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Different drying methods alter the quality parameters of ethnobotanically important *Curcuma caesia* Roxb. rhizomes

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Abstract

Curcuma caesia Roxb. of the Zingiberaceae family, is an ethnobotanically important rhizomatous perennial herb and is commonly known as "Black turmeric". Like in other species of *Curcuma*, the pharmacologically important polyphenol curcumin is also found in *C. caesia*. In traditional medicine, *C. caesia* has been used to treat piles, leprosy, asthma, cancer, diabetes, fertility, toothache, vomiting, allergies, etc. Drying is considered the simplest and most cost-effective means of preserving raw plant materials, safeguarding the integrity of nearly all biologically active substances. Medicinal plant materials are primarily utilized in their dried forms in pharmaceutical industries. However, it has been observed that there is a lack of comprehensive information on the effects of different drying methods on quality parameters, and other pharmaceutically important aspects of *C. caesia.* Hence, here, we investigated the impact of various drying methods, such as direct sunlight exposure, solar drying, shade drying, and conventional oven drying with or without previous boiling, on the quality parameters of *C. caesia* rhizome, such as moisture, ash, and curcumin content. The results suggest that oven drying at 100^0C, despite possessing favorable criteria for drug designing like the smallest particle sizes and the lowest moisture content, did not restore the maximum level of ash, curcumin, and essential mineral elements. Nevertheless, boiling in the range of 60^0C – 80^0C for 30 minutes before drying restores the pharmaceutically important qualities more favorably. The results of this study will help to formulate better treatment for restoring the market-preferable optimum quality of *C. caesia*.

Keywords

ash; boiling; curcumin; mineral element; moisture; particle size

Introduction

Plants have functioned as a natural reservoir for remedies and therapies, from ancient times, medicinal herbs have gained popularity due to their broad use and fewer side effects. The globe has seen a surge in plant research in recent years, and a wealth of data has been gathered to demonstrate the enormous potential of medicinal plants utilized in a variety of traditional systems (1-3). Indian medicinal plants are considered a vast source of several pharmacologically active principles and compounds, which are frequently utilized in home remedies against a variety of illnesses (4). Plants produce specialized metabolites as part of their natural defense mechanisms against diseases, infections, and predators. These compounds have demonstrated promising to utilize for novel drug designing. As per the World Health Organization (WHO), more than 80% of the global population

relies on herbal and natural products for medicinal treatments (5). However, apart from the pharmacologically potential phytochemical composition, particle size and morphology of raw materials of plant origin is also another important aspect to be considered as a quality parameter in the pharmaceutical design of drug dosage (6). Again, nutritional contents, including mineral elements, having considerable therapeutic and prophylactic properties play vital roles in fighting different human diseases, but is another less explored area of ethnobotany (7).

The Zingiberaceae family of advanced monocot plants is known for its aromatic, non-tuberous, and tuberous rhizomes, which have a vast potential for ethnomedical uses (8). This family is well-known for its members with medicinal properties and is widely distributed across tropical regions (9). Out of the 80 species documented in the genus *Curcuma* of the Zingiberaceae family within the Indo-Malayan region, 40 are native to India (10). *Curcuma caesia* Roxb., also referred to as "Black turmeric," is a perennial herb that exhibits a striking bluish-black coloration in the inner part of the rhizome slices (Fig. 1). Presently, *C. caesia* is classified as a critically endangered species (11). It naturally thrives in the regions of North-East and Central India (12,13). The rhizome contains essential oils that give off a distinct sweet fragrance (14). *C. caesia* has significant ethnobotanical potential as the rhizome of this species has been used to treat piles, leprosy, asthma, cancer, diabetes, fertility, toothache, vomiting, allergies, etc. in traditional medicine (12-14). The considerable medical value of *C. caesia* rhizome makes it a valuable commercial resource. The chemical makeup of natural plants has a direct correlation with the pharmacological actions of such plants. Specialized marker metabolites of the genus *Curcuma*, such as curcuminoids, tend to congregate in rhizomes. Curcuminoids are useful for a variety of applications, including spices, coloring pigments, additives, and medicines (15). As the pharmaceutical industry continues to expand, there is an increasing need for plant-based raw materials to extract a wide array of bioactive compounds (16-19). Preserving key components like phenolic and bioactive compounds such as curcuminoids from *Curcuma* spp. including *C. caesia* is essential for pharmaceutical applications and this necessitates an efficient preservation method (20).

Drying is a frequently used technique for medicinal and aromatic plant preservation because it reduces the risk of physical, chemical, and biological modification by lowering the moisture content to a point where microbial development and subsequent degradation of bioactive components is no longer feasible. The moisture level of aromatic and medicinal plants affects their bioactivity by modulating their physical and chemical characteristics. Drying is considered the simplest and most cost-effective means of preserving raw materials and safeguarding the integrity of nearly all biologically active substances and medicinal plant material is primarily utilized in its dried form in pharmaceutical industries (21). However, the drying process significantly changed the composition and oil content of aromatic plants (22). Hence, in contemporary times, the drying of medicinal plants necessitates careful consideration of several factors including the scale of production of bioactive components, the accessibility of new technologies, and adherence to pharmaceutical quality standards (23). The drying process can be broadly categorized into two main groups: natural drying methods, which encompass sun and shade drying, and artificial drying techniques conducted in specialized dryers including hot air drying, infrared drying, microwave drying, vacuum drying, freeze drying, etc. (24). Open sun drying continues to be a prevalent method in many tropical and subtropical regions due to its cost-effectiveness. However, the impact of intense solar radiation is detrimental to quality, resulting in essential oil losses or alterations in the colour of dried plants. Besides, products subjected to open sun drying are vulnerable to deterioration owing to factors such as rodent interference, unexpected rainfall, and insufficient heating (25). The conventional drying method typically involves extended drying times and higher temperatures to achieve thorough drying, which may cause depletion in nutrient and bioactive compounds as well as changes in the colour of

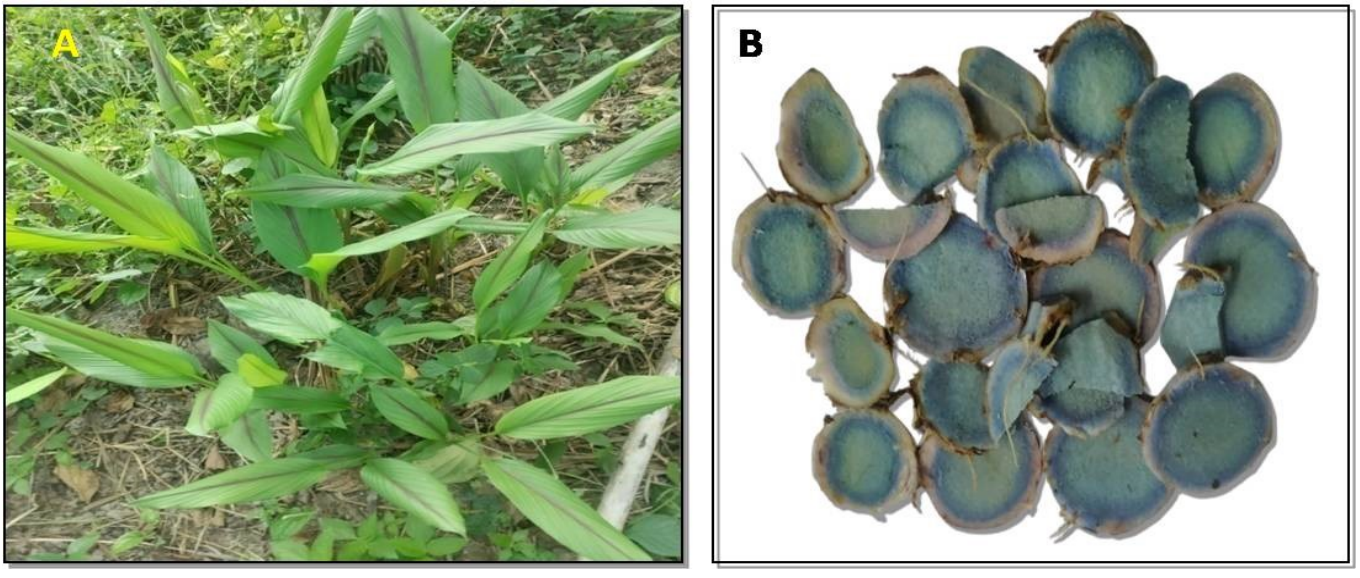

Fig. 1. The whole plants (A), and sliced rhizome (B) of *C. caesia*.

medicinally potential species including *Curcuma longa* (turmeric) and *Curcuma amada* (white turmeric) (26-28).

Traditionally, it is advised to use low drying temperatures ranging from 30°C to 50°C to maintain delicate active components. To preserve these active ingredients in medicinal plant materials, opting for relatively low drying temperatures is recommended, as a result, the drying duration is comparably long (23). Therefore, identifying an appropriate drying method is crucial for maximizing the concentration of active substances in medicinal plants. Nevertheless, there has been a lack of comprehensive information on the effects of different drying methods on quality parameters, elemental profile, and particle size of *C. caesia.* Therefore, this study aims to compare and analyze the effects of different drying methods, such as direct sunlight exposure, solar drying, shade drying, and conventional oven drying, on quality parameters including moisture, ash, and curcumin contents of *C. caesia* rhizomes. Two other important aspects of pharmaceutical consideration viz. elemental composition and particle size of *C. caesia* rhizome after various drying treatments are also investigated to scientifically suggest an optimum condition for this ethnobotanically important medicinal plant species.

Materials and Methods

Plant materials

Rhizomes of *C. caesia* were collected in October 2023 from Khat Tetelia Namgaon, Khetri, Assam, India [26^0 8$^/$ N latitude and 91^0 40$^/$ E longitude]. Rhizomes were separated from dust and dirt, before being peeled and sliced.

Drying methods

Different drying treatments were given as illustrated below (Fig. 2).

Sun drying: Slices of fresh *C. caesia* rhizomes were directly exposed to the sunlight until they gave fine power upon grinding the dried sample.

Solar drying: *C. caesia* sliced rhizome samples were exposed to the sunlight by covering the samples with black cloths.

Shade drying: *C. caesia* sliced rhizome samples were air dried in shade and at room temperature (25°C).

Conventional oven drying: *C. caesia* sliced rhizome samples were spread on a Petri plate and kept in an oven with temperature variations of 70°C, 85°C, and 100°C. The drying process was continued until the moisture level dropped to less than one percent.

Boiling before different drying treatments: The rhizomes of *C. caesia* were boiled in water for 30 minutes with temperature variations of 60°C, 80°C and 100°C in the

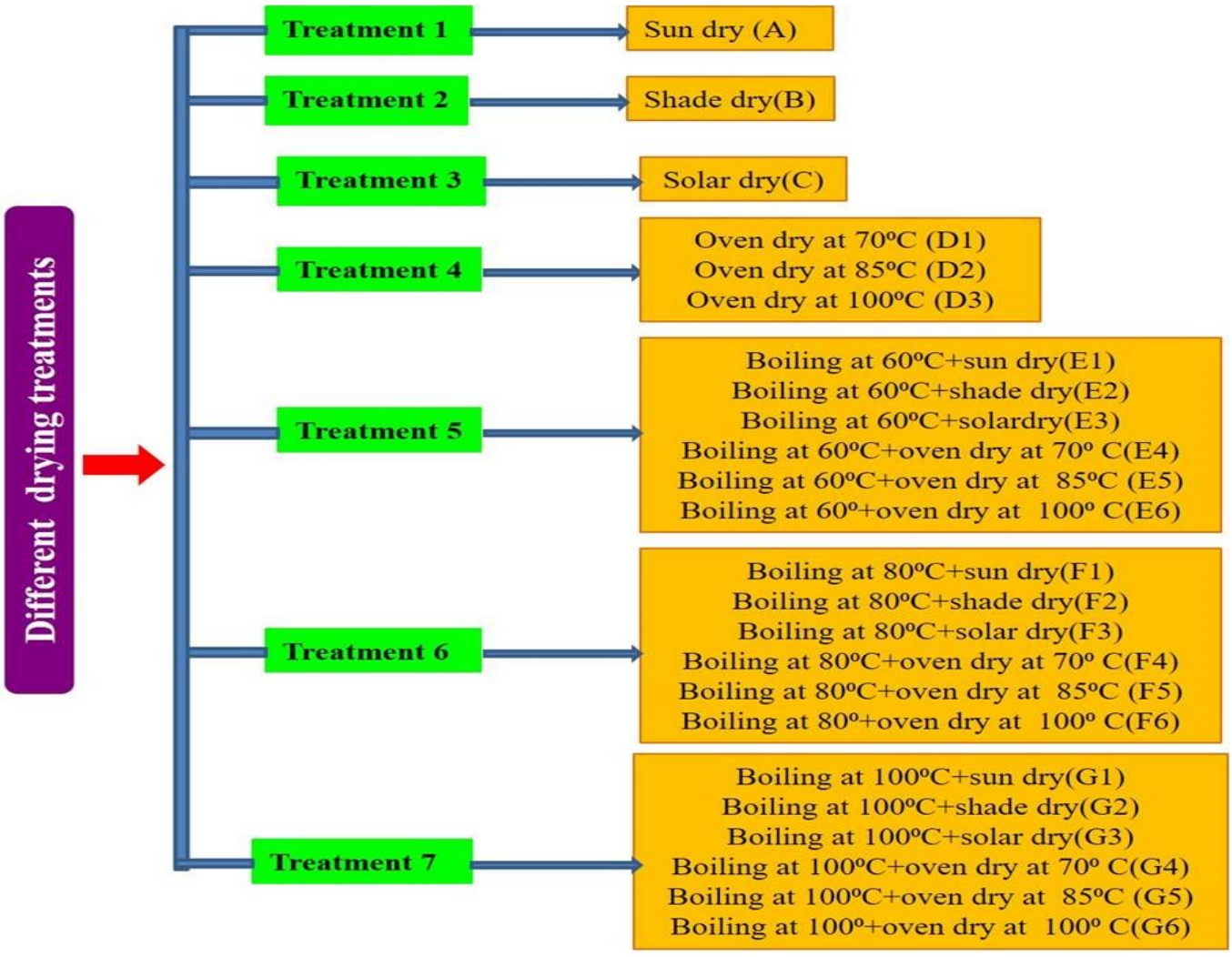

Fig. 2. Schematic diagram of different drying treatments given to *C. caesia* rhizome.

water bath to allow complete gelatinization of the starch and even distribution of curcumin, avoiding raw odor, reducing drying time and yielding a uniformly colored product. After Boiling, different drying treatments were given which included sun drying, solar drying, shade drying, and oven drying at 70°C, 85°C, and 100°C.

Analytical methods

Determination of moisture content: The moisture contents of *C. caesia* rhizomes were determined using a standard protocol as described previously (29). Accordingly, slices of rhizomes were dried at 105 °C in a hot air oven to constant weight and the moisture content was calculated by using **Equation 1.**

Where,

$$\textbf{Moisture content (\%)} = \left[\frac{W1-W2}{W1}\right] \times 100 \quad \text{.....Eqn.1}$$

W1= Weight (g) of the sample before drying

W2= Weight (g) of the sample after drying

Estimation of ash content: Ash content was determined by using a previously described standard protocol (30). The sliced rhizomes were heated in porcelain basins at 550 °C in a muffle furnace for three hours and the ash content was calculated with the help of Eqn. 2.

$$\textbf{Ash content (\%)} = \left[\frac{\text{Weight of the basin with ash-Weight of the basin}}{\text{Gram of sample taken}}\right] \times 100 \quad \text{.....Eqn.2}$$

Determination of curcumin content: Curcumin content was determined by a previous standard protocol (31). As such, 5 mL of acetone extract of rhizome powder was taken in a round flask wrapped with dark colored tape to maintain the dark condition (since curcumin is light sensitive). The UV-Vis spectrophotometer (Agilent Cary 60 UV-Vis) was used to measure the absorbance of the extract in 420 nm wavelength. Finally, curcumin content was calculated based on a standard curve prepared by using commercial curcumin powder procured from SRL. Curcumin concentrations were determined using Eqn. 3.

$$\textbf{Curcumin content (µg/mg)} = \frac{\textbf{Curcumin equivalent to standard curcumin } \left[\frac{\mu g}{mL}\right] \times \textbf{Total reaction volume (mL)}}{\textbf{Concentration of plant material in working stock solution } \left[\frac{mg}{mL}\right] \times \textbf{Volume of alequotes (mL)}}$$

Estimation of mineral elements

Differently treated dried powdered samples of *C. caesia* rhizomes were used for elemental analysis employing a scanning electron microscope (SEM) coupled with an energy dispersive x-ray (EDX) spectrometer. The SEM utilizes a high-energy electron beam to produce a range of signals on the specimen's external surface, offering insights into both external morphology and chemical composition. SEM paired with EDX represents modern and widely adopted techniques for both qualitative and quantitative assessment of trace and essential elements (32).

Particle size determination

SEM pictures for particles of various granulometries were captured. Particle sizes in the micrographs were measured using the Image software (33). Due to the irregular shapes of the particles, the widest side was selected as the parameter for measurement.

Statistical analysis

All the determinations were performed in triplicates and the values were expressed as mean ± standard deviation (SD). The data were subjected to statistical analyses, with the help of MS Excel and SPSS software.

Results and Discussion

Moisture content

In the present study, the moisture content of *C. caesia* rhizome varied in the range of 0.038 % to 0.198 % with variations in drying treatments (Fig. 3). Shade dried sample which was boiled at 100 ^{0}C showed the highest moisture content (0.198 %) and oven dry sample at 100 ^{0}C

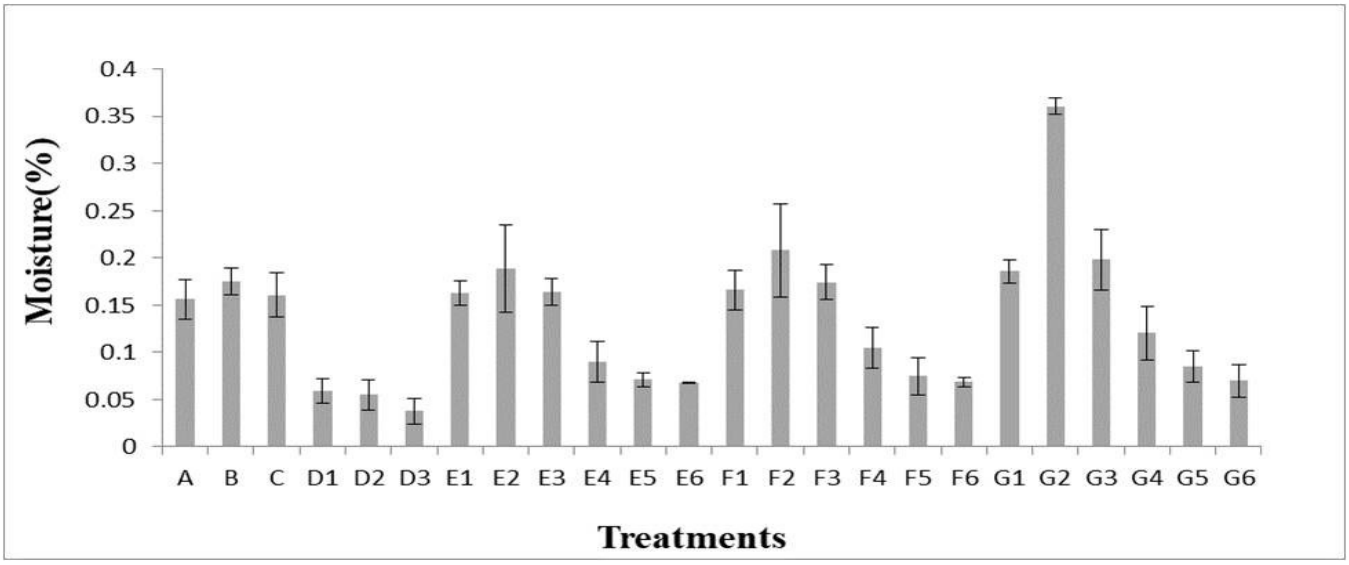

Fig. 3. Effect of different drying treatments on the moisture content of *C. caesia* rhizome.

recorded the lowest moisture content (0.038 %). This result supported the previous finding, where higher drying temperatures resulted in lower moisture content and water activity in *C. amada* (white turmeric) rhizome powder. In the same study, a freeze-dried sample of *C. amada* at 0 °C and subsequent final heating at 50 °C exhibited the lowest moisture content, compared to the sun-dried sample with the highest moisture content (27). Similar previous investigations suggested that different drying techniques, viz. shade drying, solar drying, hot air oven drying, microwave drying, and freeze drying, had dissimilar impacts on the moisture level of *C. longa* rhizome samples (34). Reports suggested that the moisture content of the oven-dried sample of *C. longa* showed the lowest moisture content in comparison to the cooked/oven-dried sample which exhibited the highest moisture content (35). Interestingly, the moisture contents of the *C. longa* rhizome flours did not differ significantly with the various oven drying temperatures such as at 45, 55, 65, and 75 °C (36). Conventional oven drying at 50 °C resulted in the lowest moisture content in rhizome samples of *Zingiber officinale* than the solar box, sun oven plus solar box, and microwave oven drying treatments (37). In a related previous study, a rhizome sample of *Boesenbergia rotunda* (L.) Mansf. which was spray-dried at 190 °C showed lower moisture content compared to those spray-dried at 150 °C (38). For dried herbs, it is crucial to keep the moisture content below 10% to prevent fungal growth and it has been revealed that reducing the water content makes *C. amada* more conducive for extending shelf life (39). Studies revealed that mechanical dryers offered superior results in terms of moisture content and functional properties compared to the sun-dry method.

Ash content

In the present study on *C. caesia*, the highest ash content (11.5 %) was obtained in the sample that was oven-dried at 70 °C after boiling at 80°C and the lowest ash content (3.23 %) was recorded in samples that were oven-dried at 100 °C after boiling at 100 °C (Fig. 4). This corroborated with the previous findings where the finger rhizomes of *C. longa* boiled at 80 °C for 30 minutes exhibited the highest total ash content, while the rhizomes boiled at 100°C for 75 minutes had the lowest ash content (40). On the contrary, the solar drying method yielded the highest total ash content in ginger, indicating solar drying as a potential method to retain maximum minerals in that species and revealing the species-wise variation in optimum preservation methods (41). Treatment-wise variations in ash content were also observed in *Curcuma domestica* and *Z. officinale.* Solar box treatment was more effective in the restoration of ash content in *Z. officinale* compared to sun oven plus solar box, and conventional oven drying at 50 °C as well as microwave oven drying treatments (37). On the other hand, *C. domestica*, Bonga51/71 variety treated with conventional curing and solar tunnel dryer had the highest total ash content, while the lowest ash content was observed in the Dame variety cured in improved curing and drying methods in greenhouse solar dryer (42). On the contrary, the ash contents showed no significant variations in *C. longa* rhizome samples with various oven drying temperatures, viz. 45, 55, 65 and 75 °C (36). However, in another study, the highest total ash content of *C. longa* was recorded in the tray drying method at a drying temperature of 80 °C, while the lowest total ash content was achieved at 60 °C (43). In another report, the highest total ash content in *C. longa* was observed in oven-dried samples and blanched/oven-dried samples with no significant difference between these two treatments, whereas the lowest total ash content was observed in cooked/oven-dried and sun-dried samples, respectively, again with no significant difference between the two treatments (35). Hence, it is evident that there are treatment-wise variations in ash content in rhizomes of different species of the Zingiberaceae family, which reveals the need for optimization of drying methods from species to species for better restoration of quality parameters.

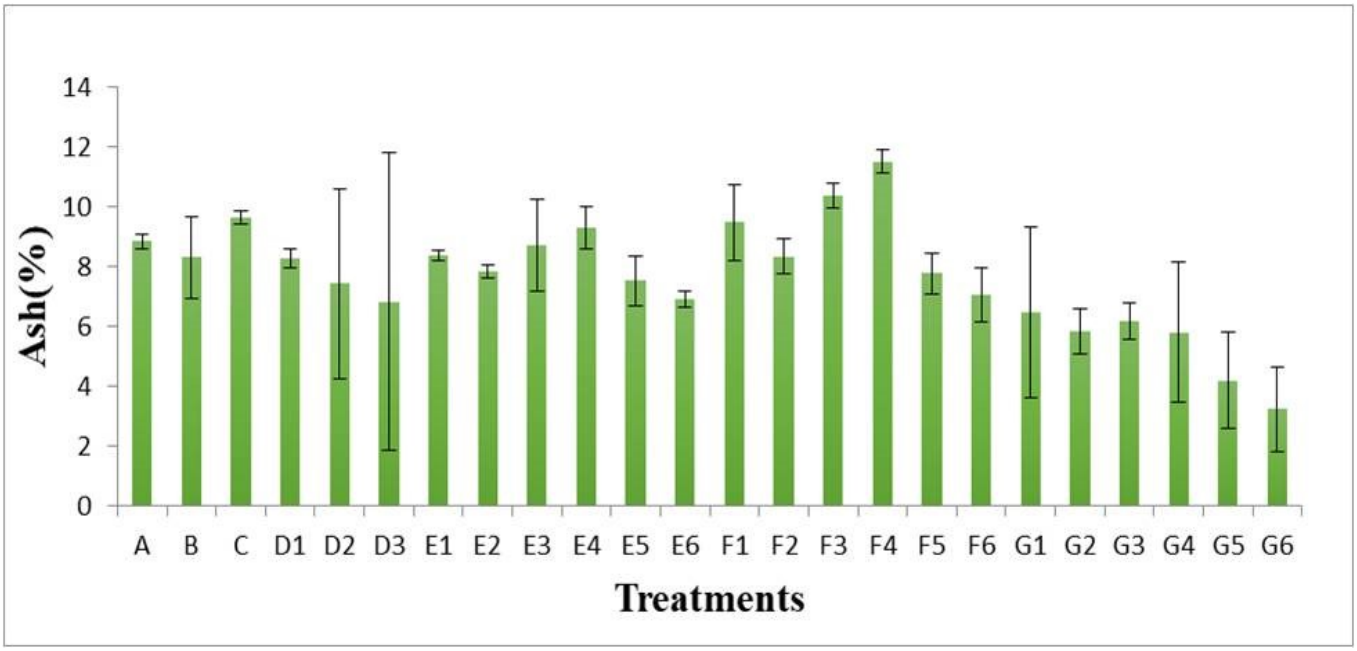

Fig. 4. Effect of different drying treatments on ash content of *C. caesia* rhizome.

Curcumin content

In the present study on *C. caesia*, maximum curcumin content was recorded in shade dried sample which was boiled at 80 ^{0}C for 30 minutes, and the minimum curcumin content was found in the sample that was oven-dried at 100^0 C followed by boiling at 100 ^{0}C (Fig. 5). This result partially aligned with other findings on *C. longa* where shade-net drying was found to be preferable over cabinet and sun drying regarding the retention of curcumin content (44). Previous reports also suggested that drying *C. longa* rhizomes with hot air at 70 °C emerged as the most effective method for maximizing the curcumin content. A decrease in curcumin content with the increased boiling time was reported in several previous studies (45). The sliced sun-dried sample of *C. longa* boiled for the longest duration of 60 minutes, exhibited the lowest curcumin content 3.53 % (46). Again, the flat-bed drying treatment at temperatures of 50, 60, and 70 °C, conducted on *C. longa* showed the retention of the highest curcumin content at 50 °C (47). On the other hand, the Bonga 51/71 variety of *C. domestica*, when cured with conventional curing and drying with a solar tunnel dryer, resulted in the highest retention of curcumin content (42). Interestingly, the cooked/oven-dried and sun-dried samples of *C. longa* exhibited the highest curcumin content, with no significant difference between the two treatments, while the blanched/oven-dried and oven-dried samples showed the lowest curcumin content with no significant difference between the two treatments (35). Studies also suggested that there was a significant reduction in the curcumin content of *C. longa* during heat processing, with the most substantial loss during pressure cooking for 10 minutes (26). Hence, the treatment-wise discrepancy is obvious from species to species regarding curcumin content too suggesting the requirement of thorough scientific investigation of drying methods for a particular species, to restore the medicinally important quality parameters in the best possible way.

Mineral elements

Presence of the elements such as carbon (C), oxygen (O), magnesium (Mg), silicon (Si), phosphorus (P), potassium (K), and calcium (Ca) was revealed in SEM-EDX analyses of a differently treated sample of *C. caesia* rhizome powder. This substantiated the previous reports on shade-dried samples of *C. caesia* rhizome, identifying the similar trend of the existence of these elements that revealed higher atomic (%) of C and O in comparison to Mg, Si, P, K, and Ca (7, 32). Here, solar dried sample had the highest C [atomic (%) =61.73] and the sample that was boiled at 100^{0}C followed by oven drying at 100^0 C had the highest O [atomic (%) = 53.26] (Table 1). However, Mg, P, and Ca were found to be the highest [atomic (%) = 0.84, 0.70, and 1.62, respectively] in sun-dried samples followed by boiling at 60 ^{0}C for 30 minutes. Another important element K was found to be the highest in oven-dried samples at 100 ^{0}C followed by boiling at 60 ^{0}C for 30 minutes, whereas Si was the maximum in solar-dried samples after boiling at 60 ^{0}C as well as 80 ^{0}C. Hence, boiling at 60 ^{0}C and subsequent drying are suggested to be the most favourable drying treatment regarding the restoration of important mineral elements in *C. caesia* rhizome which provides a necessary clue to identify the better preservation treatment for optimum pharmaceutical application of the species. Studies on the medicinal properties of plants, focusing on their organic components such as alkaloids, glycosides, essential oils, vitamins, and other active compounds, have been extensive. However, there is limited information on the medicinal properties of plants based on elemental or mineral nutrient content. It is though crucial to recognize that various elements play vital roles in combating different human diseases. Mineral elements that are present in medicinal plants in small doses possess both therapeutic and prophylactic properties (7, 48).

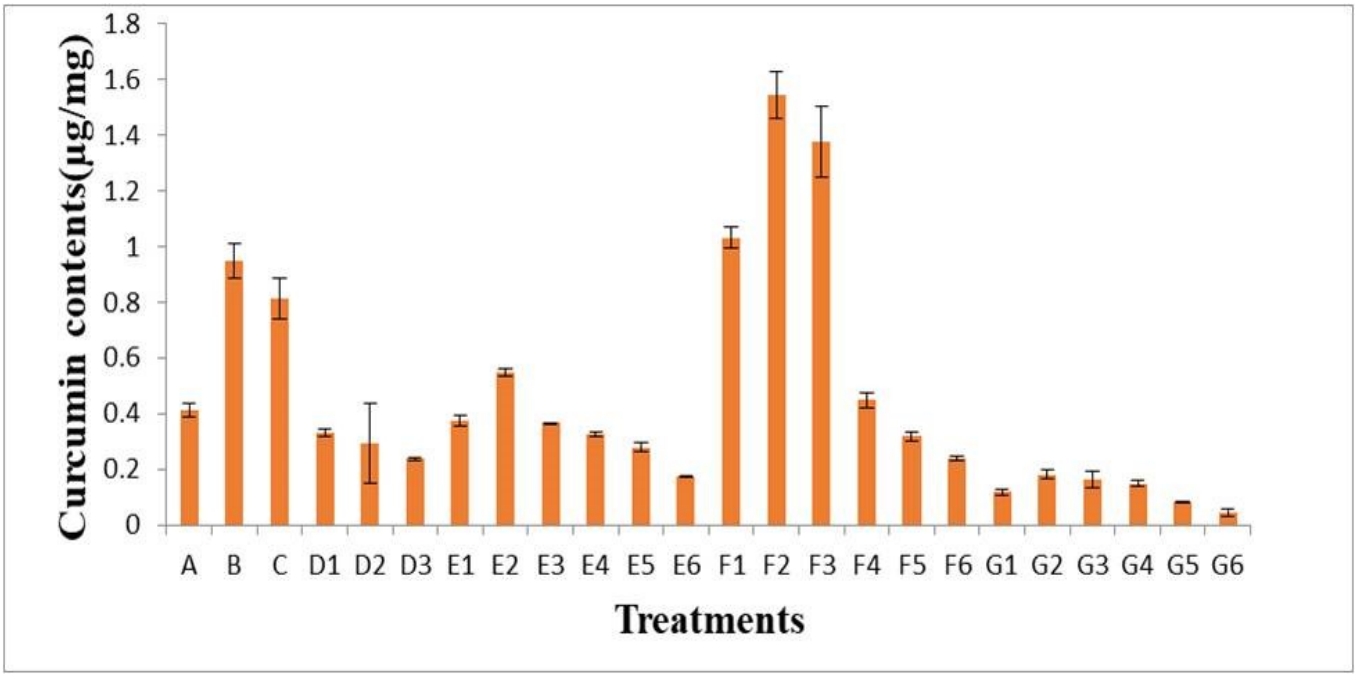

Fig. 5. Effect of different drying treatments on curcumin content of *C. caesia* rhizome.

Table 1. Elements of *C. caesia* rhizome after different drying treatments obtained from SEM-EDX.

Treatments	Atomic %						
	C	O	Mg	Si	P	K	Ca
A	52.22	45.81	0.22	0.45	0.17	0.76	0.07
B	50.74	47.26	0.24	0.37	0.25	0.82	0.05
C	61.73	35.85	0.19	0.47	0.11	1.13	0.13
D1	57.21	38.92	0.43	0.32	0.57	2.14	0.08
D2	49.18	45.25	0.12	0.87	0.41	3.55	0.24
D3	45.49	49.51	0.59	1.30	0.44	2.14	0.26
E1	39.54	52.98	0.84	1.85	0.70	1.53	1.62
E2	53.39	43.28	0.10	0.28	0.37	1.87	0.19
E3	46.52	49.04	0.12	1.90	0.20	1.54	0.23
E4	41.20	52.50	0.38	1.26	0.43	3.09	0.49
E5	48.21	47.97	0.13	0.83	0.15	2.08	0.19
E6	46.87	46.71	0.29	0.80	0.21	4.05	0.11
F1	43.80	49.53	0.73	1.54	0.14	3.12	0.62
F2	46.40	50.09	0.36	0.68	0.36	1.32	0.23
F3	54.37	40.58	0.32	1.90	0.28	1.49	0.27
F4	51.53	46.17	0.18	0.23	0.21	1.07	0.09
F5	51.24	46.07	0.36	0.74	0.26	0.67	0.08
F6	49.09	47.72	0.33	0.53	0.32	1.39	0.13
G1	44.90	48.33	0.14	1.73	0.43	3.63	0.21
G2	49.40	47.88	0.21	0.39	0.41	1.02	0.18
G3	48.93	47.10	0.22	0.88	0.51	1.72	0.12
G4	42.67	53.13	0.19	0.95	0.33	1.82	0.18
G5	52.53	45.31	0.27	0.67	0.16	0.42	0.13
G6	42.59	53.26	0.11	0.55	0.35	2.35	0.21

[A = Sun dry, B = Shade dry, C = Solar dry , D1= Oven dry at 70^0 C, D2= Oven dry at 85^0 C, D3= Oven dry at 100^0 C, E1= Boiling at 60^0 C+ sun dry, E2 = Boiling at 60^0 C+ shade dry, E3 = Boiling at 60^0 C+ solar dry, E4 = Boiling at 60^0 C+ oven dry at 70^0 C, E5 = Boiling at 60^0 C+ oven dry at 85^0 C, E6 = Boiling at 60^0 C+ oven dry at 100^0 C, F1 = Boiling at 80^0 C+ sun dry, F2 = Boiling at 80^0 C+ shade dry, F3 = Boiling at 80^0 C+ solar dry, F4 = Boiling at 80^0 C+ oven dry at 70^0 C, F5 = Boiling at 80^0 C+ oven dry at 85^0 C, F6 = Boiling at 80^0 C+ oven dry at 100^0 C,G1= Boiling at 100^0 C+ sun dry, G2=Boiling at 100^0 C+ shade dry, G3 = Boiling at 100^0 C+ solar dry, G4 = Boiling at 100^0 C+ oven dry at 70^0 C,G5 = Boiling at 100^0 C+ oven dry at 85^0 C, G6 = Boiling at 100^0 C+ oven dry at 100^0 C]

Particle size

Through analyzing the SEM images, the particle sizes of treated and powdered samples of *C. caesia* were determined. Samples that were oven-dried at 100 ^{0}C had the smallest particle size compared to other treatments, whereas the boiled samples had increased particle sizes compared to other samples (Fig. 6). Particles of raw materials at the nano- and microscales are critically observed in the pharmaceutical design of solid dosage forms. This is crucial as the morphology of particles, including size, shape, and surface characteristics have significant impacts on the performance of processes and the quality characteristics of pharmaceutical products (6). Reports suggested that the yield of extract, phytochemical composition, and metabolite profile were comparatively richer as well as antioxidant activity was stronger with smaller particle sizes of certain plant materials (49). Moreover, the smaller particle size of medicinal material corresponds to increased surface area that facilitates faster dissolution in the blood as well as enhanced penetration through the barriers resulting in more efficient delivery to the target (50). However, restoration of other quality parameters alongside smaller particle sizes is crucial for recommending the best method for restoration of the pharmaceutical potential of the medicinal plant species.

Conclusion

This study suggests that quality parameters including moisture, ash, and curcumin contents as well as mineral elements and particle sizes of *C. caesia* rhizomes vary with different drying treatments. In the current study, oven drying at 100 ^{0}C, despite producing the smallest particle sizes and the lowest moisture content (which are favourable criteria for drug designing), did not restore the maximum level of ash, curcumin, and essential mineral elements. On the contrary, boiling in the range of 60 ^{0}C to 80 ^{0}C for 30 minutes and subsequent drying restored the pharmaceutically important qualities more favourably. Hence, other mechanical strategies to obtain smaller particle sizes for the samples boiled in the range of 60 ^{0}C to 80 ^{0}C can be explored to get maximum medicinal and nutritional benefit from the species in a pharmaceutically preferable manner.

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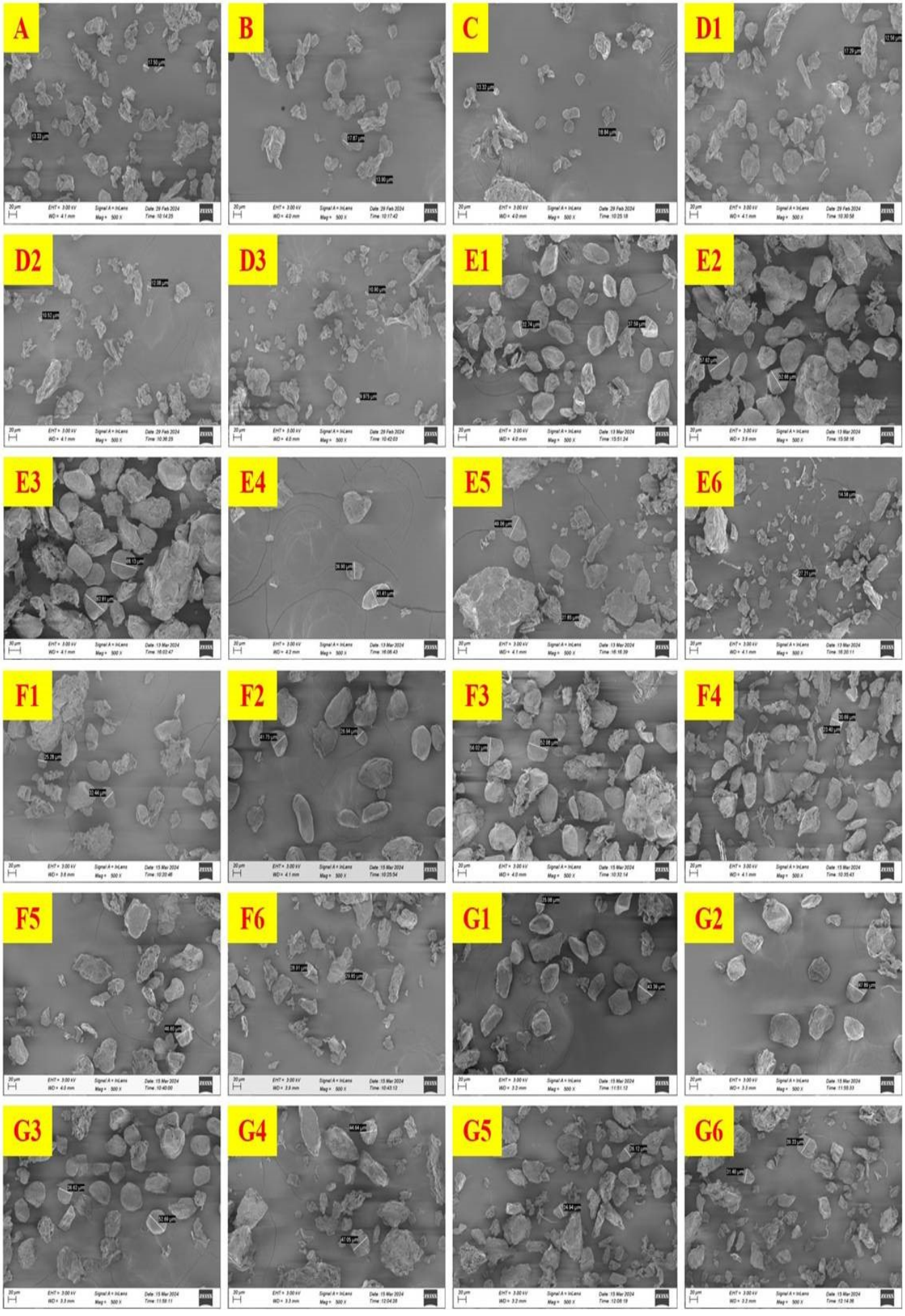

Fig. 6. SEM images of the particles of rhizome powders after different drying treatments.

Authors' contributions

NB conceived the idea of the study and participated in its design and coordination. PB carried out the material collection, sample preparation, and quality analyses on the bench. NB participated in the data analyses and interpretation as well as performed the statistical analyses. PB drafted the manuscript initially and the overall final compilation was done by NB. Both authors read and commented on the previous drafts of the manuscript and approved the final manuscript.

Compliance with ethical standards

Conflict of interest: Authors do not have any conflict of interest to declare.

Ethical issues: None.

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