World Academy of Science, Engineering and Technology
International Journal of Chemical and Materials Engineering
Vol:16, No:8, 2022

Application of Molecular Materials in the Manufacture of Flexible and Organic Devices for Photovoltaic Applications

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Open Science Index, Chemical and Materials Engineering Vol:16, No:8, 2022 publications.waset.org/10012627.pdf

Abstract**—**Many sustainable approaches to generate electric energy have emerged in the last few decades; one of them is through solar cells. Yet, this also has the disadvantage of highly polluting inorganic semiconductor manufacturing processes. Therefore, the use of molecular semiconductors must be considered. In this work, allene compounds $C_{24}H_{26}O_4$ and $C_{24}H_{26}O_5$ were used as dopants to manufacture semiconductor films based on PbPc by high-vacuum evaporation technique. IR spectroscopy was carried out to determine the phase and any significant chemical changes which may occur during the thermal evaporation. According to UV-visible spectroscopy and Tauc's model, the deposition process generated thin films with an activation energy range of 1.47 eV to 1.55 eV for direct transitions and 1.29 eV to 1.33 eV for indirect transitions. These values place the manufactured films within the range of low bandgap semiconductors. The flexible devices were manufactured: polyethylene terephthalate (PET), Indium tin oxide (ITO)/organic semiconductor/Cubic Close Packed (CCP). The characterization of the devices was carried out by evaluating electrical conductivity using the four-probe collinear method. I-V curves were obtained under different lighting conditions at room temperature. OS1 (PbPc/$C_{24}H_{26}O_4$) showed an Ohmic behavior, while OS2 (PbPc/$C_{24}H_{26}O_5$) reached higher current values at lower voltages. The results obtained show that the semiconductor devices doped with allene compounds can be used in the manufacture of optoelectronic devices.

Keywords**—**Electrical properties, optical gap, phthalocyanine, thin film.

I. INTRODUCTION

THE photo-response occurs when a photon has the necessary energy to eject an electron from molecular orbital HOMO (Highest Occupied Molecular Orbital) and sending it to molecular orbital LUMO (Lowest Unoccupied Molecular Orbital). This type of phenomenon is currently used in photocells, photoconductive devices, and solar cells. The photo-response can be present in organic and inorganic materials. Organic materials have those so-called *small molecular materials*. In this type of materials is included a subgroup that includes the photovoltaic materials within which the phthalocyanines (Pcs) are found [1], [2]. Pcs are molecular materials that are formed by four isoindol units that form a central cavity where around 63 different ions can be accommodated. If they have one or two metallic ions are called metallic phthalocyanines or metallophthalocyanines (MPc). Since the MPcs show the ability to transfer charges because of their system of 18 π electrons conjugated rings that they have in their structure, these can be efficiently used in molecular electronics, in the manufacture of optoelectronic devices, photonic devices, sensors, digital technology and photovoltaic cells [2]-[5].

A great number of MPcs are planar molecules, within the exceptions is the PbPc (Fig. 1 (a)). This molecule shows polymorphism. The predominant polymorphic structures are the monoclinic crystalline structure (Fig. 1 (b)) and the triclinic (Fig. 1 (c)) [6], [7]. In the first one, the PbPc molecules are stacked in parallel to the axis-*c* in linear columns, giving a one-dimensional conductive behavior, while in the second, the molecules are stacked along the axis-*a* orienting their concave and convex sides alternately. The PbPc shows high thermal and chemical stability, so it has been applied in thin film form, as field effect transistors (FETs) [6]-[8]. Other possible applications of PbPc films are in switch optimization, gas sensors and solar cells [6]. It is estimated that the efficiency of use in the applications described above may increase, since for example the conductivity of PbPc increases when thyoalkil groups are added [9]. PbPc with a controlled structure, forming thin films, present the properties that make them useful in electronics industry.

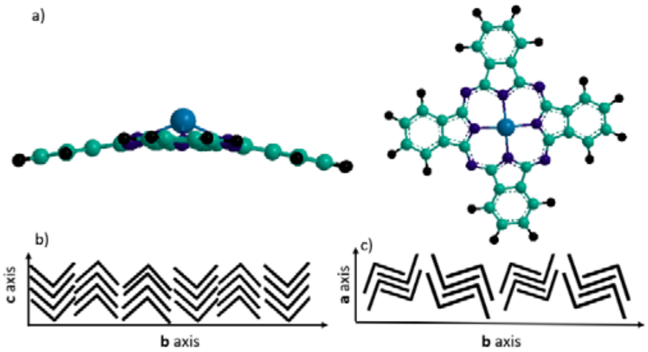

Fig. 1 (a) Representation of the molecule of PbPc; (b) and (c) molecular arrangements in monoclinic and triclinic form along its axis-b

Allenes are molecular materials discovered in the nineteenth

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century [10], characterized by containing a carbon atom which bears two double bonds with delocalized π electrons. In recent years, these molecular materials have been importantly considered because they can be used in optoelectronic devices [11]. They can participate as intermediaries in organic synthesis [12] and offer applications related to their interesting electronic and optical properties which are associated to the valence band electronic configuration [13]. For example, the tetrasubstituted allenes combine both structural and electronic properties possessed by the carboxylic acids that comprise them. These allenes are synthetic targets due to the presence of different functional groups on their structure, which can then result in a huge range of compounds and properties [14]. In studies on allenes, it is important to include their function as organic semiconductors, also called molecular semiconductors. Currently, photovoltaic devices that have gained great importance for their use in solar energy, are manufactured by inorganic semiconductors such as silicon. Silicon and the rest of the inorganic semiconductors are normally processed by polluting and expensive technology that must be replaced using molecular semiconductors. Allenes can be a viable option as organic semiconductors, especially because they can be used as dopants in MPcs, which are widely known for their semiconductor's properties. Therefore, the aim of this work is to use the allene compounds $C_{24}H_{26}O_4$ and $C_{24}H_{26}O_5$ (see Fig. 2) as dopants, to manufacture semiconductor films based on PbPc. It is intended to improve their optical and electrical properties for photovoltaic applications, like solar cells. In this study, the doping of PbPc was carried out and later, the semiconductor films were manufactured, structurally characterized, and analyzed in their optical and electrical properties.

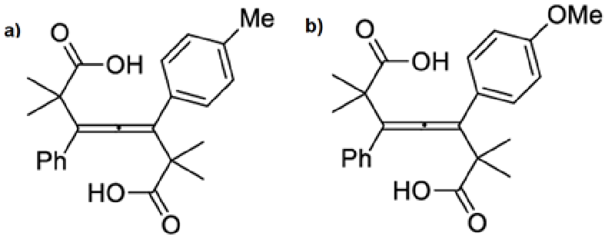

Fig. 2 Schematic representation of the investigated allene structures (a) $C_{24}H_{26}O_4$ and (b) $C_{24}H_{26}O_5$

II. EXPERIMENTAL METHOD

The PbPc and the compounds 3-(4-methylphenyl)-2,2.4,4-tetramethyl-5-phenylhepta-3,4-diene-1,7-dioic acid ($C_{24}H_{26}O_4$) and 3-(4-methoxyphenyl)-2,2.4,4-tetramethyl-5-phenylhepta-3,4-diene-1,7-dioic acid ($C_{24}H_{26}O_5$) (see Fig. 2) used as dopants were acquired from commercial sources (Sigma Aldrich) and were used without previous purification. The doping of the semiconductors OS1 (PbPc/$C_{24}H_{26}O_4$) and OS2 (PbPc/$C_{24}H_{26}O_5$) was carried out by means of bulk heterojunction (BHJ) using a pressure and temperature, controlled reactor. A reactor Monowave 50 was used, operating with a borosilicate glass vial, and manually closed by a cover with an integrated pressure (0-20 bar) and temperature sensor. The powdered material was heated at 150 °C to produce their phase change; kept in the reactor for 30 minutes, let cool in the reactor by lowering the pressure and temperature of the system and then filtered, purified from 1:1 ethanol-water and dried in a vacuum of $1x10^{-5}$ torr, at room temperature. The semiconductors based on PbPc were deposited in thin films, using high vacuum sublimation technique. The thin films were built onto different substrates to obtain their electrical and optical measurements: ITO coated PET, high resistivity monocrystalline n-type silicon wafers (c-Si) and Corning glass. Except for PET coated with ITO, substrates were submitted to a sonication cleaning process in different solvents and dried in vacuum. The deposition process took place using an evaporation equipment with a tantalum boat. The evaporation rate (0.3 Å/s), temperature (298 K) and pressure (1×10^{-5} Torr) in the vacuum chamber were the same for all the deposition processes. During the deposition processes, the films' thicknesses were monitored with a quartz crystal monitor. An IR spectroscopy was carried out to verify the vibrational modes of the organic radicals that integrate the doped semiconductor. The IR analysis was carried out using a Nicolet iS5-FT in KBr pellet and the film deposited on the silicon wafers. The semiconductor films on corning glass substrates were used to measure the UV-vis spectroscopy of the films with a Unicam spectrophotometer, model UV300, in the wavelength range of 200-1100 nm. Finally, PET/ITO/doped PbPc/CCP simple device was fabricated by sublimation process, under the same conditions of temperature and pressure. Electric conductivity measurements were performed by using the four wires method, in which the electric current is passed through the end wires and measures the potential difference between the central wires. The current-voltage (*I-V*) measurements were performed employing a programmable voltage source, a sensitizing station with lighting and temperature controller circuit from Next Robotics and an auto-ranging Keithley 4200-SCS-PK1 pico-ammeter were employed. I-V curves were obtained with the devices under darkness, white, red, orange, yellow, green, blue, and ultraviolet lights in the range of 0 V to 20 V and at room temperature.

III. RESULTS AND DISCUSSION

IR spectroscopy was realized with the intention to clarify if during the thin film deposition, a material degradation occurred, as a result of the compound sublimation and posterior deposition on different substrates. Table I shows the signals of the doped semiconductors in different forms: pellet and thin film. Regarding the presence of PbPc, the bands observed around 1611 cm^{-1} and 1476 cm^{-1} result from a C=C stretching mode. The peaks responsible for carbon/nitrogen stretching and bending occur at 1585 cm^{-1} and 1331 cm^{-1}. The peaks located around 1160 cm^{-1}, 1119 cm^{-1} and 780 cm^{-1} result from the interaction of carbon with the peripheral-ring hydrogen atoms [10]. In addition, the IR spectra show the signals of the allenes: The value around 3060 cm^{-1} for each doped semiconductor corresponds to the vibration of the O-H bond of the carboxylic acid C(O)O-H, at 2980 cm^{-1} likewise the vibration related to the C-H bonds of the methyl substituents is found. Finally, with the substituent methoxyl, it is possible to see two bands

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corresponding to the carbonyls of the two carboxylic acids; in the case of the substituent methyl, the two bands of the carbonyls overlap, showing a slightly wider band at 1700 cm^{-1} (C=O). According to Table I, the values obtained for the films change slightly with respect to the values obtained for the pellets. This is because during the deposition process, stress concentrations are generated between the molecules that integrate the film; this brings therefore, a change in the angles and energy of intramolecular bonds. However, the results suggest that low pressure evaporation is an adequate molecular process for device fabrication, due to the fact that it does not change the compound chemical composition. Furthermore, the IR absorption technique was used to identify the phase nature of the PbPc powder and thin film [11]. The main spectral features distinguishing the different crystalline forms of PbPc were found to lie in the region 800-700 cm^{-1}. The α-form gives rise to vibrations at about 770 cm^{-1}, while the β-form produces vibrations at about 720 cm^{-1} [11], [12]. From these studies, we can determine the phase and any significant chemical change which may occur in these materials during the thermal evaporation. Initially, spectra pellet of PbPc showed two crystalline forms, when the spectra of the film were analyzed, the crystalline structures α and β remain. This is an important aspect to consider since the deposition process by high vacuum sublimation generates thermal gradients between the high temperature of the doped phthalocyanine, and the room temperature to which the substrate is exposed. This derives in a nucleation and growth of the semiconductor on the substrate that can change the crystalline structure of the material.

TABLE I
CHARACTERISTIC FT-IR SIGNALS OF (OS1, OS2) IN PELLETS AND THIN FILMS (CM^{-1})

Sample	v(C=C)	v(C=N)	v(C-H)	v(C(O)O-H)	v(C=O)	α-form	β-form
OS1 Pellet	1608	1584	1160	3062	1699	771	724
	1477	1331	1119		1600		
	1617		781				
	1564						
OS1 Thin Film	1607	1483	1160	3060	1699	771	721
	1478	1328	1119		1600		
	1616		781				
	1569						
OS2 Pellet	1610	1585	1160	3058	1701	770	724
	1479	1331	1119				
	1618		782				
	1563						
OS2 Thin Film	1607	1584	1162	3059	1700	771	724
	1479	1329	1115				
	1620		785				
	1559						

UV-vis spectroscopy was performed in the semiconductors OS1 and OS2 on thin films, to obtain its optical activation energy (optical gap). Optical properties of the PbPc semiconductors with a π-conjugated system depend strongly on the molecular orientation and crystallinity. In Fig. 3 are illustrated the UV-vis spectra of the investigated films deposited in glass substrate. For OS1 film there are clearly two peaks in the visible region: the first one a high energy peak, around 620 nm, and the second, a low energy peak, around 750 nm. These two peaks correspond to the Q-band transition of the MPcs, generated by their α-form [13], [14]. Additionally, the high-energy peak of the *Q*-band is assigned to the first π-π* transition on the Pc macrocycle and the low-energy peak of the *Q*-band is explained, as a second π-π* transition, as an excitation peak, as a vibrational internal interval and as a surface state [12], [15], [16]. On the other hand, from the examination of the spectra, all the films have the *Soret*-band in the near-ultraviolet region around 350 nm [11]. The electronic n-π* transition in the PbPc corresponds to a *B*-band or *Soret*-band which gives the fundamental absorption edge [17], [18]. The high intensity *Soret*-band, together with the two “shoulders” at 400 nm and 440 nm, is an indication of the β-form in the PbPc [19]. The UV-vis spectrum of the OS2 film indicates the low optical properties in the semiconductor film. Apparently, the methyl substituent on the allene and its low polarity are the causes of the null absorbance in the film. These results must be complemented with the evaluation of the bandgap and the electrical behavior in the film, before deciding if it has the possibility of being used in the manufacture of photovoltaic devices.

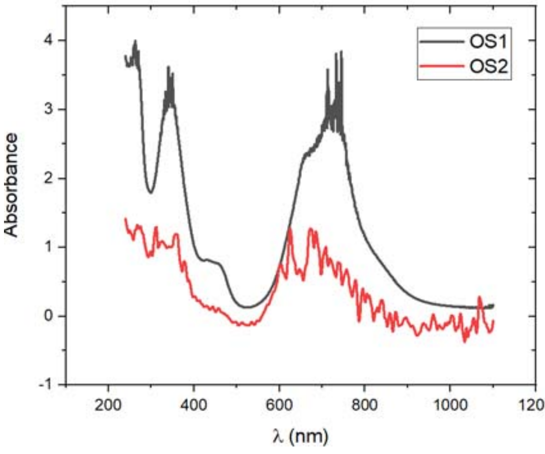

Fig. 3 UV-vis spectra of OS1 and OS2 thin films

The optical gap is a parameter to consider in the semiconductor films that is intended to use in the design and construction of optoelectronic devices. In the current study, the optical gap was obtained from UV-Visible spectroscopy and Tauc’s model [20]. This semi-empirical model is based on the fact that for thin films, the optical transition is independent of the photon energy, therefore it is possible to extrapolate linearly the graphs of the absorption coefficient of the material (α) against the photon energy ($h\nu$). The absorption coefficient describes the depth of penetration of radiation into a bulk solid. The absorption coefficient is defined by the Beer–Lambert Law and can be calculated from the optical transmittance.

$$\propto = -\ln\frac{T}{t}$$

where T is the transmittance and t is the film thickness. The optical gap was determined from the spectral dependence of the absorption near the fundamental absorption edges. The α is well described by Urbach's relation [20].

$$\propto hv = \beta(hv - E_g)^n$$

where *hv* is the energy of incident photons and E_g is the value of the optical gap corresponding to transitions indicated by the value of *n*, for direct transitions, $n = ½$, and for indirect transitions $n = 2$. Thus, the optical gaps for both transitions could be determined by the extrapolation to zero of the linear regions of the $(\alpha h\upsilon)^2 = f(h\upsilon)$ and $(\alpha h\upsilon)^{½} = f(h\upsilon)$ curves, Eg_d and Eg_i, respectively [21]. The factor β depends on the transition probability and can be assumed to be constant within the optical frequency range. Table II shows the values of both the direct and indirect optical gap for the doped PbPc. It can be observed that the dominant activation energies for this type of materials are indirect. These results are to be expected, considering that the high vacuum evaporation method, used to manufacture the thin films, generates wide thermal gradients that produce a disorder in the internal structure of the film. E_g values obtained, place the manufactured films within the range of low bandgap semiconductors [22], [23].

TABLE II
DIRECT AND INDIRECT OPTICAL ACTIVATION ENERGIES OF THE FILMS

	Eg_d	Eg_i
OS1	1.47 eV	1.33 eV
OS2	1.55 eV	1.29 eV

Based on the results obtained, a design of dispersed heterojunction was proposed according to the scheme shown in Fig. 4. In such design, the dispersed heterojunction film is deposited on ITO (anode) which in turn is deposited on the PET substrate. The circuit is closed in such a way that the cathode is deposited on top of the active layer, in this case, the CCP. To measure electrical performance, the devices were subjected to current-voltage *(I-V)* measurements. Voltages in a range of 0 V to 20 V were applied to measure its current response. It is worth mentioning that the measurements were made in the devices at room temperature under the influence of radiation between visible and ultraviolet light. OS1 film (see Fig. 5 (a)) shows a similar electrical behavior at all applied lightning conditions, since all the radiations generate practically the same current at a given voltage. All the curves have a constant slope and its current increases linearly with respect to the voltage applied, this means OS1 is an Ohmic material. Film OS2 (see Fig. 5 (b)) also shows that all radiation generates almost the same effect under different lightning conditions. The material reaches a maximum current of 0.020 A at 2.5 V. However, it becomes an electrical insulator at high voltages, since there is a saturation after 2.5 V. Film OS2 shows a better overall behavior because it reaches higher current values than OS1 with lower voltages. Therefore, OS2 is the best candidate for making photovoltaic devices.

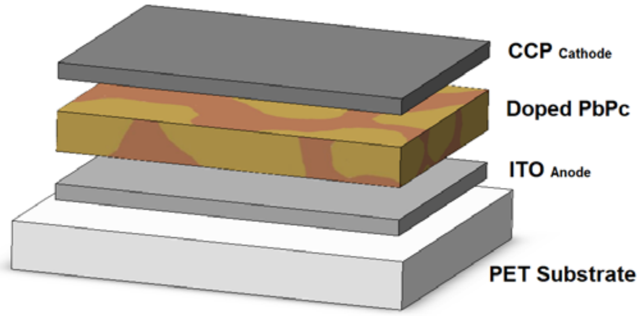

Fig. 4 Schematic representation of the flexible structures

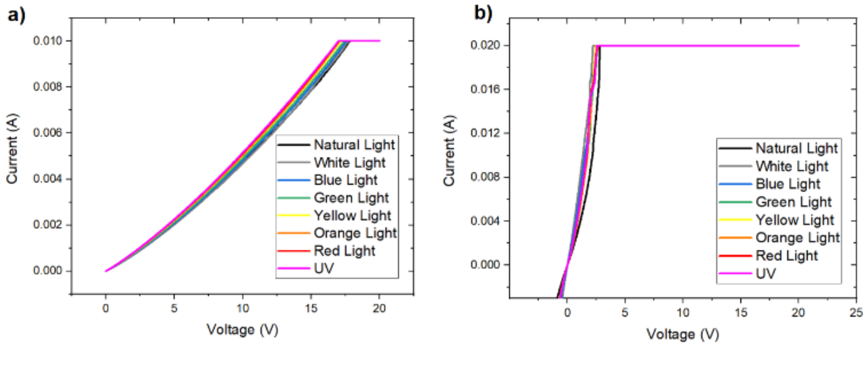

Fig. 5 Plot of the electric behavior of the films obtained from the flexible devices with (a) OS1 and (b) OS2

IV. CONCLUSIONS

Allene compounds $C_{24}H_{26}O_4$ and $C_{24}H_{26}O_5$ were used as PbPC dopants to manufacture organic semiconductors, to improve its optical and electrical properties and then be able to use it in the manufacture of thin film structures for photovoltaic applications. The high-vacuum evaporation process had no effect in the intra-molecular bonds, with this we can ensure that the chemical composition of the films did not change. The optical band gap was calculated by UV-Vis spectroscopy for the doped semiconductors, and the values were found to be 1.47 eV to 1.55 eV for direct transitions and 1.29 eV to 1.33 eV for indirect transitions. The differences in the gap are related to the differences in the structure of the molecules of the allenes. The bandgap values obtained for the two semiconductor films, places them in the low bandgap semiconductor range. The I-V relationship was evaluated under different lighting conditions in a range of 0 V to 20 V. This showed in both films that all the light radiations generate practically the same current. Film OS1 showed an Ohmic behavior, while OS2 reaches higher current values at lower voltage levels. Therefore, OS2 is a more efficient device for making photovoltaic devices. The results show that the new molecular semiconductors can be used in the manufacture of optoelectronic devices.

ACKNOWLEDGMENT

The authors wish to thank the technical assistance of Mariana Sánchez Araiza and financial support from Universidad Anahuac México, project number NNAIASEVM16070616.

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