Solar Thermal Steam Distillation for Essential Oil Production

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Abstract

This work reports the utilization of solar thermal energy to produce Eucalyptus essential oil, reducing the need for conventional energy sources and minimizing the environmental impact. The focus was on optimizing the oil extraction by conventional steam distillation using Response Surface Methodology (RSM) and applying the optimized result to a Scheffler concentrator-driven solar steam distillation set-up. The optimum operating conditions were: leaf size: 0.02 m and 0.02 m, extraction temperature: 97.76 ℃ and 96.97 ^{0}C, solid/solvent ratio: 0.61 and 0.48, and extraction time: 206 min and 209 min for normal and sundry eucalyptus leaves respectively. Notably, the extraction temperature was identified as the most influential parameter. By applying RSM input parameters range to the experiments, the major components of the oil obtained were 1-8 cineole (57.53 to 78.45 %) and α-Pinene (15.27 to 27.83 %) with normal leaves and 1-8 cineole (50.52 to 62.82 %) and α-Pinene (20.05 to 27.54 %) with sundry leaves. Over the course of the outdoor experiments, the small capacity solar boiler operating through the 2.5m^2 Scheffler concentrator achieved an average efficiency of 27 % and produced steam at a rate of 2 kg/h at an average solar insolation of 700 W/m^2. However, conventional steam distillation surpassed the solar process in terms of oil yield (0.68 % vs. 0.1 % and 0.36 % vs. 0.08 %) because of the stable processing temperatures. Major component, (% 1-8 cineole) present inside the eucalyptus oil, come up with nearer results (62.76 % vs. 61.44 % and 57.77 % vs. 54.96 %) with conventional and solar distillation for normal and sundry leaves respectively. The effort demonstrates a Eucalyptus essential oil extraction methodology which is well-suited for sunny regions and small businesses targeting niche markets.

1.Brief description on the state of the art of the research topic

In contrast to solar thermal energy, which provides a more environmentally friendly alternative, essential oil production in aromatherapy and medicine historically and conventionally relies on polluting energy sources. The conventional methods for extracting volatile oils from aromatic plants, use processes including solvent extraction, solvent distillation, cold pressing, and maceration, each of which is best suited for specific plants and results. Pure essential oils can be extracted profitably from flowers and other plant parts with volatile components via steam distillation. With the application of steam distillation, the composition and nature of the extracted oil not changed, that is why market accepted steam distilled essential oil as the normal and natural oil [1]. The concentrated extracts are widely used in a variety of industries and are considered of great importance as export products for most developing countries. According to WHO, 21,000 plant species have therapeutic potential [2]. These plants contribute to fragrances, lotions, and skincare products in the cosmetics industry by providing aromatic and medicinal properties. Essential oils are widely used in aromatherapy for their aromatic and healing properties. In Eucalyptus viminalis Labill. 1, 8-cineole was 56.43 %, and α-Pinene was 3.91 % [3]. In Eucalyptus leaves the % of 1,8-cineol was 49.07 to 83.59 % and α-Pinene was 1.27 to 26.35 % as observed by Sebei et al. 2015 [4]. Eucalyptus oil's quality is determined by elements including root structure and water movement; its primary constituents are 1-8 cineole and α-Pinene. Notably, 1-8 cineole holds an anti-inflammatory role and inhibits tumour necrosis factor-alpha [5,6]. In the context of solar energy applications for extracting volatile oils from medicinal and aromatic plants, various studies have been conducted to develop solar distillation systems and evaluate their performance. The system achieved focus receiver temperatures of 300-400 °C within a solar radiation range of 700-800 W/m^2. The recorded power and system efficiency values were 1.55 kW and 33.21%, respectively, at a solar radiation level of 739 W/m^2 [7]. According to surveys, the majority of processes are carried out at or below 180 °C. Due to their automatic tracking mechanism and parabolic shape, solar concentrators like Scheffler concentrators are in demand for low to medium-temperature applications. Scheffler concentrators are also in demand because of two axis tracking mechanisms. Which save the non-renewable energy by placing an alternative renewable source of energy. This is low cost technology as its construction required only commonly available materials and tools and/or economically available elements and maintenance can also be conducted locally at less expense. The concentration section is of parabolic in geometry. The optical characteristic of a parabola is to reflect incoming rays parallel to its vertical axis towards single focal point.

- The parabola can be tracked automatically with very little energy.
- The parabola's axis of rotation is parallel to the earth's rotation axis, so the inclination of the parabola varies according to the latitude of its location.
- Low operational cost because no need for fuel, whose price is uncertain in future.
- The system not only minimise the losses of volatile essence of the plant material but also reduce the dependence on the conventional fossil fuels in the domestic sector. [8,9,10,11,12].

Response Surface Methodology (RSM) is employed to optimize the distillation process by analysing relationships between variables and recommending optimal conditions [13]. It is used to describe and optimize the interactions between various input factors and the process's output response. Regression analysis is used to develop a mathematical model that explains the relationship between the input factors and the response after a series of experiments are conducted.

2. Definition of the Problem

To explore environment friendly methodology for extraction of essential oil, to design and fabricate the conventional and solar steam distillation assembly. Using RSM, to determine optimized input (leaf size, extraction temperature, solid/solvent ratio, and extraction time) and output parameters (% oil yield, % 1

– 8 cineole and % α-Pinene) to extract Eucalyptus essential oil by conventional steam distillation from normal and sundry Eucalyptus leaves. To Compare optimized output parameters for optimized input parameters by RSM, obtained from conventional steam distillation with solar steam distillation for normal and sundry eucalyptus essential oil.

3. Objective and Scope of work

Objective of the work is to access environment friendly technology with the application of Scheffler solar concentrator, to extract essential oil. Can be summarised as follows: -

- To select plant material and extraction methodology.
- To Design and fabricate steam distillation experimental setup unit and Scheffler solar concentrator.
- Apply RSM to find out the optimum output parameters (% oil yield, % 1 – 8 cineole and % α-Pinene) by applying input parameters (leaf size, extraction temperature, solid/solvent ratio, and extraction time) for the essential oil, extracted from normal and sundry eucalyptus leaves. To Perform experiments according to given run sheet of RSM with conventional steam distillation and then apply optimized results to solar steam distillation.
- To Characterize output parameters % 1 – 8 cineole and % α-Pinene by GC – MS analysis.
- To Compare the results obtained from conventional and solar steam distillation for normal and sundry eucalyptus leaves.

4. Original contribution by the thesis

- Designed and developed conventional and solar steam distillation system to extract eucalyptus essential oil. As shown in Figure. 1 instead of fossil energy, introduced 2.5 m^2 Scheffler solar concentrator, to extract essential oil with the application of steam distillation. Optimized results from conventional and solar steam distillation with RSM, obtained according to Table no. 5

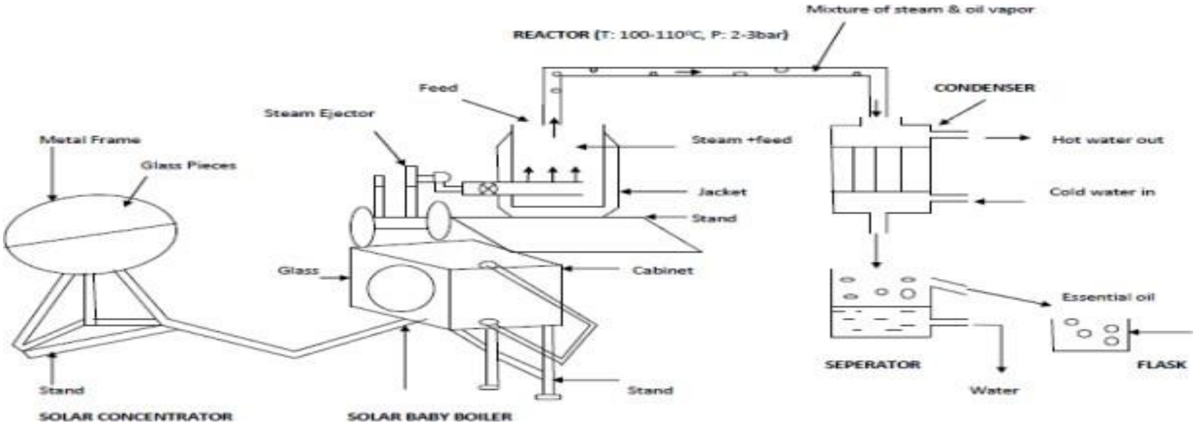


Figure 1. To extract Essential oil by solar thermal steam distillation

- According to the run-sheet of RSM, eucalyptus leaves were prepared for the 27 runs of normal leaves and 27 runs of sundry leaves.
- With the input variables as shown in Table 2.
- Obtained output variables from 27 runs in the range of;

	% oil yield	% 1-8 cineole	% α-Pinene
Normal leaves:	0.3 to 0.75 %	57.53 to 78.45 %	15.27 to 27.83 %
Sundry leaves:	0.18 to 0.39 %	50.52 to 62.82 %	20.05 to 27.54 %

The essential oil yields and extraction processes, mostly operated by solar thermal energy for the various plants are given in Table 1. Various research, including the present work, has reported different Eucalyptus yields. It showing that depending on the plant species, technique of extraction, and working conditions, a given batch of essential oil may have different % yield values. Apart from these, the effectiveness of the distillation setup and the analytical techniques used for yield determination are some of the elements that can be responsible for the variations in yield.

Table 1. Comparison of essential oil yields and extraction processes for various plants

Feedstock	Extraction method	Operating condition	%Y	Ref.
Eucalyptus	Solar distillation	-	-	[14]
Cumin	Solar distillation	3 kWh, 23-250 °C	4.06 ml	[15]
Eucalyptus Peppermint Pinus	Hybrid solar distillation		0.59% w/w 0.40% w/w 0.31% w/w	[16]
Fennel seed	Solar distillation	7.5h, 1 bar	2% w/w	[17]
Lemon grass	Solar distillation		0.358-1.533%	[18]
Lavender	Solar distillation	60 min	0.5-6.8% w/w	[19]
Eucalyptus	Steam & Solar distillation	206. 89 min, 97.76 °C	0.68% w/w 0.1% w/w	(Present study)

5. Methodology of research:

5.1 System description

5.1.1 Conventional Electric-Powered Steam Distillation System

The conventional electric-powered steam distillation system comprised an electric boiler, extraction column, condenser, and oil-water separator. The photograph and schematic diagram of the unit are shown in Figure. 2 (a) and (b). The electric boiler had a power rating of 2000 W and was used to heat 40 liters of water. It serves as the source of steam for the distillation process. The extractor column could store 5 kg of raw material and 25 l of water. It was made from food-grade stainless steel having an opening of 0.012 m for drainage. A 0.003 m nozzle was installed inside the extractor for sparging. At the bottom of the extractor column, there was a 0.005 m perforated stainless-steel wire mesh to hold the raw material during the steam distillation process. The separator was a glass tube with a capacity of 100 ml. It was used to separate the extracted oil from the condensate water. The condenser was a shell and tube type made from stainless steel. It was used to condense the steam vapor back into liquid form during the distillation process. The condenser was sealed to prevent any leaks using gaskets and silicone sealants.

Figure 2. (a) Conventional electric-powered steam distillation system

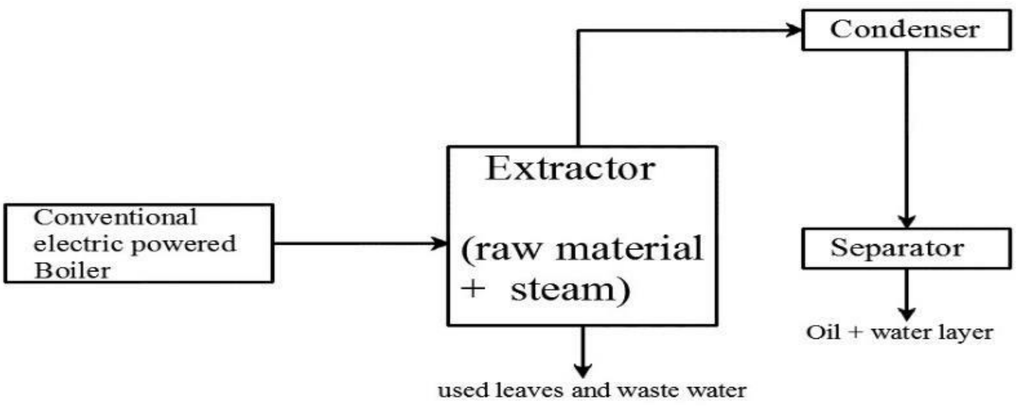


Figure 2. (b) Schematic diagram of Conventional (Electric – powered) steam distillation system

5.1.2 Solar Steam Distillation System

The solar steam distillation system consisted of a solar baby boiler with a capacity of 4 litres. It was designed to collect solar energy efficiently, which is then used to generate steam for the distillation process. The Scheffler concentrator was mounted on a two-axis tracking system that allowed it to follow the movement of the sun throughout the day. This tracking system ensured that the concentrator remained oriented towards the sun, maximizing the amount of sunlight it captured. As the concentrator tracked the sun, it reflected and concentrated sunlight onto the solar boiler, which was placed at the focal point. The Scheffler concentrator had an aperture area of 2.5 m^2. The solar steam distillation system was integrated with the distillation assembly as in Figure 2 (a) to extract the oil. The photograph and schematic diagram of the integrated system are shown in Figure 3 (a) and (b).

Figure 3. (a) Solar steam distillation system

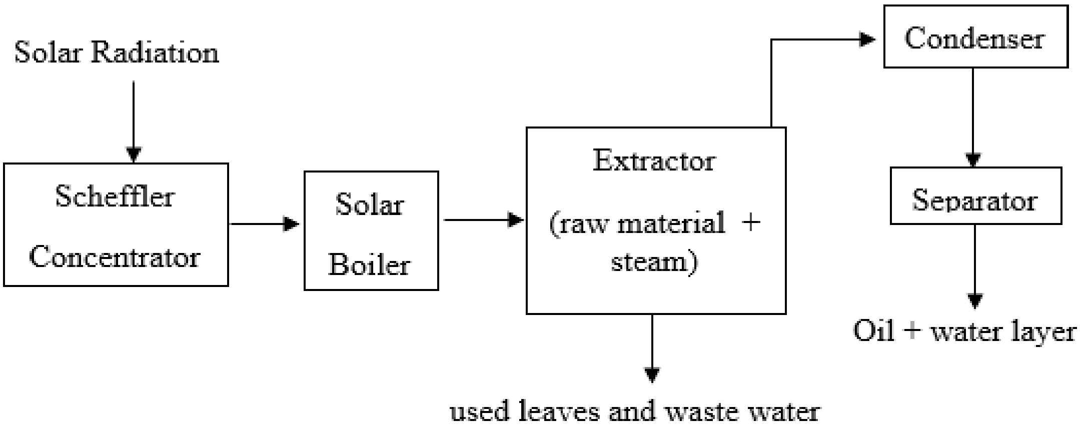


Figure 3. (b) Schematic diagram of solar steam distillation

5.2 Experimental procedure

Fresh Eucalyptus leaves were collected from Ambaji (24° 33' N, 72°.850 E') in the northern part of Gujarat, a state in western India. The first step is to thoroughly wash the leaves with water to remove any dirt, debris, or contaminants from the surface of the leaves, ensuring that the experiments are conducted on clean and pure samples. The leaves were first thoroughly washed with water and, the cleaned leaves were then kept at room temperature for 2-3 hours air-drying them partially before further processing. The cleaned leaves were then sorted according to their size as in Figure 4. The reason for this sorting was to ensure uniformity and consistency in the experimental setup, as leaves of different sizes have varying characteristics that could affect the results. The organized leaves of the same size were then used for the experiments.

Figure 4. Eucalyptus leaves with normal and different size

5.2.1 Experiments with Conventional Electric-Powered Steam Distillation System

- According to the run-sheet of RSM, Eucalyptus leaves were prepared for the 27 runs. The independent variables (input parameters) were:
- Leaf Size: Ranging from 0.02 to 0.05 meters.
- Extraction Temperature: Ranging from 92 to 98 °C.

- Solid/Solvent Ratio: Ranging from 0.4545 to 0.6250.
- Extraction Time: Ranging from 150 to 210 minutes.

Each run used 2.5 kg of Eucalyptus leaves. The leaves were kept on the wire mesh installed at the bottom of the extractor. The steam from the boiler was introduced to the extractor through a sparger. The sparger ensured the equal distribution of steam on leaves throughout the experimental run. The first drop of oil was collected after an hour and the quantity of the collected oil was recorded at regular intervals of time. The water vapors, along with the volatile essence of the leaves, flowed through the extractor and were then condensed. In the separator, oil samples were separated from the water layer based on their density. The separated oil samples were filtered and dried for 12 hours using anhydrous sodium sulfate (Na_2SO_4). The dried samples were stored in a refrigerator at 4 °C. The characteristics of the oil samples were determined through qualitative analysis by GC-MS (Gas Chromatography-Mass Spectrometry). A similar procedure was followed for all 27 runs. The rated power of the electric boiler was 2000 W. The total run time for each run was observed and found to be 3.5 h. The goal of these experiments was to optimize the conventional electric-powered steam distillation process and identify the best combination of input parameters that lead to a higher oil yield from the Eucalyptus leaves.

5.2.2 Experiments with Solar Steam Distillation System

After performing 27 runs with normal Eucalyptus leaves, experimental runs with a solar distillation system using optimum values derived using RSM were conducted. Instead of using an electric boiler, a solar baby boiler was employed to produce the steam in the setup. This change allowed to utilize solar thermal energy for steam generation, making the process more sustainable and environmentally friendly. The experimental procedure for the solar steam distillation system was the same as that for the conventional electric-powered steam distillation system. The Eucalyptus leaves were thoroughly washed, sorted based on size, and kept on a wire mesh at the bottom of the extractor. The steam from the solar baby boiler was introduced into the extractor through a sparger to ensure uniform steam distribution. The experiments were performed in May 2022 at the terrace of CSIR-Central Salt & Marine Chemicals Research Institute, Bhavnagar. The coordinates of the location are specified as 21.7645° N latitude and 72.1519° E longitude. The solar intensity during the experiment varied between 93.95 W/m^2 and 861.54 W/m^2 throughout a typical day. The Eucalyptus leaves used for each run were maintained at 2.5 kg, and the optimum values for leaf size, extraction temperature, solid/solvent ratio, and extraction time were derived from the RSM analysis based on the conventional electric-powered steam distillation runs. The goal of these studies was to compare the solar steam distillation system with those produced by the conventional electric-powered steam distillation system in order to determine how well it extracts essential oils from Eucalyptus leaves. To comprehend the effectiveness and constraints of the solar distillation setup, the effect of solar radiation and other environmental conditions on the oil output was explored.

5.3 Experimental design and data analysis

The variables for the production of Eucalyptus essential oil were optimized using Response Surface Methodology (RSM). It is a statistical technique commonly used to optimize processes by determining the best combination of input variables to achieve desired responses (output variables). In this case, the variables considered during the experimental runs were:

- Leaf Size (A): The size of the Eucalyptus leaves used in the distillation process, measured in m. Extraction Temperature (B): The temperature at which the distillation process is carried out, measured in °C. Solid/Solvent Ratio (C): The ratio of the mass of Eucalyptus leaves to the volume of the solvent used for extraction. Extraction Time (D): The duration for which the distillation process is carried out, measured in min.

The response variables selected to evaluate the performance of the process were:

- Extraction oil yield (R1): The percentage of essential oil extracted from the Eucalyptus leaves during the distillation process. 1,8-Cineole Yield (R2): The percentage of 1,8-cineole, a specific component in the essential oil. α-Pinene Yield (R3): The percentage of α-Pinene, another specific component in the oil. The responses were determined at the optimal conditions of these variables. The designed model was then validated by comparing the experimental values with the predicted values. Design expert software uses simultaneous optimization techniques (desirability function) for the optimization of multiple responses. For the optimization, described desirability approach declaration in the work [20]. The experimental runs for this case were planned according to four-factor CCD, for 27 runs and 3 imitates at the center point. The data sets were analyzed and optimum operation conditions were derived using the Design Expert 8.0.6 software. The variables for the extraction of EO and the operating conditions range for implementation of RSM is shown in Table 2. Table 3 and 4 demonstrates the complete experimental run sheet for normal and sundry eucalyptus leaves.

Table 2. Actual and coded values of preparation variables used for optimization

Factors	**Independent Variables**	**Low level**	**High level**
A	Effect of leaf size (cm)	2	5
B	Temperature on leaf surface (^{0}C)	92	98
C	Solid/solvent ratio	0.4545	0.625
D	Time for completion (min)	150	210

Table 3. Detailed experimental run sheet and experimental data for normal leaves

Run	Factor 1 A: leaf size (cm)	Factor 2 B: Tepm. (^{0}C)	Fcator 3 C: solid/solvent ratio	Factor 4 D: time for completion (min)	Response 1 Yield %	Response 2 % 1-8 cineole	Response 3 % α – pinene
1	3.50	95.00	0.54	210.00	0.5	60.67	22.17
2	5.00	98.00	0.63	150.00	0.5	57.53	17.1
3	3.50	95.00	0.45	180.00	0.63	61.32	21.97
4	3.50	98.00	0.54	180.00	0.65	62.06	20.32
5	5.00	98.00	0.63	210.00	0.49	61.4	20.84
6	2.00	92.00	0.63	150.00	0.59	78..45	17.88
7	2.00	92.00	0.45	150.00	0.61	70.49	20.56
8	2.00	98.00	0.63	210.00	0.75	63.11	21.23
9	3.50	95.00	0.54	180.00	0.7	69.57	17.22
10	2.00	92.00	0.45	210.00	0.62	62.83	15.27
11	3.50	95.00	0.54	180.00	0.63	65.66	18.87
12	5.00	98.00	0.45	210.00	0.49	67.56	22.28
13	5.00	92.00	0.63	210.00	0.48	59.88	23.45
14	2.00	98.00	0.45	150.00	0.72	62.67	27.83
15	5.00	92.00	0.45	150.00	0.49	63.71	19.59
16	3.50	95.00	0.54	150.00	0.6	58.57	20.96
17	3.50	92.00	0.54	180.00	0.59	71.67	15.66
18	2.00	92.00	0.63	210.00	0.62	64.17	19.83
19	5.00	95.00	0.54	180.00	0.3	68.45	17.01
20	2.00	98.00	0.45	210.00	0.65	62.45	19.89
21	3.50	95.00	0.54	180.00	0.68	64.92	19.7
22	3.50	95.00	0.63	180.00	0.65	60.59	23.99
23	5.00	92.00	0.45	210.00	0.58	60.01	18.42
24	2.00	95.00	0.54	180.00	0.57	77.22	18.71
25	2.00	98.00	0.63	150.00	0.73	61.27	22.03
26	5.00	98.00	0.45	150.00	0.44	60.16	22.27
27	5.00	92.00	0.63	150.00	0.4	64.71	17.3

Table 4. Detailed experimental run sheet and experimental data for sundry leaves

Run	Factor 1 A: leaf size (cm)	Factor 2 B: Temp. (^{0}C)	Factor 3 C: solid/solvent ratio	Factor 4 D: time for completion (min)	Response 1 Yield %	Response 2 % 1-8 Cineole	Response 3 % α – pinene
1	5.00	92.00	0.45	150.00	0.19	53.03	25.07
2	2.00	98.00	0.45	210.00	0.32	54.36	27.54
3	3.50	95.00	0.54	210.00	0.21	55.49	24.11
4	2.00	95.00	0.54	180.00	0.38	62.73	20.79
5	5.00	95.00	0.54	180.00	0.2	52.76	20.05
6	5.00	92.00	0.45	210.00	0.21	52.33	23.42
7	3.50	98.00	0.54	180.00	0.34	58.86	23.19
8	3.50	95.00	0.54	180.00	0.22	58.01	23.3
9	2.00	92.00	0.63	210.00	0.35	59.57	24.27
10	5.00	98.00	0.45	210.00	0.18	55.69	24.66
11	3.50	95.00	0.63	180.00	0.28	57.23	25.11
12	2.00	98.00	0.63	150.00	0.37	60.83	22.31
13	5.00	98.00	0.63	210.00	0.21	53.21	21.88
14	3.50	95.00	0.54	180.00	0.27	58.08	24.8
15	3.50	95.00	0.45	180.00	0.2	53.87	26.76
16	3.50	92.00	0.54	180.00	0.23	56.1	24.75
17	5.00	98.00	0.63	150.00	0.2	52.35	25.95
18	5.00	92.00	0.63	210.00	0.18	52.87	23.99
19	2.00	92.00	0.45	210.00	0.38	54.65	25.9
20	2.00	98.00	0.45	150.00	0.28	58.6	23.93
21	5.00	98.00	0.45	150.00	0.2	54.55	25.83
22	3.50	95.00	0.54	180.00	0.2	62.82	21.79
23	2.00	92.00	0.45	150.00	0.25	55.01	25.35
24	2.00	98.00	0.63	210.00	0.39	61.39	21.57
25	3.50	95.00	0.54	150.00	0.19	56.86	24.45
26	2.00	92.00	0.63	150.00	0.32	57.06	25.34
27	5.00	92.00	0.63	150.00	0.195	50.52	27.41

5.4 Characterization of Eucalyptus oil

The chemical characteristics of the eucalyptus oil was evaluated through GC-MS. The tests were carried out at the NFDD Department and Pharmaceutical Department of Saurashtra University, Rajkot. The test was performed on a shimadzu model GCMS-QP2010 instrument with operating conditions are as follows: Injection temperature: 100 °C, Pressure: 73 kPa, linear velocity: 0.4 m/s, purge flow: 3.5 mL/min, and split ratio: 5. The column of the instrument was 30 m long having 0.25×10^{-3} m inner diameter. The sample was first dissolved in methanol and prepared with 10 % W/V before testing.

5.5 Results & discussion

- The experiments for essential oil extraction from the solar steam distillation system were carried out in the month of May 2022. The results for 4 sunny days of the experiment, when the time of extraction and temperature matched the optimal values, are shown in Fig. 5 (a) and

5 (b). The Scheffler concentrator was positioned to face the sun eastward in the morning. The plots show that the higher solar radiation generally correlated with increased steam generation, and this also affected the boiler efficiency positively. The enhanced solar energy input resulted in higher temperature differences for better heat transfer leading to higher solar baby boiler efficiency. It can also be observed that, there was no strong relationship pattern between ambient temperature and boiler efficiency. The expanded uncertainty associated with the measurement of the thermal efficiency of the solar boiler was calculated to be $\pm$ 0.38 %.

- When steam was generated in larger quantities, it facilitated the mass transfer of essential oil components from the leaf to the vapor phase, leading to higher yields. Heat transfer also improved, ensuring the leaves under process reach the desired temperature fast and with uniformity leading to enhanced release of the essential oil.

  The results of GC-MS from optimum values are depicted in Table 5. Table 5 shows a comparison of the response variables, the % oil yield, % 1-8 cineole, % α - Pinene for the conventional steam distilled and solar steam distilled processes.
- Showed a considerable variation in the yields for both experimental conditions. The ability to maintain specific and stable conditions in the conventional system might have led to the higher yield of essential oils from the Eucalyptus leaves compared to the variable conditions in the solar steam distillation system (Figure 5a). Similar to the % yield, due to more stable temperatures achieved in conventional steam distillation by carefully controlling and regulating the energy input, a higher % of 1-8 cineole was obtained for the conventional system.
- Solar steam distillation, on the other hand, depended on solar radiation, which varied depending on the time of day and the weather the duration of distillation could have affected the yield of α-Pinene, which was 20.81 % through conventional steam distillation compared to 8.9 % for solar steam distillation. Apart from a controlled condition of operation for the conventional system, the yields of α-Pinene could have differed because of the differences in the quality or composition of the source material between the experiments.

Table 5. Predicted and Experimental values of % oil yield, % 1, 8 – cineol, % α – pinene under the optimum extraction conditions

Eucalyptus leaves condition	Optimum extraction conditions				Response variables								
					SD With Electric Boiler						SD with Solar Boiler		
					Maximum value (predicted)			Maximum value (experimental)			Maximum value (experimental)		
	Leaf size (cm)	Temp (^{0}C)	Solid/ solvent ratio	Time (min)	% yield	% 1-8 cineole	% α – pinene	% yield	% 1-8 cineole	% α – pinene	% yield	% 1-8 cineole	% α – pinene
Normal	2	97.76	0.61	206.63	0.70	63.98	20.81	0.68	62.76	16.63	0.1	61.44	8.9
Sundry	2	96.97	0.48	209.92	0.32	57.34	24.94	0.36	57.77	16.95	0.08	54.96	2.82

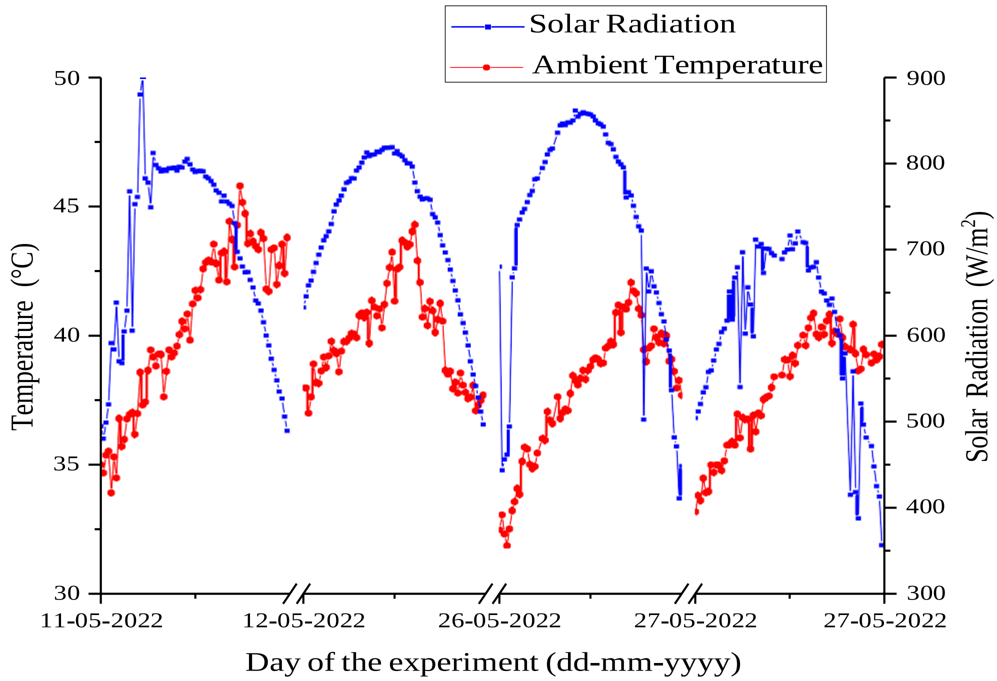


Figure 5 (a) Ambient conditions on 4 days of the experiment

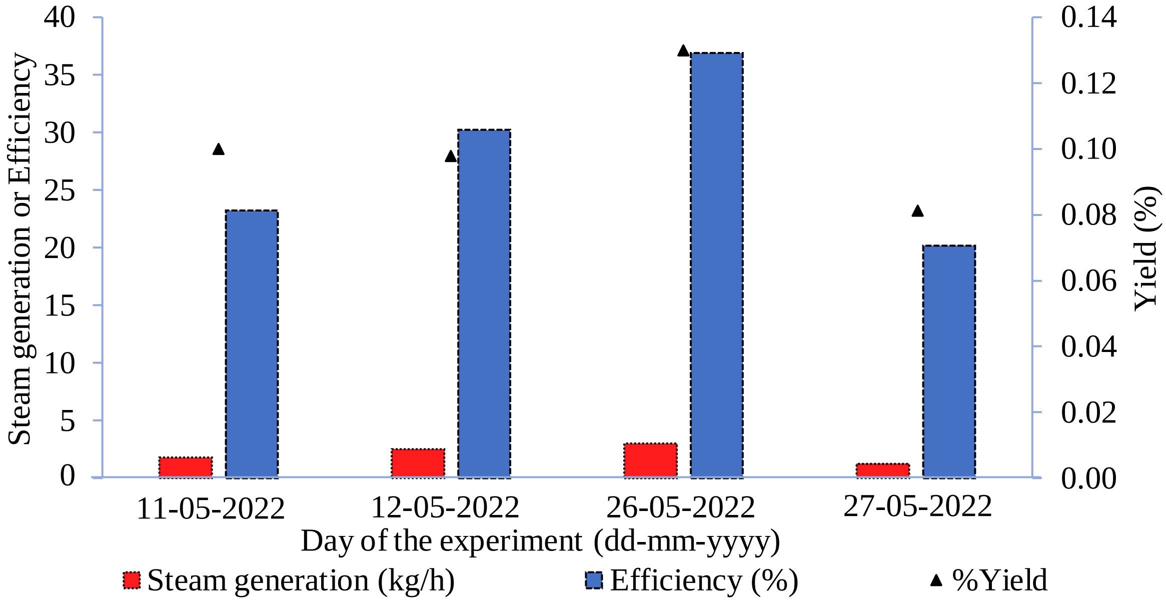


Figure 5 (b) Experimental results showing the average steam generation rate, efficiency of the solar boiler and yield of the essential oil on the days of the experiment.

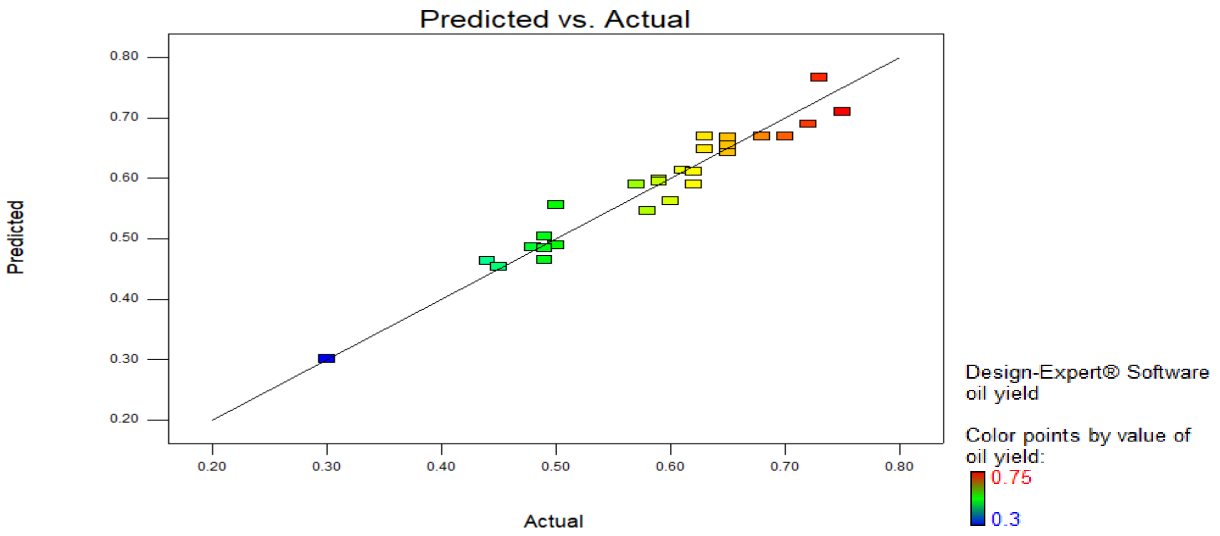
5.6 Analysis and evaluation of the fitted model

The responses were determined at the optimal conditions of these variables. The designed model was then validated by comparing the experimental values with the predicted values. Design expert software uses simultaneous optimization techniques (desirability function) for the optimization of multiple responses [20]. The variables for the extraction of Eucalyptus essential oil and the operating conditions range for the implementation of RSM are shown in Table 2. The ranges define the lower and upper bounds for each factor, in order to explore the response surface effectively and identify the optimal factor levels. Table 3 and 4 demonstrates the complete experimental run sheet, presenting the specific factor levels and corresponding responses observed in each of the 27 experimental runs. To determine the significance of the model, the model parameters were calculated using software. The acceptability of the model is evaluated based on the smaller values of the coefficient of variance (CV), which indicates higher reproducibility. A lower CV implies that the data points are less scattered relative to the mean. This validates the reliability of the model. The goodness of fit is supported by moderately lower values of the standard deviation (SD) and the closeness of the R^2 value to unity (1.0). The goodness of fit of the regression model is determined by the values of R^2 for the three responses Y, C, and P. The R^2 values of 0.9494 for Y, 0.9338 for C, and 0.9347 for P with normal leaves and 0.9215 for Y, 0.9057 for C, and 0.907 for P with sundry leaves indicate that the regression lines reasonably fit the data as shown in Table 6 and 7. The difference between the predicted R^2 and the adjusted R^2 is less than 0.2, indicating reasonable agreement and reliability for all three responses.

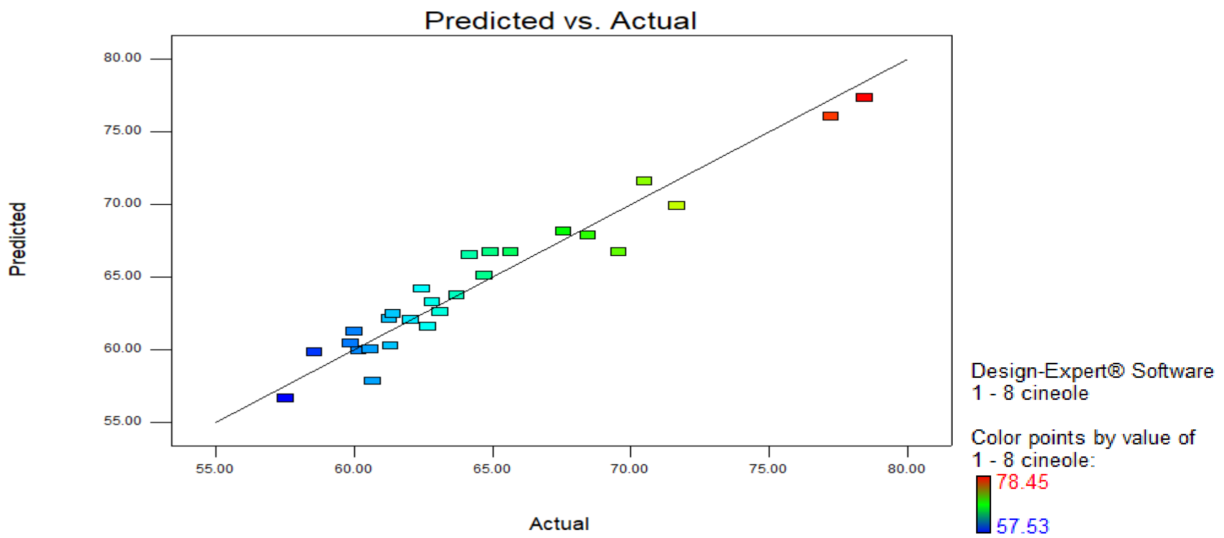
- Adequate precision is assessed by the signal-to-noise ratio, with a value greater than 4 considered desirable. In this case, the values of adequate precision for all three responses are significantly greater than 4, indicating that the existing model can effectively navigate the design space.
- The implications of the designed model are justified in Table 6 and 7, where the significance of terms is determined based on the p-value. A p-value less than 0.05 indicates significant terms, while a p-value greater than 0.05 indicates insignificant terms. An insignificant p-value is desired for lack of fit. The statistical data obtained confirms that the designed models are adequate for predicting the selected responses within the specified range of input variables.
- For the response surface model for steam distillation, the ANOVA (Analysis of Variance), as in Table 6 and 7, could help assess the significance of the model terms and identify which factors and interactions significantly influenced the response variable, the yield or quality of the essential oil. The model showed significant linear, interaction, and quadratic effects of independent parameters (A, B, C, D, AB, AC, AD, BC, BD, CD, A^2, B^2, C^2, D^2), indicated by p-values < 0.05 for significant terms and p-values > 0.05 for non-significant terms (a, b), as discussed previously.
- Furthermore, Figure 6 (a, b, c) and Figure 7 (a, b, c) shows that the predicted values are in close proximity to the experimental values for all three responses. This observation suggests that the predicted and experimental values align well with each other.

Table. 6 ANOVA for response surface model for all responses for normal leaves

	Y %					C %					P%				
Source	**Sum of Squares**	**df**	**Mean Square**	**F value**	**p value**	**Sum of Squares**	**Df**	**Mean Square**	**F value**	**P value**	**Sum of Squares**	**df**	**Mean Square**	**F value**	**p value**
Model	0.13	14	8.97E-03	16.1	<0.0001significant	2.49	14	0.18	12.1	<0.0001significant	2.28	14	0.16	12.3	<0.0001significant
A-Effect of leaf size	0.072	1	0.072	130	< 0.0001[a]	0.35	1	0.35	23.8	0.0004[a]	0.021	1	0.021	1.56	0.2361[b]
B-Temperature on leaf surface	3.10E-03	1	3.10E-03	5.55	0.0363[b]	0.35	1	0.35	23.6	0.0004[a]	0.49	1	0.49	37.1	< 0.0001[a]
C-Solid/solvent ratio	2.32E-05	1	2.32E-05	0.04	0.8418[b]	3.88E-04	1	3.88E-04	0.03	0.8739[b]	2.17E-03	1	2.17E-03	0.16	0.693[b]
D-Time for completion	3.00E-05	1	3.00E-05	0.05	0.8205[b]	0.025	1	0.025	1.67	0.2207[b]	1.66E-04	1	1.66E-04	0.01	0.9127[b]
AB	5.85E-03	1	5.85E-03	10.5	0.0071[a]	0.14	1	0.14	9.36	0.0099[a]	0.14	1	0.14	10.5	0.007[a]
AC	7.14E-04	1	7.14E-04	1.28	0.28[b]	0.062	1	0.062	4.24	0.062[b]	4.24E-03	1	4.24E-03	0.32	0.5817[b]
AD	8.73E-04	1	8.73E-04	1.56	0.235[b]	0.12	1	0.12	8.15	0.0145[b]	0.33	1	0.33	24.6	0.0003[a]
BC	3.03E-03	1	3.03E-03	5.43	0.038[b]	0.09	1	0.09	6.13	0.0292[b]	0.18	1	0.18	13.6	0.0031[a]
BD	7.61E-04	1	7.61E-04	1.36	0.2656[b]	0.45	1	0.45	30.3	0.0001[a]	0.024	1	0.024	1.8	0.2044[b]
CD	1.71E-05	1	1.71E-05	0.03	0.8641[b]	0.018	1	0.018	1.19	0.2966[b]	0.5	1	0.5	37.7	< 0.0001[a]
A2	0.034	1	0.034	60.9	<0.0001[a]	0.13	1	0.13	8.81	0.0118[b]	6.03E-03	1	6.03E-03	0.46	0.5127[b]
B2	7.54E-04	1	7.54E-04	1.35	0.2678[b]	2.91E-03	1	2.91E-03	0.2	0.6647[b]	4.74E-03	1	4.74E-03	0.36	0.5608[b]
C2	1.60E-04	1	1.60E-04	0.29	0.6016[b]	0.23	1	0.23	15.3	0.0021[a]	0.33	1	0.33	25.2	0.0003[a]
D2	6.55E-03	1	6.55E-03	11.7	0.005[a]	0.33	1	0.33	22.4	0.0005[a]	0.16	1	0.16	12.4	0.0042[a]
Residual	6.70E-03	12	5.58E-04			0.18	12	0.015			0.16	12	0.013		
lack of fit	5.72E-03	10	5.72E-04	1.17	0.547 not significant	0.13	10	0.013	0.56	0.78 not significant	0.12	10	0.012	0.53	0.79 not significant
pure error	9.81E-04	2	4.91E-04			0.046	2	0.023			0.043	2	0.022		
cor total	1.30E-01	26				2.67	26				2.43	26			
Adeq precision	18.547					13.985					15.496				
R2	0.9494					0.9338					0.9347				
Adj. - R2	0.8903					0.8565					0.8586				
Pred - R2	0.7345					0.6795					0.6866				
C.V.%	3.11					1.51					2.57				



(a)



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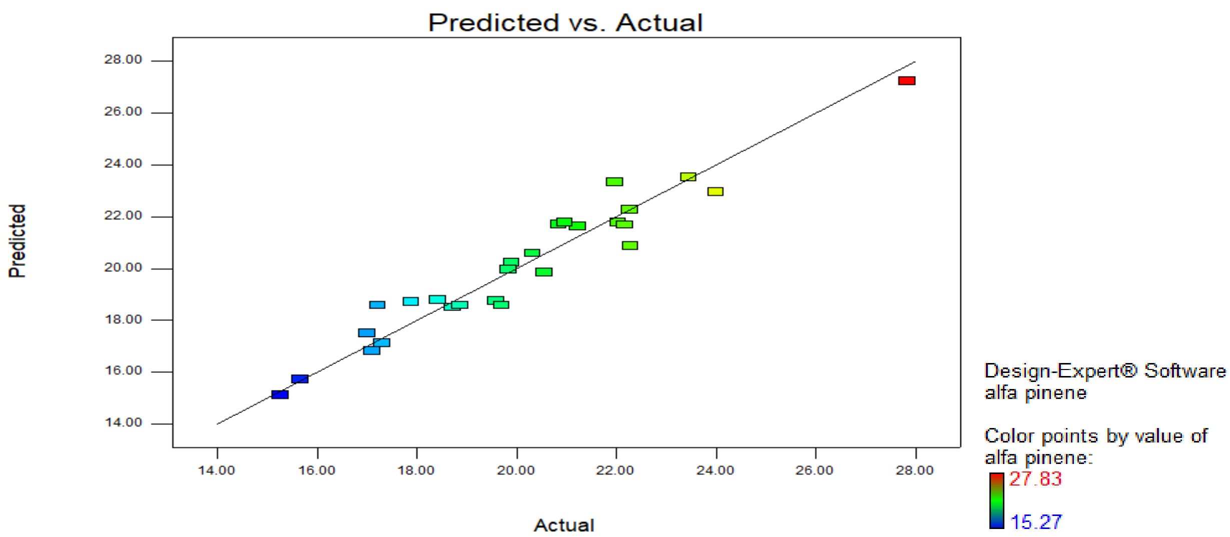

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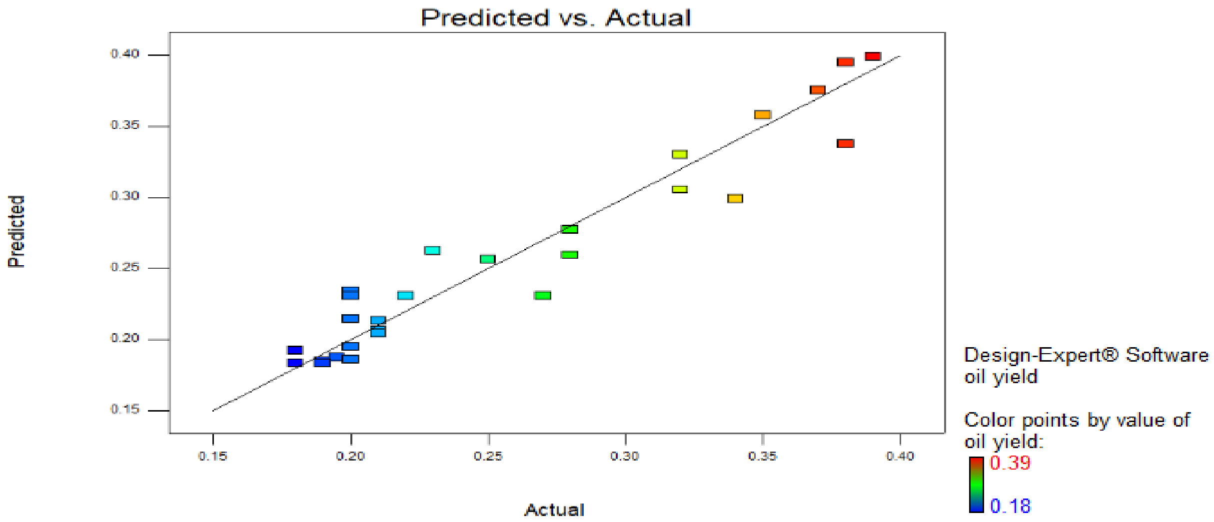
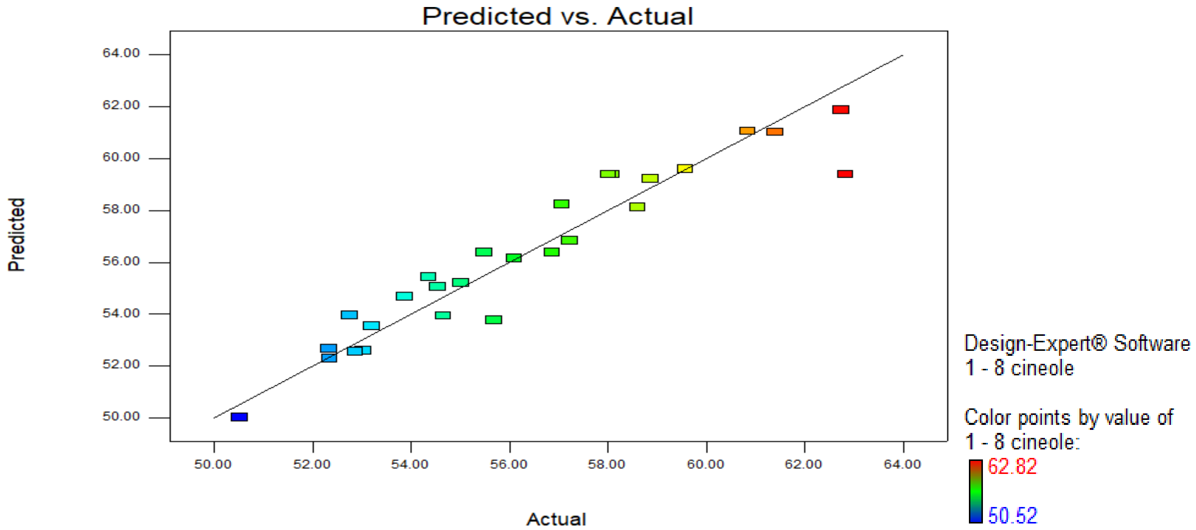
Figure 6. Variation of predicted values with actual values (a) for % Y, (b) for % C, and (c) for % P for normal Eucalyptus leaves

Table. 7. ANOVA for response surface model for all responses for sundry leaves

Source	Sum of square	df	Mean Square	F value	p-value	Sum of square	Df	Mean Square	F value	p-value	Sum of square	df	Mean Square	F value	p-value
Model	0.11	14	8.15E-03	10.07	0.0001 significant	1.15	14	0.082	8.24	0.0004 significant	0.9	14	0.065	8.36	0. 0004 significant
A-Effect of leaf size	0.085	1	0.085	104.52	< 0.0001	0.58	1	0.58	58.72	< 0.0001	3.18E-04	1	3.18E-04	0.04	0.8427
B-Temperature on leaf surface	2.57E-03	1	2.57E-03	3.17	0.1003	0.087	1	0.087	8.75	0.012	0.047	1	0.047	6.02	0.0304
C-Solid/solvent ratio	4.66E-03	1	4.66E-03	5.76	0.0336	0.045	1	0.045	4.47	0.0561	0.067	1	0.067	8.61	0.0125
D-Time for completion	2.39E-03	1	2.39E-03	2.95	0.1115	6.02E-07	1	6.02E-07	6.04E-05	0.9939	0.036	1	0.036	4.68	0.0513
AB	8.52E-05	1	8.52E-05	0.11	0.7512	6.00E-04	1	6.00E-04	0.06	0.8103	0.011	1	0.011	1.41	0.2585
AC	1.87E-03	1	1.87E-03	2.31	0.1542	0.15	1	0.15	14.59	0.0024	0.056	1	0.056	7.24	0.0196
AD	2.55E-03	1	2.55E-03	3.15	0.1013	7.99E-03	1	7.99E-03	0.8	0.3881	0.1	1	0.1	12.9	0.0037
BC	1.59E-03	1	1.59E-03	1.96	0.1867	8.70E-05	1	8.70E-05	8.73E-03	0.9271	0.086	1	0.086	11.1	0.006
BD	7.10E-04	1	7.10E-04	0.88	0.3676	8.36E-03	1	8.36E-03	0.84	0.3778	5.71E-03	1	5.71E-03	0.74	0.4069
CD	8.49E-04	1	8.49E-04	1.05	0.3261	0.031	1	0.031	3.08	0.1047	0.071	1	0.071	9.2	0.0104
A2	8.60E-03	1	4.86E-03	6.01	0.0306	0.02	1	0.02	2.04	0.1789	0.12	1	0.12	15.4	0.002
B2	4.76E-03	1	4.77E-03	5.89	0.0319	0.025	1	0.025	2.5	0.1396	0.035	1	0.035	4.56	0.054
C2	6.41E-05	1	6.4E-05	0.079	0.7832	0.12	1	0.12	11.6	0.0052	0.22	1	0.22	28.1	0.0002
D2	2.51E-03	1	2.51E-03	3.1	0.1035	0.078	1	0.078	7.86	0.0159	0.054	1	0.054	7.04	0.021
Residual	9.72E-03	12	8.09E-04			0.12	12	9.95E-03			0.093	12	7.73E-03		
Lack of Fit	6.96E-03	10	6.96E-04	0.5	0.81 not significant	0.057	10	5.65E-03	0.18	0.97	0.044	10	4.40E-03	0.18	0.97not significant
Pure Error	2.76E-03	2	1.37E-03			0.063	2	0.031			0.049	2	0.024		
Cor Total	0.12	26				1.27	26				1	26			
Adeq Precision	9.587					10.659					10.946				
R^2	0.9215					0.9057					0.907				
Adj. – R^2	0.83					0.7958					0.7984				
Pred – R^2	0.6331					0.6368					0.6588				
C.V.%	5.66					1.33					1.79				



(a)



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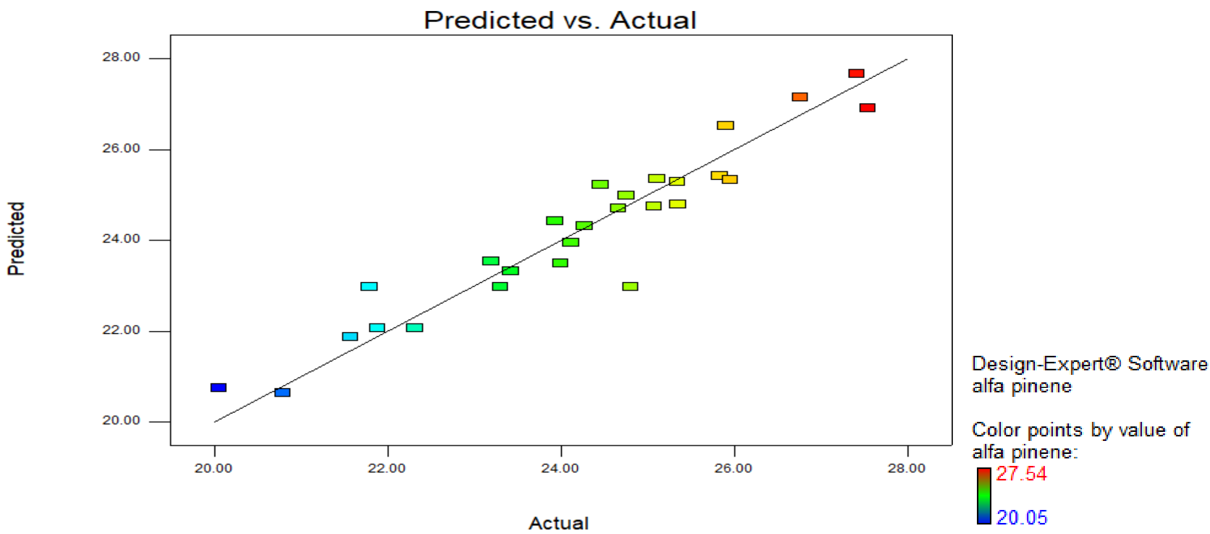

(c)

Figure 7. Variation of predicted values with actual values (a) for % Y, (b) for % C, and (c) for % P for sundry Eucalyptus leaves

6. Achievements with respect to objectives

- Designed and fabricated the conventional steam distillation unit and Scheffler solar concentrator (for solar steam distillation) with 2.5 m^2 area to extract the essential oil. Eucalyptus leaves were selected as raw material because of its medicinal and aromatherapy benefits.
- Optimization results obtained with the design expert 8.0.6 are found satisfactory, as the % 1 – 8 cineole and % α-Pinene obtained for conventional steam distillation are similar to solar steam distillation system as shown in table 5. It is also seen from table 5 that, the quality of essential oil obtain from normal eucalyptus leave is better than sundry leaves because of better volatility.
- Due to the variation in intensity of sunlight and time limit, obtained less % of oil yield with solar steam distillation. But the quality of oil has no major difference as compared to conventional steam distillation. So, the system is useful for the medium temperature range applications and for different plant materials also.

7. Conclusion

The goal of the study was to maximise the yield of essential oil from eucalyptus leaves while identifying the variables that affect both the oil yield and the yields of the two main constituents, 1-8 cineole and α-Pinene. Response surface methodology (RSM), a statistical and mathematical approach was used to describe and optimise systems with various variables. In order to investigate the impacts of various input factors on the oil yield as well as the components, the study was carried out a total of 27 experimental runs in a conventional electric-powered steam distillation system. The following conditions were determined to be the most effective for obtaining essential oil from eucalyptus leaves: size of leaf: 0.02 m, temperature: 97.76 °C, ratio of solid to solvent: 0.61 and time: 206 min for normal leaves. According to the findings, the optimized solution yielded 0.70 % oil, 63.98 % 1,8-cineole, and 20.81 % α-Pinene for normal leaves. The corresponding experimental values for yield, % 1-8 cineole, and % α-Pinene were 0.68 %, 62.76 %, and 16.63 %, respectively. 0.02 m leaf size, 96.97 ^{0}C temperature, 0.48 solid to solvent ratio and 209.92 min were the effective conditions for sundry leaves. According to the findings, the optimized solution yielded 0.32 % oil, 57.34 % 1,8-cineole, and 24.94 % α-Pinene for sundry leaves. The corresponding experimental values for yield, % 1-8 cineole, and % α-Pinene were 0.36 %, 57.77%, and 16.95%, respectively. The study further applied the RSM-derived optimal operating conditions from the conventional system to a Scheffler solar steam distillation system. By applying Scheffler solar steam distillation with normal leaves at optimal operating conditions, obtained the results: 0.1% oil, 61.44 % 1,8-cineole, and 8.9 % α-Pinene. Same as with sundry leaves obtained 0.08 % oil, 54.96 % 1,8-cineole, and 2.82 % α-Pinene. Variations in solar intensity and other ambient temperature were observed, which resulted in a lower extraction oil yield than the conventional technique. The results of this study can aid in improving the understanding of the solar thermal extraction procedure, and the optimal circumstances can be used in industrial settings to produce eucalyptus oil and its primary constituents for a variety of uses at higher yields.

8. Publications

[1] Kajal J. Sareriya, Jigar K. Andharia, Piyush B. Vanzara, Subarna Maiti, A comprehensive review of design parameters, thermal performance assessment, and medium temperature solar thermal applications of Scheffler concentrator, Cleaner Engineering and Technology 6 (2022) 100366.

[2] Kajal J. Sareriya, Piyush B. Vanzara, Dhara Upadhyay, Methodology for Extraction of Essential Oils: A Review, International Journal for Multidisciplinary Research (IJFMR) E-ISSN: 2582-2160. Volume 5, Issue 4, July-August 2023.

[3] Kajal J. Sareriya, Piyush B. Vanzara, Design and Experimentation of a Laboratory Steam Distillation setup for Extraction of Essential oil from Eucalyptus leaves. (submitted and accepted in International Journal of Plant and Environment).

[4] Kajal J. Sareriya, Piyush B. Vanzara, Subarna Maiti, Solar steam distillation for Eucalyptus essential oil extraction optimized with Response Surface Methodology (Prepared).

9. Acknowledgement

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