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Optimization & Studies on Extraction of Palma Rosa Oil by Steam Distillation

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Abstract: **Steam Distillation is one of the separation techniques employed to reduce the boiling point of heat sensitive materials and has significant application in chemical industries. Palmarosa oil has commercial value in perfumery, tobacco, soap and pharmaceutical industries. To obtain maximum yield, steam distillation is opted for extraction of oil from Palmarosa grass. Extraction is done on fresh Palmarosa grass and also on shade dried grass for six days. The volume of oil collected as a function of time, throughout the extraction process is recorded. Kinetics of extraction and optimization of yield are reported in this paper. Comparative study of the yield of oil with respect to packing density; Secondary essential oil is recovered from the distillate by solvent extraction and Characterization of Palmarosa oil using Gas Chromatograph with regard to its components,viz. Geraniol, Linalool are also presented in this paper.**

Keywords: **Steam Distillation, Yield, Gas Chromatography, Solvent Extraction.**

I. INTRODUCTION

Steam distillation is a separation technique which is used for separating mixture of components which are heat sensitive by using steam. The principle is based on difference in boiling point by reducing partial pressure of volatile component [1]. Steam distillation has significant application in petroleum refineries, extraction of volatile compounds of plants which were used in perfumery and flavouring industries. . Palmarosa is a sweet smelling grass grows in tropical climates and its origin lies in India. Scientific name of Palmarosa is Cymbopogan Martini [2]. Palmarosa grass is extracted by steam distillation. The distillated content has two parts, essential oil and distillation water. The distillate is subjected to solvent extraction where the secondary essential oil is extracted. The components of Palmarosa oil is classified based on Chromatograms of Gas- Chromatography.

II. EXTRACTION OF ESSENTIAL OIL

Extraction of oil from Palmarosa was carried out by a stream-distillation using autoclave apparatus. the Distillation apparatus made up of S.S, consists of two vessels, inner vessel (diameter 13 inch, height 21 inch) and outer vessel (diameter 14 inch, height 27 inch).Outer vessel contains heating coil, inner vessel contains holes at the bottom to pass steam through it as shown in figure 2. Pressure gauge and safety valve is provided at the top to control the pressure inside the apparatus. Autoclave and condensing section are connected by steel tube. Water is used as coolant in condensing section. The separation section consists of burette as the separator. Based on density difference of oil and water gets separated. Fresh Palmarosa grass was collected and shade dried. The inner vessel was loaded with calculated quantity of Palmarosa and outer vessel was filled with 10 lit of demineralized water. Autoclave was operated at a pressure of 0.35 to 0.4 kgf/cm2 [3] resulting in rupture of cell walls of leaves due to generated steam. The vapors were passed in to the condenser and the distillate is collected in the burette. Distillation was continued until no further increase in volume of oil.

III. RECOVERY OF SECONDARY ESSENTIAL OIL

Recovery of secondary essential oil was carried out by solvent extraction. N-heptane is used as solvent which is immiscible with aqueous layer and has an appreciable difference in boiling point with essential oil. Multiple solvent extractions is used for better recovery of oil [4]. Total distillation water of run 1 is divided in to samples of 200 ml each.

Each sample is taken in a glass stoppered bottle and 10 ml of n-heptane is added. Stoppered bottle is shaken for 20 minutes and then n-heptane is separated from aqueous layer using a separating funnel. The above process is repeated for 8 times with preceding separated aqueous layer. The separated organic layer i.e. mixture of n-heptane and oil from each sample is collected in to distillation flask. Simple distillation is carried out in heating mantle which is run at 10 volts. The n-heptane vapors are formed and oil is left out in a distillation flask.

TABLE I Yield of oil for collected for each run Parameters

	Parameters					
Run no	*No of days dried*	*Weight of shaded grass (KG)*	*Oil (ml)*	*Steam consumed (ml)*	*Steam consumed per ml of oil*	*Oil collected per Kg of grass*
1	1	2.97	10.1	3500	346	3.4
2	2	1.5	6.5	2210	340	4.33
3	3	1.5	9.3	2883	310	6.2
4	4	1.5	11.25	2398	212	7.5
5	5	1.5	10.2	2255	221	6.8
6	6	1.27	6.1	2725	446	4.80

IV. ANALYSIS OF ESSEENTIAL OIL BY GAS CHROMATOGRAPHY:

The gas chromatography works on the principle of adsorption, where PEGA (Poly Ethylene Glycol Adipate) is used as the adsorbent. It consists of three gases. They are helium or nitrogen, hydrogen and zero gas. Helium gas is called as carrier gas because it is used to carry the vapor feed and nitrogen gas is used to carry the flow rate. Zero gas is nothing but moisture free air. It contains mainly of nitrogen and oxygen. Nitrogen creates inert atmosphere and oxygen helps to burn hydrogen. On burning hydrogen it will produce flame, due to the heating effect produced by flame liquid feed will be transformed into vapor phase. This vapor moves along the column by using helium which is a carrier gas. This column was coated with PEGA. Different molecular weight components were adsorbed on PEGA at different time intervals. By comparing these graphs with the standard graphs the name of the component and also the composition of the component can be identified.

V. RESULTS & DISCUSSION

A. Analysis of oil from steam distillation):

1) Yield: For each experimental run dried leaves were taken and subjected to steam distillation. The collection of oil for every 20 min are recorded and given in table 2. The oil collected and steam consumed for each run is shown in table 1.

TABLE II Weight loss with respect to days old

DAY	*Weight of grass(KG)*
0	1
1	0.957
2	0.905
3	0.849
4	0.787
5	0.716
6	0.641

2) Kinetics theory: Extraction of essential oil from Palmarosa by steam distillation is a rate process. Also oil removed per unit time is directly proportional to oil remaining in the leaves. The rate equation is given by

Oil in leaves Extracted oil

From Rate law

-d [Oil in leaves]/ dt = k [Oil in leaves]

d[Extracted oil]/ dt = k[Oil in leaves]

Let 'a' = initial volume of oil in leaves.

'x' = volume of oil extracted in leaves at time 't'

So, a-x is the volume of oil present in leaves after time t

d(x)/dt = k(a-x)

By integrating above equation on both sides

$=>0\int x\, dx/(a-x) = k0\int t\, dt$

=> [-ln(a-x)]0x = k[t]0t

=> ln(a) –ln(a-x) = kt

kt = ln(a/a-x)

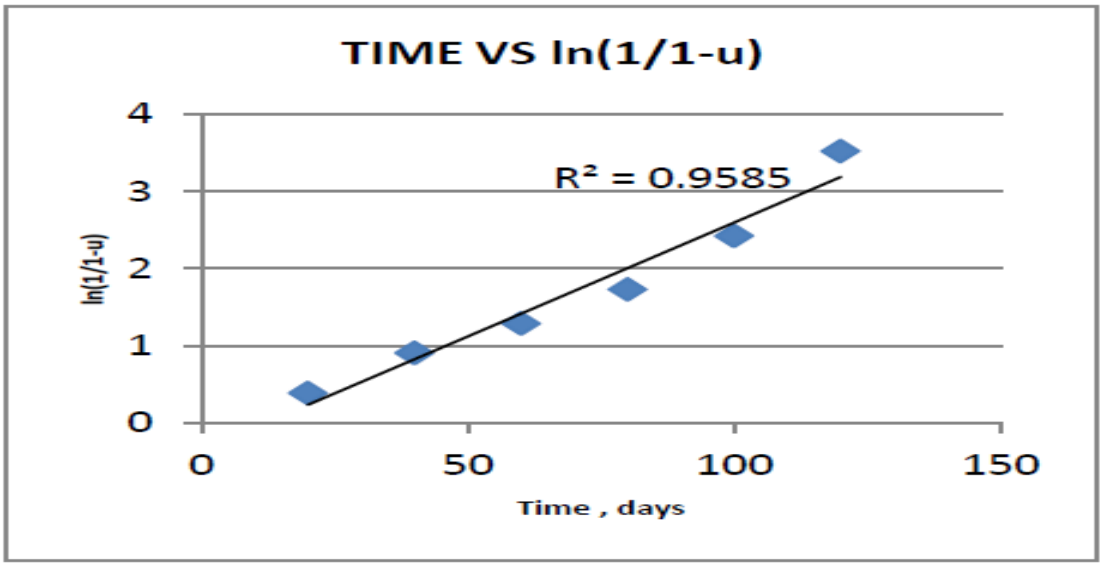
kt = ln(1/1-v)

t = time of steam distillation

v = fraction of oil extracted = x/a

TABLE III Fractional yield of oil with time in day 5

	Parameters				
s.no	*time*	*Oil collected(ml)*	*V*	*ln(1/(1-v))*	*K*
1	20	3.3	0.3235	0.3909	0.0195
2	40	6.1	0.5980	0.9114	0.0228
3	60	7.4	0.7255	1.2927	0.0215
4	80	8.4	0.8235	1.7346	0.0217
5	100	9.3	0.9118	2.4277	0.0243
6	120	9.9	0.9706	3.5263	0.0294



If the above model holds good then the plot of ln(1/1-u) Vs t will produce a straight line. The slope of the straight line obtained is 'k'.

For day 5 a = 10.2 ml

From table 2 it is observed that last column 'k' is almost constant so extraction of oil through steam distillation of Palmarosa follows first order kinetics.

3) Factors affecting yield: From table 3 it is observed that weight loss in grass is a function of time. Day to day comparison of oil with respect to weight loss provides factors affecting the yield. Day 2 ~ Day 1 represents the yield of day 2 follows yield of day 1 on the basis of day 1 weight, assuming all other factors constant. Dryness factor affect the yield as the time passes due to loss of moisture along with small amount of oil. By observing the day 2 ~ day 1 row

TABLE IV

	Oil (ml)	
Day to day comparison	Theoretical estimation	Experimental observation
Day 2 ~ Day 1	5.39	6.4
Day 5 ~ Day 1	6.8	10.2
Day 6 ~ Day 1	6.447	6.1
Day 5 ~ Day 2	8.215	10.2
Day 6 ~ Day 2	7.769	6.1
Day 6 ~ Day 5	9.64	6.1

Estimation of theoretical yields with respect to weight loss experimental yield of oil is not equal to theoretically estimated value i.e. 5.39 < 6.4 it clearly seems that apart from dryness factor there is another factor which affects the yield content. This factor is identified as packing density by varying the density of grass accommodated in autoclave between day1 and day2.

4) *Optimization:*

Since there is difference between theoretical estimation and experimental observation we presumed that dryness, packing density are the factors. To determine that they are significant factors affecting the yield ANOVA had been done and found that the two factors are affecting. Response equation has been evaluated with the response as the yield and factors as dryness, packing density and found the equation….. '-' represents low level and '+' represents high level so dryness should be low and packing density can be extended to higher weights depending on capacity of autoclave. So for the required yield and on the given day (dryness) we get required packing density which is optimum.

ANOVA – TABLE V

Source	SS	DOF	MS	F_0
Dryness	49.884	5	9.97	16.794
Packing Density	3.26	1	3.26	5.49
Error	2.9685	5	0.5937	*
Total	56.112	11	5.101	*

Regression Analysis: yield versus dryness, packing density

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The regression equation is
yield = 2.35 - 1.36 dryness + 4.53 packing density

Predictor          Coef  SE Coef      T      P
Constant          2.350    9.921   0.24  0.818
dryness          -1.362    6.457  -0.21  0.838
packing density   4.529    6.068   0.75  0.474
```

Analysis of Variance

Source	DF	SS	MS	F	P
Regression	2	3.515	1.758	0.30	0.747
Residual Error	9	52.592	5.844		
Total	11	56.107			

B. Analysis of secondary essential oil:

The oil recovered from 3500 ml of distillation water of day 1 is 5.25 ml. The oil recovered up to 51.9 % of oil obtained from distillation by solvent extraction. Solvent recovered is the percentage of ratio of solvent obtained after extraction to total solvent used for extraction.

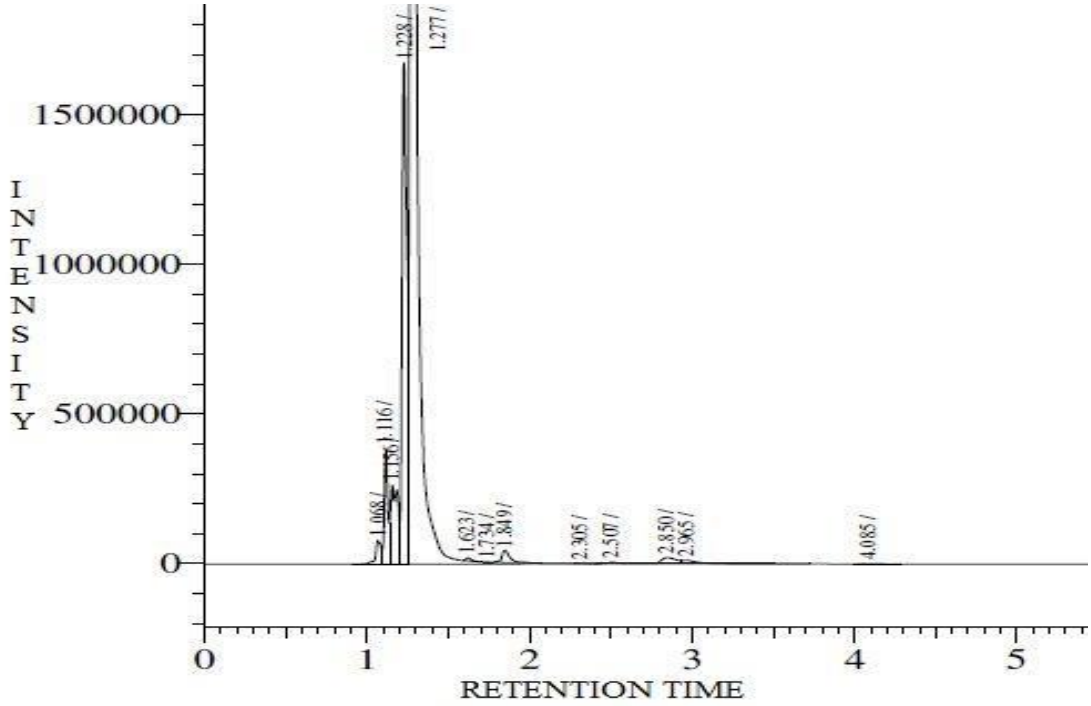
Fig2. Internal Cross section of autoclave

% Solvent loss = 100 – % solvent recovered

Solvent loss is about 8.57%

C. Chemical composition of Palma rosa oil:

From the figure the major components present in the essential oil is identified as Geraniol ($C_{10}H_{18}O$ [6]) – [5], Geranyl acetate ($C_{12}H_{20}O_2$ [6]) [5], Linalool ($C_{10}H_{18}O_2$ [6]) [5].



VI. CONCLUSIONS

Steam distillation gives an edge in extracting heat sensitive oils, but due to the dissolution of oil in distillation water at high temperature it is not completely efficient. Economical day for extraction of oil in terms of both yield obtained and steam consumed is 5th day. Steam distillation of Palmarosa oil follows first order kinetics. Dryness factor and packing density are the factors affecting the extraction of oil. Recovery of oil from solvent extraction is accounted for 51% of the total oil yield from steam distillation. The solvent loss in this process is 8.7%.

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