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Experimental Study of the Effects of Chenodeoxycholic Acid (Cdca) Additive on Dye-Sensitized Solar Cell (Dssc) Performance Using Dye Mixture of Chlorophyll and Anthocyanin

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Abstracts: In this study, we seek to investigate the effects of Chenodeoxycholic Acid (CDCA) in the improvement of photosensitizer made from the mixture of chlorophyll and anthocyanin extracted from neem leaves (Azdirachta indica) and roselle flowers (Hibiscus sabdariffa) respectively. Four DSSC samples were prepared on separate substrates made from fluorinated tin oxide (FTO) coated with titanium dioxide (TiO2) semiconductor nanoparticles double-loaded with different patterns of dye mixture and labeled AA, AB, AC and AD. The optimized dye mixture containing neem in ethanol (Ne) and Hibiscus in water (Hw), (that is, Ne+Hw), gave the best absorbance spectrum and widest band gap with highest peak at 550nm wavelength. Additive dye that contained 1.0mM of CDCA concentration gave maximum peak at 630nm wavelength (aside zero concentration), and showed significantly long-term stability. Substrate AB loaded with background optimized fresh dye and overlaid with additive dye produced the best overall photovoltaic performance with efficiency of 0.505%, fill factor of 54.97%, short-circuit current density Jsc of 0.090mA and open-circuit voltage Voc of 10.20mV. The result showed that the efficiency of cell AB surpassed the efficiency of cell AC (substrate with background old dye loading overlaid with additive dye) by 17.03%, 31.48% over cell AA (substrate double-loaded with only optimized dye), and 35.84% over cell AD (substrate doubled-loaded with only additive dye).

Keywords: Chenodeoxycholic Acid, Neem Leaves, Photosensitizer, Concentration), Roselle Flowers.

1. INTRODUCTION

The rate of energy consumption globally has accelerated the depletion of the earth's oil reserves, and the combustion exhaust of fossil fuels has resulted in the environmental contamination and greenhouse effect **(1)**. Hydroelectric power generation, for instance, can no longer meet the demand of growing rate of industrialization. Beside, this source is seasonal; it depends on the water level of the dam occasioned by the rainfall strength, yet it is grossly inadequate. This form of energy is a non-renewable energy resource. The cheap alternative to hydropower is the solar energy, a renewable source of energy of which dye-sensitized solar cell (DSSC) is one of the global focus in the area of renewable green energy. Solar energy, derived from sunlight, is a natural endowment and one of the most fundamental renewable energy sources accessible today as it is easily exploitable, inexhaustible, safe, environmentally friendly, and adjustable to enormous applications (2). Dye-sensitized solar cell (DSSC) was inspired by the energy and electron transfer mechanisms in natural photosynthesis and in dye sensitized silver halide emulsions used for photography **(3)**. A dye-sensitized solar cell (DSSC) is a device that changes light energy into electric energy by light sensitization based on a photo-electrochemical system where a semiconductor is formed between photosensitizer anode and an electrolyte **(4)**. There had been persistent research developments aiming at improving DSSCs performance using natural dye from natural plant parts such as leaves, flowers, roots, fruits, barks, seeds, among others, in diverse forms with appropriate modifications in the various materials in the past three decades. Some of these modifications involve the use of additives on either dye or electrolyte or both, dye mixtures, pH, extracting methods, dye loading, nature of redox electrolyte, annealing and sintering process, nature of substrates, to mention but few. In particular, dye mixture tends to provide wider band gap and **improve** absorbance peak value (5), (6). Chenodeoxycholic acid (CDCA) is of the additives used on natural dye for an improved DSSCs efficiency due to its influence in improving morphology of dye and semiconductor interface by providing additional carboxyl and hydroxyl groups for effective anchorage of dye on TiO_2 surface. This action increases the rate of charge transfer mobility that leads to increase in short-circuit current in DSSC operation. It also helps to suppress dye aggregation that reduces short-circuit current (7). They studied the effect of chenodeoxycholic acid (CDCA) as additive for mangosteen pericarps dye sensitizer, in related to its concentration in mole based on the potency of the dye itself. They found that the efficiency of mangosteen was increased from 0.36% to 0.56% when optimized 2.0 mM of CDCA was added. This was attributed to the additional O-H group into the sensitizer which increases the ability to anchor onto the TiO2 surface**. (8)** also studied the effectiveness of CDCA in improving the DSSC performance using Pinang Palm (*Areca catechu)* dye*.* The study revealed that the absorption spectra indicated that natural dyes with addition of CDCA have an excellent stabilizing ability. With the optimized 1.5 mM of CDCA, the DSSC generated a significant photocurrent through efficient electron injection, and the overall power conversion efficiency was enhanced by 4.6%. (9) also studied the effect of Chenodeoxycholic acid CDCA as dye-adsorbent for Rose Bengal dye in improving the performance of DSSC. The addition of CDCA onto dye helps to suppress the dye aggregation on the photoanode surface, a phenomenon that reduces the photocurrent in DSSC. The study revealed that the optimized concentration of 8 mM of CDCA increased electron injection which account for increase in power conversion efficiency from 0.50% to 0.97%. However, at optimized CDCA concentration, the reduced dye loading due to the presence of CDCA and consequently decreased light-harvesting was compensated by the increased electron injection efficiency that led to improved device efficiency.

Other additives that serve as anti-aggregation are glucose derivatives, deoxycholic acid (DCA) and bile acids (compounds based on a steroid scaffold with one to three hydroxyl groups, in addition to a flexible chain with a carboxylic acid). These additives, including chenodeoxycholic acid (CDCA), were found to anchor on the TiO_2 surface. By suppressing unfavorable dye–dye interactions, CDCA has become the most widely used additive in DSSCs. Co-sensitization with CDCA leads to a reduction in dye loading. This effect is expected to lower the conduction band edge of titania, which in most cases leads to a reduction in the open-circuit voltage (V_{oc}). To maximize the effects of CDCA, one needs to carefully choose an appropriate concentration of CDCA which may vary depending on the potency of dye or dye mixture for optimum result (10).

2. METHODOLOGY

2.1 Materials

In this study, we have used natural plants of neem leaves and roselle flowers from which chlorophyll and anthocyanin have been extracted respectively using distilled water and ethanol. The choice of our materials is informed by their accessibility, abundance, low-cost, and environmental friendliness. Besides, the chlorophyll extract from neem leaves is very stable and does not degrade easily over a long time of DSSC operation [11]. CDCA and the FTO substrates coated with titanium dioxide (TiO_2) as photoanode and platinum (Pt) coated as counter electrode were all purchased from Solaronix Chemicals, and used as supplied. Ionic liquid (IL) electrolyte was prepared locally with potassium iodide (KI), iodine (I_2), ethylene glycol, were purchased locally while 1,2, dimethyl-3-propyl imidazolium iodide (DMPII) was ordered from Solaronix chemicals, Switzerland.

2.2 Preparation of Natural Dye Sensitizers

2.2.1 Roselle flower (*Hibiscus sabdariffa*): 10g each of dried roselle flower, locally called zobo, was bought from local market and first rinsed with distilled water to remove the impurity before it was macerated with 50ml warm distilled water at 50ºC, and 50ml ethanol as extracting solvents for 18hrs in separate containers at room temperature. This is because, between temperature of 50ºC and 60ºC, anthocyanin gives a better DSSC performance in that, at higher temperature above 60ºC the thermal degradation of anthocyanin begins to set in. In the same vein, lower temperature could make the anthocyanin less soluble due to lighter colour of the extract which may result in low absorbance, leading to low efficiency of DSSC performance (4). The solutions were filtered with Whatmann paper No. 42 to remove the solid residual from the pure filtrates. The pH values for both distilled water and ethanol extracting solvents were maintained at 2.2 each. Each extract H_w (Hibiscus in water) and H_e (Hibiscus in ethanol) was used fresh in the mixture. Figure1a shows the local dried roselle flower, Figure1b displays the anthocyanin extract from roselle flower from ethanol solvent, and Figure1c shows the unfiltered anthocyanin extract of roselle flower using distilled water at 50ºC as the extracting solvent.

Fig.1(a): Dried Roselle flower

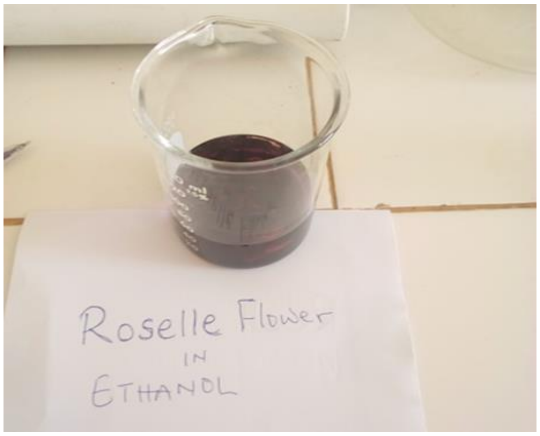

Fig.1(b) Anthocyanin extract from roselle flower using ethanol.

Fig.1(c): Anthocyanin extract from roselle flower using distilled water.

2.2.2 Neem Leaves (*Azadirachta indica*): 10g of fresh neem leaves was washed thoroughly and air dry at room temperature. The leaves were crushed in a mortal with pestle for softening and soaked in warm water at 50ºC for 18hrs. The solution was then filtered severally using Whatman paper to obtain pure chlorophyll extract denoted as N_w (Neem in water). Another 10g of neem leaves was prepared for ethanol extracting solvent at room temperature denoted as N_e (neem in ethanol). The pH 2.2 was maintained for both extracts. Although, anthocyanin performed better with pH1.0 but this pH is too acidic and can cause leaching of the substrate which subsequently can damage the cell. Hence, the choice of this pH value was mild enough to sustain our design DSSC. Figure 2(a), (b) and (c) show different stages of extraction in water and ethanol solvents.

Fig.2a: Blended fresh neem leaves

Fig.2b: Chlorophyll extract from neem leaves using ethanol.

Fig.2c Chlorophyll extract from neem leaves using distilled water.

Four dye samples were originally prepared from both extracts out of which another four mixtures were prepared in ratio 1:1. Four mixed dye samples were prepared in ratio 1:1 from original extracts, and were subjected to analyses using UV-vis absorbance spectrum and other characterization tests. The dye sample mixtures are; Neem + Hibiscus in water ($N_w + H_w$), Neem +Hibiscus in ethanol (N_e+H_e), Neem in water + Hibiscus in ethanol (N_w+H_e), and Neem in ethanol + Hibiscus in water (N_e+H_w). The purpose for these different dye samples was to get the optimized dye that will enhance photovoltaic performance. Figures 3(a), (b), (c), and (d) show the various mixtures of chlorophyll and anthocyanin extracts from distilled water and ethanol.

Fig.3a: Dyes of chlorophyll and anthocyanin in ethanol

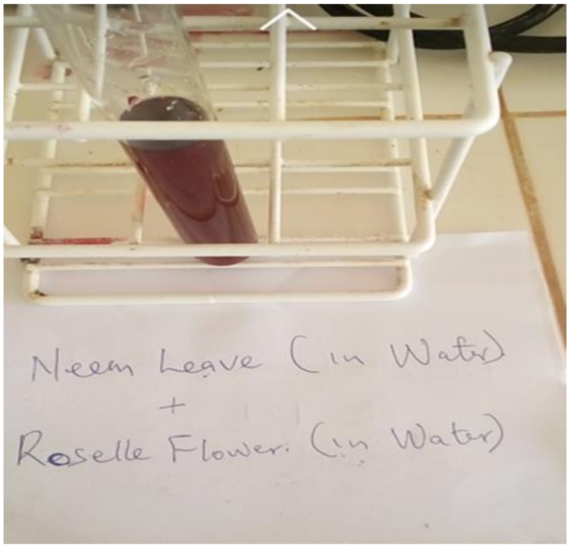

Fig.3b: Dyes of chlorophyll and anthocyanin in water.

Fig.3c: Mixed dye in water and ethanol (N_w+H_e)

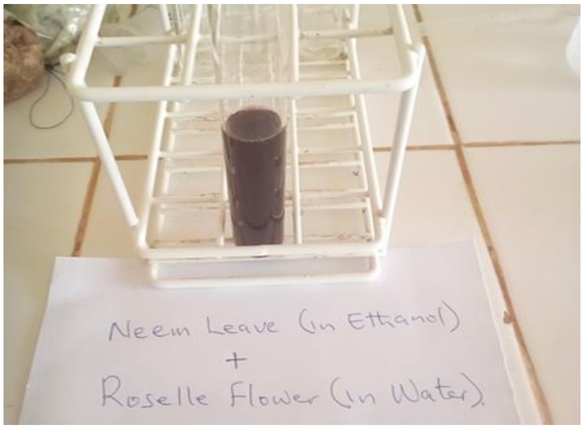

Fig.3d: Mixed dye in ethanol and water (N_e+H_w).

2.2.3 Preparation of Dye Additive with CDCA ($C_{24}H_{40}O_4$)

The optimized dye (N_e+H_w) was treated with chenodeoxycholic acid (CDCA) additive for improved photosensitizer. CDCA is an organic white powder with chemical compound $C_{24}H_{40}O_4$ having molar mass 392.57g/mol was prepared in different moles of 0.5mM, 1.0mM, 1.5mM, 2.0mM, and 2.5mM so as to determine the additive optimized concentration. Each mole was prepared using the formula;

Mass = mole × molar mass.

Each obtained gram of CDCA was mixed in the same volume (20ml) of optimized dye solution to obtain different additive molar concentration, and shake vigorously to obtain homogenous solution using the formula;

Molarity (M) = mole/ volume.

2.2.4 Preparation of Ionic Liquid-Based Electrolyte

The working electrolyte for this study was prepared in four stages using potassium iodide (KI) and iodine (I_2), ethylene glycol, 1,2 dimethyl-3-propylimidazolium (DMPII), and dye as;

(i) Ordinary liquid electrolyte (KI + I_2),

(ii) Gel electrolyte (KI +I_2 + Ethylene glycol),

(iii) Ionic liquid-based (IL) electrolyte (KI + I_2 + Ethylene glycol + DMPII)

(iv) Additive electrolyte (KI + I_2 + Ethylene glycol + DMPII + Dye).

(i). Preparation of Liquid Electrolyte: $KI + I_2 \rightarrow KI_3$

0.036mol of KI was prepared with 7.2ml of distilled water and mixed with 0.015mol of iodine prepared in 3.0ml of ethanol.

(ii) 2.5ml of ethylene glycol was added to the liquid electrolyte for form gel electrolyte in the ratio 4:1.

(iii) 1.5g of DMPII salt was dissolved in 3.5ml of ethylene glycol formed the optimized ionic salt concentration, which is 30wt% of DMPII and was added to 2.0ml of gel electrolyte to produce ionic liquid-based electrolyte [12].

(iv) Additive electrolyte was produced by mixing 0.3ml of optimized additive dye in 1.5ml of ionic electrolyte which formed 20vol% of dye concentration.

2.2.5 Preparation of Photoanode and Counter Electrode Substrates

Four separate photoanodes were prepared each on fluorinated tin oxide (FTO) substrates that have been coated with titanium dioxide (TiO_2) nanoparticles having an active area of 0.4cm x 0.4cm as dye-sensitizers for analytical and comparative studies. These substrates were labeled as **AA, AB, AC** and **AD** as explained above. The substrates were removed and rinsed separately with ethanol and air-dried before coupling. The platinum counter electrodes were first cleansed in warm distilled water of 50^o C and cooled to room temperature before coupling.

2.3 DSSCs Assembly

Four separate DSSC samples were assembled using already prepared substrates **AA**, **AB**, **AC**, and **AD**. The active areas of each substrate of both working electrode and counter electrode were protected by the gasket supplied by Solaronix, with conductive surfaces overlaid each other and held tightly together by clips at the two opposite ends of the substrates (Figure 4). The electrolyte was introduced through the drilled hole in the counter electrode via the syringe, and the hole was covered with cap supplied by solaronix kit to protect the entire system from contamination and environmental adverse condition. The diagram showing the coupling processes was shown in Figure 5, while Figure 6 shows schematic assembly of DSSC operations. The assembled DSSCs were now made ready for solar illumination using direct sunlight irradiation.

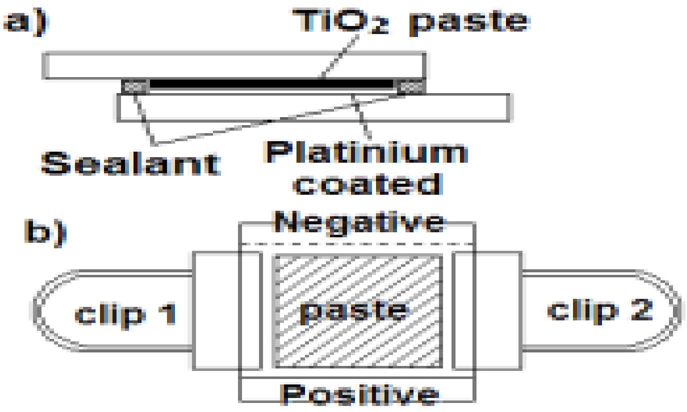

Fig 4: Assembly diagram of DSSC with: a) showing the cross-sectional view, and b) showing the top view with binder clips on both sides

Fig.5: DSSC coupling process.

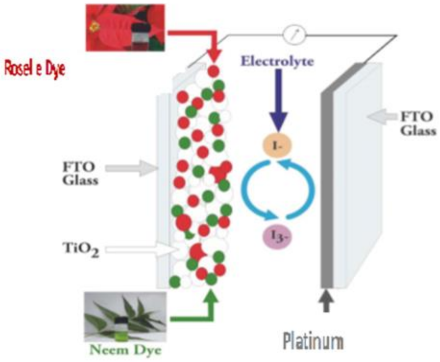

Fig. 6: Schematic assembly of DSSC operation

2.4 Measurement and Characterization

2.4.1 UV-Vis Absorbance Characterization

The UV-vis absorbance spectra of both dye mixtures of chlorophyll and anthocyanin in different combinations, and additive dyes in different concentrations were measured using BIOBASE UV-VIS (B-UV1800PC) Spectrophotometer. Functional groups of molecular bonding were analyzed using Fourier Transform Infrared spectroscopy (Nicolet iS10 FT-IR Spectrometer model) within the range of 350-4400cm^{-1}. The fabricated DSSCs were subjected to direct sunlight irradiation with an intensity of 100mA/cm^2 (AM 1.5G) where various photovoltaic parameter measurements were obtained.

3. RESULTS AND DISCUSSION

Generally, dye mixtures aim to widen the wavelength range of light absorption, which can enhance conversion efficiency (6). Figure 7 displays combined effect of absorption peaks of chlorophyll and anthocyanin in various mixes of ethanol and distilled water solvents. Dye mixture of N_e+H_w gave the outstanding absorbance performance in terms of absorbance peak value and broad band range. This could be attributed to the fact that chlorophyll dye extract from neem leaves is more stable in ethanol while roselle flower extract is better in water for long-term stability (4). (13). The combined effect from the broad band spectrum of the mixture dye incorporates the two peaks in the wider band from 480nm to 660nm with maximum peak at 550nm as shown in figure 4.3. This can be explained in terms of different solvents used where each of the solvents was affected by the level of polarity. The result is to cause shift in the absorption peak and level of absorbance, which in this case resulted longer wavelength (red shift) [6]. Although, anthocyanin is more soluble in ethanol and show a broad absorption band in the visible spectrum region, however, its efficiency drops rapidly when exposed to light radiation. This is due to the photocatalytic decomposition of anthocyanin when in contact with semiconductor oxide (TiO_2) in the presence of ethanol which makes the colour of the photoanode to become pale within a short time of light illumination. As a result, ethanol is not suitable as an anthocyanin extracting solvent for long term DSSC operation [4]. In the same vein, though water is not as good as ethanol in the absorption spectrum, however, in this novel mixture, water seems to serve as antidote to reduce ethanol volatility for long time stability of DSSC performance as can be seen from the result of UV-vis absorbance spectroscopy in figure 4.3, where this mixture gives the highest absorbance value among the rest of the mixtures. The dye mixture (N_e+H_w) was used as the optimized dye for the study.

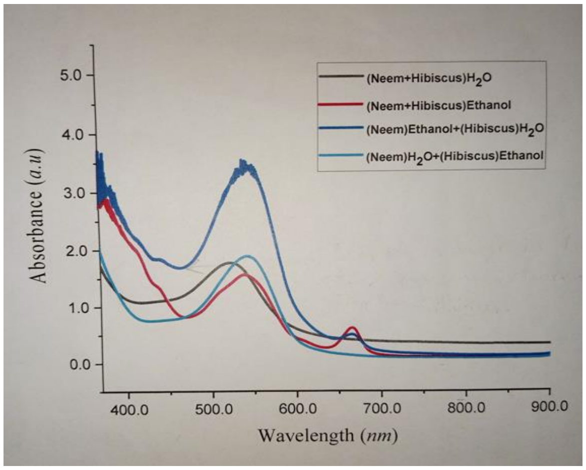

Fig. 7: UV-vis spectra of dye mixtures combinations.

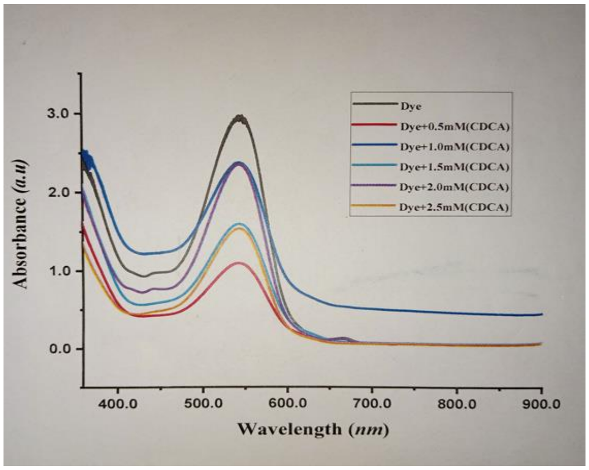

Fig. 8: UV-vis spectra of dye mixtures with CDCA Additive

Figure 8 displayed the absorption spectra of additive dye mixtures. The slight drop in the absorbance value was as a result of decrease in dye concentration caused by the addition of CDCA unto the dye.

3.1 Fourier Transform Infrared (FTIR) Spectroscopy Analyses

Fourier Transform Infrared analysis is the study of the chemical properties of dye solution and nanoparticles (TiO_2). It reveals the various chemical bond formations and associated functional groups in a compound with their stretching band peaks at specific wavenumber. The FTIR analysis enables the predication of how effective a dye can attach with the semiconductor nanoparticles for an enhanced light harvesting ability. FTIR spectrum of pure TiO_2 is shown in Figure 9 with its associated peaks. The FTIR spectra of optimized dye and additive dye containing chlorophyll and anthocyanin are displayed in Figure 10 showing different chemical bond formations. All the spectra showed the presence of both hydroxyl and carboxyl groups, and various chemical bond formations. In the case of pure TiO_2 in Figure 9, the FTIR spectrum showed wide band of the presence of O-H group in TiO_2 as indicated in the wavenumber 3307cm^{-1} and also a C-H stretching belonging to an alkane in the wavenumber 2929cm^{-1}. The presence of these groups will ensure good anchoring of dye on to TiO2 [14]. A peak at 1656.42cm^{-1} is indicative of carbon double bond in the carboxyl group, while aromatic ring of single bond of carbon C-C occurs at wavenumber 1555.52 cm^{-1}. There is also a weak C-H representing the presence of phenol at 1455.00cm^{-1}. A peak at 1059.00 cm^{-1} represents the C-O aromatics ring, and the presence of alkyl halide (halogen group) typical of C-Br bond at 592.0cm^{-1} [14,15]. The FTIR spectra of dye mixture and additive dye are displayed in Figure 10 showing different chemical bond formations. The FTIR spectrum of optimized dye shown in figure 4.11(a), produced a broad band but weak peak, which is typical of a stretching absorption band at wave number 3376cm^{-1} corresponding to the vibration of the O-H associated bond. The weak peak can be attributed to the mixture of ethanol and water that possibly weakens the absorbance capacity. Another band related to valence bond of C=C aromatic ring peaks at wave number 1369cm^{-1} [16]. The peak at wavenumber 2918cm^{-1} shows the C-H of aromatic ring which is characteristics of anthocyanin. This is closely followed by another weak peak of C-H aromatic ring at wavenumber 2855.01cm^{-1}, typical of chlorophyll extract from neem leaves[14]. The peak at 2360.17cm^{-1} indicates the presence of chlorophyll in O-H group, while chlorophyll appears in the carboxyl group in form of aromatic C-C bond at 1595.00cm^{-1} 15. This could probably accounts for long term stability of neem. Similarly, anthocyanin also appears in the carboxyl group as C-O stretching bond corresponding to a wave number 1002.00cm^{-1} [11]. The wave number at 367.33cm^{-1} represents the presence of alkyl halide (halogen) compound caused by the stretching C-Br which is an indication of the presence of both chlorophyll and anthocyanin in a blended dye [17,18].

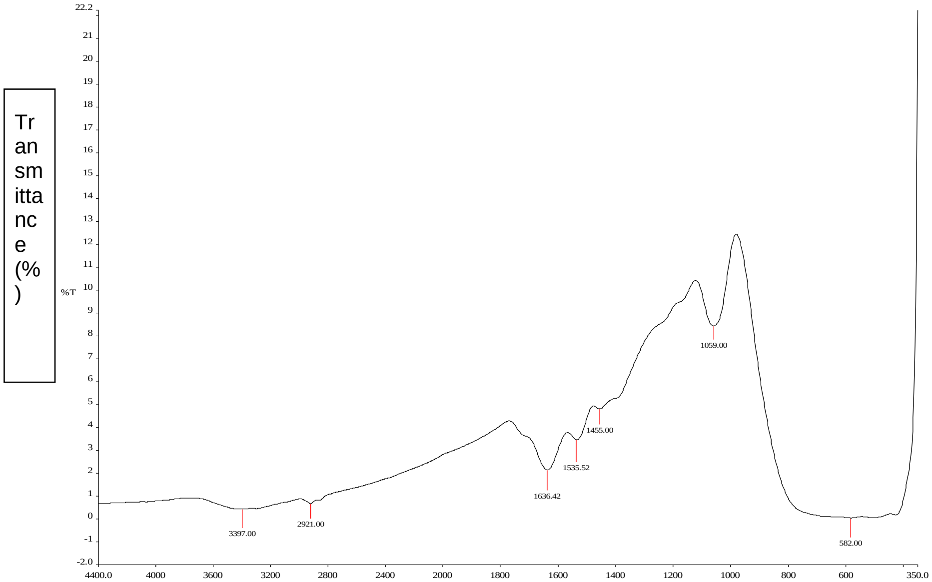

Fig. 9: FTIR Spectra of pure TiO_2 nanoparticles

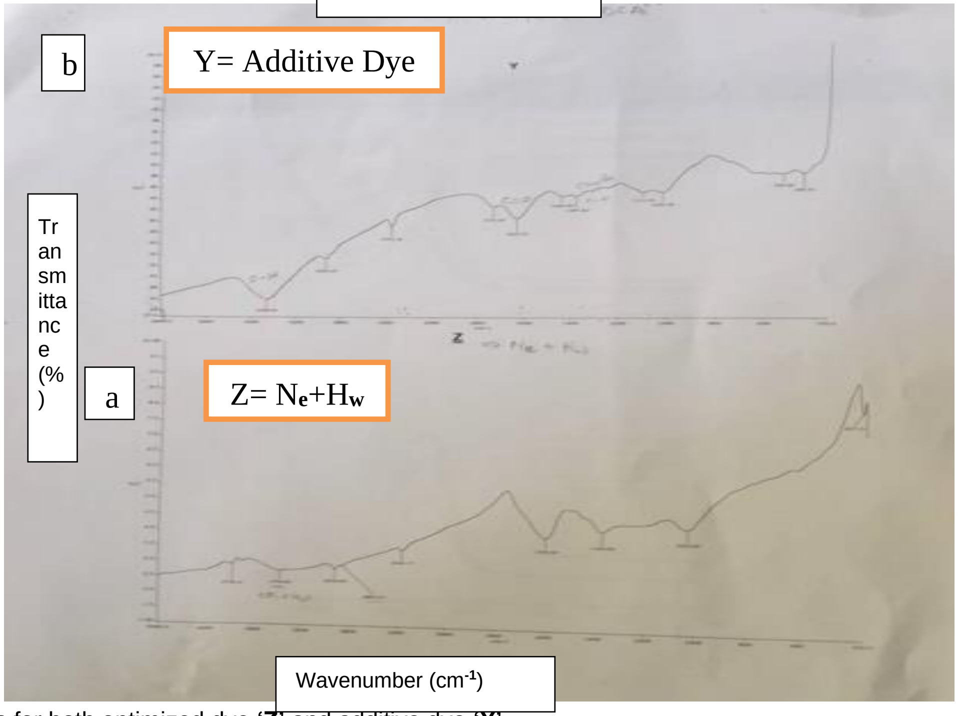

Fig.10: FTIR analysis for both optimized dye **‘Z’** and additive dye **‘Y’**

3.2 Current Density versus Voltage Curve (J-V) Analyses

The corresponding plots for each reading are shown in figures 11a-11d from where the open circuit voltage (V_{oc}), and short circuit current density (J_{sc}) are obtained at the intercepts of each axis (that is y-axis and x- axis) respectively. The kneel point, which represent the maximum power point, of each curve gives the J_{max} and V_{max} values at the intercept of each axis.

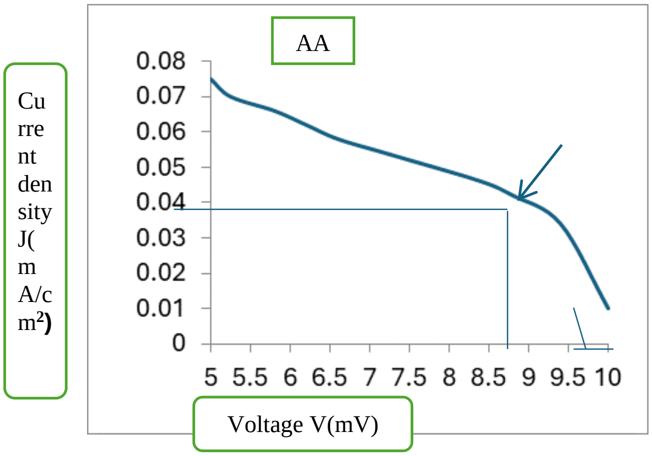

Fig. 11a: J-V plot for cell sample AA.

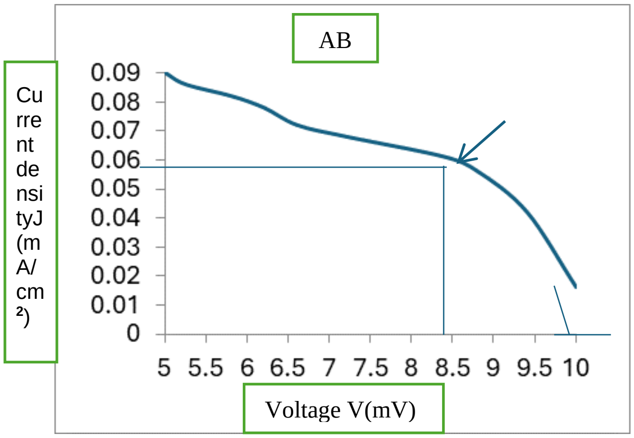

Fig. 11b: J-V plot for cell sample AB

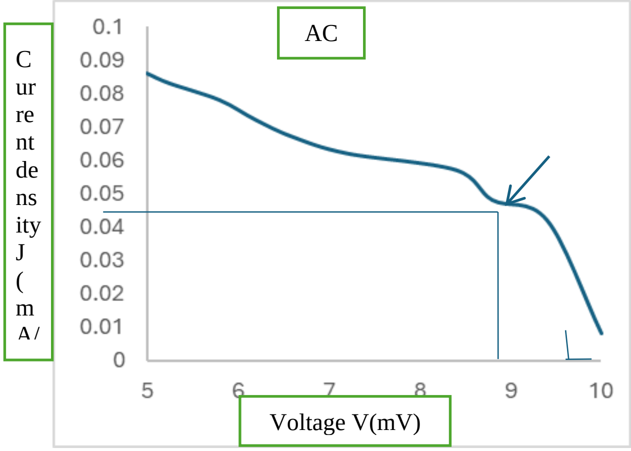

Fig. 11c: J-V plot for cell sample AC.

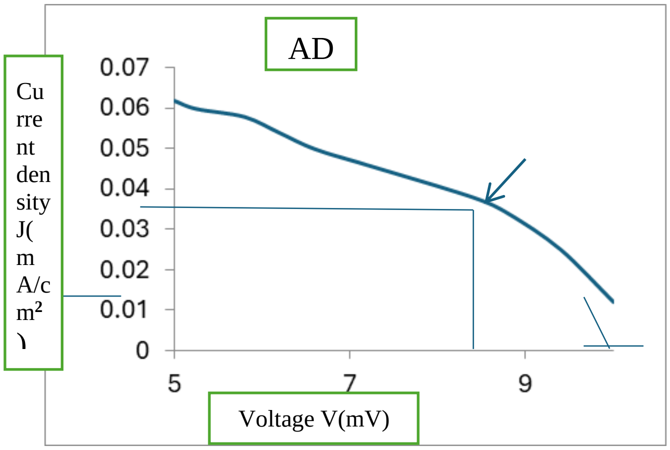

Fig. 11d: J-V plot for cell sample AD

With these obtained values for each curve, fill factor (FF) and efficiency (η) for each cell sample was calculated from the formulae:

$$FF = \frac{J_{max} \times V_{max}}{J_{sc} \times V_{oc}} \text{ -- (1),}$$

or,

$$FF = \frac{J_{max} \times V_{max}}{J_{sc} \times V_{oc}} \times 100\% \text{ , ------------------------------ (2)}$$

and,

$$\text{Efficiency, } \eta = \frac{V_{oc} \times J_{sc} \text{ x FF}}{P_{in}} \text{ ------------------------------- (3)}$$

where P_{in} is the standard radiation power at 100mW/cm^2 or (AM 1.5).

For sample AA, we have,

$$FF = \frac{0.037 \times 9.35}{0.075 \times 10.20} \times 100\%$$

$$= 0.4522 \times 100\% = 45.22\%, \text{ and}$$

$$\text{Efficiency } \eta = \frac{0.075 \times 10.20 \times 45.22\ \%}{100\text{mW/cm}^2}$$

$$= 0.3459 = 0.346\% \text{ (approx.).}$$

Similarly for sample AB;

$$FF = \frac{0.058 \times 8.70 \times}{0.090 \times 10.20} 100\%$$

$$= \frac{0.5046 \times 100\%}{0.918} = 54.97\%$$

$$\text{Efficiency } \eta = \frac{0.918 \times 54.97\%}{100\text{mW/cm}^2} = 0.505\%$$

For sample AC;

$$FF = \frac{0.045 \times 9.30}{0.086 \times 10.18} 100\% = 47.80\%$$

$$\eta = \frac{0.086 \times 10.18}{100\text{mWw/cm}^2} \times 47.80\% = 0.4185\%$$

For sample AD;

$$FF = \frac{0.037 \times 8.75}{0.062 \times 10.24} \times 100\%$$

$$= \underline{32.375\ \%} = 50.99\%$$

0.63488

$$\eta = \frac{0.63488 \times 50.99\%}{100mW/cm^2} = 0.324\%.$$

Table 4 gives the summary of the result of photovoltaic parameters for each of the samples of DSSC fabricated.

Table 4: Result of photovoltaic parameters for each fabricated DSSC sample.

Sample	J_{sc}(mA)	V_{oc}(mV)	J_{max} (mA)	V_{max}(mV)	FF(%)	η(%)
AA	0.075	10.20	0.037	9.35	45.22	0.346
AB	0.090	10.20	0.058	8.70	54.97	0.505
AC	0.086	10.18	0.045	9.30	47.80	0.419
AD	0.062	10.24	0.037	8.75	50.99	0.324

CONCLUSION

The mixture of chlorophyll dye extract from neem leaves using ethanol and anthocyanin dye extract from roselle flowers using distilled water has been used to study the effect of chenodeoxycholic acid (CDCA) dye additive on the performance of dye-sensitized solar cell (DSSC) stability and efficiency. The optimized dye mixture containing neem in ethanol (N_e**)** and Hibiscus in water (H_w**)**, (that is, N_e+H_w), gave the best absorbance spectrum and widest band gap with highest peak at 550nm wavelength. Additive dye that contained 1.0mM of CDCA concentration gave maximum peak at 630nm wavelength (aside zero concentration), and showed significantly long-term stability. Substrate **AB** loaded with background optimized fresh dye and overlaid with additive dye produced the best overall photovoltaic performance with efficiency of 0.505%, fill factor of 54.97%, short-circuit current density J_{sc} of 0.090mA and open-circuit voltage V_{oc} of 10.20mV. The result showed that the efficiency of cell AB surpassed the efficiency of cell **AC** (substrate with background old dye loading overlaid with additive dye) by 17.03%, 31.48% over cell **AA** (substrate double-loaded with only optimized dye), and 35.84% over cell **AD** (substrate doubled-loaded with only additive dye).

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