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Effect of drying conditions on phytochemicals in vacuum drying

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Abstract
Basil leaves scientifically proved to have phytochemicals compound which has potential of bioactivities. To preserve the quality of thisherbs, drying process must be carried out.
Drying treatment and experimental method for vaccum drying was carried out at the temperatures of 45 °C, 55 °C and 65 °C to find and suggest the optimum drying condition for acquiring quality dried basil leaves and phytochemicals like uginol, caryophyll of basil leaves. Results have revealed that 'total drying time' is considerably reduced with the increase in drying air temperatures from 45 °C to 65 °C. It could be recommended that for the best drying temperature is 45 °C of basil leaves to retain the various phytochemicals.

Keywords: Aroma, drying treatment, basil leaves, vaccum drying, phytochemical

Introduction
The major focus for drying basil leaves often remained centered around a point where its aroma needs to be preserved beside the appearance and nutritional characteristics. Researchers (Diaz-Maroto *et al*., 2002; Fleisher, 1981) [8, 10] have reported that improper drying may cause losses in volatilities or formation of new volatilities as a result of oxidation and esterification reactions. The volatile composition of basil is found to be dependent on the variety and/or geographical cultivation of the basil plant depending upon main components (Linalool, methyl cinnamate, eugenol, methyl eugenol, and etc.) of this precious herb.
The majority of findings have revealed this fact where the drying is reported to influence changes in the volatile compounds present in basil.
If we look from quantitative points of view, these decreases in the total amounts of essential oils have been reported to varied tune, say being 36% to 45% for sweet basil during drying at ambient temperature (Nykanen and Nykanen, 1987; 1989) [12]. A study by Yousif *et al*. (1999) [14] showed significant difference in concentrations of linalool and methyl chavicol in air-dried basil samples compared to those present in fresh samples, while that of vacuum dried samples showed substantial increase of about 2.5 fold for linalool and 1.5 fold for methyl chavicol, compared to that present in air-dried samples. Di Cesare *et al*. (1994; 2000; 2001; 2002; 2003) [3, 4, 5, 6, 7] found microwave drying to retain high percentages of characteristic volatile compounds (eucalyptol, linalool, eugenol, and methyl eugenol) in basil (*Ocimum basilicum* L.) compared to samples dried by air-drying and freeze-drying with blanching, except freeze-dried unblanched leaves. Other studies on drying methods on volatilities of leaf (Diaz-Maroto *et al*., 2002) [8], and spearmint (Diaz-Maroto *et al*., 2003) [9] too have given such logical variability.
Yuparat *et al*. (2014) [15] utilized some of the predictive models to evaluate the performances as well as influences of certain parameters towards drying of leaves and other similar materials by fitting prevailing moisture versus time data to five different crop drying models. The drying constants were well related to the drying temperatures. The ultimate findings of Abdollah *et al*. (2014) [1] reflected the facts that (1) drying temperatures can decrease essential oil contents of basil, (2) drying methods can change the chemical profile of essential oil of basil, and (3) oven drying at 40 ^{0}C had the least effect on essential oil.
Looking into plethora of such studies and their findings towards temperature effects on drying and volatile components of basil leaf, present research was conceived and conducted in India whose preliminary results are reported herein. The major aim of the work was to examine the influence of various drying methods and retention of phytochemical on a couple of crop varieties as cultivated in India, leaves cultivated in this specific region of India, to promote its

market/utility and also to maintain its nutritional value and other qualitative parameters in an optimum manner.

Materials and Methods

Fresh basil leaves were taken from the plants grown in campus and thoroughly cleaned before manual nibbing. The soft stems were removed and basil leaves were separated and cleaned manually to remove soil and dust particles if any attached to it.

Cleaned basil leaves were weighed in digital balance (Simanzu make, Capacity: 220 g, least count: 0.01 g). Samples were prepared and placed in tray for vacuum drying.

Weighed basil leaves were pretreated by steam blanching for 30 seconds by keeping them in a sieve above the boiled hot water to receive the steam coming from it.

Steam blanched samples were placed over a perforated tray to separate the stuck leaves during steaming. After that weight sample of leaves was loaded into perforated stainless steel tray in vaccum dryer. Fresh sample without pretreatment was used as control

The drying of basil leaves was carried out using solar dryer. Treatment was carried out with samples in triplicate.

Each 100 g blanched and unblanched samples were uniformly spread in the perforated stainless steel tray and dried at the desired temperature. The moisture loss was recorded by using top pan digital balance.

An electrically heated laboratory vacuum dryer, manufactured by Gansons Ltd., Mumbai and externally fitted with rotary vacuum pump and thermostat was used. The dryer was pre-adjusted to the selected temperature (45, 55, 65 ^{0}C) for about half an hour before starting of the experiment to achieve the steady state.

Each 100 g blanched and unblanched samples were uniformly spread in single layer on a perforated stainless steel tray and dried at the desired temperature with a constant vacuum of 700 mm Hg. The moisture loss was recorded at every 60 min interval using top pan digital balance.

The drying process was stopped when the last two consecutive observations of moisture loss were found constant. The product was cooled for 10 min after drying and packed in LDPE bags for further analysis. All the experiments were conducted in triplicate and the average values are reported

Estimation of Volatile Oil

For the estimation of volatile oil in the dried basil leaves, minimum 50 g dried sample was taken. To collect 50 g dried sample, at least 300 g fresh leaves were collected to dry in hot air tray dryer. For duplication total 600 g. Fresh leaves were collected and dried for volatile oil sample analysis.

The volatile oil content of basil leaves was estimated as described by the Bureau of Indian Standards (SP: 18(part Vii)-1982)

50 g of basil leaves were transferred into 1 liter round bottomed flask and then water was added to fill the flask slightly less than half full and mixed by swirling. To this few glass beads were also added. The flask was connected through calibrated oil trap to the condenser. The mixture was distilled for four hours until there was no increase in the oil content over a period of 1 hour. The setup was cooled to room temperature and allowed to stand until the oil layer was clear. The volatile oil was collected in the trap, was measured in ml.

GC - MS Conditions

For the identification of the volatiles compounds, some samples were subjected to GC- MS analysis on a Perkin Elemer Autosystem Excel with Turbomass. Conditions were as follows

1. Mode: TIC (Total Ion Chromatogram)
2. Column Type: PE- 5 (MS)
3. Column Oven Temperature: 70 °C (5 minute) -80 °C (10 minute)
4. Injector Temperature: 250 °C
5. Detector: Quadrupole
6. Ion Source Temperature: 250 °C
7. Carrier Gas: Helium
8. Flow Rate: 1ml/min
9. Split Ratio: 1: 5

Results and Discussion

The basil leaves were collected from the plants grown in campus. samples of the fresh basil leaves was 81.68% (w. b.) at the time of harvest. The range of moisture content varied from 81.00 - 83.00% (w. b.), which shows that the basil leaves can be considered under highly perishable group.

Drying Characteristics of Basil Leaves

The drying characteristics of basil leaves were analyzed using the experimental data on moisture of product at various time intervals for different drying conditions. The experimental data of the drying behaviour of basil leaves in relation to moisture content,was recorded and summarized. After pretreatment, the samples were dried up to the safe moisture content level of 4 to 6% (% d.b.).

Relation of time, temperature, moisture content (% d.b.) was attempted to characterize the drying behaviour of basil leaves. The moisture content was compared for the blanched and unblanched samples with different time, temperature for different dryers.

The basil leaves were steamed and thus had some moisture on the surface of basil leaves due to which blanched shows higher moisture content then actual

Vacuum drying of basil leaves

The freshly harvested basil leaves were placed over stainless steel tray, which was kept over the platform for drying in vacuum oven at 45, 55 and 55 ^{o}C temperature and 700 mm of Hg vacuum. The drying in vacuum oven was done at each till the final weight reached to predecided level. The observations in regards to weight loss were noted during each drying run at an interval of 60 minutes. The observations on drying under vacuum oven at different drying temperatures are presented in Appendix I.

In vacuum drying, moisture loss was more in the blanched basil leaves samples than the unblanched samples at 45, 55 and 65 °C drying temperatures. From the Figs 1, 2 and 3, it was observed that the moisture loss was less in case of blanched samples at the 55 °C temperature. The effect of blanching at the 55 °C temperature was comparatively lower than the 45 and 65 °C drying temperatures. The initial moisture loss was rapidly increasing at 45 °C in the unblanched sample. This was due to the effect of the vacuum in 45 °C temperature in unblanched sample. No constant rate drying period was observed during drying. Complete drying took place only in falling rate period.

The effect of drying air temperature on drying behaviour of basil leaves for blanched and unblanched samples indicate that as inlet temperature increased, the rate of moisture reduction was increased.

The effect of temperature was found more prominent for blanched basil leaves. The initial moisture content 466.67,

436.84 and 500.00 (% d.b.) were reduced to final moisture content of 5.56, 5.26 and 5.88 (% d.b.) in 360, 300, and 180 minute for 45, 55 and 65 °C temperatures, respectively. In case of unblanched samples, the initial moisture content 488.24, 417.95 and 471.43 (% d.b.) were reduced to final moisture content of 5.88, 5.98 and 4.76 (% d.b.) in 480, 360 and 300 minute for 45.55 and 65 °C temperatures, respectively.

Effect of vacuum drying on the colour, appearance, texture of vacuum dried leaves was found better for lower temperature in comparison to higher temperature. In blanched samples the colour was very near to colour of fresh samples, though colour varied with temperature. Texture and appearance was also found better for blanched samples in comparison to the unblanched samples.

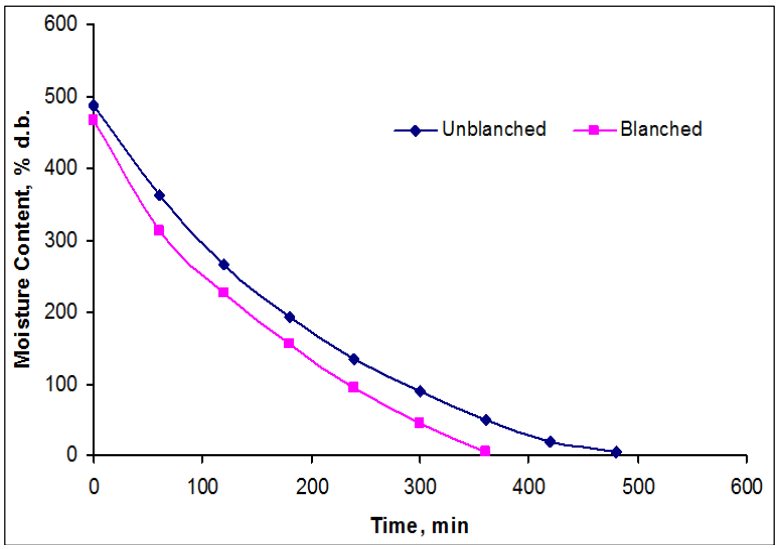


Fig 1: Variation in moisture content of basil leaves with drying time for vacuum drying at 45 °C

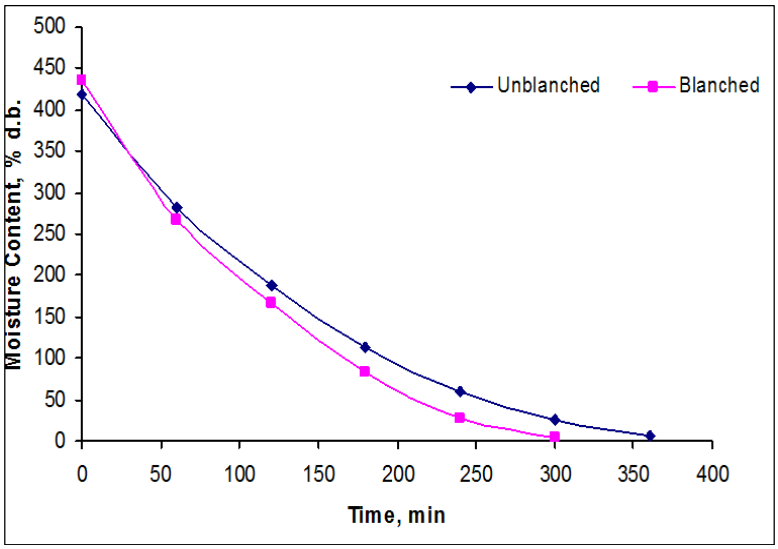


Fig 2: Variation in moisture content of basil leaves with drying time for vacuum drying at 55 °C

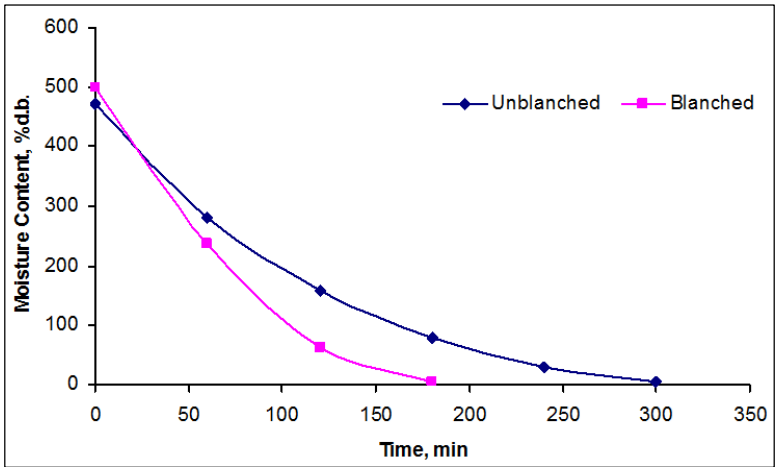


Fig 3: Variation in moisture content of basil leaves with drying time for vacuum drying at 65 °C

Effect of Drying Conditions on Volatile Oil Content

Volatile oil content of dried basil leaves is presented in Table 1. It showed that volatile oil of samples varied from 0.51 to 0.73 ml/100g d.m. In most cases of unblanched samples, oil content loss was higher as compared to blanched sample. This might be be due to content loss during blanching treatment.In vacuum drying of basil leaves at 45, 55 and 65 ^{0}C temperatures, the volatile oil content was found as 0.73, 0.60 and 0.53 ml/100g d.m., respectively, in the unblanched samples. Whereas, 0.71, 0.56 and 0.51 ml/100g d.m., respectively, in case of blanched samples. The volatile content of fresh sample was 1.36-ml/100g d.m. The loss of volatile oil content was very high ranging from 46.33% at 45 °C drying temperature (unblanched,) to 62.50% (unblanched) for vacuum drying at 65 °C. Loss of volatile oil content was higher when basil leaves were dried at higher temperature. This might be because of the breakage of oil cell due to heating, which leads to loss of volatile oil. It was also observed that although at higher temperature the drying time was shorter, the loss of volatile oil was higher.

From the Table 1 it was revealed that volatile loss was minimum 46.33% d.m. in the unblanched samples at the 45 °C temperature in the vaccum dryer, in blanched samples loss was minimum to 47.80%.

Table 1: Experimental data on volatile oil content on dried basil leaves

Type of dryer	Temperature (°C)	Treatment Unblanched (UB) / blanched (B)	Volatile oil (ml/100 g d.m.)	Loss in Volatile oil (%)
Vacuum dryer	45	UB	0.73	46.33
		B	0.71	47.80
	55	UB	0.60	55.89.
		B	0.56	58.83
	65	UB	0.53	62.50
		B	0.51	61.03

Effect of Drying Conditions on Active Ingredients

Blanching resulted into the good colour. Hence, blanched samples were taken for further GC-MS analysis to identify the volatile compounds in the fresh basil leaves and as well as in blanched basil leaves dried at the temperature 45, 55 and 65 °C in the vaccum dryer..

Fig. 4 shows a typical chromatogram obtained from a fresh sample from GC-MS analysis and Table 2 shows the percentage composition of the identified compounds as calculated from the respective chromatographic areas. There are, mainly eugenol (61.69%) and in minor proportion caryophyllene (28.77%) remaining peaks correspondence to compounds that were not identified.

Table 2: Active ingredients available in fresh basil leaves

Peak	Compound	Area (%)
1	Eugenol	61.69
2	Caryophyllene	28.77

The two major volatile compounds in fresh basil leaves samples were eugenol and caryophyllene usually considered responsible for the typical basil aroma as per figure 4. Similar results were reported by (Anon., 1966) [2]. The area under the chromatographic peaks of the other compounds is small.

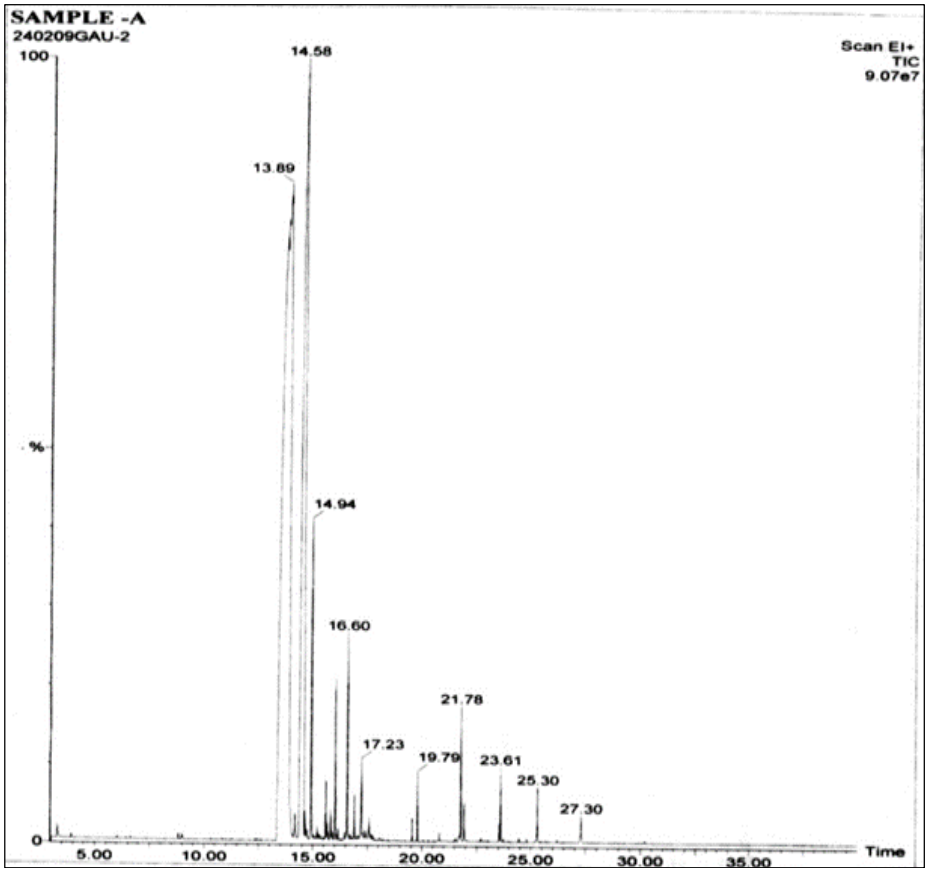


Fig 4: Gas chromatograph of fresh basil leaves

Effect of drying conditions on eugenol compound

Table 3 shows the eugenol percentage in the dried blanched basil leaves in vacuum drying at 45, 55 and 65 °C temperature. In the fresh basil leaves eugenol was 61.69% and Caryophyllene was 28.77%.

As shown in figure 5, in the vacuum drying of blanched basil leaves, the eugenol was observed 33.74% at 45 °C temperature, 32.10% at 55 °C temperature and 30.97% at 65 °C temperature which indicates that as temperature increases, the eugenol was decreases

Table 3: Eugenol content in the dried basil leaves

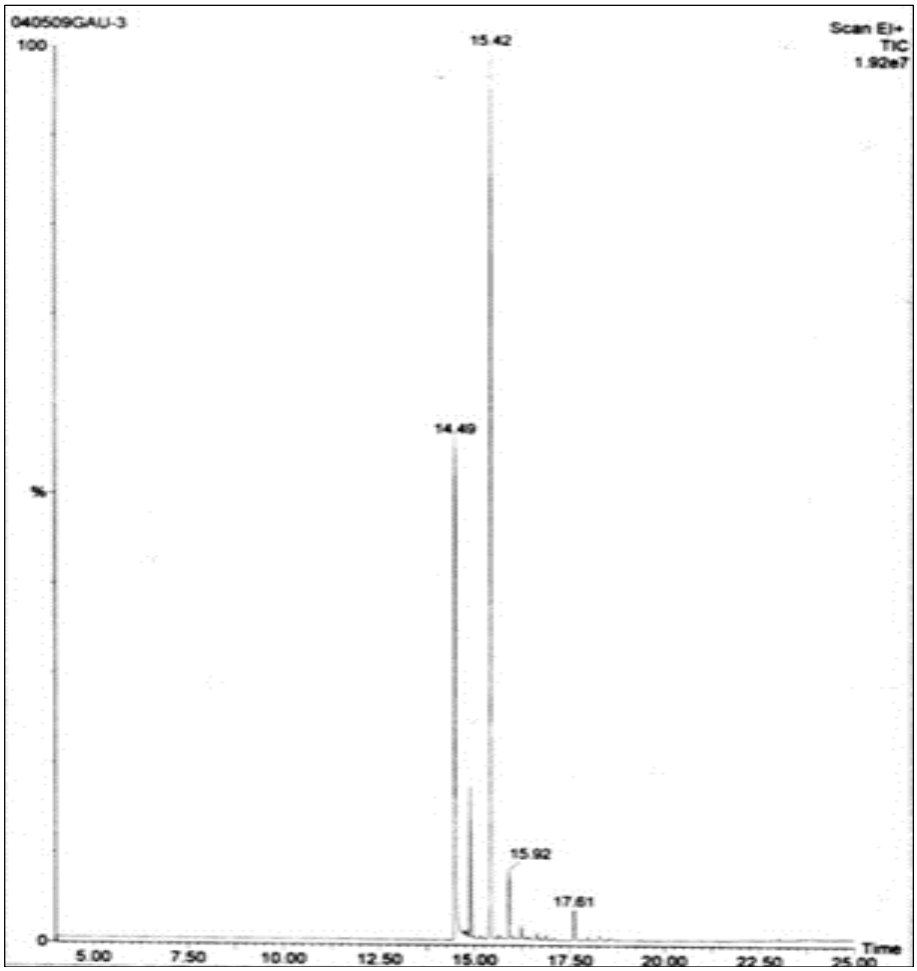
Type of dryer	Temperature (°C)	Treatment Blanched (B)	Eugenol Content in fresh leaves (%)	Eugenol content in dried leaves (%)
Vacuum Dryer	45	B	61.69	33.74
	55	B	61.69	32.10
	65	B	61.69	30.97

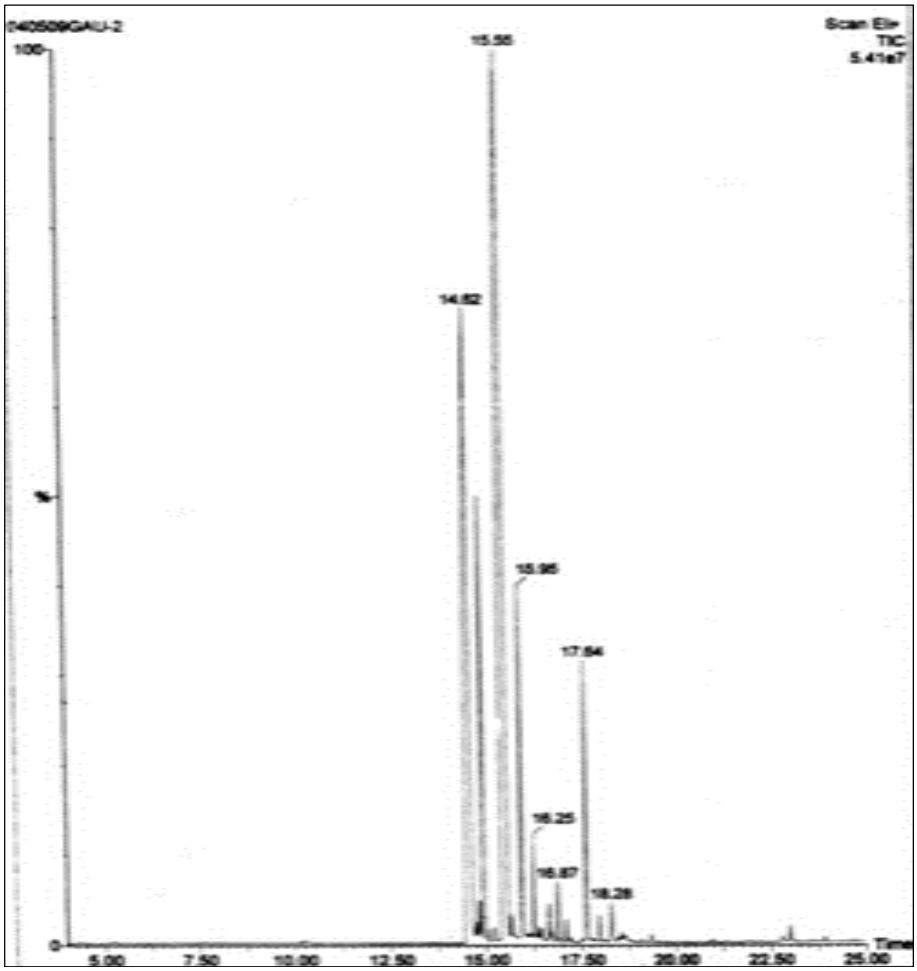
Effect of drying conditions on caryophyllene compound

The percentage retentions of the second major volatile compound i.e caryophyllene in the blanched basil leaves was less in lower temperature than the higher temperature in vacuum drying are reported in Table 4. It shows that as compared to caryophyllene in the fresh sample of basil leaves it increased in basil leaves dried in a 45 to 65 °C the temperature as per figure 5. This trend was observed similar to trend observed by Mondal (2007) [11].

Table 4: Caryophyllene content in the dried basil leaves

Type of dryer	Temperature (°C)	Treatment Blanched (B)	Caryophyll Content in fresh leaves (%)	Caryophyll content in dried leaves (%)
Vaccum dryer	45	B	28.77	47.82
	55	B	28.77	53.76
	65	B	28.77	60.36





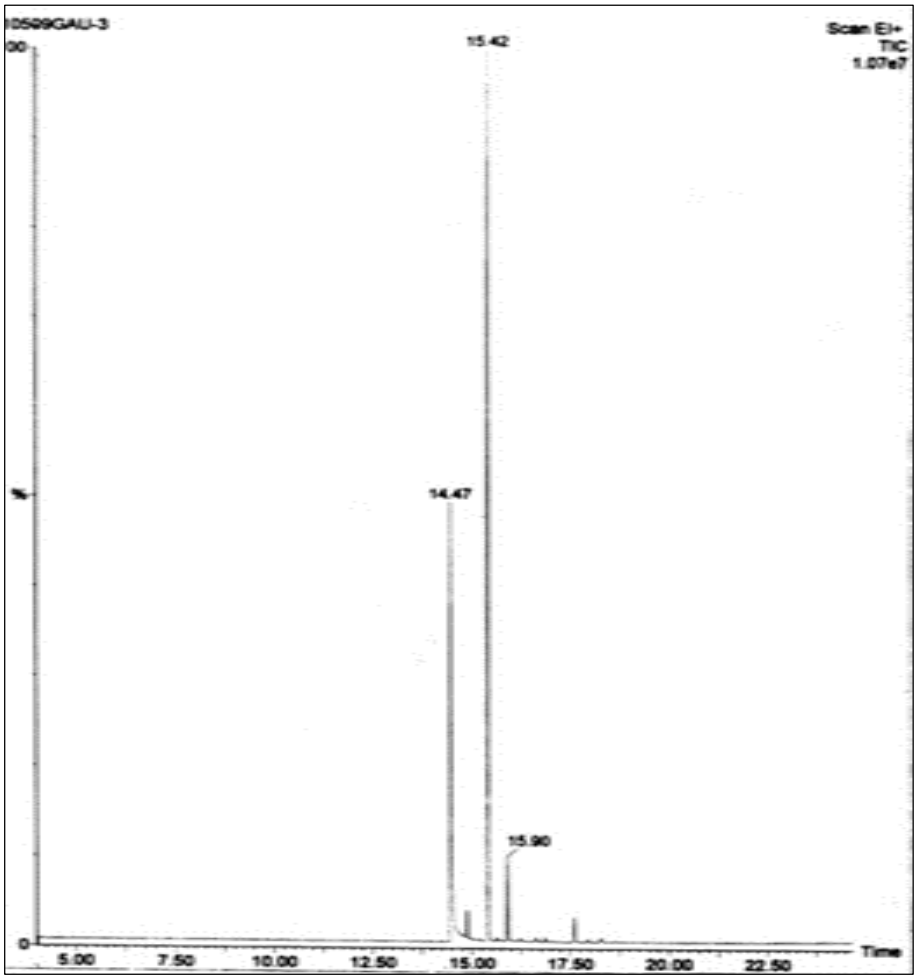


Fig 5: Eugenol and Caryophyllene content of vacuum dried basil leaves at different temperatures: (A) 45 °C; (B) 55°C and (C) 65 °C

Conclusions

On the basis of experimental results and data analysis the following conclusions are drawn given as under.

1. Total drying time considerably reduced with the increase in drying air temperature from 45 °C to 65 °C temperatures in most cases.
2. Blanched sample took less time for drying compared to unblanched samples in each dryer at every temperature from 45, 55 and 65 °C temperature.
3. Volatile oil was found slightly less in the blanched samples in comparison to unblanched samples of basil leaves.
4. Volatile oil was higher at lower temperature and decreased at higher temperature.
5. Eugenol was found as major active ingredient in the fresh sample of basil leaves, while caryophyllene was the second major active ingredient.
6. In the dried basil leaves, the caryophyllene increased, while eugenol decreased most at higher temperatures.

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