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Extraction of natural dye (Flavonoid) from common marigold flower

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ABSTRACT

I tested the type of solvent to be used on sonicator and found out that alkaline solvent gives darker colour, while acids give lighter colour, and alcohol tend to give somewhat original colour of the flower petals from these batches I could conclude that ethanol was the best solvent. I tested the ratio of natural source (gm) and solvent (ml) on heating mantle and found that ratio of 1:20 was better than ratio of 1:10 and temperature of 80°C and 100°C were optimum. I carried out batches on ultrasound probe and found that temperature of 50°C was optimum. After this batches on microwave reactor were carried out and found that power of 320 watt was optimum. In soxhlet extraction optimum time was 1 & ½ hour and after 1 hour solution retained was constant. Pre mordanting dyeing was done for all the different methods.

Keywords: UV spectrophotometer, flavonoid, & marigold.

INTRODUCTION

Natural dyes are dyes or colorants derived from plants, invertebrates, or minerals. Biological pigments or biochromes are substances produced by living organisms that have a color resulting from selective color absorption. The primary function of pigments in plants is photosynthesis, which uses the green pigment chlorophyll along with several red and yellow pigments that help to capture as much light energy as possible. Other functions of pigments in plants include attracting insects to flowers to encourage pollination. Plant pigments include a variety of different kinds of molecule, including porphyrins, carotenoids, anthocyanins and betalains. All biological pigments selectively absorb certain wavelengths of light while reflecting others. The light that is absorbed may be used by the plant to power chemical reactions, while the reflected wavelengths of light determine the color the pigment will appear to the eye. A plant usually contains a mixture of natural dyes. This mixture is often extracted and used to dye textiles. Common marigold contains flavanoid. Which exhibits yellow to red color. Flavonoids the word is derived from the Latin word flavus meaning yellow. There are four main types of flavonoids and other less common ones. Major ones are flavones, flavonols, anthocyanidins and anthocyanins. Flavones and flavonols have yellowish colors. Anthocyanidins and anthocyanins are the most highly colored of the flavonoids. Biochromes are extracted and used as dyes, dietary supplements, and as food flavor [6].

MATERIALS AND METHODS

1) Procedure for Sonicator Method:

In this method I took 10 gm of natural source i.e. flaked marigold flower petals and 100ml of solvent. Solvents used were water, methanol, ethanol, butanol, benzene, toluene, 1ml H2SO4+ 99ml H2O, 1ml HNO3 + 99ml H2O, 1ml HCl + 99ml H2O, 1ml acetic acid + 99ml H2O, NaOH, KOH, methyl acetate, ethyl acetate, and n-butyl acetate. These batches were kept at 45°C for 1 hour and then filtration was done. After filtration analysis of dye was done on UV spectrophotometer to find out maximum absorbance for maximum wavelength.

2) Procedure for Direct Heating (1:20):

In this method I took 10 gm of natural source i.e. flaked marigold flower petals and 200ml of solvent. Solvent used was distilled water. Time was 1hour. Temperature was changing parameter. Batches carried at different temperature were 100°C, 80°C, 60°C, 40°C, and 20°C. And then filtration was done. After filtration analysis of dye was done on UV spectrophotometer to find out maximum absorbance for maximum wavelength.

3) Procedure for Direct Heating (1:10):

In this method I took 10 gm of natural source i.e. flaked marigold flower petals and 100ml of solvent. Solvent used was distilled water. Time was 1hour. Temperature was changing parameter. Batches carried at different temperature were, 80°C, 60°C, 40°C, and 20°C. And then filtration was done. After filtration analysis of dye was done on UV spectrophotometer to find out maximum absorbance for maximum wavelength.

4) Procedure for Ultrasound Probe Method:

In this method I took 10 gm of natural source i.e. flaked marigold flower petals and 200ml of solvent. Solvent used was ethanol. Time was 1hour. Frequency of the probe is 20 kHz. Temperature was changing parameter. Batches carried at different temperature were 30°C, 50°C, 70°C, and 90°C. And then filtration was done. After filtration analysis of dye was done on UV spectrophotometer to find out maximum absorbance for maximum wavelength.

5) Procedure for Microwave Method:

In this method I took 10 gm of natural source i.e. flaked marigold flower petals and 200ml of solvent. Solvent used was ethanol. Time was half an hour. Power was changing parameter. Batches carried at different power were 160W, 320W, 480W, 640W, and 800W. And then filtration was done. After filtration analysis of dye was done on UV spectrophotometer to find out maximum absorbance for maximum wavelength.

6) Procedure for Soxhlet Extraction Unit Method:

In this method I took 5 gm natural source i.e. flaked marigold flower petals and 200ml of solvent. Solvent used was ethanol. Temperature was 100°C. Time was changing parameter and batches were carried out at ½ hour, 1 hour 1&½ hour, and 2 hour. And then filtration was done. After filtration analysis of dye was done on UV spectrophotometer to find out maximum absorbance for maximum wavelength.

7) Procedure for different types of mordanting:

Pre mordanting dyeing was done for all the different methods. For microwave batch of 320 W pre mordanting, meta or simultaneous mordanting and post mordanting was done.

i. Procedure for pre mordanting dyeing:

It was done for all the different methods. Washing of khadi cotton cloth at 45 °C for 45 minutes in distilled water. Preparation of mordanting solution by adding 2gm alum in 40 ml water. Mordanting of khadi cotton at temperature of 45 °C in mordanting solution for 45 minutes. Dyeing of mordanted khadi cotton in 40 ml of extracted dye at temperature of 45 °C in for 45 minutes.

ii. Procedure for meta mordanting dyeing:

It was done only for microwave batch at 320 W. Washing of khadi cotton cloth at 45 °C for 45 minutes in distilled water. Preparation of mordanting solution by adding 2gm alum in 40 ml water. Simultaneous dyeing and mordanting of khadi cotton cloth in 40 ml dye and 40 ml mordanting solution 45 °C for 45 minutes.

iii. Procedure for post mordanting dyeing:

It was done only for microwave batch at 320 W. Washing of khadi cotton cloth at 45 °C for 45 minutes in distilled water. Preparation of mordanting solution by adding 2gm alum in 40 ml water. Dyeing of khadi cotton in 40 ml of extracted dye at temperature of 45 °C for 45 minutes. Mordanting of dyed khadi cotton at temperature of 45 °C in mordanting solution for 45 minutes.

RESULTS AND DISCUSSION

A. Analytical Results:

Analysis was done under UV Spectrophotometer. Maximum wavelength for maximum absorbance was observed and also absorbance at 1100nm. Following are the analytical results.

1) Sonicator Method Results:

Natural source: 10 gm marigold flower

Temperature: 45 °C Time: 1 hour

Ratio of natural source v/s solvent is 1 gm:10 ml

Solvent (100 ml)	Absorbance at Maximum Wavelength	Maximum Wavelength (nm)	Absorbance at 1100 nm	Amount of Solvent Retained (ml)
Water	2.503	205	0.036	97
Methanol	2.531	324	0.519	78
Ethanol	2.517	324	0.807	85
Butanol	2.533	324	0.099	85
Benzene	2.508	207	0.112	79
Toluene	2.204	202 & 260	0.102	79
1ml H_2SO_4+ 99ml H_2O	2.525	324	0.152	90
1ml HNO_3 + 99ml H_2O	2.524	324	0.162	91
1ml HCl + 99ml H_2O	2.540	324	0.172	85
1ml acetic acid + 99ml H_2O	2.326	324	0.160	93
NaOH	2.526	324	0.172	80
KOH	2.515	324	0.206	68
Methyl acetate	2.518	324	0.271	81
Ethyl acetate	2.522	324	0.077	85
n-butyl acetate	2.513	324	0.453	77

Graph: Absorbance of Solvent's at 1100 nm

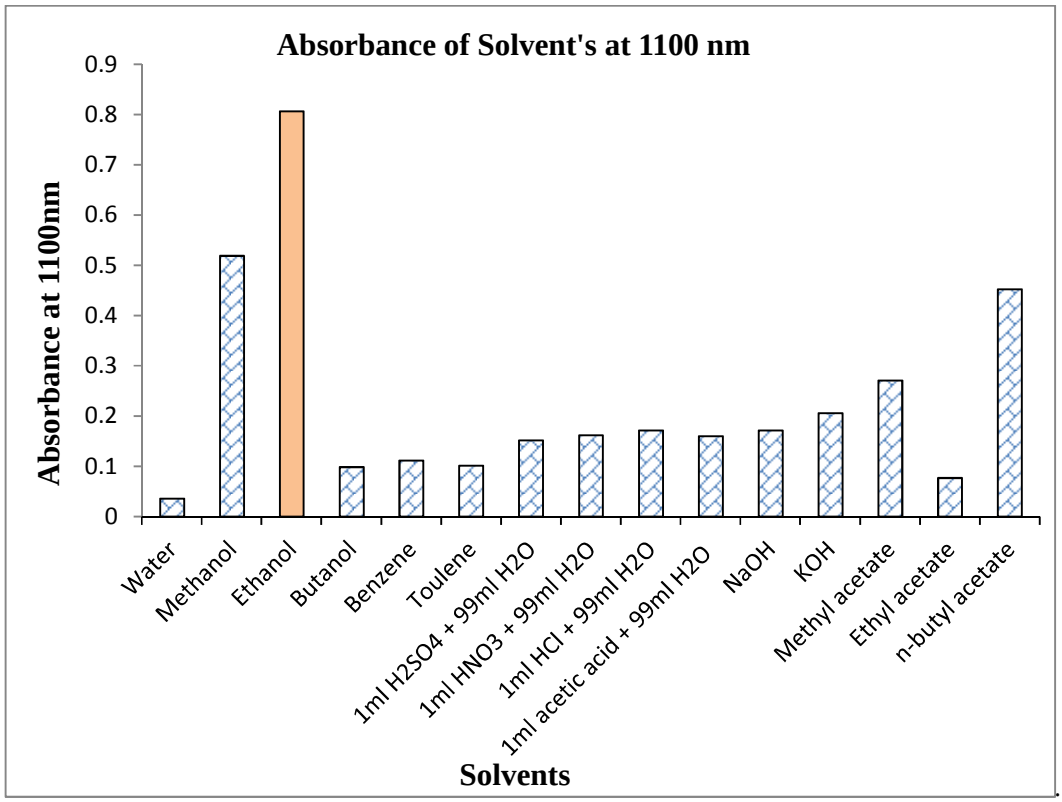


Fig. 1: Sonicator solvent selection bar graph

2) Direct Heating Method (1:20) Results:
Natural source: 10 gm marigold flower
Solvent: 200 ml distilled water Time: 1 hour
Ratio of natural source v/s solvent is 1 gm:20 ml

Temperature °C	Absorbance at Maximum Wavelength	Maximum Wavelength (nm)	Absorbance at 1100 nm	Amount of Solvent Retained (ml)
100	324	2.509	0.656	72
80	324	2.525	0.154	100
60	324	2.529	0.416	140
40	324	2.538	0.161	187
20	302	2.504	0.160	198

3) Direct Heating Method (1:10) Results:
Natural source: 10 gm distilled marigold flower
Solvent: 100 ml water Time: 1 hour
Ratio of natural source v/s solvent is 1 gm:10 ml

Temperature °C	Absorbance at Maximum Wavelength	Maximum Wavelength (nm)	Absorbance at 1100 nm	Amount of Solvent Retained (ml)
100	324	2.508	0.587	15
80	324	2.529	0.288	50
60	324	2.520	0.222	65
40	324	2.534	0.199	77.5
20	211	2.173	0.173	89

Graph: Absorbance at 1100 nm v/s Temperature °C

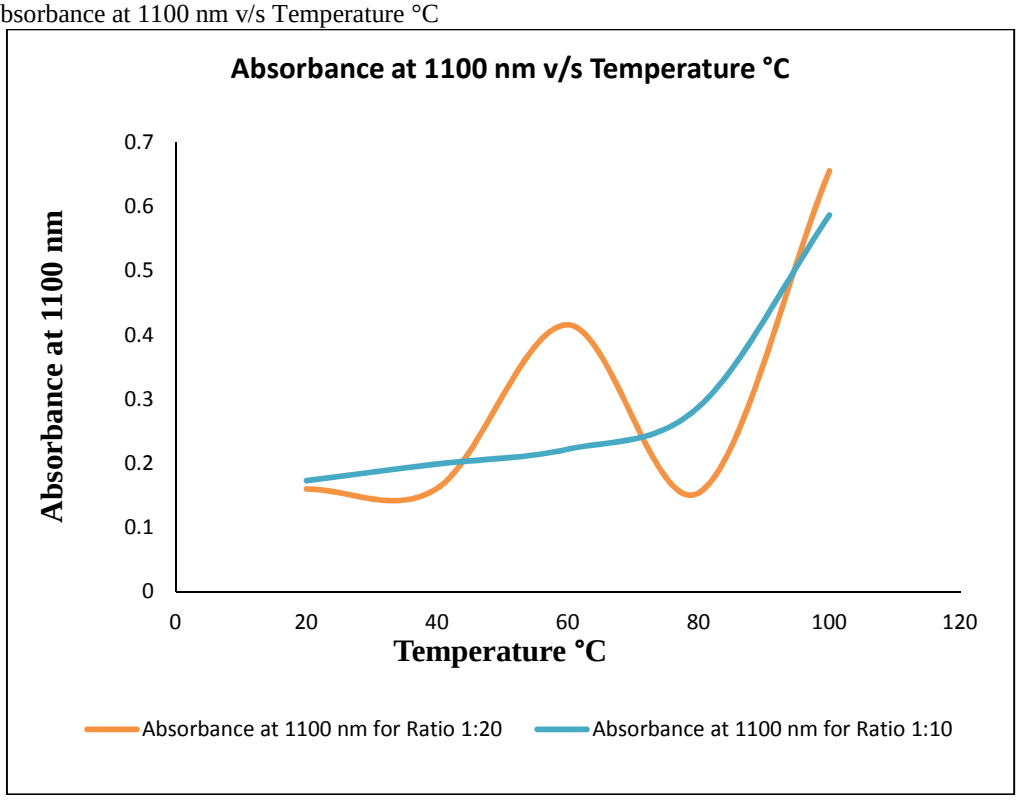


Fig. 2: Direct heating method graph

4) Ultrasound Probe Method Results:
Natural source: 10 gm marigold flower Frequency: 20 kHz
Solvent: 200 ml ethanol Time: 1 hour
Ratio of natural source v/s solvent is 2 gm:10 ml

Temperature °C	Absorbance at Maximum Wavelength	Maximum Wavelength (nm)	Absorbance at 1100 nm	Amount of Solvent Retained (ml)
30	2.512	324	0.258	181
50	2.499	324	0.762	179
70	2.517	324	0.333	150
90	2.500	324	1.230	138

Graph: Absorbance at 1100 nm v/s Temperature °C

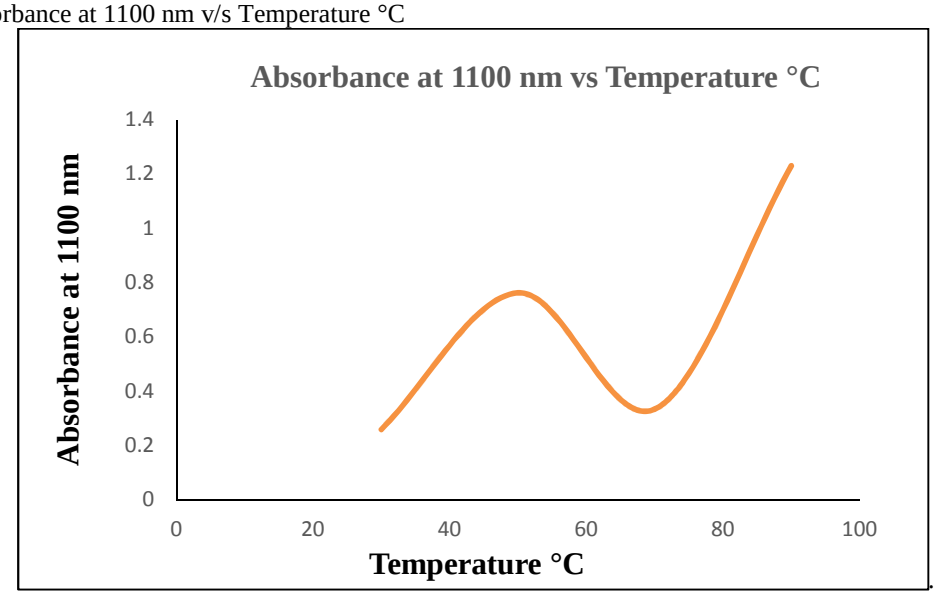


Fig. 3: Ultrasound probe method graph

5) Microwave Method Results:
Natural source: 10 gm marigold flower
Solvent: 200 ml ethanol Time: half an hour
Ratio of natural source v/s solvent is 2 gm:10 ml

Power watt	Absorbance at Maximum Wavelength	Maximum Wavelength (nm)	Absorbance at 1100 nm	Amount of Solvent Retained (ml)
160	2.513	324	0.456	189
320	2.499	324 & 336	1.350	175
480	2.498	324	1.151	170
640	2.540	321 & 324	1.162	163
800	2.530	323 & 324	1.171	159

Graph: Absorbance at 1100 nm v/s Power W

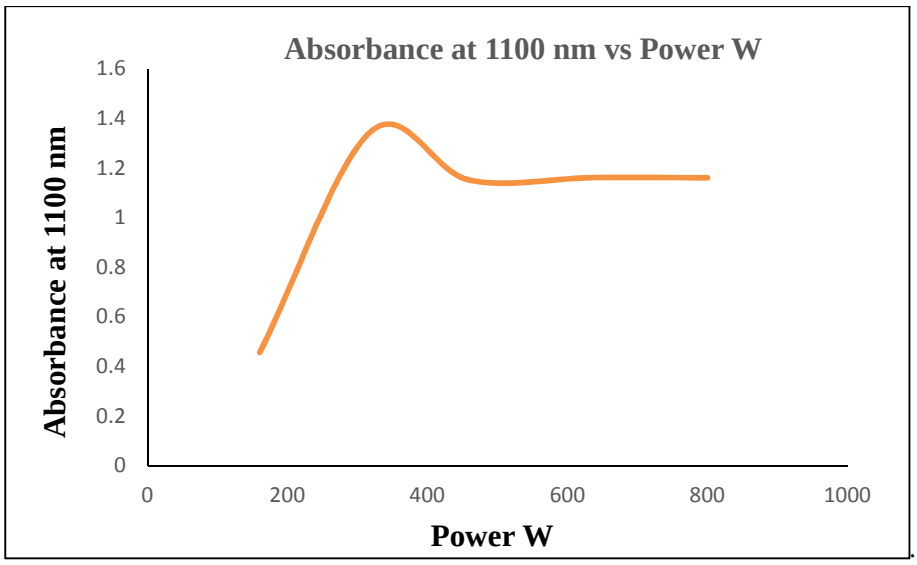


Fig. 4: Microwave method graph

6) Soxhlet Extraction Unit Method Results:
Natural source: 5 gm marigold flower
Solvent: 200 ml ethanol Temperature: 100 °C
Ratio of natural source v/s solvent is 1 gm:40 ml

Time hour	Absorbance at Maximum Wavelength	Maximum Wavelength (nm)	Absorbance at 1100 nm	Amount of Solvent Retained (ml)
½	2.478	324	0.275	192
1	2.505	324 & 337	0.403	186
1 & ½	2.517	324 & 343	1.295	186
2	2.525	324 & 347	1.573	186

Graph: Absorbance at 1100 nm v/s Time hour

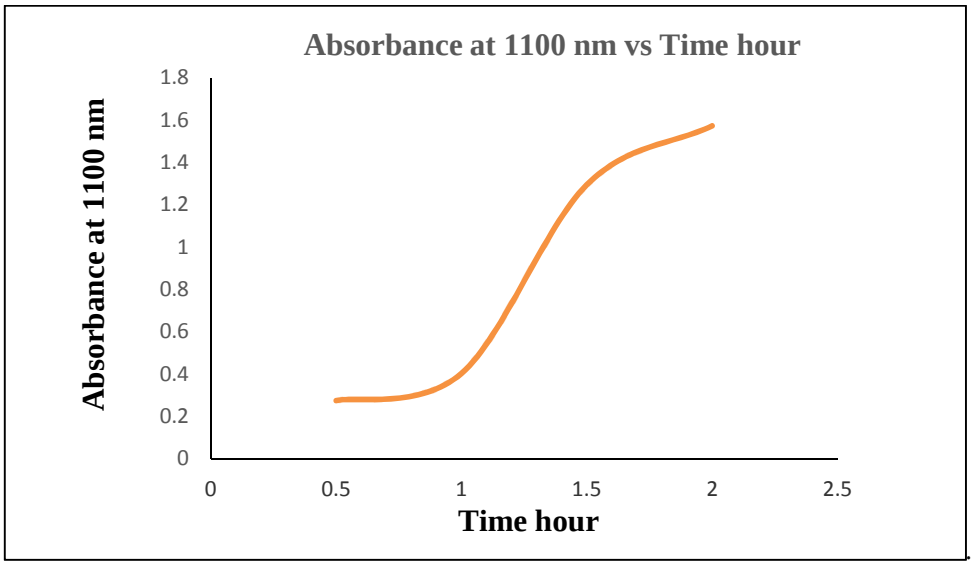


Fig. 5: Soxhlet extraction unit method graph

B. Dyed Cloth Results

1) Sonicator Method: Pre mordaning dyeing was done on all the batches.

i. Acids: Acids gave light color shade for marigold dye.

1ml H_2SO_4+ 99ml H_2O

1ml HNO_3 + 99ml H_2O

1ml HCl + 99ml H_2O

1ml acetic acid + 99ml H_2O

Fig. 5: Sonicator Method Dyed Cloth (Acids)

ii. Alkalis: Alkalis gave darker color shade marigold dye.

NaOH

KOH

Fig. 6: Sonicator method dyed cloth (Alkalis)

iii. Alcohols: Alcholos gave best color shade than any other solvents for marigold dye. Ethanol was optimum solvent as it gave best color quality.

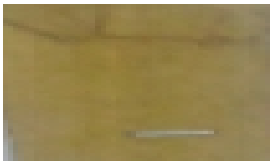
Methanol

Ethanol

Butanol

Fig. 7: Sonicator method dyed cloth (Alcohol)

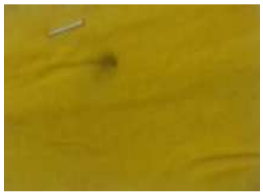
iv. Other Solvents: Different shades are obtained by using different solvents.



Water

Benzene

Toluene



Methyl acetate

Ethyl acetate

n-butyl acetate

Fig. 8: Sonicator method dyed cloth (Other Solvents)

2) Direct Heating Method (1:20): In this method temperature was changing parameter and pre mordaning dyeing was done. From the color shade optimum temperature is 100 °C.

20 °C 40 °C 60 °C 80 °C 100 °C

Fig. 9: Direct heating method (1:20) dyed cloth

3) Direct Heating Method (1:10): In this method temperature was changing parameter and pre mordaning dyeing was done. From the color shade optimum temperature is 100 °C.

20 °C 40 °C 60 °C 80 °C 100 °C

Fig. 10: Direct heating method (1:10) dyed cloth

4) Ultrasound Probe Method: In this method temperature was changing parameter and pre mordaning dyeing was done. From the color shade optimum temperature is 50 °C.

30 °C 50 °C 70 °C 90 °C

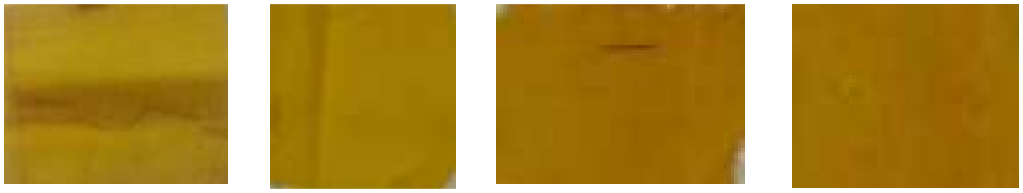
Fig. 11: Ultrasound probe method dyed cloth

5) Microwave Method: In this method power was changing parameter and pre mordaning dyeing was done. From the color shade optimum Power is 320 W.

160 W 320 W 460 W 640 W 800 W

Fig. 12: Microwave method dyed cloth

6) Soxhlet Extraction Unit Method: In this method time was changing parameter and pre mordaning dyeing was done. From the color shade optimum time was 1 & ½ hour.



½ hour 1 hour 1 & ½ hour 2 hour

Fig 13. Soxhlet extraction unit method dyed cloth

7) Different Types of Mordanting: Three types of mordanting were done on microwave batch of 320 W they are as follows: Meta mordanting dyeing gave lighter shade than pre mordanting. Post mordanting dyeing gave lightest shade as compared to pre mordanting and meta mordanting.

Pre mordanting dyeing Meta mordanting dyeing Post mordanting dyeing

Fig. 14: Different types of mordanting

C. Discussions

1) Sonicator Method:

Ethanol is the optimum solvent for extraction as the color quality obtained from it is very good. Ethanol also had maximum absorbance. Acids tend to give lighter color shade. Alkalis give darker color shade. Alcohol tend to give original color shade. Extracted dye from marigold flower was flavonoid and sub type for marigold is flavones. This was concluded from color extracted and from maximum wavelength obtained, that was 324 nm for most of the batches and which is within the flavonoid range of 200-500 nm, and for flavones 310-350 nm and 250-280 nm.

2) Direct Heating Method (1:20):

From the color shade optimum temperature is 100 °C. More amount of solvent is retained in this batch as compare to direct heating method (1:10). Extracted dye from marigold flower flavonoid and sub type is flavones. This was concluded from color extracted and from maximum wavelength obtained, that was 324 nm for most of the batches and which is within the flavonoid range of 200-500 nm, and for flavones it is 310-350 nm and 250-280 nm. Even though absorbance is decreasing and then increasing in the graph the color quality is also increasing.

3) Direct Heating Method (1:10):

From the color shade optimum temperature is 100 °C. Less amount of solvent is retained in this batch as compare to direct heating method (1:20). Extracted dye from marigold flower flavonoid and sub type is flavones. This was concluded from color extracted and from maximum wavelength obtained, that was 211 nm, and 324 nm for most of the batches, which is within the flavonoid range of 200-500 nm, and for flavones it is 310-350 nm and 250-280 nm. In this absorbance is increasing in the graph and so is the color quality.

4) Ultrasound Probe Method:

From the color shade optimum temperature is 50 °C. As after 50 °C the color quality is constant. Extracted dye from marigold flower flavonoid and sub type is flavones. This was concluded from color extracted and from maximum wavelength obtained, that was 324 nm for most of the batches and which is within the flavonoid range of 200-500 nm, and for flavones it is 310-350 nm and 250-280 nm. Even though absorbance is decreasing and then increasing in the graph the color quality is remaining constant after 50 °C. Color quality obtained is better than direct heating method (1:20) and direct heating method (1:10).

5) Microwave Method:
From the color shade optimum power is 320 W. As after 320 W the color quality is constant. Extracted dye from marigold flower flavonoid and sub type is flavones. This was concluded from color extracted and from maximum wavelength obtained, that was321 nm, 323 nm,324 nm, and 336 nm for most of the batches and which is within the flavonoid range of 200-500 nm, and for flavones it is 310-350 nm and 250-280 nm. Even though absorbance is increasing and then decreasing in the graph the color quality is remaining constant after 320 W. Color quality obtained is better than direct heating method (1:20) and direct heating method (1:10) and ultrasound probe method. Time required is also less for extraction it half an hour and for other batches it is 1 hour. Because of the above reasons this batch is favorable than other batches in terms of time and color quality obtained.

6) Soxhlet Extraction Unit Method:
From the colour shade optimum time is 1 & ½ hour. As after 1 & ½ hour the colour quality is constant. Solvent retained is constant after 1 hour. Extracted dye from marigold flower flavonoid and sub type is flavones. This was concluded from colour extracted and from maximum wavelength obtained, that was 324 nm for most of the batches and which is within the flavonoid range of 200-500 nm, and for flavones it is 310-350 nm and 250-280 nm. Even though absorbance is increasing in the graph the colour quality is remaining constant after 1 & ½ hour.

7) Different Types of Mordanting:
Different color shade is obtained from different mordanting dyeing. Pre mordanting gives bright color. Meta mordanting gives light color. And post mordanting gives lightest color shade.

CONCLUSION

Ethanol is the optimum solvent for extraction as the colour quality obtained from it is very good. For direct heating method optimum temperature is 100 °C. More amount of solvent is retained in direct heating method (1:20) as compare to the direct heating method (1:10).For ultrasound probe optimum temperature is 50 °C, as after 50 °C the colour quality is constant. For microwave optimum power is 320 W, as after 320 W the colour quality is constant. Different colour shade is obtained from different mordanting dyeing. For soxhlet optimum time is 1 & ½ hour based on colour quality and solvent retained after 1 hour is constant. Extracted dye is of flavonoid family and sub group is flavones.

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