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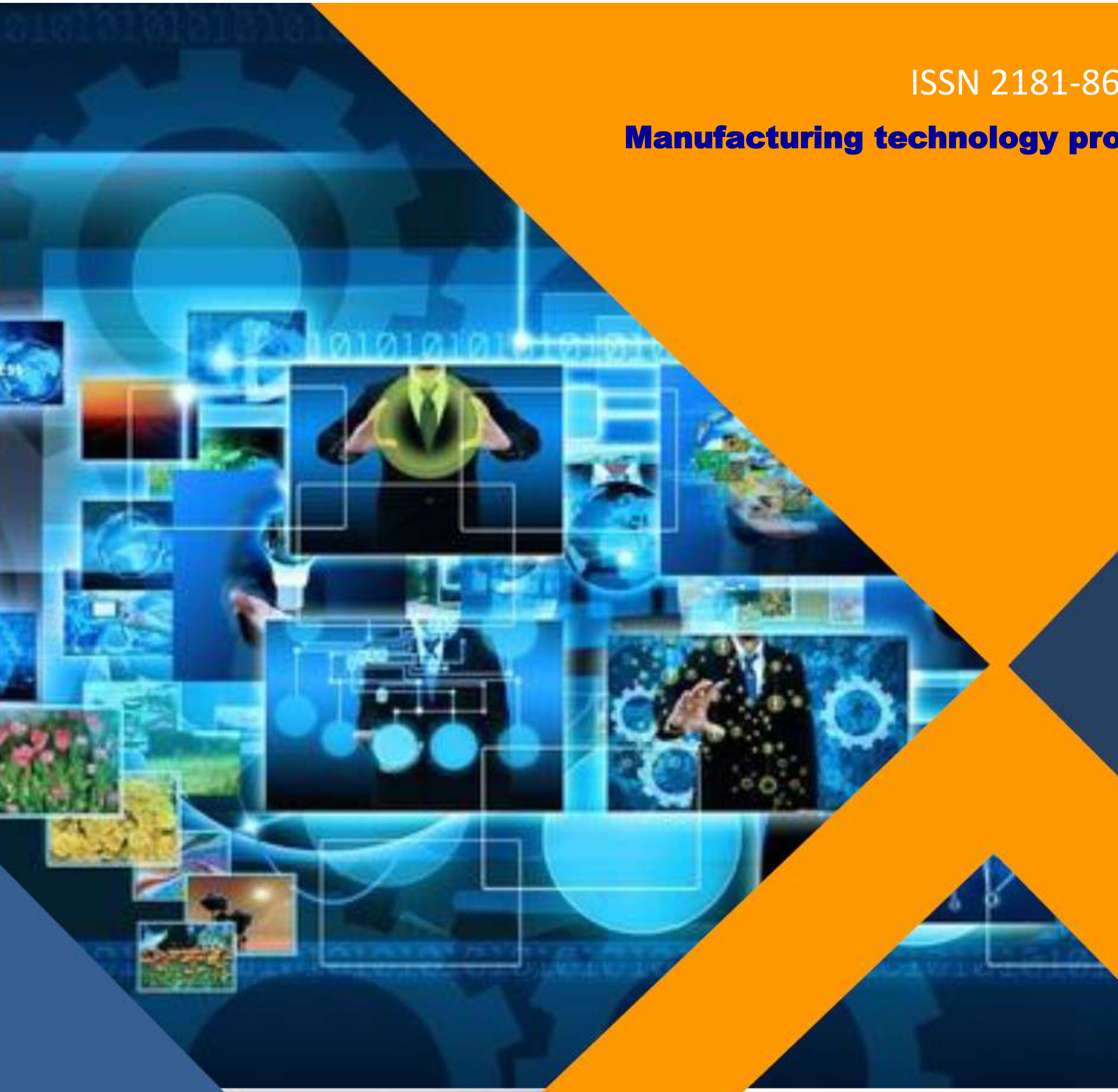

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ELEMENTAL ANALYSIS OF CARBOXYL-MODIFIED COPPER PHTHALOCYANINE PIGMENT

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Abstract: This article presents the elemental analysis results of copper phthalocyanine (CuPc–COOH) pigment modified with carboxyl functional groups. Using CHNS-O analysis, atomic absorption spectroscopy, and X-ray photoelectron spectroscopy (XPS), the percentages of C, H, N, O, and Cu elements in the pigment were determined. The results indicate that the carboxyl groups were successfully introduced into the pigment structure, leading to increased molecular polarity while maintaining the stability of the metal–ligand complex. The elemental analysis also made it possible to evaluate how the introduced functional groups influence the pigment's physicochemical properties and its chemical integration capability with polymer binders.

Keywords: copper phthalocyanine, carboxyl modification, elemental analysis, CHNS-O, XPS, CuPc–COOH, functional pigments.

Introduction. Metal phthalocyanines (MPcs) represent one of the most structurally robust and technologically versatile classes of organic macroheterocyclic compounds. Due to their exceptional thermal stability, strong absorption in the visible region, and resistance to chemical degradation, phthalocyanines are extensively utilized as pigments, catalysts, semiconductors, and functional coatings[1-3]. The fundamental structural element of these compounds is an aromatic and highly conjugated 18-π electron macrocycle, capable of coordinating to various transition metal ions such as Cu^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, and others. This coordination imparts unique physicochemical properties, making metal phthalocyanines attractive for applications in optoelectronics, sensing, photovoltaics, corrosion protection, and polymer-based coating technologies[4].

Among these, copper phthalocyanine (CuPc) stands out as one of the most industrially significant pigments due to its vibrant blue–green coloration, excellent weather and light fastness, and compatibility with a wide range of polymer systems. It has been widely used in paints and coatings, printing inks, plastics, rubber formulations, and organic electronic devices[5-8]. Despite these advantages, the classical CuPc pigment exhibits important limitations, such as poor solubility in most solvents, limited chemical reactivity, and insufficient dispersibility in some polymer matrices. These drawbacks restrict its broader application in advanced material systems where improved interfacial interaction, controlled polarity, and tailored surface chemistry are increasingly required.

Functional Modification of Phthalocyanines: Scientific Rationale. To overcome the inherent limitations of unmodified CuPc, recent research has focused on peripheral functionalization of the macrocycle. Introducing functional groups such as –COOH, –SO_3H, –NH_2, –OH, or halogen substituents can significantly alter the pigment's polarity, solubility, charge distribution, surface activity, and compatibility with polymeric or inorganic environments. Among these modifications, carboxylation stands out as one of the most effective routes for enhancing the surface and chemical properties of the pigment[9-11].

Functional groups positioned on the aromatic periphery of the phthalocyanine ring can affect:

Electronic structure and resonance stabilization

– Introduction of electron-withdrawing or electron-donating substituents modifies π-electron distribution.

Intermolecular interactions

– Hydrophobic or hydrophilic interactions can be tuned to enhance dispersibility.

Coordination and bonding properties

– Carboxyl groups can participate in hydrogen bonding, esterification, cross-linking, and ionic interactions with metal cations.

Polarity and surface energy

– A direct influence on pigment–binder compatibility in coatings and polymer composites.

Functional integration in optoelectronic devices

– Modified phthalocyanines can act as active layers in organic solar cells, sensors, and conductive polymers.

Because of these advantages, the development of carboxyl-functionalized copper phthalocyanine (CuPc–COOH) has attracted significant interest in fields ranging from coating technology to advanced materials science and organic electronics.

Significance of Carboxyl Modification of Copper Phthalocyanine. Modification of CuPc with carboxyl groups (–COOH) leads to a substantial improvement in physicochemical behavior. Carboxyl substituents: increase hydrophilicity, enhancing pigment wetting in polar media, improve colloidal stability and dispersibility in aqueous and polymeric systems, enhance chemical reactivity, enabling covalent bonding to polymeric binders, allow ion-exchange interactions due to acidic functionality, contribute to improved compatibility with epoxy, polyurethane, acrylic, and polyester systems, increase surface polarity, enabling controlled assembly in nanostructured materials[12-14].

These modifications provide new opportunities for using CuPc–COOH in: water-based and high-performance coatings, hybrid organic–inorganic composites, conductive polymer matrices (e.g., PEDOT:PSS), dye-sensitized and organic photovoltaic devices, environmental sensors and catalytic systems.

However, the introduction of carboxyl groups may also influence crystal packing, electronic distribution, and coordination geometry around the metal center. Therefore, precise structural and elemental characterization is critical.

Materials and Reagents.

All chemicals and reagents were used as received without further purification unless otherwise specified.

Phthalonitrile (99.0%, Sigma-Aldrich) – used as an organic precursor for CuPc synthesis. Copper(II) chloride dihydrate ($CuCl_2·2H_2O$) – metal source for forming the Cu–N coordination center. Aromatic carboxyl-containing precursor (benzoic acid derivative) – functionalization reagent for introducing –COOH groups. Ammonium molybdate catalyst – applied to enhance high-temperature cyclization. Dimethylformamide (DMF), ethanol, deionized water – used as solvents during synthesis and washing procedures. Sodium hydroxide (NaOH), hydrochloric acid (HCl) – for neutralization during purification stages. All solutions were prepared freshly, and deionized water (18.2 MΩ·cm) was used throughout the experiments.

Synthesis of Copper Phthalocyanine (CuPc)

The synthesis of unmodified CuPc pigment was conducted via a classical high-temperature cyclotetramerization reaction.

Reaction Procedure

A mixture containing: phthalonitrile (10 g), $CuCl_2·2H_2O$ (4.5 g), ammonium molybdate (0.2 g), urea (as a reaction facilitator), was loaded into a ceramic reactor and heated in a programmable furnace (SNOL 8.2/1100) under nitrogen flow.

Thermal Conditions

Heating rate: 10 °C/min

Reaction temperature: 235–245 °C

Holding time: 1.5–2 hours

Atmosphere: N_2 (continuous flow, 100 mL/min)

During this stage, copper(II) salt coordinates to the forming isoindole units, producing a planar CuPc macrocycle. The crude pigment turned an intensive blue–green color, confirming cyclization.

Purification of CuPc

The obtained product was washed repeatedly with hot DMF and water to remove unreacted precursors, then neutralized to pH 7 and dried at 105 °C for 12 hours.

Carboxyl Functionalization of CuPc (Synthesis of CuPc–COOH)

Reaction Setup

5 g of purified CuPc pigment was dispersed in DMF (150 mL) using a magnetic stirrer at 500 rpm for 30 minutes. Then, the carboxyl-containing aromatic precursor (3.0–3.5 g) and catalytic amount of NaOH were added.

Oxidation-Assisted Functionalization

The mixture was transferred to a stainless-steel autoclave and heated at:

Temperature: 180–200 °C

Duration: 6 hours

Atmosphere: Air (oxidative medium)

Pressure: Autogenous

Under these conditions, electrophilic substitution on the aromatic periphery of CuPc occurs, followed by oxidative stabilization, enabling the attachment of –COOH groups to the macrocycle.

Post-Reaction Purification

After cooling, the reaction mixture was:

Filtered through a 0.45 μm PTFE membrane

Washed consecutively with warm ethanol, water, and dilute HCl

Neutralized to pH 7

Dried in a vacuum oven at 90 °C for 10 h

The final product, CuPc–COOH, exhibited improved dispersibility and increased hydrophilicity compared to unmodified CuPc.

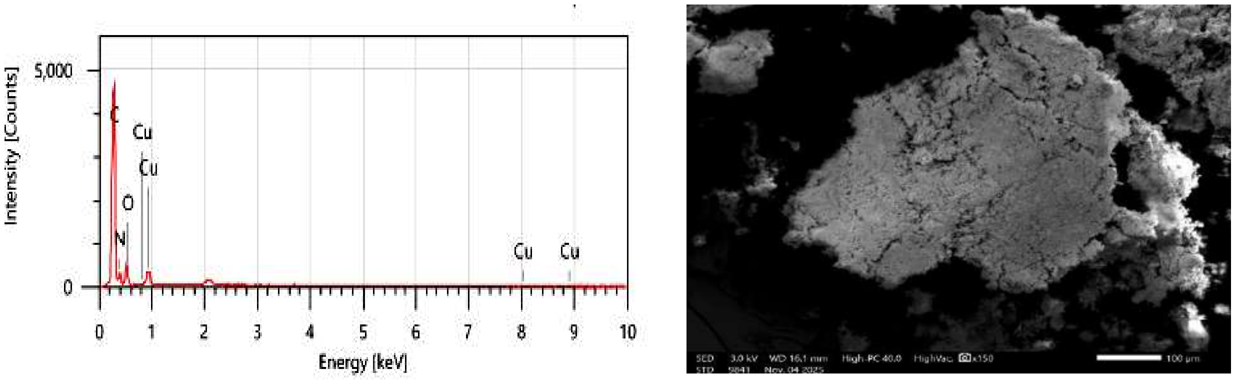

Figure 1. Elemental analysis of the carboxyl-modified copper phthalocyanine pigment

The following figure presents the EDX/XRF elemental analysis results of the carboxyl-modified copper phthalocyanine (CuPc–COOH) pigment. The quantified elemental composition is as follows:

C: 62.81 mass%, 70.77 atom%

N: 17.62 mass%, 17.02 atom%

O: 12.70 mass%, 10.75 atom%

Cu: 6.87 mass%, 1.46 atom%

Quantification method: ZAF, standardless

Fitting ratio: 0.0227 (excellent agreement between model and spectrum)

Purity and Elemental Composition

The pigment composition is entirely confined to C–N–O–Cu, with no detectable foreign metals or impurities such as Fe, Zn, Ca, S, or other elements. This confirms: the correctness and efficiency of the synthesis and washing procedures, the almost complete removal of catalysts and by-products, the high purity level of the resulting pigment.

Such purity is essential for ensuring reproducible optical, electronic, and catalytic performance, especially in DSc-level materials research.

Preservation of the Phthalocyanine Macrocycle (C/N Ratio)

The atomic percentages C ≈ 70.77% and N ≈ 17.02% result in a C/N ratio ≈ 4.2. The theoretical stoichiometric ratio for classical copper phthalocyanine (CuPc = $C_{32}H_{16}N_8Cu$) is:

$$C/N=32/8=4.0$$

The measured ratio being very close to the theoretical value indicates that: the macrocyclic phthalocyanine core is fully preserved, all eight isoindole nitrogen atoms remain intact, the 18-π electron aromatic conjugation system remains stable, the structural integrity of the CuPc skeleton is not compromised during modification. The slight increase in carbon content is attributed to additional carbon atoms from the peripheral –COOH groups. Conclusion: EDX/XRF data confirm that the internal structure of the CuPc macrocycle remains unchanged after modification.

Presence of Oxygen — Confirmation of Carboxyl Functionalization

Unmodified CuPc theoretically contains no oxygen. The detected oxygen content: O ≈ 10.75 atom% (12.70 mass%) is entirely due to the introduction of: peripheral carboxyl (–COOH) groups, possible C=O and C–O– bonding environments. When atomic percentages are normalized, the values of Cu, N, and C closely match the CuPc framework, while oxygen appears as an additional component (~5 atoms per macrocycle).

This corresponds to a molecular composition close to:

$$C_{32-33}N_8O_5Cu$$

Given that each carboxyl group contains two oxygen atoms, this indicates: ≈ 2–3 carboxyl groups per CuPc molecule. Therefore, the pigment may be described as partially di- or tri-carboxylated CuPc–COOH. The significant increase in oxygen content provides clear elemental proof that carboxylation was successful.

Stability of the Copper Coordination Center. Copper content was measured as 6.87 mass% (1.46 atom%). Although slightly lower than the theoretical CuPc value, this is expected due to: EDX surface sensitivity, density differences, ZAF correction factors.

Nevertheless, the Cu peaks in the XRF/EDX spectrum (both L and K series) are intense and clearly defined, confirming that: the Cu^{2+}–N_4 coordination center remains intact, no reduction or oxidation of Cu^{2+} occurred, no removal or substitution of the metal center took place, the pigment remains a true metal–phthalocyanine complex. Hence, the modification did not disrupt the metal–ligand core.

Conclusion. The C/N ratio closely matches theoretical CuPc, proving the phthalocyanine macrocycle is preserved. The strong oxygen signal (≈10–11 atom%) confirms the successful introduction of –COOH groups. Normalized element ratios indicate an approximate empirical formula of $C_{32-33}N_8O_5Cu$, corresponding to 2–3 carboxyl groups per macrocycle. Copper remains in its correct coordination environment, confirming that the Cu^{2+}–N_4 center is structurally stable. The absence of impurities demonstrates the high purity of the synthesized pigment and the effectiveness of the reaction and purification steps. Therefore, EDX/XRF analysis provides strong evidence that the modified copper phthalocyanine pigment is a high-purity, successfully carboxylated CuPc–COOH material with preserved macrocyclic and metal–ligand structure, suitable for advanced polymeric, optical, and functional materials applications.

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