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Influence of drying methods on retention of carotenoids and their antioxidant activity in marigold flowers

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ABSTRACT

The present investigation was carried out to find the effect of drying methods on retention of carotenoids and their antioxidant activity in marigold flowers. The data revealed that vacuum drying method had retained highest total carotenoids (2765.76 mg/100g) and lutein (295.15µg/g) in dried petals of cv. Pusa Narangi Gainda (PNG), whereas sun drying exhibited lowest total carotenoids (79.92 mg/100g) and lutein (8.87µg/g) in dried petals of cv. Pusa Basanti Gainda (PBG). Highest β-carotene (16.45 µg/g) was retained in petals dried by vacuum drying method in cv. Pusa Arpita. Vacuum drying method also had retained highest total phenolic content (93.00 mg GAE/g) and total flavonoid content (47.74 mg RE/g) in dried petals of cv. PBG. Vacuum drying method had retained highest antioxidant activity in terms of FRAP values (838.83 µmol $FeSO_4$/g DW) in dried petals of cv. PNG and DPPH values (76.63 %) in dried petals of cv. Pusa Arpita, whereas sun drying resulted in lowest antioxidant activity (388.55 µmol $FeSO_4$/g DW and 53.41%) in dried petals of cv. PBG.

Key words: *Tagetes* sp., vacuum drying methods, HPLC, lutein, antioxidant.

INTRODUCTION

Marigold (*Tagetes* sp.) is an important flower crop grown worldwide. It is a native of Mexico and belongs to the family Asteraceae. It is cultivated commercially in most of the parts of India and become most popular flower to be used for decoration and in landscaping. Marigold fresh or dried flowers have also been used as food colourant and ingredient in cooking and also used in animal feeds. Besides, these are used in preparation of tea, spice and medicines and have therapeutic activities. Carotenoid pigments are found in all parts of marigold plant such as leaf, roots, flowers, seeds, etc., however, marigold petals are considered as an important source of carotenoid pigments (Akshaya *et al.,* 5), especially the xanthophylls (lutein, zeaxanthin) and the yellow carotenoids (β-carotene), (Ahluwalia *et al.,* 2) and polyphenols (Siriamornpun *et al.,* 15). Lutein is one of the potential natural plant based antioxidant which reduces the risk of chronic diseases such as cancer and enhance immune function, reduces auto oxidation of cellular lipids and age related macular degeneration, provides protection against oxidant induced cell damage, *etc,.* Many biochemical changes happen in between harvesting and consumption of the flowers which affect the nutraceutical value in marigold. Dehydration is an important process which extends the storage life of flowers by retaining their physical and biochemical properties, leads to easy extraction of pigments and also ensures the inhibition of enzymatic degradation and microbial growth. Sun drying is the most commonly used method for dehydrating marigold flowers and is used in production of powder for use in food and feed industries. However, this method of drying has disadvantages like contamination especially in the rainy season, longer drying times, inability to handle large quantities to achieve quality standards. The commonly used drying processes could lead to the degradation of bioactive compounds and cause damage to the physical qualities of the marigold powder. There are other drying methods such as hot air oven drying; Microwave and vacuum drying, *etc*. which are used for drying marigold flowers in powder production for further use in the food and feed industries. These methods retain product quality in terms of shelf -life, recover more carotenoids and other bioactive compounds and reduce transport and storage costs. However, there are very few published reports on the effect of efficient drying techniques on retention of carotenoids and antioxidant properties of marigold flowers. Hence, the present investigation was carried out to study the influence of different dehydration techniques for maximum recovery of carotenoids and other bioactive compounds and higher retention of antioxidant properties of pigments in marigold flowers.

MATERIALS AND METHODS

The plant material utilized for conducting the experiments consisted of two cultivars of African

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marigold (*Tagetes erecta* L.) namely Pusa Narangi Gainda (PNG) and Pusa Basanti Gainda (PBG) and one cultivar of French Marigold (*Tagetes patula* L) namely Pusa Arpita. The features and photograph of cultivars used in the present investigation are given in Table 1 and Fig. 1, respectively. These were grown and maintained at research farm of the Division of Floriculture and Landscaping, ICAR-Indian Agricultural Research Institute, New Delhi. Fresh marigold flowers were harvested at full bloom stage for drying of petals using different drying methods. In sun drying method, petals were uniformly spread in single layer on aluminium trays and kept under open sunlight until the flower petals were fully dried and have constant weight. The position of trays was also interchanged according to sunlight or shadows. In hot air drying, temperature was maintained at 60°C and air velocity of 0.12-.16 m/sec and petals were dried till constant weight was attained. In microwave drying, marigold flower petals were spread uniformly in the microwave oven for duration of 90 seconds. In vacuum drying, petals were spread uniformly in the trays of vacuum oven (LVO-2030, Daihan Labtech Co. Ltd.) at pressure of 0.08k Pascal at 45 °C till the constant weight was obtained. The dried petals were further used for extract preparation (petroleum ether extract for carotenoids, ethanolic extract for antioxidant, and lutein extract for lutein and β-carotene estimation). The total carotenoids were extracted and estimated using method given by Ranganna (12) with minor modifications. Sample preparation for Lutein and β-carotene was done using a modification of procedure described by Barba *et al.* (6).

Analysis of lutein and β-carotene was carried out using High Performance Liquid Chromatography (HPLC) where peaks were detected at respective l_{max} of 444 and 453 nm for lutein and β-carotene. The phenolic compounds in dried petals of marigold were extracted using a modification of the procedure described by Uzelac *et al*. (16).Total phenolic content (TPC) was estimated according to procedure given by Singleton and Rossi (14). The colorimetric method described by Abu Bakar *et al.* (1) was used to determine total flavonoid content (TFC). The sample was extracted using the procedure as in case of phenolic compounds The antioxidant activity of the extracts was determined using FRAP (Ferric Reducing Antioxidant Potential) method as described by Benzie and Strain (7) and DPPH assay described by Braca *et al.* (8). The data was statistically analyzed in completely randomized design (CRD) using Statistical analysis system (SAS) software. All determinations were done at least in triplicate and all were averaged. The confidence limits used in this study were based on 95% confidence ($P<0.05$).

RESULTS AND DISCUSSION

The identification of the carotenoid components-lutein and β-carotene (as chromatographic peaks)

Table 1. Salient features of marigold cultivars.

Cultivar	Species	Flower type	Flower size	Flower colour	Flowering time	Source
Pusa Arpita	*Tagetes patula* L.	Semi double	Medium	Orange	Mid Dec.,- Mid Feb.,	ICAR-IARI
Pusa Narangi Gainda (PNG)	*Tagetes erecta* L.	Semi double	Medium	Orange	Mid Feb., Mid April	ICAR-IARI
Pusa Basanti Ganida (PBG)	*Tagetes erecta* L.	Semi double	Medium	Yellow	Mid Feb.,- Mid March	ICAR-IARI

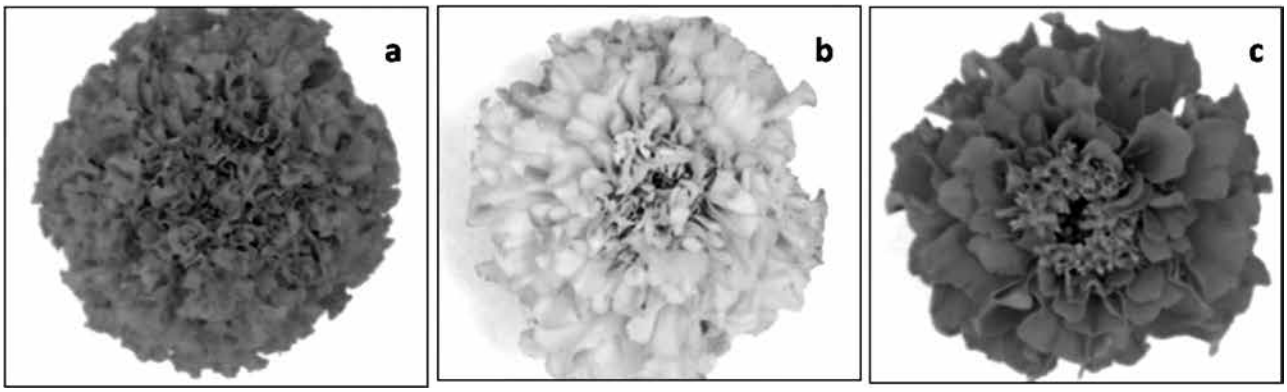

Fig. 1. Marigold cultivars used for present study-a) PNG; b) PBG; c) Pusa Arpita.

was carried out by comparing their retention times with those obtained with a standard mixture of β-carotene and lutein. HPLC generated chromatograms of lutein and β-carotene of dried marigold flowers of cultivars Pusa Narangi Gainda (PNG), Pusa Basanti Gainda (PBG) and Pusa Arpita, respectively by different drying methods are shown in Fig. 2, 3 and 4. Chromatographic peaks were observed for lutein and β-carotene content of dried flowers of African marigold cv. PNG under sun drying at 6.623 and 14.128 minutes; microwave drying at 6.000 and 14.196 minutes; hot air oven drying at 6.199 and 14.154 minutes and vacuum drying at 7.065 and 15.455 minutes, respectively (Fig. 2). Similarly, dried flowers of African marigold cv. PBG exhibited chromatographic peaks for lutein and β-carotene under sun drying at 6.637 and 14.079 minutes; microwave drying at 6.641 and 14.104 minutes; hot air oven drying at 6.675 and 14.218 minutes and vacuum drying at 6.621and 14.086 minutes, respectively (Fig. 3). Chromatographic peaks were observed for lutein and β-carotene of dried flowers of French marigold cv. Pusa Arpita under sun drying at 6.937 and 15.001 minutes; microwave drying at 6.996 and 15.224 minutes; hot air oven drying at 7.057 and 15.339 minutes and vacuum drying at 6.812 and 14.668 minutes, respectively (Fig. 4). Lutein and β-carotene of vacuum dried flowers and extract of marigold was also studied using HPLC in our earlier study (Akshaya *et al*. 3; Akshaya *et al*. 4).

Drying methods namely hot air oven, microwave oven, vacuum drying exhibited high recovery of total carotenoids, lutein and β-carotene as compared to control i.e. sun drying. The data presented in the Table 2 depicts that vacuum drying method

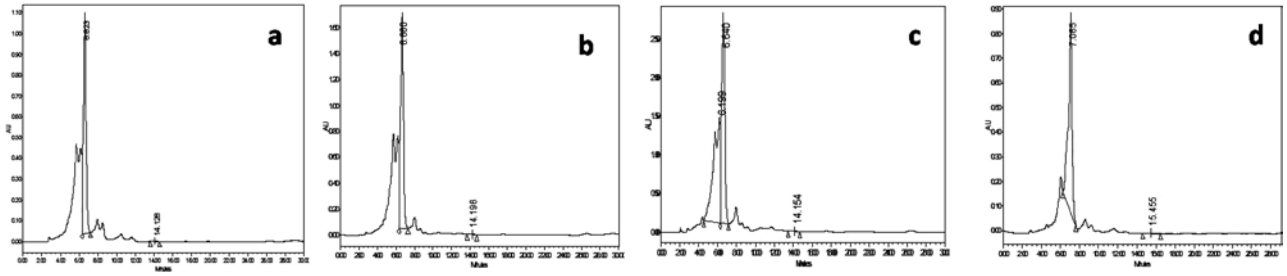

Fig. 2. HPLC chromatograms of lutein and β-carotene of cv. PNG flowers dried by different drying methods- a) Sun drying; b) Microwave drying; c) Hot air drying; d) Vacuum drying.

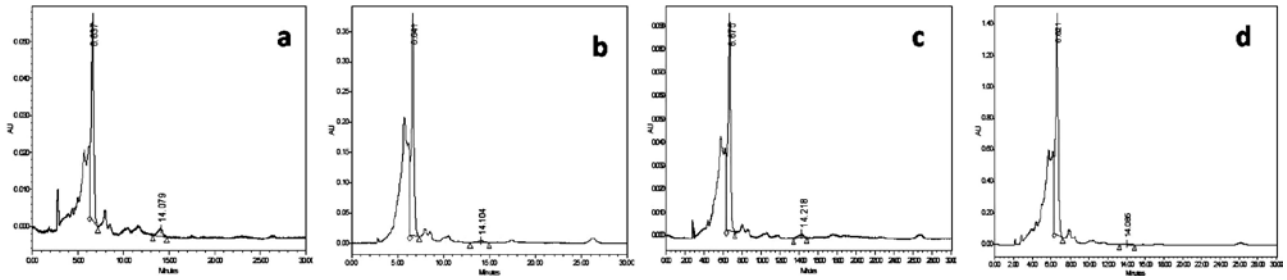

Fig. 3. HPLC chromatograms of lutein and β- carotene of cv. PBG flowers dried by different drying methods - a) Sun drying; b) Microwave drying; c) Hot air drying; d) Vacuum drying.

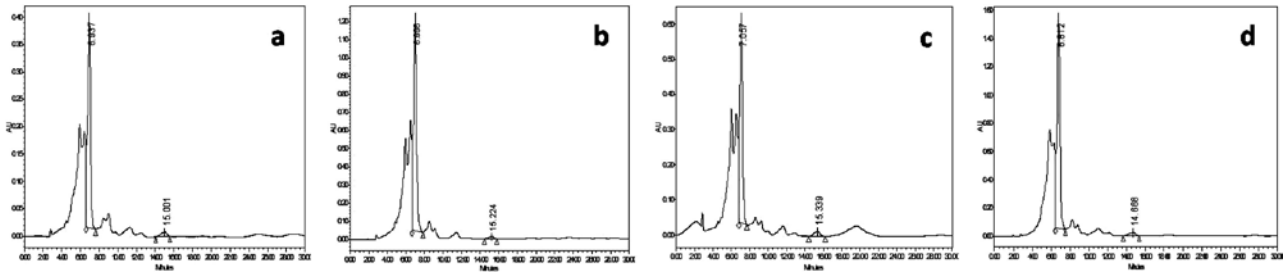

Fig. 4. HPLC chromatograms of lutein and β- carotene of cv. Pusa Arpita flowers dried by different drying methods - a) Sun drying; b) Microwave drying; c) Hot air drying; d) Vacuum drying.

Table 2. Effect of drying methods on total carotenoids, lutein and β-carotene in marigold flowers.

Drying Method	Cultivar	Total carotenoids (mg/100g)	Lutein (µg/g)	β-carotene (µg/g)
Sun drying	Pusa Arpita	593.40	65.29	6.18
	PNG	1738.28	170.59	1.68
	PBG	79.92	8.87	1.29
Microwave drying	Pusa Arpita	799.16	209.46	10.86
	PNG	2518.08	267.71	3.75
	PBG	97.40	54.45	3.51
Hot air oven drying	Pusa Arpita	659.19	97.05	10.21
	PNG	1968.54	186.68	6.62
	PBG	111.78	13.82	1.05
Vacuum drying	Pusa Arpita	1108.76	252.51	16.45
	PNG	2765.76	295.15	10.80
	PBG	144.90	79.21	4.65
CD (p≤0.05)	Drying Method (A)	33.30	19.21	0.98
	Cultivar (B)	33.29	16.64	0.85
	A×B	57.67	33.28	1.69

had retained highest total carotenoids and lutein (2765.76 mg/100g and 295.15µg/g) followed by microwave drying method (2518.08 mg/100g and 267.71 µg/g) in dried petals of cv. PNG, whereas sun drying exhibited lowest total carotenoids (79.92 mg/100g) in dried petals of cv. Pusa Arpita and lutein (8.87µg/g) in cv. PBG. Highest β-carotene (17.00 µg/g) was also retained in petals dried by vacuum drying method in cv. Pusa Arpita which is at par with petals of cv. PNG (16.44 µg/g) followed by micro wave drying method (10.86 µg/g) in dried petals of cv. Pusa Arpita. However, the lowest β-carotene was found in hot air dried petals of cv. PBG (1.05 µg/g) followed by sun drying of petals of cv. PBG (1.29 µg/g) which is at par with var. PNG(1.68 µg/g). In vacuum drying methods, petals of African marigold cv. PNG had retained highest total carotenoids (2765.76 mg/100g) and lutein (295.15 µg/g) followed by French marigold cv. Pusa Arpita (1108.76 mg/100g and 252.51 µg/g), while lowest was observed in cv. PBG (144.90 mg/100g and 79.21 µg/g) on dry weight basis. After Vacuum drying, petals dried under microwave drying methods exhibited total carotenoids of 2518.08 mg/100g and lutein of 267.71 µg/g in cv. PNG followed by cv. Pusa Arpita (799.16 mg/100g and 209.46), whereas, lowest in cv. PBG (97.40 mg/100g and 54.45 µg/g). In sun drying, petals of cv. PNG exhibited highest total carotenoids (1738.28 mg/100g) and lutein (170.59 µg/g) followed by cv. Pusa Arpita (593.40 mg/100g and 65.29 µg/g) and it was lowest in petals of cv. PBG (79.92 mg/100g and 8.87 µg/g). β-carotene of vacuum dried marigold flowers was found highest in cv. Pusa Arpita (16.45 µg/g) followed by cv. PNG (10.80 µg/g) and lowest was observed in cv. PBG (4.61 µg/g) on dry weight basis. In microwave drying, flowers of cv. Pusa Arpita exhibited highest β-carotene of 10.86 µg/g following by cv. PBG (3.75 µg/g) which is statistically at par with cv. PNG (3.51 µg/g) on dry weight basis. However, in sun drying, petals of cv. Pusa Arpita exhibited β-carotene (6.18 µg/g) followed by cv. PNG (1.68 µg/g) which is statistically at par with cv. PBG (1.29 µg/g). As lutein is not synthesized by humans and thus must be obtained by the ingestion of fruits leafy green vegetables, edible flowers and other food, etc. containing it. Lutein is a major carotenoid in marigold flower and remarkably, its amount was more than 5-fold in dried samples compared with fresh petals (Siriamornpun *et al.,* 15). Among various drying methods studied in marigold, vacuum drying was found to be best method in high retention of carotenoids, lutein and β-carotene followed by methods like microwave drying and hot air oven drying. Our findings are also in confirmation with the results of Ahluwalia *et al*. (2) as reported that vacuum drying method is the best method that yielded maximum preservation of carotenoids in marigold flowers. In present study, hot air oven dried petals of marigold also retained more carotenoids, lutein and β-carotene. Saha (13) also reported the hot air oven method was better than sun drying method for more retention of total carotenoids and β-carotene.

Table 3. Effect of drying methods on total phenolic content, total flavonoid content and antioxidant activity in marigold flowers.

Drying Method	Cultivar	Total phenolic content (mg GAE/g)	Total flavonoid content (mg RE/g)	FRAP (µmol $FeSO_4$/g)	DPPH (%)
Sun drying	Pusa Arpita	52.51	22.71	486.89	55.40
	PNG	63.00	32.87	424.28	64.09
	PBG	60.58	21.96	388.55	53.41
Micro wave drying	Pusa Arpita	66.35	24.17	576.93	69.00
	PNG	78.33	43.97	705.14	75.58
	PBG	88.08	33.23	530.73	66.23
Hot air oven drying	Pusa Arpita	60.73	21.11	515.43	72.71
	PNG	70.83	35.58	605.29	68.76
	PBG	85.42	25.89	493.89	59.32
Vacuum drying	Pusa Arpita	84.24	47.74	655.75	76.63
	PNG	83.89	41.95	838.83	71.99
	PBG	93.00	46.61	716.05	73.15
CD ($p \leq 0.05$)	Drying Method (A)	2.40	2.65	43.85	2.45
	Cultivar (B)	2.29	2.07	37.97	2.11
	A×B	4.58	4.14	75.94	4.24

The data presented in the Table 3 depicts that vacuum drying method had retained highest TPC (93.00 mg GAE/g) followed by microwave drying method (88.08 mg GAE/g) in dried petals of cv. PBG, whereas sun drying exhibited lowest TPC (52.51 mg GAE/g) in dried petals of cv. Pusa Arpita. Highest TFC (47.74 mg RE/g) was also retained in vacuum dried petals of cv. Pusa Arpita followed by micro wave dried petals of cv. PNG (43.97 mg RE/g). However, lowest TFC was found in hot air dried petals of cv. Pusa Arpita (21.11 mg RE/g). followed by sun drying of petals of cv. PBG (21.96 mg RE/g) which is at par with cv. Pusa Arpita (22.71 mg RE/g). In vacuum drying methods, petals of African marigold cv. PBG had retained highest TPC (93.00 mg GAE/g) followed by cv. Pusa Arpita (84.24 mg GAE/g) which is statistically at par with French marigold cv. PNG (83.89 mg GAE/g) on dry weight basis. Highest TFC was retained in vacuum dried flowers of French marigold cv. Pusa Arpita (47.74 mg RE/g) which is at par with cv. PBG (46.61 mg RE/g), however, cv. PNG exhibited lowest TFC (41.95 mg RE/g). After vacuum drying, petals dried under microwave drying methods exhibited TPC (88.08 mg GAE/g) in cv. PBG followed by cv. PNG (78.33mg GAE/g), while lowest TPC (66.35 mg GAE/g) was found in cv. Pusa Arpita. In microwave drying, highest TFC was retained in dried petals of cv. PNG (43.97 mg RE/g), however, cv. Pusa Arpita exhibited lowest TFC (24.17 mg RE/g). In sun drying, petals of cv. PNG exhibited high TPC (63.00 mg GAE/g) followed by cv. PBG (60.58mgGAE/g), while lowest TPC (52.51 mg GAE/g) was found in cv. Pusa Arpita. Highest TFC was retained in dried petals of cv. PNG (32.87mg RE/g), however, cv. PBG exhibited lowest TFC (21.96 mg RE/g). It was concluded from the present results that drying methods significantly affect the bioactive compounds in the marigold flowers and vacuum dried petals of cultivars Pusa Arpita, PNG and PBG retained highest total phenolic and flavonoid content followed by microwave dried petals and hot air oven dried petals. However, lowest total phenolic and flavonoid content was retained in sun dried petals which was in confirmation with the findings of Ramamoorthy and Bono (11) who reported highest total phenolic content in vacuum dried samples than dried by other methods in extracts of *Morinda citrifolia* fruit. Khattak (10) also reported lowest phenolic and flavonoid content in sun dried sample of marigold flowers. Losses in TPC and TFC by thermal processing have been reported in many studies, mostly for vegetables, therefore suitable food processing can recover the properties of naturally occurring antioxidants and induce the formation of new compounds with antioxidant activities, so that the overall antioxidant activity increases or remains unchanged.

The FRAP and DPPH values of the marigold flowers dried under different drying methods are shown in Table 3. The data depicted that vacuum

drying method had retained highest antioxidant activity in terms of FRAP (838.83 µmol $FeSO_4$/g DW) in dried petals of cv. PNG and DPPH (76.63 %) values in dried petals of cv. Pusa Arpita followed by microwave drying method (FRAP: 705.14 µmol $FeSO_4$/g DW and DPPH: 75.58%) in dried petals of cv. PNG, whereas sun drying exhibited lowest antioxidant activity (388.55 µmol $FeSO_4$/g DW and 53.41%) in dried petals of cv. PBG. In vacuum drying methods, petals of cv. PNG had retained highest antioxidant activity in terms of FRAP (838.83 µmol $FeSO_4$/g DW) followed by cv. PBG (716.05 µmol $FeSO_4$/g DW) on dry weight basis. Whereas, petals of cv. Pusa Arpita had retained highest antioxidant activity in terms of FRAP (76.63%) followed by cv. PBG (73.15%) on dry weight basis. In sun drying, petals of African marigold cv. Pusa Arpita had also retained highest antioxidant activity in terms of FRAP (486.89µmol $FeSO_4$/g DW) and DPPH (64.09 %) followed by cv. PNG (FRAP: 424.28 µmol $FeSO_4$/g DW and DPPH: 55.40%). In our studies, highest retention of antioxidant activities was reported in vacuum drying as compared to microwave drying and hot air oven. Similar results were reported by Duy *et al*. (9) by observing significant losses in the antioxidant capacity as measured by DPPH scavenging activity of oven dried samples of different vegetables. Lowest antioxidant activities in sun dried sample of marigold flowers was also reported by Khattak (10). The enhanced antioxidant activity of the thermally processed marigold could be explained by the increased amount of β-carotene and lutein, which are major phytochemicals in marigold, and other bound phytochemicals released from the matrix with thermal processing. Siriamornpun *et al.* (15) also reported that hot air drying had the lowest antioxidant activities, phenols, flavonoids compared to FIR-hot air and Freeze drying of marigold flowers. The interaction between drying methods and cultivars is significant at 5% level of significance. These results demonstrate that vacuum drying may be considered as a suitable drying method for marigold flowers with respect to preserving carotenoid pigment and its antioxidant activity and will provide useful information for commercial production of marigold flower powder.

DECLARATION

The authors declare no conflict of interest.

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