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EFFECT OF INITIAL REAGENT RATIOS AND TEMPERATURE ON THE YIELD IN THE SYNTHESIS OF COPPER–COBALT-CONTAINING PHTHALOCYANINE COMPOUNDS: FTIR SPECTRAL ANALYSIS OF THE OBTAINED PRODUCT

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Abstract

This article investigates the effect of initial reagent ratios and temperature on the synthesis of a bimetallic copper–cobalt phthalocyanine (Cu–Pc–Co) compound. The synthesis process was carried out within the temperature range of 180–240 °C using different molar ratios of phthalic anhydride (FA), urea (Kar), $CuCl_2$, and $CoCl_2$. The results showed that both temperature and the ratio of initial reagents significantly influence the product yield. In particular, the maximum yield of 87,1% was achieved at an FA: Kar: $CuCl_2$: $CoCl_2$ ratio of 1:6,0:1:2 and a temperature of 220 °C. Based on graphical analysis, the optimal reaction conditions were determined. These findings demonstrate that controlling synthesis parameters plays an important role in improving the efficiency of bimetallic phthalocyanine production. Infrared (IR) spectral analysis of the synthesized copper–cobalt phthalocyanine compound confirmed the presence of characteristic phthalocyanine ring bonds (–C–N–C–), as well as metal–nitrogen coordination bonds (Cu–N and Co–N).

Keywords: *Phthalocyanine, phthalic anhydride, urea, reaction yield, temperature, $CuCl_2$, $CoCl_2$, bimetallic, IR spectrum, stretching vibration, bending vibration*

Introduction

Phthalocyanines (Pc) belong to a class of tetrapyrrolic macrocyclic compounds characterized by a delocalized 18 π-electron system, in which all atoms of the molecule are arranged in a single geometric plane. Due to their electroactivity, high thermal stability, and resistance to light, phthalocyanine derivatives are widely used in the production of dyes, sensors, catalysts, solar cells, and anticorrosion coatings. Among metal-containing phthalocyanines, copper phthalocyanine (CuPc) and cobalt phthalocyanine (CoPc) are of particular importance (Fazlı H. et al. 2023; BaŞ, Hüseyin, Nuran Kahrİman, and Zekeriya BiyiklioĞlu. 2020; Gokce, C., Aslan, Y., Durmuş, M., 2015).

Bimetallic phthalocyanines, containing two different metal ions within their structure, exhibit enhanced electronic and physicochemical properties and are capable of functioning effectively even under complex conditions (Author(s) Unknown. 20220). In recent years, pigments based on bimetallic phthalocyanines have attracted increasing interest among researchers due to their high thermal and chemical stability, intense coloration, and efficient catalytic properties (Maqbaş, H., Kahriman, N., Bıyıklıoğlu, Z., 2020; Author(s) Unknown. 2022). In this study, phthalocyanine complexes containing copper and manganese ions were synthesized. The influence of different ratios of initial reagents and temperature on the reaction yield was investigated. It was found that a higher product yield was achieved at an initial reagent ratio of 3:3:0.5:0.5 and a reaction temperature of 240 °C. However, when the temperature was increased to 260 °C, a decrease in the reaction yield was observed (Nazarov N. I., Fayziyev J. B., Beknazarov H. S., Mirzoyeva G. A., 2025). Copper phthalocyanine and nickel phthalocyanine were synthesized, and the IR spectra of the obtained compounds were analyzed. The analysis revealed characteristic absorption bands corresponding to the bonds between atoms forming the phthalocyanine molecule: for copper phthalocyanine, absorption bands were observed at 3047 cm^{-1} (aromatic –CH), 1609 cm^{-1} (–C=C–), 1504 cm^{-1} (–N=), and 1408, 1329 cm^{-1} (–C=C–N–); for nickel phthalocyanine, bands appeared at 3059 cm^{-1} (aromatic –CH), 1620 cm^{-1} (–C=C–), 1488 cm^{-1} (–N=), and 1380, 1301 cm^{-1} (–C=C–N–) (Aleksanyan K. G. i dr., 2018). Although data on the synthesis and spectroscopic properties of bimetallic phthalocyanines have been reported, the optimal conditions for their synthesis, including the ratio of initial reagents and the effect of reaction temperature on product yield, have not been fully investigated (Zengin, H., Kantekin, H., 2011).

Materials and Methods

Phthalic anhydride, urea, $CuCl_2{\cdot}2H_2O$, $CoCl_2{\cdot}6H_2O$, and 0,05 g of catalyst were weighed in the required proportions, and the synthesis of phthalocyanines was carried out. The data on the effect of the initial reagent ratios and temperature on the yield during the synthesis of copper–cobalt ion-containing bimetallic phthalocyanine complexes are presented in Table 1.

Table 1. *Effect of FA: Kar: $CuCl_2$: $CoCl_2$ ratio and temperature on the yield of phthalocyanine*

No.	FA: Kar: $CuCl_2$: $CoCl_2$	T, °C	ω,%	No.	FA: Kar: $CuCl_2$: $CoCl_2$	T, °C	ω,%
1.	1: 4,0: 1: 2	180	25,1	9.	1: 6,0: 1: 2	180	67,9
2.		200	28,2	10.		200	80,2
3.		220	31,5	11.		220	87,1
4.		240	36,9	12.		240	81,2
5.	1: 5,3: 1: 2	180	46,2	13.	1: 7: 1: 2	180	64,5
6.		200	59,8	14.		200	75,9
7.		220	64,3	15.		220	79,2
8.		240	54,5	16.		240	72,6

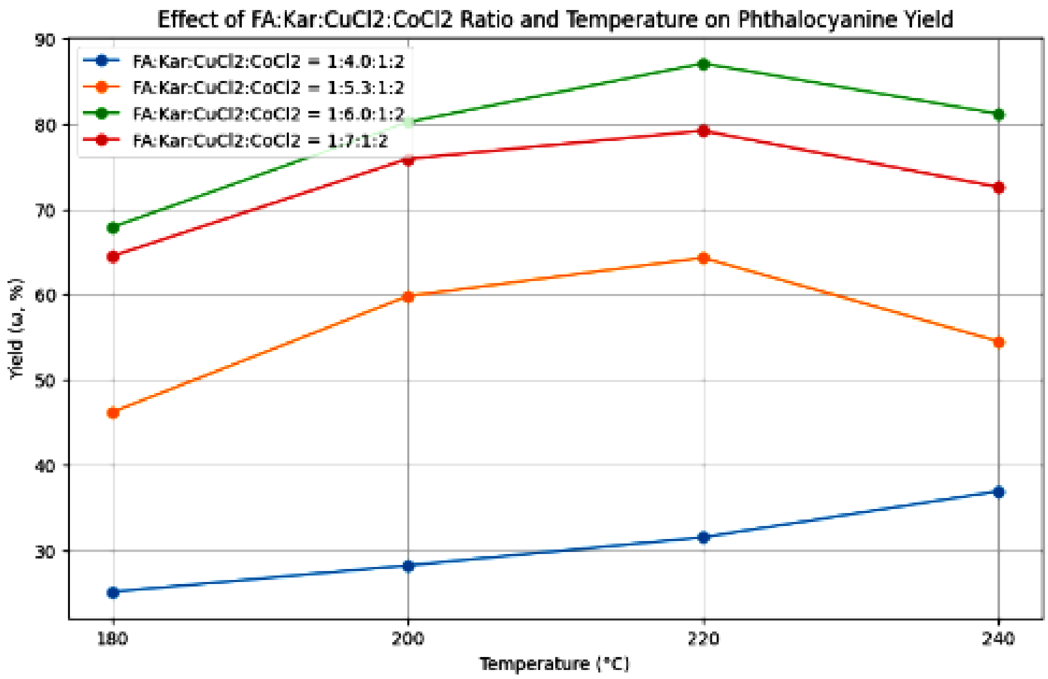
Effect of Initial Reagent Ratios and Temperature on the Yield of Phthalocyanine in the Synthesis of Copper–Cobalt-Containing Phthalocyanine

The table presented above shows the results of the analysis of the relationship between temperature (T, °C) and product yield (ω, %) in the synthesis of bimetallic phthalocyanine in the presence of copper(II) chloride and cobalt(II) chloride. In this analysis, the synthesis results obtained using different ratios of FA: Kar: $CuCl_2$: $CoCl_2$ (1:4.0:1:2; 1:5.3:1:2; 1:6.0:1:2; 1:7:1:2) were compared. It was observed that an increase in the amounts of phthalic anhydride and urea leads to an increase in product yield. In all cases, the yield increased as the reaction temperature rose from 180 °C to 220 °C. However, at 240 °C, a decreasing trend in product yield was observed, which may indicate a reduction in product mass or the occurrence of thermal degradation at higher temperatures. When the initial reagent ratios were 1:6,0:1:2 and 1:7:1:2, and the reaction was carried out at 220 °C, the highest product yields (87,1% and 79,2%, respectively) were obtained. This suggests that an optimal balance between phthalic anhydride and urea plays a crucial role in achieving maximum yield.

The presence of metal salts is essential for phthalocyanine synthesis. The optimal temperature range for the reaction was found to be 180–220 °C, while temperatures above 240 °C resulted in a decrease in product yield.

The diagram above illustrates the effect of FA: Kar: $CuCl_2$: $CoCl_2$ ratios on the yield (ω, %) of bimetallic phthalocyanine complexes as a function of temperature. The obtained results indicate that both the composition of the reaction medium and temperature significantly influence the formation of the macrocyclic ring.

Figure 1. *Graphical Representation of the Effect of Initial Reagent Ratios and Temperature on the Yield of Copper–Cobalt-Containing Phthalocyanine Complex*



The diagram above illustrates the effect of FA: Kar: $CuCl_2$: $CoCl_2$ ratios on the yield (ω, %) of bimetallic phthalocyanine complexes as a function of temperature. The obtained results indicate that both the composition of the reaction medium and temperature significantly influence the formation process of the macrocyclic ring.

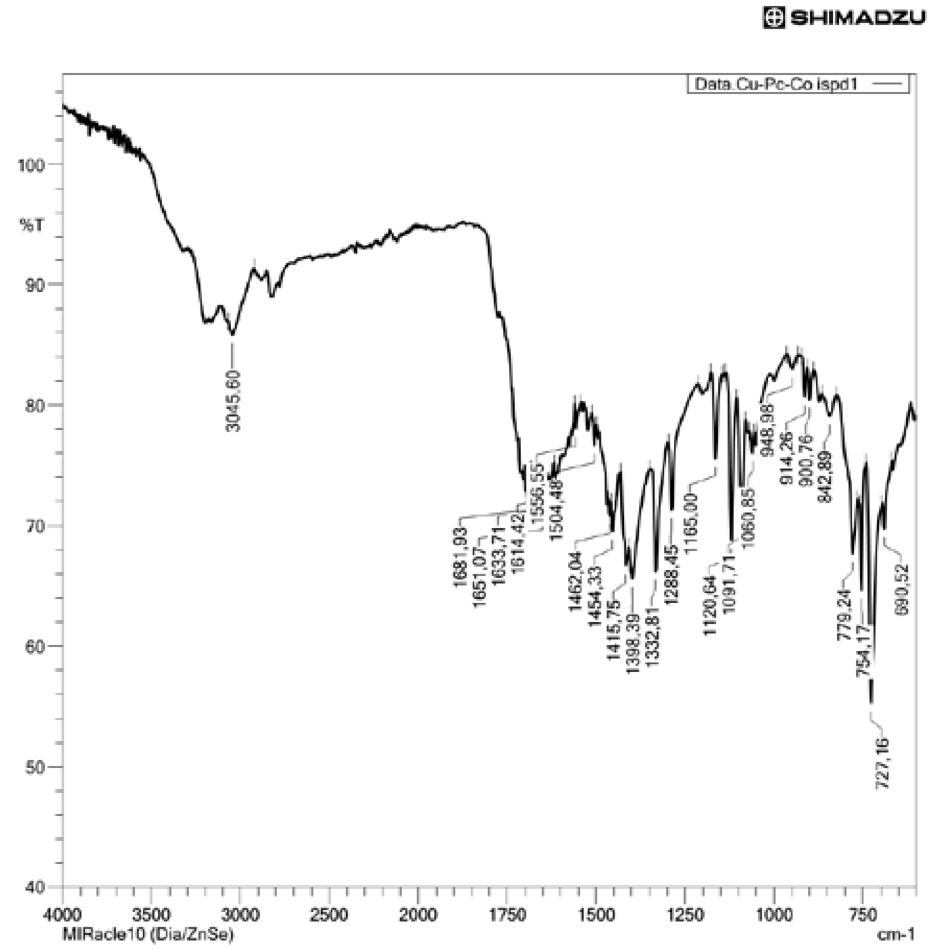
According to the results, at initial reagent ratios of 1:6,0:1:2 and 1:7.0:1:2, the maximum reaction yields of 87.1% and 79.2%, respectively, were achieved at 220 °C. This suggests that, at 220 °C, the cyclotetramerization process proceeds under kinetically and thermodynamically optimal conditions. In particular, at the 1:6,0:1:2 ratio, the sufficient amount of urea ensures complete conversion of phthalonitrile intermediate products and promotes coordination of metal ions (Cu^{2+} and Co^{2+}) with the macrocyclic ligand center.

When the temperature was increased to 240 °C, a decrease in product yield was observed for all studied ratios. This may be attributed to partial thermal degradation of the macrocyclic system, activation of side reactions, and decomposition of intermediate compounds. Although the process becomes kinetically faster at 240 °C, the stability of the product decreases, leading to reduced overall selectivity.

A similar trend was observed for the ratios 1:4.0:1:2 and 1:5,3:1:2; however, the product yields in these cases were significantly lower. Based on the obtained results, it can be concluded that the optimal conditions for the synthesis of copper–cobalt-containing bimetallic phthalocyanine complexes are an initial reagent ratio of 1:6,0:1:2 and a temperature of 220 °C, at which the yield reaches 87,1%. These conditions can therefore be considered optimal for the synthesis of Cu–Pc–Co complexes.

The FTIR spectrum of the synthesized copper–cobalt phthalocyanine (Cu–Pc–Co) complex was recorded in the range of 4000–600 cm^{-1}. The observed absorption bands correspond to the characteristic stretching and deformation vibrations of the phthalocyanine ring (Figure 2).

Figure 2. *FTIR Spectrum of Copper–Cobalt-Containing Phthalocyanine*



The absorption band at 3045 cm^{-1} corresponds to the C–H bonds of the aromatic ring. Since no vibrations characteristic of aliphatic groups were observed in the phthalocyanine molecule, these signals can be attributed solely to the aromatic ring of phthalocyanine.

The peaks in the range of 1681–1614 cm^{-1} correspond to the stretching vibrations of the C=N bonds in the phthalocyanine molecule, as well as the C=C bonds of the aromatic ring. The slight shift of these absorption frequencies toward higher wavenumbers indicates the formation of coordination bonds with the

Cu^{2+} and Co^{2+} ions. In metal-free phthalocyanine, these absorption peaks typically occur around 1620–1600 cm^{-1}. Upon formation of the metal complex, the peaks shift to higher frequencies, confirming coordination interactions between the metal and nitrogen atoms in the phthalocyanine macrocycle.

The symmetric and asymmetric absorption bands in the range of 1556–1504 cm^{-1} correspond to the C=C and C=N bonds within the phthalocyanine ring. Absorption bands at 1462–1398 cm^{-1} are associated with the stretching vibrations of C–C bonds and deformation vibrations of C–N bonds in the aromatic ring, confirming the formation of the phthalocyanine macrocycle.

The band at 1332–1288 cm^{-1} corresponds to deformation vibrations of the C–N bonds, while the absorption region at 1165–1060 cm^{-1} is related to vibrations of –C–H and C–N–C bonds within the phthalocyanine ring. Peaks in the range 948–842 cm^{-1} correspond to –C–H bonds and metal–nitrogen coordination interactions. In particular, the 900–850 cm^{-1} region indicates the formation of Cu–N and Co–N bonds. Finally, the absorption bands at 754–690 cm^{-1} correspond to deformation vibrations of the M–N (Cu–N, Co–N) bonds in the phthalocyanine complex, confirming the successful formation of metallophthalocyanines.

Conclusion

In this study, the synthesis of bimetallic copper–manganese phthalocyanines was successfully carried out, and the optimal conditions for the synthesis were determined. It was found that, at an initial reagent ratio of 1:6:1:2 and a reaction temperature of 220 °C, the yield of the resulting phthalocyanine reached 87,1%. These results indicate that these conditions are optimal for the synthesis of copper–cobalt-containing bimetallic phthalocyanines.

FTIR spectral analysis of the obtained compound showed that the main vibration bands in the 1300–1500 cm^{-1} region, corresponding to C–N and C=N bonds, are associated with the aromatic ring of phthalocyanine. Shifts in these bands, along with absorption in the 690–640 cm^{-1} region, confirm the coordination of the central metal ions with the ligand and the formation of the metallophthalocyanine complex.

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