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Research Paper :

Fractionation of Lemon grass oil for commercial exploitation

NADEEM AKBAR AND B.K.SAXENA

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See end of the article for authors' affiliations

Correspondence to:

NADEEM AKBAR
Fragrance and Flavour Development Centre, KANNAUJ (U.P.) INDIA

ABSTRACT

Water-steam distilled lemon grass (*Cymbopogon flexuosus* L.) oil contains citral 72-78% as main content. Citral was isolated under reduced pressure up to the maximum purity of 90% and further it is chemically converted into highly valuable aroma chemical, geranyl nitrile. The results indicated that isolation of citral and production of geranyl nitrile was a profitable processing for the perfumery industry.

Key words : Essential oil, Perfumery, *Cymbopogon flexuous*, Geranyl nitrile, Lemon grass

Lemon grass (*Cymbopogon flexuosus* L.) is an aromatic species of *Cymbopogon* belonging to family Graminae. It has been known for its essential oil for a long time and widely distributed to different agro-climatic zones of the country. This aromatic grass is perennial in nature and once planted properly can give economic yield for numbers of years depending upon the management practices, climate, soil fertility etc. This crop is sensitive to environmental conditions *i.e.* rainfall, humidity, temperature and soil fertility. Therefore, there is wide variation in both yield and quality of the oil produced at different locations. In India, this plant is found growing in wasteland, saline soils, alkaline soils, hill slopes and marginal lands of semi-arid regions with low to moderate rainfall. The essential oil is obtained from the distillation of whole plant. Lemon grass is of four high yielding varieties like CKP- 25, Kalam Krishna, Pragati and Praman.

Essential oil is the volatile oil produced by steam, or water-steam distillation of whole plant material. The vapours are condensed to yield a water condensate and an essential oil that can be separated off, usually by gravity. Essential oil is a complex mixture of hundred constituents. These constituents can be separated into single isolate by using fractional distillation unit. Fractionation is a process in which the oil is redistilled in vacuum so individual components, or fractions, are separated out as they evaporate one after the other. This is possible because fractions or constituent has its own rate of volatility based on time and temperature.

In 1997, Bhattacharya *et al.* reported four *C. flexuosus* cultivars OD -19, Pragati, Cauvery and SKK –7 rich in neral/ geranial, one rich in geraniol GR-1 and one hybrid (CKP- 25) *C. flexuosus* x *C. khasianus* Hackstapf ex. Bor.) rich in neral/ geranial that has been released for commercial cultivation. In addition to the above named lemon grass cultivars, Kulkarni (2000) listed SD 68 and Krishna as additional cultivars grown in India. Baratta *et al.* (1998) screened a commercial oil of lemongrass for it antimicrobial and antioxidant properties

The aim of this study was to fractionate the oil for getting the maximum recovery of citral and conversion of citral into geranyl nitrile for the benefit of new entrepreneurs.

MATERIALS AND METHODS

Lemon grass oil was collected locally and subjected to GC analysis by using Hewlett Packard 5890 series II gas chromatograph equipped with flame ionization detector (FID) and Carbowax 20mm polar fused silica capillary column (30m x 0.32mm.). The injector and detector temperature were maintained 210^{0}C and 220^{0}C, respectively. Nitrogen was used as carrier gas, flow rate 1.5 ml/min. The amount of sample injected was 0.1ml (split ratio 60:1). The oven temperature was programmed from 60-210^{0}C at 3c/min.

After the analysis of lemon grass oil, it was then separated into various fractions using a 25-litres capacity glass fractionating column equipped with 4-nech flask capacity 25 liters, double walled column 4"/ 1 meter with stainless steal wire sulzer packing, reflux divider 4" with thermometer, condenser 4"x24" long, receivers of 5000ml and 2000 ml of 2 necked 1"/1" with bottom outlet 1". The vacuum pump of 600 liters capacity per minute was used with the unit. Glass reaction unit of 25 liters capacity 4 neck flask fitted with Teflon stirrer, thermometer pocket, reflux divider 4" with 1"thermometer, heat exchanger 4"/ 24" receivers of 5000ml and 2000 ml of 2 necked 1"/1"

with bottom outlet 1". The experiment was carried out at Process Technology Division, Fragrance and Flavour Development Centre, Kannauj, U.P.

Lemon grass oil contains a number of fragrant fractions of which citral-a and citral-b are the major components with other minors like limonene, linalool,, geraniol,nerol,alpha pinene,beta pinene, nerol and geranyl acetate (Pino and Rosado, 2000; Kasali *et al.*, 2001). Fractional distillation of lemon grass oil yielded 7 fractions that were tested in quality assurance lab (Table 3). In a 25 liters capacity reaction unit equipped with stirrer, thermometer, additional funnel and reflux condenser charged water and then dissolved hydroxylamine hydrochloride along with sodium hydroxide and heated. Citral of 90% purity was charged into this solution carefully at a required temperature and maintaining the temperature for two hours. After completion of the reaction, the oxime product was washed in presence of a solvent. In the second step, this oxime compound was dehydrated into nitrile in presence of acetic anhydride at a certain temperature. Finally fractionate, the crude geranyl nitrile along with neryl nitrile at about 10-12 mm pressure through a packed column.

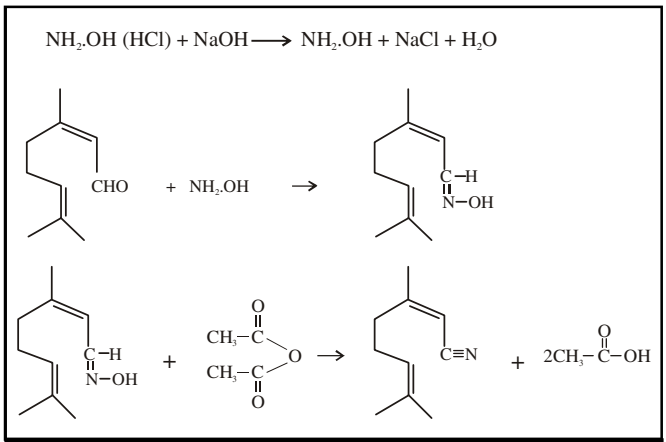

Fig. 1 : Reaction mechanism:

Table1 : International standards for lemon grass Oil (ISO 4718-1981)

Properties	Specifications
Appearance	Clear, mobile liquid
Colour	Pale yellow to yellowish brown
Relatively density at $20/20^0$ C	Minimum: 0885 Maximum:0. 905
Refractive index at 20^0 C	Minimum: 1.4830 Maximum: 1.4890
Optical rotation at 20^0 C	Range: -3^0 to $+1^0$
Miscibility in 70% (V/V) ethanol at 20^0C	One (1) Volume in three (3) volumes of 70%(V/V) to give a clear solution, which sometimes becomes opalescent on further dilution.
Carbonyl value	Minimum: 268- corresponding to 73% of carbonyl compounds, expressed as Citral.
Residue from vacuum distillation	Maximum: 10%(m/m)

Table 2 : Quality parameters of lemon grass oil used in the processing

Sr. No	Quality parameters	Values
1.	Refractive index at 27^0C	1.4852
2.	Specific gravity at 27^0C	0.8895
3.	Limonene	4.1%
4.	Linalool acetate	1.13%
5.	Linalool	3.03%
6.	Citral-a	32.59%
7.	Citral-b	41.26%
8.	Geranyl acetate	3.67%
9.	Nerol	0.47%
10.	Geraniol	2.44%

RESULTS AND DISCUSSION

The composition of lemon grass oil, which is used in the processing, is displayed in Table 2 along with ISO specification in Table 1. The oil sample was analyzed by GC and found that lemon grass oil contained 32.5% citral-a and 41.2% citral-b. First, this oil is fractionated at reduced pressure for the recovery of total citral components (Table 2). Thereafter, the fractionation, all possible citral containing fractions (4,5,6) were blended and (Table 4) it was found that the recovery of the main product as citral was 76%. The average purity was 93%. In the fractionation of the oil, various fractions were collected at required temperature and reduced pressure. Apart from citral containing fractions, other fractions were also checked and may be utilized in the making of reconstituted lemon grass oil and cheaper rate fragrances for agarbatti and dhoopbatti. During very sophisticated operation of fractionation unit, vacuum was maintained 5-3mm and the processed temperature was maximum $145\text{-}160^0$ C. The losses were 4-5% during fractionation of oil. Ten kgs material was taken in this experiment.

Citral was now charged into the glass reaction unit and added alkaline solution of hydroxylamine hydrochloride for making the intermediate oxime compound. In this

Table 3 : Fractionation of lemon grass oil

Fractions No.	Vacuum in mm Hg	Pot temperature (^{0}C)	Vapour temperature (^{0}C)	Yield of fractions
1	750	90-120	68-70	4.50%
2	752	120-128	70-74	2.00%
3	752	128-130	74-84	3.50%
4	754	130-135	84-90	12.50%
5	755	135-140	90	25.00%
6	755	140	90	38.50%
7	755	140-148	90-92	3.50%
			Residue	5.50%

Table 4 : Conversion of citral into geranyl nitrile

Sr. No.	Fractions used	Citral contents in fractions	Conversion	Purity of product by GLC
1.	4	91.67%	84.21%	91.85%
2.	5	92.58%	85.63%	92.10%
3	6	94.84%	85.33%	92.45%

reaction hydroxylamine hydrochloride was added into aqueous sodium hydroxide solution and heated for 2 hours at required temperature. Further this oxime compound was dehydrated into nitrile in presence of acetic anhydride. Citral containing fractions were taken separately in this chemical reaction. The average yield of geranyl nitrile was 85% of Citral and average purity of geranyl nitrile was 92%. The results are shown in Table 4. Chagonda *et al.*(2002) had also evaluated the essential oils of *Cympopogon* spp. from Zimbabwe.

Authors' affiliations:
B.K. SAXENA, Department of Chemistry, D.A.V. (P.G.) College, KANPUR (U.P.) INDIA

REFERENCES

1. **Clevenger, J.E.** (1928). Apparatus for the determination of volatile oil. *J. American Pharm. Assoc.*, **17** : 346.
2. **Bhattacharya, A.K.,** Kaul, P.N., Rajeswara Rao, B.R. Mallavrapu, G.R. and Ramesh, S.I. (1997). Interspecific and intercultiver variation in the essential oil profile of lemongrass. *J. Essent. Oil Res.*, **9** : 361-364.
3. **Baratta, T.,** Dorman, H.J. Deans, S.G. , Figueirodo, A.C., Barroso, J.G. and Roberto, G. (1990). Antimicrobial and antioxidant properties of some commercial essential oils. *Flav. Fragr. J.,* **13**: 235 244 (1998).
4. **Oberhofer, A.,** Nikiforov, Buchbauer, G. , Jirovetz, L. and Bicchi, C. (1999). Investigation of the alteration of the composition of various essential oils used on aroma lamp application flv. *Fragr. J.,* **14** : 293-299.
5. **Chagonda, L.S.,** Makanda, C. and Chalchat, J.C. (2000). Essential oils of cultivated *Cymbopogon winterianus* (Jowilt) and of *C. citratus* (DC.) Stapf. from Zimbabwe. *J. Essent. Oil Res.*, **12** : 478-480.
6. **Pino, A.** and Rosado, A. (2000). Chemical composition of the essential oil of *Cymbopogon citrates* DC. Stapf. form *Cuba. J. Essent. Oil Res.,* **12** : 301-302.
7. **Kasali., A.A.,** Oyedeji, A.O. and Ashilokun, A.O. (2001). volatile leaf oil constituents of *Cymbopogon citrates* DC. Stapf. *Flav Fragr. J.*, **16** : 377 –378.

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