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Natural Pigment –Poly Vinyl Alcohol Nano composites Thin Films for Solar Cell

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Abstract:

Solar cells thin films were prepared using polyvinyl alcohol (PVA) as a thin film, with extract of natural pigment from local flower. A concentration of 0.1g/ml of polyvinyl alcohol solution in water was prepared for four samples, with various concentrations of plant pigment (0, 15, 25 and 50) % added to each of the four solutions separately for preparing (PVA with low concentrated dye , PVA with medium concentrated dye and PVA with high concentrated dye) thin films respectively . Ultraviolet absorption regions were obtained by computerized UV-Visible (CECIL 2700). Optical properties including (absorbance, reflectance, absorption coefficient, energy gap and dielectric constant) via UV- Vis were tested, too. Fourier transform infrared (FTIR) spectrophotometer was employed to test the samples. Thermal analysis of thin films, including melting point (Tm), onset degree, endset degree, and crystallinity% were tested by differential scanning calorimeter (DSC). Three dimensional morphologies of thin films were inspected by atomic force microscopy (ATM). Contact angle also was tested as an index to hydrophilicity. Results proved that the ultraviolet and FTIR absorption increase after adding the natural pigment to PVA thin film, as well as it increases with increasing concentration of natural pigment. DSC analysis revealed an increase of PVA melting point when adding 15% concentration and it decreases with a 50% concentration of pigment. AFM results show an increase in surface roughness, hence the surface bearing index of PVA thin films is inversely proportional to pigment concentration. Contact angle decreases from 46.5° for pure PVA thin film to 44. 8°, 42. 6° and 35.2° after adding (15, 25, and 50)% concentration of natural dye respectively. Optical properties were enhanced by adding the natural dye, hence energy gap decreased from 3 eV for pure PVA to 2.3 eV for the PVA with a high concentrate dye. Dielectric constant increased with increasing concentration of dye, which leads to high polarization of solar cell.

Key words: Hydrophilic, Natural pigments, PVA, Solar cell, Thin films, UV-spectrum.

Introduction:

Materials are classified according to their wetting behavior into two categories: hydrophobic and hydrophilic, the former have a surface with sturdy affinity to water whereas the latter fend off water. There are different solid materials which have varying wetting properties, and the ability of solid surfaces to accept or reject water is affected by several factors, such as roughness and surfaces topography (1). It is usual to recognize three types of solar cells, and these include mono and poly crystalline silicon-based solar cells which represent 90% of the market shares, and the third type of solar cells is the dye-sensitive solar cell(2). Solar cells have a key role in renewable energy, since it’s one of more interesting sources for sustained energy. Plant dye is a very safe, cost effective and bioactive factor when added to polymers to improve optical and structural properties for solar cell applications. Semiconducting oxides nanostructures like TiO_2, SnO_2, and ZnO nanoparticles, with a wide band gap and high surface area are mostly used for high efficiency solar-power, since they corporate with dye for solar cell synthesis. The incorporation of new nanoparticles to solar cell matrix containing a natural dye can enhance free electrons scattering, hence creating a new position which consequently limits the electron mobility and increases light absorption. Solar cell involves an electrolyte media for performing its designated function, although employing the solid electrolyte may leads to a leak, hence antistatic hydrophilic materials may be used for this purpose (3). The traditional polymer solar cells, also known as all polymer solar cells (APSC)have long been used in the last few decades, nevertheless they haven't gained the desired high performance compared to their new counterparts, manufactured by modern techniques, due to many challenges: the poor polymer-polymer miscibility may lead to a high phase segregation between donor-acceptor, which enhances short circuit current density (JSC), and fill factor, hence decreasing efficiency of solar cells (4).The efficiency of polymer solar cells is also affected by the diffusion length of the exaction, i.e. the distance travelled by exactions until being recombined, which is in the range (3-10) nm in organic semiconductors. The exactions taking place in a large distance from the hetero junction are difficult to be gained, which impedes the efficiency of the solar cell due to the thin layer near the interface which may not be sufficient to make use of solar radiation (5). Another issue associated with organic semiconductors is the speed through which the exaction is generated and moved to the donor to acceptor being dissociated in the diffusion length, and this doesn't directly creates free charge carriers due to Coulomb attraction. In an attempt to solve this problem, researchers are focusing on nanotechnology to manufacture solar cells with diffusion length twice the traditional one (6). Polyvinyl alcohol (PVA) is a hydrophilic, antistatic, non-toxic polymer. Molecular weight of PVA is about (31000 to 50000) g/mol.It has good thermal and chemical stability, as well as easy processing, high mechanical strength and biocompatibility, as such it's a suitable material for solar cell fabrication. The natural dye from a local yellow flower contains positions for light absorption of sun light, in addition to many elements which could create hydrogen bonds when they react with PVA matrix (7-9). Several studies have been published in this field; Mohammed I. et. al (2012), prepared a natural dye from a mixture of flame tree flower and Pawpaw leaf for dye-sensitized solar cells (DSSC). Photoelectric behavior of DSSC showed an open-circuit voltage (VOC) of 0.50V, short-current circuit density of about 0.668 mA/cm^2 (10). Abodunrin et.al. (2015), fabricated a dye-sensitized solar cells (DSSC) which was mixed with natural mango leaf dye extracts, and studied the effect of electrolyte on properties of fabricated solar cell. The researchers have found that the (DSSC had the least parameter value because of degradation of lupeol at increased temperatures and vigorous acidic medium producing in the filtrate of the adsorbed dye from the TiO2 surface(11). The DSSC showed the highest conversion value of photocurrent density (J_{SC}) (1.3 mA/ cm^2) at pH 2.58. Mango leaf extract dye is environmental friendly, non toxic ,cheap, available and active dye for solar cell fabricated under different conditions.

Jamalullai et. al. in 2017 published a review for using three types of natural dyes: *Anthocyanin*, *Clorophyll* and *Betalain*, to compare their performance to synthesize solar cells, they concluded that these three natural dye were ecofriendly, non-toxic, cheap and available dye sensitizer for DSSC. Based on data gained in this work, they found that the *Betalain* pigments extracted from purple wild Sicilian prickly pear dye have the highest efficiency of conversion energy around 2.06%, followed by *Anthocyanin* and *Chlorophyll*.(12)

The current work aims to provide a method to manufacture solar cells using low cost, ecofriendly, and locally available materials, using polyvinyl alcohol (a water-soluble polymer) and a natural dye extracted from (*Tar n-yzat*) flower, a wild local flower that grows naturally without human intervention, and is abundant in the middle east countries. Thus reducing the ecological effect, while keeping a rational cost of synthesis.

Materials and Methods:

Poly vinyl alcohol (PVA) purchased from Sigma- Aldrich ™ with a chemical formula $[C_2H_4]_n$ is a white powder with 0.95% purity, weight average molecular weight ($\overline{M}_w$) in a a range (31000 to 50000) g/mol. and hydrolysis rate (86-90 mol/mol) was used, De-ionized water as a solvent was used. Extracted of natural dye from *Tar-n-yazt* plant flower was used, as shown in Fig.1

Figure 1. *Tar n-yazt* Flower

Tar N-yazt is a herb plant that grows in spring and is 15 cm length, and its leaves are yellow. The Tar n-yazt leaves pigments were extracted by crushing 250 g of herb plant in an electric mixer and immersing it in 5000 ml of acetone. The yellow color of the flowers comes from the *carotin* and *xanthin*, two pigments that give plants their distinctive yellow color, and both are insoluble in water (10). This mixture was distilled and evaporated via a rotary evaporator to recover the pigment from the mother liquor-acetone. The resultant pigment was divided to 3 parts including 15, 25, as well as 50 ml and added to 3 samples of 1% g/ml of PVA dissolved in deionized water separately. Table 1. shows the samples prepared in this research.

Table 1. The samples used in this search

No. of samples	Contents	Ratios of dye
1	PVA thin film	1g of PVA + 99 g of H_2O (free dye)
2	PVA + 15 ml of pigments: LCD	Sample 1 (85) ml + 15 ml of pigment
3	PVA + 25 ml of pigment :MCD	Sample 1 (75) ml + 25 ml of pigment
4	PVA+50 ml of pigment :HCD	Sample 1 (50) ml + 50 ml of pigment

The previous solutions were poured into a plastic container with dimensions of 10 * 10 cm^2 and were left to solidification for 24 hours.

The test apparatuses

Analysis of water/dye solution contents

This test was carried out by V10S Compact Volumetric KF Titrator, an automatic titration apparatus that gives precise content analysis. The solution (solvent+ the material to be analyzed) is placed in an automatic burette, which was in turn placed in an automatic titrator, and then the solution is analyzed down to its main ingredients. Table 2 shows the contents of dye solution.

Table 2. Dye analysis contents

Sample type	Ph	EC µS/cm	N Ppm	O.M %	Cl Ppm	Ca Ppm	SO_4 ppm	CO_3 Meq/L
Water sample	6.3	310	20.2	0.4	99.4	60	24	0

E.C : electrical conductivity, O.M : metal oxides

Testing devices

UV-Vis CECIL-2700

The optical properties were studied by the UV. Visible (CECIL-2700). The UV spectrum analysis is used to measure the wavelengths and absorption intensity of specimens in a wide range of spectrum. The solution (solvent+ the material to be analyzed) is placed in a small transparent container, which in turn is subjected to an omnidirectional wavelength, and another container, filled with the solvent only, is subjected to the same omnidirectional wavelength used for the sample container. The intensities of the beams transmitted through the containers is measured by electronic detectors and compared to standards, thus the difference in contents and their concentrations can be measured accurately.

Absorption coefficient calculated according to Eq 1.

$$\propto = \frac{2.303A + 2(\ln(1-R))}{d} \quad \ldots \quad 1$$

Where α : absorption coefficient , A: absorbance , R: reflectance , d:sample thickness

Electrical properties were performed according to the results of UV-Visible spectrometer via refractive index (n) as the following Eqs (2&3) for real and imaginary parts of dielectric constant respectively.

$$\boldsymbol{\varepsilon_1 = n^2 - k^2} \quad \ldots\ldots\ldots 2$$

$$\boldsymbol{\varepsilon_2 = 2\,n\,k} \quad \ldots\ldots\ldots 3$$

energy gap calculated according to Eq. 4

$$Eg = (\alpha h\upsilon)^2 \quad \ldots\ldots\ldots 4$$

Where h = Plank's constant, υ=photon frequency = light velocity/ (wavelength)

Eq 5. used for calculation of reflectance property

$$Ab+Tr+Rf = 1 \quad \ldots\ldots\ldots\ldots 5$$

Where : Ab is absorbance , Tr. : is transmittance , Rf: is reflectance

(FTIR-600) Spectrum

The Fourier transform infrared spectrometer (FTIR-600) from SIDCO-England was used to study the bond energy of the PVA and its solutions. The FTIR analysis is used to measure the wavelengths absorbed by the material in the infrared region of spectrum, which gives the molecular structure of the material. The sample to be tested is dissolved in the proper solvent to form a solution, which is subjected to infrared spectrum, thus producing a signal that is characteristic to the material, and this signal is in turn decoded using a mathematical method (known as Fourier's transformation), and the resulting spectral map is compared to standard references to obtain the chemical features of the material.

Other properties involve thermal properties and hydrophilicity test performed via Shimadzu ™, and by drop shape analyzer – DSA100 ™ by KRUSS ™, respectively. Also, Atomic Force Microscopy (AFM by nanosurf™) was used in this study to 3D-analysis of used samples.

Results and Discussion:

Optical Properties

Absorbance

Fig. 2.a shows the UV- Spectrum of extract dye, and Fig. 2.b shows the absorbance of all samples: The high concentration dye (HCD), medium concentration dye (MCD) and low concentration dye (LCD), together with the zero dye concentration are shown in Fig. 2.b

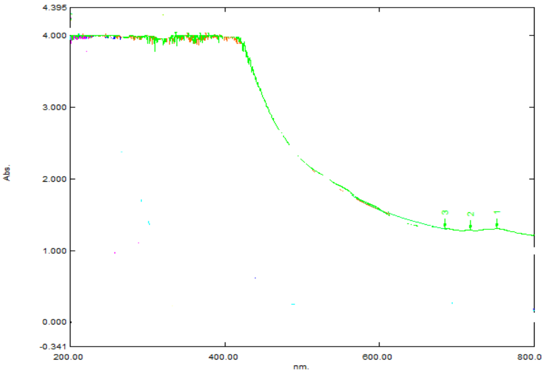

Figure 2.a The Aabsorbance Spectrum of Extract Dye

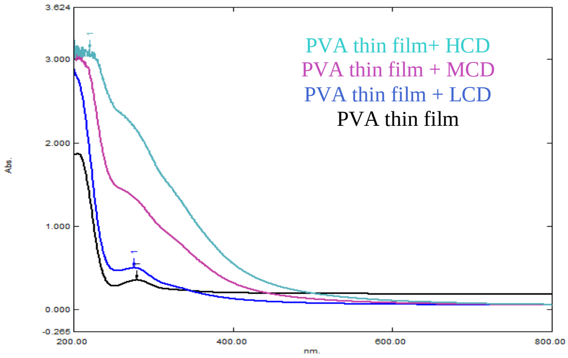

Figure 2.b The Absorbance of PVA Thin Films HCD: high concentration dye, MCD: medium concentration dye, LCD: low concentration dye

Fig 2-a refers to the absorption spectrum located at the visible region and the highest peak is at 450 nm. Fig 2.b shows that the absorption peak of UV spectrum is noticed to increase with increasing the concentration of dye, due to the presence of nitrogen, oxygen, sulfur and halogens present in the natural contents of dye extracted from the flower, and these in turn contain (n) electrons and can absorb visible or ultraviolet radiation as these spectra have higher energy than that needed to stir up electrons (13), This corresponds to the results obtained by Muhammed et.al (10). We notice that the prepared thin film is primitive in the wavelength after 450 nm, and there are not any peaks of absorbance wavelength found.

Reflectance:

Fig 2.c shows the reflectance of all samples as shown below, as they are obtained by the UV-visible apparatus (CECIL-2700) according to Eq 5. We note from Fig 2.c The reflectance of samples increase in the UV- wavelengths region about (200-300 nm), as well as noting the PVA + HCD sample have highest reflectance peak in the wavelengths about (380-390 nm) , this is because the molecules of dye reflect the light strongly (14), this corresponds to the results obtained by Adnan et al. (14).

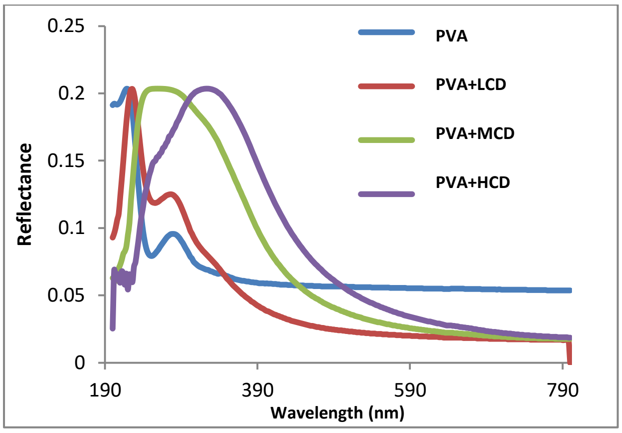

Figure 2.c the reflectance of PVA Thin Films

Absorption coefficient

Absorption coefficient calculated according to Eq 1, Fig 2.d shows the absorption coefficient of all samples as shown below:

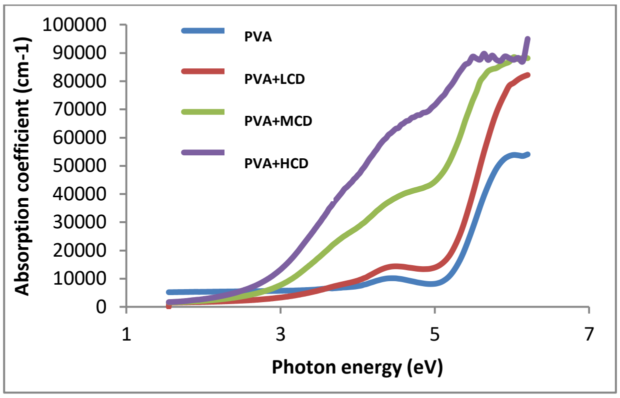

Figure 2.d the absorption coefficient of PVA Thin Films

It is noticed from Fig. 2.d that the absorption coefficient of samples increases with increasing of photon energy, as well as noting PVA + concentrated dye has the highest absorption coefficient, this is because the dye extract contains Nitrogen, oxygen, sulfur and halogens, which contain n electrons and can absorb visible or ultraviolet radiation because these rays have more energy than the energy needed to stir up electrons. This corresponds to the results obtained by Adnan et al. (14).

Direct Energy Gap

The energy gap is calculated from the energy gap curve on the y-axis by calculating the absorption coefficient according to Eq 1 and applying Eq. 4 to calculate the energy gap versus the photon energy equal to Planck's constant * wavelength on the x-axis, then a straight line representing the tangent of the resulting curves is projected to represent the required energy gap value.(15) as in Fig 2.e and table 3.

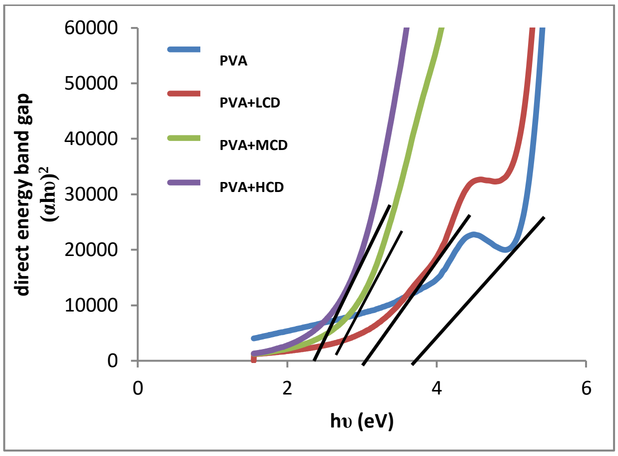

Figure 2.e Energy gap of PVA samples with 0%, 15%, 25% and 50% dye concentration

Table 3. The energy gaps of samples

No. of samples	Contents	Energy gap (eV)
1	PVA (free dye)	3
2	PVA + LCD	2.8
3	PVA +MCD	2.5
4	PVA + HCD	2.3

We observe, the direct energy gap decreases with increasing of dye concentration, this is because the natural dye with the lowest energy gap works on assisting the electron to excite from valence band to the conduction band with very low energy and very short time , this leads to prepare solar cells with high efficiency. This corresponds to the results obtained by Adnan et al. (14).

Dielectric constant

Figs 2.(f-g) show the real and imaginary dielectric constant respectively which calculated according to Eqs (2&3 respectively)

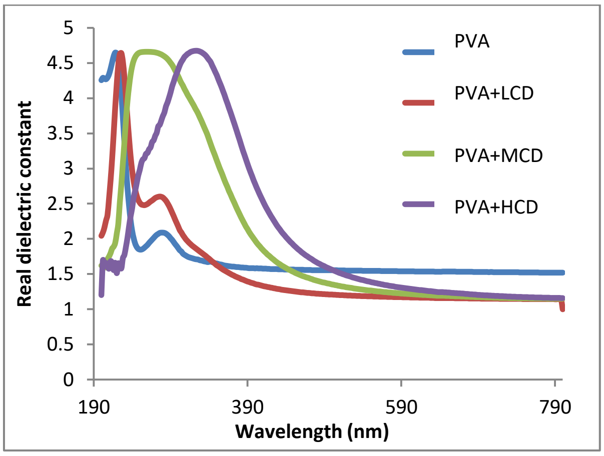

Figure 2.f real dielectric constant

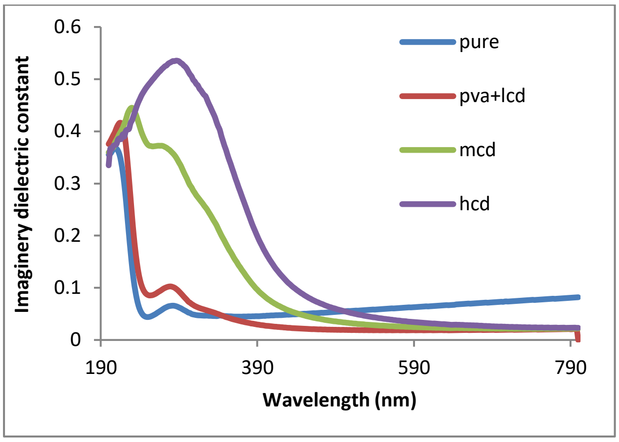

Figure 2.g imaginary dielectric constant

We note that, the real and imaginary dielectric constants have the highest value to the (PVA + HCD) sample , this means that the concentrated dye works on increasing of real dielectric constant, which leads to increases of polarizing of solar cell. (13)

Structural properties

FTIR Analysis

Fig. 3 shows the FTIR analysis of PVA thin film and PVA thin film mixed with different concentrations of dye: low, medium and high concentrations dye respectively. The bonds between 3550 and 3200 cm^{-1} were noted and attached to the stretching O–H of the intramolecular and intermolecular hydrogen bridges. This corresponds to the results obtained by Adnan et al. (14). Vibrational group between 2840- 3000 cm^{-1} were noticed and stretched C–H of alkyl groups.

The peak of IR-rad transmittance refers to there are an changing of peak positions, and a new peak created at 3016.3 cm^{-1}, this refers to the interaction between PVA and dye (16).

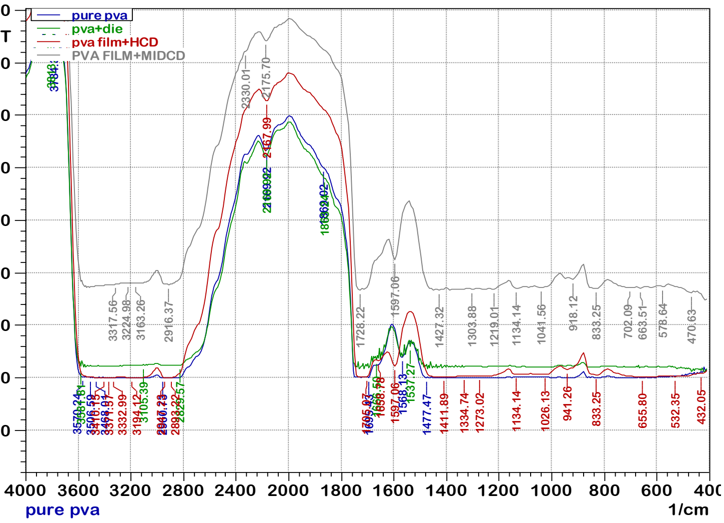

Figure 3. FTIR analysis of PVA thin films and PVA mixed with three different percentages of dye

3D – Analysis by Atomic Force Microscopy (AFM):

Figs 4 (a-d) and Table 4. show the morphology of PVA thin films (a. thin film of PVA, b. PVA thin film+ 10% w/v pigment, c. PVA thin film+ 25% w/v of pigment, and d. PVA thin film+50% w/v of pigment) respectively.

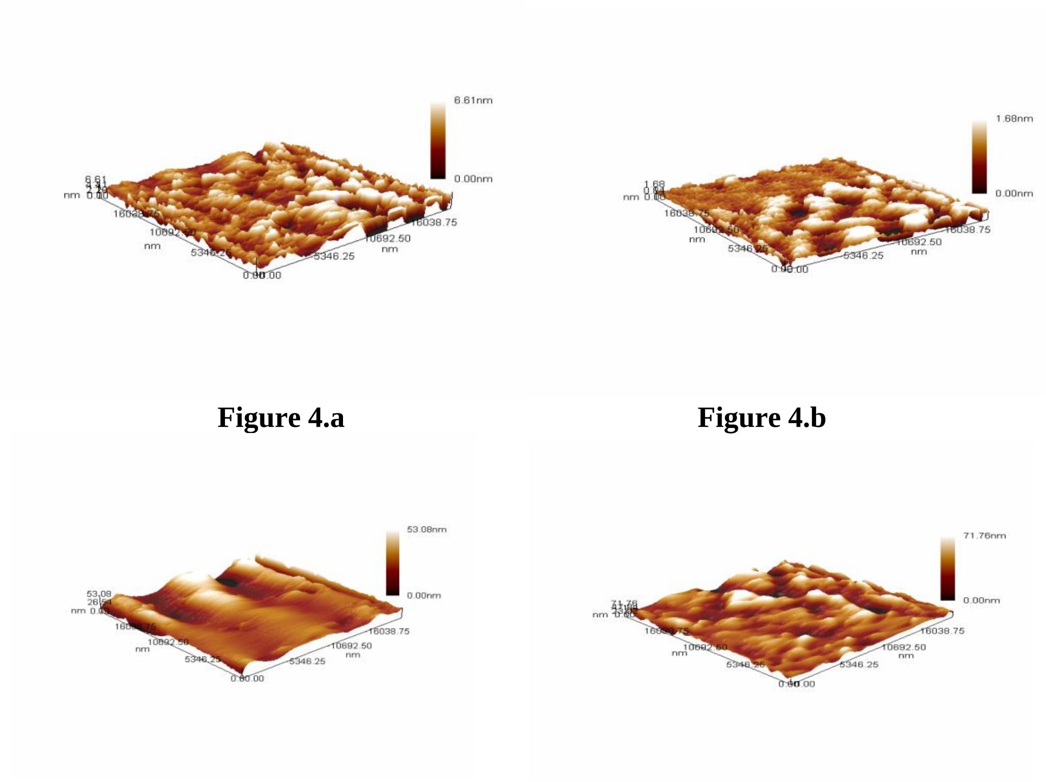

Figure 4.a

Figure 4.b

Figure 4.c

Figure 4.d

Figure 4. AFM Analysis of a. PVA thin film b. PVA thin film + 15% w/v of pigment c. PVA thin film + 25% w/v of pigment PVA thin film + 50% w/v of pigmen

Table 4. Surface parameters by AFM Tested

Sample No.	Sample	Roughness average nm	Sdr(Surface area ratio)	Bearing index
1	PVA	1.2	0.00327	1.6
2	PVA + LCD w/v Pigments	0.319	0.000249	2.7
3	PVA + MCD w/v Pigments	7.3	0.0198	0.9
4	PVA + HCD w/v Pigments	10.7	0.05	1.06

Surface area ratio parameter was noticed to decrease after adding 15% concentration of natural dye, while the bearing index of surface have increased after adding 15% concentration of the natural dye, this is because the pigment is homogenized with polyvinyl alcohol since low concentrations prevent aggregations on the thin film surface, hence the increase of bearing ability of the surface. The surface area ratio and average surface parameters are directly proportional to bearing ability of the surface. The above-mentioned parameters are also directly proportional to the dye concentration in the range of 25-50% respectively. This may be due to heterogeneity of the dye-base material mixture and the creation of conglomerates on the surface of the resulting thin film (17).

Hydrophilicity Test

Fig. 5 (a-d) shows the contact angle of PVA thin film and PVA+ aqueous extract of natural dye respectively.

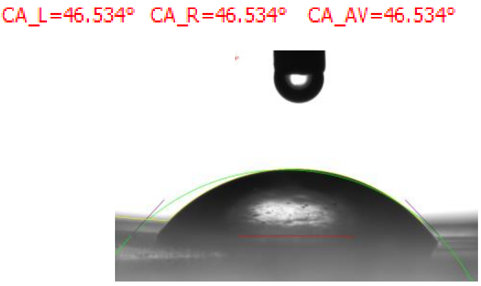

Figure 5.a Contact angle of a. PVA thin film

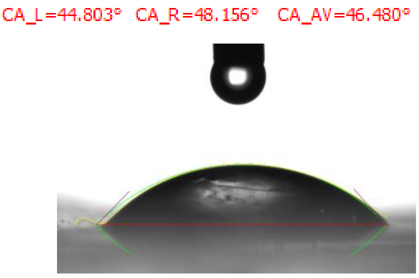

Figure 5. b. PVA+15% of natural dye

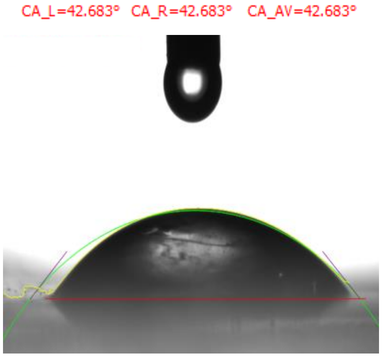

Figure 5.c PVA thin film 25% of natural dye

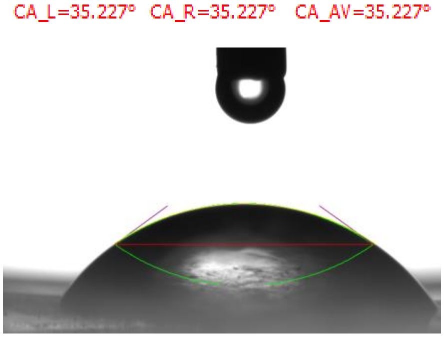

Figure 5.d PVA+50% of natural dye

It is important to show the difference in concepts between hydrophobic and hydrophilic materials: hydrophilic materials have low affinity with water, such that their contact angle when they come in contact with water is less than 90°, while hydrophilic materials have a high wettability, so their contact angle with water is larger than 90°(18).

It is observed from Figure 6 that adding aqueous solution of natural dye with 15%, 25%, and 50% concentrations respectively to PVA thin films separately, lead to decrease the contact angle of pure PVA thin film from 46.534° to 44.80°, 42.683°, and 35.227° respectively, according to dye concentrations, this is because of the increase of hydrogen bonds that resulted by the addition of dye which contain nitrogen, oxygen, and sulfur (19).

Differential Scanning Calorimetry (DSC) Analysis of Thin Films

Fig. 6 and Table 5 show the thermal analysis of PVA and its nanocomposites mixed with extracted natural dye. The thermal characterization was carried out using differential scanning calorimeter type (Shimadzu-4)

Table 5. Shows the Thermal Analysis of Thin Film by DSC

Sample	Onset ^{o}C	Endset ^{o}C	Tm^{o}C	Crystallinty %(-) J/g
PVA (pure)	157	206	201	2.25
PVA + LCD	174	199	203	2.18
PVA + HCD	187	198	108	1.71

As noticed in the Table above and Fig.6 below, the melting point is directly proportional to the concentration of the dye, this may have been caused by the presence of new bonds created between PVA matrix and the added dye, due to the oxygen, calcium, nitrogen and sulfate (SO_4) (20).

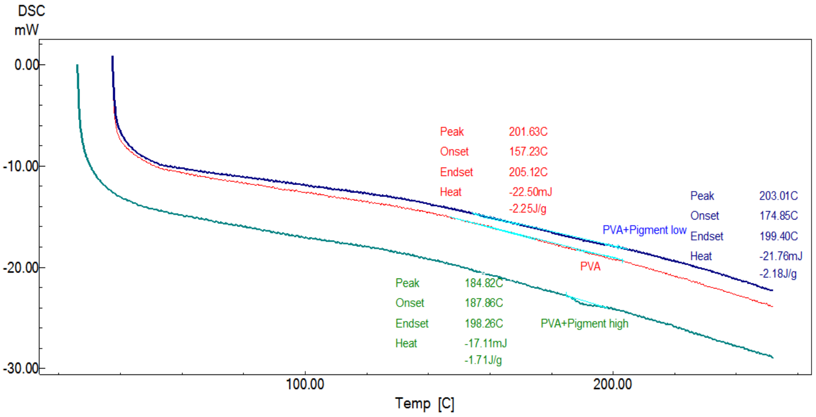

Figure 6. DSC curve for thermal analysis of PVA thin film, and PVA with three different concentrations of dye

Conclusions:

From the work carried out in this research, the following can be concluded:

The addition of the natural dye improves the optical properties of the solar cell including absorbance, transmittance, reflectivity, and coefficient of absorption, as well as reducing the energy gap of the resulting cell, in addition to the increased dielectric constant, which leads to increased polarization of solar cell. The dye extracted from yellow flowers can be used to improve the optical properties of polyvinyl alcohol for use as a hydrophilic, anti-static and safety electrolyte in solar cells and the lowest energy gap is to PVA + high concentrated dye about 2.3 eV.

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Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Babylon.

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اغشية رقيقة من متراكبات نانوية لبولي فينيل الكحول / صبغة طبيعية للخلايا الشمسية

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الخلاصة:

تلعب الخلايا الشمسية دورًا مهمًا في الطاقة المتجددة ، حيث إنها محور التركيز الرئيسي للباحثين في مجال الطاقة المستدامة. تهدف هذه الورقة إلى تصنيع مواد مركبة نانوية ، باستخدام بولي فينيل الكحول (PVA) مع مستخلص بيولوجي صديق للبيئة تم استخدامه لإعداد عينات من الأفلام الرقيقة. تم تحضير تركيز 0.1 غم / مل من محلول بولي فينيل الكحول في الماء لأربعة عينات، مع إضافة تركيزات مختلفة من الصبغة النباتية (15٪ و 25٪ و 50٪) إلى كل من المحاليل الأربعة بشكل منفصل. تم اختبار مناطق امتصاص الأشعة فوق البنفسجية بواسطة مطياف الأشعة فوق البنفسجية والمرئية المحوسبة (CECIL 2700) ، وتم اختبار الخصائص البصرية بما في ذلك (الامتصاصية، الانعكاس ، معامل الامتصاص ، فجوة الطاقة وثابت العزل). تم اجراء فحص طيف الأشعة تحت الحمراء (FTIR) لاختبار العينات. تم اختبار التحليل الحراري للأغشية الرقيقة، بما في ذلك نقطة الانصهار (Tm)، ودرجة البدء، ودرجة النهاية ، والبلورة٪ ، بواسطة المسعر التفاضلي الحراري (DSC) (Shimadzu-4). كما تم فحص مورفولوجيا السطح للأغشية الرقيقة بواسطة مجهر القوة الذرية (AFM). تم اختبار زاوية التلامس أيضًا كمؤشر على قابلية تبلل السطح. أثبتت النتائج أن امتصاص الأشعة فوق البنفسجية و FTIR يزيد بعد إضافة الصبغة الطبيعية إلى غشاء البولي فينيل الكحول ، كما يفعل مع زيادة تركيز الصبغة الطبيعية. كشف تحليل DSC عن زيادة في درجة انصهار غشاء البولي فينيل الكحول عند إضافة تركيز بنسبة 15٪ ويتناقص بتركيز 50٪ من الصبغة. تظهر نتائج AFM زيادة في خشونة السطح ، وبالتالي فإن عامل التحمل السطحي للأغشية الرقيقة يتناسب عكسيا مع تركيز الصبغة. تقل زاوية التبلل من 46.5 درجة لاغشية PVA النقية إلى 44.8 ° و 42.6 ° و 35.2 ° بعد إضافة تركيز (15 و 25 و 50) ٪ من الصبغة الطبيعية على التوالي. تم تعزيز الخواص البصرية عن طريق إضافة الصبغة الطبيعية مثل فجوة الطاقة انخفضت من 3 الكترون فولت لغشاء البولي فينيل الكحول الخالية من الأصباغ إلى 2.3 الكترون فولت للبولي فينيل الكحول بعد إضافة صبغة عالية التركيز. يزداد ثابت العزل الكهربائي بزيادة تركيز الصبغة ، مما يؤدي إلى ارتفاع استقطاب الخلايا الشمسية.

الكلمات مفتاحية : محب للماء، صبغة طبيعية، بولي فينيل الكحول، خلايا شمسية، اغشية رقيقة، طيف الاشعة فوق البنفسجية.