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Impact of species and particle size on essential oil yield of *citrus* peel (*Citrus* Spp.)

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Abstract
Essential oil was extracted from the peel of four *citrus* species namely, Jatti Khatti, Soh Sarkar, grapefruit and pummelo using Clevenger apparatus. All the four species yielded significantly different essential oil. The mean essential oil yield of 8.08, 4.64, 4.77 and 3.79% (db) was obtained from Jatti Khatti, Soh Sarkar, grapefruit and pummelo, respectively. Particle size of peel was found to have significant influence on oil yield. Oil yield of 0.68% was obtained from whole peel, whereas peel of 0.5 mm size exhibited maximum yield, increasing it by about 13%. Analysis of oil indicated that number of volatile compounds in grapefruit was 16, whereas in Jatti Khatti, Soh Sarkar and pummelo, these compounds were 14, 11 and 6, respectively. Limonene, the major compound was found to vary from 93.57-96.41% in the essential oil. Physical and chemical properties of essential oil revealed better quality of essential oil from grapefruit.

Keywords: *Citrus* species, essential oil extraction, gas chromatography, storage conditions

Introduction
Citrus represent the third most important group of fruits next to mango and banana and comprise about 10% of total fruits produced in India (Mishra *et al*. 2005) [9]. *Citrus* fruit comprises of about 25-30% peel, consisting of two parts; inner part known as albedo, which is white in colour and outer part, called as flavedo, which is green in colour. Theflavedo layer contains essential oils in range of 0.5 to 3.0 kgper tonne of fruits (Ahmad *et al*. 2006a) [2]. These oils are veryuseful and are widely being used as flavouring and maskingagents in many foods, cosmetics and pharmaceuticals, as antimicrobial agent, mosquito-larvicide and also as aninsecticide. This oil is confined in oblate to spherical shaped oil glands, which are known as oil sacs. The oil sacs are intercellular cavities embedded irregularly at different depthin the flavedo beneath pericarp and hypoderm and above the albedo. Therefore, efficient extraction of this oil poses technological challenge.
Studies have been reported on extraction of essential oil from the peel of *citrus* fruits using different methods (Sud and Badyal 1993, Brat *et al*. 2001, Mishra *et al*. 2005, Ahmad *et al*. 2006, Bousbia *et al*. 2009) [13, 4, 9, 1, 5]. Sud and Badyal (1993) [13] extracted peel oil from the rind at various stages offruit maturity from various *citrus* cultivars such as oranges, Kinnow, Cleoptara mandarin, lime and hill lemon using steam distillation and reported maximum yield in terms of peel oil as 2.05 ml/100 g peels in orange and minimum in hill lemon. Brat *et al*. (2001) [4] reported essential oil extraction by flash vacuum expansion of peel of lemon, orange, mandarin and grapefruit with maximum yield of 2.41 kg/tonne of fruits. Mishra *et al*. (2005) [9] evaluated the effect of pre-treatments with enzyme (0.1 to 0.3%) and found a maximum increase in yield by 13% over control. Ahmad *et al*. (2006a) [2] studied on essential oil extraction from peel of *citrus* species, Malta, grapefruit, Kinnow, Fewtrell's early, Mosambi, and Eureka lemon by cold pressing method and determined that Malta peel had the highest oil yield (1.21%), Eureka lemon (1.12%), Mosambi (0.98%), grapefruit (0.73%), Kinnow (0.32%), Fewtrell's early (0.22%). Bousbia *et al*. (2009) [5] extracted peel essential oil from *citrus* varieties such as Eureka, Villa França, lime, Marsh Seedless, Tarocco, Valencialate, Washington Naval, Tengelo Seminole were extracted by Clevenger apparatus using microwaved hydro-diffusion and gravity and reported a significant reduction in extraction time (15 min). Thus, the major research efforts have been on extraction of essential oil from various *citrus* species but Indian varieties have not received attention. The objective of present research work was to evaluate the effect of species and particle size on yield of the essential oil from *citrus* peel.

The size reduction of peel was aimed to increase the yield of essential oil.

Materials and Methods

The *citrus* species such as Jatti Khatti (*Citrus* jambhiriLush.), Soh Sarkar (*Citrus* karna Raf.), grapefruit (*Citrus* paradisi Macfad.), pummelo (*Citrus* grandis Osbeck) were obtained from the orchard of the Division of Fruits and Horticultural Technology, Indian Agricultural Research Institute, New Delhi (India) for the experiment. For extraction of essential oil, peel was first removed manually from the fruit using knife. The whole peel as well as flavedo was cut into pieces of size 32 mm × 12 mm and then flavedo were further ground into finer particle sizes. Grinding was carried out using mixer-grinder (Vector make) having two blades for 2, 5 and 8 minutes to a peel size of 1.4 mm, 1.1 mm and 0.5mm, respectively. The average particle size of above ground and dried (12% wb) sample of peel was calculated in terms of volume surface mean diameter.

Extraction of essential oil was carried out using Clevenger apparatus. Two hundred gram of peel along with distilled water was taken into round bottom flask of the apparatus using peel: water in the ratio of 1:3 (w/V). Thereafter, heating mantle was "PUT ON" and kept for boiling of water for 2 hr. The variac position in the heating mantle was fixed. As the boiling of water progressed, the water and volatile oil in the form of vapour started rising which got condensed in the condenser tube. The condensed water and oil were collected into a calibrated tube. Excess water flowed back into the boiling flask containing the *citrus* peel. On completion of extraction process, essential oil and water mixture was removed from the tube and put into separating funnel to separate water from oil. Diethyl ether (Approx 3-4 ml) was also added into separating funnel. The oil sample collected in the glass tube was kept open in ambient condition (for about 30 minutes) so that diethyl ether could get evaporated. Further, to ensure complete removal of moisture from oil, sodium sulphate (about 1mg) was added into oil sample and stored in refrigerator at 4ºC. The oil yield was calculated using equation 1 and 2, as follows:

Volume of essential oil collected (ml)

$$\text{Oil yield at wet basis (\%)} = \frac{\text{Volume of essential oil collected (ml)}}{200\ (\text{g})} \times 100 \quad (1)$$

$$\text{Oil yield at dry basis (\%)} = \frac{\text{Volume of essential oil collected (ml)}}{\text{Dry weight of peel (g)}} \times 100 \quad (2)$$

Analysis of components of peel essential oil obtained from peels of freshly harvested fruits (i.e. without storage) was done using Gas Chromatography (GC). GC analysis was performed using PerkinElmer Auto system gas chromatograph equipped with flame ionisation detector and a carbowax 20M capillary column (50 m × 0.25 mm; film thickness 0.25 m). The oven temperature was programmed from 60ºC (10 min) to 180ºC at 3ºC/min. Injector and detector temperature was maintained at 200 and 250ºC, respectively. Nitrogen was used as the carrier gas at the flow rate of 40 ml/min. Oil sample (0.1 l) was injected in split ratio (1:40). The relative amount of individual components was calculated based on area under peak.

Statistical analysis of data was carried out using SPSS16.0. As mentioned above, the experiment comprised of two factors, *viz*. species, and particle size having four and five levels, respectively. Accordingly, experiment was planned as factorial experiment in completely randomized design with three replications. Analysis of variance in oil yield was performed using the univariate option of general linear model by confounding higher order interaction in order to improve the efficiency of main effects and first order interactions. Characteristics of the data were obtained using descriptive statistics module of SPSS.

Results and Discussion

Effect of Species on Essential Oil Yield

Jatti Khatti, Soh Sarkar, grapefruit, and pummelo had the peel content of 36.58%, 43.11%, 22.89%, 34.05%, respectively and the thicknesses of peel were found to be 5.05, 10.36, 3.59 and 11.29 mm, respectively. The corresponding thicknesses of flavedo were found to be 3.31, 2.59, 1.23 and 2.10 mm, respectively. Maximum oil yield was obtained from Jatti Khatti, followed by grapefruit, Soh Sarkar and pummelo (Table 1). It is apparent that thickness of flavedo cannot be related with the oil yield.

Table 1: Descriptive Analysis of Oil Yield (% db) Between *Citrus* Species

Species	Mean	Standard Error
Jatti Khatti	8.0859	0.4515
Soh Sarkar	4.6432	0.2542
Grapefruit	4.7772	0.3092
Pummelo	3.7944	0.2957

Previous studies too have indicated significant variation in oil yield between species. For instance, Ahmad *et al*. (2006b) [3] found the total essential oil content of *citrus* fruits namely, Malta, Mosami, grapefruit and Eureka lemon to be 1.21, 0.98, 0.73 and 1.12%, respectively.

Effect of size of peel on essential oil yield

The essential oil yield on dry basis varied from 1.1 to 12.98% (0.2 to 2.1% wb) in Jatti Khatti whereas in Soh Sarkar, grapefruit and pummelo, the corresponding values were found to be 0.52-6.91% (0.1-1.25% wb), 0.38-7.92 (0.1-2.05% wb) and 0.73-10.07% (0.15-2.05% wb), respectively (Fig 1). Essential oil yield increased with decrease in particle size (i.e., increase in grinding time) of the peel in all the 4 *citrus* species, and reached to almost maximum at 5 min of grinding (corresponding to particle size of about 1.1mm) (Fig. 1). It is evident that oil yield varied widely among species and particle size of peel (Table 2). The oil recovery was less (mean yield of 0.68%) when extraction was carried out from whole peel. This may be due to presence of albedo layer, causing resistance to flow of water in oil sac as well as resistance to flow of oil-water mixture back to the solution. However, the mean yield increased to 2.24% when extraction was done from flavedo of same cross-sectional size but of reduced thickness. The obvious reason to increase in oil yield was the availability of both surfaces of peel to waterflow inside the peel, which was otherwise unavailable due to albedo layer. The mean oil yield increased to 7.03% on grinding of flavedo.

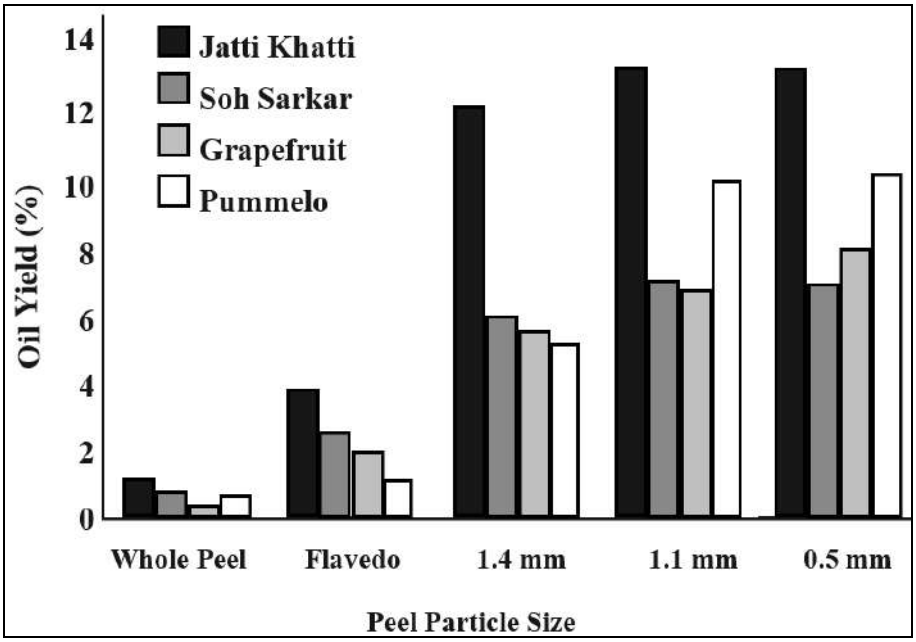

Fig 1: Effect of Peel Particle Size on Essential Oil Yield of Different *Citrus* Specie

for 2 min (particle size of 1.4 mm). With further grinding of flavedo to 1.1 mm and 0.5 mm, the oil yield increased further to a level of 8.13 and 8.86%, respectively. Thus, a very significant increase in oil yield was found on reducing particle size of peel. This increase in oil yield might be due to increased surface area of peel and thereby having close contact with water during extraction as well as having reduced resistance to oil water mixture fromoil sac to the solution.

Comparing the essential oil yield from grapefruit, the obtained maximum (2.05%) yield in the present study was found to be quite higher than previous reported yield of 0.73% (Ahmed *et al*. 2006b) [3]. Similarly, the maximum essential oil yield of 2.05% obtained in pummelo in the present study was much higher than 0.93% reported by Jin-hua *et al*. (2006), primarily due to size reduction. Thus, size reduction could be considered as an effective pre-treatment for increasing essential oil yield.

Table 2: Effect of peel particle size on essential oil yield of different *citrus* species

	Particle Size		***Citrus* Species**	
	Jatti Khatti	**Soh Sarkar**	**Grapefruit**	**Pummelo**
Whole peel	1.13	0.53	0.38	0.74
Flavedo	3.82	2.64	1.93	1.23
1.4 mm	11.85	5.91	5.55	5.16
1.1 mm	12.95	6.86	6.76	9.83
0.5 mm	12.97	6.91	7.92	10.07

Composition of Volatiles in Essential Oil

The volatile components in essential oil were found to be hydrocarbons and oxygenated classes such as monoterpenes, sesquiterpenes, monoterpenole, aldehyde, alcohol and ester (Table 3).

Quantitatively, the most representative monoterpene was observed to be limonene and their concentration varied widely among species (Table 3). Other monoterpenes were α-pinene, β-pinene, myrcene, in different concentrations in Soh Sarkar (2.67%), grapefruit (1.8%), and pummelo (2.05%). However, camphene was also identified in Jatti Khatti (3.42%). This variation in components of different species could be due to differences in physiological factors such as tissue age (maturity), difference in species and genetic make-up (Rehman *et al*. 2008) [11].

It was also found that the limonene contents obtained in the present study (93-96%) were higher as compared to the previous findings (Ahmad *et al*. 2006a, Chowdhury *et al*. 2007, Rehman *et al*. 2008, Tao *et al*. 2008) [2, 6, 11] in different *citrus* species.

Table 3: Componential concentration (%) of volatiles in *citrus* peel essential oils extracted from the fruit peel of different *citrus* species

Volatiles	***Citrus* Species**			
	Jatti Khatti	**Soh Sarkar**	**Grapefruit**	**Pummelo**
Monoterpenes				
α-pinen	0.48	0.47	0.11	0.25
Camphene	0.03			
β-pinene	0.74	0.10	0.07	
Myrcene	2.17	2.10	1.62	1.80
Limonene Sesquiterpenes	95.13	96.23	93.57	96.41
γ-terpinene	0.17	0.19	0.15	
β-caryophyllane Monoterpenole	0.04		0.09	

Terpinine-4-ol	0.10	0.13	0.05	
Nerol	0.37	0.08	0.28	
α-terpineol			0.18	
Geraniol Aldehydes	0.04	0.15	0.07	
Citronellal			0.26	0.17
Linalool	0.11	0.18	0.30	0.23
Neral			0.18	0.06
Geranial Ester	0.08		0.19	
Linalyl acetate	0.31	0.20	0.57	
Geranyl acetate	0.05	0.12	0.13	
Total (%)	99.82 (14)	99.95 (11)	97.82 (16)	98.92 (6)

Total % value is below 100 as some of the compounds could not be identified

It was inferred from the study that essential oil yield isstrongly dependent upon species as well as the particle size of peel. Maximum oil yield was obtained from Jatti Khatti (8.08%) followed by grapefruit (4.77%), Soh Sarkar (4.64%), and pummelo (3.79%). Size of peel was considered as an effective pre-treatment for increasing essential oil yield. By reducing the particle size, the essential oil yield increased by about 1050-1950%. Limonene content, representing the quality of essential oil, was found to be higher in pummelo (96.41%) followed by Soh Sarkar (96.23%), Jatti Khatti (95.13%) and grapefruit (93.57%).

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