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A comparative study on distillation methods for oil content, chemical composition, yield and economics in *Eupatorium adenophorum*

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Abstract
An experiment was conducted to assess oil content (%), chemical composition, oil yield and economics in An experiment was conducted to assess oil content (%), oil yield (kg/10q) and economic returns (Rs/10 q) in *Eupatorium adenophorum* via two different distillation methods: Hydro-steam and Steam distillation. Maximum oil content (0.23%) was achieved by Hydro-steam distillation method as compared to Steam distillation (0.18%). Further, Hydro-steam distillation (2.27 kg/10q) exceeded Steam distillation (1.82 kg/10q) in term of oil yield. The economic returns were also significantly higher for Hydro-steam distillations (Rs 10696.60/ 10 q) as compared to steam distillation (Rs.7376.40/10 q). Experimental finding reveals Hydro-steam distillation to be significantly better than Steam distillation in terms of oil content (%), oil yield (kg/10 q) and economic returns (Rs/ 10q). The suitability and feasibility of Hydro-steam distillation were found to be more appropriate under field/ farm conditions in comparison to Steam distillation. Further, the chemical compositions of essential oil via two different methods were largely similar. GC-MS analysis of the essential oil showed that the presence of p-cymene (12.87%), α-Phellandrene (9.35%), Cis-Cadin-4-en-7-ol (7.46%), Bornyl acetate (5.97%), Camphene (5.75%), γ-patchoulene (5.18%), δ-2-carene (4.26%) as major constituents in steam distillation and p-cymene (11.31%), α -Phellandrene (7.92%), Cis-Cadin-4-en-7-ol (8.52%), Bornyl acetate (5.78%), Camphene (5.08%), γ-patchoulene (4.40%), δ-2-carene (4.00%) in Hydro-steam distillation respectively.

Keywords: Eupatorium, distillation, hydro-steam, chemical composition

1. Introduction
Eupatorium adenophorum Spreng syn. (*Ageratina adenophora* King and Robinson) belong to the family Asteraceae is a perennial herbaceous shrub, having woody rootstock and numerous upright branching stems is native to Central America (Mexico). Commonly known as Crofton weed, Banmara (Nepal), Kala Bansa (Uttarakhand) is highly invasive and fast-growing weed. It comprises mainly 600 species [1]. This weed is widely distributed from tropical to the temperate regions mainly all over the world in ravine slopes and grassy localities, up to 2200 meters above mean sea level [2]. The weed is characterized by its great reproductive capacity, broad tolerance, and enormous dispersal ability to various environmental conditions [3]. The weed is reported to possess diverse medicinal properties and uses in traditional medicines. *E. adenophorum* is used in folk medicine since ancient times due to its antiseptic, allelopathic, antioxidant, antifungal, analgesic properties. It also posses properties like blood coagulant, antipyretic, acaricidal, anti-nematodal activity and suppose to be enhancer of phenobarbitone that induced sleep [4] It is also used to treat jaundice, fever, insomnia, and ulcers [5]. The essential oil of the E. adenophorum is also known to possess insect repellent activity [6]. Due to the significant essential oil content in its aerial parts, it is considered as a valuable raw material for perfumery industry [5] & (Sharma *et al*., 1998 [7]; Adhikari and Kraus, 1994). The essential oil of *E. adenophorum* is extracted from aerial part of the plant has been investigate for its chemical composition [8-10]. Mainly oil extractions of Aromatic plants are done by the various distillation processes that include water distillation (hydro distillation), hydro-steam distillation, and steam distillation. In view of its important medicinal properties a study was conducted to emphases on the cost-effective distillation technique for maximizing the essential oil extraction/yield of *E. adenophorum*.

2. Material and Method

2.1 Sample collection and Extraction of essential oil

The fresh aerial part of *E. adenophorum* Plants [10 quintals] were collected from Sahiya (Chakrata), Dehradun (30°35'47.41" N, 77°52'38.21" E) and 1035 m amsl). The collected materials were chopped, and finally weighed before proceeding for distillation via two different distillation processes. total portion of plant above ground was harvested and weighed. After the plant sample collection, the plant materials were freshly chopped and then passed through following distillation methods

2.1.1 Hydro-Steam Distillation method

10kg freshly chopped material was loaded in 20kg pilot distillation unitplant chamber (cylindrical) keptep above the water level. A perforated grid (or plate) was placed fashioned to ensure so that the plant materials were as raised above the water level. The water is boiled at 100 C below the firewood and "wet" steam generated by boiling the water passed through the plant material vaporizing the essential oil. The complete distillation process extraction of essential oil took 4.30 hrs. The steam along with the water and oil were collected in the tube (Figure: 1). The traces of water in the essential oil were removed by adding anhydrous sodium sulfate (Na2SO4) and stored at 4 C for further analysis. Finally the essential oil yield was calculated using below formula

The yield of essential oil (%) = Essential oil obtained / Total raw material) X 100

2.1.2 Steam Distillation method

10kg fresh chopped material (10 kgs) was loaded in steam distillation units (50 kg capacity) 50kg plant chamber (cylindrical). Steam was generated using steam boiler (separate unit) at temperature of above 100 ºC and "dry" steam was channeled into the 50kg plant chamber (separate unit) containing the chopped material *E. adenophorum* aerial parts. The complete distillation process took 4.30 hrs. The water steam along with the and oil were collected in the separator (Figure: 2). The temperature of the steam was kept high enough (above 100 C) but adequately maintained below the extreme levels by adjusting temperature control knobs so that the chemical compositions of essentials oils were not affected .to vaporize the oil present, but not so high that destroys the plants or burns the essential oil. The traces of water in the essential oil were removed by adding anhydrous sodium sulfate (Na2SO4) and stored at 4 C for the use of analysis. Finally the essential oil yield was calculated using below formula:

The yield of essential oil (%) = Essential oil obtained / Total raw material) X 100

2.2 Chemical composition

2.2.1 Gas-Chromatography

Gas chromatography (GC) analyses of the essential oils were carried out by Agilent (model 6890N) gas chromatograph equipped with Flame Ionization Detector (FID) using N2 as the carrier gas. The column was HP-5 fused silica capillary column (30 m × 0.32 mm, 0.25µm film thickness) and temperature program was used as follows: initial temperature of 60 °C (hold: 2 minutes) programmed at a rate of 3 °C/minute to a final temperature of 220 °C (hold: 5 minutes). Temperatures of the injector and FID were maintained at 210 °C and 250 °C, respectively. The sample injection volume was 0.2µL. (neat essential oil)

2.2.2 Gas-Chromatography and Mass-Spectrometry

The gas chromatography-mass spectrometry (GC/ MS) analyses of the oils were performed with a Perkin Elmer Clarus 500 gas chromatograph equipped with a split/split less injector (split ratio 50:1) data handling system. The column was Rtx-5 capillary columns (60 m × 0.32 mm, 0.25µm film thickness). Helium (He) was the carrier gas at a flow rate 1.0mL/min. The mass spectra were recorded over 40–350amu that revealed the total ion current (TIC) chromatograms, using an ionizing voltage of 70eV. The identification of compound was performed by MS library search with Wiley and NIST and compare with MS literature search (11]) and the quantification was done by peak area percentage of GC compound was performed by MS library search with Wiley and NIST and compare with MS literature search [(Adams R.P, 2009).

3. Statistical Analysis

The data was collected on parameters viz. oil content (%) and oil yield (kg/10q) were worked out. Further economics of oil yield via. two different methods: Hydro-steam distillation and Steam distillation were worked out to find out economic returns (Rs./10q). All data were subjected to student's t-test for statistical analysis.

4. Results & Discussion

4.1 Hydro-steam and Steam Distillation

The experiments revealed that the essential oil content, oil yield, and economics of *E. adenophorum* differed significantly due to different distillation methods. By Hydro-steam distillation method essential oil content, oil yield & economic return are found to be (0.23%), (2.27kg/10q) and (Rs 10696.9/10q) respectively & Steam distillation method essential oil content, oil yield & net return are found to be (0.18%), (1.82kg/10q) and (Rs 7376.4/10q) respectively. Under student's t-test (%) oil content, oil yield and economic returns differ significantly at p=0.05 (Table: 1). Therefore, from the experimental finding it may be concluded that Hydro-steam distillation was the better in comparison to Steam distillation method for maximizing essential oil content, oil yield and economics return. Similar results were also reported by [12] with maximum essential oil yield (1.5%) under Hydro-distillation method, while minimum yield under Steam distillation method (1.1%). Another study also confirms the higher yield of essential oil through Hydro distillation than the Steam distillation [13]. Previous studies also reveal that there were statistically significant differences found for interaction between the concentration of the main oil constituents and the method of distillation [14]. The essential oil extraction by Hydro-steam distillation method also proved suitable and feasible under field and natural conditions. Further it was also observed that the Steam distillation required more precisions in operation which is not likely feasible under field conditions so the Hydro-steam distillation method may be used alternatively. Hydro-steam distillation can also be modified to get maximum output as well as maximum recovery by using various distillers [15].

Chemical composition of essential oils

The chemical composition of the essential oils of *E. adenophorum* by Steam distillation is presented as Table 2. GC-MS analysis of the essential oil of *E. adenophorum* leaves showed that the presence of p-cymene (12.87%), α-

Phellandrene (9.35%), Cis-Cadin- 4-en-7-ol (7.46%), Bornyl acetate (5.97%), Camphene (5.75%), γ-patchoulene (5.18%), δ-2- carene (4.26%) as major constituents (Table 2). The earlier workers had reported the major constituents in essential oil of the aerial parts of *E. adenophorum* were p-cymene (11.6%), α-phellandrene (5.7%), γ-curcumene (5.0%), δ-2-carene (5.0%), and camphene (4.8%), and endo-bornyl acetate (4.4%), [10] .

Further, the chemical composition of the essential oils of *E. adenophorum* by Hydro- steam distillation revealed the presence of p-cymene (11.31%), α-Phellandrene (7.92%), Cis-Cadin-4-en-7-ol (8.52%), Bornyl acetate (5.78%), Camphene (5.08%), γ-patchoulene (4.40%), δ-2-carene (4.00%) as major constituents (Table 2).

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a. Steps in Hydro-steam distillation
b. Steps in Steam distillation

Table 1: Average mean values for different parameters *via*. Different distillation methods in *E. adenophorum*

Treatment	Oil content (%)	Oil yield kg/10 q	Net return Rs./10q
Hydro-steam distillation	0.23a	2.27a	10696.9a
Steam distillation	0.18b	1.82b	7376.4b

The value of p=0.05 was considered statistically significant. All results refer to mean±SD. In each column, different letters mean significant differences between the samples mean.

Table 2: Chemical composition of *E. adenophorum* (Steam & Hydro-Steam Distillation)

S. No	RT	Component	RI^{exp}	RI^{lit}	(%) (Steam Dist.)	(%) (Hydro- Steam Dist.)
1.	11.329	camphene	950	946	5.75	5.08
2.	13.330	δ-2-carene	1002	1001	4.26	4.00
3.	13.520	α-Phellandrene	1005	1002	9.35	7.92
4.	14.400	p-cymene	1026	1020	12.87	11.31
5.	14.520	DL-limonene	1029	1024	0.99	0.88
6.	15.230	β- ocimene	1045	1044	0.28	0.22
7.	17.610	Linalool	1098	1095	0.13	0.27
8.	20.821	Borneol	1170	1165	0.28	0.54
9.	21.971	P-mentha-1,5-dien-8-ol	1175	1172	0.14	0.62
10.	23.452	Thymol methyl et her	1235	1232	0.14	0.13
11.	26.122	Bornyl acetate	1287	1284	5.97	5.78
12.	31.813	Trans-caryophyllene	1419	1417	1.47	1.25
13.	32.303	α-Bergamotene	1435	1432	1.08	0.90
14.	33.074	β- farnesene	1443	1440	3.32	2.67
15.	34.104	β- Himachalene	1450	1449	1.40	1.09
16.	34.244	Epizonarene	1504	1501	2.17	1.88
17.	34.374	γ-patchoulene	1497	1502	5.18	4.40
18.	34.964	Trans-β guaiene	1504	1503	1.39	1.21
19.	35.274	α-farnesene	1512	1505	4.81	4.00
20.	35.554	β -curcumene	1515	1514	0.44	0.36

21.	35.874	β -sesquiphellandrene	1519	1521	2.44	2.02
22.	36.184	α -bisabolene	1526	1529	1.18	1.16
23.	37.324	Elemol	1548	1548	0.31	0.36
24.	37.534	Nerolidol	1557	1561	0.12	0.14
25.	38.455	Spathulenol	1579	1577	0.29	0.39
26.	40.355	Cis-Cadin-4-en-7-ol	1639	1637	7.46	8.52
27.	40.515	<epi-α>cadinol	1638	1640	0.59	0.71
28.	41.035	α-cadinol	1655	1652	0.44	0.55
29.	41.505	<epi-β>Bisabolol	1672	1670	0.27	0.33
30.	42.065	<epi-α>Bisabolol	1686	1685	2.73	4.04
31.	42.705	Widdrenal	1704	1708	2.08	2.91

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