MANAS Journal of Engineering
ISSN 1694-7398 | e-ISSN 1694-7398

Volume 10, Issue 1, (2022) Pages 81-87
https://doi.org/10.51354/mjen. 1076925

Novel indium(III) acetate phthalocyanine: synthesis and spectroscopic properties

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ABSTRACT

A new phthalocyanine 1 was synthesized, containing 2-aminophenoxy groups as tetra from its peripheral positions and indium (III) acetate in its cavity. The structure of **1** was illuminated by elemental analysis, FTIR, UV-*vis* and fluorescence spectroscopy techniques It is well soluble in solvents such as polar and nonpolar solvent types. Its aggregation properties have been studied both in the solvents mentioned above and in Dimethyl sulfoxide at different concentrations. Its aggregation properties were examined in these solvents as well as in Dimethyl sulfoxide at certain different concentration ranges. With these determined spectroscopic properties, it can be a candidate for use in various technological applications.

ARTICLE INFO

Research article

Received: 21.02.2022
Accepted: 25.04.2022

Keywords:
Phthalocyanine, indium(III) acetate, 2-aminophenol, spectroscopic, fluorescence

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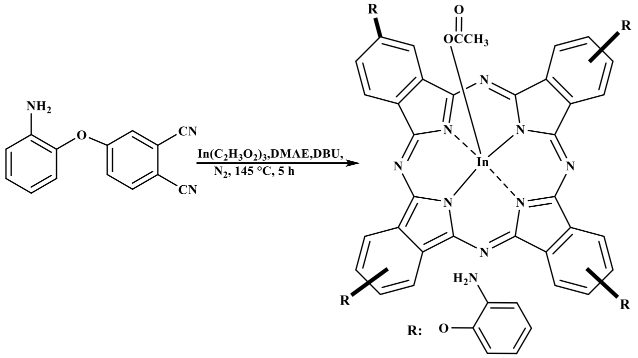
1 Introduction

Phthalocyanines (Pcs) are synthetic molecules that are similar in structure to natural porphyrins but are not natural [1,2]. Pcs, which are formed by the cyclization of four iminoisoindoline groups over azo bridges, are generally with or without metal depending on the metal substitution in the space on the ring [1, 2]. According to the variety of metal bound to Pcs, their properties and usage areas vary [3,4]. By substituting different functional groups on the peripheral or non-peripheral positions of Pc, such as tetra or octa, as well as the presence of different metal species containing ligands in the axial position in its cavity, their aggregation can be prevented by increasing their solubility [5-9]. Pcs, which have high thermal and chemical stability, and light stability, have extraordinary physicochemical properties [5-9]. Therefore, they have different advanced technological application areas. The main areas of use are electrochemical applications [10, 11], gas sensors [12, 13], pigments and dye industry [14, 15], photovoltaic materials [16, 17], liquid crystals [18, 19], organic field transistors [20-22], non-linear optical materials [23-25], light-emitting devices [26, 27], optical switching and limiting devices [28-30], solar cells [31, 32] and as a photosensitizer for photodynamic therapy (PDT) [33-41]. The type of metal in the metal-containing phthalocyanine (MPc) cavity, changes MPc's planar geometry, solubility, thermal and optical stability, as well as electrical and chemical properties [42-50]. 2 -Aminophenol is a reducing agent, a useful reagent for the synthesis of dyes and heterocyclic compounds [51]. In this research work, a new phthalocyanine complex was designed, which can be used in different technological application areas, is well soluble in different solvent types, contains monomeric species, has fluorescent properties, and contains heavy metal ions. For this purpose, 2 (3), 9 (10), 16 (17), 23 (24)-tetra-(2-aminophenoxy) phthalocyaninato indium(III) acetate [In(OAc)Pc] **1** was synthesized and its properties were determined by widely known spectroscopic techniques (Scheme 1).



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Scheme 1. *Synthesis of peripheral-tetra-2-aminophenoxy substituted indium(III) acetate phthalocyanine [In(OAc)Pc] 1*

2 Experimental section

2.1. Material and methods

Indium(III) acetate metal salt and 4-(2-aminophenoxy)phthalonitrile compound were purchased from Sigma-Aldrich and ET Co.,Ltd., respectively. The solvents were purified, dried, and retained in molecular sieves, 4Å. All reactions were carried out under a dry nitrogen atmosphere. It was purified by successive washings with various solvents using a Soxhlet apparatus. The purity of the product was tested by thin layer chromatography after washing with each solvent and drying. FTIR and electronic spectra were recorded on a Shimadzu FTIR-8300 (ATR) and a Shimadzu UV–1601 spectrophotometer, respectively. Elemental analysis using LECO CHN 932 was carried out by TUBITAK Ankara Instrumental Analysis Laboratory. Fluorescent excitation and emission spectra were recorded on a Varian Eclipse spectrofluorometer using 1 cm path length cuvettes at 25 °C.

2.2. Synthesis of the In(OAc)Pc 1

4-(2-aminophenoxy)phthalonitrile (0.15 g, 0.64 mmol), indium(III) acetate (0.09 g, 0.32 mmol) and and catalytic amount of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) in 1.50 mL of 2-Dimethylaminoethanol (DMAE) was heated with stirring at 145 °C in a sealed glass tube for 5 hours under nitrogen atmosphere. The resulting suspensions were cooled to room temperature and then poured into 25 ml of ethanol. The precipitates were filtered and washed sequentially with water, ethyl acetate, hexane, acetone, dichloromethane (DCM), and diethyl ether and dried over phosphorus pentoxide in a vacuum desiccator. Mp>300° C. Yield: 54.56 mg (27.85%). Calculated for $C_{58}H_{39}InN_{12}O_6$. Elemental Analysis: C, 62.49; H, 3.53; N, 15.08%; found C, 64.65%; H, 3.18; N, 14.87%. FTIR (ATR) λ_{max}/cm^{-1}: 670-1058(C–H str.), 10165-1201(C–N str.), 1241-1371(C–O str.), 1469-1572 (C–H bend.), 1608 (C=N- str.),1722-1773(C=C str.), 3082(=C–H str.), 3200 (-NH_2 str.). UV–*vis* (DMSO, 1.00 ×10^{-5} M): λ_{max}(nm), (log ε): 354 (4.83), 614 (4.43), 656 (4.50), 684 (5.08).

3 Results and discussion

3.1. Synthesis and characterization

The In(OAc)Pc **1** was synthesized by cyclotetramerization reaction of 4-(2-amino phenoxy)phthalonitrile presence of DMAE as the solvent, DBU as the catalyzer and anhydrous indium(III) acetate under the nitrogen atmosphere (Scheme 1). It was isolated as dark green solids in 27.85% yield. It was washed sequentially with each of the solvents of water, ethyl acetate, hexane, acetone, DCM and diethyl ether. Its structure was illuminated by elemental analysis, FTIR, UV-*vis*, fluorescence spectroscopic techniques. The elemental compositions of C, H, and N obtained from its elemental analysis are in agreement with the proposed structure. The fact that the moderately sharp -C≡N stretching vibration band at 2233 cm^{-1} belonging to 4-(2-aminophenoxy) phthalonitrile does not appear in the FTIR spectrum belonging to the In(OAc)Pc **1** confirms the proposed structure (Fig. 1). In its FTIR spectrum, respectively, at 3200 cm^{-1} moderate NH stretching, at 3082 cm^{-1} weak aromatic CH stretching, at 1722-1773 cm^{-1} moderately sharp aromatic -C=C- stretching, at 1608 cm^{-1} weak aromatic C=N- stretching, at 1469-1572 cm^{-1} aromatic C–H bending, and at 1241-1371 cm^{-1}, moderate Ar–O-Ar stretching characteristic vibrational bands were observed, respectively (Fig. 1).

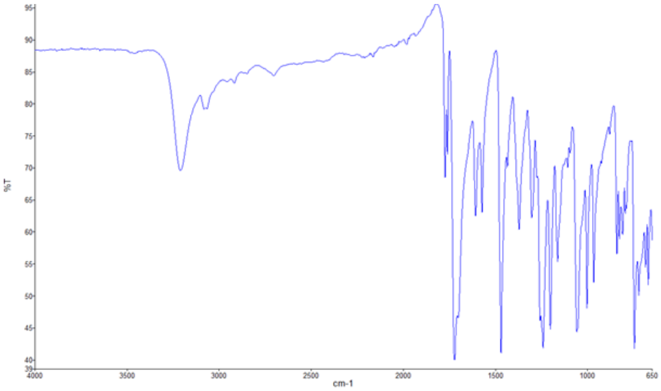

Figure 1. *FTIR spectrum of the In(OAc)Pc* ***1****.*

In its UV-*vis* absorption spectrum, a monomeric behavior was observed as evidenced by a single (narrow) Q band at 1.00 × 10^{-5} M solution in DMSO. This is characteristic for metal-containing Pcs [47-50]. It demonstrated a single Q band due to the π-π* transition at 684 nm, with a high molar extinction coefficient of 5.08 L.mol^{-1} cm^{-1} (Fig. 2). It demonstrated another characteristic band, the Soret band (B band), at 354 nm due to n-π* electronic transitions (Fig. 2). It showed 3 nm blue-shifted Q band with respect to unsubstituted indium(III) acetate phthalocyanine [49, 50]. This is the type of substituent on the phthalocyanine skeleton, which is related to the presence of 2-aminophenoxy groups. The data and analyzes from these spectroscopic techniques for the In(OAc)Pc **1** are in agreement with the proposed structure.

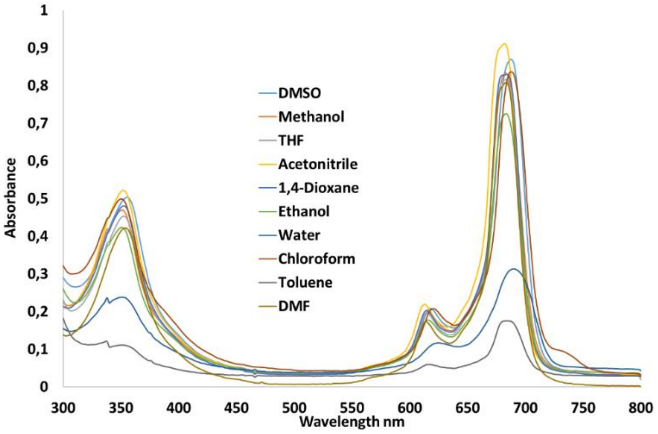

Figure 2. *UV-vis absorption spectra of the In(OAc)Pc **1** in different solvents. Concentration=* $\sim 1\times 10^{-5}$ *M.*

3.2. Aggregation studies

Aggregation is known by the incorporation of monomers, dimers, and rings into higher-order complexes in different types of solvent. In this study, the aggregation behavior of the In(OAc)Pc **1** was investigated in different solvents It can dissolve well without aggregation in different kinds of solvents such as chloroform ($CHCl_3$), toluene and 1,4-dioxane as non-polar solvents and ethanol (EtOH and water as polar protic solvents, as well as tetrahydrofuran, N,N-dimethylformamide (DMF), dimethyl sulfoxide (DMSO) and acetonitrile as polar aprotic solvents (Fig. 2). In addition, its aggregation behavior was determined by investigating whether it obeys the Lambert-Beer law in DMSO at certain concentration ranges at room temperature. Ten solutions of its in DMSO with decreasing concentrations from 1.00 $\times 10^{-6}$ M to 1.00$\times 10^{-6}$ M were prepared separately. The UV-*vis* spectra in Figure 3 show its aggregation behavior in DMSO. A linear regression analysis was performed between the intensity of its Q-band and its concentration. It has been determined that it obeys the Lambert-Beer law and contains monomeric species. It was also observed that the absorbance of the Q-band also increased due to the increase in its concentration and there were no new (normally blue or red shifted) bands. These obtained results confirm that it does not tend to aggregate and does not include aggregated species.

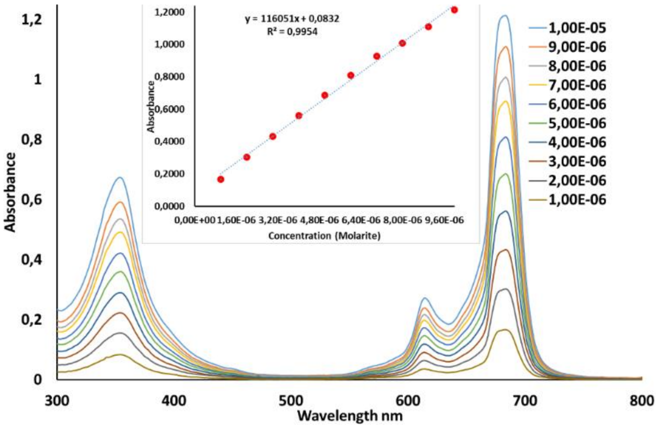

Figure 3. *Aggregation behavior of the In(OAc)Pc **1** in DMSO at different concentrations. (Inset: plot of absorbance vs. concentration).*

3.3. Fluorescence spectra

Figure 4 displays fluorescence excitation and emission spectra of the In(OAc)Pc **1** in DMSO. Its bands with maximum intensity in the fluorescence spectrum were monitored at 690 nm for excitation and 702 nm for emission, respectively. It has a Stokes shift of 18 nm and is longer than that of unsubstituted In(OAc)Pc [27]. Its fluorescent emission in DMSO is low intensity and characteristic due to the heavy atom effect of indium metal [44-50].

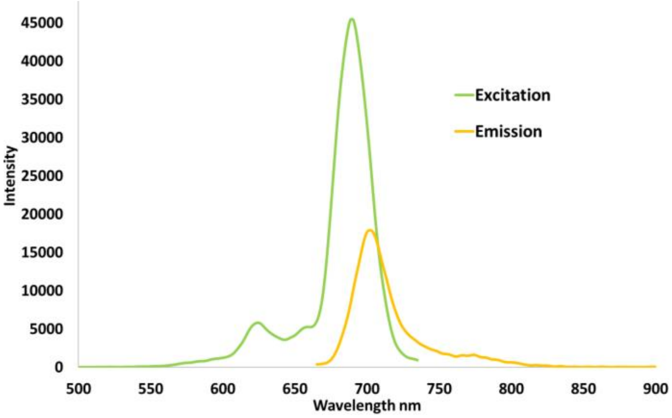

Figure 4. *Excitation, and emission spectra of the In(OAc)Pc **1** in DMSO. Excitation wavelength= 655 nm.*

4 Conclusions

In this research study, a novel phthalocyanine, containing heavy metal ion, which is 2(3),9(10),16(17),23(24)-tetra-(2-aminophenoxy)phthalocyaninato indium(III) acetate was synthesized. Its structure was illuminated and confirmed using elemental analysis, FTIR, UV-*vis*, and fluorescence spectroscopic techniques. It dissolves well in polar protic, polar aprotic and non-polar solvents, contains mainly monomeric species, and can absorb at long wavelengths with its high molar extinction coefficient. It showed low fluorescence emission due to the presence of indium metal ion as heavy metal in its cavity and with large atomic radius. It can be used in various technological applications because it dissolves very well in different solvent types, has monomeric species and sufficient fluorescent properties, and can be useful for systems involving changes in forms similar to the studied complex.

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