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Original Article

Synthesis of Polyaniline Dye Pigment and Its Study in Dye-Sensitive Solar Cells

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Abstract *- The aim of this study is to synthesize and physicochemically study a semiconductor polymer dye pigment used as a dye pigment for dye-sensitive solar cells, which are currently considered third-generation solar cells as one of the alternative energy sources. to achieve this goal, polyaniline was synthesized in an acidic environment in the presence of aniline and ammonium persulfate by the oxidative polymerization method. the synthesized semiconductor polymer dye was first used in the preparation of solar elements sensitive to new dyes from pigment. Measurements in the prepared paint-sensitive solar cells were performed outdoors for 12 days using a DT 9205A multimeter. the highest values of voltage and current were recorded on the first day as 700.6 (mV), 65.45 (mA) and the lowest as 335 (mV), 26.6 (mA) on the last day of measurement was found.*

Keywords **-** *Dye-sensitive solar cell, Photophysics, Polymer films, Polyaniline, pigments.*

1. Introduction

Many high-molecular compounds are semiconductor polymer substances, and today these properties are becoming the focus on intensive research by many research scientists due to their excellent photochemistry of polymeric materials [1]. Semiconductor polymer materials are used mainly as nanocomposite materials in solar cells, microelectronics, optical materials, photoelectron engineering industry based on polymer materials for energy production [2]. Electrically conductive polymers are also called organic metals because they do not contain metal atoms, but their electrical conductivity covers the permeability region of inorganic semiconductors, and is close to the permeability limits of metals [3, 4].

Semiconductor polymers are used in the flexible, and can produce as environmentally friendly manufacturing. Nowadays, suppernanocompositors on the bases of semiconductor polymers are used in organic solar batteries, biosensors, the flexible transparent displays, LCD screens based on liquid crystals, for example: computer monitors, televisions, telephones, digital cameras, e-books, navigators, calculators, clocks and we can say that indium is also giving as a substitute for thallium oxides for liquid screens in electronic devices used in other demonstrations [5, 6]. the electrical conductivity of metals, and semiconductor polymers are differenr from each other not only in quantity sides but also in high qualitative aspects. for example, as the temperature increases, the electrical resistance of metals increases (electrical conductivity decreases), while that of semiconductors decreases, the electrical resistance increases [7]. the electrical conductivity of metals does not change under the influence of light, however, that of semiconductors may change many times. the electrical and optical properties of semiconductors are incomparably more sensitive to external influences than those of metals. Polymer semiconductors different in their permeability illumination as anisotropic semiconductor coatings and photovoltaic devices [8].

Semiconductor polymers will be used to open new production lines, as well as in the manufacture of photovoltaic devices that provide high conversion efficiency at low cost, also we should taking into account their electrical and physical properties. New nanostructured conductive polymer forms with large photoelectric field, high sensitivity, and diffusion provide new ideas in nanocomposite technology [9, 10]. Research in this area has shown that organic semiconductor polymer composites have a million times higher conductivity than conventional semiconductor polymers. at present time, semiconductor polymers and organic semiconductors are used in many fields of electrical engineering and electronics. Because organic polymers are cheaper, easier to manufacture, lighter, as well as more flexible than their inorganic analogues, they are now of great interest to many research scientists [11, 12]. Common representatives of the class of electrically conductive polymers: polyamide, polyaniline, polypyrrol, polyacetylene, polyphenylene, polytophene, polyvinyl chloride, poly (3,4-ethylenedioxythiophene) and others. These polymers have high electrical conductivity combined with high stability. Therefore, the existing and possible range of application of polyaniline, polytiophene, polyacetylene, polyvinyl chloride, polypyrrole, poly (3,4-ethylenedioxythiophene) and similar semiconductor polymers is very wide [13,14,15].

These high molecular weight compounds consist of linear stereoregular macromolecules with a conjugated double bond. the properties of electrically conductive polymers are similar to those of inorganic semiconductors [16]. Given that photovoltaic solar cells that use semiconductor organic polymers in absorbing and converting light to convert sunlight into electricity have great potential, we are currently conducting intensive research [17].

2. Materials and Methods

2.1 Materials

Raw materials: aniline, ammonium persulfate, 1m hydrochloric acid or sulfuric acid, potassium per sulfate $K_2S_2O_8$, potassium bichromate $K_2Cr_2O_7$, potassium bromate K_3BO_3, potassium iodate KIO_3, hydrogen peroxide H_2O_2, ammonia, dimethylformamide and distilled water.

2.2 Polyaniline Synthesis

Polyaniline synthesis is divided into two different methods: chemical and electrochemical methods. the chemical method is on the basis of the oxidation of polyaniline under the influence of oxidants in an acidic environment. Initially, the reactants were weighed in a 1:1 mole ratio of ammonium per sulfate and aniline. Added 75 ml of 1M HCl to the weighed aniline, dissolve in a 400 ml beaker and store at 25 °C. the mixture by mixed the addition of hydrochloric acid solution is up colourless. Then, a 10% solution of the measured ammonium persulfate was dissolved in 70 ml of 1M HCl in another 400 ml beaker, the mixture was also stored at 25 °C, and the reaction was carried out at the high temperature by slowly dripping over an aniline and hydrochloric acid mixture for one minute. the temperature was kept constant at 25 °C while stirring the reaction at medium speed for 30 min. the fact that the color of the solution gradually turns blue and then turns reddish-green indicates the formation of polyemeraldin, ie an intermediate product. After another 30 minutes, the precipitate collects on the Buyuxner funnel and is washed several times with a water aspirator. the precipitate washed with distilled water is filtered, washed again with hydrochloric acid and dried in a drying cabinet for 3 hours. the resulting product is a polyemeraldine salt, which is green in color. to obtain the polyemyraldine base, the polyemyraldine hydrochloride was processed in an aqueous solution with ammonia water for 1 h and filtered using a Buckner funnel to separate the polyaniline powder from the resulting solution, then dried for 12 h in a vacuum drying oven at 60ºC to rehydrate the filtrate. During the polymerization process, the colour of the reaction mixture changes from blue to dark blue. the general appearance of the polymerization reaction of polyaniline can be expressed as shown in Fig.1.

The synthesis of high-permeability polymer substances and their study in various fields is one of the current tasks before us. the unique physical-mechanical, chemical and electrical properties of semiconductor polymers allow these materials to be used in various fields of technology. Many researchers suggest that the field of synthesis of organic semiconductor polymer substances is giving us great opportunity, and will be an energy source for other types of energy sources [18, 19].

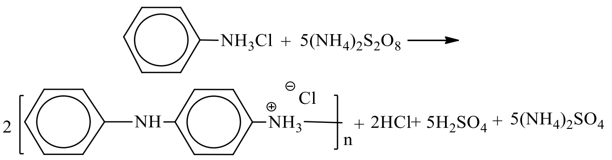


Fig. 1 Formation reaction of polyaniline

2.3 Characterization

the morphologic properties of the synthesized polymer (JEOL JSM-5800LV) were studied using IR spectroscopy to obtain information on the presence of functional groups in the scanning electron microscope (SEM) and the polymer, for what length it is located in the field. Also, the thermal analysis of the differential thermal and pigment was carried out by thermogravimetric methods of obtaining polyaniline paint (DTG-60, DTA-TG APPARATUS SHIMADZU simultaneously, Japan). in addition, the analysis of X-ray Space analysis of polymer dye pigment (XRD-6100 X-RAY SHIMADZU Defraktometer) was taken tick. the power output of the solar element, obtained on the basis of the most dye pigment, was measured using a digital multimeter combination of Щ300 and, importantly, polyaniline DT 9205A with the values of voltage (mV) and current (mA).

2.4 To study the effect of factors influencing the process of polyaniline synthesis

The analysis of the effect of aniline concentration, reaction time and reaction temperature on the yield of the product formed by the process of obtaining polyaniline in the presence of aniline, ammonium persulfate, 1M hydrochloric acid, and ammonia solution is given in Table-1

Table 1. To study the effect of concentration, time and temperature of reactants on the yield of the product

the mol ratio of initially products	time of continue reaction, (hours)	Tempe-rature dependence of the reaction, ºC	the reaction efficiency %
0.1:1:1	3	-10	75
0.5:1:1	2.5	-5	80
1:1:1	2	0	89
1.5:1:1	1.5	10	70
2:1:1	1	25	72

During the experiments, aniline and ammonium persulfate were obtained in different proportions. in the process, the results of the reactions carried out with different proportions of aniline to ammonium persulfate to hydrochloric acid were lower than those obtained with both low and high proportions of aniline. in a 1: 1 mol reaction of ammonium persulfate with aniline, it was found that the yield of the process was so high (Fig.2).

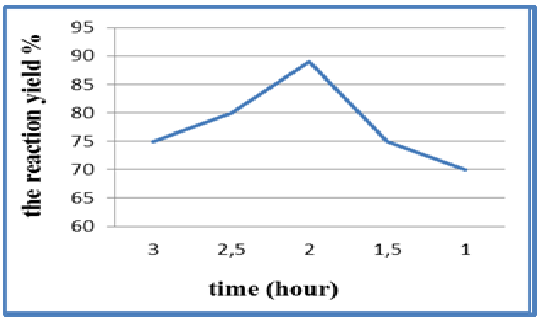


Fig. 2 The effect of molar ratios of starting materials on the reaction yield during the production of polyaniline

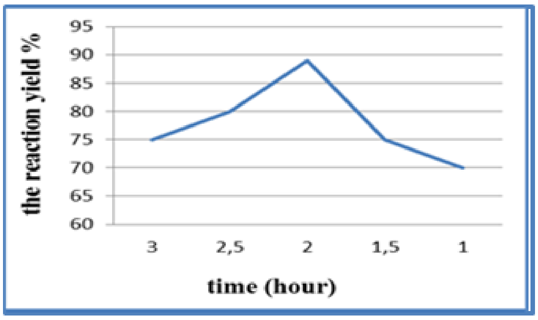


Fig. 3 The effect of temperature on the reaction yield during the production of polyaniline

The experiments were performed at different temperatures. the yield of the reactions performed at different temperatures in the process was tested at both low and high temperatures, and the results are shown in Fig.3. the yield of the product formed by the reaction of aniline with ammonium persulfate at 0°C was found to be high.

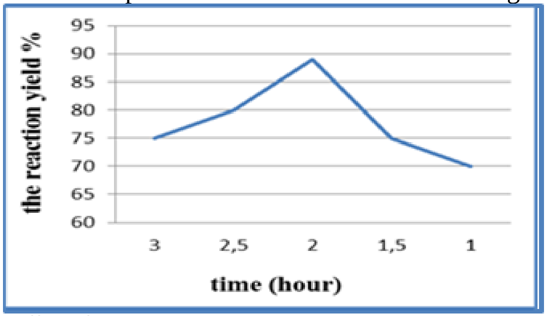


Fig. 4 Effect of time on reaction yield during polyaniline extraction

The experiments were performed at different times. the yield of the product obtained at different time intervals during the reaction, i.e., from 1 h to 3 h, was tested and the results are shown in Fig.4. the yield of the product formed by the reaction of ammonium persulfate with aniline for 2 hours was found to be high.

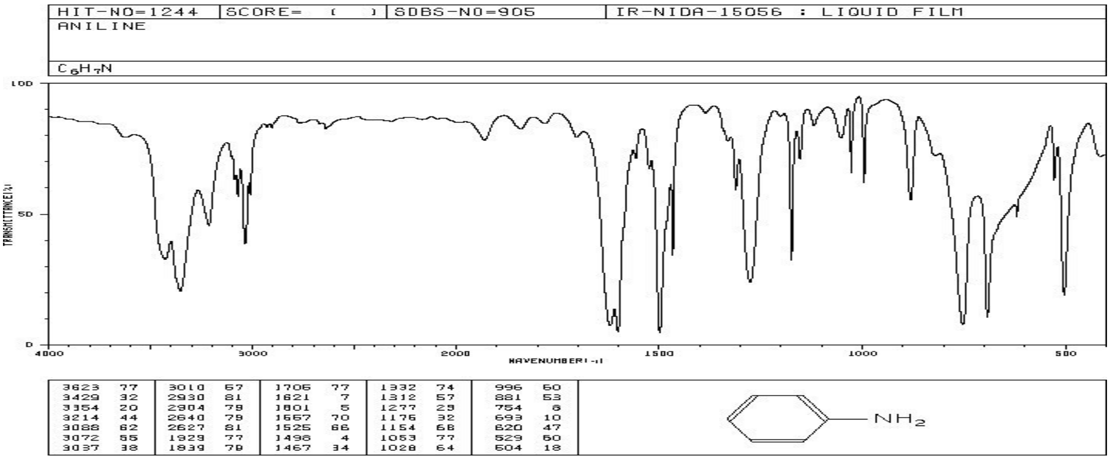
3. Results and Discussion

3.1 IR Analysis

The morphological properties of the obtained polyanilin were measured and analyzed by infrared spectroscopy to obtain information about the presence of functional groups in the SEM scanning electron microscope and the polymer, the wavelength range, and the results of the study are shown in Fig.5 (a - b). in the IR spectra of the resulting new polymer, it was observed that the absorption frequencies of 3483 cm^{-1} belonging to the N-H -group are in the range of high intensity of valence oscillation type. Absorption frequencies were observed in the 2927-3000 cm^{-1} area (Ar) = C-H group, and in the weak 1655 cm^{-1} area -C = N- valence intensive group.

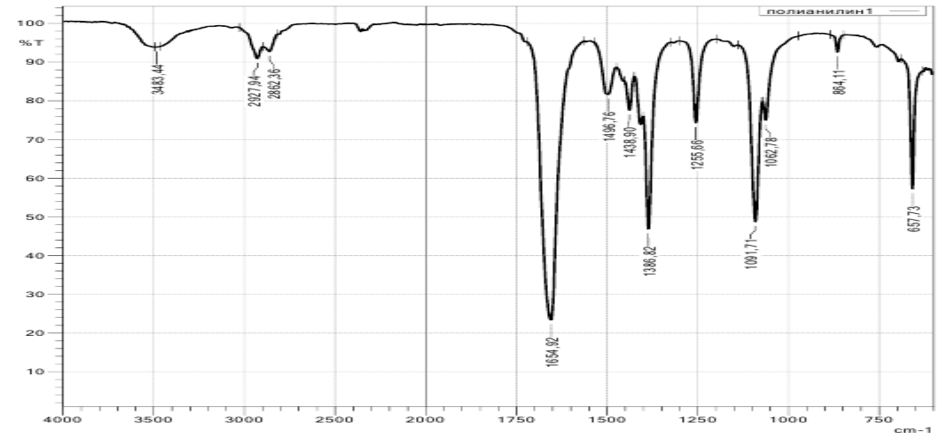
The absorption frequencies around 1500–1600cm^{-1} are related to the elongation of the CN bonds in the kinetic rings with gasoline [20, 21]. Given that peaks in the region of about 1300 cm^{-1} are related to the presence of aromatic amines present in all types of polyanilines discussed here, the intensity of these absorption frequencies is about the oxidation state of polyaniline, if they exhibit similar intensity, the colour of synthesized polyaniline is reddish-blue associated with the appearance of green [22, 23].

The absorption frequencies of the synthesized polyaniline appear to have wider absorption frequencies instead of higher intensity, which is due to the presence of the groups analyzed above at higher concentrations. There is evidence that the resulting polyaniline sample has the lowest point of low intensity in the region where the absorption frequency extending around 3400 cm^{-1} depends on the O-N bond due to the presence of the O-N bond in the solvent.



3623	77	3010	67	1706	77	1332	74	996	60
3429	32	2930	81	1621	7	1312	57	881	53
3354	20	2904	79	1601	5	1277	29	754	8
3214	44	2640	79	1567	70	1176	32	693	10
3088	62	2627	81	1525	66	1154	68	620	47
3072	55	1929	77	1498	4	1053	77	529	60
3037	38	1839	78	1467	34	1028	64	504	18

(a)

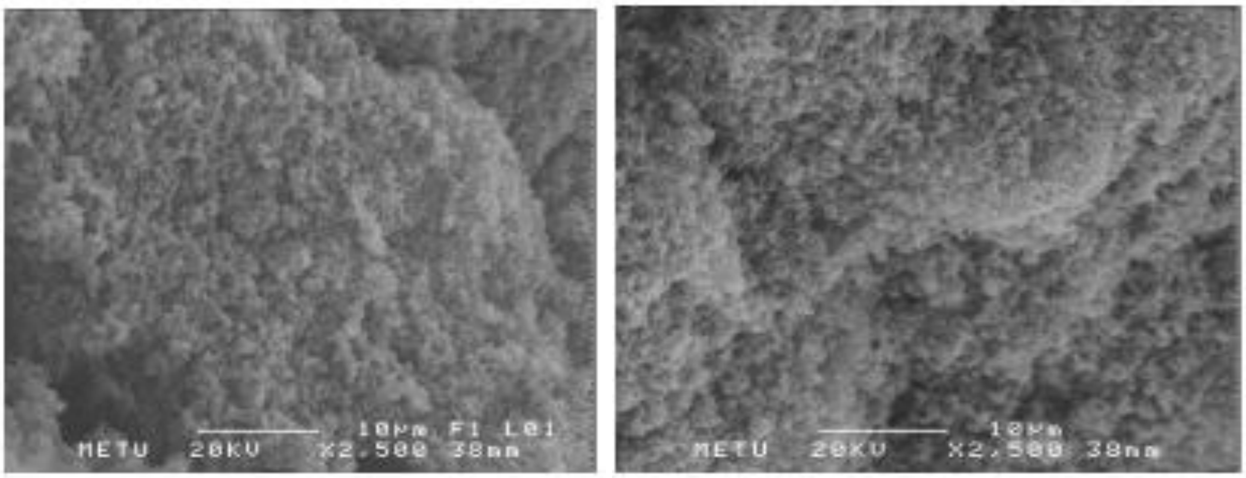


(b)

Fig. 5 IR-spectra of a-aniline and b-polyaniline were given

3.2 SEM analysis

Scanning electron microscopy (SEM) allowed us to see that the polymer morphology was similar to other polymers described in the literature. SEM photographs show an image of the polymer magnified 500 times Fig.6 (a - b). AS can be seen from these photographic images, it consists mainly of unevenly placed grains and sharp corners of the edges, moreover, the structure is more porous. This suggests that the length of the polymer chains increases with decreasing synthesis temperature and therefore yields a large molecular weight, as proven by many researchers [24].

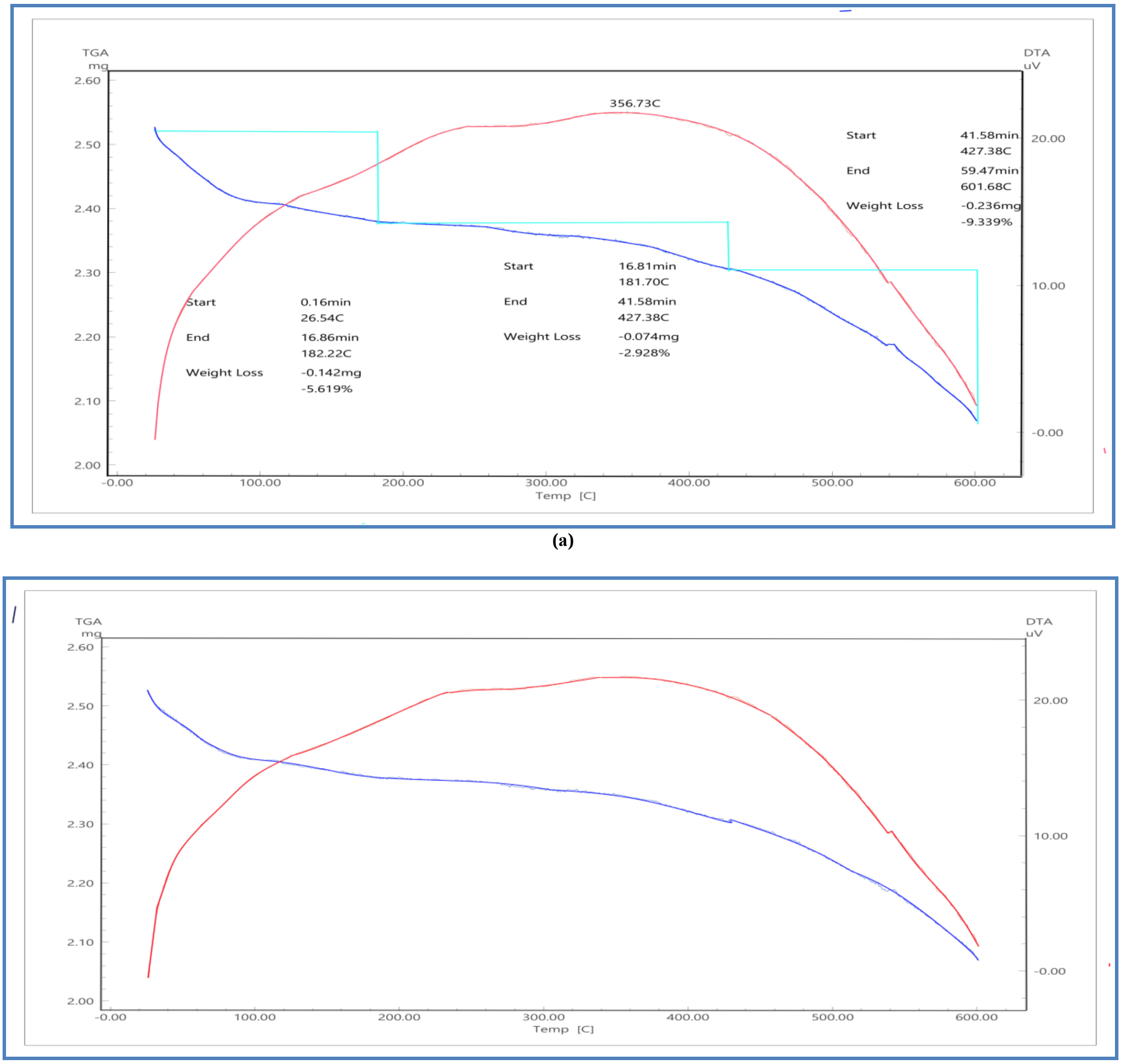


(a) **(b)**

Fig. 6 Scanning electron microscopy image of synthesized polyaniline

3.3 Study of Thermal Analysis of Synthesized Polyaniline

the thermal stability of the synthesized semiconductor polyaniline pigment was analyzed by differential-thermal and thermogravimetric methods (DTG-60, SIMULTANEOUS DTA-TG APARATUS SHIMADZU, Japan). the resulting derivatogram is shown in Fig.7(a - b), which consists of 2 curves. Thermal analysis is performed to obtain similar information about the thermal properties of the polyaniline under study, its temperature tolerance, how it changes under the influence of temperature, i.e., at what temperature and in return for mass loss. Thermal analyzes were performed in a temperature range of 20 °C to 600 °C, in an argon environment at a rate of 10 degrees per minute.



(a)

(b)

Fig. 7 Study of thermal analysis of synthesized polyaniline

The endothermic curve of the obtained derevatogram was mainly performed in 3 intensive decomposition temperature ranges. the 1st decomposition interval lasted 16.86 minutes from 45 °C to 182 °C and the mass loss was 0.142 mg 5.62%. the 2nd decomposition interval ranged from 182.22 °C to 427 °C for 41.58 minutes with a mass loss of 0.074 mg 2.93%. the 3rd decomposition interval ranged from 427.38 °C to 601.68 °C for 59.47 minutes with a mass loss of 0.236 mg 9.4%.

This means that the mass is reduced by 2-3% due to moisture loss up to 100 °C. Subsequent mass loss begins at 182 °C, with a possible breakdown of the acid bound to the water. Above 350 °C the substance liquefies and above 427.38 °C decomposition occurs in the basic structure of the substance.

3.4 Study of X-ray phase analysis of synthesized polyaniline

The synthesized polyaniline dye pigment (XRD-6100 SHIMADZU X-RAY Defractometer) was analyzed on an X-ray spatial analyzer apparatus. Measure the average distance between layers or rows of atoms of a polymeric material, determine the direction of a single crystal or molecule, determine the crystalline or amorphous structure and quantity of an unknown material, measure the size, shape and internal structure of small crystalline regions, and analyzed the quality and quantity of matter allowed to take.

Table 2. Information about Diffractogram Data of Polyaniline

№	2theta- angle of scan [° 2th]	Peak [cts]	FWHM wide of integral [°2θ]	d- the distance ofamong the planes [Å]	I- density of peak
1	43.98	478.85	0.2400	2.0572	1000.00
2	64.34	501.11	0.2800	1.4468	897.00
3	77.46	400.63	0.2400	1.2312	836.66

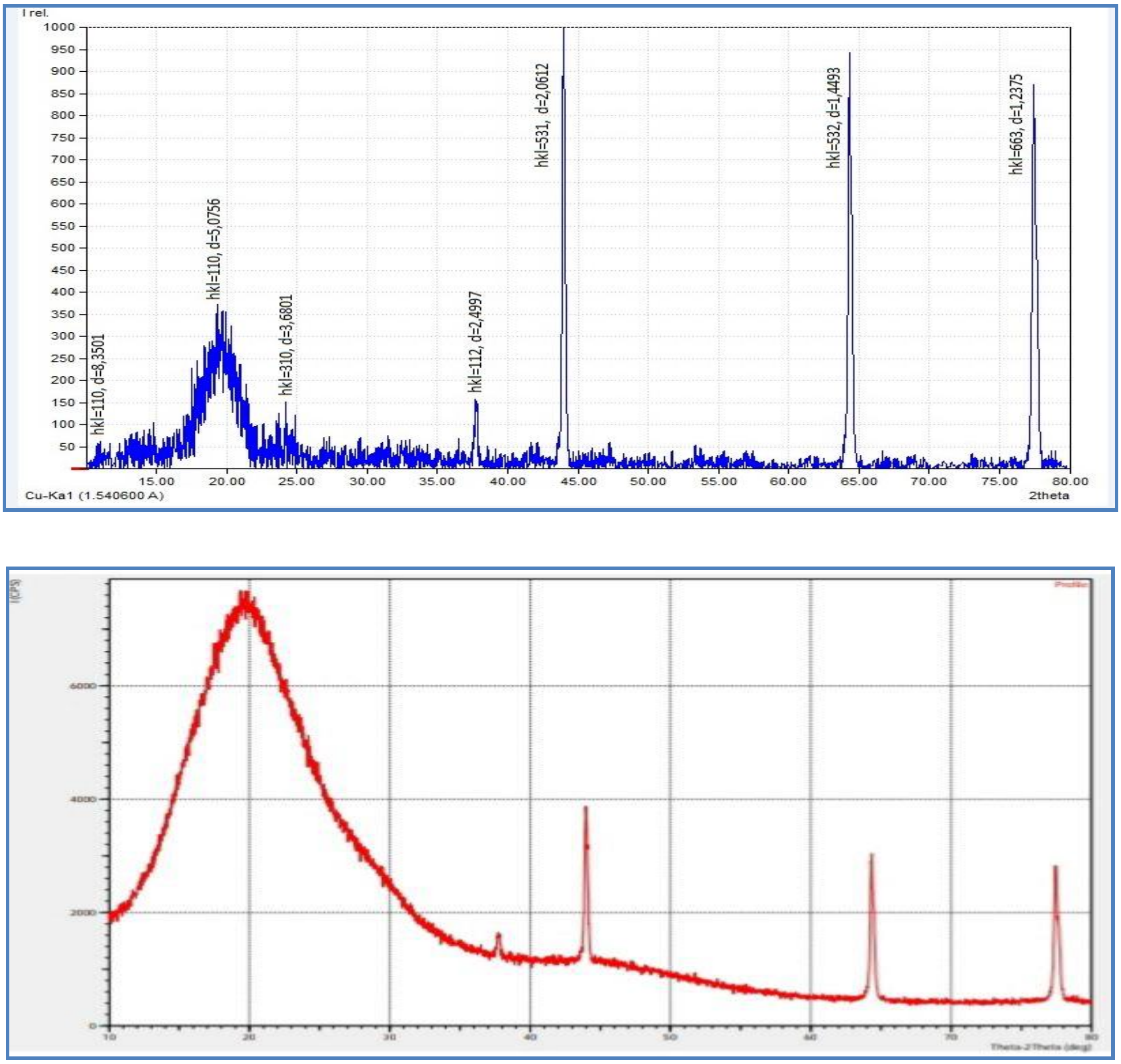


Fig. 8 Study of X-ray phase analysis of synthesized polyaniline

On the average crystal size and shape of nanoparticles are often used by the (Scherrer) method to study X-ray phase analysis for experimental study of their distribution laws [25].

Coherent scattering area (KTM) dimensions (nanocrystal dimensions) are calculated classically using the Williamson-Hull eqn.1: [26].

$$B(2\theta)=\frac{K\lambda}{L\cos\theta} \qquad (1)$$

Here: V (2θ) is the average size of the crystals (nm) the coefficient form of K-dimensional particles (Williamson-Hull) ranges from 0.68 to 2.08. for spherical crystallites with cubic symmetry, K = 0.94 [27].

L- FWHM (Full Width at Half Maximum) is the representation of the integral width in a diffractogram. (in 2nd radian and unit) the l-ray wavelength is Cu Ka = 1.540600 Å [28]. costh is the cosine of X-rays of the diffraction angle. (Breggovsky corner). costh = 0.2 value is obtained.

Polyaniline is calculated from the values given in the formula when calculating the particle size according to the Williamson-Hull eqn.2: [29].

$$B(2\theta)=\frac{K\lambda}{L\cos\theta} \tag{2}$$

Example:
B (2θ) =0,94•1.540600/ 0.2400•0,2=30,1700

Table 3. Calculated the particle size of polyaniline according to the Williamson-Hull equation

№	2theta- angle of scan [°2θ]	FWHM wide of integral [°2θ]	*d* (nm)- on the average size of the crystals	*d* (nm)on average
1	10.98	0.2400	30.1701	28,73
2	26.60	0.2800	25.8600	
3	37.74	0.2400	30.1700	

According to the Williamson-Hull equation, polyaniline was calculated to have an average particle size d (nm) of 28.73, and the particle size was proved to be a nano particle.

It was found that 86.01% of the polyaniline polymer molecule has a crystalline and 13.99% amorphous structure Table-4.

Fig.8 shows the results of measuring the solution of polyaniline polymer dye pigment in dimethylformamide under a visible light spectrum of 190–1100 nm on a UV-Vis spectrophotometer. Polyaniline polymer dye pigment was found to have a high degree of assimilation in the range of visible wavelengths ranging from 400 nm to 600 nm. the highest absorption was found to be at a wavelength of 450 nm. AS a result, the polyaniline polymer dye pigment was used as a dye for dye-sensitive solar cells due to its ability to absorb photons from light.

Table 4. Results of determination of amorphous and crystallinity of polyaniline polymer molecule

Profile area	Counts	Amont
Total area	203501	100 %
Diffraction peaks	175039	86.01 %
Background	28462	13.99 %
Instrumental background	0	0.00 %
Amorphous phases	28462	13.99 %
Degree of crystallinity (DOC) = 86.01 % **Amorphous content (weight %) = 13.99 %**		

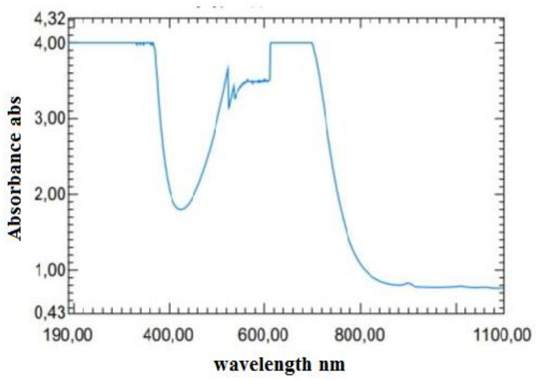


Fig. 9 The ratio of the wavelength of light absorption of a solution of polyaniline in dimethyl formamide on a UV-Vis spectrophotometer

3.5 Volt-Ampere Properties of Polyaniline Dye Pigment

Research

Fig.10 and 11 below show the results obtained by measuring the power output, i.e., voltage (mV) and current (mA) values for 12 days using a DT 9205A digital millimeter on contact based on a semiconductor polymer pigment. A solution of the on the base of polyaniline dye pigment in dimethylformamide was used during the testing of the sunlight absorption properties of the polyaniline-based dye pigment. AS a result of immersing the semiconductor polymer pigment solution in a polymer paint pigment solution as an assimilation semiconductor layer using titanium dioxide on one side of a transparent glass plate, the absorption layer of this titanium dioxide suspension is formed by contact with the paint pigment solution. It is very important to obtain more accurate voltage and current values of the volt-ampere properties of the semiconductor polymer pigment we recommend for 12 days. This is because it is an important indicator for studying the stability of the pigment when applying the polymer pigment to dye-sensitive solar elements [30, 31].

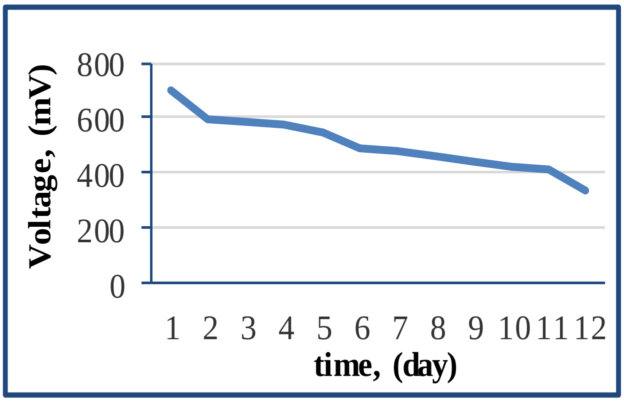


Fig. 10 Polyaniline-based paint is the power (mV) generated by a sensitivesolar cell

It was noted that the method of obtaining the dye pigment we used was simple and resistant to external aggressive environments, most importantly the power output volt-ampere values were higher than the values given in the literature [32, 33]. During our measurements, the values of power output voltage and current varied slightly depending on the daily weather conditions and part of the day, taking into account the measurements taken at different parts of the day and giving their average values. from the above results, the highest values of voltage and current were recorded on the first day as 700.6 (mV), 65.45 (mA) and the lowest as 335 (mV), 26.6 (mA) on the last day of measurement.

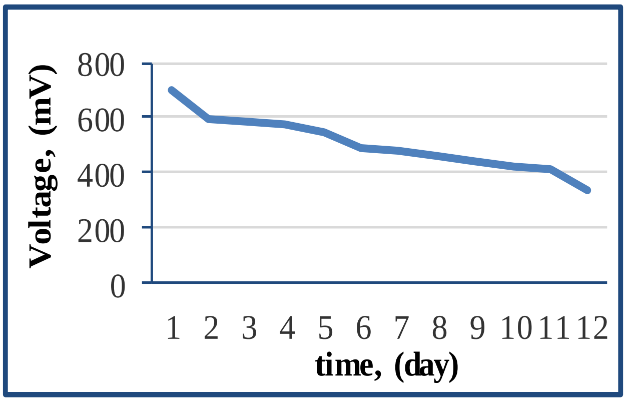


Fig. 11 Polyaniline-based paint is the power (mA) generated by a sensitive solar cell

Despite its small efficiency, this polyaniline dye pigment has shown its strength as an effective semiconductor dye pigment for the new generation solar cells. AS a result, we recommend polyaniline dye pigment as an alternative semiconductor polymer pigment in the preparation of new generation of dye sensitive solar cells. the results of the study show that the synthesis temperature affects the nature of the solvent and the efficiency of the pH power output of the medium [34].

Synthesis, and research of semiconductor polymers give us excellent photochemical properties, high energy conversion, economic-effectiveness, flexible plastic transistors and electrodes also solar cells, electromagnetic interference capture, static elimination, corrosion prevention materials, diodes and related electrical engineering in their widespread use the most important thing is to expand the use of semiconductor polymer materials in the production of materials and increase the efficiency, the most important is the creation of alternative variants of sensitive dye in the new third-generation dye-sensitive solar cells.

4. Conclusion

Polyaniline was synthesized by the oxidative polymerization method. the effect of temperature, concentration, and time on the reaction yield of the synthesized polyaniline has been studied. the morphology, surface area, and porosity (SEM) of the synthesized polyaniline at 100 nm were determined by scanning electron microscopy, the nature of the bonding of the atoms in it, the absorption frequency (IR) by infrared spectroscopy, and the conductivity, volt-ampere properties at DT9205A analyzed on the basis of measured data. Despite its low efficiency, this polyaniline dye pigment has proven its strength as an effective semiconductor polymer pigment for new generation dye-sensitive solar cells.

Conflict of Interest

The authors declare that there is no conflict of interests regarding the publication of this article.

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