

# Electronic and Optical Properties of 3,3',3'',3'''- and 4,4',4'',4'''- Tetraaminophthalocyanincopper(II) Grafted with Different Polymers

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## Abstract

Phthalocyanines and other related compounds are an important class of macrocyclic with many commercial applications. Therefore, their electronic and optical properties of 3,3',3'',3'''-tetraaminophthalocyanincopper(II), and 4,4',4'',4'''-tetraaminophthalocyanincopper(II) compounds and their grafted polymers based on poly(ethylene glycol) with adipic acid or sebacic acid were studied. Their absorption spectra resulted from UV-Visible measurements showed the characteristic phthalocyanine peaks (Q and B bands). In general, the two bands exhibit blue shifts of both bands for polymers grafted-compounds in comparison with 3,3',3'',3'''- (NH<sub>2</sub>)<sub>4</sub>PcCu(II) and 4,4',4'',4'''-(NH<sub>2</sub>)<sub>4</sub>PcCu(II) alone. The energy gaps of tetraaminophthalocyanincopper(II) compounds and with grafted polymers were calculated from optical measurements and showed an increasing in the energy gap due to grafted polymers in comparison with the ungrafted tetraaminophthalocyanincopper(II) and they were affected by their particle size.

**Keywords:** electronic properties, optical properties, tetraaminophthalocyanine, grafting polymer, energy gap.

## 1. Introduction

Phthalocyanine and its derivatives constitute one of the most studied classes of organic functional materials in nonlinear optics (Zawadzka et al., 2014; Yao et al., 2013; and Venkatram et al., 2008), liquid-crystalline electronic charge carriers (Yuksel et al., 2011; Ambily, et al., 1999), exciton-transport materials (Tejada et al., 2011), optical data storage (Donker et al., 2005), photodynamic cancer therapy (Tynga et al., 2013; Nombona et al., 2012), solar cells (Klumbies et al., 2014), catalysis (Mahyari et al., 2014; Forde et al., 2012), as the active layers of gas sensors (Cui et al., 2013) and as electrochromic substances (Riou and Clarisse, 1988).

There is a considerable interest in the characterization of the electronic structure of phthalocyanines. The absorption spectra of many metallated as well as free Pc's have been reported in the vapor phase (Fielding and Mackay, 1964), various solvents (Stillman and Thomson, 1974), and thin films (Collins et al., 1993) and in thermoplastic media (Lucia et al., 1968).

The spectra of Metallophthalocyanine complexes consist of an intense absorption band in the visible region traditionally near 670nm called the Q band and a generally weaker band near 340 nm called the Soret or B band (De la Torre et al., 2010). The typical molar extinction coefficient of the Q-band of a Phthalocyanine is in ca. 10<sup>5</sup> M<sup>-1</sup>cm<sup>-1</sup> (Jiang et al., 2010; Li et al., 2008).

The location of the Q band in phthalocyanine complexes can be adjusted by attaching suitable substituents onto the peripheral and non-peripheral positions of the ring and by the change in the nature, size and number of substituents. Addition of electron donating groups such as -NH<sub>2</sub>, OR and SR at the non-peripheral (1,4,8,11,15,18,22, and 25) or peripheral (2,3,9,10,16,17,23, and 24) positions of the phthalocyanine ring results in red shift to the NIR region. Substitution at the non-peripheral position shows more red-shift than at the peripheral position (Nyokong and Isago, 2004).

Furthermore, measurement of the absorption of light is considered as one of the most important techniques for optical measurements in solids because the optical properties of solids provide an important tool for studying energy band structure, impurity levels, localized defects, lattice vibrations, and certain magnetic excitations (Yao et al., 2013).

In previous work, we reported (Haider, 2014) gravitation of some prepared phthalocyanine tetraamino derivatives with different polymers to produce polygrafted molecules able to be used for different applications. This paper will concentrate on the effect of grafting process on the electronic properties of the resulting tetraaminophthalocyanincopper(II) solid derivatives and studying their optical properties and measuring their energy gaps.

## 2. Methods

### 2.1 Preparation of Phthalocyanine Derivatives and Grafted Polymers

Two phthalocyanine compounds were prepared, namely 3,3',3'',3'''- tetraaminophthalocyanincopper(II) and 4,4',4'',4'''- tetraaminophthalocyanincopper(II) compounds with four substitution of nitro groups of 3 position

and 4 position respectively. The nitro groups were reduced to amino groups to produce 3,3',3'',3'''-tetraaminophthalocyanatocopper(II) (3Pc-NH<sub>2</sub>) and 4,4',4'',4'''-tetraaminophthalocyaninato copper(II) (4Pc-NH<sub>2</sub>) by addition of excess of sodium sulfide nonahydrate solution.

Poly(ethylene adipate) and poly(ethylene sebacate) were prepared using ethylene glycol and adipic acid and sebacic acid monomers respectively. Also, poly(ethylene glycol)-sebacic acid polyester using different chain length of poly(ethylene glycol) molecular weight (6000, 10000 and 20000) were prepared too. All polymers were prepared by condensation polymerization and to have carboxylic terminal groups. They were characterized by FTIR spectroscopy and molecular weight determination by end-group analysis.

3Pc and 4Pc compounds were grafted by the prepared polymers to produce ten phthalocyanine grafted compounds. The FTIR spectra revealed that real grafting were obtained. The code of the phthalocyanine compounds and their grafted polymers are shown in Table 1.

## 2.2 Electronic (UV-Visible) Spectra

The U.V-Visible electronic spectra were recorded in the range (800-200) nm, using L.R1156 Spectrophotometer. The spectra of the compounds were recorded in DMSO as a solvent, using 1cm quartz cell at ambient temperature.

Table 1: Code of prepared phthalocyanine compounds and their grafted polymers

Sample	Code
3,3',3'',3'''-tetranitrophthalocyanatocopper(II)	3Pc-NO <sub>2</sub>
3,3',3'',3'''-tetraaminophthalocyanatocopper(II)	3Pc-NH <sub>2</sub>
3Pc grafted poly(ethylene adipate), PEA	3Pc+PEA
3Pc grafted poly(ethylene sebacate), PES	3Pc+PES
3Pc grafted poly(ethylene glycol)-sebacic acid with PEG 6000	3Pc+PEG <sub>1</sub> S
3Pc grafted poly(ethylene glycol)-sebacic acid with PEG 10000	3Pc+PEG <sub>1</sub> S
3Pc grafted poly(ethylene glycol)-sebacic acid with PEG 20000	3Pc+PEG <sub>3</sub> S
4,4',4'',4'''-tetranitrophthalocyanatocopper(II)	4Pc-NO <sub>2</sub>
4,4',4'',4'''-tetraaminophthalocyanatocopper(II)	4Pc-NH <sub>2</sub>
4Pc grafted poly(ethylene adipate)	4Pc+PEA
4Pc grafted poly(ethylene sebacate)	4Pc+PES
4Pc grafted poly(ethylene glycol)-sebacic acid with PEG 6000	4Pc+PEG <sub>1</sub> S
4Pc grafted poly(ethylene glycol)-sebacic acid with PEG 10000	4Pc+PEG <sub>1</sub> S
4Pc grafted poly(ethylene glycol)-sebacic acid with PEG 20000	4Pc+PEG <sub>3</sub> S

## 3. Results and Discussion

### 3.1 Electronic Properties

The main and most characteristic feature of the absorption spectra of phthalocyanines is the presence of two very intensive bands (Q and B). The Q-band represents absorption of light and consequently excitation of electrons from the highest occupied molecular orbital (HOMO), namely the a<sub>1u</sub> (π), to the lowest unoccupied molecular orbital (LUMO), namely the eg (π\*). Furthermore, the transition from a<sub>2u</sub> to eg results in the B-band formation (Lezno and Lever, 1989).

Figures 1 and 2 show the electronic spectra (UV-Visible) of the of 3- and 4- nitro derivatives of phthalocyanine compounds deposited as thin film.

It is quite obvious that both spectra exhibit a general feature of phthalocyanine compounds and they are the two characteristic bands with the main absorption in the range 298-337 nm (B- band) and in the range 680-740 nm (Q-band). Table 2 summarizes their values.

The reduction of nitro groups to amino groups causes changes toward the red shifts, (+56 nm for Q band) and (27 nm for B band) for 3Pc-NH<sub>2</sub> while in the 4Pc-NH<sub>2</sub>, red shifts are (+45 nm for Q band) and (25 nm for B band), Figures 3 and 4 respectively. This may be attributed to the addition of electron donating groups (NH<sub>2</sub>) at the non-peripheral (1,4,8,11,15,18,22, and 25) or peripheral (2,3,9,10,16,17,23, and 24) positions of the Pc ring result in red shift to the NIR region due to the presence of the pair of electrons of the amine group (Nyokong, 2008; Lezno and Lever, 1989).

Substitution at the nonperipheral position (3Pc) shows more red shift than at the peripheral position (4Pc). These results are in a good agreement with the reported results (Bhardwaj, 2002), that a large red shift was obtained for benzene rings substituted at the 3 positions with substituent groups that allow conjugation with the 18 system.

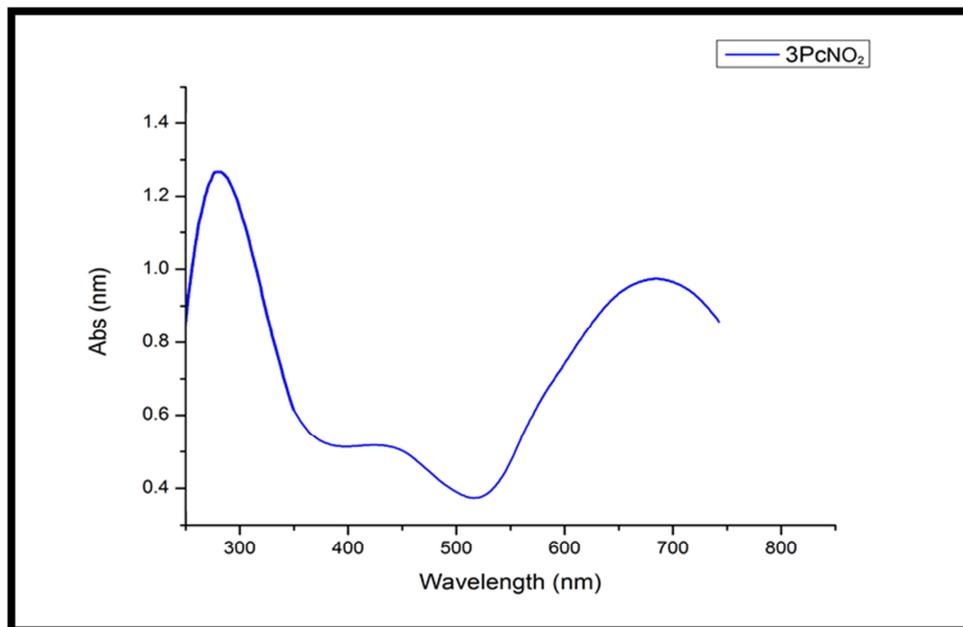


Figure 1: Electronic absorption spectrum of 3Pc-NO<sub>2</sub>

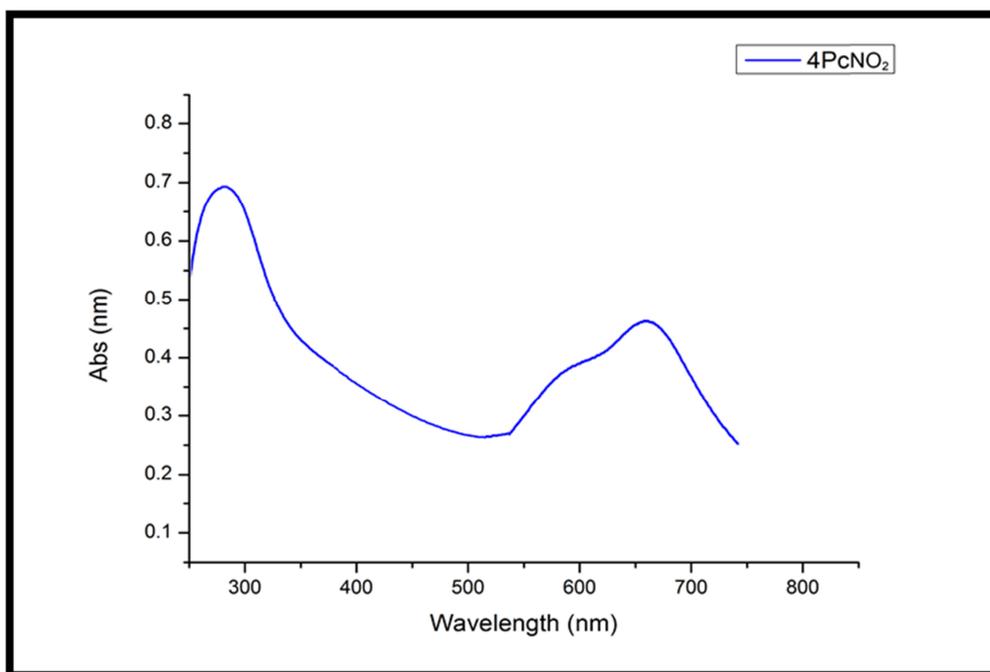


Figure 2: Electronic absorption spectrum of 4Pc-NO<sub>2</sub>

Table 2: Values of Q and B bands of phthalocyanine derivatives and their grafted polymers

Sample	B bands	Q bands
3Pc-NO <sub>2</sub>	298	684
3Pc-NH <sub>2</sub>	325	740
3Pc grafted poly(ethylene adipate), PEA	332	730
3Pc grafted poly(ethylene sebacate), PES	330	725
3Pc grafted poly(ethylene glycol)-sebacic acid with PEG 6000, PEG <sub>1</sub> S	330	728
3Pc grafted poly(ethylene glycol)-sebacic acid with PEG 10000, PEG <sub>2</sub> S	330	735
3Pc grafted poly (ethylene glycol)-sebacic acid with PEG 20000, PEG <sub>3</sub> S	335	735
3Pc-NO <sub>2</sub>	302	680
4Pc-NH <sub>2</sub>	326	725
4Pc grafted poly(ethylene adipate), PEA	318	720
4Pc grafted poly(ethylene sebacate), PES	337	718
4Pc grafted poly(ethylene glycol)-sebacic acid with PEG 6000, PEG <sub>1</sub> S	318	720
4Pc grafted poly(ethylene glycol)-sebacic acid with PEG 10000, PEG <sub>2</sub> S	320	721
4Pc grafted poly (ethylene glycol)-sebacic acid with PEG 20000, PEG <sub>3</sub> S	328	722

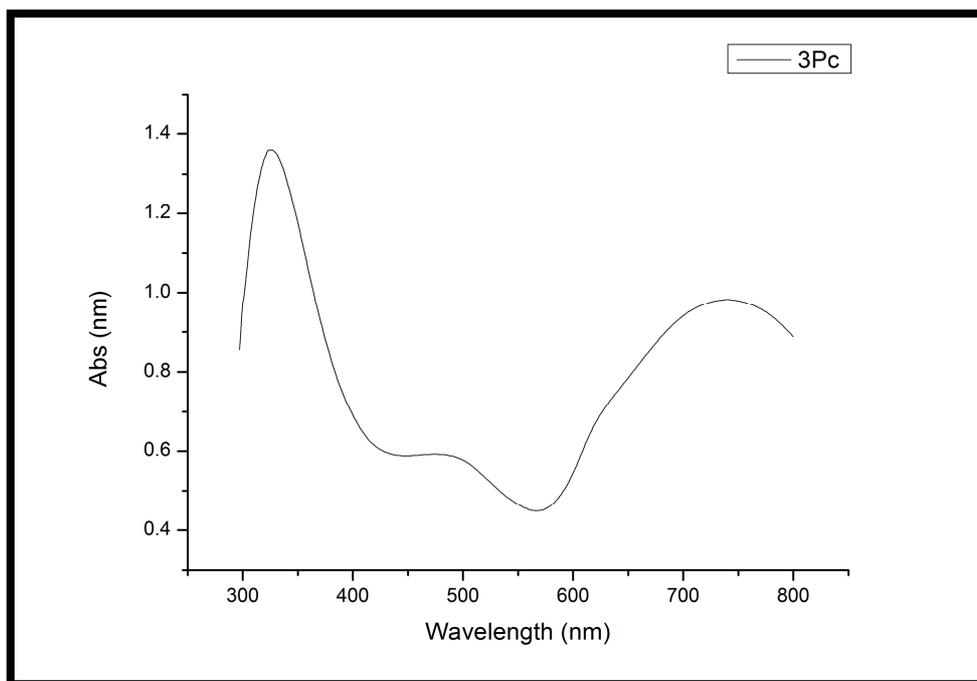


Figure 3: Electronic absorption spectrum of 3Pc-NH<sub>2</sub>

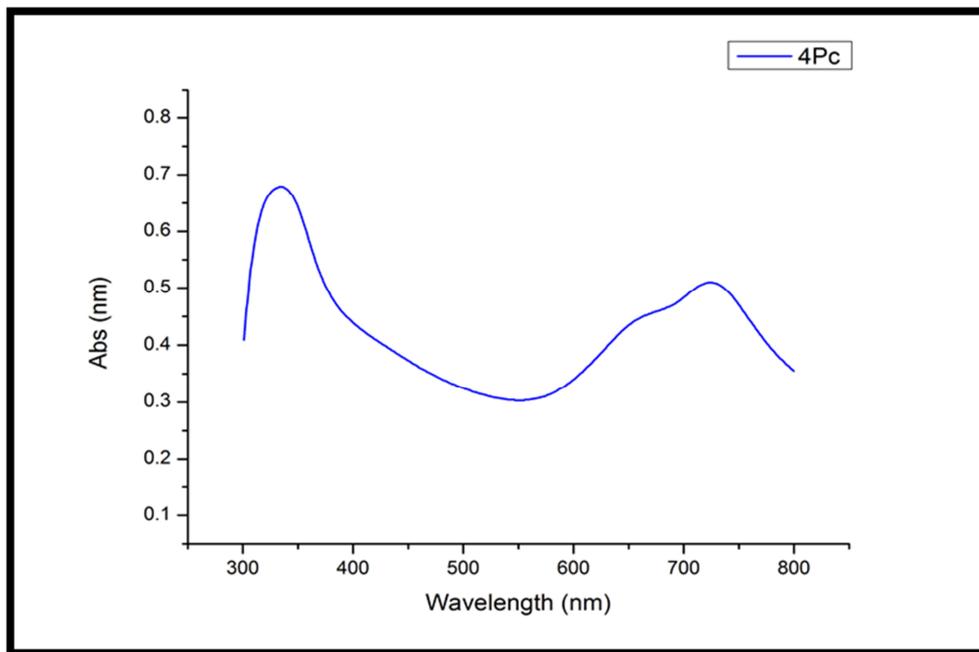


Figure 4: Electronic absorption spectrum of 4Pc-NH<sub>2</sub>

Grafting phthalocyanine amino derivatives with different prepared polymers having carboxyl end groups causes a little further red shift due to the effect of the electron-withdrawing carbonyl groups (Al-Assadi, 2002).

The electronic spectrum (UV-Visible) for the 3Pc-grafted PEA show a red shift (+46 nm for Q band) and (+34 nm for B band), while the red shifts at 3Pc-grafted PES are (+41 nm for Q band) and (+32 nm for B band) compared to the spectrum given by ungrafted 3PcNH<sub>2</sub>, Table (2).

Grafting 3Pc derivative with PEGn-S polymers having different PEG chain lengths (different molecular weights), i.e., 3Pc-grafted PEG1-S, 3Pc-grafted PEG2-S and 3Pc-grafted PEG3-S, gave different electronic spectra in UV-Visible measurements and seems also enhancing toward the red shifts, results are shown in Table (2). 3Pc-grafted PEG1-S gave (+44 nm for Q band) and (+32 nm for B band), 3Pc-grafted PEG2-S (+53 nm for Q band) and (+32 nm for B band), and 3Pc-grafted PEG3-S (+53 nm for Q band) and (+37 nm for B band).

The second group of peripheral substitutions, 4Pc, its electronic spectrum (UV-Visible) for the 4Pc-grafted PEA indicated red shifts (+40 nm for Q band) and (+16 nm for B band) while spectrum of 4Pc grafted PES exhibits a (+38 nm for Q band) and (+32 nm for B band) compared with ungrafted 4PcNH<sub>2</sub>.

Grafting 4Pc-NH<sub>2</sub> derivative with PEGn-S polymers having different PEG chain lengths (different molecular weights), i.e., 4Pc-grafted PEG1-S, 4Pc-grafted PEG2-S and 4Pc-grafted PEG3-S, gave also different electronic spectrum in UV-Visible measurements and seems also enhancing toward the red shifts, Table 2.

4Pc-grafted PEG1-S gave (+40 nm for Q band) and (+16 nm for B band), 4Pc-grafted PEG2-S (+41 nm for Q band) and (+18 nm for B band), and 4Pc-grafted PEG3-S (+42 nm for Q band) and (+26 nm for B band).

### 3.2 Optical Properties

The optical band gap and the nature of optical transitions can be obtained from the dependence of the absorption coefficient on the photon energy. The optical absorption coefficient of the film was calculated from the relationship in Equation 1 and 2 (Yakuphanoglu, 2007).

$$A' = A - A'' \quad \dots\dots\dots (1)$$

$$\alpha = (2.303 (A - A'')/d) \quad \dots\dots\dots (2)$$

Where: A= Absorbance, A'= Correction Absorbance, A''= Correction value of Absorbance, d= Thickness of thin film, and  $\alpha$ = Absorption coefficient

It is reported (Kim et al., 2000) that, there are two types of transition and both of them depend on the absorption coefficient value ( $\alpha$ ). If  $\alpha$  is  $\geq 10^4$ , this means that the transition is of the direct type, and if  $\alpha$  is  $< 10^4$ , the transition will be indirect. For direct transition, the energy gaps can be obtained from Tauc's formula as in Equation 3, and for indirect transition as in Equation 4:

$$\alpha = \alpha^0 (h\nu - E'g)^2 / h\nu \quad \dots\dots\dots (3)$$

$$\alpha = A' (h\nu - E_g \pm E_p)^{1/2} / h\nu \quad \dots\dots\dots (4)$$

Where:

$\alpha$  and  $A'$  are Absorption Coefficient and Correction Absorbance respectively as mentioned before, and  $h\nu$ =Photon energy,  $E'_g$ = Energy of direct transition,  $E_g$ = Energy of indirect transition, and  $E_p$ = Photon energy of indirect transition.

The direct and indirect transition optical energy gaps can be determined from the intercept of the extrapolated linear part of the curve to zero absorption of the plot of  $(\alpha h\nu)^{1/2}$  for indirect transition and  $(\alpha h\nu)^2$  for direct transition as functions of photon energy.

The absorption coefficients ( $\alpha$ ) calculated from equation 2 exhibit that 3Pc, 4Pc and their grafted polymers have values  $\geq 10^4$ , which implies that the transitions are direct.

The energy gaps of 3Pc-NH<sub>2</sub> and 4Pc-NH<sub>2</sub> are measured from Figures 5 and 6 for ungrafted 3Pc and 4Pc respectively, and the obtained results are shown in Table 3 for ungrafted phthalocyanine and for phthalocyanine grafted polymers.

From the data in Table 3, one can observe that the 3Pc-NH<sub>2</sub> and 4Pc-NH<sub>2</sub> have lower values of energy gap compared with the Pc-grafted polymers which causes toward blue shifts in energy gap. This may be attributed to reduction happened in the particle sizes going from 3Pc-NH<sub>2</sub> and 4Pc-NH<sub>2</sub> to Pc-grafted polymers as it was discussed in literature (Table 4) (Haider, 2014) leading to increase in energy gap. The same observation was made by Ahemen and his co-workers (Iorkyaa et al., 2013) on their study of the effect of different chain length of poly(ethylene glycol) on particle size of ZnS nanoparticles and measuring their energy band gaps.

It was found that the 3Pc-grafted PEA has blue shift from the 3Pc-NH<sub>2</sub> with (0.3ev) with particle (20 nm) size while the 3Pc-grafted PES has blue shift with (0.3ev) with particle (20 nm) compared with 3Pc which has particle size (40 nm), Table 4.

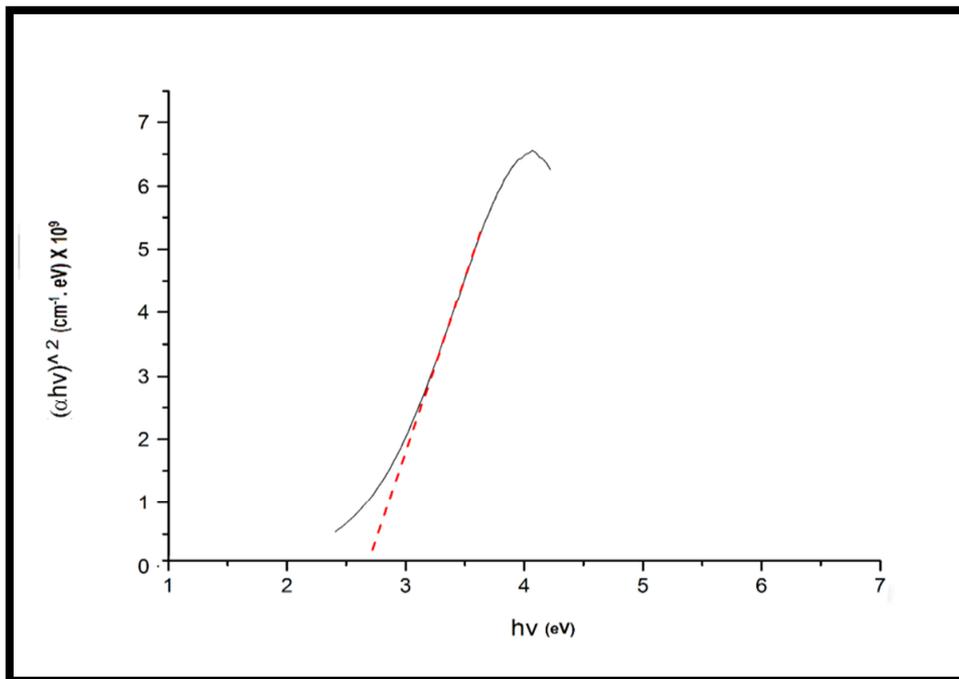


Figure 5: The variation of  $(\alpha h\nu)^2$  versus photon energy ( $h\nu$ ) for 3Pc-NH<sub>2</sub>

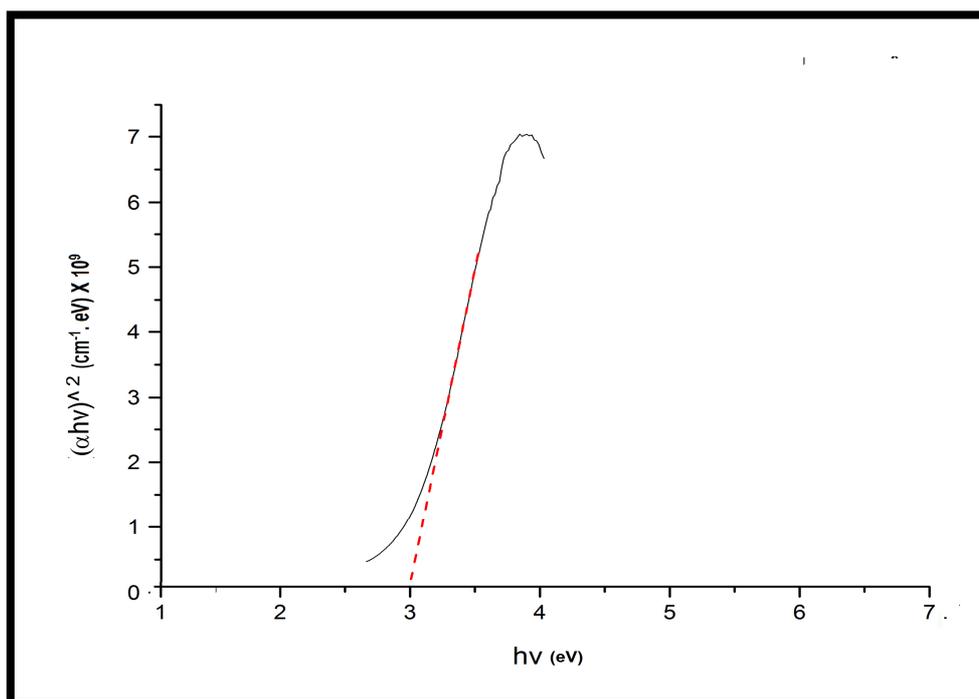


Figure 6: The variation of  $(\alpha h\nu)^2$  versus photon energy ( $h\nu$ ) for 4Pc-NH<sub>2</sub>

Table 3: Energy gap values for 3Pc-NH<sub>2</sub>, 4Pc-NH<sub>2</sub> and the grafted polymers

Sample	Eg (ev)	Sample	Eg (ev)
<b>3Pc</b>	2.7	<b>4Pc</b>	2.6
<b>3Pc+PEA</b>	3	<b>4Pc+PEA</b>	2.9
<b>3Pc+PES</b>	3	<b>4Pc+PES</b>	3
<b>3Pc+PEG<sub>1</sub>S</b>	2.9	<b>4Pc+PEG<sub>1</sub>S</b>	2.9
<b>3Pc+PEG<sub>2</sub>S</b>	2.75	<b>4Pc+PEG<sub>2</sub>S</b>	2.8
<b>3Pc+PEG<sub>3</sub>S</b>	2.8	<b>4Pc+PEG<sub>3</sub>S</b>	2.8

The 3Pc-NH<sub>2</sub> grafted different polymers, namely PEG<sub>1</sub>-S, PEG<sub>2</sub>-S and PEG<sub>3</sub>-S, their blue shifts are (0.2 eV), (0.05 eV) and (0.1 eV) respectively compared to 3Pc-NH<sub>2</sub>. This is might attributed to the reduction in sizing of 3Pc-grafted different polymers (15 nm), (30 nm) and (36 nm) respectively compared to 3Pc-NH<sub>2</sub> that has particle size of (40 nm), Table 4.

In the second group of 4Pc with grafted polymers, the 4Pc-NH<sub>2</sub> grafted-PEA has blue shift with (0.3 eV) with particle size (24 nm), while the 4Pc-grafted PES has blue shift with (0.4 eV) with particle size (24 nm) compared with 4Pc-NH<sub>2</sub> which has 30 nm particle size. Also, the 4Pc grafted different polymers, namely PEG<sub>1</sub>-S, PEG<sub>2</sub>-S and PEG<sub>3</sub>-S, their blue shifts are (0.5 eV) with particle size (18 nm), (0.8 eV) with particle size (23 nm) and (0.1 eV) with particle size (24 nm) respectively compared with 4Pc-NH<sub>2</sub> which has particle size (30 nm), Table 4.

All these results confirm the reductions in sizing leading to an increase at the energy gap and they are in good agreement with results reported in the literature. Iorkyaa et al., 2013 have used polyethylene glycol (PEG) and carboxyl methyl cellulose (CMC) as capping agent for ZnS particles, they found that absorption band of both ZnS/PEG and ZnS/CMC were slightly blue shifted from that of the bulk ZnS, and a report on the influence of poly(vinyl alcohol) and poly(methacrylic acid) on ZnS nanoparticles revealed also there was particle sizes effect (Iorkyaa et al., 2014).

Table 4: Specific surface area, surface weight mean, volume weight mean and [d (0.1), d (0.5) and d (0.9)] parameters of ungrafted and polymer-grafted 3- and 4- tetranitrophthalocyanocopper(II)<sup>(30)</sup>

Item	Particle Size	Surface Weight Mean	Vol. Weight Mean	d(0.1)	d(0.5)	d(0.9)	Uniformity	Specific Surface Area
3Pc-NH <sub>2</sub>	40	12.235	35.602	4.423	27	76.32	0.873	0.2
3Pc +PEA	20	8.615	19.441	3.468	14.30	42.43	0.869	0.284
3Pc +PES	20	8.251	17.695	3.43	13.5	38.23	0.816	0.297
3Pc +PEG <sub>1</sub> S	15	7.636	16.717	3.521	11.51	33.33	0.932	0.321
3Pc +PEG <sub>2</sub> S	15	6.461	13.533	4.21	6.24	32.36	1.41	0.379
3Pc +PEG <sub>3</sub> S	36	9.341	29.846	3.37	23.16	67.72	0.872	0.262
4Pc-NH <sub>2</sub>	30	15.568	30.711	7.131	24.21	58.86	0.722	0.156
4Pc + PEA	24	12.42	25.87	5.453	19.79	52.62	0.77	0.197
4Pc +PES	16	10.155	16.143	5.231	13.19	29.74	0.608	0.241
4Pc +PEG <sub>1</sub> S	18	11.557	19.065	5.93	15.12	33.12	0.648	0.212
4Pc +PEG <sub>2</sub> S	23	13.437	21.839	6.592	18.87	40.07	0.568	0.182
4Pc +PEG <sub>3</sub> S	24	11.786	22.29	4.868	19.53	53.31	0.68	0.208

#### 4. Conclusions

The results of UV-Visible and fluorescence showed the characteristic phthalocyanine peaks (Q and B bands), and they exhibit clear blue shifts of both bands for the grafted-phthalocyanine in comparison with the 3Pc-NH<sub>2</sub> and 4Pc-NH<sub>2</sub> alone.

The energy gaps measured for tetraaminophthalocyaninecopper(II) compounds and their grafted polymers were in good relation with their particle sizes.

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