6.1 UV-Visible Spectra

6.1.1 UV-Visible Spectra of Pomegranate Dye Solution

The UV-Visible spectra obtained for the pomegranate dye solution as shown in fig. 6.1. Spectra exhibited $\lambda_{\text{Max}}$ at 424nm and 460 nm.

![UV-Visible Spectrograph of Aqueous Pomegranate Dye](image)

Fig. 6.1: UV-Visible Spectrograph of Aqueous Pomegranate Dye

6.1.2 UV-Visible Spectra of Marigold Dye

UV-Visible spectra of marigold dye shown in figure 6.2 shows $\lambda_{\text{Max}}$ at 474nm characteristic peak obtained for xanthophylls.
6.1.3 UV-Visible spectra of Red Onion Dye

The colorant was dissolved in methanol-HCl (pH-2) and scanned between 200-800 nm (Systronics-PC based Double Beam Spectrophotometer-2202). The peaks at 368 and 526 nm are characteristics of anthocyanins. (Fig. 6.3a) These peaks are suggestive of cyanin and peonidinglycone, which are the anthocyanin responsible for deep red color (Da costa et al., 2000). The methanol-HCl extracts of red onion peels having deep red color mainly contains anthocyanins and flavonols. Color of aqueous dye solution was found to change with pH. The pH of the solution was maintained by adding either HCl or Na₂CO₃. The extract solutions were scanned in the range of 200-800 nm for the pH ranges 1.5-2 and 6-6.5. (Figure 6.3b,c). When the pH of the dye bath was decreased below 4, a bathochromic shift took place and the color became deep red having $\lambda_{\text{max}}$ 368 and 526 nm. However, at pH 6-6.5 the dye solution was dirty blue-green having $\lambda_{\text{max}}$ 412 and 471 nm. This change in color may be attributed to change in conjugation in anthocyanins and flavonols at different pH values. (Scheme 1)
Fig. 6.3 a: UV-Visible Spectrograph of Aqueous Red Onion Dye

Fig. 6.3 b: UV-Visible spectrum of colorants from red onion in HCl-water at pH-2
Fig. 6.3 c: UV-Visible spectrum of colorants from red onion in aqueous Na2CO3 at pH-6.4.

6.1.4 UV-Visible Spectra of Dahlia dye

In UV spectroscopic scanning of dye-methanol solution shown in Fig. 6.4a, the peaks 281 and 502 nm are characteristics of anthocyanins. The peak are suggestive of cyanin and delphidine, which are the anthocyanin responsible for deep red colour.

UV spectroscopic analysis showed that the colour of aqueous dye solution changes with pH, which was accomplished by adding either HCl or Na2CO3. The scanning between 200-800 nm was carried out at pH 2.0 and 8.0 (Fig. 3) by diluting the colorant solution in order to get absorbance below 4.00. When the pH of the dye bath was decreased 8 to 2, a bathochromic shift took place and the colour of the dye bath solution became deep red and the $\lambda_{\text{max}}$ were 281 and 521 nm. However, at pH 8.0 the dye solution was blue-green and the peak at 521 vanished as shown in Fig. 6.4b.
Again this change in colour may be attributed to change in conjugation in anthocyanins and flavonols at different pH values. (Scheme 1)

Fig. 6.4 a.- UV-Visible spectra of Dahlia dye in methanol

Fig. 6.4 b - UV-Visible Spectra of Dahlia dye: Effect of changing pH from 2 to 8.0 on colour
Scheme 6: Effect of pH on chemical structures of anthocyanidins in aqueous solution
6.2 High-Performance Liquid Chromatography

Fig 6.5 A: HPLC analysis of the Marigold dye solution dyeing

B: HPLC analysis of the Marigold dye after dyeing
Fig. 6.6A: HPLC of red onion dye solution before dying

B: HPLC of red onion dye solution after dyeing
From the above plotted graphs (Fig. 6.5A, B and 66A, B) it can be observed that there is a considerable decrease in the absorbance peaks after dying resembling the attachment of the dyes to the textile. This makes us understand that the extracted dye has the colouring component that can be applicable for textile dying.

6.3. FTIR

6.3.1 FTIR of Pomegranate dye

Infrared (IR) spectra of the pomegranate rind dye (Fig. 6.7) has been recorded on KBr, revealed the presence of hydroxyl group at 3421 cm\(^{-1}\), CH stretching at 2935 cm\(^{-1}\), C=O stretch at 1648 cm\(^{-1}\) (due to conjugation with aromatic nucleus peaks shifted towards lower value), C-O stretching at 1184 cm\(^{-1}\) and 1066 cm\(^{-1}\). Aromatic C-H ring bending appeared at 638 cm\(^{-1}\).

![FTIR Spectra of Dye from pomegranate rind](image)
6.3.2 FTIR of Marigold Dye

Infrared (IR) spectra of the marigold dye (Fig. 6.8) has been recorded on KBr, revealed the presence of hydroxyl group at 3332 cm$^{-1}$, CH stretching at 2935 cm$^{-1}$, C=O stretch at 1722, Aromatic nucleus at 1512 cm$^{-1}$ and 1450 cm$^{-1}$, C-O stretching at 1266 cm$^{-1}$ and 1165 cm$^{-1}$. Aromatic C-H ring bending appeared at 825 cm$^{-1}$.

Fig. 6.8 : FTIR of Marigold Dye
6.3.3 FTIR of Red Onion Dye

Infrared (IR) spectra of the red onion dye (Fig. 6.9) has been recorded on KBr, revealed the presence of hydroxyl group at 3383 cm\(^{-1}\), CH stretching at 2927 cm\(^{-1}\), C=O stretch at 1623 cm\(^{-1}\) (due to conjugation with aromatic nucleus peaks shifted towards lower value), Aromatic nucleus at 1512 cm\(^{-1}\), C-O stretching at 1262 cm\(^{-1}\) and 1066 cm\(^{-1}\). Aromatic C-H ring bending appeared at 639 cm\(^{-1}\).

Figure 6.9. FTIR of Red Onion dye
6.3.4 FTIR of Dahlia Dye

In the FT-IR spectrum of dye synthesized by UAE shown in Fig. 6.10, a broad peak appearing at 3100–3600 cm\(^{-1}\) assigned to fundamental stretching vibration of O–H hydroxyl groups, The absorption band at 1640 cm\(^{-1}\) is due to >C=O conjugated with aromatic ring, 1508 and 1468 cm\(^{-1}\) correspond to Aromatic C=C and 1275 for Phenol C-O.

![FTIR peaks obtained for all dyes are indicative of flavonoids, most possibly anthocyanins, like cyanidin and delphinidin glycones, and flavonols like quercetin aglycone](image)

**Fig. 6.10 : FTIR of Dahlia Dye**

FTIR peaks obtained for all dyes are indicative of flavonoids, most possibly anthocyanins, like cyanidin and delphinidin glycones, and flavonols like quercetin aglycone (*Bilyk and cooper, 1984*).

6.4 SEM

The microstructure of tissue of red red onion after solvent extraction, UAE, EAE and EUAE was investigated by SEM. The significant difference were revealed in figures 6.11a,b,c and d.
The structure of red onion peels after UAE, EAE and EUAE was looser than that after solvent extraction. It seems that ultrasonic acoustic cavitation results in an explosive disruption of the physical structure of peels, leading to a direct migration of colouring material into the surrounding solvent because the acoustic cavitation, heating and mechanical action in peels occur under ultrasonic irradiation and thus increasing the dye extraction yield.

As plant tissues contain cellulose and pectin as binding materials, enzymes including Cellulase and Pectinase have been used to loosen the surrounding material leading to the extraction of dyes molecules with high yields.

Fig. 6.11 a : SEM of Red Onion Peel after solvent extraction
Analysis

Fig. 6.11b : SEM of Red Onion Peel after UAE

Fig. 6.11c : SEM of Red Onion Peel after EAE
Fig. 6.11d : SEM of Red Onion Peel after EUAE