ISSN 2349-7203
IJPPR
INTERNATIONAL JOURNAL OF PHARMACY & PHARMACEUTICAL RESEARCH
An official Publication of Human Journals
HUMAN

Human Journals
Research Article
July 2016 Vol.:6, Issue:4

Extraction of Essential Oil from Flowers of *Mammea suriga* (Buch.-Ham. Ex Roxb.) KOSTERM. GC-MS Analysis and Incorporation in Cosmetic Product

ISSN 2349-7203
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Submission: 7 July 2016
Accepted: 12 July 2016
Published: 25 July 2016

HUMAN JOURNALS

www.ijppr.humanjournals.com

Keywords: Clusiaceae, cold maceration, n-hexane extract, Headspace, olfactory evaluation

ABSTRACT

Mammea suriga (Buch, -Ham. Ex Roxb) Kosterm. belongs to family Clusiaceae is endemic to Western Ghats of India and is popular for its fragrant flowers. Extraction of essential oil from flowers was standardized by cold maceration method and soxhlet extraction with n-hexane. Essential oil extracted from flowers was also subjected to GC-MS and headspace analysis. The major components identified by cold maceration method were Trans-Alpha-Bergamotene (32.32%), Kaurene (10.60%) and Squalene (07.74%). Essential oil extracted in n-hexane showed following components, Trans-Alpha-Bergamotene (30.73%), Kaurene (13.09%), Alpha Farnesene (08.28%) and Squalene (07.45%). Trans-Alpha-Bergamotene component was found in higher percentage by cold maceration and in soxhlet method. The major components identified in flowers by headspace technology were Trans-Alpha-Bergamotene (55.91%), Alpha farnesene (10.91%), Trans-Beta-Farnesene (5.21%), Pentadecane (4.82%) and Bisabolene (Cis-gamma) (2.94%). The essential oils extracted from *M. suriga* were also subjected to olfactory evaluation and was incorporated in cosmetic products (Hair oil, Massage oil and Body cream).

INTRODUCTION

Mammea suriga (Buch, -Ham. Ex Roxb) Kosterm. is one of the important aromatic tree species of the Western Ghats, belonging to family Clusiaceae, commonly known as Nagasampige in Sanskrit and Suragi in Kannada. [1] It is an endemic tree species confined to Western Ghats of India. [2] The plant is dioecious. It is one of important non-timber forest product of the central Western Ghats. It bears staminate flowers once in a year during the months of March and April and the flower buds are globous, white or pinkish in colour (Fig.1). It is cultivated for its sweet scented flower and handsome foliage. Fresh flowers are used for worshiping in temples and for personal adornment. Part of the plant which is of economic importance is dried flower, which persists its fragrance for a long period of time and thus is used in perfume industries. [3]

No standard extraction protocol of essential oil of *M. suriga* has been reported till date. The aim of this study was to standardized protocol of extraction of essential oil of *M. suriga*.

Figure. 1: Flowers of *Mammea suriga*

MATERIALS AND METHODS

Plant material –

The fresh flowers of *Mammea suriga* were collected from College of Forestry, Dr. B. S. Konkan Krishi Vidyapeeth, Dapoli, Ratnagiri, Maharashtra, India.

Extraction of essential oil -

Various methods of extraction of essential oil from *M. suriga* were tried viz., hydro-distillation, Soxhlet extraction with ethanol and n-hexane and cold maceration method using mineral oil and n-hexane. Flowers (300gms in 500ml n-hexane) were used for maceration. The setups were left overnight without disturbance. Next day flowers parts were removed from the solvent. Essential oil was recovered by evaporating the solvent.

Essential oil recovery -

Volatile oil was recovered by completely evaporating n-hexane from the solvent obtained above using rotary vacuum evaporator.

Evaluation of essential oils by headspace analysis -

Fresh flower samples were collected and placed into plastic bag and transported to the laboratory. 3 ± 0.2 gm of flowers was accurately weighed and placed into glass sampling bottles (11 cm long by 4 cm in diameter) without damaging the tissue. The bottles were immediately sealed with headspace sampling cap containing Teflon-coated septa and aluminum seals.

GC-MS -

Analysis was carried out on Agilent 5975C inert mass selective detector (MSD) with triple axis detector, directly coupled to a HP 7890A gas chromatograph. A 30 m × 0.25 mm × 0.25 micron fused silica HP-5ms column was employed. The column oven temperature was programmed from 80^{o}C (after 3min) to 250^{o} C at 2.5 ^{o} C/ min. The injector was maintained at 280 ^{o} C respectively. Electron ionization mass spectra were acquired over the mass range 40 - 550 Da.

Application of essential oils in Cosmetic Products -

The essential oils were further evaluated for its application in cosmetic products by incorporating in three different products specifically hair oil, massage oil and cream base.

Hair oil and Body massage oil -

The various ingredients used in the formulation of hair oil and body massage oil are presented in (Table-1). Accurately weighed ingredients were mixed together.

Table 1: Hair oil and Massage oil formulation.

Ingredients	Hair oil	Massage oil
Olive oil	10%	-
Mineral oil	79.2%	78%
Sesame oil	10%	14.2%
PP	0.1%	0.1%
BHT	0.2%	0.2%
Silicon oil	-	7%
Essential oil	0.5%	0.5%

Cream formulation -

Oil in water (O/W) emulsion-based cream (semisolid formulation) was formulated. The emulsifier (stearic acid) and other oil soluble components (Cetyl alcohol, coconut oil and coco butter, propylparaben) were dissolved in the oil phase and heated to 75° C. The water soluble components (Methylparaben, Glycerine etc) were dissolved in the aqueous phase and heated to 75° C. After heating, the oil phase was added in portions to the aqueous phase with continuous stirring until cooling of cream took place. After cream was prepared, *M. suriga* flower oil was incorporated in it by mixing using stirrer. The formula for the cream is given in (Table 2).

Same procedure was used for incorporation of petal oil.

Table 2: Body cream formulation

Ingredients	Percentage
Stearic acid	7%
Cetyl alcohol	2%
IPM	2%
Coco butter	1%
Coconut oil	2%
Dimethicone	1%
PP	0.2%
BHT	0.2%
Water	77.55%
TEA	1%
Glycerine	5%
MP	0.35%
EDTA	0.2%
Essential oil	0.5%

RESULTS AND DISCUSSION

Extraction of essential oil

The flowers were collected in morning hours. Hydro-distillation, soxhlet extraction and cold maceration methods were used to extract essential oil from flowers. The hydrodistillation method was found to be unsuitable, as there was no essential oil extracted from the flowers.

In soxhlet extraction method, ethanol and n-hexane were used for extraction of essential oil. n-hexane extract was found to be better than ethanol extract. In cold maceration, mineral oil and n-hexane were used. In n-hexane cold maceration, oil extracted had similar odour profile to that of natural flower. Mineral oil had hindered the natural smell.

Hence most suitable method was cold maceration and soxhlet extraction with n-hexane.

GC-MS Analysis

Essential oil was extracted using n-hexane by cold maceration and soxhlet method. The major components identified by cold maceration method were Trans-Alpha-Bergamotene (32.32%), Kaurene (10.60%) and Squalene (07.74%). Essential oil was extracted in n-hexane by soxhlet method showed following components, Trans-Alpha-Bergamotene (30.73%), Kaurene (13.09%), Alpha Farnesene (08.28%) and Squalene (07.45%). Longicyclene (01.13%), Palmitic acid (00.80%), Palmitate ethyle (01.66%) and Linoleate ethyle (00.64%) components were identified in cold maceration method but were missing in soxhlet extraction. Trans-Alpha-Bergamotene component was found in higher percentage by cold maceration and in soxhlet method. Phenyl ethyl alcohol (00.45%) and Alpha Santalene (00.72%) were found lesser in amount by soxhlet method however they were totally absent in cold maceration method (Table 3).

Table 3: GC-MS Analysis of *M. suriga* flower oil

Component Name	Percentage (%) Cold Maceration	Percentage (%) Soxhlet Extraction
Gamma Octalactone	00.98	O1.14
Alpha Copaene	01.08	01.13
P. E. Alcohol	-	00.45
(+) Longicyclene	01.13	-
Alpha Muurolene	00.80	00.51
Alpha-Santalene	-	00.72
Trans-Beta-Farnesene	02.95	02.33
Alpha Farnesene	08.67	08.28
Trans-Alpha-Bergamotene	32.32	30.73
Beta Bisabolene	01.04	01.29
Beta-Sesquiphellandrene	01.14	01.52
Palmitic Acid	00.80	-
Palmitate Ethyle	01.66	-
Kaurene	10.60	13.09
Linoleate Ethyle	00.64	-
Squalene	07.74	07.45

Headspace analysis

In present study, *M. suriga* flowers were subjected to headspace analysis. The major constituents identified in flower were Trans-Alpha-Bergamotene (55.91%), Alpha farnesene (10.91%), Trans-Beta-Farnesene (5.21%), Pentadecane (4.82%) and Bisabolene (Cis-gamma) (2.94%). Alpha Longipinene (0.22%), Beta Elemene (0.08%), Beta Caryophyllene (0.51%), Zingiberene (0.41%), Gamma Muurolene (0.62%) and Valencene (0.32%) components were found in minor amount. The major components identified in headspace analysis were Trans-Alpha-Bergamotene, Alpha Farnesene and Kaurene were also detected in essential oil extracted by maceration in hexane (Table 4).

Table 4: *M. suriga* Flower Headspace At 100DEG

Component Name	**Percentage (%) Flower Oil**
Alpha Longipinene	0.22
Alpha-Copaene	1.25
Beta Elemene	0.08
Alpha-Santalene	1.41
Beta Caryophyllene	0.51
Zingiberene	0.41
Trans-Beta-Farnesene	5.21
Alpha Muurolene	0.92
Gamma Muurolene	0.62
Isoledene	1.33
Valencene	0.32
Trans-Alpha-Bergamotene	55.91
Alpha Farnesene	10.91
Beta-Sesquiphellandrene	1.98
Bisabolene <Cis-Gamma>	2.94
Kaurene	3.86
Pentadecane	4.82

Odour Evaluation

The essential oils extracted from *M. suriga* flowers were also subjected to olfactory evaluation. The olfactory analysis was done for flower oil. The evaluated notes are described in (Table 5).

Table 5: Odour profile of *M. suriga* flower oil

Odour profile of *M. suriga* flower in cold maceration extract	**Odour profile of *M. suriga* flower in soxhlet method extract**
White floral	Light floral
Spicy	Oily
Sharp	Stale
Pungent	Sweet
Green	Spicy
Sweet	-
Stale	-

On comparison of the notes it was observed that the cold maceration extract of flower oil gave a white floral, spicy, sharp, pungent, green, and sweet with stale notes in the background, which was more enhanced than the soxhlet extract of flower oil which had a light white, oily, stale with hints of sweet, spicy notes.

Incorporation of Essential Oil in Cosmetic Products

M. suriga flower essential oil was incorporated into hair oil, massage oil and body creams. The stability of essential oil into different products is presented in Table 6. When it was incorporated into hair oil, massage oil, body Cream it masks the original base and it retained its original odour intensity.

Table 6: Incorporation of flower oil

Product	Parameters				
	Colour	**Odour intensity in product**	**Change in viscosity**	**Odour intensity on application**	**Odour description**
Hair oil	Yellowish	Similar to essential oil	Constant	Similar to product	Oily note, light floral, sharp, spicy
Massage oil	Yellowish	Similar to essential oil	Constant	Similar to product	Oily note, light floral, sharp, green
Cream	Pale yellow	Similar to essential oil	Constant	Similar to product	White floral, Sweet, stale

Essential oil from flowers of *M. suriga* was not only able to mask the base odour of the cosmetic products but also gave it a pleasant fragrance (Fig. 2).

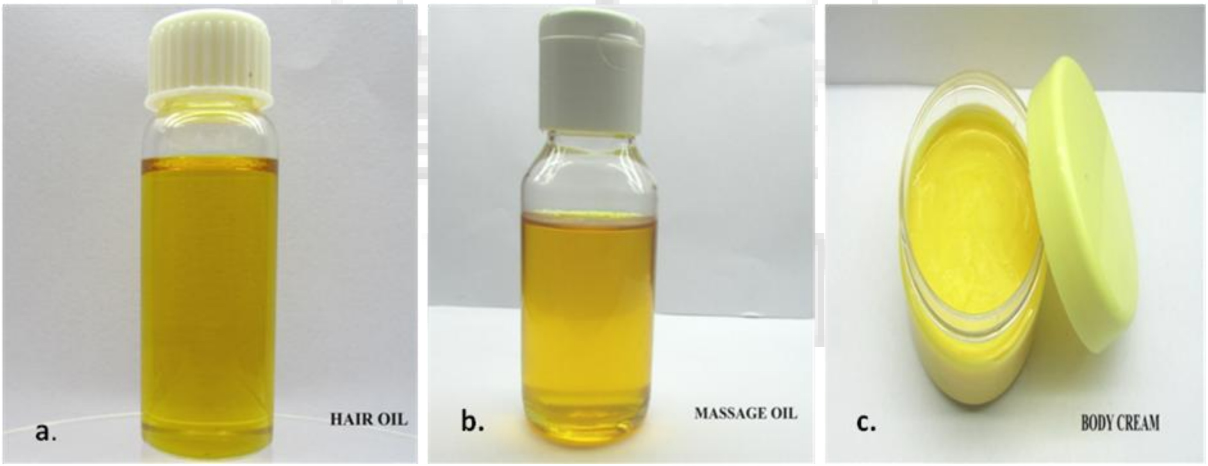

Fig.2- Cosmetic products: a) Hair oil, b) Massage oil, c) Body cream

CONCLUSION

The cold maceration by n-hexane was the suitable method for extraction of essential oils of *M. suriga*. The essential oil components were analyzed by GC-MS as well as head space analysis. The oil was subjected to odour evaluation. Essential oil from flowers of *M. suriga* was most suitable for hair oil, massage oil and body creams.

ACKNOWLEDGMENT

We are grateful to Department of Analytical Science, SH Kelkar Company, Mulund (W) Mumbai, India for helping GC-MS and headspace analysis. We would like to thank Dr. Shakti Vinay Shukla (Director of Fragrance & Flavour Development Centre Kannauj, India) for his invaluable suggestion in extraction of essential oil.

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