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Research Article

Impact of Different Drying Techniques on Neem Seeds Drying Kinetics and Oil Quality

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Neem oil is a promising alternative for synthetic chemicals in food preservation and an active functional agent in food packaging. Drying studies were conducted on neem seeds using different drying methods, and the oil yield profile and nutritional content such as azadirachtin content, functional groups, and elemental composition were analysed. Tray drying at 60°C showed a faster drying rate with a minimum quality of the oil. The process parameters were statistically optimized by analysing the effect of drying methods and thickness (15 and 30 mm) on azadirachtin content and oil yield. Maximum oil yield and azadirachtin content of 42.1 and 0.053% were obtained in solar drying with 15 mm bed thickness. Fourier-transform infrared spectroscopy analysis showed that there is no change in the functional group when the neem seeds were dried, and the peak absorption wavenumber confirmed the presence of O-H stretching, C-H stretching, C-O stretching, C-H bending, O-H bending, C=O stretching, and N-H stretching. Sun- and solar-dried neem seeds showed maximum retention in elemental composition when compared to the tray-drying method.

1. Introduction

Neem (*Azadirachta indica*) which is known as "Geed Hindi" in Somalia which means that "the Indian tree" belongs to the mahogany family "Meliaceae." There are about 14 million neem trees in India which can grow well up to 40 to 60 feet high in all types of soil [1]. Neem oil has excellent antibacterial, antifungal, antioxidant, and plasticizer properties and finds its applications in food preservation, packaging, and storage due to the presence of bioactive phytochemicals such as azadirachtin, salannin, nimbidin, gedunin, nimbin, isomargolonone, margolone, nimbolide, and margolone. Neem oil is an attractive choice for active food packaging applications due to its antibacterial and antioxidant characteristics with maximum tensile strength, elongation, and transparency [2]. Neem fruit pulp represents, about half the weight of neem fruits which serves as a carbohydrate, a rich substrate for industrial fermentations [3].

Neem tree seeks attention worldwide due to its medicinal properties in the field of ayurveda and due to the presence of azadirachtin ($C_{35}H_{44}O_{16}$), a tetranortriterpenoid. Apart from azadirachtin, it also contains other bioactive components such as nimbin, nimbinin, nimbidin, oleic stearic and palmitic acids, quercetin, and other limonoids. Neem seed area, oil content, and azadirachtin content vary among individual tree and agroclimatic zones of India [4].

The neem tree starts fruiting after 3 to 5 years of planting and yields upto 50 kg of neem fruits annually from the 10^{th}

year onwards. Neem fruit is usually oval or nearly round in shape which has a thin exocarp, yellowish white mesocarp (bittersweet pulp) of thickness in the range of 3–5 mm, and the endocarp has a white hard inner shell containing brown kernel which contains 25–45% oil [5].

Neem oil is a vegetable oil which contains high fatty acids and low terpene content which adds an advantage over tea tree oil. It is also used in medicines, soap industries, and cosmetics and can also be used as a mosquito repellent [4]. Every year neem tree yields 3.5 million tonnes of kernels which can produce 7 lakh tonnes of neem oil. Due to a lack of processing equipment, India produces only 2.5 lakh tonnes of the total oil which is just 30% of the total potential [6].

Neem fruit contains 40 to 60% of water which makes it a highly perishable product and the depulping followed by drying has to be performed within 3 to 4 days to maintain its oil quality [7]. It is reported that 50% of the neem seeds collected have been wasted as there is no attention among researchers and there is a lack of depulping facilities. The manual processing of neem fruits is laborious and time-consuming and the quality of the oil is greatly affected [1]. Timely depulping and drying are required to get a good quality oil.

Previous works are available on the aspects of different methods for extracting oil from neem seeds and analysing the azadirachtin content. However, to the best of our knowledge there is no study regarding the effect of different drying methods on azadirachtin content, oil yield, and quality parameters of neem oil and seed cake. To address this limitation and to reduce the wastage of neem seeds, this study has been attempted to dry the neem seeds using different drying methods with the following objectives:

(1) To study the drying characteristics of neem seeds under different drying methods

(2) To analyse and compare the effect of drying methods on the quality of neem seeds

2. Material and Methods

2.1. Collection of Neem Fruits. Fresh neem fruits were collected from Forestry College and Research Institute, Mettupalayam, India, and were used for the oil production. The pulp of the fresh neem fruits (38% w.b.) was removed using the single drum neem fruit depulper (capacity: 125 kg/h and depulping efficiency: 94.29%) developed by the Department of Food Process Engineering, Tamil Nadu Agricultural University (TNAU), India.

2.2. Drying Studies. During March 2022, 50 kg of depulped neem seeds having a moisture content of 30% (w.b.) were sun-dried (2 days) at an average temperature of $35 \pm 5^{\circ}$C and $45 \pm 5\%$ RH. Similarly, 100 kg of seeds were dried in a compound parabolic collector (CPC) solar dryer at $45 \pm 5^{\circ}$C and a tray dryer at 40 ± 2, 50 ± 2 and $60 \pm 2^{\circ}$C until they reached a final moisture content of 8% (w.b.). The weight of the neem seeds was measured using an electronic weighing balance (Ohaus Corporation, NJ, USA) with an accuracy of ±0.01 g at every one-hour interval. The drying characteristics of the neem seeds and the quality of the oil obtained from the neem seeds were evaluated.

2.2.1. Open Sun Drying. Neem seeds were dried under the open sun from 9.00 a.m. to 5.00 p.m. Since black painted trays ($41 \times 81 \times 4$ cm) absorb more heat, they were used instead of regular trays. Ten kilograms of fresh neem seeds with an initial moisture content of 30% (w.b.) were kept in black-painted trays in the open sun until they reached a moisture content of 8% (w.b.).

2.2.2. Solar Drying. Solar drying of neem seeds was carried out in a CPC type of solar dryer (1.98 m dia. × 2.30 m height with a capacity of 100 kg) provided with temperature and humidity controllers available in the Department of Renewable Energy Engineering, TNAU, India. The experiment was carried out between 9.00 am and 5.00 pm.

2.2.3. Tray Drying. Depulped neem seeds with a moisture content of 30% (w.b.) were dried in a tray dryer (M/s. Teqto Scientific, Coimbatore) available at the Department of Food Process Engineering, AEC & RI, TNAU, India. The constant set temperature of $40 \pm 2^{\circ}$C, $50 \pm 2^{\circ}$C, and $60 \pm 2^{\circ}$C were used in the tray dryer for drying 50 kg of neem seeds until it reached 8% (w.b.) moisture content.

2.3. Estimation of Azadirachtin Content. The azadirachtin content in fresh and dried neem seeds was estimated using the high-performance liquid chromatography method described by Kaushik [8]. Hexane was used as a solvent to extract oil from the seed kernel powder and ethanol was used to extract azadirachtin. Azadirachtin was separated using acetonitrile-water (40 : 60) at a rate of 1 $\mathrm{mL{\cdot}min^{-1}}$ with a monitoring peak of 214 nm. 10 μL of the sample was injected into the HPLC using an autoinjector, and helium was used as a degassing agent.

2.4. Oil Yield. To extract oil from the dried neem seeds, a standardised oil extraction method using a Soxhlet apparatus (NF ISO 734-1) was used. The oil yield percentage was calculated using the following equation as mentioned by Tesfaye and Tefera [9]:

$$\mathrm{OY}\,(\%) = \frac{W_{\mathrm{IN}} - W_{\mathrm{FI}}}{W_{\mathrm{IN}}} \times 100, \tag{1}$$

where OY represents the oil yield, %. W_{IN} and W_{FI} represent the initial weight of the sample placed in the thimble and the final sample weight of the dried sample in the oven, g.

2.5. Physiochemical Properties of Oil. The specific gravity was determined using the AOAC method (1984). Standardized methods were used to determine the density (AFNOR T60−214), viscosity (ASTM D−445), iodine value (AFNOR T60−203), and peroxide value (AFNOR T60−220). The acid and saponification values were determined using the AOAC recommended methods (1990). The color value (lightness (L^*),

Table 1: Experimental design for the optimization of azadirachtin content and oil yield.

Variable	Independent variable				Dependent variable
Number	Temperature, °C			Bed thickness, mm	(1) Azadirachtin content, % (2) Oil yield, %
Levels	Sun drying (35 ± 5)	Solar drying (45 ± 5)	Tray drying (40 ± 2, 50 ± 2 and 60 ± 2)	15 ± 1 and 30 ± 1	

redness to greenness (a^*), yellowness to blueness (b^*), and ΔE) of the extracted neem oil was determined using a Lovibond Tintometer (Lovibond, LC 100, The Tintometer Ltd., UK).

2.6. Particle Size Analysis. Fresh and dried neem seeds were powdered using a ball mill (Yatherm Scientific, India). The particle size analyser (Horiba Scientific, SZ-100) was used to measure the particle size of the powdered samples (fresh and dried neem seeds).

2.7. FTIR Analysis. FTIR (fourier-transmission infrared) spectrometer (model number: FT/IR-6800, incident angle: 45°, detector: TGS, accumulation: 64, resolution: 4 cm^{-1}, zero filling: on, apodization: cosine, gain: auto (8), aperture: auto (7.1 mm), scanning speed: auto (2 mm/sec), and filter: auto (10000 hz)) was used to analyse the functional groups present in the samples. It consisted of a source, interferometer, beam splitter, fixed mirror, movable mirror, sample holder, and a detector. FTIR sampling procedure was used to analyse the functional groups in powdered fresh and dried neem seeds as mentioned by Elzey et al. [10].

2.8. Elemental Analysis. Inductively coupled plasma mass spectrometer (model: Thermo Scientific™ ICAP™ RQ, type: single quadrupole ICP-MS, hertz: 2 MHz, nebulizer: borosilicate glass, and spray chamber: quartz, cyclonic) was used to analyse the elements present in the samples. The elemental composition of 7 Li (lithium), 24 Mg (magnesium), 39 K (potassium), 48 Ti (titanium), 51 V (vanadium), 52 Cr (chromium), 55 Mn (manganese), 57 Fe (iron), 59 Co (cobalt), 60 Ni (nickel), 75 As (arsenic), 133 Cs (cesium), 63 Cu (copper), 23 Na (sodium), 44 Ca (calcium), 11 B (boron), 121 Sb (antimony), 31 P (phosphorus), 66 Zn (zinc), 95 Mo (molybdenum), 111 Cd (cadmium), 118 Sn (tin), and 208 Pb (lead) in fresh and dried neem seeds were evaluated using the standard ICPMS procedure as mentioned by Novotnik et al. [11].

2.9. Statistical Optimization. The experimental data were statistically analysed using the central composite design (CCD) technique from Design Expert Software (Version 13.0) and SPSS 2020 software (IBM Corporation, New York, USA). The regression coefficients were calculated using the quadratic model. The analysis of variance (ANOVA) was used to identify the model's significant terms. Drying methods and bed thickness were investigated for their effects on azadirachtin content and oil yield. Table 1 shows the dependent and independent variables used in the experimental design.

3. Results and Discussion

3.1. Drying Studies. The drying characteristics curve (moisture content and drying time) of neem seeds dried in a tray dryer, solar dryer, and sun drying is shown in Figures 1(a)–1(c)–3. The moisture content of neem seeds decreased from 30% to 8% (w.b.) in 300 and 240 minutes of tray drying, respectively, a with bed thickness of 30 and 15 mm at a constant temperature of 60 ± 2°C. It was also noted that there was an increase in the drying time of 480 and 420 minutes at 30 and 15 mm bed thicknesses when the temperature was reduced to 50 ± 2°C. Furthermore, a decrease in temperature to 40 ± 2°C resulted in an increased drying time of 660 and 590 minutes, at 30 and 15 mm bed thicknesses for drying neem seeds from 30% to 8% (w.b.) moisture content.

In solar drying, the time required to dry neem seeds with a bed thickness of 30 and 15 mm from an initial value of 30% (w.b.) to a final value of 8% (w.b.) was recorded at 540 and 420 minutes, respectively, with the maximum recorded temperature of 50 ± 5°C. Corresponding to sun drying with the same two-bed thicknesses, resulted in a maximum drying time of 780 and 630 minutes to dry neem seeds to 8% (w.b.) moisture content with the maximum recorded temperature of 35 ± 5°C.

It is clear that a higher temperature of 60 ± 2°C reduced the drying time to 300 minutes, whereas sun drying at a low temperature of 35 ± 5°C resulted in the longest drying time of 780 minutes. This is because of higher heat transfer at higher temperatures [12, 13]. Higher vaporisation of water also resulted in faster drying of neem seeds [14].

Figures 4(a)–4(c) to 6 show the drying rate curve of neem seeds dried in a tray dryer, solar dryer, and sun drying. Initially, tray-dried neem seeds at 40, 50, and 60°C showed a drying rate of 0.02 ± 0.01–0.049 ± 0.01 (g/g. min) for different bed thicknesses (15 and 30 mm) until critical moisture content was reached, and then it showed a drying rate from 0.002 ± 0.001 to 0.007 ± 0.001 (g/g. min), respectively. The constant high temperature in tray drying caused faster drying. The drying rate increased as the drying temperature and time increased.

Similarly, an initial drying rate of 0.036 ± 0.01–0.041 ± 0.01 (g/g. min) was observed for solar-dried neem seeds under 15 and 30 mm bed thicknesses, respectively, until a critical moisture content was reached. It showed a falling drying rate of 0.0027 ± 0.0001 (g/g. min) in solar drying after 6 hours. Likewise, the sun-dried neem seeds showed an initial drying rate of 0.022 ± 0.01–0.028 ± 0.01 (g/g. min) followed by a falling drying rate of 0.0006 ± 0.0001 (g/g. min) after 6 hours. This decrease in drying rate was due to the subsurface removal of water from the neem seeds at a lower rate.

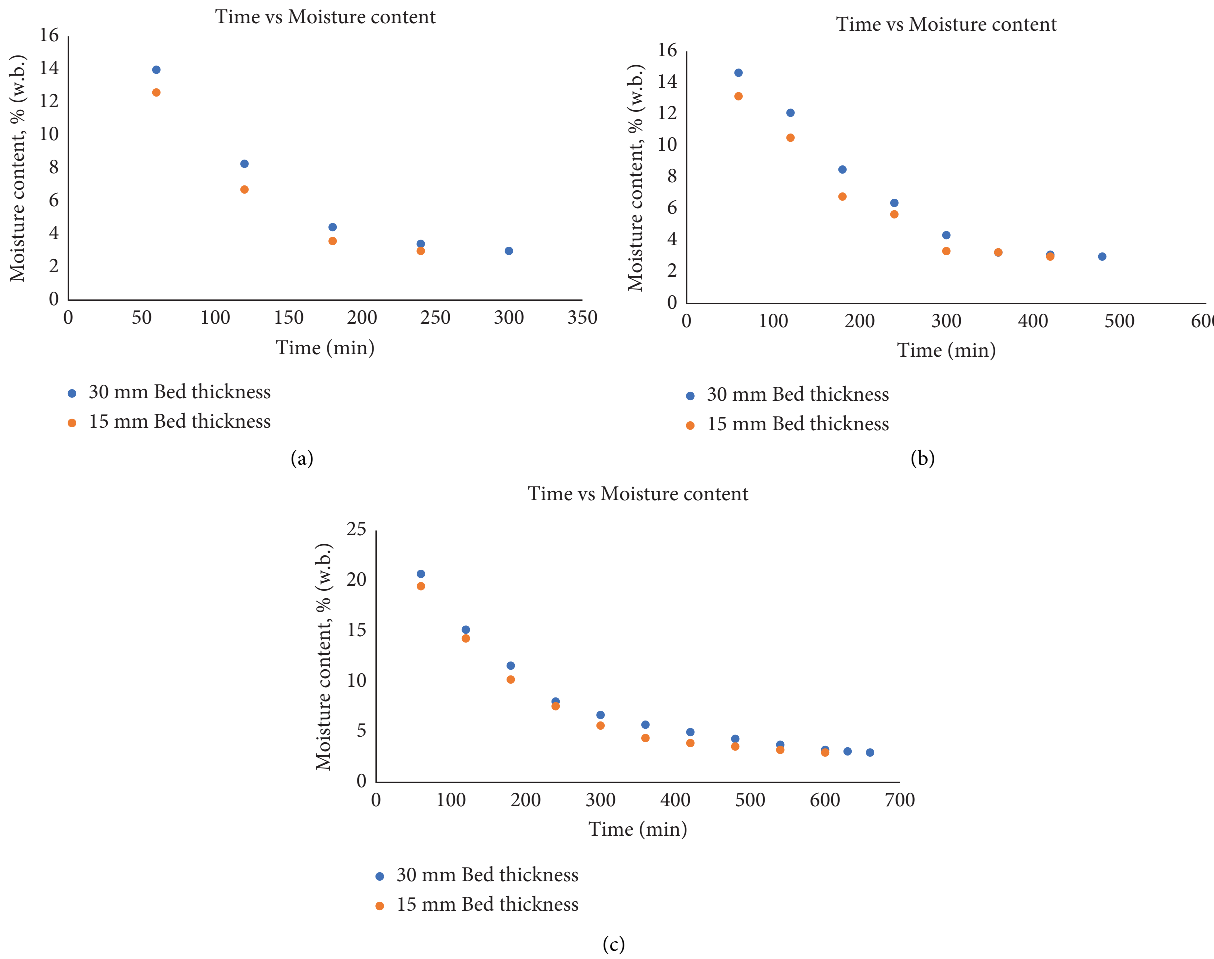


Figure 1: (a) Moisture content of neem seeds under tray drying at 60°C. (b) Moisture content of neem seeds under tray drying at 50°C. (c) Moisture content of neem seeds under tray drying at 40°C.

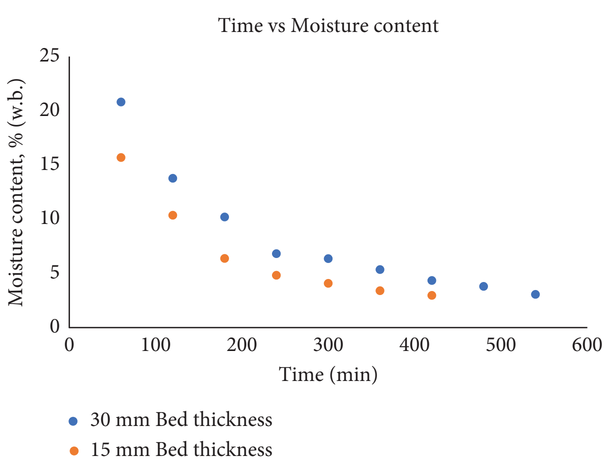


Figure 2: Moisture content of neem seeds under solar drying.

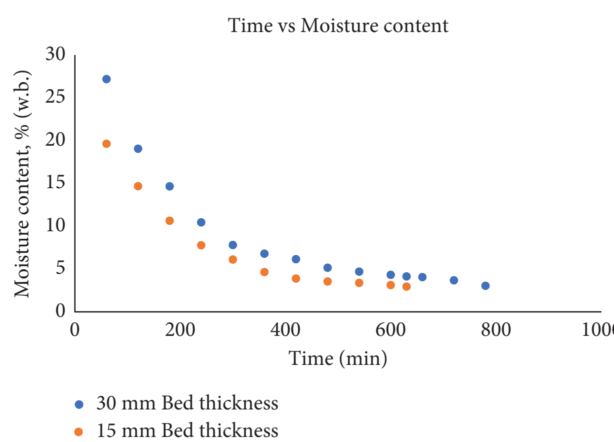


Figure 3: Moisture content of neem seeds under sun drying.

Similar results were obtained by Bhaskara Rao and Murugan [15] for neem leaves drying, Firdissa et al. [16] for Arabica coffee variety drying, Curtis et al. [17] for galip nuts drying, and Olalusi et al. [18] for drying of locust beans Figure 5.

3.2. Effect of the Drying Method and Bed Thickness on Azadirachtin Content. Figures 7(a) and 7(b) show the effect of drying methods and bed thickness on the azadirachtin content of neem seeds. Azadirachtin content of fresh neem seeds was found to be 0.057%. It was observed that the azadirachtin content ranged between 0.040 and 0.054% at

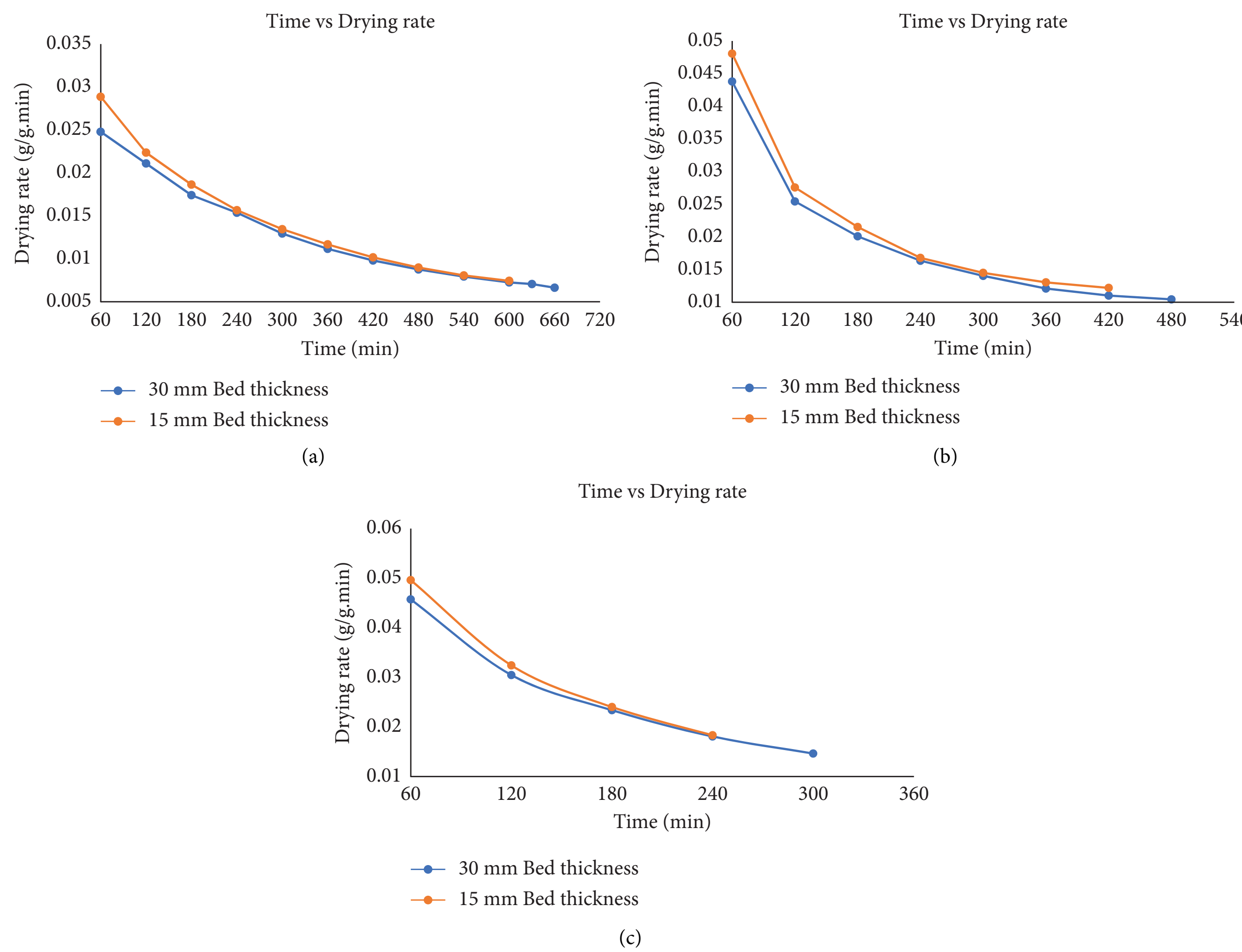


Figure 4: (a) Drying rate of neem seeds under tray drying at 40°C. (b) Drying rate of neem seeds under tray drying at 50°C. (c) Drying rate of neem seeds under tray drying at 60°C.

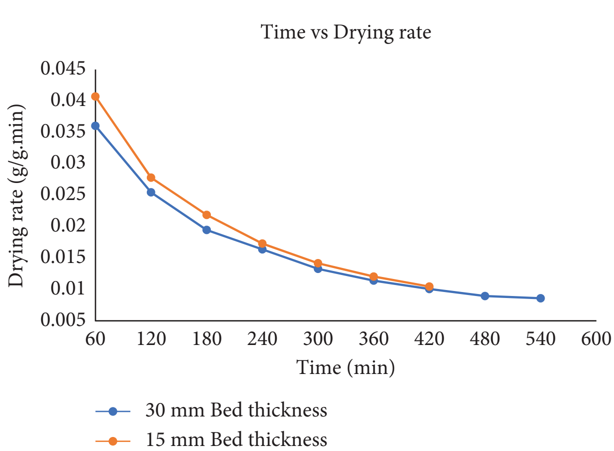


Figure 5: Drying rate of neem seeds under solar drying.

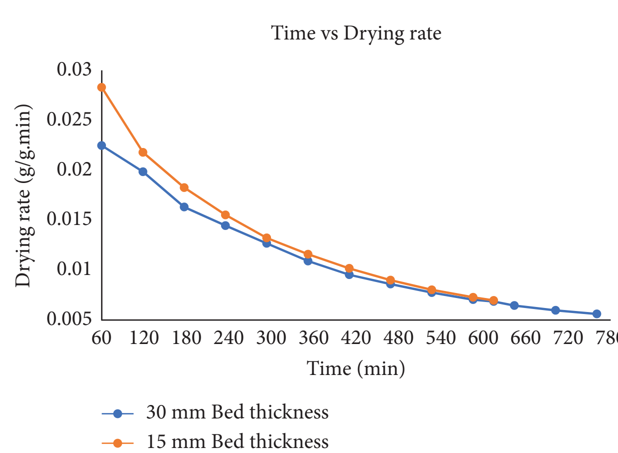
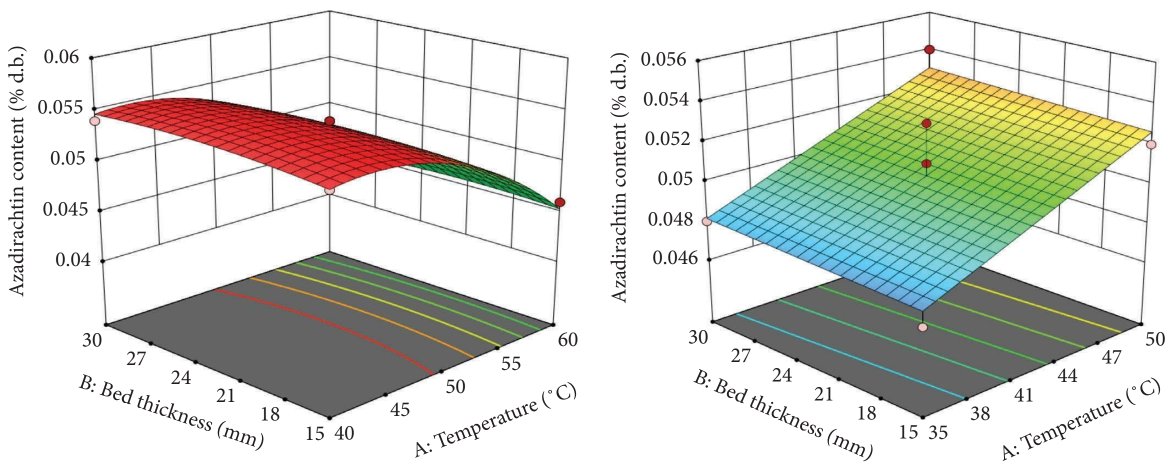


Figure 6: Drying rate of neem seeds under sun drying.

different combinations of drying methods and bed thicknesses. Minimum azadirachtin content in the range of 0.040–0.046% was obtained in the tray drying method at temperature of 60 ± 2°C and a bed thickness of 15 mm. It was noted that with an increase in temperature above 50 ± 2°C in tray drying, a decrease in azadirachtin content was observed. This might be due to the exposure of neem seeds to more heat which resulted in the reduction of the azadirachtin content.

Similar results were reported by Bhaskara Rao and Murugan [15] for neem leaves drying and Firdissa et al. [16] for Arabica coffee variety drying. Maximum temperature of up to 50 ± 2°C in tray drying resulted in azadirachtin content



(a) (b)

Figure 7: (a) Effect of tray drying and bed thickness on azadirachtin content. (b) Effect of drying methods (sun and solar drying) and bed thickness on azadirachtin content.

in the range of 0.049–0.054%. Decreasing the bed thickness to 15 mm, resulted in the azadirachtin content of 0.046–0.051% for different combinations of tray drying with the temperature of 40 ± 2 and $50 \pm 2^{\circ}$C. Azadirachtin content of 0.040–0.049% and 0.048–0.054% was obtained in sun and solar drying at the temperatures of 35 ± 5 and $45 \pm 5^{\circ}$C with 15 and 30 mm bed thicknesses, respectively.

Tables 2 and 3 show the F value of the statistically analysed azadirachtin content of neem seeds dried under different drying methods (sun, solar, and tray drying) and bed thicknesses. The R^2 value was found to be 0.988 for the different combinations of tray drying (40, 50, and 60°C) and bed thickness (15 and 30 mm). Whereas, sun and solar drying with 15 and 30 mm bed thickness had an R^2 value of 0.712. The suggested quadratic model revealed that different drying methods and bed thickness had a significant effect on azadirachtin content ($p \le 0.05$). The azadirachtin content was statistically analysed and mentioned in the following equations:

$$\text{Azadirachtin content (\%)} = +0.0536 - 0.0046A + 0.0001B - 0.0002AB - 0.0033A^2 - 0.0006B^2, \quad (2)$$

$$\text{Azadirachtin content (\%)} = +0.050 + 0.0024A + 0.0001B. \quad (3)$$

3.3. Effect of the Drying Method and Bed Thickness on Oil Yield. The effect of drying methods and bed thickness on oil yield is shown in Figures 8(a) and 8(b). It was observed that a minimum oil yield of 33–37% was obtained at a minimum temperature of 35 ± 5 and $40 \pm 2^{\circ}$C in sun and tray drying with a maximum bed thickness of 30 mm, respectively. This decrease in oil yield might be due to less exposure of heat to the neem seeds. Increasing the temperature and decreasing the bed thickness to $50 \pm 2^{\circ}$C and 15 mm in tray drying resulted in an increase in oil yield from 40.3 to 42.5%. The solar ($45 \pm 5^{\circ}$C) and tray drying (50 ± 2 and $60 \pm 2^{\circ}$C) with bed thickness of 15 mm resulted in a maximum oil yield of 42.5–45.6%, respectively.

Similar work of oil extraction from neem seeds by Tesfaye and Tefera [9] and Djibril et al. [19] showed an oil yield of 43.71% and 48.98 ± 0.34%, respectively. A little lower oil yield of 32.5% was obtained in neem seed kernels using 50% of ethanol as solvent by Saha et al. [20]. Also, a maximum oil yield of 53.5% was obtained by Subramanian et al. [21] at an optimum condition of extraction time (6 h) using a solvent mixture of 50 : 50 (n-hexane : ethanol).

The combination of drying methods and bed thickness showed a significant effect at ($p \le 0.05$) on oil yield with an R^2 value of 0.981 and 0.966 using the suggested quadratic model as shown in Tables 2 and 3. The obtained data were statistically analysed and are represented by the following equations:

$$\text{Oil yield (\%)} = +40.92 + 4.34A - 2.19B + 0.7750\text{AB} - 0.6350A^2 - 0.9100B^2, \quad (4)$$

$$\text{Oil yield (\%)} = +41.52 + 5.50A - 1.81B + 0.025\text{AB} - 1.81A^2 - 1.33B^2. \quad (5)$$

3.4. Optimization of Process Parameters. The experimental results showed a maximum azadirachtin content of 0.050–0.054% at different combinations of drying methods and bed thicknesses (solar and tray drying with 15 and 30 mm). The central composite design was used to find the optimum combination of temperature and bed thickness, which resulted in maximum azadirachtin content and oil yield. The RSM showed that the maximum azadirachtin content and oil yield of 0.052 and 0.054%, 42.1, and 45.6%, respectively was obtained under solar ($45 \pm 5^{\circ}$C) and tray drying ($50 \pm 2^{\circ}$C) at a bed thickness of 15 mm with

Table 2: Analysis of variance of effects of different combinations of tray drying and bed thickness on azadirachtin content and oil yield.

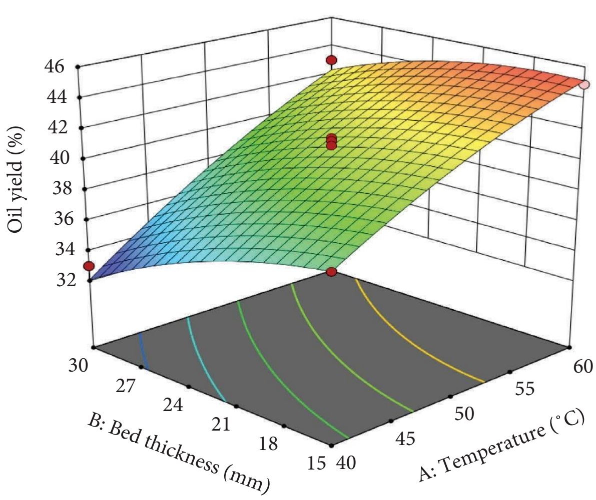
Source	Degrees of freedom	Azadirachtin content (%) *F* value*	Oil yield (%) *F* value
Model	5	118.15	72.36
Temperature (°C) (*A*)	1	407.48	273.89
Bed thickness (mm) (*B*)	1	0.0516	69.64
AB	1	0.6018	4.37
A^2	1	182.37	5.10
B^2	1	5.07	10.47

* *F*value-ANOVA coefficient (it is the ratio between the mean sum of squares between the groups to the mean sum of squares within the groups).

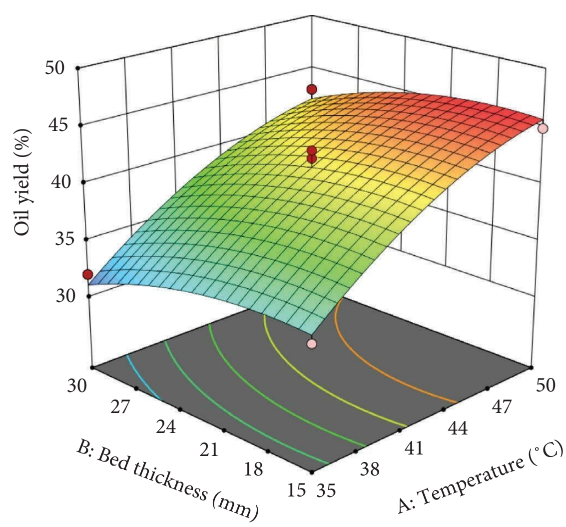
Table 3: Analysis of variance of effects of different combinations of drying methods (sun and solar drying) and bed thickness on azadirachtin content and oil yield.

Source	Degrees of freedom	Azadirachtin content (%) *F* value*	Oil yield (%) *F* value
Model	5	12.37	37.97
Temperature (°C) (*A*)	1	24.59	153.26
Bed thickness (mm) (*B*)	1	0.1628	16.69
AB	1	—	0.0016
A^2	1	—	14.46
B^2	1	—	7.87

* *F*value-ANOVA coefficient (it is the ratio between the mean sum of squares between the groups to the mean sum of squares within the groups).



(a)



(b)

Figure 8: (a) Effect of tray drying and bed thickness on oil content. (b) Effect of drying methods (sun and solar drying) and bed thickness on oil yield.

a desirability value of 0.88 and 0.93 and was found to be statistically significant at $p \leq 0.05$. The optimum combination of azadirachtin content and oil yield is shown in Figure 9.

3.5. Quality Analysis. Quality analyses were carried out for the dried neem seeds and oil obtained from different drying methods i.e., sun drying (35 ± 5°C), solar drying (45 ± 5°C), and tray drying (50 ± 2°C). Since the optimized drying combination of tray drying (50 ± 2°C with 15 mm bed thickness) showed maximum azadirachtin content and oil yield, this combination was used for quality analysis.

3.6. Physiochemical Properties of Oil. Figures 10(a)–10(e) show the physicochemical properties of oil obtained from the sun-dried, solar-dried, and tray-dried neem seeds. The oil obtained from the tray-dried neem seeds had a higher specific gravity (0.93 ± 0.008), acid value (8.3 ± 0.18 mg/g), saponification value (189.4 ± 2.90 KOH/g), viscosity (45.21 ± 1.25 mm^2/s), and peroxide value (1.45 ± 0.023 meq of O_2/kg), respectively. When compared to the solar- and sun-dried neem seed oil, the higher temperature in the tray dryer caused lipid hydrolysis, which resulted in oil degradation and indirectly increased acid and peroxide value [9]. The constant set temperature in the tray dryer resulted in

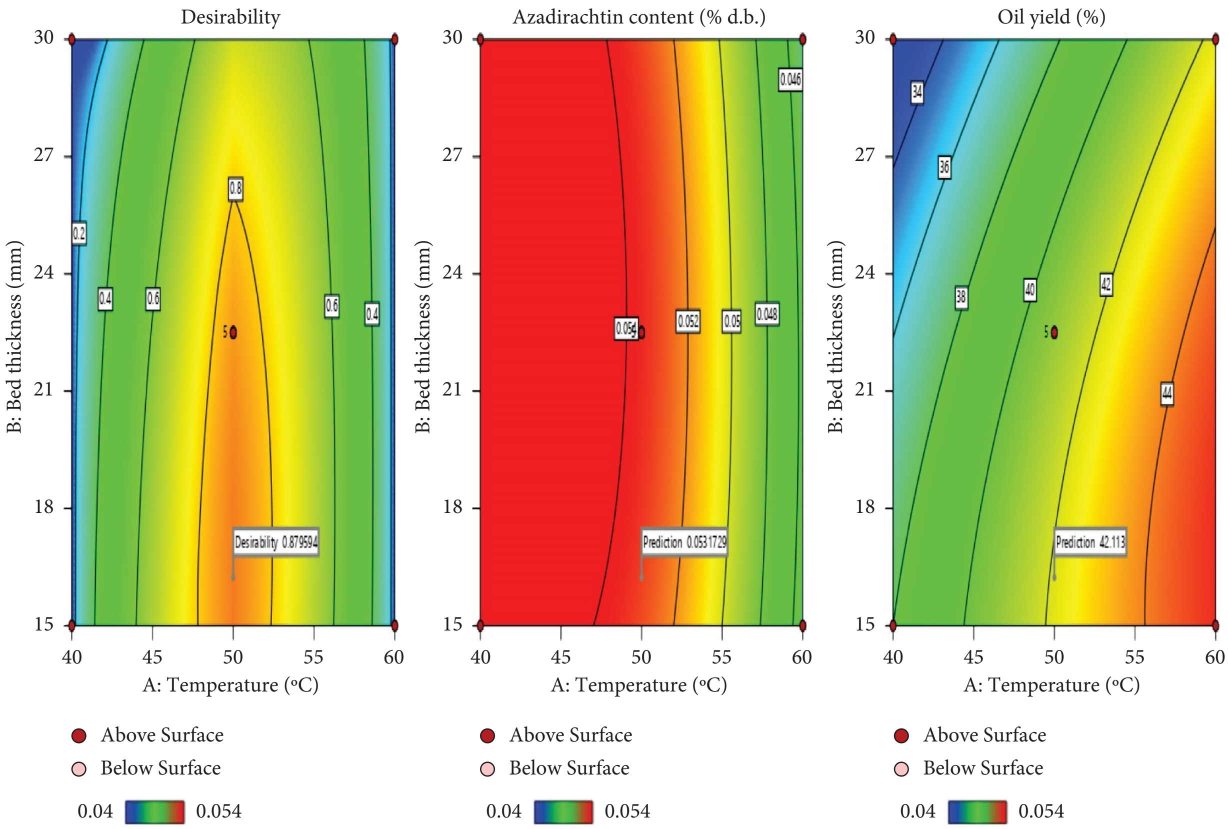


Figure 9: Optimum combination of azadirachtin content and oil yield.

lipid breakdown and a reduction in the molecular weight of the triglycerides present in the oil, resulting in an increased saponification value [9]. The oil of the tray-dried neem seeds was found to be darker than that of the solar- and sun-dried neem seeds.

Similar studies were carried out by Tesfaye and Tefera [9], and they obtained the minimum acid value (14.46 mg KOH/g), saponification value (194.48 mg KOH/g), specific gravity (0.90), and pH (4.86). Also, Djibril et al. [19] found that neem oil had an acid value of 10.2 $mg{\cdot}g^{-1}$, a saponification value of 200 $mg{\cdot}g^{-1}$, and an iodine value of 72.8 g. 100 g^{-1} for neem seed. The variations in the quality and quantity of neem oil obtained might be due to the varietal differences and extraction methods.

The color values in terms of L^*, a^*, and b^* were found to be 53.9 ± 0.127, 12.8 ± 0.24, and 35.9 ± 0.42 for sun-dried neem seeds, 36.7 ± 1.173, 10.54 ± 0.344, and 20.21 ± 0.3163 for solar-dried neem seeds, and 26.5 ± 0.143, 7.4 ± 0.153, and 6.3 ± 1.104 for tray-dried neem seeds, respectively. The effect of drying methods on the color value of neem seed oil was statistically significant at $p \leq 0.01$. Due to the increased heat exposure, the L^* value of neem oil decreased as the drying temperature increased [22].

3.7. Particle Size Analysis. Table 4 shows the particle size analysis of fresh and dried powdered neem seed kernels under different drying methods. The particle size of the powdered fresh, sun-, solar-, and tray-dried neem seeds was recorded as 711.5 ± 92.2, 265.3 ± 26.1, 662.0 ± 81.1, and 1335.5 ± 273.3 nm, respectively. The particle size of the tray-dried neem seed powder was found to be higher than the solar- and sun-dried seed powders. This is because of the uniform drying of neem seeds in the tray drying method. Similar to the present work, Shewale and Rathod [23] analysed the different particle sizes of neem leaf powder (0.1–0.2, 0.2–0.3, and 0.3–0.4 mm) to study the effect of drying methods on the phenolic contents of neem. Also, Tesfaye and Tefera [9] analysed the powder size of neem seed (355 μm) for extracting neem oil using the Soxhlet extraction methods. The particle size is also equally important for the effective oil extraction.

3.8. FTIR Analysis. Figure 11 shows the results of the FTIR analysis of powdered fresh and dried neem seeds. It showed the presence of different functional groups in different absorption wave numbers and it was found to be varied among samples based on the drying method opted. The total number of functional groups found in the seeds is 6. Fresh neem seeds showed peaks at a wavenumber of 2970.44, 1737.73, 1375.12, 1220.27, 904.42, and 518.05 cm^{-1}, respectively. Similarly, sun-dried neem seeds showed peaks in the wavelength of 2928.27, 1732.36, 1375.12, 1214.13, 1005.62, and 535.68 cm^{-1}. The solar-dried neem seeds exhibited the peaks at the wavenumber of 2916.78, 1743.86, 1375.12, 1214.13, 1029.38, and 518.05 which was followed by the tray-dried neem seeds at 2910.64, 1732.36, 1380.49,

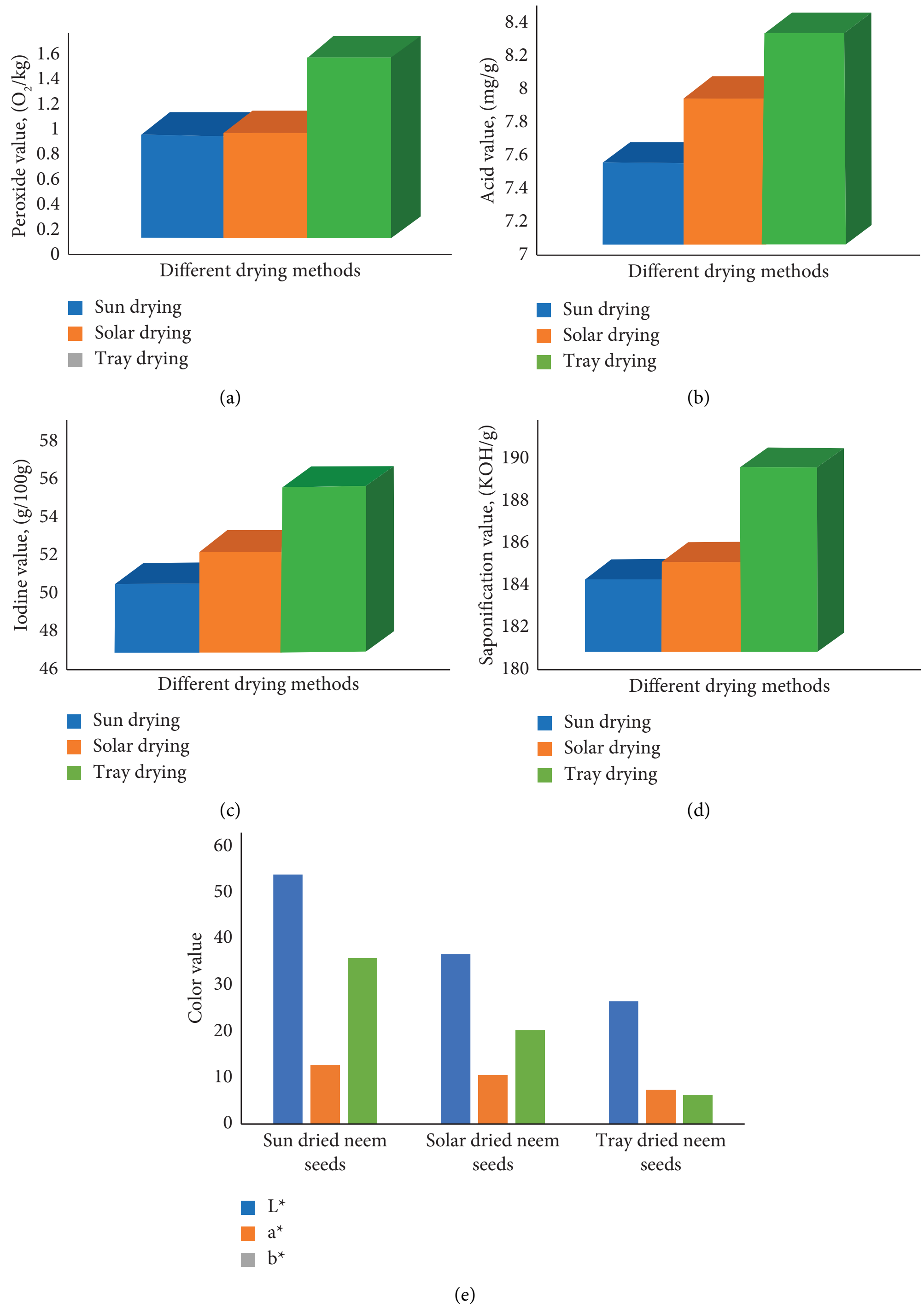


Figure 10: (a) Peroxide value of neem oil under sun, solar, and tray drying. (b) Acid value of neem oil under sun, solar, and tray drying. (c) Iodine value of neem oil under sun, solar, and tray drying. (d) Saponification value of neem oil under sun, solar, and tray drying. (e) Color value of neem oil under sun, solar, and tray drying.

1214.13, and 541.82 cm^{-1}. The spectra of FTIR verified the occurrence of amines, aromatic compounds, carboxylic acid, amino acids, alkyl halide, alkanes, and phenols in the seeds.

The peak absorption wavelength of 2910.64, 2916.78, 2928.27, and 2970.44 represents the C-H stretching vibration modes in the hydrocarbon chain which was observed in fresh, sun-dried, solar-dried, and tray-dried neem seeds. Whereas, the peak values at 1737.73 cm^{-1} matching with the stretching vibrations of the C=O in carboxylic acids, aldehydes, and ketones were found in tray-dried neem seeds and at 1732.36 cm^{-1} in solar-dried neem seeds, and at 1743.86 cm^{-1} in sun-dried neem seeds compared to the fresh neem seeds with a peak value of 1732.36, respectively [24].

Table 4: Particle size analysis of powdered neem seed kernels under different drying methods.

S. No	Method of drying	Particle size of neem seed powder (nm)
1	Fresh neem seeds	711.5 ± 92.2
2	Sun-dried	265.3 ± 26.1
3	Solar-dried	662.0 ± 81.1
4	Tray-dried (50 ± 2°C with 15 mm bed thickness)	1335.5 ± 273.3

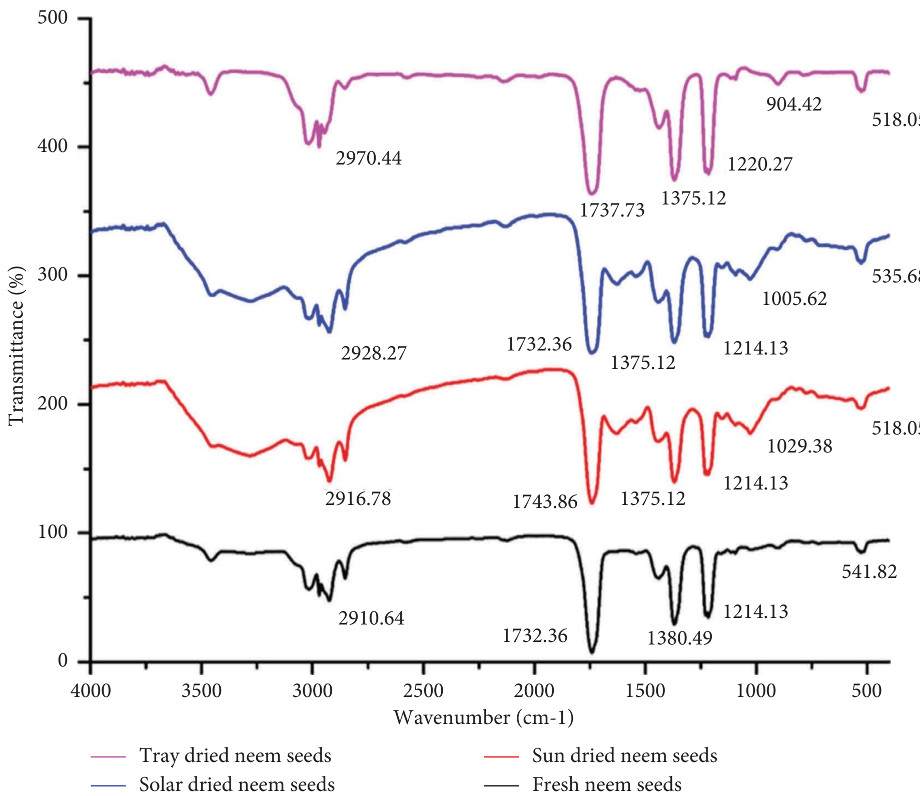


Figure 11: FTIR analysis of powdered neem seeds.

The absorption bands of dried neem seeds in the range of 1375 cm^{-1} were designated to the frequency stretching that comes from the C-O bonds of acetyl esters, C=O, CH_2 wagging, and C-H stretching compared to the peak value of fresh neem seeds at 1380.49 cm^{-1}. It was reported that the aliphatic C-C position occurred in the range of 1214.13–1220.27 cm^{-1} and frequency values of around 1005.62 cm^{-1} in neem seeds was the stretching vibration C-O. The FTIR spectra verified the occurrence of halides, aliphatic amines, aromatic, carboxylic groups, amides, alkanes, and alkenes. Similar to the present study, the presence of the terpenoid group was confirmed with the same level of peak by Senthilkumar and Sivakumar [25].

Similar studies of FTIR spectroscopy on the determination of the composition of adulterated neem and flaxseed oil, performed by Elzey et al. [10] showed that 2900, 1700, and 1100 cm^{-1} confirmed the presence of C-H stretching, C=O stretching, and C-O stretching. Correspondingly, Iqbal et al. [26] studied FTIR analysis for highly stabilized neem oil and mixture (neem and grass oil) emulsion and found that the wavenumber of 1642 and 2928 cm^{-1} was not changed when neem oil was added with grass oil which confirmed that there is no interaction between these oils and other ingredients.

3.9. Elemental Analysis. Elemental compositions such as Li, Mg, K, Ti, V, Cr, Mn, Fe, Co, Ni, As, Ce, Cu, Na, Ca, B, Sb, P, Zn, Mo, Cd, Sn, and Pb are shown in Figure 12.

Increases in the percentage of 117.51, 19.791, 20.662, 20, 15.404, 25.011, 16, 166.667, 192.727, and 10.417 ppm were observed in the elemental composition of Na, Ca, B, Sb, P, Zn, Mo, Cd, Sn, and Pb. The elemental composition was found to be 92.113 ± 0.87, 1359.02 ± 24.04, 4.922 ± 0.137, 0.005 ± 0.0001, 927.7 ± 20.19, 18.636 ± 0.35, 0.075 ± 0.0007, 0.009 ± 0.0002, 0.055 ± 3.742, and 0.432 ± 0.0061 ppm for fresh neem seeds, respectively. The minimum elemental composition of 78.773 ± 0.64, 1442.79 ± 14.72, 5.939 ± 0.044, 0.006 ± 0.34,

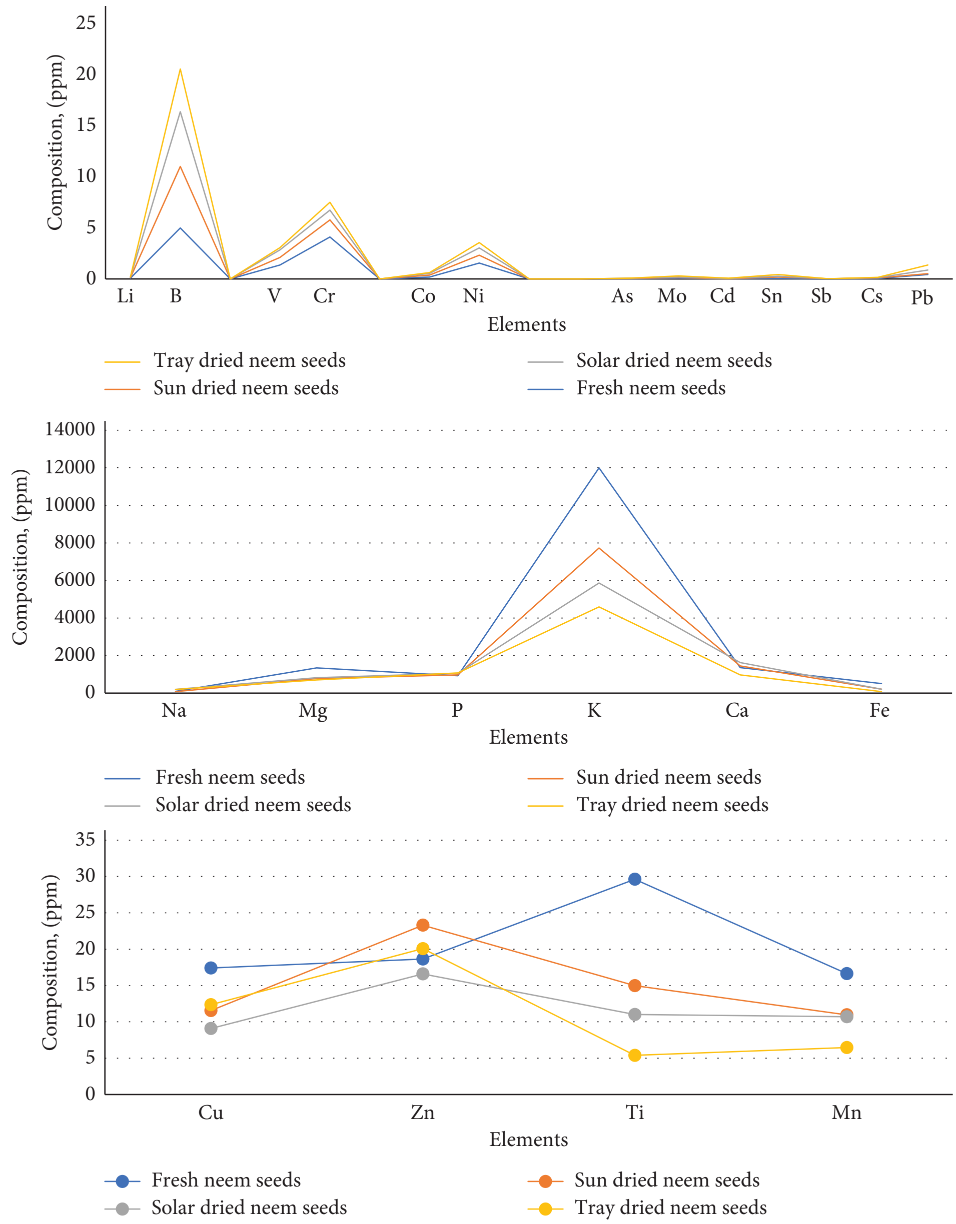


FIGURE 12: Elemental composition of fresh and dried neem seeds.

964.602 ± 14.43, 23.297 ± 0.634, 0.063 ± 0.0003, 0.023 ± 0.0006, 0.136 ± 0.003, and 0.0675 ± 0.002 ppm were obtained for sun-dried neem seeds. Solar-dried neem seeds showed maximum retention in the elemental composition of 200.356 ± 2.95, 1627.99 ± 34.33, and 5.289 ± 0.028 for Na, Ca, and B. The elemental composition of Sb, P, Zn, Mo, Cd, Sn, and Pb was found to be maximum for tray-dried neem seeds with 0.004 ± 6.25, 1070.6 ± 5.82, 20.076 ± 0.150, 0.087 ± 0.0002, 0.024 ± 0.001, 0.161 ± 0.0004, and 0.477 ± 0.0021 ppm, respectively. This increase in composition was observed since the seeds were concentrated during drying by the removal of water.

Similar studies by Novotnik et al. [11] on neem powder showed that trace elements, namely, Al, Fe, Co, Cu, Ni, Cd, V, Zn, As, Se, M, Pb, and Cr were present in the composition of 3100 ± 10, 1510 ± 40, 1.56 ± 0.02, 19.9 ± 0.2, 17.8 ± 0.8, 0.034 ± 0.001, 9.3 ± 0.4 29, 16.2 ± 0.7, 0.63 ± 0.02, 0.54 ± 0.01, 1.57 ± 0.06, 4.6 ± 0.1, and 197 ± 5 $mg{\cdot}kg^{-1}$, respectively. Correspondingly, Zhang et al. [27] mentioned that 17 trace elements, namely, B, Na, Ca, Cr, Cd, Cu, Fe, Se, Pb, Al, Mn, Ni, As, Mg, P, K, and Zn was recorded with the composition of 18.86 ± 0.08, 10215 ± 248, 6967 ± 338, 1.75 ± 0.4, 0.024 ± 0.001, 2.7 ± 0.1, 107 ± 20, 0.21 ± 0.00007, 0.257 ± 0.100, 101 ± 4.00, 17.40 ± 0.11, 0.92 ± 0.17, 0.066 ± 0.001, 2487 ± 67, 4873 ± 185, 15860 ± 947, and 26.4 ± 3.7 $\mu g{\cdot}g^{-1}$ in freeze-dried blueberry and the strawberry. The quality of neem oil depends on the elemental composition and the difference in content might be due to the variety, varying climatic conditions, and extraction techniques [28].

4. Conclusion

In this study, three different drying methods (sun drying, solar drying, and hot air at 40, 50, and 60°C) were applied to neem seeds and the effects of these drying methods on the azadirachtin content, functional groups, and elemental composition were studied. The drying methods significantly affected the oil yield and azadirachtin content of the neem samples. The optimum conditions for obtaining maximum azadirachtin and oil yield were found in solar drying at a bed thickness of 15 mm. Based on the study, there was a change in the quality of the oil for different drying methods and this might be because of an increase in temperature and drying time. Regarding the functional groups, FTIR results showed that there is no change in functional groups when the neem seeds are dried under different drying methods without allowing the functional compounds to degrade in the specified temperature ranges. Among the drying methods, sun-dried samples showed the least values of elemental composition while solar-dried and hot air-dried samples contributed to the maximum retention of elements. This study will be highly useful for food sectors that use neem oil in preservation, packaging, and storage.

5. Disclosure

The first author and third author presented the same work "Impact of different drying techniques on neem seeds drying kinetics and oil quality" in International Symposium "Agricultural Engineering Innovation for Global Food Security and India @2047: Agricultural Engineering Perspective" organized by Indian Society of Agricultural Engineers, New Delhi, and Agricultural Engineering College and Research Institute, Tamil Nadu Agricultural University, Coimbatore, India.

Data Availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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References

[1] M. A. Adedeji and O. K. Owolarafe, "Some physical properties of neem seeds & kernels (*Azadirachta indica*) as a function of moisture content," *Carbon*, vol. 86, pp. 78–92, 2015.

[2] S. Sanuja, A. Agalya, and M. J. Umapathy, "Synthesis and characterization of zinc oxide – neem oil – chitosan bio nanocomposite for food packaging application," *International Journal of Biological Macromolecules*, vol. 74, pp. 76–84, 2015.

[3] P. Kumar, N. Singh, L. S. Devi et al., "Neem oil and its nanoemulsion in sustainable food preservation and packaging: Neem oil and its nanoemulsion in sustainable food preservation and packaging: Current status and future prospectsurrent status and future prospects," *Journal of Agriculture and Food Research*, vol. 7, Article ID 100254, 2022.

[4] S. Dubhashi, V. Pranay, M. Singaiah, J. Satwik, V. V. L. N. Prasad, and P. V. Diwan, "Studies on extraction and HPLC analysis of azadirachtin from kernels of neem seeds," *Journal of Advanced Pharmacy Education & Research*, vol. 3, no. 1, pp. 27–30, 2013.

[5] S. H. Heroor and S. R. Bharadwaj, "Production of bio-fuel from crude neem oil and its performance," *International Journal of Environmental Engineering and Management*, vol. 4, no. 5, pp. 425–432, 2013.

[6] R. C. Solanki, S. N. Naik, S. Santosh, and A. P. Srivastava, "Design, development and evaluation of neem depulper," *Agricultural Mechanization in Asia Africa and Latin America*, vol. 48, no. 4, p. 46, 2017.

[7] N. D. Bup, E. N. Aweh, and I. N. Mbangsi, "Physical properties of neem (*Azadirachta indicaa. juss*) fruits, nuts and kernels," *Sky Journal of Food Science*, vol. 2, no. 8, pp. 14–23, 2013.

[8] N. Kaushik, "Determination of azadirachtin and fatty acid methyl esters of Azadirachta indica seeds by HPLC and GLC," *Analytical and Bioanalytical Chemistry*, vol. 374, no. 7-8, pp. 1199–1204, 2002.

[9] B. Tesfaye and T. Tefera, "Extraction of essential oil from neem seed by using soxhlet extraction methods," *International Journal of Advanced Engineering, Management and Science*, vol. 3, no. 6, pp. 646–650, 2017.

[10] B. Elzey, D. Pollard, and S. O. O. Fakayode, "Determination of adulterated neem and flaxseed oil compositions by FTIR spectroscopy and multivariate regression analysis," *Food Control*, vol. 68, pp. 303–309, 2016.

[11] B. Novotnik, T. Zuliani, J. Ščančar, and R. Milačič, "Content of trace elements and chromium speciation in Neem powder and tea infusions," *Journal of Trace Elements in Medicine and Biology*, vol. 31, pp. 98–106, 2015.

[12] G. Jeevarathinam, R. Pandiselvam, T. Pandiarajan et al., "Infrared assisted hot air dryer for turmeric slices: Infrared assisted hot air dryer for turmeric slices:Effect on drying rate and quality parametersffect on drying rate and quality parameters," vol. 144, Article ID 111258, 2021.

[13] D. S. A. Delfiya, K. Prashob, S. Murali et al., "Drying kinetics of food materials in infrared radiation drying: Drying kinetics of food materials in infrared radiation drying: A review review," *Journal of Food Process Engineering*, vol. 45, no. 6, Article ID e13810, 2022.

[14] R. Richa, N. C. Shahi, U. C. Lohani et al., "Design and development of resistance heating apparatus-cum-solar drying system for enhancing fish drying rate," *Journal of Food Process Engineering*, vol. 45, no. 6, Article ID e13839, 2022.

[15] T. S. Bhaskara Rao and S. Murugan, "Experimental investigation of drying neem (*Azadirachta indica*) in an evacuated tube solar dryer: performance, drying kinetics and characterization," *Solar Energy*, vol. 253, pp. 270–284, 2023.

[16] E. Firdissa, A. Mohammed, G. Berecha, and W. Garedew, "Coffee Coffee Drying and Processing Method Influence Quality of Arabica Coffee Varieties (Coffee arabica L.) at Gomma I and Limmu Kossa, Southwest Ethiopiarying and processing method influence quality of Arabica coffee varieties (*Coffee arabica L.*) at gomma I and limmu kossa, southwest Ethiopia," *Journal of Food Quality*, vol. 2022, Article ID 9184374, 8 pages, 2022.

[17] T. Curtis, R. Corkish, R. J. Fuller, and A. B. Sproul, *A mixed mode low profile solar tunnel dryer for canarium indicum nuts*, Deakin University, Geelong, Australia, 2015.

[18] A. P. Olalusi, M. O. Omosebi, and O. Y. Agbola, "Influence of drying methods on the drying characteristics and nutritional quality of fermented Locust beans," *International Journal of Environment, Agriculture and Biotechnology*, vol. 4, no. 6, pp. 1695–1703, 2019.

[19] D. Djibril, F. Mamadou, V. Gérard, M. D. C. Geuye, S. Oumar, and R. Luc, "Physical characteristics, chemical composition and distribution of constituents of the neem seeds (*Azadirachta indica A. Juss*) collected in Senegal," *Research Journal of Chemical Sciences*, vol. 3, no. 2, pp. 606–612, 2015.

[20] S. Saha, D. Singh, S. Rangari et al., "Extraction optimization of neem bioactives from neem seed kernel by ultrasonic assisted extraction and profiling by UPLC-QTOF-ESI-MS," *Sustainable Chemistry and Pharmacy*, vol. 29, Article ID 100747, 2022.

[21] S. Subramanian, R. T. Bachmann, and M. S. Hossain, "Simultaneous extraction and separation of oil and azadirachtin from seeds and leaves of Azadirachta indica using binary solvent extraction," *Natural Product Sciences*, vol. 25, no. 2, pp. 150–156, 2019.

[22] S. Turan, S. Keskin, and R. Solak, "Determination of the changes in sunflower oil during frying of leavened doughs using response surface methodology," *Journal of Food Science and Technology*, vol. 59, no. 1, pp. 65–74, 2022.

[23] S. Shewale and V. K. K. Rathod, "Extraction of total phenolic content from *Azadirachta indica* or (neem) leaves: kinetics study," *Preparative Biochemistry & Biotechnology*, vol. 48, no. 4, pp. 312–320, 2018.

[24] A. A. Oyekanmi, U. S. U. Kumar, N. G. Olaiya et al., "Functional Functional Properties of Antimicrobial Neem Leaves Extract Based Macroalgae Biofilms for Potential Use as Active Dry Packaging Applicationsroperties of antimicrobial neem leaves extract based macroalgae biofilms for potential use as active dry packaging applications," *Polymers*, vol. 13, no. 10, p. 1664, 2021.

[25] S. R. Senthilkumar and T. Sivakumar, "Green tea (Camellia sinensis) mediated synthesis of zinc oxide (ZnO) nanoparticles and studies on their antimicrobial activities," *International Journal of Pharmacy and Pharmaceutical Sciences*, vol. 6, no. 6, pp. 461–465, 2014.

[26] N. Iqbal, N. Kumar, A. Agrawal, and J. Kumar, "Development of highly stabilized neem oil microemulsion system: a green approach," *World Journal of Pharmaceutical Research, Microemulsion Formulation of Botanical Oils as an Efficient Tool to Provide Sustainable*, vol. 8, no. 3, pp. 1507–1517, 2019.

[27] H. Zhang, Z. Y. Wang, X. Yang et al., "Determination of free amino acids and 18 elements in freeze-dried strawberry and blueberry fruit using an Amino Acid Analyzer and ICP-MS with micro-wave digestion," *Food Chemistry*, vol. 147, pp. 189–194, 2014.

[28] P. Sudha and S. Ganga Kishore, "Impact of Different Drying Techniques on Neem Seeds Drying Kinetics and Oil Quality," *International Symposium on Agricultural Engineering Innovation for Global Food Security and India @2047: Agricultural Engineering Perspective*, Indian Society of Agricultural Engineers, New Delhi and Agricultural Engineering College and Research Institute, Tamil Nadu Agricultural University, p. 359, Coimbatore, India, 2022.