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Evaluation of size reduction on the yield and quality of celery seed oil

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Abstract

The effect of grinding/flaking with and without pre-cooling of celery seeds on the yield and physical and chemical characteristics of volatile oil was evaluated. For smaller batches (200 g) with pre chilling and flaking, yields of oil were marginally but consistently higher (2.20%), compared to grinding celery at ambient temperature using a mini plate mill (1.9%) and with waring blender (1.8%). With flaking at room temperature the yield of oil was 2.0%. However, in large batches (10 kg), with steam distillation the yield of steam distilled oil was significantly higher for flaking (1.76%) as compared to the hammer mill powdering (1.4%), both at room temperature. Extraction of volatile oil from celery powder or flakes follows first order kinetics with an variance value of 0.04. Gas chromatograph (GC) and gas chromatograph–mass spectra (MS) analysis showed that in case of flaking, the volatile oil had higher levels of limonene, the major volatile compound and sedanenolide, the major character impact compound being present in almost equal quantities in both the cases of flakes and powder. Selective collection of volatile oil at different intervals of time of distillation gave products of different flavour profiles. Flaking had the advantage of higher yields of the volatile oil with better flavour quality. It was also observed that flaking of celery helped in overcoming the problem of clogging and choking which is associated with the conventional grinding.

Keywords: Flaking; Powdering; Steam distillation; Quality; Volatile oil; Sedanenolide

1. Introduction

Celery seed is the dried fruit of *Apium graveolens* Linn. belonging to the Umbelliferae family. It is valued as a spice and imparts a warm and pleasing flavour to foods such as canned soups, sauces, pickles, tomato products and meats. Celery is extensively cultivated for the seeds in India, France and United States. Celery essential oil lends a floral like odour to oriental perfumes to which it imparts warm and clinging notes (Lewis, 1984). The ground seed is mixed with table salt to give "celery salt" which is used in flavouring fish, salads, and eggs. Out of a world production of 6000 t, India produces 4000 t and exports about 3000 t. Celery is used in various forms such as fresh herb, seeds, oil and oleoresin for flavouring foods. The seed contains on an average 2.5% volatile oil and 15%–17% fixed oil.

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The composition of celery volatile oil has been studied by many workers (Lawerence & Reynolds, 1998; Gupta & Baslas, 1978; Wilson, 1970) and GC–MS analysis has shown that δ-limonene and selinene form about 60% and 20% of the oil, respectively. However, the important flavour constituents of the oil responsible for the typical aroma are 3-*n*-butyl-4-5-dihydro phthalide (sedanenolide), 3-*n*-butyl phthalide, sedanolide and sedanonic anhydride which are present in very low levels (Choudhary & Kaul, 1992). Six major compounds viz.,piperitone, eugenol, β-pinene, terpinolene 3-carene, myrcene and menthone have been reported in steam distilled celery seed oil (Guenther, 1990). Distillation of fresh celery juice and identification of flavour compounds such as phthalides and hydro phthalides have been reported (Gold & Wilson, 1963). Myrcene, Limonene, butyl phthalide, pentyl benzene and β-carryophyllene have been reported in the oil of a selected Indian variety of celery seed. A review on celery with composition of oil has been published (Verghese, 1990). Celery seed oil is a valued product both in flavour and fragrance industries. Owing to its high fixed oil or fatty oil content, powdering of celery poses problems such as clogging of the mill, over-heating and loss of volatile oil when subjected to conventional grinding techniques using plate mill or hammer mill. Flaking as an alternative to cryo grinding an expensive operation was adopted for improved oil recovery from celery. The objective of the present study was to evaluate the effect of flaking on the yield and physico-chemical quality of volatile oil fractions obtained by steam distillation and on the recovery of oil from condensate.

2. Materials and methods

2.1. Materials

Celery seeds grown in Punjab, India, was used in the present study reference flavour chemicals viz., were purchased from Sigma Aldrich Co. Solvent acetone and hexane were of analytical grade. For powdering celery Laboratory model mini plate mill Buhler miag, Italy and laboratory model grinder–Sumeet, India, were used for powdering and Laboratory model flaker Pascal Engineering, Sussex, England was used for flaking for laboratory scale (200 g) experiments. For bigger batch (10 kg) Hammer mill Batliboi, India was used for powdering, and Twin drum roller fabricated at CFTRI, Mysore, India, was used for flaking.

2.2. Laboratory scale experiment (200 g batch)

Celery seeds were powdered in a dry grinder at laboratory level and 200 g of powder was subjected to hydro-distillation by Clevenger method (ASTA, 1991) for 5 h. In another batch, celery seeds were flaked in a laboratory model flaker with a gap adjustment of 0.1 mm between the rollers. Flakes (200 g) were subjected to hydro-distillation for 5 h. The yield of volatile oils were expressed as percentage (ml/100 g).

2.3. Scale up experiment (10 kg batch)

Volume of oil collected every 30 min was measured and altogether four fractions F1, F2, F3 and F4 were collected. The fractions were analysed for their physical properties viz., specific gravity, refractive index and optical rotation (ISI, 1982) and chemical composition by GC with FID detector. Confirmation of phthalides was carried out by GC–MS analysis.

2.4. Analysis of volatile oil by gas chromatography

Shimazdu 15-A gas chromatograph with column- SE-52 on chromosorb B (10 ft length 1/8 i.d.) with a temperature programme of 75/5/180/2/200 °C with a injector temperature: 150 °C, detector temperature: 210 °C, carrier gas flow: 30 ml/min. The oil (0.05 ml) was diluted in acetone (1 ml) and 1 μl was injected to GC.

2.5. Recovery of oil from condensate

During steam distillation, the condensate was found to be turbid, indicating that certain amount of oil was getting dispersed in water. An attempt was made to recover the oil from condensate by hydro-distillation and hexane extraction. Well mixed condensate (5 l) obtained during 5 h of steam distillation was subjected to hydro-distillation for 30 min. The well mixed condensate from steam distillation (2 l) was taken in a separating funnel and 200 ml of hexane was added ,shaken well and allowed for the separation of two layers. Hexane layer separated at the top was collected. The extraction was repeated with fresh 200 ml hexane and pooled extract was distilled to get the oil.

2.6. Kinetics

Kinetics of the extraction of celery volatile oil during steam distillation was calculated (Levenspiel, 1972). The general equation for kinetic study is

$$-\frac{dC_A}{dt} = kC_A^n \tag{1}$$

C_A is the yield of oil at any time t, k is the rate constant and n is the order of change. It is the common experience that the first order ($n = 1$) changes are encountered in most of the cases. Assigning $n = 1$ in Eq. (1) and rearranging, we get

$$-\frac{dC_A}{C_A} = k\,dt \tag{2}$$

Integrating Eq. (2) at conditions
$C_A = C_{A_0}$ at any time $t = 0$ and C_A at any time t, we get

$$-\ln\frac{C_A}{C_{A_0}} = kt \tag{3}$$

The rate constant k was obtained by using Eq. (3).

3. Results and discussion

3.1. Lab scale studies

In case of small batch size (200 g), the yield of oil was almost equal both in case of powder and flakes, probably because difference between the material temperature during grinding (32 °C) and flaking (27 °C) was only 5 °C. The yield of oil was 1.9% and 1.8% in the powders obtained in plate mill and dry grinder, respectively, which favorably compares with 1.98% oil yield from flakes (Table 1). Pre cooling of celery seeds to 5–8 °C by keeping the celery seeds in freezing compartment of refrigerator, resulted in higher oil yield 2.2%, 2.05% and 2.2% for powder obtained in plate mill, dry grinder and flaker, respectively The higher yield of oil may be due to the fact that temperature attained by the product during grinding or flaking was lower compared to normal operation without pre-cooling. The temperature attained by the product by pre-cooling followed