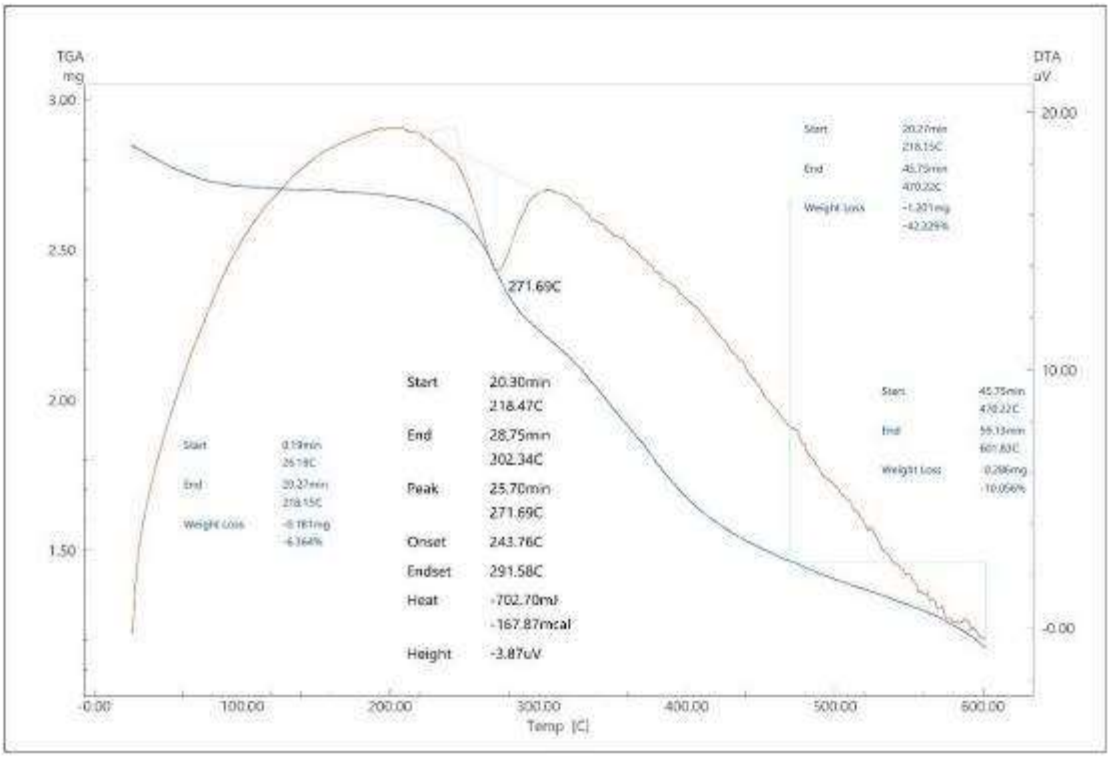


Figure 2.23. Derivatogram of K-2 brand organic pigment

The maximum temperature of 600°C was selected for the organic pigment of K-2 brand, which was synthesized in the dry mass presented in Fig. 2.23, and the analysis results of the pigment were studied according to the presented thermogravimetric derivatogram and differential thermogravimetric analysis. An endothermic effect was observed at a temperature of 271.69°C. K-2 brand organic pigment was taken in an open-mouth crucible made of aluminum resistant to temperature of 600°C, 2.844 mg of pigment was taken, starting from 20°C, the temperature was gradually increased. Figure 2.23.

The analysis of the thermogravimetric curve of the organic pigment shows that the TGA curve mainly takes place in the 3 intensive mass loss temperature range. The 1st mass loss range corresponds to the temperature of 26.19 - 218.15°C, the 2nd mass loss range corresponds to the temperature of 218.15 - 470.22°C, and the 3rd mass loss range corresponds to the temperature of 470.22 - 600°C.

Table 2.5.

Analysis of TGA and DTA curve results of new organic pigments exposed to high temperature

№	T,°C	Lost mass, mg (4.373 mg)	Mass lost,%	Amount of energy consumed (μV*s/mg)	Time spent (min)	Residual mass, dw (mg)	dw/dt (mg/min)
4,373 mg of M-1 brand organic pigment was obtained in total mass							
1	100	0.089	2.035	12.5	8.52	4.284	0,01
2	200	0.136	3.1	16.524	18.72	4.237	0,007
3	300	1.7	38.9	2.438	29	2.67	0,06
4	400	3.3	75.5	12.434	38.95	1.07	0,08
5	500	3.55	81.23	16.61	49.05	0.821	0,07
6	600	4.11	94.03	0.604	59.37	0.26	0,07
3.8 mg of M-2 brand organic pigment was obtained in total mass							
1	100	0.064	1.69	13.777	8.45	3.74	0.0076
2	200	0.086	2.27	17.189	18.58	3.72	0.0046
3	300	1.636	43.03	2.433	28.8	2.167	0.057
4	400	2.79	73.4	11.8	38.8	1.01	0.072
5	500	2.9	76.2	7.27	49.1	0.906	0.06
6	600	3.32	87.2	4.627	59.2	0.488	0.056
A total of 2.23 mg of K-1 brand organic pigment was obtained							
1	100	0,06	2,69	13.18	8.88	2.17	0,006
2	200	0,15	6,72	16.92	19.5	2.08	0.007
3	300	1,08	48,4	7.94	29.25	1.15	0,036
4	400	1,82	81,6	13.04	39.3	0.41	0,046
5	500	1,91	85,6	13.19	49.4	0.32	0,038
6	600	2,22	99,5	5.01	59.6	0.012	0,037
2,844 mg of K-2 brand organic pigment was obtained in total mass							
1	100	0,134	4,7	15.02	8,32	2.71	0,02
2	200	0,168	5,9	19.37	18,45	2.676	0,009
3	300	0,615	21,6	16.8	28,5	2.229	0,02
4	400	1,172	41,2	12.7	38,6	1.672	0,03
5	500	1,443	50,7	5.49	48,8	1.401	0,03
6	600	1,665	58,5	0.44	58,9	1.179	0,028

The analysis shows that in mass loss range 1, mass loss of 0.181 mg, i.e. 6.364%, is observed, while mass loss 2 is the most intense decay. The main amount of mass loss in this decay is 1.201 mg, i.e. 42.229%. The mass loss in mass loss interval 3 is 0.286 mg, which is 10.056%.

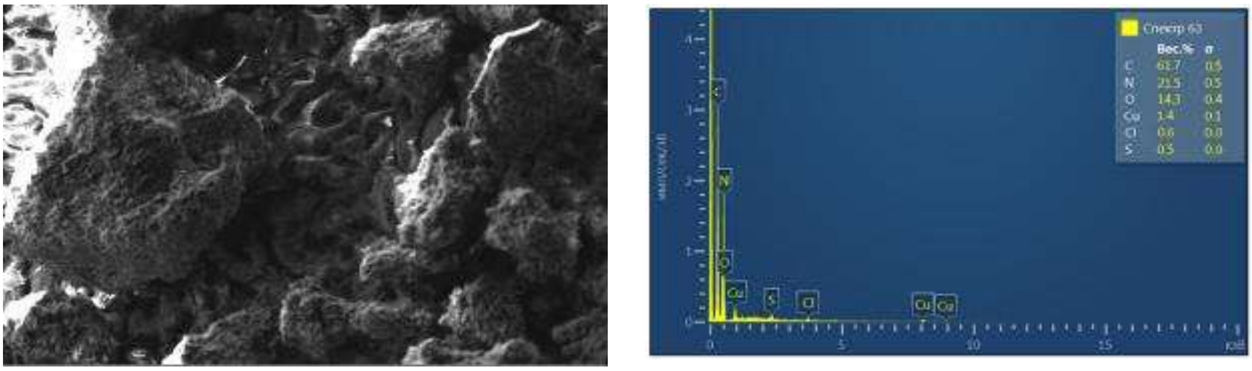
It can be seen that the first mass loss is the loss of unbound water. In the second main stage of decomposition, in the case of lower molecular compounds such as carbon dioxide, ammonia, after the decomposition of the organic part, mainly metal carbonates, a mixture of oxides and partially coal remain. In the third stage of decomposition, carbonates are also decomposed, leaving metal oxides and coal residues.

Differential thermogravimetric analysis of an organic pigment is given in Figure 2.24. Differential thermogravimetric analysis of the organic pigment shows that energy absorption occurred in the range of 218.47 - 302.34°C. The highest heat absorption occurs at a temperature of 271.69°C.

The analysis of the results of the thermal decomposition of the substance at different temperatures is given in Table 2.5.

2.4.4-Scanning electron microscore (SEM) and elemental analysis of the synthesized new organic pigments

Scanning Electron Microscopic (SEM) and Elemental Analysis of M-1 Organic Pigment. The newly synthesized M-1 brand organic pigment was studied with a MIRA 2 LMU scanning electron microscope equipped with an INCA Energy 350 energy dispersive microanalysis system. The resolution of the microscope is 1 nm, and the sensitivity of the INCA Energy detector is 133 eV/10 mm2, allowing the analysis of elements from beryllium to plutonium. Scanning electron microscope analyzes were performed under high vacuum. Microanalysis of organic pigment chemical elements was carried out in the same device, studied in fields with an accelerating voltage of 20 keV and a current of 1 nA.



a)

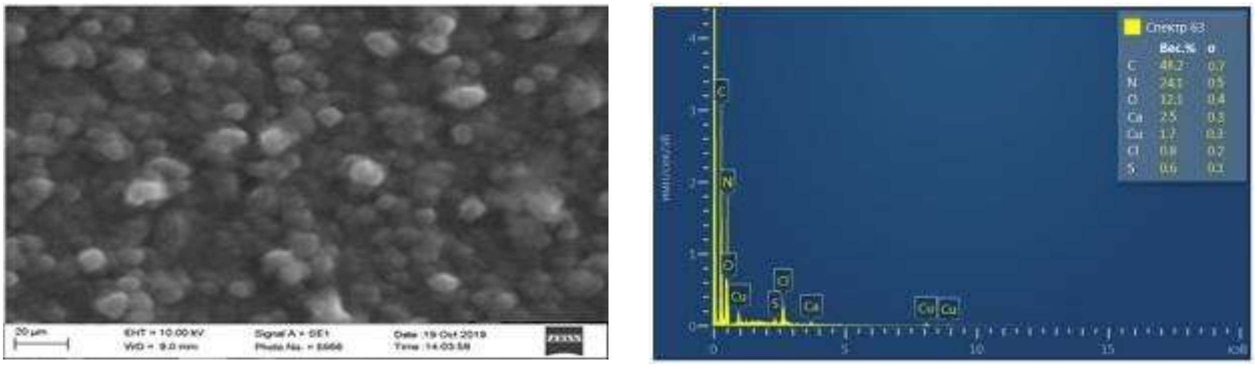
b)

Figure 2.25. Scanning electron microscopy (a) and elemental analysis (b) data of M-1 organic pigment

When 200 times magnified SEM images of M-1 brand organic pigment sample with new composition are studied, the remains of unreacted starting substances are not visible. This makes it possible to obtain information about the completion of the reaction and, in parallel, about the composition of the substances formed in the reaction. Studies have shown that organic pigment particles range in size from 30.55 to ~35.00 nm. At the same time, elemental analysis was carried out on a separate surface in large clusters. Large clusters indicate the presence of sulfur and chlorine residues within the experimental error level, except for the M-1 brand organic pigment in the studied points when the element is analyzed (Fig. 2.25) [107].

Scanning Electron Microscopic (SEM) and Elemental Analysis of M-2 Brand Organic Pigment.

The newly synthesized M-2 brand organic pigment was studied with a MIRA 2 LMU scanning electron microscope equipped with an INCA Energy 350 energy dispersive microanalysis system. The resolution of the microscope is 1 nm, and the sensitivity of the INCA Energy detector is 133 eV/10 mm2, allowing the analysis of elements from beryllium to plutonium. Scanning electron microscope analyzes were performed under high vacuum. Microanalysis of the chemical elements of the composite pigment was carried out in this device and was studied in fields with an accelerating voltage of 20 keV and a current of 1 nA.

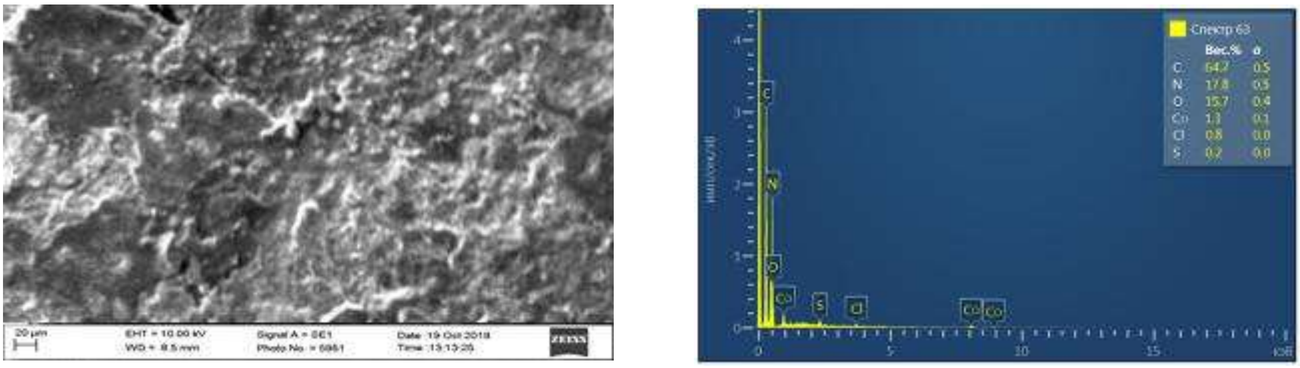


a) b)

Figure 2.26. Scanning electron microscope (a) and elemental analysis (b) data of M-2 organic pigment

When 200 times magnified SEM images of M-2 brand organic pigment sample with new composition are studied, the remains of unreacted initial substances are not visible. This makes it possible to obtain information about the completion of the reaction and, in parallel, about the composition of the substances formed in the reaction. Studies have shown that organic pigment particles range in size from 28.40 to ~36.85 nm. At the same time, elemental analysis was carried out on a separate surface in large clusters. Large clusters indicate the presence of chlorine and sulfur residues within the experimental error level, except for the M-2 brand organic pigment at the studied points when the element was analyzed (Fig. 2.26).

Scanning Electron Microscopic (SEM) and Elemental Analysis of K-1 Brand Organic Pigment. The scanning electron microscope (SEM) image and elemental analysis of the synthesized K-1 organic pigment are presented below [90].

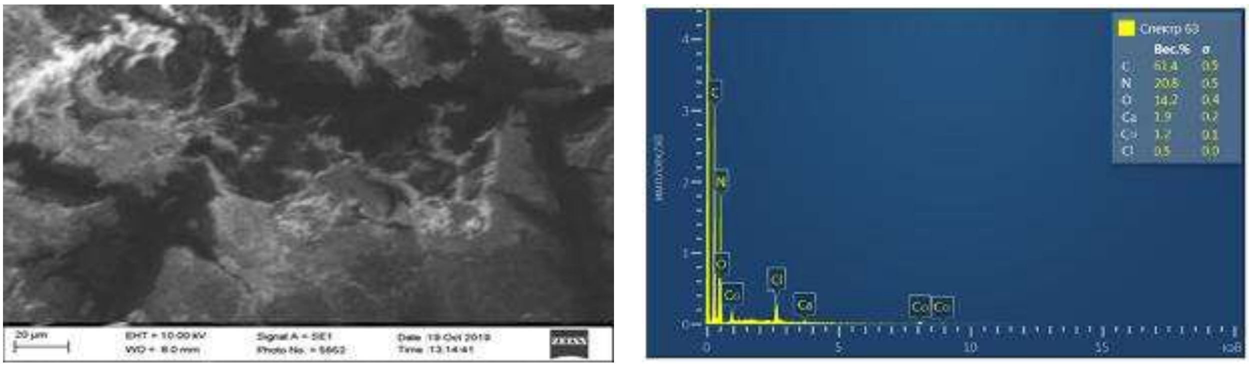


a) b)

Figure 2.27. Scanning electron microscope (a) and elemental analysis (b) data of synthesized K-1 organic pigment

When examining the 200x magnification SEM images of the K-1 brand organic pigment sample with the new composition, no traces of unreacted starting materials are visible. This makes it possible to obtain information about the completion of the reaction and, in parallel, about the composition of the substances formed in the reaction. From the obtained images, it can be seen that the organic pigment molecules are uniformly distributed. At the same time, elemental analysis was carried out on a separate surface in large clusters. Large clusters indicate the presence of sulfur and chlorine residues within the experimental error level, except for the K-1 brand organic pigment at the studied points when the element is analyzed [108] (Fig. 2.27).

Scanning Electron Microscopic (SEM) and Elemental Analysis of K-2 Brand Organic Pigment. The scanning electron microscope (SEM) image and elemental analysis of the synthesized K-2 organic pigment are presented below [109]. When examining the 200x magnification SEM images of the K-2 brand organic pigment sample with the new composition, no traces of unreacted starting materials are visible. This makes it possible to obtain information about the completion of the reaction and, in parallel, about the composition of the substances formed in the reaction. From the obtained images, it can be seen that the organic pigment molecules are uniformly distributed. At the same time, elemental analysis was carried out on a separate surface in large clusters. Large clusters indicate the presence of chlorine residues within the experimental error, except for K-2 brand organic pigment in the studied points when the element is analyzed (Fig. 2.28).



a) b)

Figure 2.28. Scanning electron microscope (a) and elemental analysis (b) data of synthesized K-2 organic pigment

Conclusion on Chapter II

In this chapter, research methods and tools, synthesis of M-1, M-2, K-1 and K-2 pigments and the effects of various factors on synthesis conditions and their analysis are presented. The optimum temperature for the synthesis of M-1 and K-1 pigments was 195°C, and for the synthesis of M-2 and K-2 pigments it was 185°C. The ratio of starting materials in M-1 brand pigment is 1:1:5:0.5 according to

TA:FA:U:CuCl; TA:FA:U: CuCl:$CaCl_2$ in M-2 brand pigment 1:1:5:0.5:1 respectively; 1:1:5:0.5 in K-1 brand pigment TA:FA:U:$CoCl_2$; In K-2 pigment, it was found that the reaction yield was the highest when TA:FA:U:$CoCl_2$:$CaCl_2$ was 1:1:5:0.5:1.

IR-spectroscopic analysis of newly synthesized organic pigments showed that the relevant vibrations have changed compared to those of the original substances. Also, the result of measurements under the visible light spectrum of 190-1100 nm using a UV-spectrophotometer is given for each pigment. Thermogravimetric and differential thermal analysis of new organic pigments are given, and in the analysis of all four pigments, loss of unbound water generally occurs in the first mass loss. In the second main stage of decomposition, in the case of lower molecular compounds such as carbon dioxide, ammonia, the organic part is decomposed, and after that, a mixture of metal carbonates, oxides, and partially coal remains. In the third stage of decomposition, carbonates are also decomposed, leaving metal oxides and carbon residues. Based on the data obtained from the analysis, the proposed formulas are proved.

CHAPTER III. RESULTS OF RESEARCH ON THE APPLICATION OF SYNTHESIZED ORGANIC PIGMENTS

3.1-§. Preparation of alkyd enamel using synthesized organic pigment

Industrial alkyd enamel was prepared according to the composition of raw materials in Table 3.1.

Table 3.1.

New alkyd enamel composition of blue color PF-115 class based on new organic pigment M-2

№	**Name of raw materials**	**Composition, mass.%**
1	PF-060 brand lok (52%)	69,8
2	R-706 brand pigment - titanium dioxide	0,5
3	BG-4 brand bentogel	0,4
4	Mel (microcalcite)	21,5
5	MIX brand siccative	0,2
6	New organic pigment M-2 brand	5,2
7	Methylethylketoxime	0,3
8	C-4 135/220 brand nephros	2,1

After adding Lok, titanium dioxide, M-2 brand organic pigment and bentogel, it was dispersed in a bead mill at room temperature for 2 hours (dsh = 3-3.5 mm, 1500-2000 min-1) until it became homogeneous, and a pigment paste was prepared, then Enamel is obtained by adding siccative and methylethylketoxime [110].

The new M-2 organic pigment was prepared using a small paint preparation device - a laboratory pearl mill. The requirement for paint products is that the coloring pigment should be well mixed into a homogeneous system. As a result of this, agglomerates of pigment particles are sharply crushed and spread agglomerates or a thin suspension is obtained. In the preparation of paint, pigment and paint raw materials were mixed in a special iron container at a high speed with the help of a dispersion knife, and the solids were transferred to the same suspension medium. M-2 organic pigment was coated on a metal plate with complete mixing in this device for 2 hours [111] (Fig. 3.1).

Figure 3.1. Alkyd enamel coated on a metal plate (based on new pigments).

§ 3.2. Thermal analysis of lacquer products based on new organic pigments

3.2.1. Thermal analysis of alkyd enamel made on the basis of M-1 organic pigment

All samples of the thermal analysis of the given alkyd enamel paint were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar.

4.9 mg of alkyd enamel paint prepared on the basis of M-1 organic pigment was taken, and the process was carried out at a temperature of 20-600°C. Alkyd enamel paint was studied by thermogravimetric analysis (TG) and differential thermal analysis (DTA). Three endothermic effects were observed at temperatures of 290°C, 382°C, 427°C and one exothermic effect at 538°C Figure 3.2.

Analysis of the thermogravimetric curve of the obtained alkyd enamel paint shows that the TG curve mainly takes place in the 3 intensive mass loss temperature range. The mass loss range 1 corresponds to the temperature of 26.83 - 229.69°C, the mass loss range 2 corresponds to the temperature of 229.69 - 497.48°C, and the mass loss range 3 corresponds to the temperature of 497.48 - 600°C.

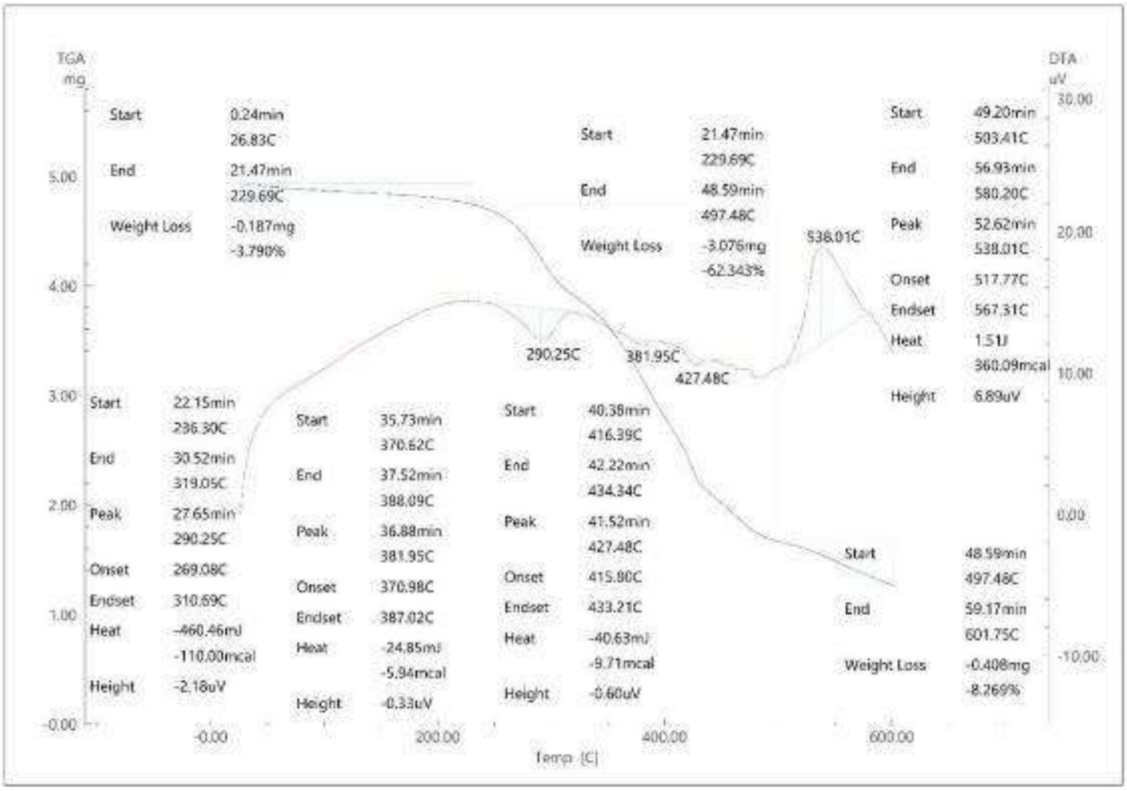


Figure 3.2. Derivatogram of alkyd enamel made on the basis of M-1 organic pigment

The results of the analysis show that 3.79% mass loss is observed in mass loss interval 1, while 62.343% of mass loss 2 occurs. The mass loss in the 3rd mass loss interval is 8.269%.

Differential thermal analysis of alkyd enamel paint shows that energy absorption occurs in the range of 236.3 - 319.05°C, 370.62 - 388.09°C and 416.39 - 434.34°C. At the same time, energy is released in the range of 503.41 - 580.2°C [112].

3.2.2. Thermal analysis of alkyd enamel made on the basis of M-2 organic pigment

10.07 mg of alkyd enamel paint prepared on the basis of M-2 organic pigment was taken, and the process was carried out at a temperature of 20-600°C. Alkyd enamel paint was studied by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). Three endothermic effects were observed at temperatures of 303, 425, 489°C (Fig. 3.3).

Analysis of the thermogravimetric curve of the obtained enamel shows that the TGA curve mainly takes place in the temperature range of 3 intensive mass losses. The 1st mass loss range corresponds to the temperature of 32.06 - 236.92°C, the 2nd mass loss range corresponds to the temperature of 236.92 - 500.24°C, and the 3rd mass loss range corresponds to the temperature of 500.24 - 600°C.

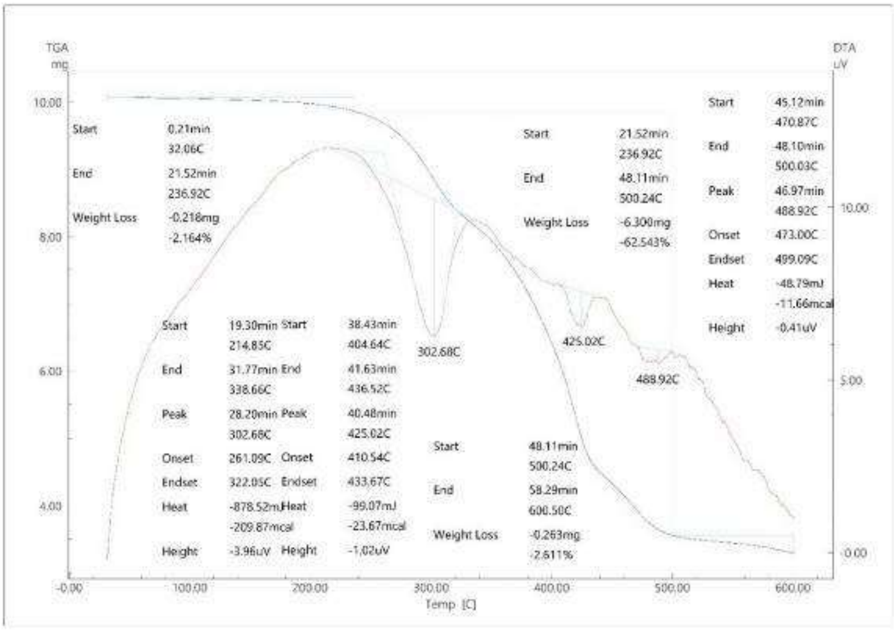


Figure 3.3. Derivatogram of alkyd enamel made on the basis of M-2 organic pigment

The analysis shows that mass loss 1 is the most intensive decay, with mass loss 0.218 mg, i.e. 2.164% observed in mass loss range 1. The main amount of mass loss in this decay is 6.3 mg, i.e. 62.5%. In mass loss interval 3, the mass loss is 0.263 mg, which is 2.6%.

It is known from the differential thermal analysis of alkyd enamel paint that energy absorption occurs in the range of 214.85 - 338.66°C, 404.64 - 436.52°C and 470.87 - 500.03°C.

3.2.3. Thermal analysis of alkyd enamel made on the basis of K-1 organic pigment

All samples of the thermal analysis of the given alkyd enamel paint were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar.

6,593 mg of alkyd enamel paint prepared on the basis of K-1 organic pigment was obtained, and the process was carried out at a temperature of 20-600°C. Alkyd enamel paint was studied by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). Endothermic effects were observed at a temperature of 449°C (Fig. 3.4).

Analysis of the thermogravimetric curve of the obtained alkyd enamel paint shows that the TGA curve mainly takes place in the temperature range of 3 intensive mass losses. The 1st mass loss range corresponds to the temperature of 27.24 - 157.02°C, the 2nd mass loss range corresponds to the temperature of 157.02 - 476.28°C, and the 3rd mass loss range corresponds to the temperature of 476.28 - 600°C [113].

The results of the analysis show that 3.125% mass loss is observed in mass loss interval 1, while 86.9% of mass loss 2 takes place. In the 3rd mass loss interval, the mass loss is 11.3%.

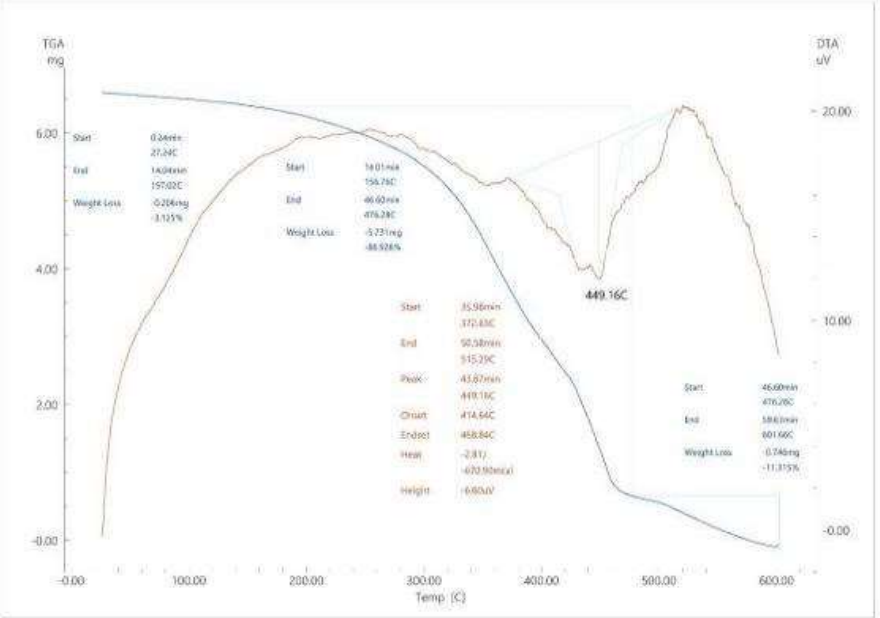


Figure 3.4. Derivatogram of alkyd enamel made on the basis of K-1 organic pigment

Differential thermal analysis of alkyd enamel paint shows that energy absorption occurs in the range of 372.6 - 515.29°C.

3.2.4. Thermal analysis of alkyd enamel made on the basis of K-2 organic pigment

All samples of the thermal analysis of the given alkyd enamel paint were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar.

8,096 mg of alkyd enamel paint prepared on the basis of K-2 organic pigment was obtained, and the process was carried out at a temperature of 20-600°C. Alkyd enamel paint was studied by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). Two endothermic effects were observed at temperatures of 322 and 432°C Fig. 3.5.

Analysis of the thermogravimetric curve of the obtained alkyd enamel paint shows that the TGA curve mainly takes place in the temperature range of 3 intensive mass losses. The 1st mass loss interval corresponds to the temperature of 28.19 - 222.28°C, the 2nd mass loss interval corresponds to the temperature of 222.28 - 453.28°C, and the 3rd mass loss interval corresponds to the temperature of 453.28 - 600°C [114].

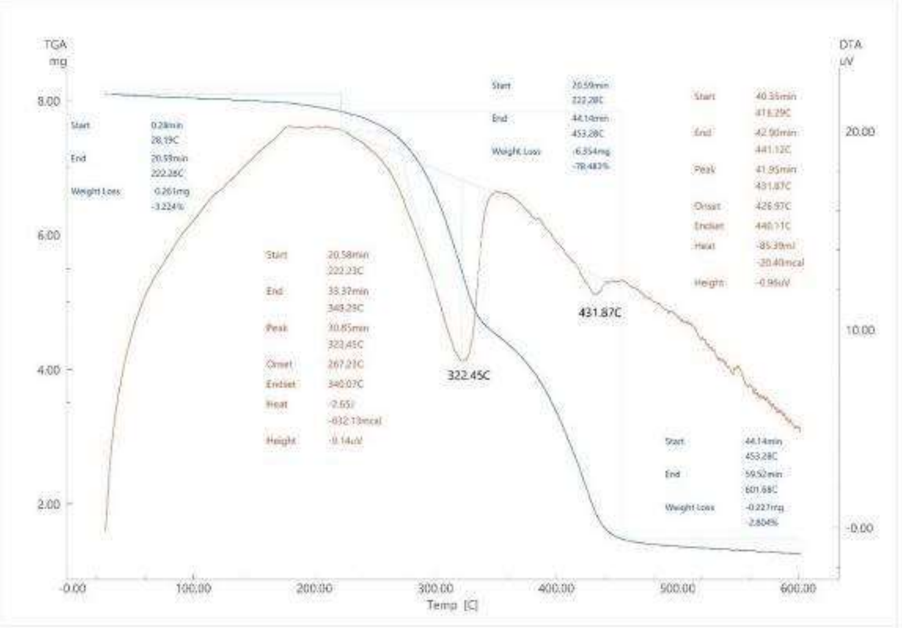


Figure 3.5. Derivatogram of alkyd enamel made on the basis of K-2 organic pigment

The results of the analysis show that 3.224% mass loss is observed in mass loss interval 1, while 78.483% of mass loss 2 takes place. The mass loss in the 3rd mass loss interval is 2.804%.

Differential thermal analysis of alkyd enamel paint shows that energy absorption occurs in the range of 222.23 - 348.29°C and 416.29 - 441.12°C.

3.2.5. Derivatogram of alkyd enamel with addition of CuPc pigment for control

Figure 3.6 shows the derivatogram of the alkyd enamel prepared by adding the CuPc pigment taken for control. According to it, 2 intensive decomposition temperatures are known in the dynamic thermogravimetric analysis of alkyd enamel. These divisions correspond to the intervals of between 60-215°C and 215-380°C.

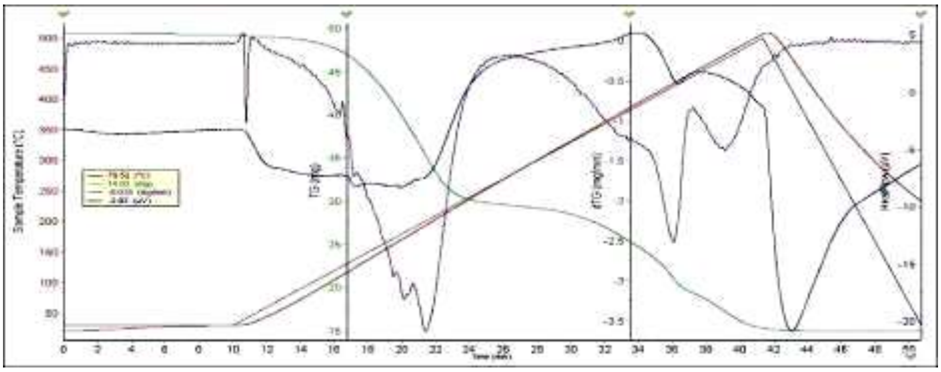


Figure 3.6 shows the derivatogram of the alkyd enamel prepared by adding the CuPc pigment taken for control.

According to it, 2 intensive decomposition temperatures are known in the dynamic thermogravimetric analysis of alkyd enamel. These divisions correspond to the intervals of 60-215°C and 215-380°C.

According to the researches, it was found that 11.76 mg of copper phthalocyanine-added enamel remains from 50 mg of initial enamel at 500°C temperature, which is 76.48%.

Table 3.2.

Table of comparison of thermal analyzes of lacquer products based on new organic pigments

№	Temperature °C	Residual mass, mg	Lost mass, mg	Lost mass,%	Amount of energy consumed (μV*s/mg)
General of the paint made of M-1 organic pigment массада 4,934 мг олинган					

1	100	4,8	0,068	1,37	8,23
2	200	4,8	0,138	2,8	18,47
3	300	4,1	0,83	16,8	28,6
4	400	2,79	2,14	43,4	38,7
5	500	1,66	3,27	66,4	48,8
6	600	1,26	3,67	74,4	59,1

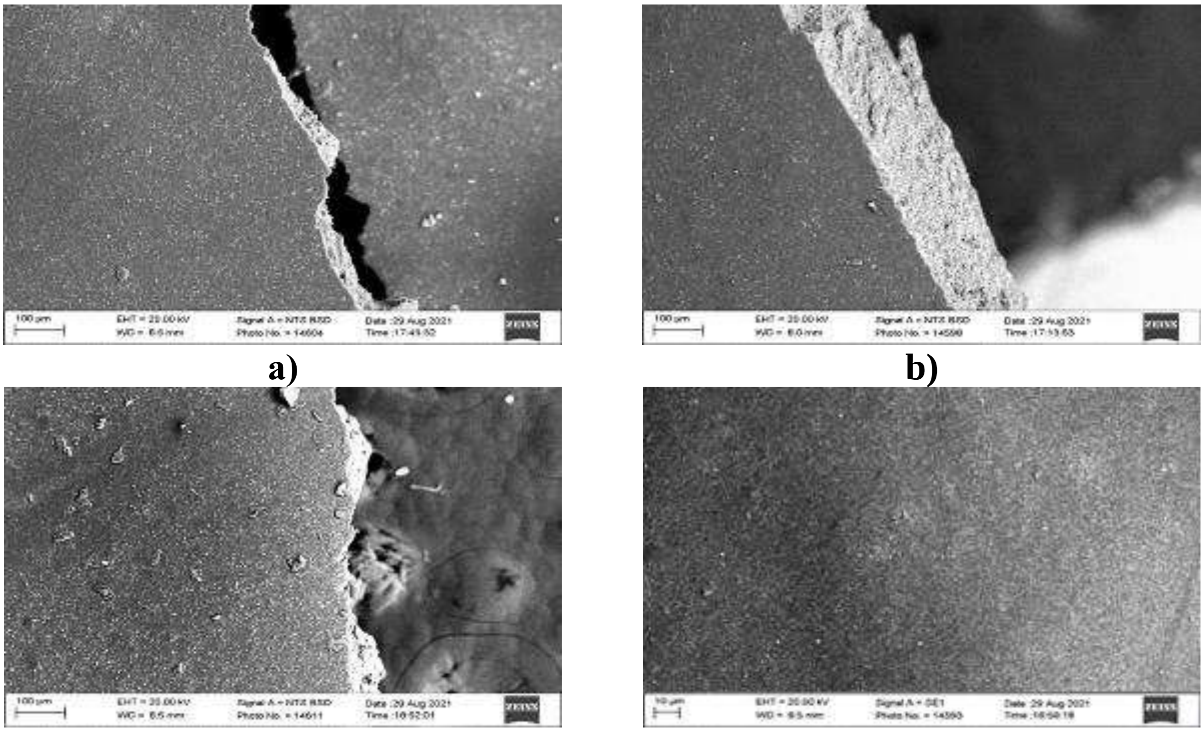
Table 3.2 continues

10.07 mg of the total mass of the paint made from M-2 organic pigment was obtained					
1	100	10,04	0,03	0,3	7,7
2	200	9,9	0,1	1,02	17,8
3	300	8,9	0,16	11,55	27,9
4	400	6,4	3,6	36.36	38.1
5	500	3,55	6,5	64,7	48,1
6	600	3,29	6,78	67,3	58,2
6,593 mg of the total mass of paint made from K-1 organic pigment was obtained					
1	100	6.49	0,11	1,67	13.9
2	200	6.2	0,4	6,07	18.7
3	300	5.5	1,1	16,69	18.17
4	400	2.9	3,7	56,12	14.8
5	500	0.5	6,1	92,5	17.5
6	600	0.1	6,5	98,6	14.57
8,096 mg of the total mass of paint made from K-2 organic pigment was obtained					
1	100	8,03	0,07	0,87	15,48
2	200	7,9	0,2	2,47	20,19
3	300	6,59	1,51	18,66	11,9
4	400	3,35	4,75	58,67	14,38
5	500	1,36	6,74	83,25	10,59
6	600	1,25	6,85	84,61	5,04
A total mass of 50 mg of paint made of control CuPc pigment was obtained					
1	50	48,903	1,097	2,194	1,45
2	100	31,309	18,691	37,382	2,91
3	200	30,209	19,791	39,58	4,09
4	300	22,406	27,594	55,19	5,08
5	400	21,896	28,104	56,208	6,93
6	500	16,66	33,34	66.68	8,07

The general thermal analysis of alkyd enamel at high temperature, prepared by adding M-1, M-2, K-1 and K-2 organic pigments based on terephthalic acid and CuPc pigments taken for control, is given in table 3.2 below. According to the obtained results, it was found that the obtained organic pigments have higher thermal stability than the imported product.

§ 3.3. SEM and elemental analysis of paints based on new organic pigments

The newly synthesized paints based on M-1, M-2, K-1 and K-2 brand organic pigments were studied in MIRA 2 LMU scanning electron microscope equipped with INCA Energy 350 energy dispersive microanalysis system (Figs. 3.7, 3.8). The resolution of the microscope is 1 nm, and the sensitivity of the INCA Energy detector is 133 eV/10 mm2, allowing the analysis of elements from beryllium to plutonium. Scanning electron microscope analyzes were performed under high vacuum. The elemental analysis of pigments was carried out in this device and studied in fields with an accelerating voltage of 20 keV and a current of 1 nA. In this work, electron scan images were obtained at 30 keV acceleration, at magnifications of 500 and 200 times, and at 0.66 and 1.653 µm of the visible region. 200 times magnified images of paints based on synthesized M-1, M-2, K-1 and K-2 organic pigments are presented.

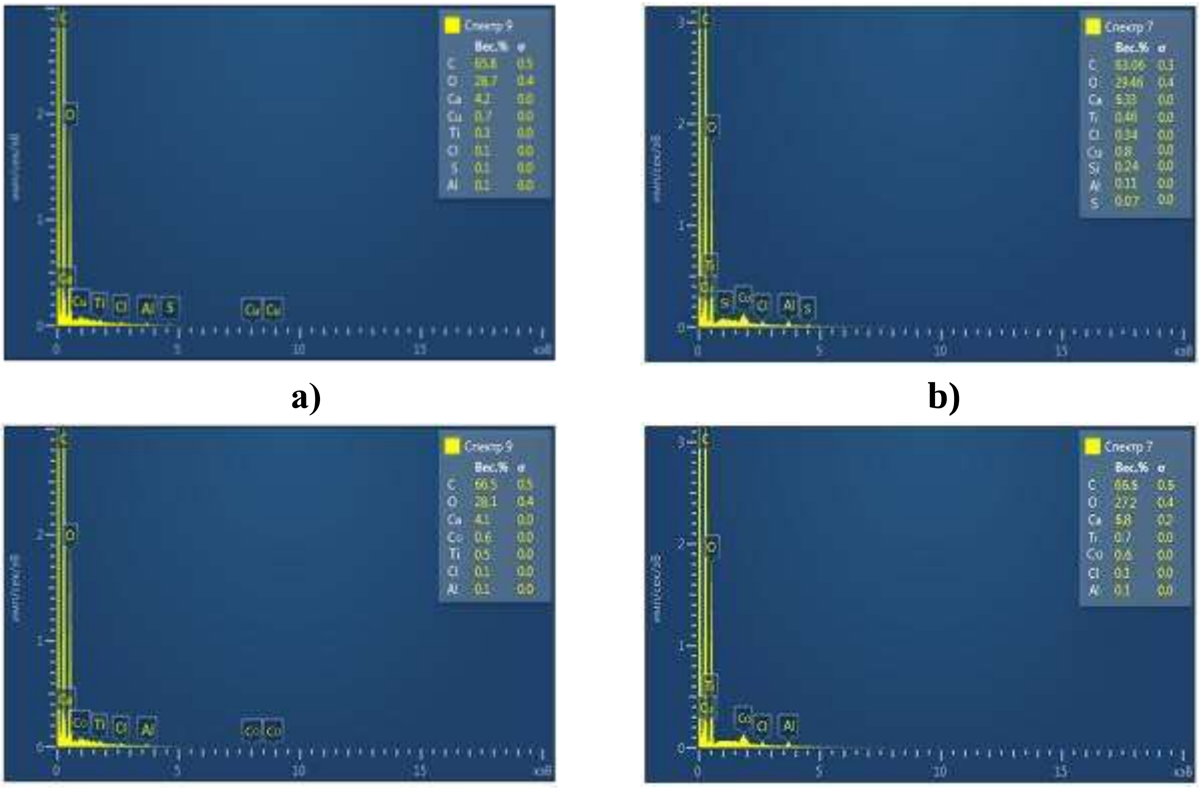


a) **b)**

c) **d)**

Figure 3.7. SEM images of paints based on new organic pigments. a) M-1, b) M-2, c) K-1, d) K-2

From the obtained images, it can be seen that the pigment molecules are uniformly distributed on the surface of the paint paste samples. Elemental analysis of large clusters was also carried out on a separate surface using a scanning electron microscope. When large clusters of elements were analyzed, no excess element was found except for pigment in the studied points.



a)

b)

c)

d)

Figure 3.7. Results of elemental analysis of paints based on new organic pigments a) M-1, b) M-2, c) K-1, d) K-2

§ 3.4. Checking the compliance of loco-paint products based on new organic pigments with state standards.

Table 3.3.

Comparison of physical and mechanical properties of PF-115 enamel prepared on the basis of new organic pigments according to GOST 6465-76

№	**The name of the indicators**		**Indicator standard**	**Result**
1	The color of the enamel coating		Number according to "Kartoteka".	Fits
	a)	The blue 423	423	
	б)	Blue	427, 428	
2	The exterior of the coating Appearance		After drying, the enamel forms a smooth, homogeneous coating without foreign particles	Fits
	a)	The blue 423		
	б)	Blue		
3	The glossiness of the coating according to the photoelectric blescomer device is not less than %		Standard according to GOST 896-69	
	a)	The blue 423	50	51
	б)	Blue	50	50
4	Conditional viscosity at a temperature of 20±0.5°C (measured on a viscometer VZ-246)		Standard according to GOST 8420-	
	a)	The blue 423	80-120	105
	б)	Blue	80-120	110
5	Mass fraction of non-volatile substances in the composition,%		Standard according to GOST 17537-72	
	a)	The blue 423	60-66	61
	б)	Blue	60-66	62
6	Degree of liquefaction for 28-30 s at a temperature of 20±0.5°C (measured on a viscometer VZ-246), not more than %		Standard according to GOST 17537-72	
	a)	The blue 423	20	20
	б)	Blue	20	20
7	Grinding degree, µm, not much		Standard according to GOST 6589-74	
	a)	The blue 423	25	25
	б)	Blue	25	25
8	Density of dried coating, not		Standard according to	

	more than g/m2	GOST 8784-75	

Table 3.3 continues

	a)	The blue 423	60	60
	б)	Blue	40	40
9		Construction time at a temperature of 20±2°C is no more than an hour	Standard according to GOST 19007-73	
	a)	The blue 423	24	24
	б)	Blue	24	24
10		The elasticity of the coating in bending is not more than mm	Standard according to GOST 6806-73	
	a)	The blue 423	1	1
	б)	Blue	1	1
11		The impact strength of the coating (measured on the U-1 instrument) is not less than cm	Standard according to GOST 4765-73	
	a)	The blue 423	40	40
	б)	Blue	40	40
12		The hardness of the coating (measured with a pendulum M-3 tool) is not less than ShB (conditional symbol)	Standard according to GOST 5233-89	
	a)	The blue 423	0,25	0,25
	б)	Blue	0,25	0,25
13		Қоплама адгезияси, балл дан кўп эмас	Standard according to GOST 51140-98	
	a)	The blue 423	1	1
	б)	Blue	1	1
14		At a temperature of 20±2°C, the resistance of the coating to the static effect of water is not less than an hour	Standard according to GOST 9,403-80	
	a)	The blue 423	2	2
	б)	Blue	2	2
15		Resistance to the static effect of a 0.5% solution of coating detergents, not less than min	Standard according to GOST 9,403-80	
	a)	The blue 423	15	15
	б)	Blue	15	15
16		The resistance of the coating to the static effect of transformer oil at a temperature of 20±2°C is not less than s	Standard according to GOST 9,403-80	

	a)	The blue 423	24	24
	б)	Blue	24	24

PF-115 enamel samples prepared on the basis of new organic pigments of M-1, M-2, K-1 and K-2 brands synthesized by GOST 6465-76 have been confirmed [100].

§ 3.5. Study of the effect of new organic pigments added to 0320 polyethylene on polyethylene properties

In order to determine other physico-mechanical properties of the synthesized new organic pigment, it was added to 0320 polyethylene and its effect on the physico-mechanical properties was studied. For this, polyethylene was added in the amount of 3%. As a result, blue-colored, intensively dyed and evenly distributed polyethylene was obtained. The mechanical and physicochemical properties of the obtained colored polyethylene were determined in a universal testing machine. The obtained results are presented in Table 3.4.

Table 3.4.

Physical and mechanical properties of polyethylene with 3% organic pigment M-1

The name	**Modulus of resilience, MPa**	**Max. flexibility,%**	**Separation power, N**	**Separation stress, MPa**	**Separation resistance,%**
1. PE+3% M-1	158.5	593.5	186.02	14.8	593.4
2. PE +3% M-1	154.08	611.5	188.8	15.1	611.4
3. PE +3% M-1	153.5	593.7	186.72	14.48	593.54
Average	155.74	602,5	187,4	14,9	602,64
Standard deviation	3.2	31.4	15.3	1.2	931.4
Maximum	158.5	611.5	188.8	15.1	611.4
Minimum	154.08	593.5	186.02	14.8	593.4

Table 3.4 shows the values of the tensile strength of painted polyethylene based on 3 samples taken, and the average value of the results compared to GOST requirements.

The tensile modulus of the polyethylene sample with 3% M-1 organic pigment was 155.4 MPa in the range of 0% to 3% deformation of the sample. The force used to break the polyethylene sample with organic pigment is 187.4 N. The tensile strength of the sample is 14.9 MPa. The amount of deformation of the sample before failure shows 602.64%, that is, the area of the sample involved in the test was stretched by 6.025 times (Fig. 3.12).

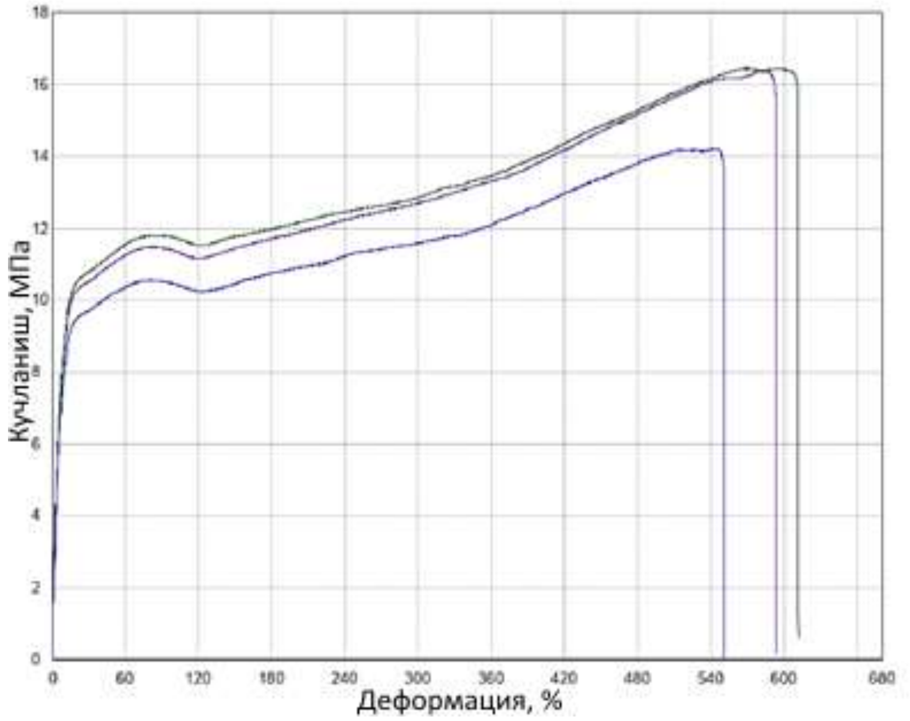


Figure 3.12. Tensile strength diagram of M-1 organic pigmented polyethylene

The tensile modulus of the polyethylene sample with PE+3% M-2 organic pigment was 172.8 MPa in the range of 0% to 3% deformation. The force used to break the polyethylene sample with organic pigment is 193.3 N. The tensile strength of the sample is 15.5 MPa. The amount of deformation of the sample before failure shows 659.6%, that is, the area of the sample involved in the test was stretched by 6.597 times (Fig. 3.13).

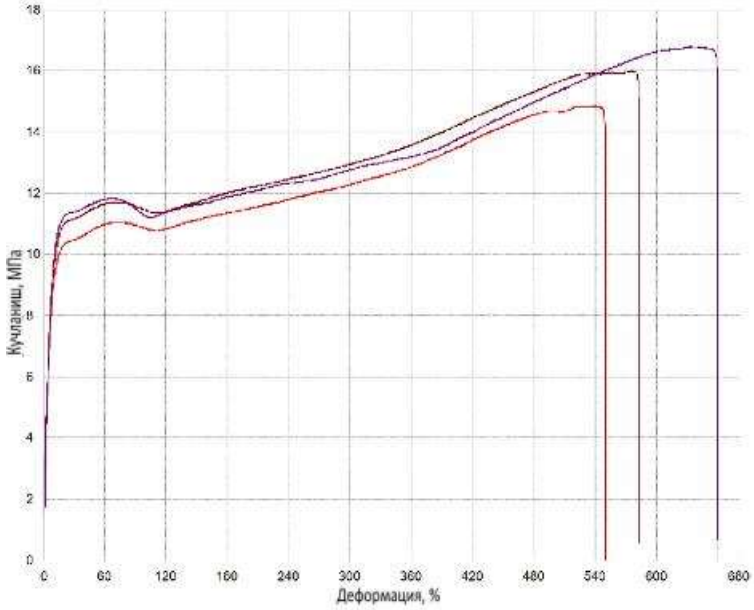


Figure 3.13. Tensile strength diagram of M-2 organic pigmented polyethylene

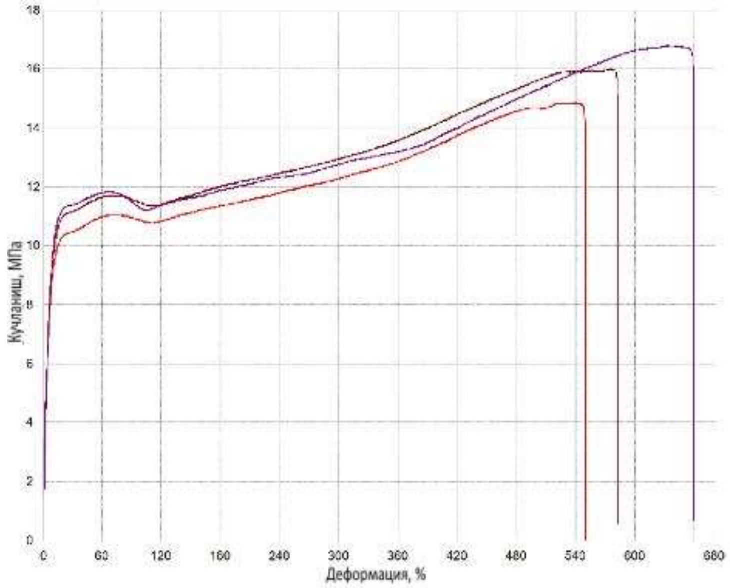


Figure 3.13. Tensile strength diagram of M-2 organic pigmented polyethylene

In this picture, the results of the deformation-strength indicators of the polyethylene sample with organic pigment added in accordance with the requirements of GOST are presented by taking 3 samples.

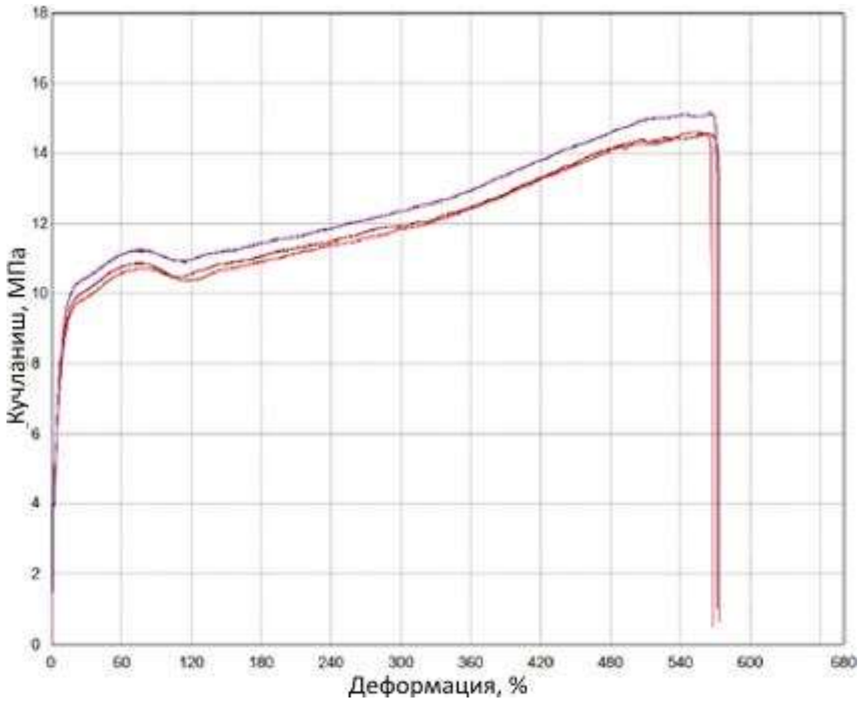


Figure 3.14. Tensile strength diagram of K-1 grade organic pigmented polyethylene

The tensile elastic modulus of the polyethylene sample with PE+3% K-1 organic pigment was 145.4 MPa in the range of 0% to 3% deformation of the sample. The force used to break the polyethylene sample with organic pigment is 171.4 N. The tensile strength of the sample is 13.7 MPa. The amount of deformation of the sample before failure shows 571.5%, that is, the area of the sample involved in the test was stretched by 5.716 times (Fig. 3.14).

The tensile modulus of polyethylene with organic pigment addition of PE+3% K-2 sample was 158.7 MPa in the range of 0% to 3% deformation of the sample. The force used to break the polyethylene sample with organic pigment is 192.01 N. The tensile strength of the sample is 15.4 MPa. The amount of deformation of the sample before failure shows 570.7%, that is, the area of the sample involved in the test was stretched by 5.708 times (Fig. 3.15).

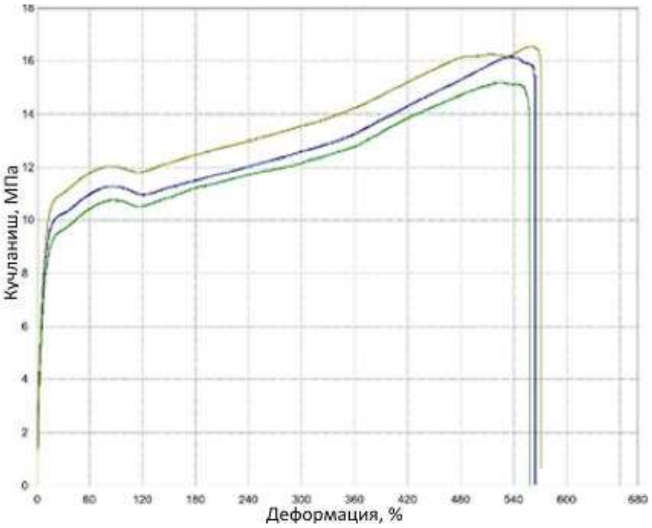


Figure 3.15. Tensile strength diagram of K-2 grade organic pigmented polyethylene

The tensile modulus of the 0320 PE sample was 134.9 MPa in the range of 0% to 3% strain. The force used to break the painted polyethylene sample is 170.6 N. The tensile strength of the sample is 13.6 MPa. The amount of deformation of the sample before breaking is 568.9%, that is, the area of the sample involved in the test is stretched by 5,691 times (Fig. 3.16).

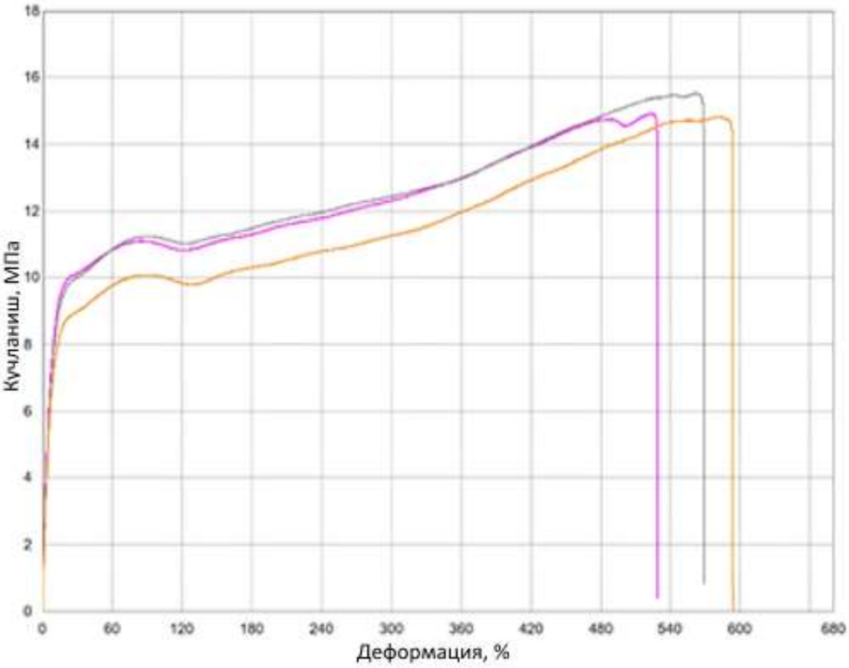


Figure 3.16. Tensile strength diagram of polyethylene without organic pigment addition

According to the results of the above analysis, it was found that adding new organic pigment M-2 to 0320 polyethylene did not adversely affect the physical and mechanical properties of polyethylene.

It was found that the relative tensile strength of polyethylene samples with organic pigment increases in the following order (Table 3.5).

PE < K-1 < M-1 < K-2 < M-2

Table 3.5.

Physico-mechanical properties of polyethylene with and without new organic pigments

Name	Modulus of elasticity, MPa	Max. Flexural strength, N	Max. Bending stress, Mpa	Max. Elongation, %	Interruption Kuchi, N	Tensile strength, MPa	Separation resistance
1. PE+3% M-1	155.74	4.7	0.4	602,5	187,4	14,9	604,53
2. PE +3% M-2	172,8	7,9	0,6	659,7	193,3	15,5	661,5
3. PE +3% K-1	145,4	12,5	0,9	571,6	168,2	13,5	572,6
4. PE +3% K-2	158.7	8.08	0.6	570.8	192.01	15.4	571.9
0320-ПЭ	134.9	9.9	0.8	569,1	170.6	13.6	567.8

Thermal analysis of M-1 organic pigmented polyethylene. Thermal analysis of polyethylene with 3% M-1 organic pigment was performed in the temperature range of 20-600°C. All samples of thermal analysis of painted polyethylene were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar. In addition, endothermic and exothermic points of obtained polyethylene were shown.

The thermal stability of the samples prepared by adding 3% of the obtained pigments to polyethylene was studied by thermogravimetric method.

The maximum temperature of 600°C was selected for the polyethylene with 3% M-1 organic pigment shown in Figure 3.17, and the analysis results of the polyethylene were studied according to the given thermogravimetric derivatogram (TGA) and differential thermogravimetric analysis. Two endothermic effects were observed at temperatures of 123.82°C and 483.46°C. Painted polyethylene was taken in an amount of 5.75 mg in a 600°C heat-resistant aluminum crucible, and the temperature was gradually increased starting at 20°C.

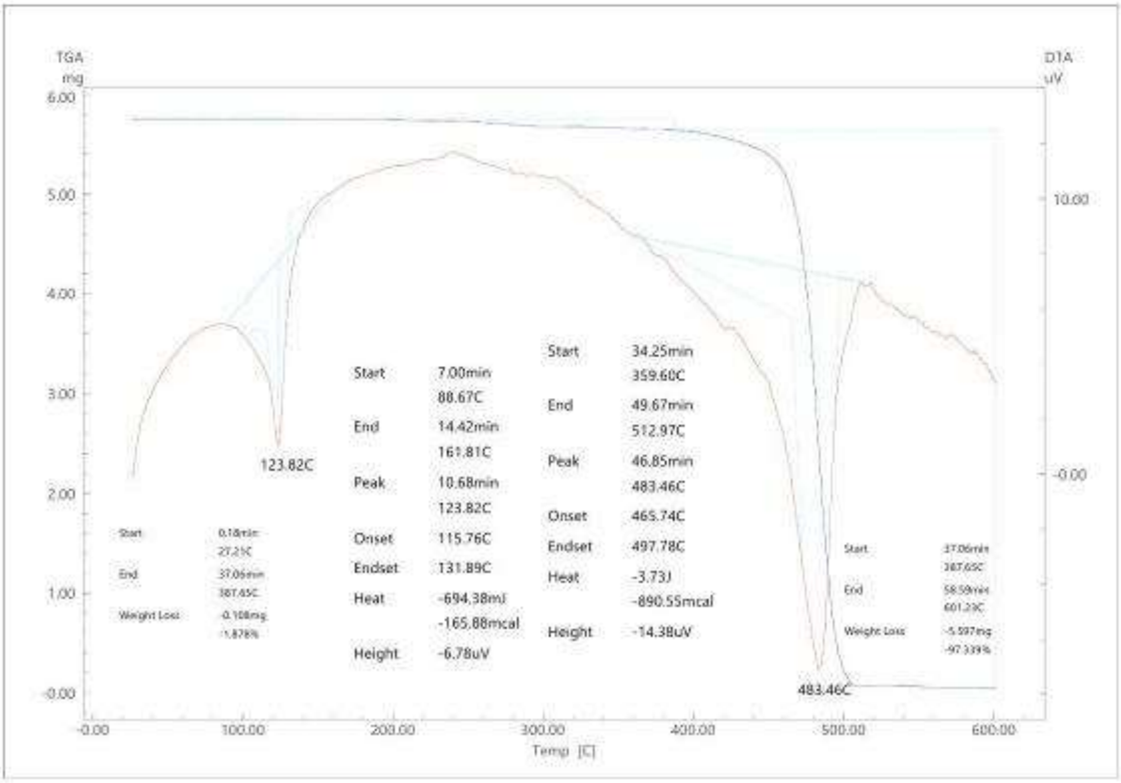


Figure 3.17. Derivatogram of polyethylene with 3% organic pigment M-1.

Analysis of the thermogravimetric curve of polyethylene with 3% M-1 grade organic pigment shows that the TGA curve mainly takes place in the temperature range of 2 intensive mass losses. The 1st mass loss interval corresponds to the temperature of 27.21-387.65°C, the 2nd mass loss interval corresponds to the temperature of 387.65 - 600°C. The analysis shows that in mass loss interval 1, mass loss of 0.108 mg, i.e. 1.878%, is observed, while mass loss 2 is the most intensive decay. The main amount of mass loss in this decay is 5.597 mg, which is 97.339%.

Differential thermogravimetric analysis of polyethylene with 3% M-1 organic pigment is presented in Figure 3.17. Differential thermogravimetric analysis of organic pigment shows that energy absorption occurred in the range of 88.67 - 161.8°C and 359.6 - 512.9°C.

Thermal analysis of polyethylene with 3% M-2 organic pigment was performed in the temperature range of 20-600°C. All samples of the thermal analysis of the given polyethylene were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar. In addition, the endothermic and exothermic points of the composition were proved.

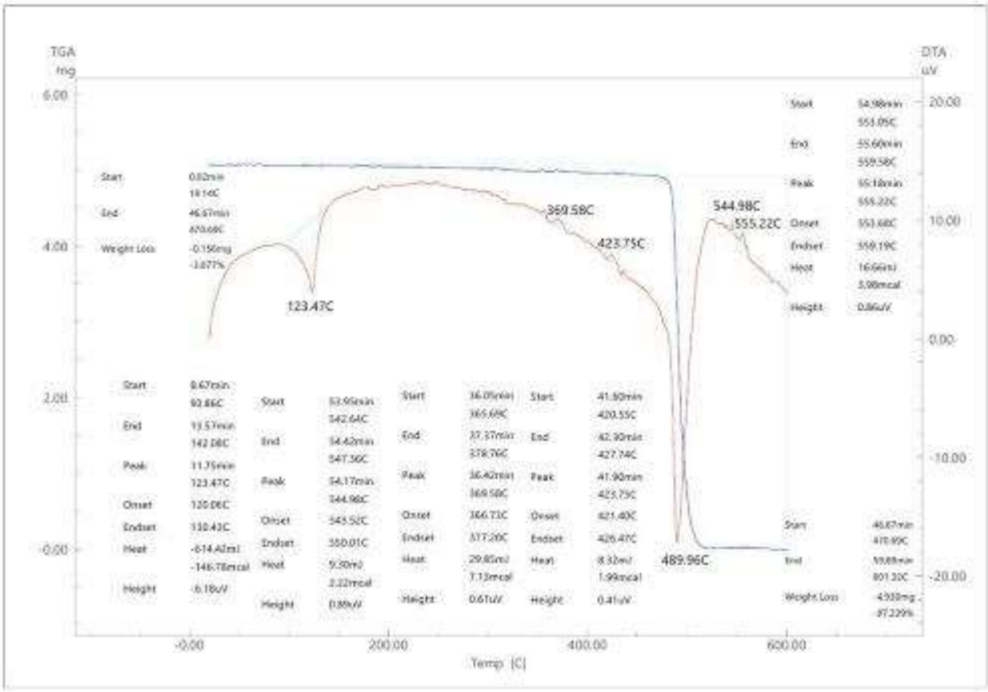


Figure 3.18. Derivatogram of polyethylene with 3% M-2 organic pigment added

The maximum temperature of 600°C was selected for polyethylene with 3% K-1 brand organic pigment shown in Figure 3.18, and the analysis results of polyethylene were studied according to the given thermogravimetric derivatogram (TGA) and differential thermogravimetric analysis. Two endothermic effects were observed at temperatures of 123.47°C, 489.9°C and four exothermic effects at temperatures of 369.58°C, 423.75°C, 544.98°C, 555.22°C. Painted polyethylene was taken in an amount of 5.07 mg in an open-mouth crucible made of aluminum heat-resistant to 600°C, and the temperature was gradually increased starting at 20°C.

Analysis of the thermogravimetric curve of polyethylene with 3% M-2 grade organic pigment shows that the TGA curve mainly takes place in the temperature range of 2 intensive mass losses. The 1st mass loss interval corresponds to the temperature of 20-470.69°C, the 2nd mass loss interval corresponds to the temperature of 470.69 - 600°C. The analysis shows that mass loss 1 is the most intensive decay, with mass loss 0.156 mg, i.e. 3% observed in mass loss range 1. The main amount of mass loss in this decay is 4.9 mg, which is 97%.

Differential thermogravimetric analysis of polyethylene with 3% M-2 organic pigment showed that energy release occurred in the range of 93.8 - 142.08°C. Energy absorption occurs between 93.8 - 142.08°C and 371.9 - 516.6°C.

Thermal analysis of polyethylene with 3% K-1 brand organic pigment was performed in the temperature range of 20-600°C. All samples of the thermal analysis of the given polyethylene were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar. In addition, the endothermic and exothermic points of the composition were proved.

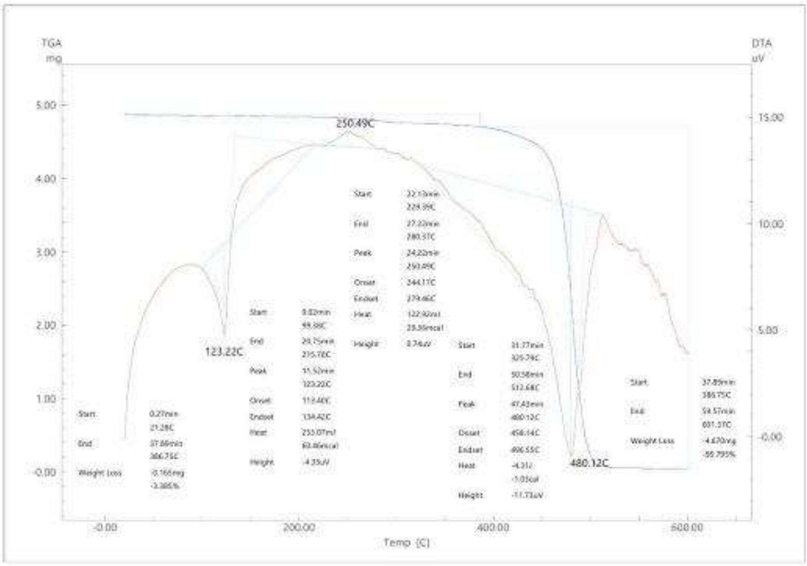


Figure 3.19. Derivatogram of polyethylene with 3% organic pigment K-1

The maximum temperature of 600°C was selected for polyethylene with 3% M-2 brand organic pigment shown in Figure 3.19, and the analysis results of polyethylene were studied according to the presented thermogravimetric derivatogram (TGA) and differential thermogravimetric analysis. Two endothermic effects were observed at 123.22°C, 480.12°C and one exothermic effect at 250.49°C. 4.875 mg of dyed polyethylene was taken in an open-mouthed crucible made of 600°C heat-resistant aluminum, and the temperature was gradually increased starting at 20°C.

Analysis of the thermogravimetric curve of polyethylene with 3% K-1 grade organic pigment shows that the TGA curve mainly takes place in the temperature range of 2 intensive mass losses. The 1st mass loss range corresponds to the temperature of 21.28-386.75°C, the 2nd mass loss range corresponds to the temperature of 386.75 - 600°C. The analysis shows that mass loss 1 is the most intensive decay, with mass loss 0.165 mg, i.e. 3.385% observed in mass loss range 1. The main amount of mass loss in this decay is 4.67 mg, which is 95.795%.

Differential thermogravimetric analysis of polyethylene with 3% K-1 organic pigment showed that energy release occurred in the range of 229.39 - 280.37°C. Energy absorption occurs between 99.38 - 215.78°C and 325.79 - 512.68°C.

Thermal analysis of polyethylene with 3% K-2 organic pigment was performed in the temperature range of 20-600°C. All samples of thermal analysis of painted polyethylene were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar. In addition, the endothermic and exothermic points of the composition were proved.

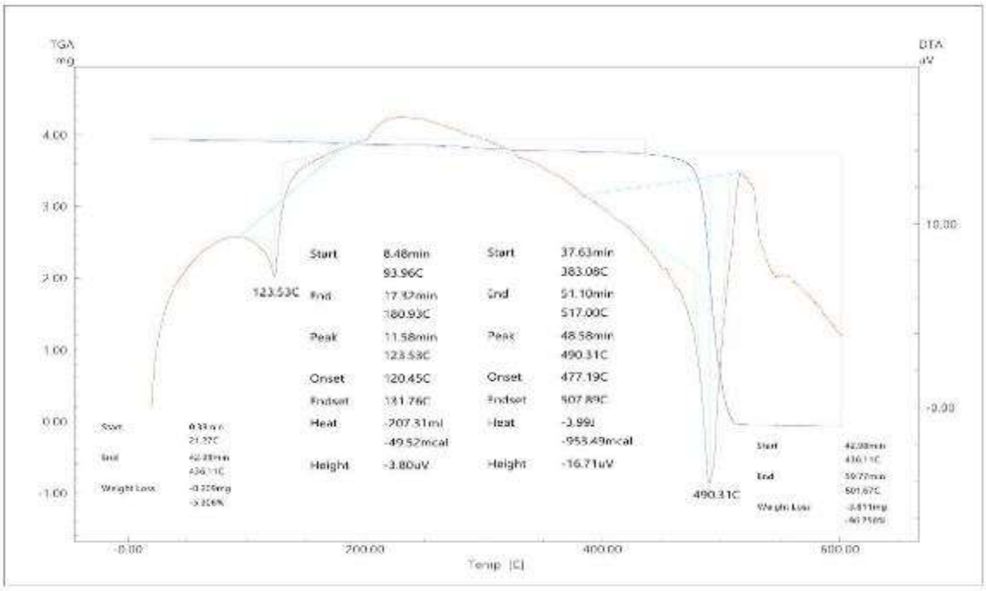


Figure 3.20. Derivatogram of polyethylene with 3% organic pigment K-2

The maximum temperature of 600°C was selected for polyethylene with 3% K-2 brand organic pigment shown in Figure 3.20, and the analysis results of polyethylene were studied according to the presented thermogravimetric derivatogram (TGA) and differential thermogravimetric analysis. Two endothermic effects were observed at temperatures of 123.53°C and 490.3°C. 3.939 mg of polyethylene was taken in an open-mouthed crucible made of heat-resistant aluminum at 600°C, and the temperature was gradually increased starting at 20°C.

Analysis of the thermogravimetric curve of polyethylene with 3% K-2 grade organic pigment shows that the TGA curve mainly takes place in the temperature range of 2 intensive mass losses. The 1st mass loss range corresponds to the temperature of 20-436°C, the 2nd mass loss range corresponds to the temperature of 436 - 600°C. The analysis shows that mass loss 1 is the most intensive decay, with mass loss 0.209 mg, i.e. 5.3% observed in mass loss interval 1. The main amount of mass loss in this decomposition is 3.8 mg, which is 96.7%.

Differential thermogravimetric analysis of polyethylene added with 3% K-2 organic pigment shows that energy absorption occurs in the range of 93.9 - 180.9°C and 383.1 - 517°C.

Derivatogram of polyethylene without organic pigment addition

Thermal analysis of 0320 Polyethylene was performed in the temperature range of 20-600°C. All samples of thermal analysis of painted polyethylene were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar. In addition, the endothermic and exothermic points of the composition were proved.

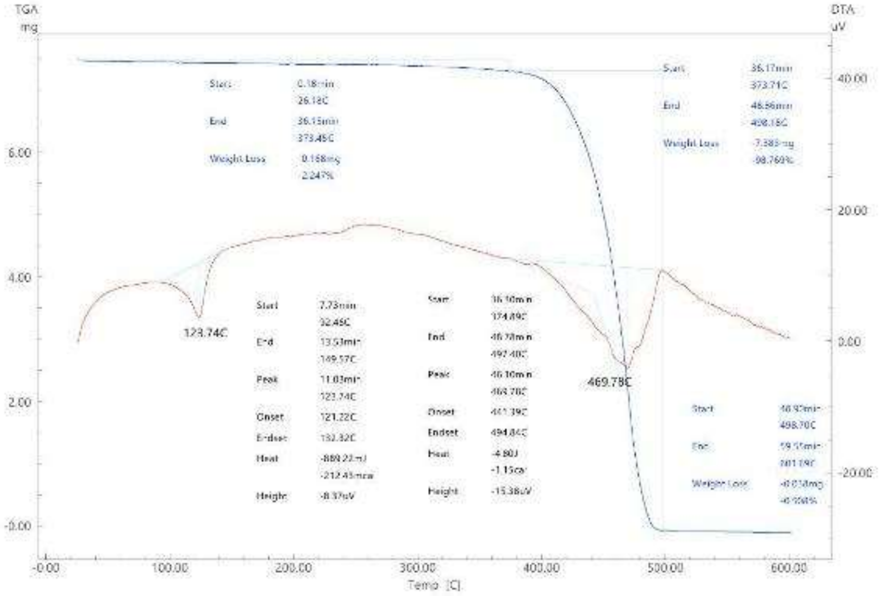


Figure 3.21. Thermogravimetric derivatogram of polyethylene brand 0320.

The maximum temperature of 600°C was chosen for the 0320 polyethylene shown in Figure 3.21, and the analysis results of the polyethylene were studied according to the given thermogravimetric derivatogram (TGA) and differential thermogravimetric analysis. Two endothermic effects were observed at temperatures of 124.07°C and 473.07°C. 6.482 mg of polyethylene was taken in an open-mouthed crucible made of heat-resistant aluminum at 600°C, and the temperature was gradually increased starting at 20°C.

Analysis of the thermogravimetric curve of 0320 polyethylene shows that the TGA curve mainly occurs in the 3 intensive mass loss temperature ranges. Mass-loss range 1 corresponds to temperatures of 22 - 386.29°C, mass-loss range 2 corresponds to temperatures of 386.29 - 504.147°C, and mass-loss range 3 corresponds to temperatures of 504.147-600°C. The analysis shows that mass loss 1 is the most intensive decay, with mass loss 0.054 mg, i.e. 0.8% observed in mass loss interval 1. The main amount of mass loss in this decay is 6.389 mg, which is 98.6%. In mass loss interval 3, the mass loss is 0.003 mg, i.e. 0.05%.

Differential thermogravimetric analysis of 0320 polyethylene shows that energy absorption occurs between 103.7 - 155.6°C and 393 - 508°C.

The results of comparing the derivatograms of polyethylene with new organic pigments and polyethylene without pigments are given in Table 3.6.

It can be seen from Table 3.6 that all the samples of polyethylene with added organic pigment were found to be less than the level of decomposition of polyethylene at temperatures up to 400°C. The resulting pigments were found to change the decomposition temperature of polyethylene. If the decomposition temperature of polyethylene is 373°C, the sample of polyethylene with added organic pigment K-1 increases to 386°C, M-1 to 387°C, K-2 to 436°C and M-2 to 470°C. Thermal decomposition temperatures were found to be 470°C, the maximum value for M-2 pigmented polyethylene.

Table 3.6.

Comparison table of derivatograms of polyethylene with new organic pigments and polyethylene without pigments

№	**Temperature, °C**	**Lost mass, mg**	**Lost mass,%**	**Amount of energy consumed (μV*s/mg)**	**Time spent (min)**	**Residual mass dw (mg)**	**dw/dt (mg/min)**
5.75 mg of polyethylene with 3% M-1 brand organic pigment was obtained							
1	100	0.001	0,02	5.031	8,2	5.749	0,00012
2	200	0.006	0,1	11.159	18,2	5.744	0,0003
3	300	0,074	1,28	10.802	28,25	5.676	0,0026
4	400	0,127	2,2	6.595	38,3	5.623	0,0033
5	500	5,528	96,2	4.512	48,4	0.222	0.114
6	600	5,7	99,1	3.436	58,46	0.043	0,097
4.875 mg of polyethylene with 3% M-2 brand organic pigment was obtained							
1	100	0,03	0,6	7,85	9,08	4,845	0,003
2	200	0,04	0,8	13,53	19,18	4,839	0,002
3	300	0,12	2,46	13,3	29,2	4,758	0,004
4	400	0,2	4,1	9,002	39,25	4,674	0,005
5	500	4,6	94,35	7,58	49,38	0,218	0,09
6	600	4,8	98,46	3,9	59,4	0,036	0,08
5.07 mg of polyethylene with 3% K-1 brand organic pigment was obtained							
1	100	0,02	0,39	7.6	9,3	5.05	0,002
2	200	0,03	0,59	12.8	19,4	5.04	0,002
3	300	0,05	0,98	12.4	29,4	5.023	0,002
4	400	0,17	3,35	8.2	39,5	4.9	0,004
5	500	4,37	86,2	4.9	49,7	0.7	0,08
6	600	5,05	99,6	3.8	59,7	0.02	0,08
3.939 mg of polyethylene with 3% K-2 grade organic pigment was obtained							

1	100	0,04	1,02	9.17	9,1	3.9	0,004
2	200	0,07	1,7	14.5	19,2	3.87	0,004
3	300	0,14	3,5	14.58	29,6	3.8	0,005
4	400	0,24	6,04	10.8	39,3	3.7	0,006
5	500	3,4	86,3	2.9	49,5	0.5	0,07
6	600	3,8	96,5	3.9	59,6	0.08	0,06

Table 3.6 continue

For the control, 7.475 mg of 0320 polyethylene was taken in total mass							
1	100	0,106	1,42	9.65	9,05	7,36	0,011
2	200	0,15	2,08	13.29	19,4	7.32	0,007
3	300	0,3	4,2	14.09	29,3	7.17	0,01
4	400	0,56	7,56	11.24	39,8	6.9	0,02
5	500	7,43	99,5	4.5	49,3	0.045	0,15
6	600	7,46	99,8	2.67	59,8	0.015	0,13

Conclusion on Chapter III

This chapter presents the results of research on the application of synthesized organic pigments. The resulting pigments were used in lok-paint and polyethylene coloring. Based on pigments, a new colored alkyd enamel of the PF-115 class was prepared and coated on a metal plate. The adhesion properties of the coating and its resistance to the static effect of water were studied. It has been proven to meet the requirements of GOST 6465-76.

The thermal analysis of lok-paint products made on the basis of new organic pigments was carried out, and it was proved that they are thermally more stable than CuPs PR 15:1 pigment imported from abroad. Scanning electron microscope analysis of paints based on newly synthesized M-1, M-2, K-1 and K-2 organic pigments shows that the pigment is evenly distributed throughout the volume of the paint mass.

It was found that the relative tensile strength of polyethylene samples with organic pigment increases in the following order:

PE < K-1 < M-1 < K-2 < M-2.

The resulting pigments were found to change the decomposition temperature of polyethylene. If the decomposition temperature of polyethylene is 373°C, K-1 increases to 386°C, M-1 – 387°C, K 2 – 436°C and M-2 – 470°C in the sample of polyethylene with added organic pigment. The maximum temperature of thermal decomposition for M-2 pigmented polyethylene was found to be 470°C.

CHAPTER IV. TECHNOLOGICAL SCHEME OF OBTAINING ORGANIC PIGMENTS BASED ON TEREPHTAL ACID

4.1-§. Technological scheme of obtaining organic pigments based on terephthalic acid

New high-performance organic pigments based on additional metal salts and terephthalic acid were obtained for the synthesis of organic pigments, and efficient production technologies were proposed along with the efficiency of their use.

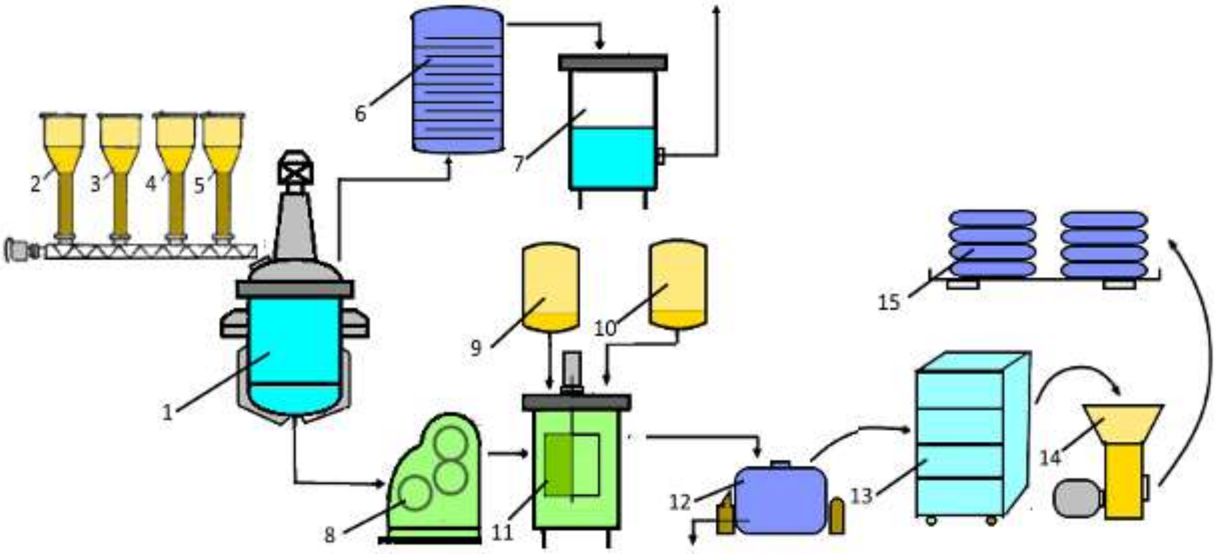


Figure 4.1. Principle technological scheme of organic pigment production

1-Synthesis reactor, 2,3,4,5-dosing capacity, 6-phthalimide trapping device, 7-capacity for ammonia retention, 8-crusher, 9-capacity for sulfuric acid, 10-capacity for water, 11- neutralization reactor, 12-centrifuge, 13-drying cabinet, 14-disintegrator, 15-finished product

Based on the results of our research, the production technology of M-1, M-2, K-1 and K-2 organic pigments synthesized on the basis of terephthalic acid was developed at Termiz State University. The special features of the proposed technological scheme for the new organic pigments obtained on the basis of terephthalic acid are that the production of the obtained organic pigments was carried out in a reactor, by the method of heating at high temperature and in a more simplified order of the existing technologies. In addition, the organic pigments obtained were mainly obtained on the basis of domestic and local raw materials.

The technological scheme for the production of new organic pigments is presented in Figure 4.1 below.

In order to obtain organic pigments based on terephthalic acid, terephthalic acid, urea, phthalic anhydride, metal salts (CuCl; $CaCl_2$; $CoCl_2$;) and catalyst are measured from the 2, 3, 4, 5 dispensers in the required amount, and the 1st synthesis is lowered into the reactor, and the temperature is slowly raised to 130 It is heated up to -135°C until liquefaction. At this temperature, phthalic anhydride and urea liquefy, terephthalic acid dissolves in this liquefaction. After complete liquefaction, the mixture is stirred using an anchor stirrer, and the temperature is slowly raised to 185°C and heated for 1 hour. During the reaction, phthalimide and NH3 gas are released, the released phthalimide 6 is captured in a special trapping device. 7. In order to retain ammonia, ammonia gas is absorbed by water and ammonia water is obtained. After the completion of the reaction, a porous brown substance is formed in the 1st synthesis reactor. The resulting substance is cooled to room temperature and sent to the 11th neutralization reactor after grinding through the 8th crusher. It is exposed to 9th concentrated sulfuric acid. In this case, the reactor temperature is 60°C for 15 minutes and acid is added to get rid of unreacted substances. Then 10-boiling water is poured into it and mixed in 11-neutralizing reactor, and organic pigment precipitates. The resulting aqueous solution is washed in a 12-centrifuge and washed with water until the medium reaches pH = 7. The final product is dried in drying oven 13 at 60°C until it reaches the final mass. The dried organic pigment is crushed in the 14th disintegrator. is sieved and the finished organic pigment is weighed. The finished product, which is the 15th harvest, is transferred to the storage warehouse and packaged and distributed to the consumer market.

CONCLUSION

1. The technology for obtaining M-1, M-2, K-1 and K-2 organic pigments synthesized by heating in the presence of terephthalic acid, urea, phthalic anhydride, metal salts and a catalyst was developed.

2. Properties of M-1, M-2, K-1 and K-2 organic pigments were studied using scanning electron microscopy (SEM), thermal analysis and UV-spectrophotometry. According to the results of the thermal analysis, the obtained organic pigments were found to be a high temperature stable pigment, unlike CuPc PR 15:1 pigment obtained for comparison.

3. Alkyd enamel was prepared by adding 5% of newly synthesized organic pigments and CuPc PR 15:1 pigments obtained for comparison. Thermal analysis of obtained enamel showed that the enamel prepared by adding M-1, M-2, K-1 and K-2 organic pigments has more thermally stable properties than the control enamel. According to the results, it was found that CuPc PR 15:1 pigments have a number of advantages compared to the synthesized organic pigment.

4. The dependence of the content of synthesized organic pigments based on terephthalic acid on temperature, duration of synthesis, ratio of initial components and pH of the environment was determined.

5. Thus, the production of M-1, M-2, K-1 and K-2 organic pigments passed the tests successfully. Based on the standard requirements of PF-115 enamel of different colors, the newly synthesized pigment has been proven to show good results according to GOST 6465-76. PF-115 enamel prepared by adding organic pigment was recommended to be used as a coating for building materials, iron, wood, etc.

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