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Synthesis and photodiode properties of chalcone substituted metallo-phthalocyanine

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ABSTRACT

In this study, we aimed to investigate the photodiode properties of the chalcone substituted-phthalocyanine complex due to the important physical properties of the phthalocyanine and chalcone compounds. For this purpose, in the first step, a heterocyclic chalcone compound **(HC)** bearing a pyridine and hydroxyl group was obtained from the reaction of 4′-hydroxyacetophenone with pyridine-3-aldehyde. In the second step, the chalcone-substituted phthalonitrile compound **(HCA)** was obtained by interacted 4-nitroftalonitrile with the compound **HC** in the presence of K_2CO_3 in DMF in an argon atmosphere. The new chalcone substituted metallo-phthalocyanine **(HCA-Co)** was synthesized from the reaction of compound **HCA** with Co (II) acetate according to heating of solid phase method under an inert atmosphere. The structure of **HC** and **HCA-Co** were fully characterized by using ^{1}H, and ^{13}C-APT NMR, FT-IR, elemental analysis, UV–Vis spectroscopy. The **HCP-Co** thin-film planar heterojunction diode was prepared. The organic layer was deposited onto the p-Si substrate to obtain an Al/p-si/phthalocyanine**(HCP-Co)**/Al diode. The current (I)- voltage (V) characteristics of heterojunction diode was measured under dark and illuminated conditions (20–100 mW/cm^2) at room temperature. The electrical characteristics of the device were measured. The ideal factor value was found to be 5.20 and according to this value, the diode exhibits non-ideal behavior. The frequency dependence of capacitance and the role of interface states were examined. The results showed that chalcone substituted metallo-phthalocyanine has a photodiode and photocapacitor characteristic. Therefore, chalcone-linked metallo-phthalocyanine can be applied in solar tracking systems.

1. Introduction

The increasing use of phthalocyanines as photodevice materials has gained attention in terms of the central metal ion or peripheral substituents. The existence of different peripheral substituents and central ions make phthalocyanines more modifying in terms of the various properties [1–10]. Phthalocyanines are high-conjugated synthetic macrocyclic compounds containing 8 carbon atoms and 8 nitrogen atoms with 18 π electrons. The center in the structure of the phthalocyanine compound determines the chemical property of the compound. The spectroscopic properties of phthalocyanines can vary widely due to their structure [1–12]. They can be seen in different colors, mostly blue and green, by absorbing visible light. It is used in Q bands with π-π * transitions and B or Soret bands with n-π* transitions to distinguish between metallic and non-metal phthalocyanine compounds.

Metallic phthalocyanines give a single band. Metallic phthalocyanines (MPc) are used as electrocatalysts, photocatalysts for the reduction of water to hydrogen, high energy density batteries, and inks because they have a dark blue color [12–16]. The chalcone compounds which are defined as propane containing two aromatic rings, one ketone group and one double bond in the main chain are also a flavonoid class compound. Because of the unsaturated carbonyl structures of α, β, chalcone compounds are highly chemically effective. The main method used in the synthesis of chalcone

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compounds is the Claisen-Schmidt condensation reaction. This method uses solutions in methanol or ethanol with strong base NaOH/KOH. In these solvents, substituted acetophenones and substituted benzaldehyde derivatives are reacted to give chalcone compounds [17–20]. As a result of various studies, chalcone compounds have been found to have physical and biological properties such as anticancer, anti-inflammatory, anti-invasive effects, solar cell applications and photophysical properties [21–25].

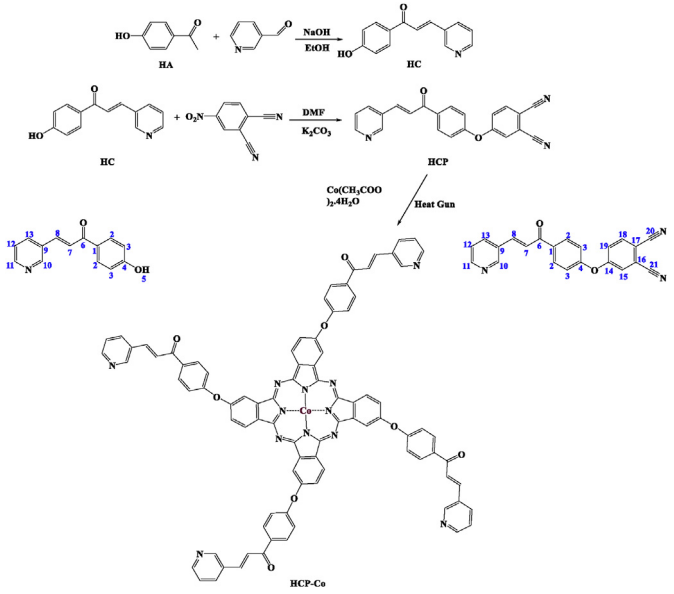
The synthesis and physical properties of phthalocyanine complexes were reported in the literature [26–31]. But no studies were found about photodiode properties of heterocyclic chalcone substituted phthalocyanines.

In this study, the heterocyclic chalcone compound and the phthalocyanine ring were used to obtained cobalt-phthalocyanine in Scheme 1. A new phthalonitrile derivative 4-((4′-oxyphenyl)-3-(3-pyridine)-2-propen-1-one)phthalonitrile (**HCP**) was synthesized by a reaction of 4-nitrophthalonitrile with 1-(4′-oxyphenyl)-3-(3-pyridine)-2-propen-1-one **(HC)**. Metallo-phthalocyanine complex (**HCP-Co**) was obtained according to the heating of solid-phase method under inert atmosphere. The cyclotetramerization of 4-((4′-oxyphenyl)-3-(3-pyridine)-2-propen-1-one)phthalonitrile (**HCP**) with Co(II) acetates was taken place to obtained new metallo-phthalocyanine (**HCP-Co**). New chalcone phthalonitrile compound (**HCP**) containing cobalt (**HCP-Co**) phthalocyanine complex was investigated in terms of photodiode characteristics. The HCP-Co/p-Si thin-film heterojunction diode was prepared. The electrical parameters of the diode such as the rectification rate (RR), the ideality factor (n) and the barrier height (Φb) were evaluated in terms of structure-reactivity relationship. The photocapacitance characteristic of shows the diode can also work as a photocapacitor.

2. Experimental

2.1. Materials and methods

4-Nitrophthalonitrile, pyridine-3-aldehyde, K_2CO_3, 4′-hydroxyacetophenone, 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU), $Co(OAc)_2.4H_2O$, dimethylformamide (DMF) and ethyl alcohol were purchased from Merck and Sigma-Aldrich. ^{1}H NMR and ^{13}C-APT NMR spectra were recorded in DMSO-d_6 (d 2.52 and 3.35 for ^{1}H and 40 ppm for ^{13}C-APT NMR) using a Bruker DPX-400 spectrometer with and TMS as the internal standards. UV–vis spectra were measured on a T80+PG model spectrophotometer and Infrared spectra were recorded on a PerkinElmer FT-IR instrument using KBr pellets spectrometer. MALDI-TOF spectra were recorded on Bruker Daltonics flex Analysis. Elemental analysis was carried out by a LECO 932 CHNS-O apparatus.



Scheme 1. Synthetic Scheme and Numeration of each P, H and C atom at comp. **HC** and **HCP**.

2.2. Synthesis

2.2.1. Synthesis of 1-(4′-oxyphenyl)-3-(3-pyridine)-2-propen-1-one *(HC)*

Heterocyclic chalcone compound (**HC**) was obtained according to Claisen–Schmidt condensation protocol in Scheme 1 [17,18]. A yellow solid, yield: 71%. FT-IR (KBr, cm^{-1}): 3441 ν_{OH}, 3067 and 3096 $\nu_{C\text{-}H(Ar.)}$, 2808, 2894 and 2952 $\nu_{C\text{-}H(Aliphatic)}$, 1656 $\nu_{C=O}$, 1513, 1590 and 1511 $\nu_{C=C.}$ ^{1}H NMR (400 MHz, DMSO-d_6, ppm): 6.91–6.93 (2H, d, J = 8.4 Hz, H^3, (–Ar-H)), 7.48–7.51 (1H, t, H^{12}, (–Ar-H)), 7.70–7.74 (1H, d, J = 15.6 Hz, H^7, (=CH-)), 8.06–8.10 (1H, d, J = 15.6 Hz, H^8, (-CH =)), 8.10–8.13 (2H, d, J = 8.8 Hz, H^2, (–Ar-H)), 8.35–8.36 (1H, d, J = 8 Hz, H^{13}, (–Ar-H)), 8.60–8.62 (1H, d, H^{11} –Ar-H), 9.02 (1H, s, H^{10} –Ar-H) and 10.53 (1H, s, H^5, (Ph-OH)). ^{13}C-APT NMR (DMSO-d_6, ppm): 129.34C^1, 131.83C^2, 115.92C^3, 162.88C^4, 187.35C^6, 124.48C^7, 139.74C^8, 131.19C^9, 150.68C^{10}, 151.22C^{11}, 124.37C^{12}, 135.44C^{13}. Anal. Calcd. for $C_{14}H_{11}NO_2$: C, 74.65; H, 4.92; N, 6.22. Found: C, 74.71; H, 4.98; N, 6.31%. MALDI-MS: m/z calc. 225.25; found: 226.08 $[M+H]^+$.

2.2.2. Synthesis of 4-((4′-oxyphenyl)-3-(3-pyridine)-2-propen-1-one)phthalonitrile *(HCP)*

A mixture of **HC** (2 g, 8.9 mmol), 4-nitrophthalonitrile (1.54 g, 8.9 mmol) and K_2CO_3 (2.45 g, 18 mmol) was stirred in the presence of dry DMF (20 mL) and then was reacted at room temperature for 48 h under argon atmosphere. The reaction was terminated by following the thin layer. The reaction was terminated and then the solid formed by settling in ice water (400 mL) was filtered and washed with water. A light yellow solid product was obtained after drying. Yield; 2.5 g (80%). FT-IR (KBr, cm^{-1}): 3016, 3052 and 3080 $\nu_{C\text{-}H(Ar.)}$, 2896 and 2959 $\nu_{C\text{-}H(Aliphatic)}$, 2234 $\nu_{C\equiv N}$, 1667 $\nu_{C=O}$, 1583, 1602 and 1607 $\nu_{C=C.}$ ^{1}H NMR (400 MHz, DMSO-d_6, ppm): 7.35–7.37 (2H, d, J = 8.4 Hz, H^3, (–Ar-H)), 7.49–7.52 (1H, t, H^{12}, (–Ar-H)), 7.56–7.59 (1H, d, H^{19}, (–Ar-H)), 7.78–7.82 (1H, d, J = 15.6 Hz, H^7, (=CH-)), 7.93 (1H, s, H^{15}, (–Ar-H)), 8.06–8.08 (1H, d, J = 15.6 Hz, H^8, (-CH =)), 8.15–8.17 (1H, d, J = 8.8 Hz, H^{18}, (–Ar-H)), 8.29–8.31 (2H, d, J = 8.8 Hz, H^2, (–Ar-H)), 8.34–8.35 (1H, d, H^{13}, (–Ar-H)), 8.63–8.64 (1H, d, H^{11}, (–Ar-H)), 9.04 (1H, s, H^{10}, (–Ar-H)). ^{13}C-APT NMR (DMSO-d_6, ppm): 134.66C^1, 132.01C^2, 117.38C^3, 160.21C^4, 188.01C^6, 124.42C^7, 141.22C^8, 130.94C^9, 150.92C^{10}, 151.56C^{11}, 124.39C^{12}, 135.68C^{13}, 158.79C^{14}, 123.96C^{15}, 120.23C^{16}, 109.84C^{17}, 136.92C^{18}, 124.07C^{19}, 115.81C^{20}, 116.32C^{21}. Anal. Calcd. for $C_{22}H_{13}N_3O_2$: C, 75.20; H, 3.73; N, 11.96. Found: C, 75.27; H, 3.77; N, 12.01%. MALDI-MS: m/z calc. 351.37; found: 352.10 $[M+H]^+$.

2.2.3. Synthesis of cobalt (II) metallo-phthalocyanine *(HCP-Co)*

To a mixture of **HCP** (0.5 g, 1.42 mmol) and $Co(CH_3COO)_2.4H_2O$ (0.18 g, 0.712 mmol) was added catalytic amount of DBU (four drops). The mixture was heated with a heat gun at 150 °C for 30 min and then was heated up to 230 °C. The reaction was terminated when the greenish mixture was observed. After the reaction mixture was dissolved in hot DMF (5 mL), it was transferred into ice water and the formed solid was filtered and dried. Yield: 0.526 g % 25. FT-IR (KBr, cm^{-1}): 3036 and 3060 $\nu_{C\text{-}H(Ar)}$, 2935 $\nu_{C\text{-}H(Alp)}$, 1661 $\nu_{C=O}$, 1503, 1522 and 1593 $\nu_{C=C,}$ $\nu_{C=N}$. UV–vis (DMF): λ_{max}/nm: 314, 610 and 666. Anal. Calcd. for

$C_{88}H_{52}CoN_{12}O_8$: C, 72.18; H, 3.58; N, 11.48. Found: C, 72.22; H, 3.61; N, 11.50%. MALDI-MS: *m*/*z* calc. 1464.39; found: 1464.515.

2.3. Preparation of HCP-Co/p-Si thin-film planar heterojunction diode

To fabricate the device, the p-Si was cleaned by deionized water for 5 min in an ultrasonic bath. Subsequently, the Si substrate is washed for 5 min each into an ultrasonic bath of methanol and acetone and then finally dried by nitrogen gas. The back contact was formed by thermally evaporating the Al metal onto the substrate and annealing at 570 °C for 5 min under the nitrogen environment. The **HCA-Co** solution was drop cast onto the p-Si substrate to form the organic layer and dried for 15 min at 50 °C. The prepared films were dried on a hot plate for 24 h at 50 °C. The top contact was formed by evaporating Al metal through the mask. The *I-V* (current-voltage) and capacitance-voltage (*C-V*) characteristics of the fabricated diode were determined at room temperature by using an FYtronix Electronic Device characterization system (Fig. 1).

3. Result and discussion

3.1. Synthesis and characterization

In this study, a new type metal phthalocyanine compound with the symmetrical structure containing chalcone groups in peripheral position were synthesized and characterization processes were performed by spectroscopic methods. The structures and general reaction notation of **HC, HCP** compounds and **HCP -Co** complex are shown in Scheme 1. In the synthesis step, starting from the compound 4-hydroxyacetophenone (**HA**) and 3-pyridinecarboxaldehyde, 1-(4′-hydroxyphenyl)-3-(3-pyridine)yl-prop-2-en-1-one **(HC)** was obtained in 71% yield.

Phenolic OH peak at 3441 cm^{-1}, carbonyl peak at 1656 cm^{-1}, and the olefin peaks at 1513, 1590 and 1611 cm^{-1} are proved that the formation of 1-(4′-oxyphenyl)-3-(3-pyridine)-2-propen-1-one (**HC**). The evaluation of NMR spectrum at Fig. 2 is also supported the formation of **HC**. The peak of number 5 proton aroused at 10.53 ppm belongs to phenolic proton, and another characteristic peaks for olefin protons show up at 7.74 and 8.10 ppm for numbers 7 and 8, respectively. In addition, the existence of aromatic protons and the integration of the integral heights with the protons in the compound are the most important proofs that compound HC is formed. In addition to proton NMR analysis, ^{13}C APT spectrum analysis is also proved that compound **HC** is formed. Carbonyl peak at 187.35 ppm, olefin carbons at 124.48 and 139.74 ppm and phenolic ipso carbon at 162.88 ppm can be shown as a proof of formation of 1-(4′-oxyphenyl)-3-(3-pyridine)-2-propen-1-one (**HC**). The presence of aromatic, olefin and other carbons indicates that compound HC is formed. In addition, characteristic peaks of the starting reagents (especially Aldehyde proton and carbon, aliphatic-CH_3 protons and carbon of ketone) were not observed in the ^1H and ^{13}C-APT NMR spectra.

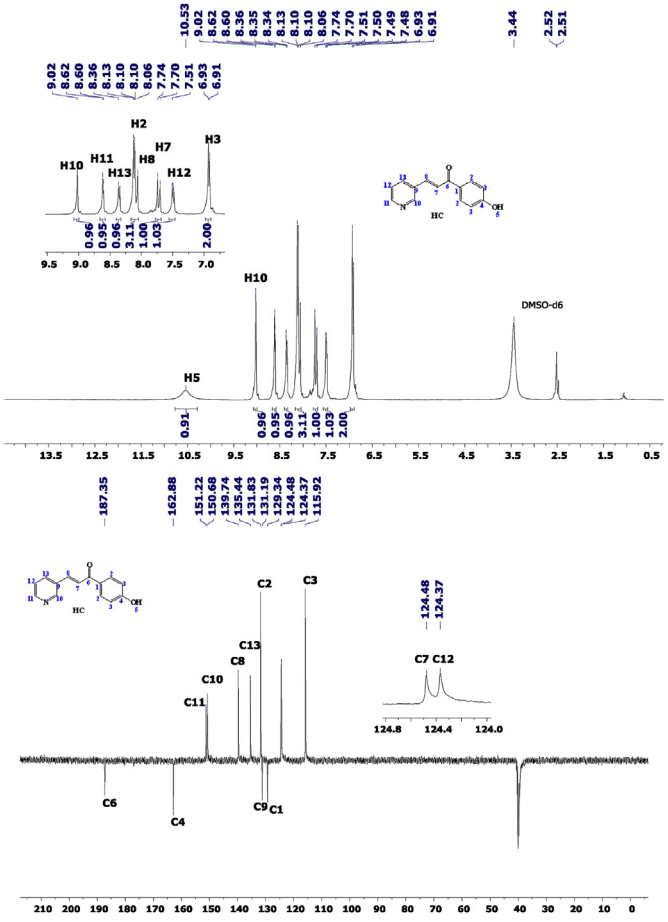


Fig. 2. ^1H and ^{13}C-APT NMR spectrum of **HC** (DMSO-d_6).

Chalcone substituted phthalonitrile **HCP** was obtained in 80% yield from the reaction of compound (**HC**) with 4-nitrophthalonitrile (**NP**). According to FT-IR spectrum of HCP, phenolic OH peak at 3441 cm^{-1} and nitro group peaks (at 1357 and 1537 cm^{-1}) is disappeared and nitrile stretching peak is observed at 2234 cm^{-1}. Integral heights are compatible with the structure. When ^{13}C-APT NMR spectrum is examined, the presence of nitrile carbon peaks in addition to the characteristic peaks indicates that the HCA compound is formed. Fig. 3 shows the ^1H and ^{13}C-APT NMR spectra of the HCA compound. These changes are supported the formation **HCP.**

A symmetrical metal phthalocyanine (**HCP-Co**) containing 1-(4′-phenyl)-3-(3-pyridine)-2-propen-1-one groups at peripheral positions were obtained from the reaction of **HCP** with metal salt (cobalt (II) acetates) according to the heating of solid-phase method under an inert atmosphere. In the structure of **HCP-Co complex**

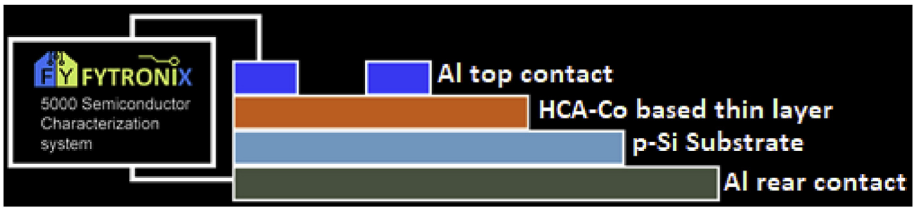


Fig. 1. Schematic diagram of the Al/p-si/HCA-Co/Al fabricated device.

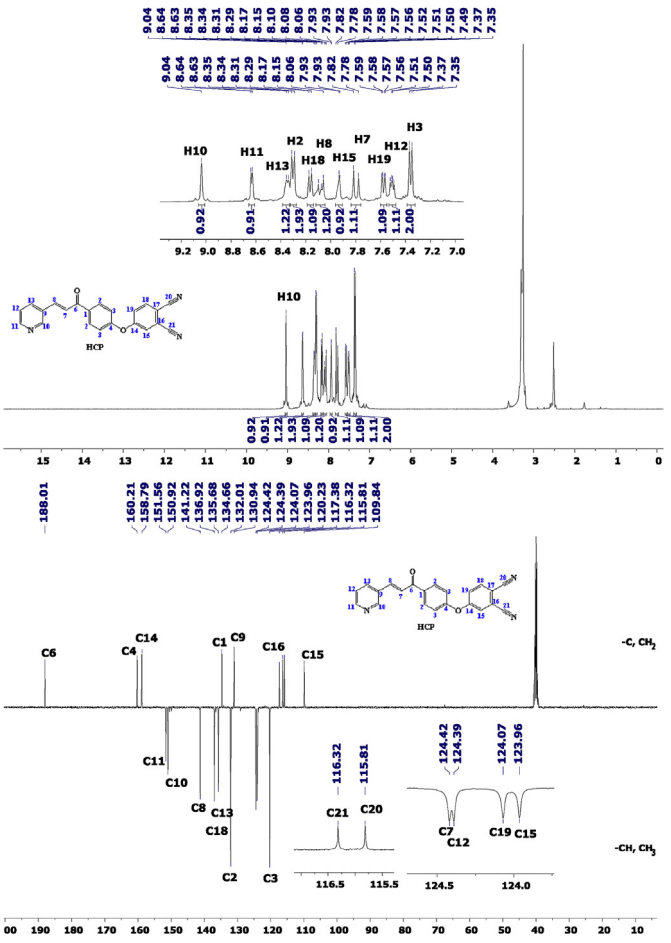


Fig. 3. ^{1}H and ^{13}C-APT NMR spectrum of **HCP** (DMSO-d_6).

was confirmed by the use of characterization methods (FT-IR, UV–Vis, MALTI-TOF MS and elemental analysis).

In the FT-IR spectrum of **HCP-Co** (Fig. 4), the existence of the aromatic peaks at 3036, 3060 cm^{-1}, the olefin stretching peaks at 1522, 1503 cm^{-1} and the disappearance of the nitrile peak at 2234 cm^{-1} proved that the target compound was formed. The disappearance of nitrile peak at 2234 cm^{-1} shows that metallic binding of cobalt was taken place.

When the UV/Visible spectra obtained in DMF are examined: characteristic Q band (π-π* transitions) for metallic phthalocyanines at 672 nm and B band (n-π* transitions) at 330 nm are observed. In addition, shoulder is seen as a result of aggregation at 610 nm.

When the MALDI-TOF MS spectra of **HCP-Co complex** is investigated, it appears they have almost the same molecular weights with the theoretically calculated masses (Fig. 5). These results are supported the formation **HCP-Co complex.**

3.2. Current-voltage (I-V) characteristics of the diode

The forward and reverse biased log I–V characteristics of the HCP-Co/p-Si thin-film heterojunction Schottky diode are shown at rt. under different light intensities (20–100 mW/cm^2) in Fig. 7. The *I-V* characteristics were analyzed by using the thermionic emission theory. According to the theory of thermionic emission (TE), the current through the Schottky contacts at forward feed voltage is determined by equation (1) [32].

$$I = I_0 exp\left(\frac{qV}{nkT}\right) \tag{1}$$

Where V is applied voltage, n is ideality factor and I_0 is the saturation current in the case of reverse polarity and calculated by equation (2).

$$I_0 = AA^* exp\left(\frac{-q\varphi_b}{kT}\right) \tag{2}$$

Where A is diode field, A^* effective Richardson constant, which equals 112 A/cm^2 K^2, T is absolute temperature, q is electron charge, Φb barrier height and k Boltzmann constant. n is a measure of the suitability of the diode to pure thermionic emission theory, the value of which is calculated from the slope of the linear region of the graph (ln I-V) using equation (3).

$$n = \frac{q}{kT}\frac{dV}{d(lnI)} \tag{3}$$

In the analysis of the experimental data of Schottky contacts, the

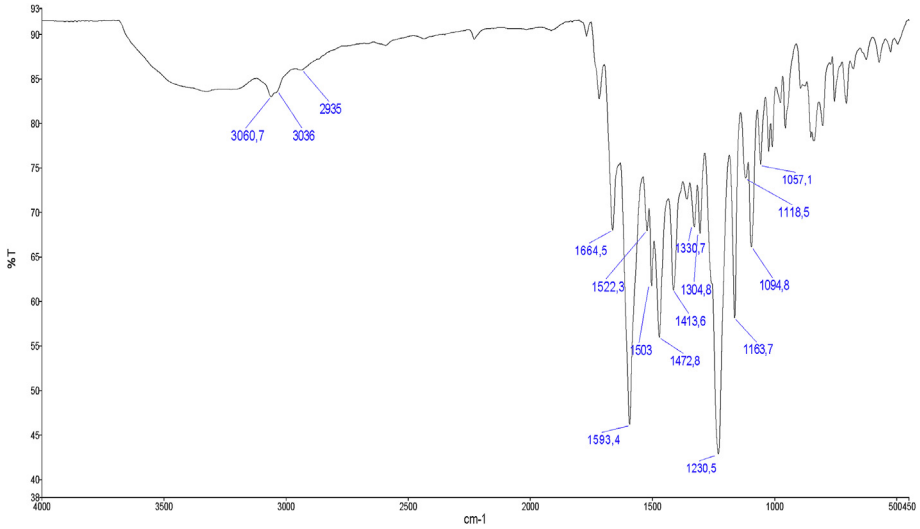


Fig. 4. FT-IR Spectrum of **HCP-Co** (KBr pellets).

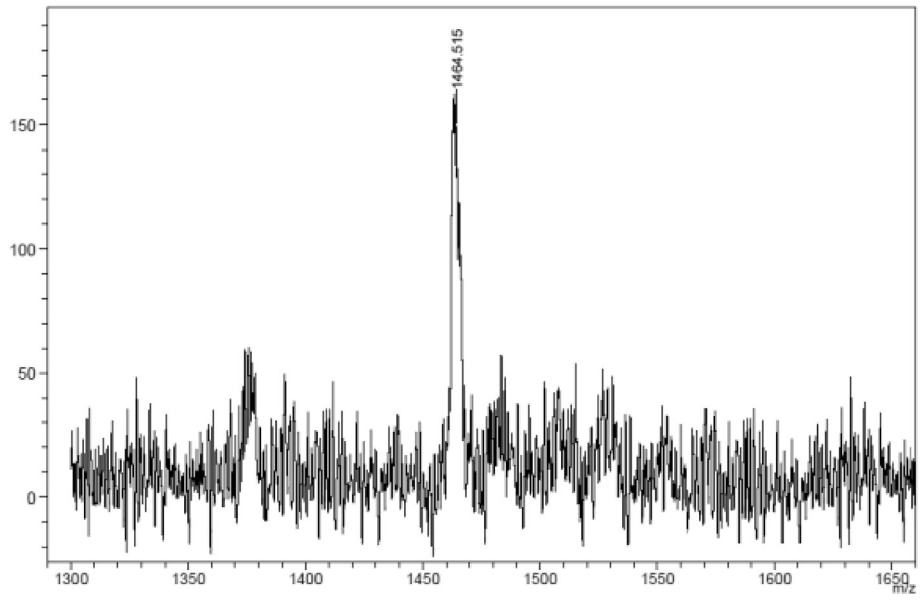


Fig. 5. MALDI TOF-MS spectrum of **HCP-Co complex**.

barrier height is usually determined from the intersection of the linear axis of the feed-forward I-V graph with the current axis. However, when an interface layer is present, this is the apparent barrier height. The effective or apparent barrier height is given as φ_b.

$$\varphi_b = \frac{kT}{q}\left(\frac{AA^*T^2}{I_0}\right) \tag{4}$$

The photosensitivity of the heterojunction diode was investigated. I-V characteristics of compound **HCP-Co** diode was evaluated under solar light in Fig. 6. The reverse current was increased with solar illumination due to the large hole separation of electrons that are jumped from the valence band to the conduction band. This behavior is a proof for HCP-Co to be used as a diode. The current–voltage curve gives us information about the electrical properties of diode such as the reverse saturation current,

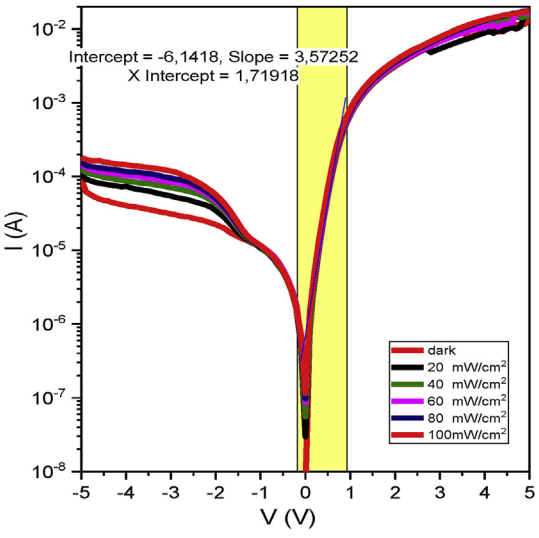


Fig. 6. Current-voltage characteristics plot of the Al/p-si/***HCP-Co***/Al diode under various illumination conditions.

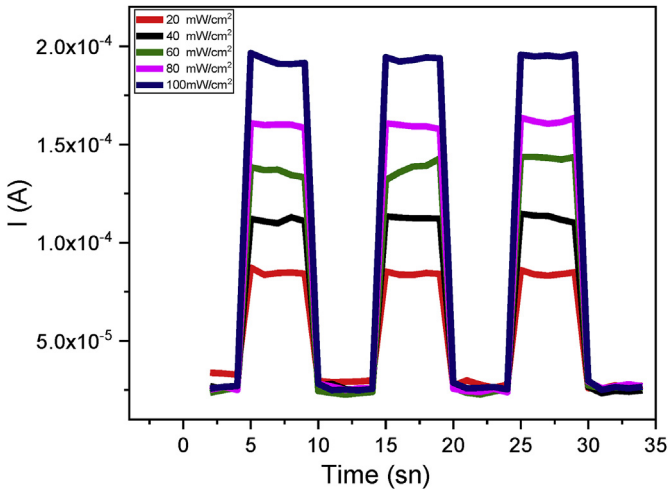


Fig. 7. Transient response of the Al/p-si/HCP-Co/Al diode.

rectification ratio and ideality factor. The ideality factor value 5.20 was found and it confirms the diode exhibits the non-ideal behavior. This non-ideality behavior arises due to the existence of surface states between silicon and an organic layer, non-homogeneity of the height of Schottky barrier, silicon dioxide layer and series resistance.

Similar studies on the electrical parameters of diodes prepared with phthalocyanine structures are available in the literature. For example, Canlıca et al. [33] have fabricated an Ag/Pc/p-Si Schottky barrier (SB) diode. They have found the ideality factor as 2.2. Islam et al. [34] have fabricated Ag/ZnPc/PEDOT:PSS/ITO cell for explore photovoltaic properties. They reported that the ideality factor was calculated as 3.8. Under various light intensity conditions, the photoresponse properties of the device were measured in reverse bias and shown in Fig. 6. The diode exhibits a high photocurrent.

3.3. Phototransient characteristics

In Fig. 7, the photo transient characteristic under various light

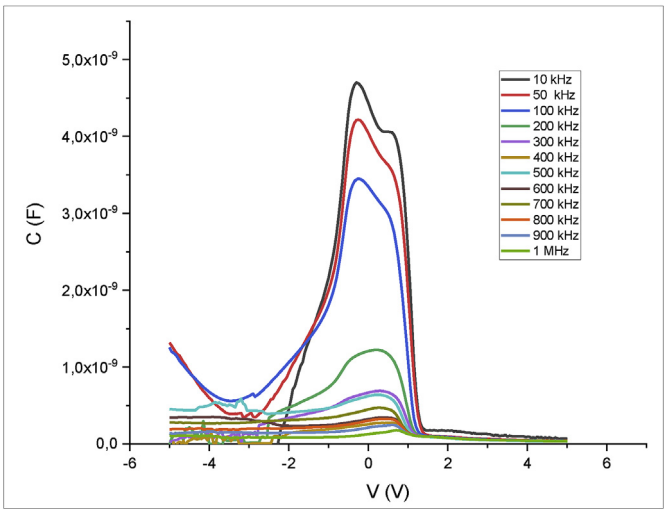


Fig. 8. C-V plots of the Al/p-si/HCA-Co/Al diode.

illuminations was evaluated based on current–time measurement. When photocurrent increases by the solar light it reaches a stable state and in the switch off situation it comes back to initial position. The photo-generated charge carries the interface region of the diode make us understand photo-charge mechanism. The rise and the decay in photocurrent are because of the trapping and detrapping in the charge carriers. According to the obtained results, the photo device can be evaluated as a photodiode. Another important parameter is the photo-response time which determines its fast varying light signal and also crucial for optical-switch applications [35,36].

3.4. Capacitance–voltage characteristics

One of the well-known methods to characterize the Schottky diode is the capacitance-voltage measurement at various frequencies. With this method, capacitance-voltage plots of diodes at various frequencies (10 kHz-1 MHz) are shown in Fig. 8. While the capacitance changes suddenly in the negative voltage region, it does not change much in the positive voltage region [37]. The reason for this change of diode in the negative region is thought to be due to the expansion in the depilation region. The capacitance is changed with bias voltages which indicates that the width of depletion is expanded with applied voltages [38]. However, capacitance shows a peak at a negative voltage and the height of this peak decreases with an increase in frequency.

4. Conclusion

In conclusion, the chalcone substituted metallophthalocyanine conjugate (**HCP-Co**) was successfully prepared. The **HCP-Co** thin film planar heterojunction diode was prepared. The photoelectrical properties of heterocyclic chalcone substituted metallo phthalocyanine conjugate were evaluated at solar light illuminations. Rectification ratio (RR) of the diode was calculated as 158,81 at ±3 V in dark conditions. The heterojunction diode showed diode characteristics with lower reverse saturation current as $2.15x10^{-3}$ A at 3 V and the ideality factor as 5.2. The photo transient characteristic under various light illuminations was evaluated based on current–time measurement. According to results, the photodevice can be evaluated as a photodiode. The capacitance–voltage under various frequencies (10 kHz-1MHz) of the diodes were measured. The diode exhibited the highest photocapacitance at 10 kHz and under the illumination intensity of 100 mW/cm^2. Hence, our device can also be used as a photocapacitor.

CRediT authorship contribution statement

Murat Demirol: Formal analysis, Investigation. **Lütfiye Sirka:** Formal analysis, Investigation. **Eray Çalışkan:** Formal analysis, Investigation. **Fatih Biryan:** Software, Investigation. **Kenan Koran:** Conceptualization, Writing - original draft, Writing - review & editing. **Ahmet Orhan Görgülü:** Visualization, Supervision, Project administration. **Fahrettin Yakuphanoğlu:** Investigation, Software, Methodology.

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