IJPSR (2016), Vol. 7, Issue 12 (Research Article)

INTERNATIONAL JOURNAL OF PHARMACEUTICAL SCIENCES AND RESEARCH

Received on 27 June, 2016; received in revised form, 07 August, 2016; accepted, 27 August, 2016; published 01 December, 2016

EXTRACTION OF ESSENTIAL OIL FROM FLOWERS OF *MESUA FERREA* LINN. GC-MS ANALYSIS AND INCORPORATION IN COSMETIC PRODUCT.

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Keywords:

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

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ABSTRACT: *Mesua ferrea* Linn. is a flowering tree found in Western Ghats of India, belonging to family Clusiaceae. Extraction process was standardized by cold maceration method with n-hexane. Essential oil extracted from different parts of the flower was also subjected to GC/MS and headspace analysis. The GC/MS analysis of essential oils from whole flowers of *M. ferrea* shows following components. Alpha-Copaene (13.52%), Trans-Alpha-Bergamotene (09.12%), Benzoate Geranyle-1 (09.82%), and Dioctyl Phtalate (20.21%), where as essential oil extracted from the petals of *M. ferrea* contained Alpha-Copaene (22.72%), Trans-Alpha-Bergamotene (17.42%), and Dicotyl Terephthalate (13.90%). The major components identified in stamens of the flowers by headspace technology were Alpha Copaene (28.49%), Beta caryophyllene (5.98%), Trans-Beta-Farnesene (9.24%), Trans-Alpha-Bargamotene (30.91%), and Alpha Bisabolene (5.15%) where as in petal of the flowers were Alpha Copaene (27.40%), Beta Selinene (5.18%), Trans-Alpha-Bargamotene (31.56%), Alpha Bisabolene (6.06%). The essential oils extracted from *M. ferrea* were also subjected to o*lfactory evaluation and* was incorporated in cosmetic products (Hair oil, Massage oil and Body cream).

INTRODUCTION: *Mesua ferrea* Linn. is a tree of tropical Asia and belongs to the family Clusiaceae. Various parts of the plant are used medicinally in India, Pakistan, Indo-china, Malaysia and Thailand [1, 2]. In India it is reported in the tropical rain forest of north-east. Western Ghats and Andamans in island [3]. This tree species are well known for its beautiful fragrant flowers. The *M. ferrea* produces blossoms in the month of February-May.

QUICK RESPONSE CODE

DOI: 10.13040/IJPSR.0975-8232.7(12).5106-10

Article can be accessed online on: www.ijpsr.com

DOI link: http://dx.doi.org/10.13040/IJPSR.0975-8232.7 (12).5106-10

The stamens are known as "Nagkeshar". In ancient Ayurvedic literature this plant was recommended for bleeding piles. The product -"Pile Off" capsule contains Nagkesar, which help to stop bleeding, shrinks the piles and relives pain [4]. *The* M. ferrea *oil is used in soap making* [5]. The incense sticks made from the flowers of this plant are popular worldwide for their intense fragrance and in some countries; the flowers also have been extracted and used in perfumes [6]. No standard extraction protocol of essential oil of *M. ferrea* has been reported till date. The aim of this study was to standardized protocol of extraction of essential oil of *M. ferrea.*

MATERIALS AND METHODS:

Plant material: The fresh flowers of *Mesua ferrea* were collected from forest research center of

Padave Mazgaon, Sawantwadi, Sindhudurg district, Maharashtra, India.

Extraction of essential oil:
Various methods of extraction of essential oil from *M. ferrea* were tried viz., hydro distillation, Soxhlet extraction with ethanol and n-hexane and cold maceration method using mineral oil and n-hexane. Two sets of cold maceration were done using different flowers parts. In one, whole flowers (300gms in 500ml n-hexane) and in other petals (300gms of petals in 500ml n-hexane) were used. Both 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 completely by evaporating n-hexane from the solvent obtained above using rotary vacuum evaporator.

Evaluation of essential oils by head space analysis - Fresh flower samples were collected and placed into plastic bags, and transported to the laboratory. (3±0.2 gm) of different flower parts was accurately weighed and placed, into headspace sampling bottles that were (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. These headspace bottles are transferred to the preheated oven at 80 ± 5^{o} c temperature for 30 min.

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 finally.

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, propyl paraben) were dissolved in the oil phase and heated to 75°C. The water soluble components (Methyl paraben, 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. ferrea* whole 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%
Dimeticone	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. Hydrodistillation,

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 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 with n-hexane.

GC/MS Analysis:

Essential oil extracted by cold maceration method and was subjected to GC/MS analysis. The GC/MS analysis of essential oils from whole flowers of *M. ferrea* shows the following components. Alpha copane (13.52%), Trans-Alpha- Bargamotene (09.12%), Benzoate Geranyle-1 (09.82%) and Dioctyl Phtalate (20.21%) where as in essential oil extracted from petals the major component Dioctyl terephthalate that was present high as (13.90%) which was totally missing in essential oil of whole flowers. In petal oil the Dioctyl Phtalate was less (02.51%) as compared to the amounts found in whole flower oil (20.21%).

In petal oil the components like Alpha Copaene (22.72%), Beta Selinene (08.85%) and Trans-Alpha-Bargamotene (17.42%) were also present comparatively in higher amounts. In petal oil the concentration of Alpha Copaene was (22.72%) where as in essential oil of whole flower it was only (13.52%). The concentration of Beta Selinene in petal oil was (08.85%) where as in whole flower oil it was (04.53%). In petal oil Trans-Alpha-Bargamotene was (17.42%) where as in whole flower oil it was only (09.12%) **Table 3.**

TABLE 3 GC/MS ANALYSIS OF *M. FERREA* WHOLE FLOWER OIL AND PETAL OIL

Component Name	Percentage % Whole flower oil	Percentage % Petal oil
Alpha copaene	13.52	22.72
Beta elemene	03.55	01.45
Beta caryophyllene	04.49	02.64
Trans-beta-farnesene	-	01.06
Gamma selinene	-	01.63
Beta selinene	04.53	08.85
Beta ionone	00.60	-
Alpha-humulene	00.10	-
Alpha selinene	04.00	06.79
Alpha farnesene	02.38	02.78
Delta cadinene	-	01.36
Trans-alpha-bergamotene	09.12	17.42
Alpha bisabolene	-	01.52
Benzoate geranyle 1	09.82	01.80
Benzoate geranyle 2	05.33	-
Dioctyl phtalate	20.21	02.51
Dioctyl terephthalate	-	13.90

Headspace analysis:

The different parts of flower like petals and stamens were subjected to headspace analysis. The major components identified in stamen of the flowers were Alpha Copaene (28.49%), Beta Caryophyllene (5.98%), Trans-Beta-farnesene (9.24%), Trans-Alpha- Bargamotene (30.91%), and Alpha Bisabolene (5.15%). The major components in flower petals were Alpha Copaene (27.40%), Trans-Alpha-Bargamotene (31.56%) and Alpha Bisabolene (6.06%). Bisabolene (Cis-Gamma) (4.12%) was found in a petal which was totally missing in stamens. Both the flower parts contained higher amounts of Alpha Copaene and Trans-Alpha- Bargamotene (**Table 4**).

TABLE 4: *M. FERREA* FLOWER STAMEN AND PETAL HEADSPACE AT 100DEG

Component name	Percentage % Flower stamen	Percentage % Flower Petals
Methyl heptenone	0.04	-
Bicycloelemene	0.37	-
(-)Alpha cubebene	0.86	0.49
(-)-Alpha-copaene	28.49	27.40
Beta elemene	3.06	3.14
Beta caryophyllene	5.98	4.50
Beta cedrene	0.15	0.22
Trans-beta-farnesene	9.24	1.75
Alpha-humulene	1.02	0.65
(-)Isolongifolene	0.87	-
Cuparene	0.39	-
Beta selinene	4.59	5.18
Trans-alpha-bergamotene	30.91	31.56
Alpha bisabolene	5.15	6.06
Bisabolene <cis-gamma>	-	4.12
Nerolidol-1	0.64	-
Delta cadinene	4.54	4.61

Odour Evaluation:

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

TABLE 5: ODOUR PROFILE – WHOLE FLOWER AND PETALS OIL OF *M. FERREA*.

Odour profile of whole flower oil of *M. ferrea* Linn.	Odour profile of flower petal oil of *M. ferrea* Linn.
White floral	Comparatively light floral
Sharp	Green
Pungent	Sweet
Spicy	Citrusy
Stale bitter notes	Smooth
Nutty	-

On comparison of the notes it was observed that the whole flower oil gave a sharp white floral, pungent and spicy note with stale bitter notes in the background, Where as the petal oil had a light white floral note with hints of sweet, green, citrus notes.

Evaluation of Cosmetic Products:

Both whole flower and petal essential oil were incorporated into massage oil, hair oil and body creams. The stability of whole flower oil into different products is presented in (**Table 6**). When it was incorporated to massage oil it could not mask the original base oil but in body cream it retained its original odour intensity. The petal oil had sweet floral aroma but it was not able to mask the base odour in both oils and creams (**Table 7**).

TABLE 6: INCORPORATION OF WHOLE FLOWER OIL.

Product	Parameters				
	Colour	Odour intensity in product	Change in viscosity	Odour intensity on application	Odour description
Massage oil	Yellowish	Less smell	Constant	Lesser then product	Oily note, light floral, Sharp, Pungent
Hair oil	Yellowish	Less smell	Constant	Lesser then product	Oily note, light floral, Green,
Cream	Pale yellow	Similar to oil	Constant	Similar to product	White floral, Sweet, Smooth

TABLE 7: INCORPORATION OF PETAL OIL

Product	Parameters				
	Colour	Odour intensity in product	Change in viscosity	Odour intensity on application	Odour description
Massage oil	Light green	Less smell	Constant	Lesser then product	Oily note, light floral, Green
Hair oil	Light green	Less smell	Constant	Lesser then product	Oily note, light floral, Green
Cream	White	Less smell	Constant	No perceivable odour	light floral, Sweet, Smooth

It was observed that though the petal oil has a sweet floral aroma of its own it was not able to mask the base odour of the cosmetic products and hence large quantities of the essential oils will be required to mask the base odour whereas the whole flower oil was not only able to mask the base odour of the cosmetic products but also gave it a pleasant fragrance.

CONCLUSION: The cold maceration by n-hexane was the suitable method for extraction of essential oils of *M. ferrea*. The essential oil components were analyzed by GC/MS as well as headspace analysis. The oil was subjected to odour evaluation. Essential oil from whole flowers of *M. ferrea* was most suitable for body creams and hair oil.

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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How to cite this article:
Jadhav NM, Thergaonkar RS and Deodhar MA: Extraction of essential oil from flowers of *mesua ferrea* linn. GC-MS analysis and incorporation in cosmetic product. Int J Pharm Sci Res 2016; 7(12): 5106-10.doi: 10.13040/IJPSR.0975-8232.7(12).5106-10.