

Original Article

Innovative Synthesis and Properties of Terephthalic Acid-Based Phthalocyanine Pigments

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Received: 19 August 2025

Revised: 12 November 2025

Accepted: 21 November 2025

Published: 19 December 2025

Abstract - In this article, the optimal conditions for obtaining a new phthalocyanine pigment based on terephthalic acid were studied. Also, phthalic anhydride, terephthalic acid, urea, copper(I) chloride, calcium chloride, and ammonium heptamolybdate were used for synthesis. To establish the optimal synthesis conditions for the newly developed phthalocyanine pigment, various molar ratios of the initial reagents were examined. The best composition was determined to be 0.2:0.2:1:0.1:0.2:0.01, with the synthesis conducted at 130-135 °C for one hour, yielding an average reaction efficiency of 91.1%. The chemical structure of the resulting phthalocyanine was characterized using Infrared (IR) and Ultraviolet (UV-Vis) Spectroscopy, while its thermal behavior was evaluated through Thermogravimetric (TG) and Differential Thermal (DTA) Analyses. In addition, the surface morphology and elemental composition were examined by Scanning Electron Microscopy (SEM) and elemental analysis. Alkyd-based coatings formulated with this phthalocyanine pigment were subsequently applied to various substrate plates for performance testing.

Keywords - Ammonium Heptamolybdate, Copper(I) Chloride, Calcium Chloride, Phthalocyanine Pigment, Phthalic Anhydride, Terephthalic Acid, Urea.

1. Introduction

After the serendipitous re-discovery of metal complex phthalocyanines by industry, and the elucidation of their structures, Copper Phthalocyanine (CuPc) pigments were introduced industrially in the 1930s to become, and remain, by far the most important blue and green organic pigments, finding almost universal use as colorants for paints, printing inks, plastics, and a wide range of other applications [1]. It is well established that phthalocyanine compounds are colorless in their monomolecular state. However, when they form coordination complexes, they exhibit strong absorption in the blue region of the visible spectrum, which accounts for their characteristic color [2]. Among the various types of phthalocyanine pigments, copper phthalocyanine and its derivatives are the most widely produced and commercially available, being extensively employed in modern coatings, printing inks, plastics, and synthetic fibers [3]. These pigments possess remarkable thermal and chemical stability compared with many other organic colorants [3]. Structurally, the synthesis of the phthalocyanine macrocycle proceeds via a cyclic tetramerization reaction. Typical precursors used for this process include phthalonitrile, phthalimide, phthalic anhydride, and 1,3-diiminoisoindole. In general, the metal-

free phthalocyanine base is rarely utilized due to its limited stability and inferior coloristic performance compared to its metallated analogues [4]. As a coloring agent, it can be used even for inert polymers such as polyethylene and polypropylene. The amorphous phase in the composition was proved to be soluble in dioxane and heptane, and a tight molecular solution of CuPc, CuPc molecules have been found to be absorbed into polyethylene, polypropylene, polycapromide, and cellulose triacetate films. It has been shown that the optical spectrum of individual CuPc molecules is significantly different from that of pigment particles [5]. In this work, Au(III)-phthalocyanine (Au-Pc) and Au(III)-2(3)-tetrakis(allyloxy)phthalocyanine (Au-AoPc) are described using phthalonitrile, 3-allyloxypthalonitrile. 3-Alloxy phthalonitrile and gold nanoparticles were obtained as starting materials. Gold nanoparticles regenerated with N, N'-dimethyl amino ethanol were prepared from HAuCl₄ solution. Purple gold nanoparticles were obtained by adding N, N'-dimethyl amino ethanol and reducing HAuCl₄ solution at pH 6-7 at room temperature. Then, blue Au-Pc and green Au-AoPc paint materials are synthesized by reactions of gold nanoparticles with phthalonitrile and 3 - allyloxypthalonitrile at a temperature of 120°C [6].



Copper phthalocyanine was synthesized in a silica gel xerogel matrix using a newly developed *in-situ* method using a sol-gel process. Copper phthalocyanine molecules were gradually synthesized in situ during the transition from wet gel to xerogel [7]. Also, phthalocyanine compounds have the property of polymorphism; for example, there are α , β , and γ modifications of copper phthalocyanine. 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 [8]. Optimum conditions of the process of dyeing wool materials with tri-sulfonic acid of copper phthalocyanine were also 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. In addition, in modern methods, methods of obtaining compounds with various special properties are also used by modifying the surface with the help of nanoparticles. For example, a nanocomposite of zinc phthalocyanine with titanium oxide was used as the catalyst for the selective oxidation of aromatic nitro compounds. The selectivity was 99% [9-11].

The reaction of 4-nitrophthalonitrile with 4-aminobenzoic acid in dimethyl sulfoxide, in the presence of potassium carbonate, yielded 4-[(4'-carboxy)phenylamino] phthalonitrile [12]. In a separate synthetic route, nucleophilic substitution of the bromine atom in 4-bromophthalonitrile with anthraquinone produced 4-[(2-hydroxy-9,10-dioxo-9,10-dihydroanthracen-1-yl)oxy] phthalonitrile. This intermediate was subsequently employed in the preparation of the corresponding cobalt phthalocyanines via the nitrile cyclotetramerization method [13].

Alkoxyphenoxyphthalonitriles were synthesized by nucleophilic substitution to obtain phenolic residues substituted with 4-nitrophthalonitrile. The stability of substituted phthalonitriles and phthalocyanines synthesized on their basis (1:1 ratio) was studied by heating in an argon-oxygen atmosphere. 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. A new copper phthalocyanine containing phenoxy substituents was synthesized and studied by mass spectrometry and elemental analysis methods. Its photoluminescence absorption spectra were examined. By using this copper phthalocyanine attached to 4,4'-N, N'-carbazole-biphenyl, organic light-emitting devices were prepared. The electrophosphorescence state at room temperature is due to the transition from the mostly excited triplet state to the singlet state (T_1-S_0) of CuPc observed at about 1.1 nm [14]. It has been provided that 3,3',3'',3'''-tetraaminophthalocyanines of Cu^{2+} , Co^{2+} , Ni^{2+} , and Zn can be used as antibacterial agents. In the work, 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 [15].

The main goal of this study is to obtain a new type of phthalocyanine pigment based on terephthalic acid for the first time and to analyze the composition and use of the obtained compound.

2. Materials and Methods

2.1. Materials

Chemical substances such as terephthalic acid, phthalic anhydride, urea, copper (I) chloride, calcium (II) chloride, sulfuric acid(93 %), Lacquer PF-060 (52%), Titanium dioxide R-706, Bentogel™ BG 4, Microcalcite, and methylethyl ketoxime were employed in the synthesis of the new phthalocyanine. All chemical reagents were obtained as analytically pure ("chemically pure") from Merit Chemicals.

2.2. IR-Analysis

The structure of this obtained pigment was determined by IR-spectroscopy (Fure spectrometer manufactured in Japan. IR spectroscopic studies were carried out in the powder method on a SHIMADZU infrared Fourer spectrometer (range 4000-600 cm^{-1} , dimensions 4 cm^{-1}).

2.3. SEM Analysis

2.3.1. Scanning Electron Microscopy (SEM)

The morphology of the newly synthesized semiconducting polymer and phthalocyanine-based dyes was examined using a MIRA 2 LMU scanning electron microscope, which was coupled with an INCA Energy 350 Energy-Dispersive X-ray (EDX) microanalysis system.

2.3.2. TGA and DTA Thermal Analysis

SHIMADZU (Simultaneous Thermal Analysis) is an easy-to-use, reliable, and high-performance thermal analysis platform for TGA, simultaneous TGA-DTA, and TGA-DSC analysis methods. It is used to identify parts, measure the optical properties of samples, and also for various research purposes.

2.3.3. M-2 Organic Pigment Synthesis

33.2 g (0.2 mol) terephthalic acid, 29.6 g (0.2 mol) phthalic anhydride, 60 g (1 mol) urea, 9.9 g (0.1 mol) of copper(I) chloride, 22.2 g (0.2 mol) of calcium chloride, and 0.62 g (0.01 mol) of ammonium heptamolybdate were added and stirred. The resulting mixture was gradually heated to 130–135 °C. At this temperature, phthalic anhydride and urea melted, allowing terephthalic acid to dissolve within the liquefied medium. Once complete liquefaction was achieved, the temperature was further raised to 185 °C over the course

of one Hour. Upon completion of the reaction, a porous brown solid was formed. After cooling to room temperature, the solid was treated with 10 mL of 93% concentrated sulfuric acid, resulting in a deep dark green solution. This solution was repeatedly washed with boiling distilled water until a neutral pH was reached, thereby removing unreacted starting materials and soluble intermediates.

The desired M-2 organic pigment was then precipitated, collected by vacuum filtration using a Buchner funnel, and dried in an oven at 60 °C until constant mass was obtained.

Finally, the dried pigment was ground using a mortar, sieved, and weighed to determine the final product yield.

When calculating the finished product, the yield was 91.1%. The optimal conditions and physicochemical properties of the M-2 organic pigment were investigated, focusing on identifying the best composition that yields the highest output. Table 1 presents a study on how temperature and the ratio of starting materials influence the product yield during the synthesis of the highly effective M-2 organic pigment.

Table 1. Effect of temperature and ratio of substances on the yield synthesis

| No | Tereph acid:PhA: Urea:CuCl: CaCl ₂ | °C | ω,% | No | TA:PhA:U: CuCl:CaCl ₂ | °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 |

Note* TA-Terephthalic Acid, PhA-Phthalic Anhydride, U-urea,

A series of experiments was carried out to synthesize the M-2 organic pigment under varying conditions and reactant proportions. The resulting pigment yield was evaluated as a function of both temperature and the molar ratios of the

starting materials [16, 17]. The optimal reaction temperature was determined to lie between 185 and 200 °C, with the maximum yield achieved at a molar ratio of 1:1:5:0.5:1 for the initial components (Figure 1).

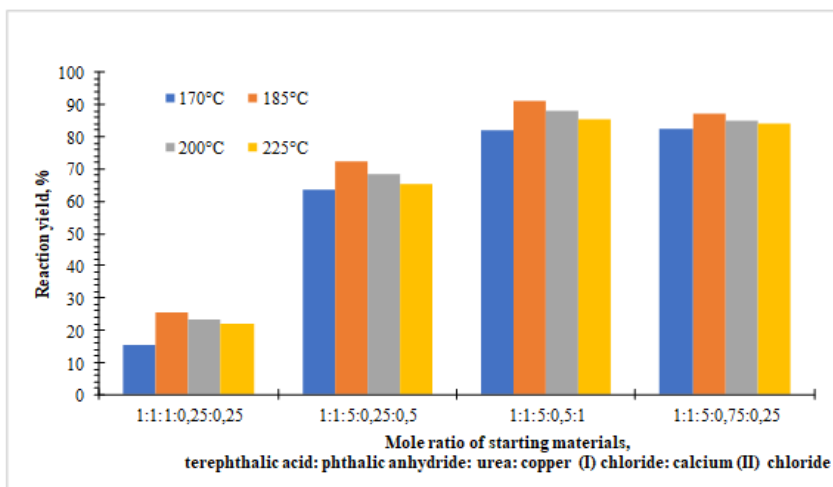


Fig. 1 The effect of the amount of starting materials and temperature on the reaction yield of M-2 organic pigment

2.4. IR Spectrum Analyses

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, valence absorption bands belong to residual water molecules. It was observed that the absorption band belonging to the carbonyl group of

terephthalic 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 [18, 19].

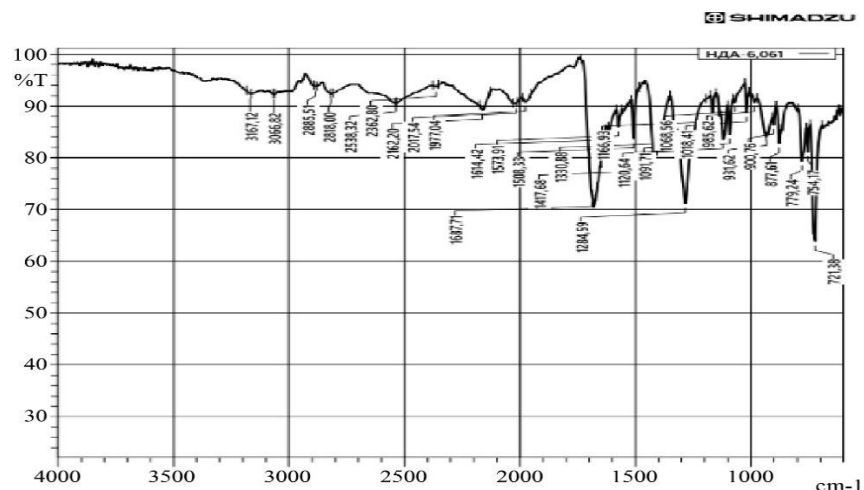
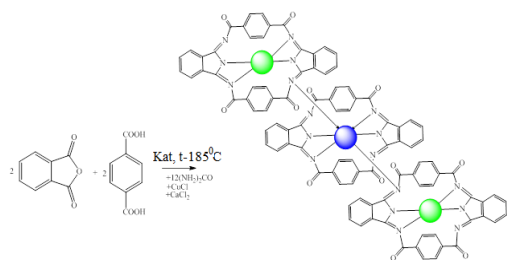


Fig. 2 IR spectrum of M-2 organic pigment

Hydrogen atoms with different positions in benzene rings have valence vibrations in the region of $2800\text{--}3000\text{ cm}^{-1}$ and deformation vibrations of low intensity in the region of $1600\text{--}1900\text{ cm}^{-1}$. 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} . M-2 brand organic pigments were synthesized based on the following reaction equation:



Scheme-1. Synthesis of the M-2 brand organic pigment

The fact that the synthesized organic pigments were obtained based on the above-mentioned reaction equations and the proposed formulas was confirmed by the conducted studies [20, 21].

2.5. UV-Spectrophotometer Test

Figure 3 shows the results of measuring M-2 brand organic pigment under the visible light spectrum of $190\text{--}1100\text{ nm}$ in a UV-spectrophotometer.

The M-2 organic pigment exhibited high absorbance within the visible wavelength range of 430 nm to 600 nm .

The highest absorption has been proven at a wavelength of 550 nm . Previous literature has demonstrated the use of natural pigments as sensitizing dyes in solar cells, suggesting a similar potential for the M-2 organic pigment in solar cell applications [22].

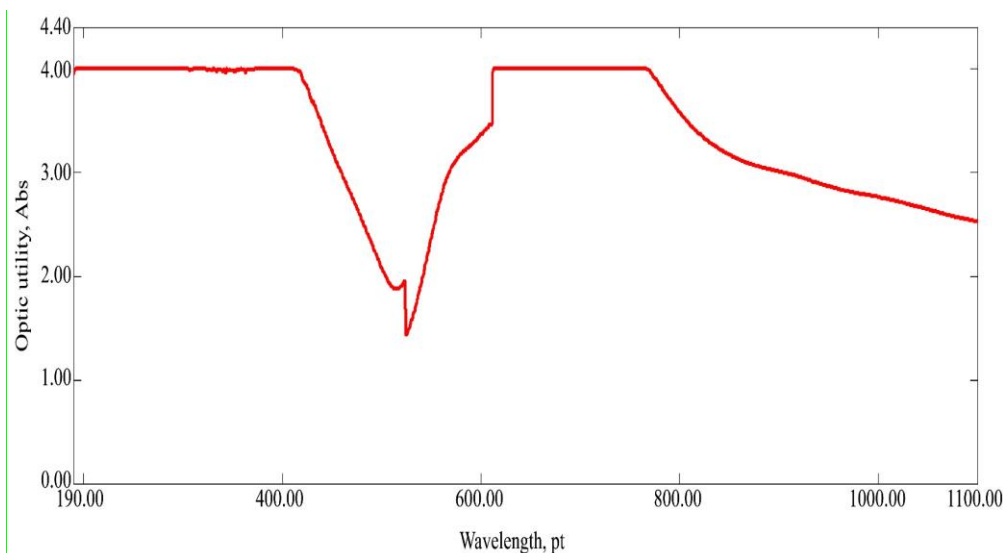


Fig. 3 The light absorption at the wavelength of the organic pigment

2.6. TGA and DTA Analysis

A 3.8 mg sample of the M-2 organic pigment was subjected to Thermogravimetric Analysis (TGA) up to 600 °C. The results of TGA and Differential Thermal Analysis (DTA) are presented in Figure 4.

The Thermogravimetric curve of the M-2 pigment reveals three distinct regions of significant mass loss across specific temperature ranges. The first mass loss occurs between 25.67 and 221.49 °C, the second between 221.49 and 331.06 °C, and the third between 331.06 and 600 °C. The initial mass loss is the most pronounced, corresponding to 0.098 mg, or 2.576% of the sample. The primary mass reduction, accounting for 2.576 mg (67.718%), occurs in the second region, while the third region exhibits a mass loss of 0.625 mg, representing 16.43% of the total sample. The Thermogravimetric Analysis (TGA) of the M-2 organic pigment indicates three distinct stages of mass loss:

The first stage involves the loss of excess moisture and absorbed water. In the second stage of decomposition, lower molecular weight compounds such as carbon dioxide and ammonia are released [23]. Following the decomposition of the organic portion, a mixture of metal carbonates, oxides, and some residual coal remains. The third stage of decomposition involves further breakdown of carbonates, resulting in the formation of metal oxides and carbon residues. Additionally, the Differential Thermal Analysis (DTA) of the M-2 organic pigment, as presented in Figure 4, reveals specific thermal events:

Two exothermic effects were observed at temperatures of 451.72°C and 552.83°C. Two endothermic effects were observed at temperatures of 311.84°C and 510.19°C. These thermal analysis results provide insights into the decomposition behavior and thermal stability of the M-2 organic pigment under different temperature conditions.

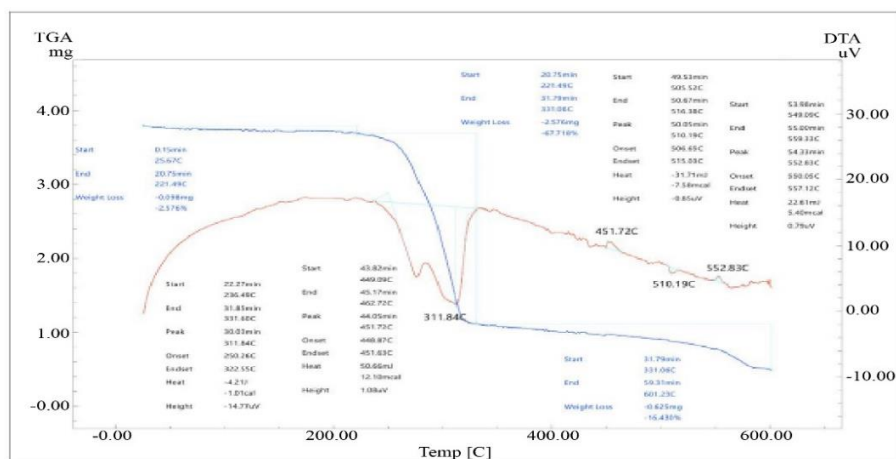


Fig. 4 Thermogravimetric analysis of organic pigment

2.7. SEM and Elemental Analysis

The newly synthesized M-2 organic pigment was examined using Scanning Electron Microscopy (SEM) under high-vacuum conditions. Elemental microanalysis of the composite pigment was performed with an accelerating voltage of 20 keV and a beam current of 1 nA. The investigation focused on determining the elemental composition within selected areas of the pigment sample [24].

Studies have shown that organic pigment particles range in size from 28.40 to ~36.85 nm. At the same time, elemental analysis was performed on a separate surface containing large clusters of the pigment [25]. Large clusters indicate the presence of chlorine and sulfur residues within the experimental error, except for the M-2 organic pigment at the studied points when the element was analyzed (Figure 5).

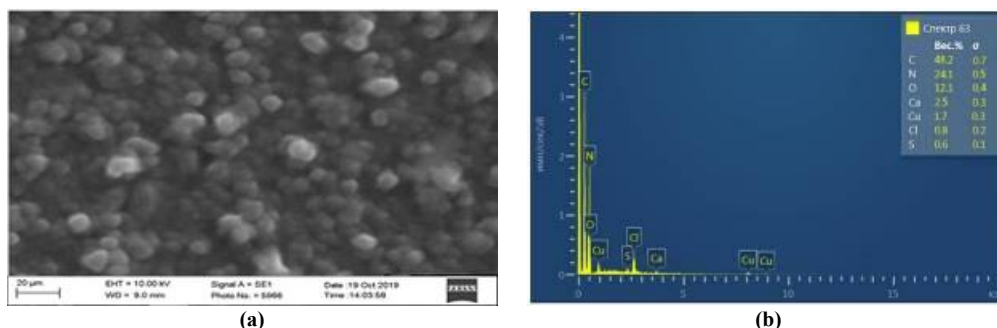


Fig. 5 Scanning electron microscope (a) Elemental analysis, and (b) Organic pigment.

3. Results and Discussion

3.1. Thermal Analysis of Alkyd Enamel based on Organic Pigment

10.07 mg of alkyd enamel paint prepared based on M-2 organic pigment was taken, and the process was carried out at a range 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, and 489°C (Figure 6) Analysis of the

Thermogravimetric curve of the enamel obtained reveals three distinct stages of intensive mass loss occurring at different temperature ranges:

The first mass loss range corresponds to temperatures between 32.06°C and 236.92°C.

The second mass loss range corresponds to temperatures between 236.92°C and 500.24°C.

The third mass loss range corresponds to temperatures between 500.24°C and 600°C.

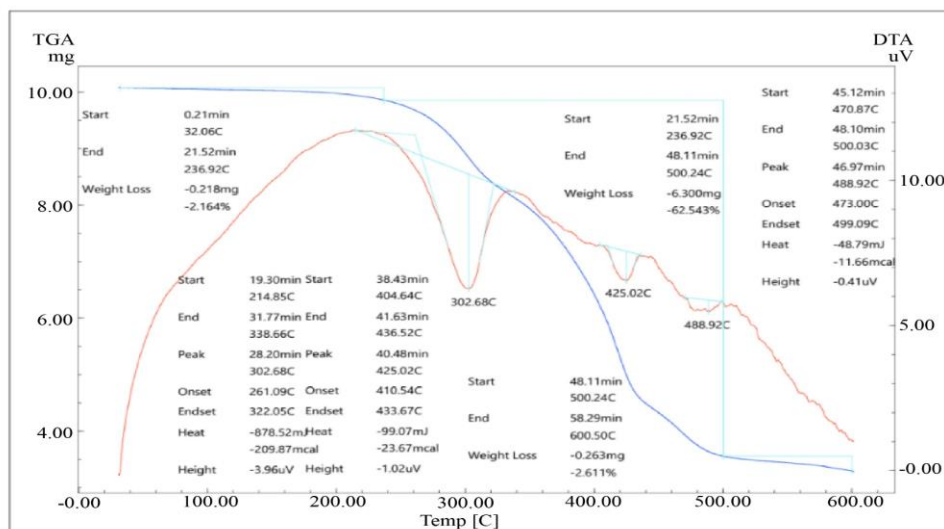


Fig. 6 Derivatogram of Alkyd Enamel made based on M-2 organic pigment

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, i.e., 2.6%. The differential thermal analysis of alkyd enamel paint indicates that energy absorption occurs within distinct temperature ranges: 214.85°C to 338.66°C, 404.64°C to 436.52°C, and 470.87°C to 500.03°C.

3.2. Synthesis of the Bimetal Phthalocyanine Pigment based on Terephthalic Acid

Utilizing the procedure outlined in existing literature, supplementary terephthalic acid and calcium chloride are introduced to the initial substrate in the process of copper phthalocyanine synthesis. Subsequently, the mixture is subjected to heating at approximately 240 °C for a duration of 90 minutes, accompanied by stirring for a span of 4 hours. The resultant porous material is subsequently cooled and dissolved in 93% H₂SO₄. This solution undergoes multiple washings with water [26]. The ensuing precipitate is isolated using a vacuum filter and subsequently subjected to drying at 60 °C for a day. The final product is further pulverized and sieved

through specialized equipment (Figure 7).



Fig. 7 M-2 organic pigment samples

3.3. Preparation of Alkyd Enamel with M-2

Table 2. New types of Alkyd composition based on the M-2 brand organic pigment

| № | The name of the components | The content of mass. % |
|---|----------------------------|------------------------|
| 1 | Lacquer PF-060 (52%) | 73,8 |
| 2 | Titanium dioxide R-706 | 0,5 |
| 3 | Bentogel™ BG 4 | 0,4 |
| 4 | Microcalcite | 20,8 |

| | | |
|---|-------------------------------|-----|
| 5 | M-2 brand new organic pigment | 5 |
| 6 | Methylethyl ketoxime | 0,5 |

The synthesis of the M-2 brand organic pigment is executed through the use of a laboratory-scale bead mill (as illustrated in Figure 2) for all preparations. The imperative requirement for paint production is the attainment of a uniform system through effective mixing of this coloring pigment. The experimental observations illustrate a pronounced reduction in the particle size of the pigment, facilitating the dispersion of agglomerates and the formation of a refined suspension. In the dyeing process, the pigment and dye raw materials undergo rapid mixing within a specialized steel container, facilitated by a dispersing knife. This results in the transfer of solids into the suspension medium (depicted in Figure 8).

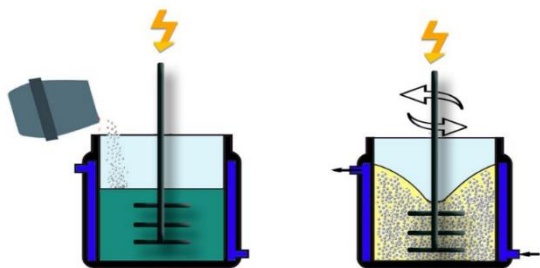


Fig. 8 M-2 Brand organic pigment is completely mixed in this device for two hour, and coated on a metal plate

To assess the coating's resistance against static water exposure, the procedure involves applying and drying paint on samples, which are subsequently left to air-dry for a period of five days prior to testing. Subsequently, the dried samples are immersed in distilled water to a level of 2/3, as per the guidelines outlined in GOST 6709, and maintained in this state for a duration of 10 hours. Post-test, the samples are subjected to a two-hour air-drying process at $(20 \pm 2) ^\circ\text{C}$, following which the coating's visual appearance is examined. After the addition of Lok, titanium dioxide, M-2 organic pigment, and bentogel, the mixture was dispersed in a bead mill at room temperature for 2 hours (bead diameter: 3–3.5 mm; rotation speed: $1500\text{--}2000 \text{ min}^{-1}$) until a homogeneous pigment paste was obtained. Subsequently, enamel was prepared by incorporating siccative and methyl ethyl ketoxime [27].

The new M-2 organic pigment was synthesized using a laboratory pearl mill, a small-scale paint preparation device commonly employed for paint production. A critical requirement in paint formulation is the complete incorporation of the coloring pigment into a homogeneous system. This involves breaking down and dispersing pigment agglomerates to achieve a fine, uniform suspension. During the preparation process, the pigment and base paint components were combined in a specialized iron vessel and mixed at high speed using a dispersion knife [28–33]. This method ensures efficient dispersion of solids into the suspension medium,

facilitating the uniform distribution of the organic pigment within the paint formulation. Organic Pigment M-2 was coated on a metal plate with complete mixing in this Device for 2 hours (Figure 9).



Fig. 9 Alkyd enamel coated on a metal plate (based on new pigments)

4. Conclusion

According to research results, methods and tools, synthesis of M-2 branded pigments, and the results of studies on the influence of various factors on synthesis conditions and their analysis are presented. The optimum temperature for the synthesis of M-2 pigments was 185°C . It was found that the reaction yield was the highest when the ratio of starting materials was 1:1:5:0.5:1 in M-2 pigment TK:FA:Kar:CuCl:CaCl₂. 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 M-2 brand pigments was studied.

Acknowledgment

The authors thank Termez State University and Termez Institute of Engineering and Technology for supporting this research work.

Authors' Declaration

- I/We hereby confirm that all the Figures and Tables in the manuscript are mine/ours.
- Ethical Clearance: The project was approved by the local ethical committee in the University of Termez State University and the Termez Institute of Engineering and Technology.

Authors' Contribution Statement

Nabiev D.A: Writing - Original Draft. Turaev Kh.Kh: Reviewing and editing paper. Toshkulov A. H: Reviewing and editing paper and Software, Validation. Nomozov A.K. and Eshankulov Kh. N: Writing - Original Draft, Conceptualization, Investigation, Visualisation.

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