International Conference on Recent Innovations in Nanoscience and Technology 2018 (ICRINT2018)
26th – 28th December, 2018
Yangon, Myanmar

Natural Dye Extraction from Gourd Leaves and Teak Leaves Using Soxhlet Extraction Method and Determination of Their Properties

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Abstract

Natural dyes such as gourd leaves and teak leaves were extracted with ethanol solvent by Soxhlet Extraction Method. The optical properties of natural dyes were analyzed by UV-visible spectroscopy. The optical absorption method was used to determine the absorption coefficient (α) and optical band gap (E_g) of the natural dyes in the wavelength range 400-700 nm. The maximum band gap energy was found 2.7 eV at gourd leaves while teak leaves exhibited the minimum band gap energy of 2.15 eV. Fourier Transform Infrared Radiation Spectral analysis was employed to observe the functional groups in the natural dyes. From the experimental data, these dyes are quite potential candidate for use in working electrode of Dye Sensitized Solar Cell (DSSC) application.

Keywords: Dye Extractor, Fourier Transform Infrared Radiation Spectral (FTIR), Gourd Leaves, Teak Leaves, UV visible Spectroscopy

1. INTRODUCTION

Natural dyes or pigments extracted from leaves, flowers, fruits, and seeds through simple procedures can be applied as sensitizers in DSSCs. The dyes include chlorophyll, betalains, carotenoids, anthocyanins, flavonoids, tannins, and so on[1,2]. Chlorophyll found in the leaves of most of green plants, absorbs light from red, blue, and violet wavelengths and obtains its color by reflecting green[1]. Green pigments have been used in dyeing food, drink, soap, and cosmetics. Due to their stability, these pigments have been practised as coating materials and anti-knocking agents in gasoline engines[3]. The gourd plant (*Lagenaria siceraria*) is one of the green plants that contain chlorophyll. Natural colorants from plants or algae, e.g., chlorophyll, are safe and could provide health benefits, owing to their anti-mutagenic and anti-oxidant contents are utilized to avoid chronic diseases[3,4]. Natural pigments can be extracted from the various parts of the plant using different types of organic solvents.The solvents affect the absorption of the dye and the binding between the dye and semiconductor surface [1]. Anthocyanin pigments are responsible for the attractive red to purple and blue colors of many fruits and vegetables[5]. Anthocyanin comes from Latin, the Latin anthos is flower and cyan is blue[6,7]. Anthocyanins naturally cause pigments of red and purple. Red anthocyanin pigments are strong and sharp. They are widely applied in various industries such as food coloring or drink[7]. They comprise the largest group of the water-soluble pigments in the plants. Therefore they attach themselves very well oxide materials, such as titanium dioxide (titania) due to a number of hydroxyl (-OH) bonds on both the titania and the dye [6,8,9,12]. Anthocyanin as health beneficial compounds is another reason for the increased scientific interest in these pigments[8]. Plants show characteristic colors owing to the presence of strong pigments. Teak leaves (*Tectona grandis*) can be used as natural dye. Teak leaves contain anthocyanin, a source of brown color[10]. The bark of roots and young leaves produce a yellowish brown-reddish dye which is exploited for paper, edibles, clothes, and matting by the rural people of Manipur[11]. Scientists are greatly interested in building inexpensive and effective solar cells. The current solar technology uses silicon.Silicon, an abundant element but it is expensive to process. In these cells, silicon absorbs light and converts it to electrons. An alternative to the silicon solar cell is one made from natural dyes that can also absorb light and convert it to electrons. Natural dyes may not be as efficient as silicon cells, but they are much less expensive to produce[1,12,13]. In this research pigments, chlorophyll were extracted from gourd leaves (*Lagenaria siceraria*) and anthocyanins were extracted from leaves (*Tectona grandis*).

2. Preparation of natural dyes

In various leaves, gourd leaves and teak leaves were firstly collected to extract the natural dyes. After collection, these leaves were cleaned with water and dried about one week at room temperature. These dried leaves were made into small pieces. The natural dyes from these dried leaves were extracted with ethanol solvent by soxhlet extractor. A Soxhlet extractor has three main section, a percolator (boiler or water bath and reflex condenser) which circulates the solvent, a thimble (a bag is made of a cotton) which retain the solid to be extracted, and a siphon mechanism, which periodically empties the thimble. The dried leaves of weight 3 g containing the compound to be extracted were placed inside the thimble. The thimble was loaded into the main chamber of the Soxhlet extractor. The extraction ethanol solvent of 200 ml to be used was in a round bottom flask or a distillation flask. The round bottom flask was placed into the water bath full of water. The Soxhlet extractor was placed at the top of the flask. A reflux condenser was placed at the top of the extractor. The reflux condenser has two holes, one is cooling water inlet at the lower part and another one is cooling water outlet at the upper part. The ethanol solvent of 200 ml was heated to reflux condenser. When the water in the water bath was heated at 85°C, the ethanol solvent started to vaporize during 1 h. The boiling point of ethanol solvent is 78.37°C. The solvent vapour traveled up a distillation arm and flooded into the chamber housing the thimble of dried leaves. The condenser ensured that ethanol solvent vapour cooled, and dripped back down into the chamber housing the thimble of dried leaves. The chamber containing the dried leaves slowly filled with warm solvent. Some of the desired dried leaves dissolved in the warm solvent. When the Soxhlet chamber was almost full about 4 h, the chamber was emptied by the siphon. The solvent was returned to the distillation flask. The thimble ensured that the rapid motion of the solvent did not transport dried leaves to the still pot. This cycle might be allowed to repeat two times over 6 h. During each cycle, a portion of the pieces of the dried leaves in the thimble dissolved in the solvent. After two cycles, the dried leaves in the thimble were concentrated in the distillation flask. After extraction the solvent was removed typically by means of rotary evaporator or condenser, yielding the extracted dyes. The non-soluble portion of the extracted leaves remained in the thimble, and was dried again. Finally, the solution of the natural dyes were obtained. Chlorophyll from gourd leaves, anthocyanin from teak leaves and mixed dyes from mixed leaves were obtained. The solution can be used as a dye sensitizer. The sequence diagram for natural dye extraction was shown in Figure 1. Gourd plant and teak plant were shown in Figure 2 and 3. Figure 4 was dye extractor, figure 5 was dye solutions from gourd, teak leaves, and mixed leaves, and figure 6 was condenser.

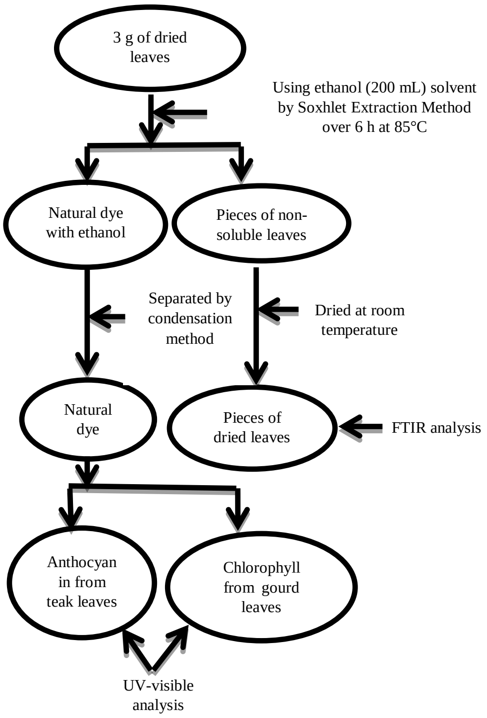

Fig. 1. Natural dye preparation

Fig. 2. Gourd plant

Fig. 3. Teak plant

Fig. 4. Soxhlet Extractor

Fig. 5. Dye solutions of gourd, teak and mixed leaves

Fig. 6. Condenser

2.1 Characterization of Natural Dyes

UV-visible absorption spectra of natural dyes were recorded on SHIMADZU 1800 sphectrophotometer. FTIR analysis was employed to study the functional groups of natural dyes. The diffraction pattern was recorded on Nicolet iS 5 spectrophotometer, over the range from 500 cm^{-1} to 4000 cm^{-1} wavenumbers.

3. **Results and Discussion**

3.1. UV-vis analysis

Figure (7), (8) and (9) showed the absorption spectra of teak leaves, gourd leaves, and mixed leaves extracted with ethanol solvent. The extracts showed visible absorption range within 400 nm-700nm. Three main peaks were found 671 nm at gourd leaves, 671 nm at 673 nm at teak leaves and for mixed leaves. From the spectra, the absorbance and relation wavelengths of gourd leaves, teak leaves, and mixed leaves were described in Table 1, 2, and 3. The variation in the absorption coefficient as a function of photon energy for allowed direct is given by

$$\alpha\,(h\,\nu)=A\,(\,h\,\nu\text{-}E_g)^{1/2}$$

where α is the absorption coefficient, A is a constant, h is Plank's constant, ν is the frequency, and E_g is the band gap energy. A plot of $(\alpha(h\,\nu))^2$ versus photon energy $(h\,\nu)$ showed intermediate linear region the extrapolation of the linear part can be used to calculate the E_g from intersect with $(h\,\nu)$ axis as shown in Figure 10, 11, and 12. The resultant value of E_g for gourd leaves, teak leaves, and mixed leaves were found to be about 2.2 eV, 2.7 eV, and 2.5 eV.

3.2. FTIR Analysis

The recorded FTIR spectra of pigment extractions were shown in figure 13, 14, and 15.The spectra indicated the functional groups of dyes from gourd leaves, teak leaves, and mixed leaves. The various peaks at different wavenumbers were 3275.51, 2921.64, 1624.18, 1400.10, 1235.59, 1017.15cm^{-1} at gourd leaves, 3275.16, 2918.21,2850.17, 1603.00, 1419.52, 1318.94, 1238.43, 1017.02cm^{-1} at teak leaves, and 3278.93, 2918.63, 1621.50, 1405.92, 1236.37, 1018.42 cm^{-1} at mixed leaves. As observed in these leaves, the sharp peaks contain 1624.18 cm^{-1}, 1603.00 cm^{-1}, and 1621.50 cm^{-1} that belong to the C=C group in NO_2 region. The Carboxylic Acid in H bonded OH region was found at wavelengths 3275.51 cm^{-1}, 3275.16 cm^{-1}, and 3278.93 cm^{-1}. The presence of Alkane in CH region was found at wavelengths 2921.64 cm^{-1}, 2918.21 cm^{-1}, and 2918.63 cm^{-1}.These FTIR spectra confirmed the presence of Carboxylic Acids, Alkane, Oxygen compound, Nitrogen compound, H-bonded OH in the extractions of gourd leaves and teak leaves.

Table 1. Absorbance and wavelength of gourd leaves from absorption spectrum

No	Absorbance(a.u)	Wavelength(nm)
1	1.117	671.00
2	0.665	613.20
3	0.884	540.00
4	3.506	427.60

Table 2. Absorbance and wavelength of teak leaves from absorption spectrum

No	Absorbance (a.u)	Wavelength(nm)
1	1.153	673.00
2	1.470	504.50
3	1.862	416.00
4	4.000	272.50

Table 3. Absorbance and wavelength of gourd and teak leaves from absorption spectrum

No	Absorbance (a.u)	Wavelength (nm)
1	0.822	672.00
2	3.691	363.50
3	4.000	326.50
4	4.000	303.50
5	4.000	295.50

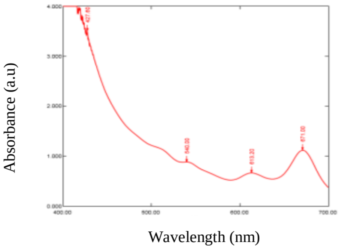

Fig.7. Absorbance spectrum of gourd leaves

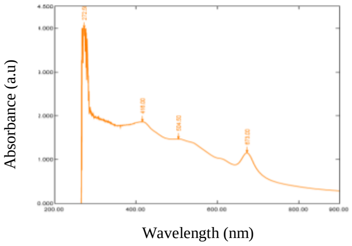

Fig. 8. Absorbance spectrum of teak leaves

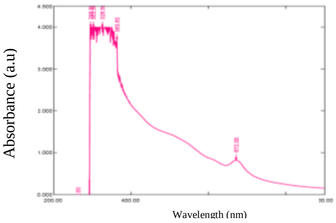

Fig.9. Absorbance spectrum of gourd and teak leaves

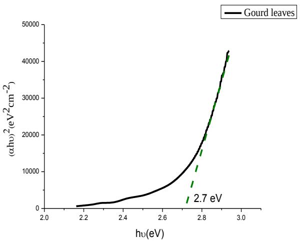

Fig.10 Plot of $(\alpha h\upsilon)^2$ versus photon energy $(h\upsilon)$ of gourd leaves

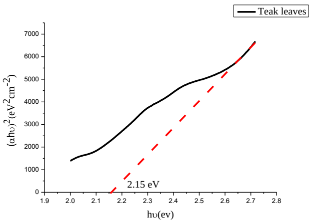

Fig.11 Plot of $(\alpha h\upsilon)^2$ versus photon energy $(h\upsilon)$ of teak leaves

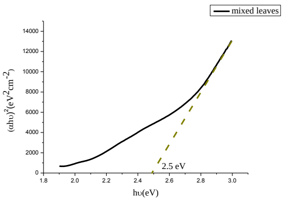

Fig.12 Plot of $(\alpha h\upsilon)^2$ versus photon energy $(h\upsilon)$ of mixed leaves

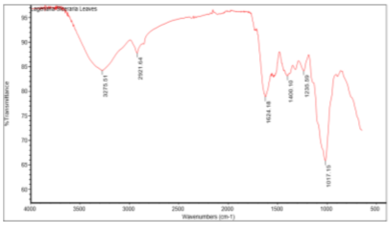

Fig. 13. FTIR spectra for dyes extracted from gourd leaves

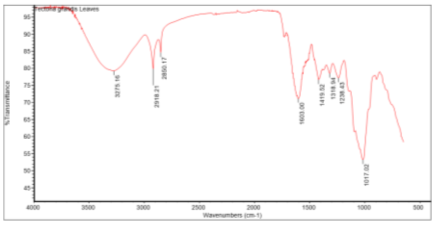

Figure 14 FTIR spectra for dyes extracted from teak leaves

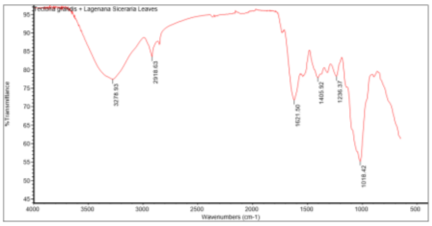

Fig. 15. FTIR spectra for dyes extracted from mixed leaves

4. CONCLUSIONS

The natural dye extracted from leaves of gourd and teak were used to build dye-sensitized solar cells. It was characterized by UV-vis spectrophotometer. The absorption spectrum from UV-vis can give to calculate the maximum energy band gap. The results from this work for gourd and teak leaves dyes were 2.7 eV, 2.15 eV, and 2.5 eV at 85°C of water. The results of FTIR showed gourd and teak dye solutions contained Carboxylic Acidic group and H bonded OH group. So the dye extracted in this research work may be applied in use for a part of dye sensitized solar cells.

ACKNOWLEDGEMENTS

We would like to thank the Materials Science Lab members, Department of Physics, University of Yangon.

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