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Quality and Utilization of pigment extract from marigold petals

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ABSTRACT
Pigment was extracted from dry marigold flower petals of two types via Yellow and Orange in two seasons i.e. summer and winter by using solvents ie. Acetone, Acetone: Hexane (1:1) and Ethanol (100%). Dye was prepared from extract of best solvent treatments and used for dyeing cotton fabric with different mordants. The colour strength of the coloured samples was analyzed. Acetone: Hexane solvent treatment of orange flower possessed highest carotenoids content (468.68 mg/100g) and lowest in yellow flower. The response of Acetone + Hexane extract of orange and yellow petal in dyeing fabrics with different mordants showed appreciable difference in colour intensity on fabrics and indicated suitability of both flower colour as dye. Potassium dichromate gave a good colour (yellowish-brown) followed by Copper Sulphate (moss green) among the mordant. Sodium carbonate gave poor result with uneven tinge of colour all over fabrics. Thus considering the overall quality it can be concluded that Acetone + Hexane extracted, summer season orange flower possessed maximum carotenoids compared to orange colour, winter season flower. However flavonoids was noted to be highest in Acetone extracts of orange colour summer season flower petals followed by Acetone + Hexane extract of summer season yellow colour flower petal. Dyeing fabrics with Potassium dichromate gave a good colour among the mordants used.

Keywords: Marigold, extraction, Lutein, solvent, natural dye, chemical mordant, cotton fabrics

I. 1.INTRODUCTION

Marigold (Tageteserecta L.)belongs to the family Asteraceae, is a leading In India a huge amount of flowers are wasted every day. Marigold is the leading loose flower of India, covering an area of 66.13 thousand hectares with 603.18 thousand metric tonnes of production in 2015-16 [1].It produces natural dye from its flowers (petals) consisting mainly of carotenoid-lutein and flavonoid-patuletin, which could be isolated and exploited commercially [2]. Marigold extract (lutein) is a xanthophyll that has strong antioxidant ability [3, 4]. It has the prospect of utilization as food colouring agent and nutrient supplement (food additive) in baked food, beverages, sauces, dairy products etc. [6]

Presently there is an excessive use of synthetic dyes, causing serious health hazards and disturbing the eco-balance of nature [7]. The awareness about the harmful effects of usage of synthetic colours and chemicals obviously enhanced the demand for natural food in the international market abruptly [6, 8]. Japan and all European countries have banned trading of synthetic colour made products. In some developed countries encouragement for using natural colours in novel products like infant toys and crayons, organic textile printing, handmade paper etc., has been implemented and followed [5,6]. At present, large and small-scale industries have begun exploring the use of natural colorants as a possible means of producing an ecologically sound product which would also appeal to the “Green-minded” consumer [7].

Anthocyanin and betalain pigments which are water soluble are extracted from raw material with water and sometimes with aqueous methanol. For carotenoids extraction, organic solvents are good choice for initial extraction of pigment from plant material [6]. Some simple innovative technique of extraction of marigold pigment by organic solvents needs to be standardized through proper method of refinement[8].

There is also possibility to increase income by extraction of pigments through sustainable harvest of marigold flowers and by reusing the huge flower waste offered to the deities in temple. Solvent extract from marigold usually contains carotenoids and flavonoids which have to be concentrated and subjected to expensive methods of purification steps to make it suitable for human consumption [9, 6]. Recent information indicated the prospect of using marigold petal extract for less expensive natural dyeing purpose on textile fibre with suitable eco-friendly (metal) mordanting agents [2, 7, 9, 10,11]. This colour dye has no harmful effect on the environment. Extraction of natural dye from marigold temple waste can be regarded as a method of sustainable waste management which adheres to the concept of zero waste [12].

II. MATERIALS AND METHODS

The marigold flower of Tageteserecta L. (African marigold) consisting of two types of flowers i)Yellow and ii) Orange, grown in the Horticulture Research Station at Monduri, BCKV, was utilized for the present investigation. The flowers were brought to the laboratory of Department of Post Harvest Technology of Horticultural Crops for conducting the experiments.

Extraction, preparation and application of natural dyes and mordants

The pigment was extracted from the marigold flower of two type types i) Yellow ii) Orange in two seasons i.e. summer and winter by using several solvents. The pigment was utilized for dyeing fabrics. Since pigment extract of Acetone: hexane (1:1) of summer season flower was superior, it was used to assess as the dye on fabrics.

After extraction, the extract was filtered and the filtrate was collected, and the solvent was evaporated and recovered to dryness. Distilled water was added to this extract for dye preparation. This was used for dyeing purpose.

Scouring of fabrics

The fabrics were washed with 2 % non-ionic soap (Tide soap) at 50 ^{0}C for 20 min, maintaining the material-to-liquor ratio at 1:80. The scoured material was thoroughly washed with plenty of tap water and dried at room temperature. The scoured material was soaked in distilled water for 30 minutes prior to dying. All the fabrics were cut and sized into 5×6 cm and used for dyeing experiments [13].

Mordanting and dyeing procedures

The mordant (5%) was dissolved in distilled water and applied to each fabric samples. The dye extract of the marigold flower petals was used for the dying of the cotton fabrics. The mordants used are Ferrous Sulfate, Copper Sulfate, Potassium dichromate, Potash alum (Potassium aluminum sulphate) and Sodium Carbonate.

Mordanting of the fabrics

In this method mordanting was carried out prior to dying. The required amount of mordant was dissolved in 100 ml of distilled water in a beaker. Fabric sample was placed in the beaker and the dye bath was gradually brought to boiling point. The sample was stirred from time to time and the mordanting was continued for 1 hour at the temperature range of 80 -85 ^{0}C. The sample was squeezed lightly after cooling and dyed immediately after mordanting and kept for overnight in the dye solution [14]. Colour of the sample was measured.

Biochemical characters

Determination of carotenoids pigment

The carotenoids (β-carotene) were estimated by the methods as detailed in [15] and [16]. The carotenoid content of the sample was estimated at 436 nm using acetone, acetone - hexane and ethanol as blank for respective solvents. Carotenoid was calculated as:

$$\text{mg of } \beta\text{-carotene per 100gram} = \frac{\mu\text{g of carotene per ml as read from the curve} \times \text{Dilution} \times 100}{\text{Weight of sample} \times 1000}$$

Total flavonoids

Total flavonoid content was determined using aluminium chloride method [15]. An aliquot (1 ml) of the extract was taken in 10 ml of the volumetric flask containing 4 ml of distilled water, 0.3 ml portions of 5 % $NaNO_2$, and 0.3 ml portions of 10 % $AlCl_3.6H_2O$. The mixture was allowed to stand for 6 min at room temperature. Two milliliters of 1 M NaOH was added and the solution was diluted to 10 ml with distilled water. The mixture was mixed well by vortexing. The absorbance was measured immediately at 510 nm using a UV–vis spectrophotometer (spectrophotometer 166).

The standard curve was prepared using 0, 50, 100, 150, 200, 250 mg/L solutions of rutin, which is a common reference compound. Total flavonoids were expressed as mg rutin equivalents/100 g of dry weight.

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$$\text{mgRutin equivalence per gram} = \frac{\text{O.D x Factor x volume made up}}{\text{Aliquot taken x weight of sample}}$$

III. RESULTS AND DISCUSSION

Carotenoids pigments of marigold petals as influenced by different treatments, flower type and season is depicted in Table 1. The treatments (solvent), type of flower and the interaction between treatments and type were all significant at 5 % level (Table 1a). Mean carotenoids content of Acetone: Hexane (1:1) treatment was maximum (360.34 mg/100g) followed by Acetone (292.12 mg/100g) and Ethanol (244.51 mg/100g) respectively. Irrespective of treatments, mean carotenoids content of orange flower (396.49 mg/100g) was significantly higher than yellow flower (201.49 mg/100g). Interaction of treatment and flower type revealed that Acetone: Hexane solvent treatment of orange flower possesses highest carotenoids content (468.68 mg/100g) followed by Acetone with orange flower (399.62 mg/100g) and Ethanol with orange flower (321.18 mg/100g). Low carotenoids content was recorded in yellow flower Ethanol and Acetone solvent treatments.

The response of solvent treatments with seasons (Table 1b) showed significant difference with season and treatment x seasons interaction with regard to carotenoids content. It was observed that irrespective of treatments, carotenoids content of summer season flower was maximum (350.53 mg/100g) and significantly higher than winter seasons flower (247.45 mg/100g). Interaction effect of treatment and season showed that carotenoids content of Acetone: Hexane treated summer flower was maximum (389.60 mg/100g) and significantly high followed by Acetone extracted summer flower (339.95 mg/100g), Acetone: Hexane extracted winter flower (331.07 mg/100g), Ethanol extracted summer flower (322.05 mg/100g) and so on in that decreasing order.

Seasons vs flower type (Table 1c) showed significant effect of seasons, flower type and also an interaction between season x type. Orange flower of summer seasons (orange x summer) possessed highest carotenoids content (483.46 mg/100g) followed by orange flower of winter season (orange x winter) (309.52 mg/100g) and yellow flower of summer seasons (yellow x summer) (217.60 mg/100g).

Combined effect of treatment, flower type and season (Table 1d) exhibited that, Acetone: Hexane extracted summer season orange flower possessed highest carotenoids content (511.30 mg/100g and lowest in Acetone: Hexane x Winter x Orange flower (426.05 mg/100g).

Table 1. Carotenoids (mg/100g) of petals extract as influenced by solvent treatments, type of flower and season of flowering of marigold

1a. Treatment (Tr) vs Flower type (Ty)

Total carotenoids content (mg/100g)			
Type / Treatment (Solvent)	**Orange**	**Yellow**	**Mean**
Acetone	399.62	184.62	292.12
Acetone+Hexane	468.68	252.00	360.34
Ethanol	321.18	167.85	244.51
Mean	396.49	201.49	

1b. Treatment (Tr) vs Season (S)

Season / Treatment (Solvent)	**Summer**	**Winter**	**Mean**
Acetone	339.95	244.28	292.12
Acetone+Hexane	389.60	331.07	360.34
Ethanol	322.05	166.98	244.51
Mean	350.53	247.45	

1c. Season (S) vs Flower type (Ty)

Type / Season	Orange	Yellow	Mean
Summer	483.46	217.60	350.53
Winter	309.52	185.38	247.45
Mean	396.49	201.49	

1d. Interaction of Treatment (Tr) x Type (Ty) x Season (S)

Type	Summer		Winter	
Treatment (Solvent)	**Orange**	**Yellow**	**Orange**	**Yellow**
Acetone	472.67	207.24	326.57	162.00
Acetone+Hexane	511.30	267.91	426.05	236.10
Ethanol	466.42	177.67	175.93	158.03

Stastistical measure	Treatment (Tr)	Type (Ty)	Season (S)	TrxTy	Tr x S	S xTy	Tr x Ty x S
S.Em ±	0.172	0.141	0.141	0.243	0.243	0.199	0.344
C.D. (5 %)	0.490	0.400	0.400	0.692	0.692	0.565	0.979

Total flavonoid content of marigold types as influenced by different solvent treatment in various seasons is shown in Table 2. Significant difference in flavonoid content for solvent treatments, flower type and treatment x flower type interaction was noted (Table 2a). Mean flavonoid content of Acetone treatment was observed to be maximum (33.92 mg RE/100g) and significantly higher than Ethanol treatment (30.12 mgRE/100g) followed by Acetone: Hexane (29.49 mg RE/100g). Irrespective of solvent treatment average flavonoid content of orange colour (type) marigold was significantly higher (35.37 mg RE/100g) than yellow type (26.98 mg RE/100g). Flavonoid content of acetone extracted orange type marigold was highest (38.87 mg RE/100g) followed by Ethanol extracted orange type (33.68 mg RE/100g) and Acetone: Hexane extracted orange type (33.57 mg RE/100g) flower. However, the effect Ethanol x orange type and Acetone: Hexane x orange type was at par and not significantly different.

The effect of treatments (solvents) with seasons indicated significant difference of seasons and treatments with season of marigold (treatment x season) on flavonoid content (Table 2b). Mean flavonoids content of summer season flower was significantly higher (33.64 mg RE/100g) than winter seasons flower (28.71 mg RE/100g). Flavonoid content of summer season flower extracted from Acetone (Acetone x summer season) was maximum (37.02 mg RE/100g) and significantly higher than Acetone: Hexane treated summer season (Acetone: Hexane x summer) is 32.16 mg RE/100g and Ethanol extracted summer seasons flower (Ethanol x summer) is 31.76 mg RE/100g) respectively.

The effect of season on flower type for flavonoid contents was significant and has been presented in Table 2c. Flavonoid content was recorded to be maximum in summer season orange flower (38.38 mg RE/100g) followed by winter season orange flower (32.37 mg RE/100g).

The combined effect of treatment, season and flower type for flavonoid content was significant at 5% level (Table 2d). Highest flavonoid content was recorded in acetone extracted summer season orange type marigold flower (42.17 mg RE/100g) followed by Acetone : Hexane treated summer season orange flower (37.06 mg RE/100g), Ethanol treated summer season orange flower (35.90 mg RE/100g), Acetone extracted, winter season orange flower (35.58 mg RE/100g) and so on in that decreasing order.

Table 2. Total flavonoids (mg RE/100g) of petals extract as influenced by solvent treatments type of flower and season of flowering of marigold

2a. Treatment (Tr) vs Flower type (Ty)

Total Flavonoids (mg RE/100g)			
Type / Treatment (Solvent)	**Orange**	**Yellow**	**Mean**
Acetone	38.87	28.96	33.92
Acetone+Hexane	33.57	25.40	29.49
Ethanol	33.68	26.55	30.12
Mean	35.37	26.98	

2b. Treatment (Tr) vs Season (S)

Season / Treatment (Solvent)	**Summer**	**Winter**	**Mean**
Acetone	37.02	30.82	33.92
Acetone+Hexane	32.16	26.83	29.49
Ethanol	31.76	28.47	30.12
Mean	33.64	28.71	

2c. Season (S) vs Flower type (Ty)

Type / Season	**Orange**	**Yellow**	**Mean**
Summer	38.38	28.91	33.64
Winter	32.37	25.05	28.71
Mean	35.37	26.98	

2d. Interaction of Treatment (Tr) x Type (Ty) x Season (S)

Type / Treatment (Solvent)	**Summer**		**Winter**	
	Orange	**Yellow**	**Orange**	**Yellow**
Acetone	42.17	31.86	35.58	26.07
Acetone+Hexane	37.06	27.25	30.08	23.58
Ethanol	35.90	27.62	31.45	25.49

Stastistical measure	**Treatment (Tr)**	**Type (Ty)**	**Season (S)**	**TrxTy**	**Tr x S**	**S xTy**	**Tr x Ty x S**
S.Em ±	0.130	0.106	0.106	0.183	0.183	0.150	0.259
C.D. (5 %)	0.369	0.301	0.301	0.521	0.521	0.426	0.737

Dyeing of cotton fabrics, Acetone + Hexane extracts pigments of summer season flower was chosen. Different shades of colour were obtained from the dyeusing different mordants like Potassium Dichromate, Potash Alum (Potassium Aluminium Sulphate), Copper Sulphate, Ferrous Sulphate and Sodium Carbonate on cotton fabrics. The colorimetric value of dyeing (with orange and yellow petal extracts) on cotton fabrics with different mordants exhibited different shade depending upon mordant which ranged from light to dark i.e. shades of beige (Sodium Carbonate), mustard yellow (Potash Alum), moss green (Copper Sulphate), black (Ferrous Sulphate) and yellowish-brown (Potassium Dichromate) in orange type marigold flower (Fig 2-12). Table 3 representsL* a* and b* values, it can be seen that mordants with higher values of L* show lighter shades, while lower L* value signify deeper shades for the dyed fabrics. Similarly, negative a* and negative b* represent green and blue colour respectively. Dyed fabric with orange colour petal extract as dye resulted in least value of L* in case of Ferrous Sulphate (47.67) followed by Potassium

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dichromate (61.59), Sodium Carbonate (62.46) and Copper Sulphate (63.03) used as mordant indicating depth of colour (dull and dark). Whereas, highest L* values were obtained in alum having good brightness (67.90).

Yellow type marigold gave comparatively high value of L* than orange colour. So the shades are lighter compared to orange colour flower extract. In case of yellow colour variety L* value is low in Ferrous Sulphate (52.42) and Copper Sulphate (67.79) followed by sodium carbonate (69.53) indicated depth of the colour and the highest value was recorded in Potassium dichromate (72.10) followed by Potash Alum (71.25) (Table 3).

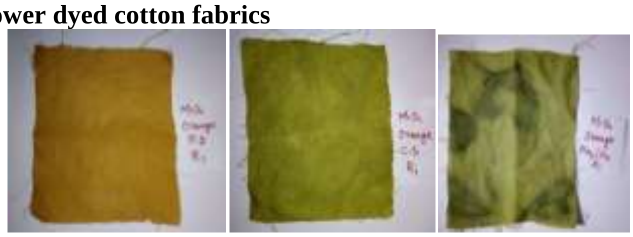
The response of mordant in dyeing showed an appreciabledifference in colour intensity on fabrics.In general orange colour petal gave deeper shade than yellow colour petal of marigoldand thus both marigold colours can be used as dye. Potassium dichromate gave a good colour (yellowish-brown) followed by Copper Sulphate (moss green) among the mordant. Sodium carbonate gave poor result with an uneven tinge of colour all over fabrics while Ferrous Sulphate gave blackish colour.The effect of mordants on dyeing fabrics exhibited different shades and intensity of colour which was found to be similar to the results [2, 10, 11] and that both marigold colour petals (orange and yellow) can be exploited as dye has also been supported by [12].Mordant are metal salts which have the ability to form coordination complexes with dye molecule. The strong coordination tendency enhances the interaction between the fiber and the dye, resulting in high dye uptake. Black deep colour of Ferrous Sulphate was due to strong coordination tendency of Fe, enhancing dye uptake, while all other metals showed similar coordination [17, 18].

Table 3. Colour measurement values of cotton fabrics dyed with marigold petal extract (orange and yellow) with different mordants.

Treatments	**Orange**				**Yellow**			
	L	**a**	**b**	**ΔE***	**L**	**a**	**b**	**ΔE***
Control	73.79	3.79	27.59	78.87	70.34	1.87	24.03	74.35
Potash alum	67.9	-1.39	35.76	76.75	71.25	-1.88	36	79.85
Copper sulphate	63.03	-3.8	27.11	68.72	67.79	-1.75	28.66	73.62
Ferrous sulphate	47.67	-0.92	8.55	48.44	52.42	-1.31	10.28	53.43
Pot. dichromate	61.59	5.14	26.11	67.1	72.1	-0.27	19.34	74.65
Na_2CO_3	62.46	-4.02	19.83	65.66	69.53	-2.38	27.7	74.88

Figure

Orange type marigold flower dyed cotton fabrics



Potassium dichromate

Copper sulphate

Sodium carbonate

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Yellow type marigold flower dyed cotton fabrics

Potassium dichromate	Copper sulphate	Sodium carbonate

IV. CONCLUSIONS

It can be concluded that the response of Acetone + Hexane extract of orange and yellow petal in dyeing fabrics with different mordants showed appreciable difference in colour intensity on fabrics and indicated suitability of both flower colour as dye. Potassium dichromate gave a good colour (yellowish-brown) followed by Copper Sulphate (moss green) among the mordant. Sodium carbonate gave poor result with uneven tinge of colour all over fabrics.Thus considering the overall quality it can be concluded that Acetone + Hexane extracted, summer season orange flower possessed maximum carotenoids compared to orange colour, winter season flower. However flavonoids was noted to be highest in Acetone extracts of orange colour summer season flower petals followed by Acetone + Hexane extract of summer season yellow colour flower petal. Dyeing fabrics with Potassium dichromate gave a good colour among the mordants used.

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REFERENCES

[1]. Anonymous (2016). Indian Horticulture Database (2015-16). National Horticulture Board, Ministry of Agriculture, Government of India, Gurgaon-122015.

[2]. Vankar, S. P., Shanker, R. and Wijayapala, S. (2009). Utilization of Temple waste flower- Tagetuserecta for Dyeing of Cotton, Wool and Silk on Industrial scale. Journal of Textile and apparel, Technology and management,**6** (1): 1-15.

[3]. Koutsos, E. A., Garcia Lopez, J. C. and Klasing, K. C. (2006). Carotenoids from dietary sources blunt systemic indices of the inflammatory response in growing chicks (Gallus gallusdomesticus). J. Nutr.,**136**:1027-1031.

[4]. Rajput, N., Naeem, M., Ali, S., Rui, Y. and Tian, W. (2012). Effect of Dietary Supplementation of Marigold Pigment on Immunity, Skin and Meat Color, and Growth Performance of Broiler Chickens. Brazil. J. Poult. Sci., **14**: 233-304.

[5]. Munhoz, M. V., longhini, R., Souza, R. P. J., Zequi, A. C. J., Mello, L. V. S. E., Lopes, C. G. and Mello, C. P. J. (2014). Extraction of flavonoids from Tagetespatula: process optimization and screening for biological activity. Rev. Bra. Farmacogn.,**24** (2014): 576-583.

[6]. Lakshmi, G. C. (2014). Food Colouring: The Natural Way. J. chem. Sci. Res.,**4** (2): 87-96.

[7]. Kannadasan, T., Gnanaprakasam, A., Harikrishnan, R., Nganyei, K. and Rajkumar, A. (2013). Extraction of Natural Dye (Xanthophyll) From Marigold Flower. J. Adv. Sci. Res., **4** (2): 48-50.

[8]. Pratheesh, V. B., Benny, N. and Sujata, C. H. (2009). Isolation, Stabilization and Characterization Of Xanthophyll From Marigold Flower- TageteErecta-L. Modern Applied Sci.,**3** (2): 19-28.

[9]. Sarkar, C.R., Das, L., Bhagawati, B. and Goswami, B. C. (2012). A comparative study of carotenoid extraction from algae in different solvent systems Asian J. Plant Sci. Res., **2** (4): 546-549.

[10]. Jha, K. C., Kumar, R., Venkat, K. S. and Rajeswari, V. D. (2015). Extraction of natural dye from marigold flower (Tageteserecta L.) and dyeing of fabric and yarns: A focus on colorimetric analysis and fastness properties. Der Pharmacia Lettre,**7**(1):185-195.

[11]. Jain, N. (2017). Extraction and application of Natural dye by utilizing temple floral waste Tageteserecta L. Int. J. Eng. Technol. Sci. Res.,**4**(3):144-150.

[12]. Saha, P. and Datta, S. (2016). Dyeing of Cotton Fibre using Marigold Flower as Dye. Industry Articles, **4** (2): 48-50.

[13]. Tiwari, H. C., Singh, P., Mishra, P. K. and Srivastava, P. (2010). Evaluation of Various Techniques for Extraction of Natural Colorants from Pomegranate rind- Ultrasound and Enzyme Assisted Extraction. Indian J. fibre textile Res.,**35** (9): 272-276.

[14]. Wang, J., Shen, X. and Chen, Y. (2012). Effect of pH, temperature and iron on the stability of anthocyanins from black-skinned peanuts (ArachishypogaeaL.). African J. Agric. Res., **8** (18): 2044-2047.

[15]. AOAC (2005). Official method of Analysis. 18th Edition, Association of Officiating Analytical Chemists, Washington.

[16]. Ranganna, S. (2007). Hand book of analysis and quality control for fruit and vegetable products (2^nd^edn.). Tata MacGraw – Hill Publishing Company Ltd., West Patel Nagar, New Delhi-8.

[17]. Zhishen, J., Mengcheng, T. and Jianming, W. (1999). The determination of flavonoid contents in mulberry and their scavenging effects on superoxide radicals. Food Chem., **64**:555–559.

[18]. Jothi, D. (2008). Extraction of natural dyes from African marigold flower (Tageteserecta L) for textile coloration. AUTEX Research Journal, **8** (2): 49-53.

[19]. Chavan, S. and Ghosh, E. (2015). Cotton and silk dyeing with Natural dye extracted from floral parts of African marigold (Tageteserecta). Int. J. Res. Advent Technol., National conference 13-14 Feb 2015.