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Steam distillation of lemon grass (*Cymbopogon* spp.)

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A simple first order kinetic model has been developed for steam distillation of lemongrass (Cymbopogon spp.). This rate model was tested on pilot scale, steam distillation units, with lemon grass batches of 70 to 1000 kg. For intimate contact of lemongrass and steam, two improved direct steam spargers were provided in these units. It is observed, that, the loose packing of plant material inside distillation still and steam injection rate, increases the oil yield. Straight line behaviour with intercept on time axis of the above rate model explains, that, oil production inside the distillation unit is not instantaneous, but some time is required for wetting of grass for the diffusion and osmosis of oil.

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Crude essential oils are obtained by steam distillation of variety of natural products like plants, grasses, wood stumps, sawdust, flowers, kernels and seeds. These oils have played an important role in the personal and social hygiene of mankind in terms of their use in cosmetics, toiletries, medicinal formulations, aroma therapy, surface coatings, etc.

Most essential oils occurring in nature, consist of mixtures of hydrocarbons[1] like terpenes, sesquiterpenes, oxygenated compounds like alcohols esters, ethers, aldehydes, ketones lactones, phenols and waxes. Out of these, oxygenated compounds are the principal odour carriers. They are more stable against oxidation and resinifying agents. The unsaturated hydrocarbons like terpenes are less stable and are responsible for degrading oils. To make essential oils more stable, so that they retain most of their odours and flavours, these terpenes are removed, so that only oxygenated compounds are retained.

Lemon grass oil is obtained by steam distillation of lemon grass (*Cymbopogon* spp.). It is the most common and cheapest oil available in the market. Two varieties of this grass code named as RRL-16 and CKP-25 have been developed at Regional Research Laboratory, Jammu. These contain 0.28 and 0.5% of essential oil respectively. The former has 79-82% citral and the latter has 82-85% citral [2].

Experimental Procedure

Pilot plant set-up

The experimental pilot plant set-up for steam distillation[3,4] of lemon grass is shown in Fig. 1. Experiments were conducted on 100 and 1000 kg scale. The above mentioned system consists of a distillation still, having capacity to hold 100 and 1000 kg of lemon grass per batch. The walls of the still are slightly tapering from top to bottom for uniform

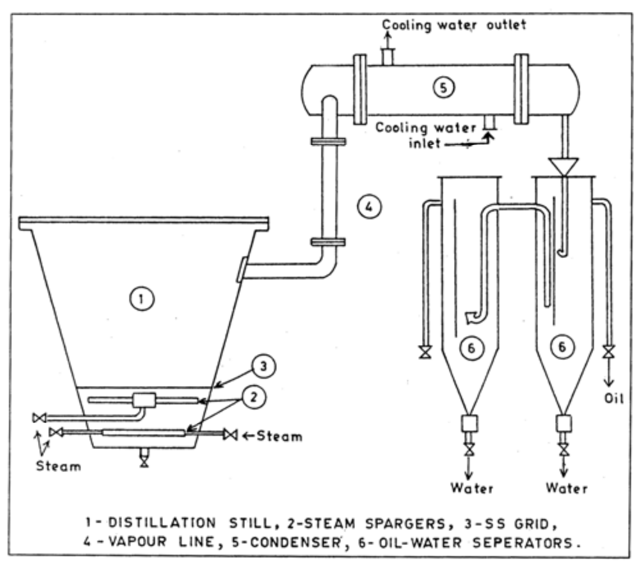

Fig. 1—Steam distillation unit
1 – distillation still , 2 – steam spargers, 3 – ss grid,
4 – vapour line, 5 – condenser , 6 – oil-water separators

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mixing of steam and plant materials. Two separate steam spargers are provided, in the lower side of the still, for the injection of open steam. The first sparger is a hollow circular pipe with cross in the middle. The sparger has holes in the bottom. Steam is injected from diametrically opposite sides. Second sparger consists of a circular box in the middle with four to six hollow pipes clinging out from the centre to the circumference and holes are drilled at the lower end of all these pipes. Steam is injected directly into the circular box, and spreads from centre towards circumference from the bottom holes of the sparger. Whereas in the 1st sparger the spreading of steam starts from circumference towards centre. On the whole, these two spargers collectively help in intimate mixing of plant material and steam. This design of the spargers help in intimate contact of steam and plant material to take away maximum oil and avoid channeling of steam. A steel grid is provided above these two spargers for holding the plant material on it. The distillation still is provided with lid, which is fixed by swivel bolts. The top side of the still is connected to the vapour line, condenser and two oil/water separators[5,6].

Method

Lemon grass (chopped or unchopped) is filled in the distillation still and its lid is fitted tightly by swivel bolts, so that oil and vapour do not leak out. The steam is injected in the still by the help of steam spargers provided at the bottom of the vessel. The upcoming steam carries away the oil from the plant material i.e. lemon grass and both oil as well as steam pass to the condenser through vapour line, where these vapours get condensed and oil and water are separated in the separators. Oil being lighter is separated out from the top and water being heavier is taken out from the bottom of separators.

The oil thus obtained is lemon grass oil having 80-85% citral content. To make it stable (i.e. terpene less) the terpenes are separated using fractional distillation.

Theory

Essential oils are highly heat sensitive, as such distillation and fractionation is to be carried out at low temperatures and sub-atmospheric pressures. In case of fractionation, vacuum pumps are used for this purpose, while as in case of steam distillation, pressure lowering effect is achieved by lower partial pressure of the feed components, enabling vapourization at sub-atmospheric conditions[7,8].

Further, direct contact heat transfer provided by steam, initially gives wettability to the grass at high temperatures, enhancing diffusion and osmosis of the oil. Rate of oil vapourization of plant material in steam distillation is not influenced by relative volatility of oil components but by their solubility in water (in vapour phase). Extraction of essential oils from lemongrass by steam distillation is a rate process. Also, oil removed per unit time is directly proportional to oil remaining in the grass. The simplest rate equation is given by first order kinetics[9,10].

$$-\frac{\mathrm{d}m}{\mathrm{d}t} = km$$

or

$$-\frac{\mathrm{d}m}{m} = k\,\mathrm{d}t$$

or

$$\ln\frac{m_0}{m} = k.t$$

or

$$\ln\frac{1}{1-u(t)} = kt \qquad \ldots (1)$$

where m= average concentration of essential oil in the grass at any time t, m_o= initial concentration of essential oil before steam distillation starts, t = time of steam distillation and $u(t)$ = fraction of oil extracted.

If the above model holds good then the plot of ln $1/1-u$ versus t shall produce a straight line passing through the origin.

Results and Discussion

Table 1 shows the results of experiments carried out on pilot scale for this study. The weight of the plant material i.e. lemon grass ranged from 70 to 1000 kg. In 1st experiment, grass was tightly packed in the distillation still, while in all other experiments grass was loosely packed. Two experiments were conducted with chopped grass as well. Steam was injected and distillation was carried out in each experiment for maximum period of 5 h. More than 75 to 80% of oil was extracted within first 2½ h in each case.

Table 1—Yields of lemon grass oil after 2½ h and 5 h

Exp. No.	Weight of plant material (kg)	Condition of plant material	Packing	Steam flow rate (L/h)	Oil produced after	
					2½h. (mL)	5 h (mL)
1	100	Unchop.	tight	12-15	255	310
2	86	Unchop	loose	12-15	330	420
3	70	Chop	loose	15	350	385
4	70	Chop	loose	12	300	330
5	1000	Unchop	loose	160	5400	5725
6	1000	Unchop	loose	125	4900	5215
7	1000	Unchop	loose	140	5000	5315

Steam flow rate was varied from 12-15 L/h for 70 to 100 kg scale experiments (i.e. Expt. No. 1 to 4), while for 1000 kg scale experiments, steam injection was varied from 125 to 160 L/h, but keeping one particular rate fixed for each experiment. The essential oil produced was recorded after every half an hour, in case of each experiment for kinetic studies. In the last column, Table 1 shows, the oil yield after 2½ h and after 5 h for each experiment.

Table 2 shows the oil yield, after rationalizing all the experiments performed using 100 kg of plant material per batch.

Table 2—Yields of oil after 2½ h and 5 h of steam distillation (on common basis of plant material and rationalizing steam flow rate = 100 kg)

Weight of plant material taken for each experiment = 100 kg

Experiment No	Steam flow rate (L/h)	Oil yield after	
		2½ h (mL)	5 h (mL)
1	12-15	255	310
2	12-15	384	489
3	15	500	550
4	12	-	472
5	16	540	573
6	12.5	490	522
7	14	500	532

Observations

Yield of oil in case of experiment No.1 is minimum in both the cases, i.e. after 2½ h and 5 h, as compared to other experiments since the quality of grass and steam flow rate is same in experiments 1 and 2, and the only difference being is tight packing and loose packing of grass, thus, it is assumed that this factor has directly contributed to increased oil yield in experiment No. 2. It may be due to the fact, that there is more intimate contact of plant material and steam, due to loose packing, as it offers less resistance to upward flow of oil-steam mixture.

In experiments No. 3 & 4, plant material was more loosely packed than in experiment No.2 (70 kg of plant material instead of 86 kg in experiment No. 2) and grass (plant material) for these experiments was chopped.The steam rate in experiment No.3 was fixed at 15 L/h and that in experiment No. 4 was fixed at 12 L/h. The oil yield in experiment No. 3 as compared to experiment No.4 is much higher due to higher steam injection rate, as all other parameters of experiment No. 3 and 4 are same. So, it is concluded from experiment No. 1 to 4 that loose packing and higher steam injection rate enhances the oil yield in 70 to100 kg scale experiments. The distillation still for these experiments was fitted with two modified steam spargers, as mentioned in experimental section.

Experiments Nos 5, 6 and 7 were actually performed on 1000 kg scale, where plant material was unchopped but loosely packed in the distillation still. From Table 2 for experiments 5, 6 and 7 it is clear, that, the higher rate of steam injection enhances the yield of oil. These results are in conformity with those obtained on smaller pilot scale (i.e. 70-100 kg scale).

Kinetic studies

The kinetic data i.e. oil yield at half hour interval of time for all the seven experiments conducted on pilot scale is presented in Table 3. Figs 2 to 5 show the plots of oil yield versus time for these experiments. Besides this, as mentioned in theory section, fractional yield of oil, u, was also calculated for all these experiments which is defined as follows:

$$u = \frac{\text{oil extracted till time } t}{\text{cummulative amount of oil extracted}}$$

In order to verify the rate model as described in theory section of this paper ln 1/1-u versus t, was plotted for all the seven experiments (Fig. 6). All the above plots show the straight line behaviour.

Normally for 1st order kinetics these straight lines should pass through the origin, but in case of the

Table 3—Experimental data for steam distillation of lemongrass oil from lemongrass

Experiment no.1 (weight of plant material - 100 kg)

Time (min)	Yield (mL)	u	ln (1/1–u)
30	70	0.22	0.25
60	150	0.48	0.65
90	190	0.61	0.94
120	225	0.72	1.27
150	255	0.82	1.71
180	285	0.91	2.40
300	310	—	—

Experiment no.2 (weight of plant material - 86 kg)

Time (min)	Yield (mL)	u	ln (1/1–u)
30	70	0.17	0.186
60	150	0.30	0.356
90	240	0.57	0.840
120	290	0.69	0.170
150	330	0.78	1.500
180	375	0.89	2.200
300	420	—	—

Experiment no.3 (weight of plant material - 70 kg)

Time (min)	Yield (mL)	u	ln (1/1–u)
30	70	0.18	0.198
60	180	0.46	0.615
90	270	0.70	0.203
120	315	0.82	1.713
150	350	0.91	1.407
180	-	-	-
300	385	-	-

Experiment no.4 (weight of plant material - 70 kg)

Time (min)	Yield (mL)	u	ln (1/1–u)
30	100	0.30	0.356
60	200	0.60	0.916
90	235	0.71	0.237
120	270	0.82	1.70
150	300	0.91	-
180	-	-	-
300	330	-	-

Experiment no.5 (weight of plant material -1000 kg)

Time (min)	Yield (mL)	u	ln (1/1–u)
30	1640	0.28	0.33
60	2990	0.52	0.73
90	4730	0.82	1.70
120	5150	0.90	2.30
150	5400	0.94	2.80
180	5540	0.96	3.20
300	5725	-	-

Experiment no.6 (weight of plant material -1000 kg)

Time (min)	Yield (mL)	u	ln (1/1–u)
30	1700	0.32	0.1385
60	3000	0.57	0.844
90	3900	0.74	1.347
120	4500	0.86	1.966
150	4900	0.94	2.813
180	5700	0.97	3.506
300	5225	-	-

Experiment no.7 (weight of plant material - 1000 kg)

Time (min)	Yield (mL)	u	ln (1/1–u)
30	1700	0.32	0.385
60	3000	0.56	0.820
90	4000	0.75	1.38
120	46000	0.86	1.96
150	5000	0.94	2.80
180	5200	0.97	3.50
300	5315	-	-

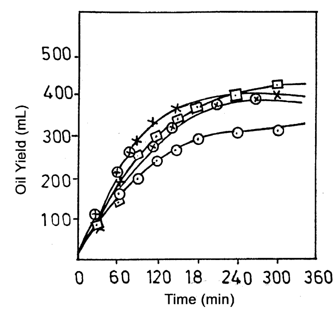

Fig. 2—Oil yield versus time
Expt – 1 ⊙: 100 kg; Expt – 2 ∗: 86 kg; Expt – 3 ×: 70 kg; Expt – 4 ⊗: 70 kg

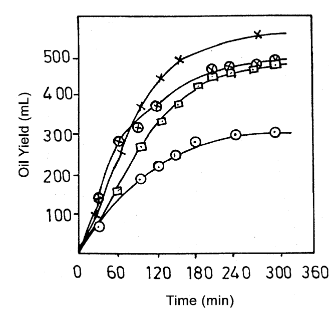

Fig. 3—Oil yield versus time converting to the basis of 100 kg plant material
Expt – 1 ⊙: 100 kg; Expt – 2 □: 100 kg; Expt – 3 ×: 100 kg; Expt – 4 ⊗: 100 kg;

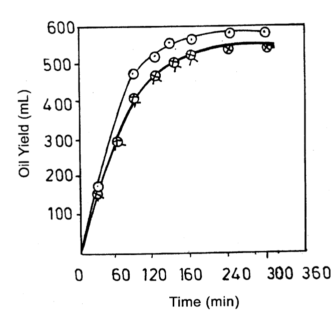

Fig. 4—Oil yield versus time.
Expt – 5 ⊙: 1000 kg; Expt – 6 ×: 1000 kg; Expt – 7 ⊗: 1000 kg

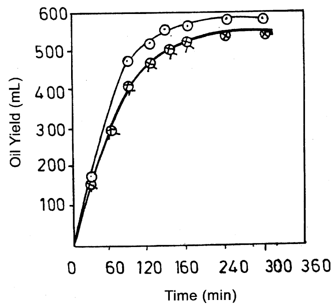

Fig. 5—Oil yield versus time converting to the basis of 100 kg plant material
Expt – 5 ⊙: 100 kg; Expt – 6 ×: 100 kg; Expt – 7 ⊗: 100 kg

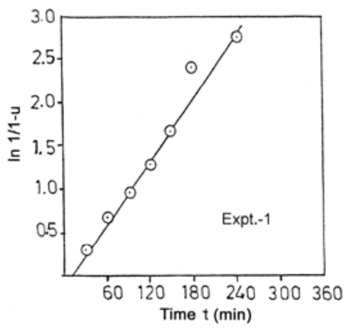

Fig. 6—First order kinetic model

above experiments these straight lines do not pass through the origin, but all have intercept on x-axis i.e. time axis, except in case of experiment No. 4, where the straight line passes through the origin which indicates that the experiment No. 4, perfectly follows the 1st order reaction kinetics. From the experimental conditions of all the experiments it was noted that in case of experiment No. 4 only, the plant material was thoroughly wetted by hot water for 10-15 min inside the distillation still and thereafter the steam was injected whereas, in all other experiments steam was injected directly to the dry plant material.

Thus, the time axis intercept in all the other experiments indicate that oil is not instantly released after injection of steam into the dry grass, but for initial 10-15 min period, the steam wets the dry plant material, which in turn helps in diffusion and osmosis of oil from inside the plant material to the surface of the individual grass strands, and once the oil is on the surface of grass/plant material, it is being carried away by steam. After this initial period of wetting, extraction of oil by steam distillation follows 1st order kinetic behaviour. Experiment No. 4, itself confirms this observation.

Also, oil removed per unit time is directly proportional to oil remaining in the grass. The slope K of the straight lines of the plot ln $1/1-u$ versus t, is on an average equal to 1.6 to 2.

Conclusion

From the pilot plant study of steam distillation process following conclusions are drawn:

(i) The loose packing of plant material in distillation still and higher (optimum) rate of steam injection, enhances the oil yield from the plant material.
(ii) Kinetic studies of the steam distillation process showed that oil is not instantly extracted , if steam is injected to the dry grass, but wetting or swelling of the grass/plant material inside the distillation still is needed for initial 10-15 min for diffusion and osmosis of the oil inside the grass.
(iii) Plot of ln $1/1\text{-}u$ versus t (where u is fractional oil uptake) shows that extraction of oil by steam distillation of grass follows first order kinetics.
(iv) Oil removed per unit time is directly proportional to the oil remaining in the grass.

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