



## Synthesis of tetrakis (4-(2-phenylprop-2-yl) phenoxy) substituted phthalocyanines using a new practical method

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### ABSTRACT

In this study, zinc, cobalt and nickel phthalocyanine compounds bearing the 4-(2-phenylprop-2-yl)phenoxy substituent were prepared. Here, a new practical method for synthesis was used. This method significantly shortens the synthesis time. This method is very effective for the reaction to take place in a short time and at a lower temperature. Additionally, these compounds were examined for their aggregation and photodegradation properties. The aggregation was studied in concentration range of  $1 \times 10^{-5}$ - $1 \times 10^{-6}$  M in tetrahydrofuran (THF). The photodegradation properties of phthalocyanine complexes under light irradiation were also investigated in chloroform ( $\text{CHCl}_3$ ).

**Keywords:** Phthalocyanine, synthesis, method, aggregation.

### Yeni pratik bir yöntem kullanarak tetrakis (4-(2-fenilprop-2-il) fenoksi) süstitüentli ftalosiyanınların sentezi

#### ÖZ

Bu çalışmada, 4- (2-fenilprop-2-il) fenoksi süstitüentini taşıyan çinko, kobalt ve nikel ftalosiyanın bileşikleri hazırlandı. Burada sentez için yeni pratik bir metot kullanıldı. Bu metot sentez süresini oldukça kısaltmaktadır. Bu yöntem, reaksiyonun kısa sürede ve daha düşük bir sıcaklıkta gerçekleşmesi için çok etkilidir. Ayrıca, bu bileşikler agregasyon ve fotoregradasyon özellikleri açısından incelenmiştir. Agregasyon tetrahydrofuran (THF) içinde  $1 \times 10^{-5}$ - $1 \times 10^{-6}$  M konsantrasyon aralığında incelenmiştir. Işık ışınımı altında ftalosiyanın komplekslerinin fotodegradasyon özellikleri de kloroformda ( $\text{CHCl}_3$ ) incelenmiştir.

**Anahtar Kelimeler:** Phthalocyanine, sentez, yöntem, agregasyon.

### 1. INTRODUCTION

Phthalocyanines have become an attractive topic for researchers because they find applications such as photodynamic therapy,<sup>1-2</sup> solar cells,<sup>3</sup> catalysts,<sup>4</sup> dyes,<sup>5-6</sup> photocatalysts,<sup>7</sup> gas sensor<sup>8</sup>, semiconductor materials.<sup>9</sup> It is known that one of the reasons preventing the widespread use of phthalocyanines in technological areas is aggregation.<sup>10</sup> The synthesis of non-aggregation phthalocyanines have been important for applications. This problem can be solved by attaching appropriate substituents at the peripheral positions of the phthalocyanine core.<sup>11-12</sup> Metal phthalocyanines have some advantages for use in both catalyst and other applications. It is the reason for preference in terms of

thermal stability and preparation technique.<sup>13</sup> These compounds are extremely stable in the chemical direction. This allows them to be used in many reaction environments.<sup>14</sup> The use of phthalocyanine complex as a catalyst in fuel cells has increased interest in these compounds.<sup>15-16</sup> As is known, the catalysts can be used not only in fuel cells but also in the deferring of many reactions and industrial wastes. Phthalocyanine compounds have a photocatalytic effect and can be applied to the breakdown of some organic waste dyes.<sup>17</sup> Phthalocyanine compounds can contribute to the key role in solving these problems. Phthalocyanines have a high photocatalytic performance potential compared to other photocatalytic materials.<sup>18-19</sup>

In this study, 4-(2-phenylprop-2-yl) phenoxy) phthalonitrile, which is the starting material, was synthesized for the mentioned phthalocyanine compounds. Zinc, cobalt and nickel phthalocyanine compounds were synthesized by using a more effective new method than conventional and the microwave method. Aggregation and solubility properties were investigated. The photodegradation preliminary studies were tested.

## 2. EXPERIMENTAL

### 2.1. General

ZnCl<sub>2</sub>, CoCl<sub>2</sub>, NiCl<sub>2</sub>, K<sub>2</sub>CO<sub>3</sub>, ethanol, methanol, 4-cumylphenol, acetone, tetrahydrofuran (THF), dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>), dichloroethane, dimethyl formamide (DMF) were purchased from Merck and Sigma. The solvents were purified according to standard procedure<sup>20</sup> and stored over molecular sieves (4 Å). All reactions were carried out under dry nitrogen atmosphere. Melting points were measured on an electrothermal apparatus. Electronic spectra were recorded on a Hitachi U-2900 Spectrophotometer. Routine FT-IR spectra were recorded on a Thermo Scientific FT-IR (ATR sampling accessory) spectrophotometer. <sup>1</sup>H NMR spectra were recorded on a Agilent 400 MHz spectrometer with tetramethylsilane as internal standard.

### 2.2. 4-(4-(2-phenylprop-2-yl) phenoxy) phthalonitrile (3)

A mixture of 4-cumylphenol 1 (0.6135g, 2.89 mmol) and 4-nitrophthalonitrile 2 (0.500 g, 2.89 mmol) in 25 ml dimethylformamide was stirred at room temperature under nitrogen atmosphere. After stirring for 15 min, K<sub>2</sub>CO<sub>3</sub> (2.2 g, 15.92 mmol) was added into the mixture over a period of 2 h. The reaction mixture was further stirred for 48 h at room temperature. The reaction mixture was poured into cold water (150 ml) and stirred. The jelly-like substance which was triturated with hexane. The precipitate was filtered off, washed with water to neutralize it, and dried. The product was recrystallized from ethanol. The product is soluble in diethyl ether, chloroform, acetone, dichloromethane, dichloroethane, acetonitrile, ethyl acetate, THF, DMF and DMSO. Yield; 0.5375 g (55%). Mp: 95°C. <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>, δ): 8.03, 7.68, 7.30, 7.24, 7.14, 7.06, 3.62, 2.48, 1.62, 1.04. <sup>13</sup>C NMR (400 MHz, DMSO-d<sub>6</sub>, δ): 161.49, 150.31, 148.26, 136.68, 129.10, 128.56, 126.84, 126.15, 122.90, 122.23, 120.16, 117.04, 115.12, 108.39, 42.65, 40.26, 40.06, 39.85, 39.64, 39.43, 39.22, 39.01, 30.82. FT-IR spectrum (cm<sup>-1</sup>): 3078, 3032, 2970, 2935 2870, 2233, 1589, 1562, 1485, 1446, 1404, 1276, 1249, 1207, 1172, 1083, 1014, 952, 898, 840, 702. Anal calculated for C<sub>23</sub>H<sub>18</sub>N<sub>2</sub>O: (338.40 g mol<sup>-1</sup>) C 81.63; H 5.36; N 8.28 %. Found C 81.46; H 5.30; N 8.23%.

### 2.3. 2(3), 9(10), 16(17), 23(24) – tetrakis (4-(2-phenylprop-2-yl) phenoxy) phthalocyaninato) zinc (II) (4)

4-(4-(2-phenylprop-2-yl) phenoxy) phthalonitrile 3 (0.100 g, 0.295 mmol) and ZnCl<sub>2</sub> (0.020 g) was powdered in a quartz crucible and heated in a sealed glass tube for 6 min under nitrogen at 230°C. After cooling to room temperature, the product was washed with water, ethanol and methanol. It was then purified by column chromatography on silica gel with tetrahydrofuran as the eluent. This compound is soluble acetone, tetrahydrofuran, dichloromethane, dichloroethane and dimethylformamide. Yield: 0.0392 g (37.40%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, δ): 7.74, 7.49, 7.33, 7.25, 7.23, 6.92, 6.56, 1.41. Calc. for C<sub>92</sub>H<sub>72</sub>N<sub>8</sub>O<sub>4</sub>Zn: (1418.99 g mol<sup>-1</sup>) C, 77.87; H, 5.11; N, 7.90%. Found: C, 77.79; H, 5.21; N, 7.84 %. UV-Vis (THF) λ<sub>max</sub> (log ε): 676 (5.30), 610 (4.72), 352 (5.04). IR spectrum (cm<sup>-1</sup>):3032, 2962, 2870, 1600, 1473, 1442, 1234, 1168, 1087, 948, 744.

### 2.4. 2(3), 9(10), 16(17), 23(24) – tetrakis (4-(2-phenylprop-2-yl) phenoxy) phthalocyaninato) cobalt (II) (5)

4-(4-(2-phenylprop-2-yl) phenoxy) phthalonitrile 3 (0.100 g, 0.295 mmol) and CoCl<sub>2</sub> (0.020 g) was powdered in a quartz crucible and heated in a sealed glass tube for 6 min under nitrogen at 230°C. After cooling to room temperature, the product was washed with water, ethanol and methanol. It was then purified by column chromatography on silica gel with tetrahydrofuran as the eluent. This compound is soluble acetone, tetrahydrofuran, dichloromethane, dichloroethane and dimethylformamide. Yield: 0.015 g (14.38%). Calc. for C<sub>92</sub>H<sub>72</sub> Co N<sub>8</sub>O<sub>4</sub>: (1412.54 g mol<sup>-1</sup>) C, 78.23; H, 5.14; N, 7.93 %. Found: C, 78.15; H, 5.20; N, 7.91%. UV-Vis (THF) λ<sub>max</sub> (log ε): 662 (5.25), 600 (4.73) 326 (5.15). IR spectrum (cm<sup>-1</sup>): 3032, 2962, 2870, 1604, 1504, 1469, 1442, 1441, 1238, 1165, 1118, 1095, 1066, 956.

### 2.5. 2(3), 9(10), 16(17), 23(24) – Tetrakis (4-(2-phenylprop-2-yl) phenoxy) phthalocyaninato) nickel (II) (6)

4-(4-(2-phenylprop-2-yl) phenoxy) phthalonitrile 3 (0.100 g, 0.295 mmol) and NiCl<sub>2</sub> (0.020 g) was powdered in a quartz crucible and heated in a sealed glass tube for 6 min under nitrogen at 230°C. After cooling to room temperature, the product was washed with water, ethanol and methanol. It was then purified by column chromatography on silica gel with tetrahydrofuran as the eluent. This compound is soluble acetone, tetrahydrofuran, dichloromethane, dichloroethane and dimethylformamide. Yield: 0.022 g (21.15%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, δ): 7.52, 7.36,

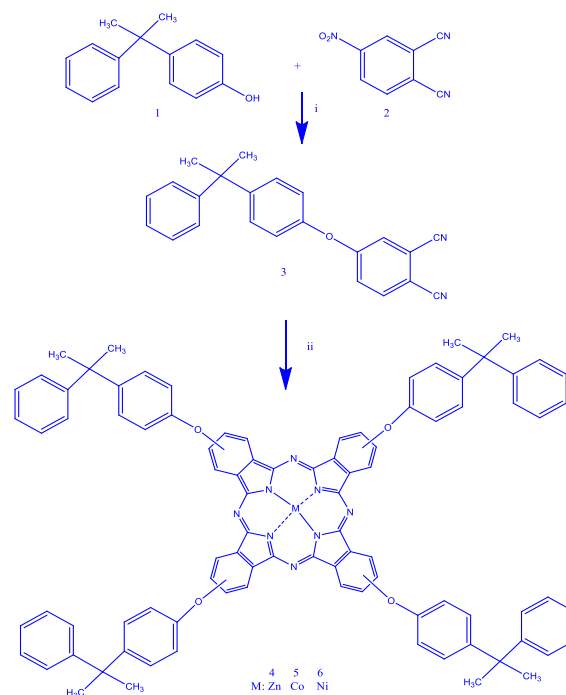
7.25, 6.98, 1.55. Calc. for  $C_{92}H_{72}N_8NiO_4$ : (1412.30 g mol<sup>-1</sup>) C, 78.24; H, 5.14; N, 7.93%. Found: C, 78.22; H, 5.10; N, 7.88%. UV-Vis (THF)  $\lambda_{max}$  (log  $\epsilon$ ): 672 (5.27), 608 (4.80), 328 (4.97). IR spectrum (cm<sup>-1</sup>): 3032, 2966, 2870, 1604, 1535, 1504, 1473, 1415, 1238, 1168, 1118, 1095, 1064, 821, 702.

### 3. RESULTS AND DISCUSSION

#### 3.1. Synthesis and characterization

The synthesis route of phthalocyanine compounds prepared in this study is shown in Scheme 1. The phthalonitrile compound was obtained by reaction of 4-cumylphenol with 4-nitrophthalonitrile in basic medium. Phthalocyanine compounds were obtained by heating with corresponding metal salts. Phthalocyanine compounds were obtained in the solvent-free medium. These reactions were completed in a very short time such as 6 minute. It was synthesized more practically than the reactions in the solvent environment.<sup>21</sup> This synthesis method is also advantageous from the synthesis made from the microwave oven. Because, the reaction was completed in a short time. This method facilitates the practical synthesis of phthalocyanines. These compounds obtained by the conventional method were tested for Langmuir-Blodgett monomolecular layer formation.<sup>22</sup> In response to the reaction times of 44 to 70 hours for the preparation of phthalocyanine complexes by these conventional synthetic methods, the same compounds were synthesized in only 6 minutes with our method. The synthesis of phthalocyanine compounds, easily and economically, provides the testing opportunity of these compounds in applications such as catalyst, sensor, photocatalytic, photodynamic therapy, dyeing.

The IR spectrum gives information about the vibration bands. This spectral data does support to structure. Compound 3 yields bands 3078, 3032 (Ar-CH<sub>3</sub>), 2970 (CH<sub>3</sub>), 2233 (CN), 1585-1589-1562 (C=C) 1249 (Ar-O-Ar). Among these bands, the 2233 (CN) band is a peak that separates this compound from phthalocyanines. The equivalent of these functional groups (Ar-CH<sub>3</sub> 3082; CH<sub>3</sub> 2976; CN 2238; Ar-O-Ar 1255; C = C 1563) was reported.<sup>22</sup> The data appear to be compatible. The observation in these compounds of the phthalocyanine is considered to be one of the most prominent features. This difference is very much in the literature. In these compounds, they give the appropriate vibration bands according to the literature.<sup>21,22</sup> The vibration bands of the compounds prepared in this study are given in the experimental part. The <sup>1</sup>H and <sup>13</sup>C NMR spectra of compound 3 are compatible with the literature values.<sup>22</sup> Except for the small shifts caused by the solvent, it is suitable. While the solvent used DMSO-d<sub>6</sub>, the previous study used CDCl<sub>3</sub>.



**Scheme 1.** Synthetic pathway of compounds (3-6). Reagents and conditions: (i) DMF, K<sub>2</sub>CO<sub>3</sub>, 48 h, rt; (ii) ZnCl<sub>2</sub>, CoCl<sub>2</sub>, NiCl<sub>2</sub>, N<sub>2</sub>, 230°C.

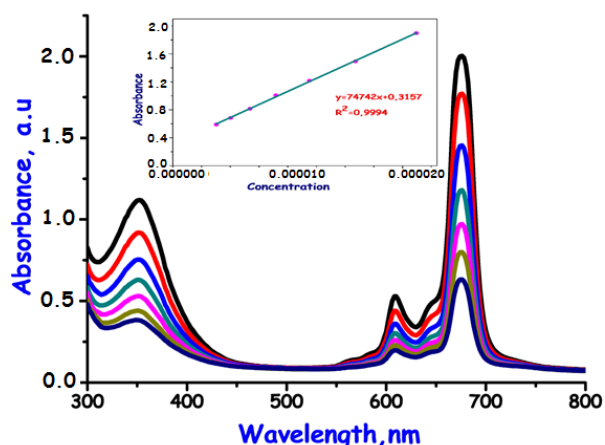
In the same way, for the NMR spectra of zinc and nickel metal phthalocyanines different solvents from the literature were used. The expected results were obtained. Structural explanation is intended to demonstrate the suitability of the method. The purpose is not to discuss the spectral data. The important thing is that the same compounds are obtained in a shorter time. For example, zinc phthalocyanine was obtained in 6 minutes at 230°C with this method, but it was 70 hours at 280°C by conventional method.<sup>22</sup> This comparison clearly demonstrates the advantage of the method.

The electronic absorption spectra of the phthalocyanine compounds obtained in this work were investigated. The absorption spectra recorded in THF give the expected B and Q bands. The electronic properties of the phthalocyanines with delocalized 18  $\pi$  electron system are explained by the fact that the Q and B bands are useful for binary applications.<sup>23</sup> It also tells you whether there is near red and whether the compound binds to the metal. Yielding Q-bands at 676, 662, and 672 nm, giving Soret bands of a typical phthalocyanine at about 352, 326 and 328 nm for compounds of 4-6 phthalocyanine.

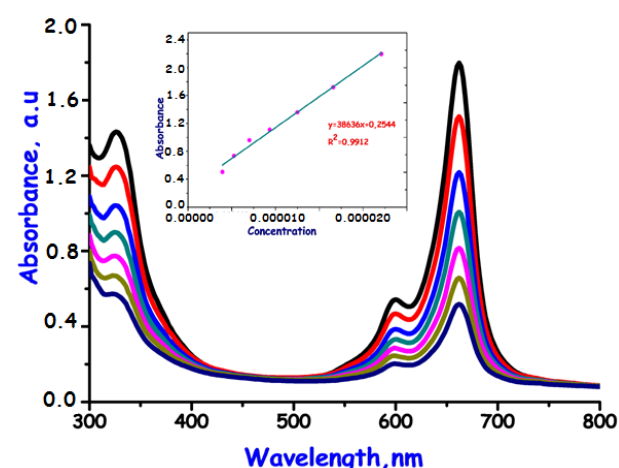
Phthalocyanine compounds show formation ability of H and J type aggregates in organic solvents, especially in aqueous solution, due to intermolecular  $\pi$ - $\pi$  interactions of electrons on the phthalocyanine rings.<sup>24</sup> H type aggregation is more common in phthalocyanine compo-

unds. Q indicates a shift in the band to blue and that the monomers come face to face. The J-type is observed to be more red-shift.

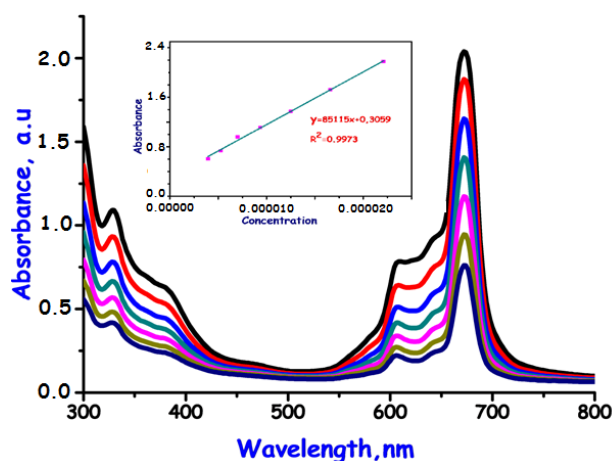
The aggregation behavior are generally displayed by changes in the UV-Vis spectra of phthalocyanines, and mostly based on the analysis of the impact of concentration on absorption spectra. Phthalocyanines (4-6) were examined in THF over a wide concentration range ( $10^{-6}$  -  $10^{-5}$  M), respectively. The working ranges of the compounds are from  $2.11 \cdot 10^{-5}$  to  $3.51 \cdot 10^{-6}$  M for compound 4, from  $2.12 \cdot 10^{-5}$  to  $3.78 \cdot 10^{-6}$  M for compound 5, from  $2.12 \cdot 10^{-5}$  to  $3.78 \cdot 10^{-6}$  M for compound 6, respectively. The aggregation studies of these compounds are given in Figures 1-3. When the shapes are examined, it is seen that the compounds are not aggregate in the study range. The Q band intensity was increased with increasing concentrations of phthalocyanines and no new bands were observed due to the aggregated species.



**Figure 1.** Aggregation study compound 4 in THF ( $2.11 \cdot 10^{-5}$ ,  $1.58 \cdot 10^{-5}$ ,  $1.19 \cdot 10^{-5}$ ,  $8.92 \cdot 10^{-6}$ ,  $6.25 \cdot 10^{-6}$ ,  $4.68 \cdot 10^{-6}$ ,  $3.51 \cdot 10^{-6}$  M concentration range).



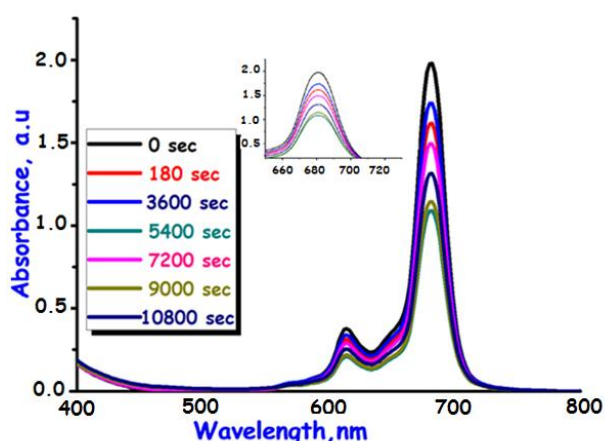
**Figure 2.** Aggregation study compound 5 in THF ( $2.12 \cdot 10^{-5}$ ,  $1.59 \cdot 10^{-5}$ ,  $1.19 \cdot 10^{-5}$ ,  $8.97 \cdot 10^{-6}$ ,  $6.72 \cdot 10^{-6}$ ,  $5.04 \cdot 10^{-6}$ ,  $3.78 \cdot 10^{-6}$  M concentration range).



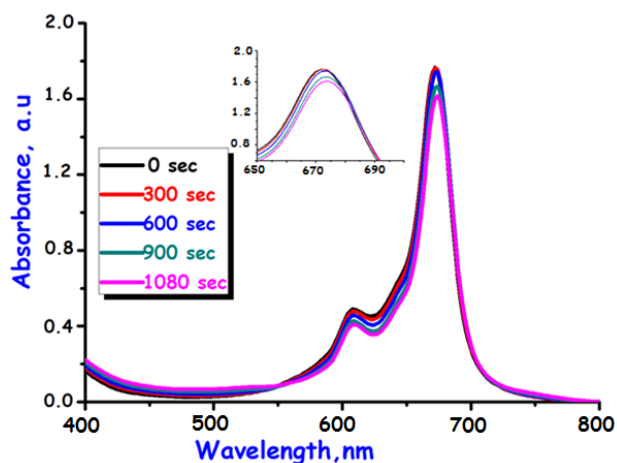
**Figure 3.** Aggregation study compound 6 in THF ( $2.12 \cdot 10^{-5}$ ,  $1.59 \cdot 10^{-5}$ ,  $1.19 \cdot 10^{-5}$ ,  $8.97 \cdot 10^{-6}$ ,  $6.72 \cdot 10^{-6}$ ,  $5.04 \cdot 10^{-6}$ ,  $3.78 \cdot 10^{-6}$  M concentration range).

Looking at the applications of phthalocyanines, it seems to be important to determine the degradation by light. Because, phthalocyanine compounds can be used as paint on many surfaces. The degradation with light effect gives information about the stability and suitability of the paint. This allows compounds of phthalocyanine to be used in many applications. However, it can be a disadvantage in terms of environmental pollution. It is also advantageous in making paint and pigment which is resistant to irradiation.<sup>25</sup> Photodegradation studies of phthalocyanine complexes have been investigated as the Q absorbance band versus time. Photodegradation studies are useful for testing these properties of substances. The same method used in the previous study was used for photocatalytic testing.<sup>17</sup> Xenon lamp (XHA-150W) water cooled photoreactor was used in this study. The photodegradation spectral changes observed by irradiation in the  $\text{CHCl}_3$  solvent are shown in Figures 4-6. It can be said that these phthalocyanines are highly resistant to photochemical degradation. The photocatalytic degradation effect of these phthalocyanine compounds on substances such as methylene blue can be investigated as another study.

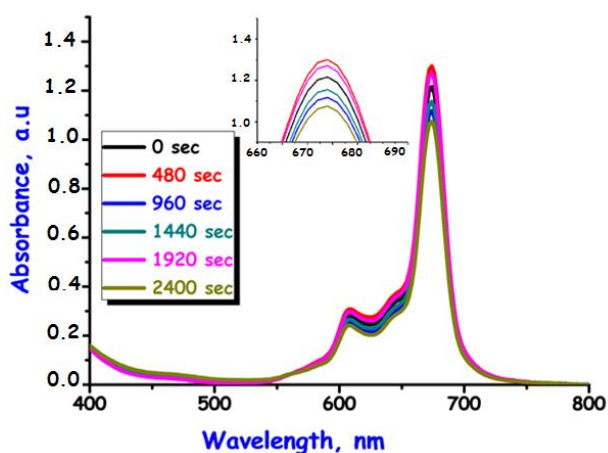
Although phthalocyanine derivatives are widely synthesized, the solubility problem is not completely exceeded.<sup>26</sup> Therefore, easy dissolving phthalocyanines are attracted by the researchers.<sup>27</sup> The method used and the solubility in a large number of solvents are important advantages. The solubility tests of the prepared phthalocyanine compounds have been found to be readily solved in organic solvents such as acetone, tetrahydrofuran, dichloromethane, dichloroethane and dimethylformamide.



**Figure 4.** The photodegradation of Zinc (II) Pc (4) in  $\text{CHCl}_3$  showing the disappearance of the Q band. Concentration =  $1.0 \times 10^{-5}$  M (Inset: plot of Q band absorbance vs time-sec).



**Figure 5.** The photodegradation of Cobalt (II) Pc (5) in  $\text{CHCl}_3$  showing the disappearance of the Q band. Concentration =  $1.0 \times 10^{-5}$  M (Inset: plot of Q band absorbance vs time-sec).



**Figure 6.** The photodegradation of Nickel (II) Pc (6) in  $\text{CHCl}_3$  showing the disappearance of the Q band. Concentration =  $1.0 \times 10^{-5}$  M (Inset: plot of Q band absorbance vs time-sec).

#### 4. CONCLUSIONS

In this study, metallo phthalocyanines compounds bearing (4-(2-phenylprop-2-yl) phenoxy) groups in the peripheral position were prepared by an effective method. These phthalocyanines compounds are readily soluble in solvents such as acetone, tetrahydrofuran, dichloromethane, dichloroethane, dimethyl formamide. These compounds show very good solubility compared to many phthalocyanine derivatives. The solubility simplifies the research for different application. The aggregation and photodegradation properties of the compounds were examined at certain concentrations and solvents. The compounds did not show any aggregation in the studied concentration range. This method shows that many phthalocyanine compounds can be synthesized practically. This method provides advantages such as time and temperature, not solvent usage.

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#### Conflict of interests




Authors declare that there is no a conflict of interest with any person, institute, company, etc.

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