Effect of thin layer drying on quality parameters of betel leaf (*Piper betle* L.)

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Abstract: Betel leaf or "Paan" is contemplated as "a neglected green gold of India" due to its nutritional, economic, medicinal, social and cultural values. However, surplus production of the leaves during the glut season and lack of adequate storage facilities compels the betel leaf growers to sell it at a throw away price. Therefore, to overcome this problem, the preservation techniques like shade drying, hot air oven drying and vacuum oven drying have been widely used, but these methods produce lower quality dried betel leaves or consume more time to dry. Freeze drying is a well-known technique to dehydrate heat sensitive products without damaging its actual quality. Hence, the current study was taken up to compare quality of freeze-dried betel leaves with shade dried, hot air oven dried and vacuum dried betel leaves. Drying conditions for shade drying, hot air oven drying, vacuum drying and freeze drying were 32 ± 3°C, 60°C, 60°C and 0.5atm, and - 40°C and 0.21 atm, respectively. Eight different thin layer drying models were studied and compared based on coefficient of determination (R^2), root mean square error (RMSE) and chi square. A sensory study on dried leaves was also conducted on 9-point hedonic scale. The results showed that Page model was the best fitted model for freeze drying ($R^2 = 0.992$) and vacuum drying ($R^2 = 0.995$) methods, whereas Peleg model and Verma et al. model were found to be best fitted for shade drying ($R^2 = 0.997$) and oven drying ($R^2 = 0.993$) methods, respectively. The results also revealed that both freeze dried and shade dried contained the highest essential oil content (1.5%) compared to oven dried (1.0%) and vacuum dried (0.8%) betel leaves. However, shade dried leaves were found to be the most preferable by the panelists during organoleptic evaluation followed by freeze drying, hot air oven drying and vacuum drying. Therefore, it can be stated that freeze dried leaves are analogous to shade dried leaves in terms of quality. However, freeze drying took much lesser time (10h) to dry compared to shade drying (10 days). Hence, freeze drying can be considered as a suitable preservation technique for drying of betel leaves.

Keywords: betel leaf, drying techniques, drying models, essential oil, sensory attributes

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1 Introduction

Betel leaf (*Piper betle* L.) is a deep green, heart shaped leaf of a vine which belongs to the family Piperacea. It is popularly known as "Paan" in India and other countries in the Indian subcontinent such as Pakistan, Bangladesh, Nepal, etc. (Sadhukhan and Guha, 2011). From the ancient time, this edible leaf has attained an esteemed socio-economic, medicinal and demographic importance apart from its general use of chewing with other ingredients like sliced areca nut, slaked lime, coriander, aniseed, clove, cardamom, etc. mainly for the purpose of attaining digestive, stimulating and mouth freshening effects. The socio-economic importance of the leaves can be

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corroborated from the scientific publications reported by Guha and Jain (1997). It is mentioned that about 20 million people are directly or indirectly associated with the farming, handling, transportation, processing, marketing and consumption of betel leaf in India alone. Moreover, the leaves possess good amount of antioxidant and antimicrobial components. These make its extract and essential oil valuable ingredients for folk medicines which are prescribed for the treatment of various diseases. These include bad breath, boils and abscesses, conjunctivitis, constipation, headache, ringworm, swelling of gum, rheumatism, abrasion, cuts and injuries and so on.

In India, betel leaf is cultivated on about 55000 ha land among which about 20000 hectares are in West Bengal encompassing about 4-5 lakh Boroj (Guha and Jain, 1997; Guha, 2006; Jana, 1996). In these Borojs, about 60-70 leaves are produced by each plant every year producing a total of 6-7 million leaves per ha worth about □9000 million (Guha, 2006). However, due to its highly perishable nature, around 35% to 70% of the leaves are subjected to spoilage by fungal infection, dechlorophyllation, loss of moisture and freshness, etc. during transportation and storage. Moreover, surplus leaves produced during the rainy season are sold at a thrown away price or used as cattle feed or disposed off in an improper manner causing environmental pollution due to the lack of proper storage facilities. In view of the above-mentioned alarming situations, a few attempts have been made by the researchers to minimize such wastage by drying the leaves (Ramalakshmi et al., 2002), controlling senescence (Mishra and Gaur, 1972), chemical treatment, manipulation of storage temperature and adopting better packaging materials and methods (Guha, 2008; Rao and Narasimham, 1977) besides curing and bleaching of the leaves (Sadhukhan and Guha, 2011; Dastane et al., 1958; Sengupta, 1996). Among these methods of enhancing the shelf life of the leaves, a few reports are also available on different drying methods used to obtain dried betel leaves which can be used later for manufacturing of different commercial products such as pouched mouth fresheners, essential oil, fortified bakeries, etc. (Guha, 2006; Maurya, 2014; Laji, 2015). Therefore, enhancing shelf life of the leaves becomes imperative during glut season as well as during dull marketing seasons to minimize the losses over 900 million. These valuable national resources are termed as green gold due to its financial, medicinal, nutritional, social and cultural importance. That apart, enhancing shelf life becomes inevitable during the period of poor export or blocked transportation due to natural or man-made calamities. Therefore, to overcome the above-mentioned alarming conditions, several attempts have been made by the research workers for enhancing shelf life of the leaves or preserving the leaves by adopting different drying techniques as discussed in the foregoing lines. The research group of Balasubramanian et al. (2011) studied the effect of drying temperature (50°C, 60°C and 70°C) on quality of betel leaves. They reported that optimum colour quality was observed at 60°C in tunnel dryer and at 50°C in cabinet dryer, but in terms of rehydration capacity, drying at 40°C yielded the best results. In another study, Pin et al. (2009) studied the effect of drying temperature on the quality of dried leaves by analyzing the changes of the major phytochemicals and concluded that 70°C was the optimum temperature for maximum retention of the major phytochemicals like hydroxychavicol and eugenol. On the other hand, Rayaguru et al. (2007) compared the quality of dried betel leaves obtained from hot air drying method with shade drying and sun drying methods. They concluded that shade drying was the best method for retaining maximum nutrition in the dried betel leaves. However, this drying method is uncertained being weather dependent and also unhygenic due to high microbial contamination. On the contrary, hot air drying at 40°C was found to be suitable for preserving considerable amount of nutritional as well as volatile components. However, hot air drying above 50°C was not recommended by them as most of the volatile and essential components were evaporated. For mathematical modellling of the drying process, several emperical equations have been developed, applied and verified using dataset obtained in various drying experiments of agricultural products. Most of these emperical equations are established to define thin layer drying kinetics. However, these equations can not be used directly to describe deep bed drying process. Therefore, in case of deep bed drying, total bed height is divided as stack of several thin layers and then, one model equation of thin layer drying along with others is used to mathematically describe the process (Da Silva et al., 2014). Therefore, search for better method for retaining these valuable components becomes necessary. The search however, shows paucity of information in this regard, particularly for freeze drying.

Therefore, the present study was planned and carried out to compare the drying kinetics and quality parameters of freeze-dried betel leaf with vacuum dried, hot air oven dried and shade dried leaves.

2 Material and methods

2.1 Raw material

Fresh and defectless betel leaves of *Tamluk Mitha* variety was procured from Technology Market, IIT Kharagpur, West Bengal. The petioles were removed and then lamina was washed properly using tap water to remove the dirt and unwanted materials. Surface water was then removed by tissue papers. The leaves were then cut into average size of 35±0.5 mm. Initial weight of the cut pieces of the leaves was measured by weighing balance for future calculation.

2.2 Drying processes

Betel leaves were subjected to four different drying methods namely hot air drying, vacuum drying, shade drying and freeze drying using thin layer of betel leaves uniformly. For hot air drying, 100 g of the leaves at three different batches were placed evenly above the cleaned shelves of the dryer at 60°C till constant weight was achieved with respect to time. Similar method was also followed for vacuum drying where leaves were placed inside the drying chamber at 60°C and 0.5 atm pressure for 4 h. Shade drying of the leaves (100 g) was performed in a closed but ventilated room at an average temperature of 32°C ± 3°C. Sample weight was measured every day to observe the change in weight and the process was continued till constant weight was achieved with respect to time. In freeze drying process, leaf samples (100 g) were crushed using mixer grinder and then lyophilised at a pressure of 0.21 atm and temperature of -40°C for 10 h in a freeze-dryer.

2.3 Mathematical modelling of betel leaves drying

In the current study, total eight thin layer drying models as listed in Table 1 were applied to select the best model considering the highest coefficients of determination (R^2) value, lowest Chi-square and root mean square error (RMSE) value. The moisture ratio and drying rate was calculated using the following eqautions:

$$Moisture\ Ratio\ (MR) = \frac{M-M_e}{M_0-M_e} \tag{1}$$

$$Drying\ rate = \frac{M_t-M_{t+dt}}{t} \tag{2}$$

Where, M = moisture content at any given instant, % db, M_e = equilibrium moisture content, % db, M_0 = initial moisture content, % db, M_t = moisture content at t, % db and M_{t+dt} = moisture content at t + dt, % db, t is drying time in h, A is drying surface area in m^2 which was same for all the drying methods and considered as constant in the calculation of drying rate.

Table 1 Emperical models to describe the drying kinetics*

S. No.	Drying model	Empirical equation	References
1	Newton Model	MR = exp(-kt)	Mujumdar and Menon (1995)
2	Page Model	MR = exp(-kt^n)	Diamante and Munro (1993)
3	Modified Page	MR = exp[-(kt)^n]	White et al. (1981)
4	Henderson and Pabis Model	MR = a exp(-kt)	Zhang and Litchfield (1991)
5	Modified Henderson and Pabis Model	MR = a exp(-kt) + b exp(-gt) + c exp(-ht)	Karathanos (1999)
6	Verma et al.	MR = a exp(-kt) + (1-a) exp(-gt)	Verma et al. (1985)
7	Peleg Model	MR = 1- t/(a + bt)	Mercali et al. (2010)
8	Two Term Model	MR=a exp(-gt)+b exp(-kt)	Sharaf-Eldeen et al. (1980)

Note: k, n, a, b, g, c & h are the drying model constants.

2.4 Extraction of essential oil

Essential oil of betel leaf was extracted from fresh and dried betel leaf using “Betel leaf oil extractor” according to Guha (2007). For the fresh samples, the leaves were rinsed thoroughly to remove foreign materials. Then, the leaves were depetiolated and weight was measured. After that, the leaf blades were minced into small pieces (approximately 4 cm^2) and put into round bottom flask of the extractor. In one batch, 200 g of the leaf blades were taken along with 400 mL of distilled water (leaf to water ratio- 1:2) to carry out the hydro-distillation process. The round bottom flask was then, placed on a heater and heated. On the other hand, cold water was circulated through condenser for condensation of oil-water vapour mixture. After condensation, the condensed oil was floated as a separate layer at the top of water in the oil collection tube of the extractor. The oil was then transferred to a 2 mL ependrof tube and stored at refrigerated condition in dark place. In case of extraction of essential oil from the dried samples, 100 g of dried leaves were used and the similar procedures were followed after hand crushing of the leaves.

2.5 Sensory evaluation

Consumer acceptability of dried betel leaf obatined from the four drying methods were evaluated based on 9 point hedonic scale (Lu et al., 2010). The sensory examination was carried out by 10 semi-trained panellists. The dried leaves which were stored in Low-density polyethelyne (LDPE) pouch at room temperature after drying, were given to the panellists along with drinking water and cream cracker biscuits (Non-salted, manufactured by Britannia, India). Ten different places or booth were arranged for unbiased evaluation. The sensory attributes of the dried leaves were compared based on colour, taste, smell and overall acceptability score.

3 Results and discussion

3.1 Drying kinetics

In the present study, drying kinetics of betel leaf in different drying methods was analysed to understand the behaviour of moisture removal from the leaves. The leaves were dried from initial moisture content (709.71% in db) to the final moisture content (% db) of 63.97%, 12.23%, 21.5% and 26% in db for shade drying, hot air oven drying, vacuum drying and freeze drying, respectively. The thin layer drying kinetics of the betel leaves in different drying methods are shown in Figure 1. It can be observed from the figure that moisture content of the leaves decreased with the increase in the drying time. Among the four methods, shade drying took maximum time (240 h or 10 days) to dry the leaves followed by oven drying (24 h), freeze drying (10 h) and vacuum drying (7 h) in order. During the vacuum drying process, boiling point of the water starts reduced with the decrease of the pressure of drying chamber, which results in quick evaporation of water molecules and generates stress at cellular material and consequently, creates pores (Mayor and Sereno, 2004). These pores then expand with further reduction of pressure which facilitates faster evaporation of moisture. On the other hand, in freeze drying process, pore size is directly proportional to size of ice crystal, which is again inversely proportional to freezing rate. Therefore, a larger crystal or pore in a dried product can be produced by decreasing the freezing rate. In this study, the fresh leaves were dried within 7 h in vacuum drying process compared to 10 h in freeze drying. The reason behind this could be the faster freezing rate which produced comparatively smaller size of pores inside the leaves than vacuum drying and therefore, rate of drying was slower in the freeze drying process. This indicates that vacuum drying has given quicker drying than freeze drying. Thus, it clearly shows that sophisticated technology, for instance freeze drying, has consumed more time, energy and consequently, cost compared to vacuum drying. This seems to be paradoxial, but it has happened due to the fast rate of cooling causing formation of smaller ice crystals yielding smaller pores. Therefore, in future, attempts should be made for slowing down the freezing rate to obtain larger crystal yielding larger pores and consequently, increased drying rate.

3.2 Drying rate curves

The plots of drying rate vs time are shown in Figure 2. From the plots, it can be observed that the leaves were predominantly dried in falling rate period and no constant rate drying period was observed. Similar results were also reported by other authors for drying of betel leaves and other agricultural produces like beans, potato and ogbono nuts and kernels, etc. (Balasubramanian et al., 2011; Senadeera et al., 2003; Aregbesola et al., 2015). From the plots, it can also be noticed that drying rate changed with change in the drying methods and faster drying rate was observed from vacuum drying process. However, a very slow rate of drying was observed for shade drying due to minimum change in the driving forces (temperature and relative humidity) with time.

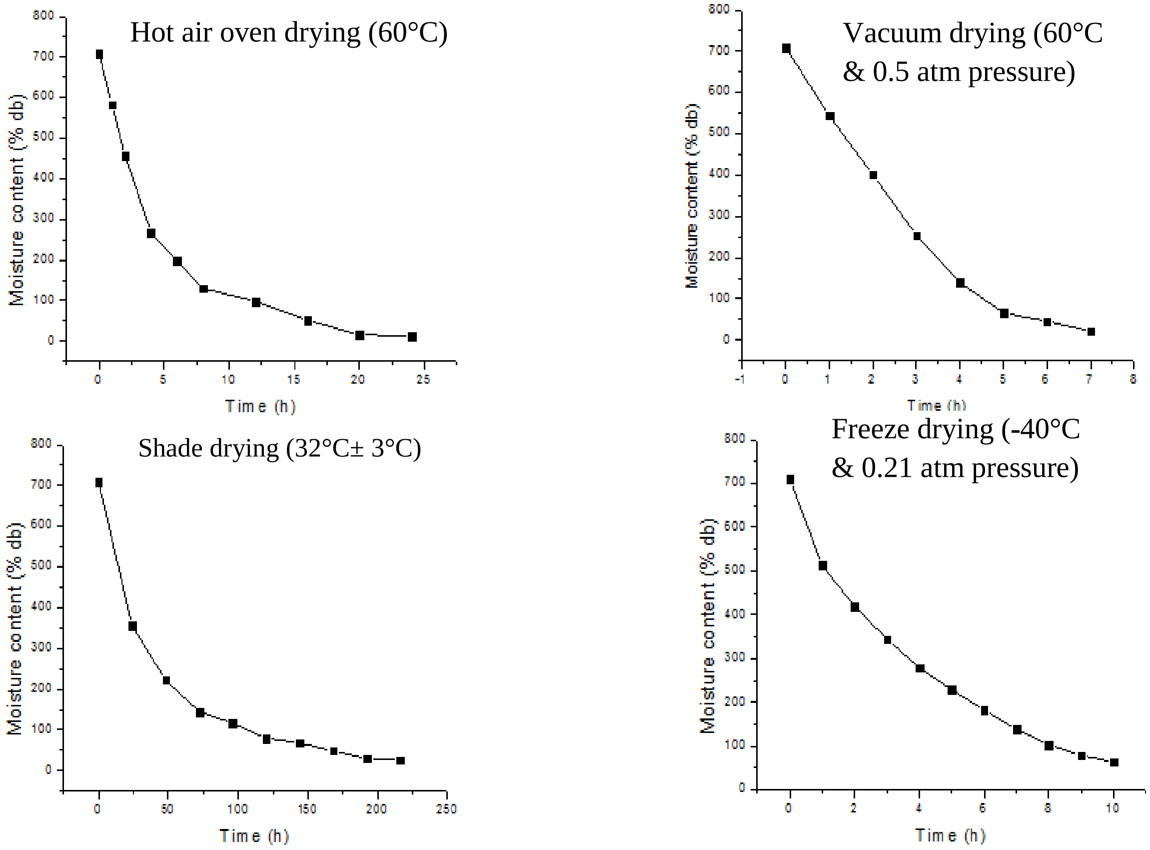

Figure 1 Drying kinetics plot of different drying methods

3.3 Mathematical models

The results of eight non-linear thin layer drying models are given in Table 2, 3, 4 & 5 for the four different drying techniques, namely oven drying, shade drying, vacuum drying and freeze drying. Based on statistical indicators such as R^2 (Maximum), Chi-square (Lowest) and RMSE (Lowest) of the emperical models, a comparison was carried out to obtain best fitted model. From Table 2, it can be observed that drying kinetics of oven drying method fitted well with R^2 value varying between 0.99 to 0.98 compared to shade drying (R^2 varies between 0.99-0.90), vacuum drying (R^2 varies between 0.99-0.95) and freeze drying (R^2 varies between 0.99-0.96) methods. On the other hand, it can also be observed from the Table 2, 3,4 & 5 that Page model was best fitted for freeze drying and vacuum drying, whereas drying kinetics of shade drying and oven drying showed best fit with Peleg model and Verma et al. model, respectively. Balasubramanian et al. (2011) studied drying kinetics of betel leaf using tunnel and cabinet dryer and found that logarithmic and Page model were the best fit model for tunnel and cabinet dryer, respectively.

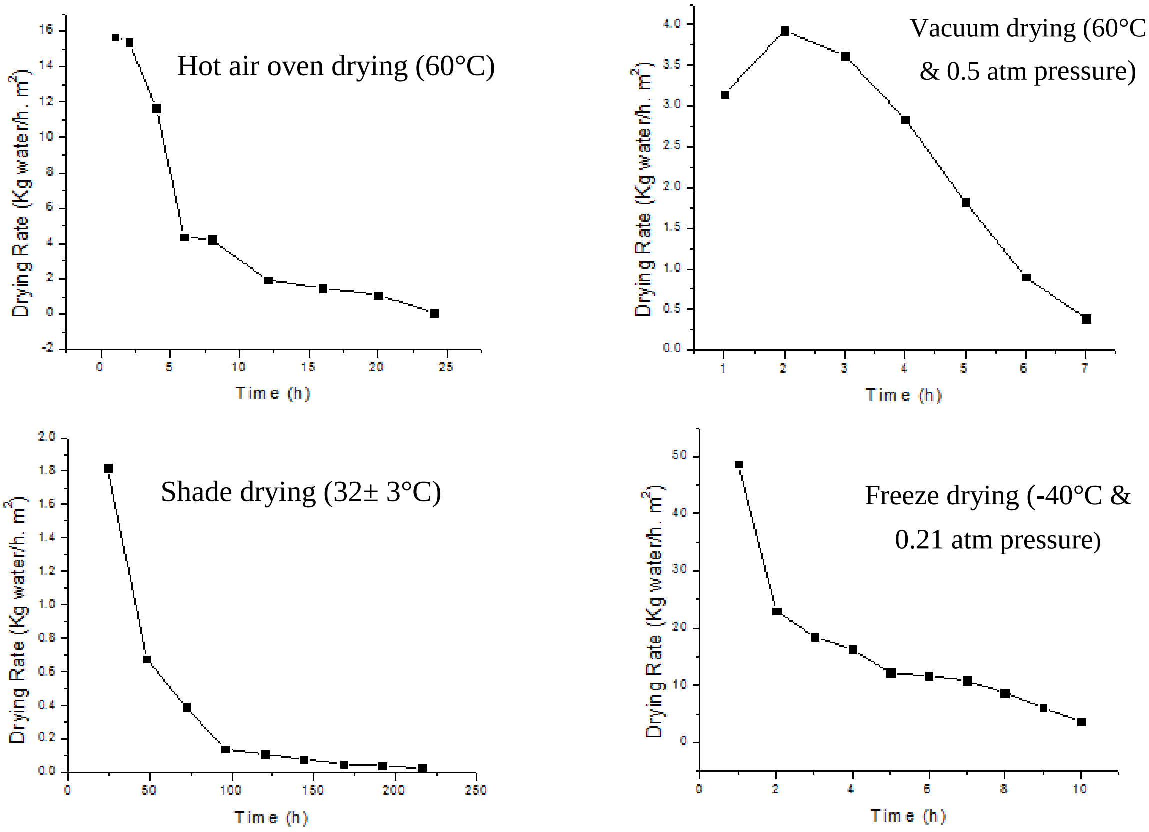

Figure 2 Drying rate curve of different drying methods

Table 2 Thin layer drying model co-efficients and its goodness of fit for oven drying

Sl. No.	Model	Constants	R^2	Chi-Square	RMSE
1	Newton Model	k=0.2128	0.987	9.94E-04	0.051478
2	Page Model	K=0.241, n=0.056	0.989	8.84E-05	0.045424
3	Modified Page	k=2.14, n=0.099	0.987	9.94E-04	0.051478
4	Henderson and Pabis Model	a=0.981,k=0.207	0.986	0.00111	0.050925
5	Modified Henderson and Pabis Model	a=646056.91, k=7062.44, b=-7.65E13, g=3.74E15, c=0.981, h=0.2076	0.967	0.00259	0.050925
6	Verma et al.	a=0.731, b=0.288, g= 0.097	0.993	6.44E-04	0.00387
7	Peleg Model	a=3.43, b=0.853	0.985	1.14E-03	0.05164
8	Two Term Model	a=0.981, b=0.2075, c=188792.35, k=114819.197	0.980	1.56E-03	0.050925

Table 3 Thin layer drying model co-efficients and its goodness of fit for shade drying

S. No.	Model	Constants	R^2	Chi-Square	RMSE
1	Newton Model	k=0.0224	0.900	0.00231	0.078507
2	Page Model	k=0.075, n=0.7028	0.996	7.89E-05	0.013572
3	Modified Page	k=1, n=0.0224	0.900	0.00231	0.078507
4	Henderson and Pabis Model	a=0.698, k=0.0154	0.986	3.21E-04	0.027386
5	Modified Henderson and Pabis Model	a=1, k=5,b=0.69885, g=0.0154, c=1, h=2	0.967	7.48E-04	0.027386
6	Verma et al.	a=0.698, b=0.015, g= 5.25E7	0.987	3.74E-04	0.0022
7	Peleg Model	a=25.88, b=0.916	0.997	5.11E-05	0.010916
8	Two Term Model	a=0.698, g=0.015, b=2.716, k=4	0.987	4.49E-04	0.027386

Table 4 Thin layer drying model co-efficients and its goodness of fit for vacuum drying

S. No.	Model	Constants	R^2	Chi-Square	RMSE
1	Newton Model	k=0.366	0.946	0.00431	0.02586
2	Page Model	k=0.226, n=1.417	0.995	4.22E-04	0.00221
3	Modified Page	k=0.366, n=1	0.946	0.00431	0.02586
4	Henderson and Pabis Model	a=1.948, k=0.891	0.980	0.00157	0.051218
5	Modified Henderson and Pabis Model	a=1.725, k=538.26, b=-2.30E8, g=752.30, c=1.26, h=0.452	0.980	0.00932	0.00932
6	Verma et al.	a=19.99, b=0.3657, g = 0.365	0.946	0.0064	0.0258
7	Peleg Model	a=3.177, b=0.522	0.979	0.00195	0.0097
8	Two Term Model	a=0.205, g=0.45, b=1.05, k=0.452	0.980	0.0031	0.0093

Table 5 Thin layer drying model co-efficients and its goodness of fit for freeze drying

S. No.	Model	Constants	R^2	Chi-Square	RMSE
1	Newton Model	k=0.210	0.960	0.00305	0.095621
2	Page Model	k=0.124, n=1.329	0.992	5.68E-04	0.038944
3	Modified Page	k=0.324, n=0.6476	0.960	0.00305	0.095621
4	Henderson and Pabis Model	a=1.172, k=0.248	0.985	0.00115	0.055287
5	Modified Henderson and Pabis Model	a=-6.641, k=47.535, b=-4.509E10, g=1.702E14, c=1.17, h=0.2483	0.970	0.00229	0.055287
6	Verma et al.	a=-1.62E-05, b=0.221, g= 0.2216	0.956	0.0043	0.0302
7	Peleg Model	a=5.44, b=0.5034	0.973	0.0021	0.074811
8	Two Term Model	a=-0.7869, g=0.7527, g=1.699, k=0.3093	0.991	6.67E-04	0.036515

3.4 Extraction of essential oil

The percentage of essential oil extracted from the dried betel leaves by various drying methods are shown in Table 6. The oil yield from fresh betel leaves was 2.1% on dry weight basis (db). This yield decreased to 1.5% (db) for both the shade drying and freeze drying techniques and was even less for hot air oven drying (1%, db) and vacuum drying (0.8%, db). The reason behind this can be explained by the difference in drying conditions of the selected drying methods. In case of hot air drying and vacuum drying, the temperature was maintained at 60°C, but the pressure was 50% less in vacuum drying (380 mmHg) than that of hot air oven drying (760 mmHg).

Table 6 Essential oil extracted from dried betel leaf obtained from different drying methods

Sl. No.	Drying type	Percentage of oil obtained (%)
1.	Fresh Betel leaf	2.1^{a}
2.	Shade drying	1.5^{ab}
3.	Hot air oven drying	1.0^{bc}
4.	Vacuum drying	0.8^{c}
5.	Freeze drying	1.5^{ab}

Note: All values are mean ± SD and the values are in same column with sharing same superscript letters are not statistically significant at significane level of 95%.

Therefore, the evaporation rate of voltile components of the betel leaves increased in vacuum drying process compared to hot air oven drying and subsequently, decreased the essential oil content in the vacuum dried leaves. On the other hand, the same amount of essential oil (1.5%) was extracted from the shadedried and freeze dried leaves. These results show that the essential oil content decreased by 28.57% in the dried leaves (Shade and freeze drying) compared to that of fresh leaves. However, this decrease in the essential oil content in the dried leaves was not statistically significant with essential oil content of fresh leaves at 95% significance level. On the other hand, the loss of the essential oil content in vacuum dried and hot air oven dried leaves was statistically significant ($p<0.05$) from the oil content of fresh leaves. Therefore, it may be stated that freeze drying and shade drying techniques are superior methods of drying in comparison to vacuum drying and hot air oven drying for retaining the essential oil in the dried betel leaves. The amount of essential oil in the leaves are very important so far as the organoleptic quality of the product is concerned. Therefore, higher the amount of the oil in the dried leaves, better is the quality of the leaves. Therefore, freeze drying and shade drying may be considered for obtaining superior dried leaves. However, shade drying took long time (10 days) and prone to microbial contamination, but freeze drying took comparatively shorter time (10 h) and not prone to mirobial contamination. However, the latter method is more costly than the former. Accordingly, one has to select a drying method based on time, quality and cost suitable to one's purpose.

3.5 Sensory evaluation

Hedonic scale is one of the unique and reliable procedure for evaluation of organoleptic properties of any developed product (Lu et al., 2010). Therefore, this method was followed in the present study. Accordingly, colour, taste and smell of the dried betel leaves were evaluated by the panelists and the data are placed in Table 7. From these data pertaining to the color, it may be stated that shade drying occupied the first position (most preferred quality) and the vacuum drying occupied the last position (inferior quality) and the other methods occupied intermediate positions. It can also be observed that shade drying was significantly better than all other methods, whereas freeze drying and hot air oven drying were at par. On the other hand, similar results were also obtained from the data pertaining to the taste. However, there was no significant difference in the sensory score of freeze drying, hot air oven drying and vacuum drying. That apart, from the data pertaining to the smell, it may be stated that shade drying occupied the first position and the freeze drying occupied the last position and the other methods occupied intermediate positions. It can also be observed that shade drying was better than all other methods, whereas hot air drying and vacuum drying, and vacuum drying and freeze drying were at par. In addition, no significant difference was found among the other methods. Therefore, from the sensory data of the dried leaves, it can be pointed out that the freeze dried leaves were the second choice among the panellists, whereas shade dried leaves were the first and vacuum dried leaves occupied the last position.

Table 7 Sensory evaluation of the dried leaves

Attributes	Shade drying	Hot air oven drying	Vacuum drying	Freeze drying
Colour	$7.8±0.4^{a}$	$6.4±0.8^{b}$	$3.8±0.2^{c}$	$6.8±0.6^{b}$
Taste	$6.8±0.3^{a}$	$5.6±0.5^{b}$	$5.3±0.4^{b}$	$5.76±0.5^{b}$
Smell	$6.6±0.5^{a}$	$5.3±0.6^{b}$	$4.7±0.8^{bc}$	$4.6±0.3^{c}$
Overall acceptability	$7.06±0.5^{a}$	$5.76±0.6^{c}$	$4.6±0.32^{d}$	$6.43±0.1^{b}$

Note: All values are mean ± SD and the values are in same row with sharing same superscript letters are not statistically significant at significance level of 95%.

4 Conclusion

In this study, four different drying methods were applied for drying of the betel leaves. From the drying rate curve, it can be observed that leaves were only dried in falling rate drying period and moisture loss was faster in vacuum drying process than the other methods. Among the eight thin layer emperical drying model investigated in this study, Page model was best fitted for hot air oven drying and vacuum drying, whereas Peleg model was found to be the best fitted model for shade drying. However, drying kinetics of freeze drying process was best described by Two Term model.

As far as sensory quality of the dried leaves is concerned, superior quality dried leaves were obtained with the shade drying. The leaf quality was better under this treatment mostly due to retention of a higher amount of essential oil in the dried product compared to other methods such as vacuum drying and hot air oven drying. However, during shade drying, the leaves are susceptable to microbial contamination due to prolonged period of drying. On the other hand, freeze drying process produced similar quality of betel leaves as that of shade dried leaves, but not susceptable to microbial contamination. Hence, from this study, it can be concluded that freeze drying can be preffered for drying of betel leaves over other three drying methods.

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