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Extraction of Dyes from Sunflower Petal and Their Fourier Transform Infrared Characterization

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ABSTRACT

Three solvents of different polarities (water, methanol and 1% NaOH solution) were used to extract dyes that produced different shades from dried sunflower (*Helianthus annuus*) petal. The extraction procedures using different solvent types were carried out separately. The dye extracts were thereafter subjected to Fourier Transform Infrared Spectrometry (FT-IR) analysis for characterization in terms of functional groups. The intensities of the extracted dyes on the shade of colours obtained on pieces of cotton material varied from yellow in methanolic extract to light yellow in aqueous and black in 1% NaOH solution extracts. The results obtained from the FT-IR analysis revealed the presence of several useful functional groups such as N-H, C=H, O-H and C=O in the extracts.

1. Introduction

Chemical processes are influenced by the properties of solvents in which they are carried out^[1]. It was reported by^[2,3] that as the polarity of a solvent increase, its solubility and ability to solvate will decrease. Solvent effects have been associated with hydrogen bonding which assists electron migration in the molecules and

stabilization of preferred structures^[4]. Factors that determine solubility at either end of the polarity scale are complex and depend on combination of hydrogen donor acceptor strength and dipolar character. A strong solvent-solute interaction makes the process of solvation more favourable. Studies on effects of solvents of varying polarity on the reactivity and solubility of compounds have been reported^[2,3,5,6].

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The decline in extracting and using of natural dyes has been resuscitated due to the fact that they are eco-friendly, biodegradable, non toxic, non allergic ^[7,8] and do not stain other fabrics when bleeding ^[9]. Natural dyes are colourants obtained from different natural sources without any synthesizing. It includes all the dyes derived from different natural sources such as plants, animals and minerals ^[8]. Uddin *et al.* ^[10] reported that majority of vegetative origin such as roots, berries, barks, leaves, wood and other biological sources like fungi and lichens. Extraction of dye from different plant sources have also been documented by several other workers ^[11-13].

Sunflower (*Helianthus annuus*) is a common plant in Nigeria. It is a tall annual plant that can grow to a height of 300 cm or more in some species. They bear one or more wide flower head with yellow ray florets at the outside and yellow or maroon disc florets inside. The plant is perceived to be a drought tolerant crop as its roots can take up water at depth not reached by other crops. The use of various plant parts as dye or colouring matter can be expanded by understanding its solubility in different solvents. Different coloured extracts from a particular plant source can be achieved depending on extraction methods ^[14].

Numerous scientific methodologies such as ultra violet (UV) -visible, fourier transform infra red (FT-IR) and gas chromatography – mass spectrometry (GC-MS) methods among others are available to analyse the effects of the solvent treatments on the characteristics of organic compounds. FT-IR spectroscopic analysis is however one of the most common and perhaps the most powerful technique for identifying the type of functional groups in biological samples and offers high speed quantitative analysis without consumption or destruction of the sample ^[15]. Its structural identification is based on the interaction of atoms with infrared radiation. When infrared radiation interacts with an organic compound, certain frequencies of energy are absorbed or reflected and these frequencies are determined by the functional groups present in the substance. IR method can be used singly or in combination with other techniques of instrumental analysis to identify the actual chemical composition of a material ^[16]. Several workers ^[17-21] reported the use of FT-IR for bioactive ingredients determination in different plant extracts. Reports on the dye extracts from sunflower petal using different solvents are relatively unavailable in the searched literature, therefore this research aimed at extracting dyes from sunflower petal using methanol, water and 1% NaOH solution. The extracted dyes are to be characterized using fourier transform infrared spectroscopy to determine the functional groups present in

each of the extracts in order to ascertain if they are good dyes or otherwise.

2. Materials and Methods

2.1 Sample Collection

The sunflower (*Helianthus annuus*) petals were collected behind the Directorate of Entrepreneurship Education Centre of Osun State Polytechnic, Iree in the month of October, 2020. After collection, attached leaf and sepals were separated from the petals. The sample was identified as *Helianthus annuus* petals by Mr Akinro Ebenezer Babatope (Biology unit) of the Department of Science Laboratory Technology, Osun State Polytechnic, Iree, Nigeria.

2.2 Sample Preparation

The sunflower petals collected were air dried at room temperature for 14 days, sorted to further remove unwanted parts, pulverised using an electric Laboratory blender, passed through a 2 mm sieve and stored in a polythene bag kept over silica gel in a dessicator and ready for further analysis.

2.3 Extraction of Dye

Extraction of the dye was done using three different solvents; water, 1% NaOH, and analytical grade methanol.

2.3.1 Aqueous Extraction

The extraction of dye using water was carried out using the following procedures 20 g of the prepared sample was weighed and 400 mL of deionised distilled water was added over a water bath for 60 min at 60 °C. The mixture was removed from the water bath and left for 48 h after which it was filtered through Whatman No. 1 filter paper. Another 200 mL of deionised water was added to the residue and left for 24 h before it was filtered as above. The residue was further washed continuously with two more portions of 50 mL deionised distilled water for adequate extraction. The supernatants was pooled together, evaporated at 60 °C until the volume was reduced to 100 mL and stored in air tight bottles ready for further analysis.

2.3.2 1% NaOH Extraction

The extraction of dye using 1% NaOH solution was carried out using the following procedures. 20 g of the prepared sample was weighed into a 500 mL conical flask; 50 mL of the solvent was added and kept for 3 days. The extracts were filtered using Whatman No. 1 filter paper

and the supernatant was collected. The residue was further extracted two more times using 25 mL of the extracting solvent with 3 days' interval for each extraction. Each of the supernatants was separately pooled together and stored in air tight bottles ready for further analysis.

2.3.3 Methanolic Extraction

The dye extractions using methanol was done with a little modification to Ashokkumar and Ramaswamy^[17] methods. 20 g of the sample was weighed into a 500 mL conical flasks; 150 mL of the solvents was added and kept for 3 days. The extract was thereafter filtered using Whatman No. 1 filter paper and the supernatants collected. The residue was further extracted two more times as above with 3 days' interval. The supernatants was pooled together, evaporated on a thermostatic water bath at 40 °C until the volume was reduced to 100 mL and stored in air tight bottle ready for further analysis.

2.3.4 FT-IR Analysis

The FT-IR analysis was carried out by encapsulating 30 mg of each extracts into 300 mg of KBr (dried at 80°C for 24 h to completely remove moisture) pellet of spectroscopic grade purity using a small agate mortar until the sample was completely mixed with the KBr powder to produce a translucent sample discs. IR spectra regions and peaks produced were recorded at room temperature on

Perkin-Elmer Fourier Transform spectrometer (RX model) Norwalk, CT, USA for the sample in each solvent with a scan range between 4000 - 400 cm^{-1} (1% NaOH), 4000 - 450 cm^{-1} (methanol) and 4000 - 500 cm^{-1} (water).

3. Results and Discussion

Table 1. Observed colours on the piece of cloth dyed with the different extracts

xtract	Piece of cloth	Colour observed
Methanol	Cotton material	Yellow
Aqueous	Cotton material	Light yellow
1% NaOH	Cotton material	Black

The visually observation of colours on the pieces of cotton materials dyed with the different extracts from the sunflower petal are shown in Table 1. The intensities of the shade of the colours obtained varied from yellow in methanolic extract to light yellow in aqueous and black in 1% NaOH extracts. The variation in colour intensities of the dyed materials may be adduced to type of solvents used and the observed different functional groups such as N-H, C=H, O-H and C=O groups in the extract. This is at variance with the report on dye extracted from *Rothmannia whitfieldii* where alkali extraction gave a deep brown colour liquid and aqueous extraction gave a black colour liquid^[14].

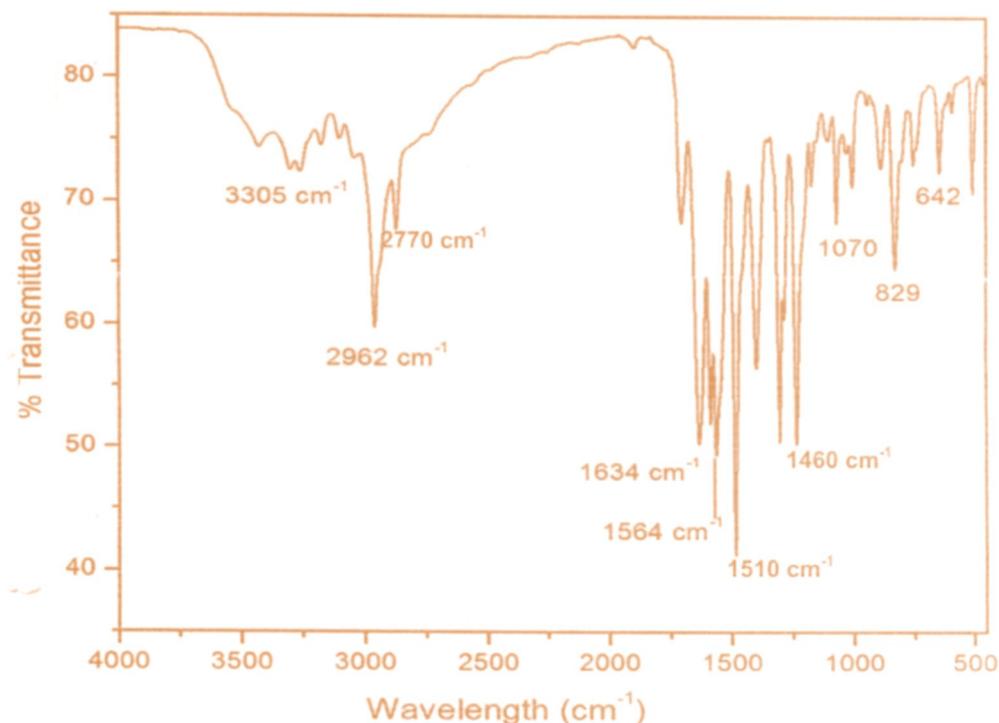


Figure 1. FT-IR spectral of aqueous dye extract

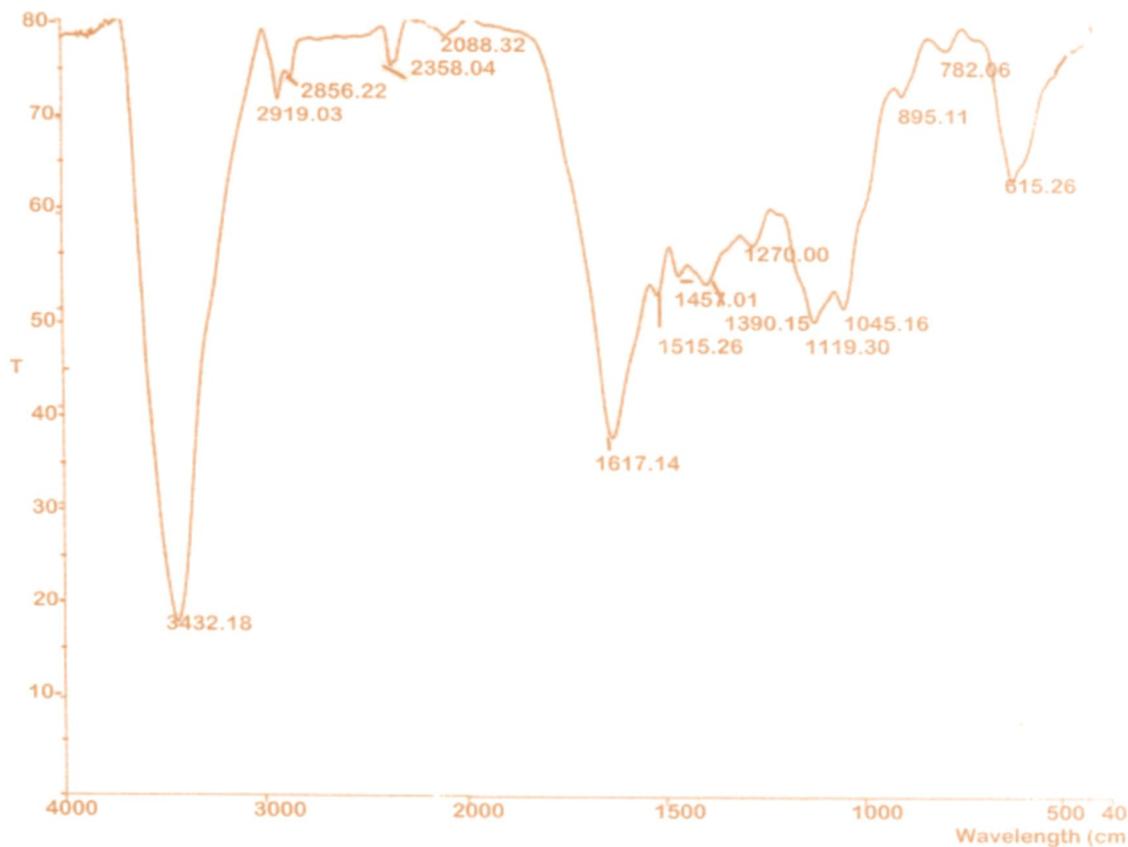


Figure 2. FT-IR of the 1% sodium hydroxide extract

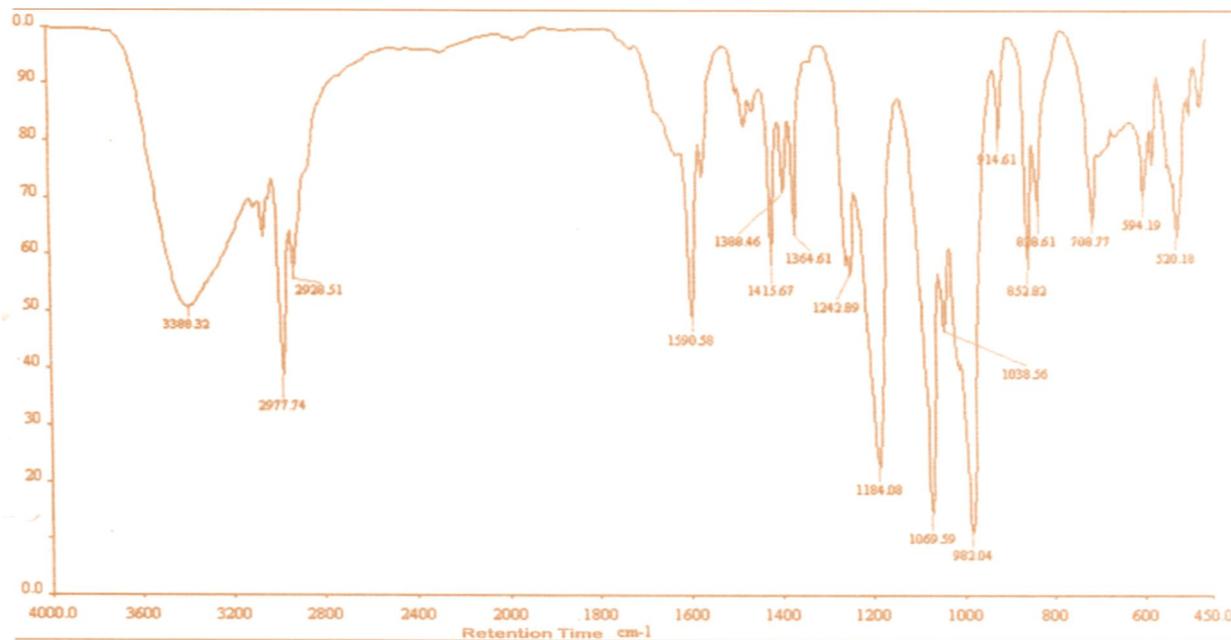


Figure 3. FT-IR of the methanolic dye extract

The results of the infrared spectral analysis of the various dye extracts from the dried sunflower petal using water, methanol and 1% NaOH respectively as the extracting solvents (Figures 1, 2, & 3) revealed the presence of various peaks ranging from C-H stretching, O-H, C=O, N-H and other several bands. The aqueous dye extract under investigation showed the presence of C-H stretching at wavenumber of 2770 cm^{-1} and amide group with N-H bending at 1564 cm^{-1} .

The presence of chromophore and auxochrome in each of the extracts reflected in the shade of colours observed. Chromophores are represented as nitrogen, carbon and oxygen which usually have single or double bonds which are primarily essential for colour formation. The parent compounds containing them are termed chromogen. The auxochromes are polar groups that greatly increase the colour yielding power of chromophores^[22] but when present alone will fail to produce that colour hence they are called colour helpers.

The following peaks were observed in the aqueous extract; 3305 cm^{-1} observed is assignable to H-bonded and O-H stretching vibration, 2770 cm^{-1} related to C-H stretching while 1460 cm^{-1} assigned for O-H bending was possibly from alcoholic group. The peaks observed at 1725 cm^{-1} or 1607 cm^{-1} could be assigned to C=O bending to stretch vibration. The methanolic extract revealed a wavelength of 1364.61 cm^{-1} of bending vibration of benzene derivatives while the C=C-C functional group is an indication of the presence of aromatic rings. The NaOH extract revealed the presence of N-H₄⁺ functional group in it while the peak value at 1617.14 cm^{-1} possibly represents C=C stretching vibration due to aromatic ring deformation. The peaks at 3432.18 cm^{-1} and 2919.03 cm^{-1} are assignable to O-H stretching vibration.

The O-H group that was found to be present uniformly in all the extracts has the ability to form hydrogen bond which is an indication of higher potential towards inhibitory activity against microorganisms^[17]. Some of the peaks observed for the aqueous and methanolic extracts of *H. annuus* petals were similar to those observed for the yellow dye of *Carthamus tinctorius* by Shin and Dong^[23]. The IR spectra obtained from this work could be compared with the standard dye databases from literature^[14,16,24] or any available literature that contain IR spectra of several classes of dyes with known chemical structures and composition to accurately placed the extracted dyes from the different solvents to enhance their uses.

4. Conclusions

The results of this research work showed that natural dye of different shades of colours can be extracted

successfully from the petal of sunflower using different solvents which could serve as an alternative to the synthetic colourants. The work also revealed that the dye extracts have different useful functional groups.

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