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Studies on intermittent and stepwise decremental microwave power drying of dill leaves

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Abstract
Green leafy vegetables are highly perishable due to high moisture content. An investigation was undertaken to study the dehydration of dill leaves using Intermittent and Stepwise Decreasing Microwave Power Drying to get quality dried food in very short drying time. The variation in chlorophyll, protein, calcium, iron and ascorbic acid between fresh and dried leaves was 7.12 %, 2.58%, 3.33%, 6.68% and 5.88% respectively. The treatment of decreasing power of microwave field with decreasing levels of moisture was given to reduce heat stroke to dill leaves, those otherwise loose useful nutrients. The pulse ratio of 1.5 to 2 shows best results with respect to product quality and reduced final moisture content. The OFF time during drying helps in retention of colour with maximum nutrient. This drying process improves energy efficiency and product quality without increasing capital cost of the drier and enables the drying without chemical pretreatment.

Keywords: *dehydration, drying, energy, intermittent, microwave*

Introduction
For human good health, vegetables are most affordable source of vitamins and minerals (Sedani *et al*., 2018) [33]. Green Leafy Vegetables (GLVs) are source of nutrient required for healthy and disease-free body. GLVs are multi-cultural components used ubiquitously in Indian cuisine. India ranks second in world for production of vegetable next to china. Over 40 groups of root crops and leafy vegetables like Solanaceous, cucurbitaceous, leguminous, cruciferous etc. are grown in tropical, subtropical and temperate regions of India (Kakade and Neha, 2014) [18].

Dill plant is scientifically named as *Anethum graveolens* and is an annual herb belonging to celery family *Apiaceae*. It is a single species in the genus *Anethum.* Fresh and dried dill leaves are widely used as herbs in Europe and central Asia (Gupta *et al*., 2012) [10]. In India, dill is known as *savaa* in hindi and by different names in different parts of India. Dill leaves are rich in minerals, proteins and fibre. The seeds are mainly used for spice, culinary and medicinal uses. It is also used as a vegetable and an aromatic (Sharma, 2014), and as an inhibitor of sprouting in stored potatoes (Score *et al*., 1997) [31]. The aroma volatiles of seed and herb of dill have been identified (Huopalahti and Linko, 1983, Shankaracharya *et al*., 2000) [13, 35] along with several therapeutic properties, and the antimicrobial activity of carvone isolated from the dill seed oil has been reported (Aggarwal *et al*., 2002) [1]. Dill contains carotenoids, vitamin C and polyphenols, the contents of which vary during different stages of its growth (Lisiewska *et al*., 2006). Hosseinzadeh *et al.* (2002) and Rifat-uz-Zaman *et al*. (2006) [20, 11, 29] reported the anti-ulcer activity of dillfruits. A. graveolens also possesses potent hypocholesterolaemic effects in rats, probably mediated through the suppression of endogenous cholesterol biosynthesis by inhibition of the activity of HMG-CoA reductase (Yazdanparast and Bahramikia, 2008) [44]. Myristin in dill is psychoactive and hallucinogenic and the apiole content may be responsible for the diuretic properties (Mazyad *et al*., 1999; Delaquis *et al*., 2002, Singh *et al*., 2002; Lopez *et al*., 2005) [24, 6, 37, 21].

Dill leaves are highly perishable due to high moisture content and need to preserve through convenient processing techniques. Serious losses occurred due to unavailability of adequate storage, transport and appropriate processing facilities at the production site (Pande *et al*., 2000) [28]. Dehydration seems to be the simplest technology for preserving GLVs, especially when they are abundantly available. The energy efficiency of the method used for and quality of dried products obtained from, are two key factors in food drying. The most serious constraint for shelf-life enhancement is the activity of micro-organisms at higher moisture content in food. During dehydration, water in food is reduced to a very low level thus better preservation from microbial action and retarding many undesirable reactions during storage is achieved (Ibarz and Barboda-Canovas, 2000) [14], owing to their reduced water activity. The GLVs are wealthy sources of micronutrients as already mentioned earlier and therefore dehydrating them can provide us a concentrated source of micronutrients. It possible to control the drying process more precisely to obtain greater yields and better quality products in shortest possible time using microwave energy-alone or in combination with conventional energy sources. In the present study, a new dehydration technology is utilized for preservation.

Soysal (2004) [39] studied the influence of microwave output power on drying kinetics and color of parsley leaves. Alibas (2007) [2] studied the efficacy of microwave, convective and vacuum drying techniques and determined the optimum drying methods for the drying of nettle leaves. Doymaz *et al.* (2006) [7] determined the effect of drying air temperature on the drying time and calculated effective diffusivity and activation energy for dill and parsley leaves. Gunhan *et al.* (2004) [9] determined the effect of air drying temperature and relative humidity for drying of bay leaves.

Sedani and Pardeshi (2018) [34] studied stepwise decreasing microwave power drying of dill leaves. It was found that using decreasing microwave power helped reduce heat stroke to dill leaves during said drying. The present study aimed to reduce heat stroke to dill leaves and to improve the quality of dried product, as it is heat sensitive.

Materials and methods

Sample preparation

Dill leaves was collected from local market of Akola. The initial moisture content of the Dill leaves was obtained by oven dry method. The dill leaves were washed and trimmed. The surface moisture was removed before drying.

Experimental set up and plan of experiment for Dill leaves

Experimental Setup consists of Microwave oven of IFB Company with 900W rated power with model No. 30SC4, Stopwatch for measuring laps time, weighing balance with precision of 0.001g. Central composite rotatable design (CCRD) was used for carrying out this experiment. In this study, two variables i.e., ON time and OFF time with range 30 s to 120 s and 0 s to 120 s, respectively, were taken. These ranges were selected on basis of preliminary trials conducted by Sedani, (2019) [32]. The experimental design was applied after selection of particular ranges. Thirteen experiments were conducted with two variables and five levels. Experiments were randomized to weaken the effect of unexplained variability within the observed responses because of extraneous factors. The centred point in the design was repeated five times to calculate the reproducibility of the method (Montgomery, 2001) [25].

The pulse ratio (Eştürk and Soysal, 2010) [40] was estimated by the Eq. 1 given below,

$$\text{PR (Pulse Ratio)} = \frac{\text{ON time+OFF time}}{\text{ON time}} \quad (1)$$

Moisture:

The moisture content of the fresh and dehydrated dill leaves was determined by AOAC method (AOAC, 2000) [5]. The moisture content of the dill leaves was computed using the following equations,

$$\text{Moisture content (\% w.b)} = \frac{M_1 - M_2}{M_1} \quad (2)$$

Where, M_1 is the weight of sample before oven drying (g), M_2 is the weight of sample after oven drying (g). The samples were placed in Hot Air Oven. The weight M_2 was taken after allowing the samples to cool down upto atmospheric temperature, by keeping the samples in desiccators having filled with silica gel in its bottom portion. The samples were weighed using electronic weighing balance with accuracy 0.001 g. The equilibrium moisture content was assumed zero for microwave oven drying (Maskan, 2000) [23] as removal of moisture continues with time and subsequently product may get burnt (Soysal, 2004) [39].

Chlorophyll

The method of McKinney (1941) [22] was used to measure chlorophyll content of the dehydrated dill leaves. Chlorophyll is extracted in Dimethyl Sulphoxide (DMSO) by heating and absorption at 645 nm and 663 nm, read in a spectrophotometer using the absorption coefficient. The amount of chlorophyll a, b and total chlorophyll were calculated by using formulae,

$$\text{Chlorophyll a} = 12.7\,(\text{OD } 663) - 2.69\,(\text{OD } 645) \times \frac{V}{1000 \times W}$$

$$\text{Chlorophyll b} = 22.9\,(\text{OD } 645) - 4.68\,(\text{OD } 663) \times \frac{V}{1000 \times W}$$

$$\text{Total chlorophyll} = 20.2\,(\text{OD } 645) + 8.02\,(\text{OD } 663) \times \frac{V}{1000 \times W} \quad (3)$$

Protein

Total nitrogen of moisture free sample was estimated by micro-Kjeldhal method employing auto analyzers (Kejlplus Pelican apparatus). Organic nitrogen was digested in digestion instrument with concentrated sulphuric acid in the presence of a catalyst, converted into ammonium sulphate and titrated to obtain nitrogen. Futher nitrogen was multiplied with conversion factor 6.25 to obtain crude protein (AOAC, 1990) [4].

$$\text{Nitrogen \%} = \frac{(\text{Sample titre} - \text{blank titre}) \times \text{Normality of HCL} \times 14 \times \text{Volume made up} \times 100}{\text{Aliquot of digestion taken} \times \text{Weight of the dried sample} \times 1000}$$

$$\text{Protein \%} = \text{Nitrogen \%} \times f \quad (4)$$

Where 'f' is multiplying factor as 6.25 for dried GLVs (Thimmaiah, 2006) [43].

Calcium

Calcium was determined by precipitating it as calcium oxalate and titrate the solution of oxalate in dilute H_2SO_4 against standard $KMnO_4$ as stated in AOAC, 1990 [4].

Iron

Total Iron from plant sample was estimated by diacid extract using atomic absorption spectrophotometer by Issac and

Kerber (1971) [16]. Diacid extract was prepared as per method outlined by Jackson (1973) [17]. The ratio of 9:4 mixture of HNO_3: $HClO_4$ was used to perform analysis.

Ascorbic acid

The capacity of a sample to reduce a standard dye solution, as determined by titration, is directly proportional to the ascorbic acid content. Standardize the dye and calculate dye factor shown in Eq. 5. Take 10 ml sample and make upto 100 ml with 3% HPO_3 and filter. Pipette 10 ml of filtrate into conical flask and titrate with the standard dye to a pink end-point (Srivastava and Kumar, 2002) [41].

$$\text{Dye factor (D.F.)} = \frac{0.5}{\text{Titre}} \quad (5)$$

Calculation:

$$\text{Ascorbic acid} = \frac{\text{Titre} \times \text{Dye factor} \times \text{volume made up} \times 100}{\text{volume of filtrate taken} \times \text{wt.or volume of sample taken}} \quad (6)$$

Color analysis

The colour (L, a and b-value) was measured using Chromameter (CR-400). Hunter L, a, b color scales based on the Opponent-Color Theory required to completely describe an object‘s color. The receptors in the human eye perceive color as the following pairs of opposites according to Opponent-Color Theory.

- L scale: Light (0-50) vs. dark (51-100)
- a scale: Red (+) vs. green (-)
- b scale: Yellow (+) vs. blue (-)

A three-dimensional representation of L, a, b color space is shown in Fig 1 below (Hunter *et al*., 1987) [12]

$$\Delta a = a_{fresh} - a^* \quad (7)$$

$$\Delta b = b_{fresh} - b^* \quad (8)$$

$$\Delta L = L_{fresh} - L^* \quad (9)$$

$$\Delta E = \sqrt{(\Delta L^2 + \Delta a^2 + \Delta b^2)} \quad (10)$$

Where, ΔE = Change in colour of dried sample with respect to fresh sample of dill leaves.

Statistical analysis

The data were analyzed for its stepwise regression analysis in Design Expert 11 Software (Stat-Ease, Inc. 2018) [42].

Results and Discussion

Effect of process parameter on final moisture content (kg/kg of dm) on intermittent and Stepwise decreasing microwave power drying (ISDMD) Dill leaves

The treatment combinations and the observations recorded are shown in Table 1. The data were analyzed for its stepwise regression analysis as shown in Table 2. It could be observed that the values of Final Moisture Content (FMC) ranged between 1.1298 to 136.43 kg/kg dm. The Reduced Cubic model was fitted to the experimental data and statistical significance for terms was calculated for final moisture content. The R^2 value was calculated by a least square technique and found to be 0.997, showing good fit of model to the data. The model F-value of 2508.82 implies that the model was significant ($P < 0.0001$). If A indicates ON time and B indicates OFF time in ANOVA table. The terms B and A^2B are significant ($P < 0.05$). The lack of fit F-value was non-significant, which shows that the developed model was satisfactory for predicting the response. Moreover, the model adequacy evaluated with predicted R^2 of 0.9965 showed it to be in reasonable agreement with the adjusted R^2 of 0.9993. This indicated that the non-significant terms have not been included in the model. Therefore this model could be used to navigate the design space. The regression equation describing the effects of the process variables on final moisture content in terms of actual levels of variables is given as,

$$\text{Final Moisture Content (FMC)} = 305.20828 - 6.76243A + 3.42660B - 0.062755AB + 0.038673A^2 - 0.017489B^2 + 0.000157A^2B + 0.000288AB^2 \quad (11)$$

The comparative effect of ON time and OFF time on the final moisture content could be observed by the F-values in the ANOVA and also by the magnitudes of coefficients of the coded variables. The F-values indicated that A was the most influencing followed by A^2, AB^2, B^2, respectively over final moisture content. The B was least influencing while AB and A^2B had moderate effect on final moisture content. To visualize the combined effect of two variables on the final moisture content, the response surface and contour plots were generated for the fitted model as a function of two variables. It could be observed from Fig. 1 (A) that final moisture content decreased with increase in ON time and increased with increase in OFF time as depicted in Eq. 11. It may be due to utilization of residual heat of product to remove moisture during OFF time. When product was heated during ON time, the total heat generated was more than that required for removing moisture during ON time. Thus accumulated residual heat was utilized during OFF time for subsequent removal of moisture.

Table 1: Treatment combinations for intermittent and stepwise decremental microwave power drying of dill leaves

Sr No.	Coded Values		Actual Values		Response 1	Response 2
	X_1	X_2	ON time	OFF time	FMC	ΔE
1.	-1	-1	43	18	102.06	3.7939
2.	1	-1	107	18	2.7056	8.2749
3.	-1	1	43	102	136.43	1.805
4.	1	1	107	102	12.28	12.6347
5.	-1.414	0	30	60	205.88	2.60671
6.	1.414	0	120	60	1.1298	6.403
7.	0	-1.414	75	0	15	8.267
8.	0	1.414	75	120	26.9864	6.18
9.	0	0	75	60	4.54	7.51872
10.	0	0	75	60	5.67	8.2918
11.	0	0	75	60	6.4645	9.38374
12.	0	0	75	60	7.6754	7.9372
13.	0	0	75	60	8.978	8.9817

Table 2: ANOVA for reduced cubic model of dill leaves for final moisture content (kg/kg of dm)

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	50665.09	7	7237.87	2508.82	< 0.0001**
A-ON time	20961.32	1	20961.32	7265.70	< 0.0001**
B-OFF time	71.84	1	71.84	24.90	0.0041*
AB	153.71	1	153.71	53.28	0.0008**
A^2	16498.46	1	16498.46	5718.76	< 0.0001**
B^2	385.46	1	385.46	133.61	< 0.0001**
A^2B	91.08	1	91.08	31.57	0.0025*
AB^2	545.43	1	545.43	189.06	< 0.0001**
Residual	14.42	5	2.88		
Lack of Fit	2.51	1	2.51	0.8419	0.4108^{NS}
R^2	0.9997				
Adjusted R^2	0.9993				
Predicted R^2	0.9965				
Std. Dev.	1.70				
C.V. %	4.12				

***p<0.001, **p<0.01, *p<0.05, ^{NS}Non Significant

Effect of process parameter on change in colour (ΔE) for intermittent and Stepwise decreasing microwave power drying (ISDMD) Dill leaves

The treatment combinations and the observations recorded are shown in Table 1. The data were analyzed for its stepwise regression analysis as shown in Table 3. It could be observed that the values of ΔE were ranged from 1.805 to 12.6347. The Reduced Cubic model was fitted to the experimental data and statistical significance for linear and quadratic terms was calculated for ΔE. The R^2 value was calculated by a least square technique and found to be 0.9217, showing good fit of model to the data. The model F-value of 23.54 implies that the model was significant ($P < 0.001$). The term A indicates ON time and term B indicates OFF time. The linear terms of A and AB is significant ($P < 0.1$) model terms while quadratic terms of A^2 and AB^2 are significant ($P < 0.01$) model terms.

The lack of fit F-value was non-significant for the model obtained shown in Eq. 12. Moreover, the predicted R^2 of 0.7905 was in reasonable agreement with the adjusted R^2 of 0.8825. This indicated that the non-significant terms have not been included in the model. Therefore this model could be used to navigate the design space. The regression equation describing the effects of the process variables on ΔE in terms of actual levels of variables is given as,

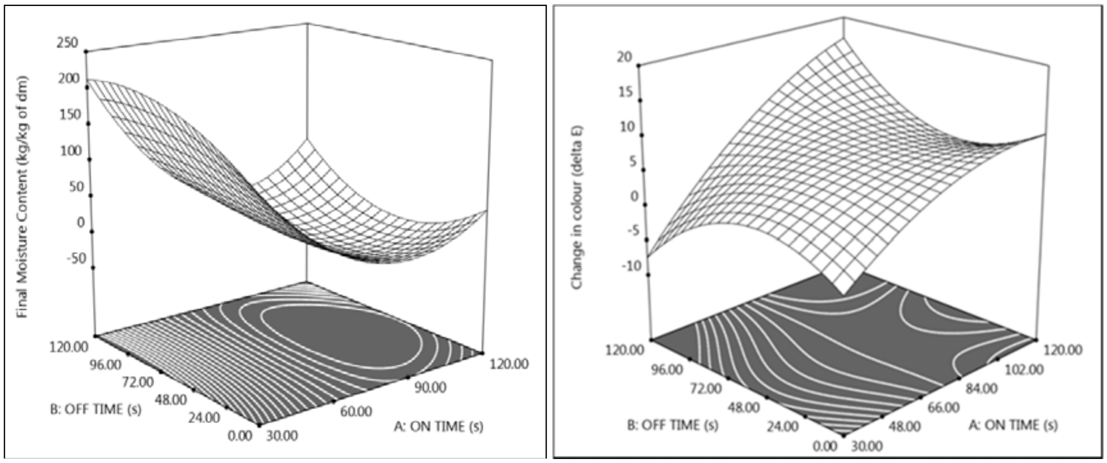
$$\text{Change in colour } (\Delta E) = 0.220691A - 0.10472B + 0.001428AB - 0.0015A^2 - 5.9E - 07AB^2 \quad (12)$$

The comparative effect of ON time and OFF time on the ΔE could be observed by the F-values in the ANOVA and also by the magnitudes of coefficients of the coded variables. The F-values indicated that A^2 was more influencing followed by AB^2, A and AB. The ΔE followed non-linear behaviour with A and AB^2 due quadratic terms of A and AB^2 in Eq. 12. The ΔE increased with increase in ON time upto certain maxima and decresased thereafter with increase OFF time.

Soysal *et al* (2009) [40] reported that the continuous microwave-convective drying resulted in darker product colour as compared to intermittent microwave-convective drying or convective drying during the drying of red pepper, possibly due to the non-enzymatic browning. Thus, for longer ON time, the product might be getting more browned during drying, it causing lowered L-value (brightness) in colour for the dried product. The improvement in L-value can be accredated to higher OFF time as it utilizes the product heat for moisture removal and decreased L-value (brightness) may be due to prolonged exposure of product to higher 8temperature (ON time), leading to the browning of dried sample. The replacement of magnesium in the chlorophyll by hydrogen takes place results in conversion of chlorophylls to pheophytins due to high temperature (Rudra *et al*., 2008) [30]. The same could be revealed from Fig. 1 (B) by visualizing the combined effect of two variables on the ΔE, the response surface and contour plots generated for the fitted model as a function of two variables. This result agreed with Onayemi and Okeibuno (1987) [27] in which slower rate of chlorophyll degradation was found with shorter drying process.

Table 3: ANOVA for reduced cubic model for change in colour (ΔE) for dill leaves

Source	Sum of Squares	Df	Mean Square	F-value	p-value
Model	96.70	4	24.17	23.54	0.0002***
A-ON time	7.21	1	7.21	7.02	0.0293*
AB	10.08	1	10.08	9.81	0.0140*
A^2	20.81	1	20.81	20.26	0.0020**
AB^2	12.36	1	12.36	12.03	0.0085**
Residual	8.22	8	1.03		
Lack of Fit	**5.91**	**4**	**1.48**	**2.56**	**0.1921^{NS}**
R^2	**0.9217**				
Adjusted R^2	0.8825				
Predicted R^2	0.7905				
Std. Dev.	1.01				
C.V. %	14.31				



(A) (B)

Fig 1: The contour and response surface plots showing the effect of (A) Final Moisture Content and (B) Change in colour during ISDMD of Dill Leaves

Optimization of intermittent and stepwise decremental microwave power drying of Dill leaves

Numerical and graphical optimization was carried out for the process parameters for Intermittent and Stepwise decremental microwave power drying (ISDMD) of Dill leaves for obtaining the best product. To perform this operation, Design Expert - version 11 software (Stat-Ease, Inc. 2018) [42], was used for simultaneous optimization of the multiple responses.

Table 4 showed that the software generated optimum condition of independent variables with the predicted values of responses. Solution No. 2, having the maximum desirability value (0.856) and was selected as the optimum conditions of ISDMD of Dill leaves.

The optimum values of process variables obtained by numerical optimization:

ON time (A): 75 s; OFF time (B): 36 s;

The superimposed contours of responses for Final Moisture Content and Change in Colour (ΔE) (Fig. 2) and their intersection zone for minimum Final Moisture Content and minimum ΔE indicated the ranges of variables which could be considered as the optimum range for best product quality.

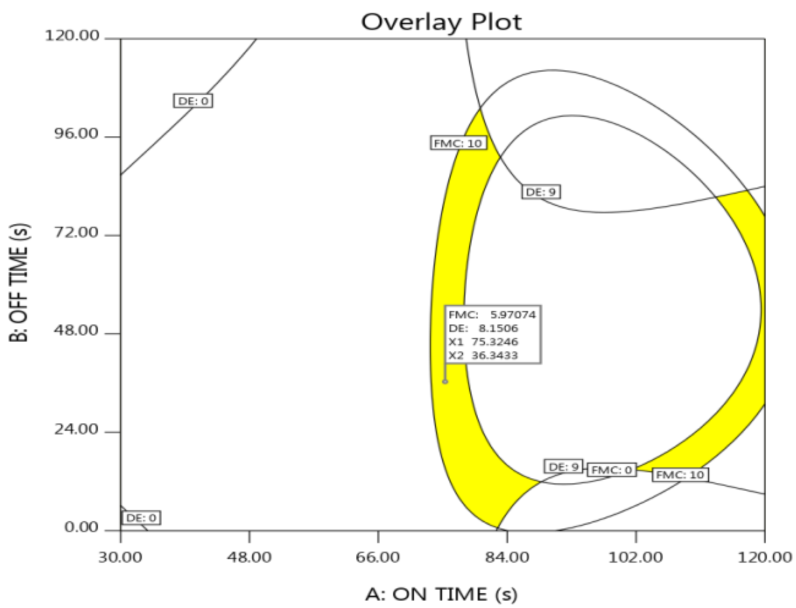


Fig 2: Superimposed counters for Final Moisture Content and Change in colour for ISDMD of Dill Leaves

Verification of the model for intermittent and Stepwise decreasing microwave power drying (ISDMD) of Dill leaves

ISDMD of Dill leaves experiments were conducted at the optimum process condition as shown in Table 4 and the quality attributes of the resulting product were determined. The observed experimental values (mean of 5 measurements) and values predicted by the equations of the model are presented in Table 5. The values of C.V. (<10%) and closeness between the experimental and predicted values of the quality parameters indicated the suitability of the corresponding models (Snedecor and Cochran, 1967; Nath *et al*., 2011) [38, 26].

Table 4: Solutions generated by the software for ISDMD of Dill leaves

No	ON time	OFF time	Final Moisture Content	ΔE	Desirability
1	78.72	35.00	0.00	8.26	0.856
2	75.32	36.34	5.97	8.15	0.856*
3	75.32	60.08	5.94	8.18	0.754
4	78.38	21.80	3.69	8.32	0.721

*selected

Table 5: Comparison between predicted and actual response variables at optimum process conditions for ISDMD of Dill leaves

Response	Predicted value	Actual Value(±SD)	Variation %	C.V. %
Final Moisture Content (kg/kg of dm)	5.9707	5.76 (±0.4489)	3.50	7.79
ΔE	8.1506	8.28 (±0.4015)	1.57	4.85

Effect of intermittent and stepwise decremental microwave drying (ISDMD) on nutritional composition of dill leaves

Dehydration, a process of moisture removal, is presumed to concentrate the nutrient and destroying the heat labile ones. Hence, an experiment was planned to find out the effect of intermittent and stepwise decremental microwave drying on nutritive value of dill leaves. At lower microwave power field, as drying proceeds, there was increase in ΔE due to longer exposure of product to microwave power field because of longer drying time, and at higher microwave power product burns before reaching to required moisture content, due to this the product lost its nutrient and chlorophyll content which makes the product quality poorer. The similar results are reported by Sedani and Pardeshi (2018) [33]. ISDMD gives better quality of product in terms of colour and chlorophyll content. The similar result was reported by Kowalski *et al*. (2013). Kowalski *et al*. (2013) [19] studied the effect of intermittent drying on the quality of dried carrot. Findings indicated that in case constant Hot Air drying was replaced by intermittent drying, a significant impact on the final quality of carrot was observed. In comparison with constant Hot Air drying conditions, intermittent drying of carrot proved more effective in improving the quality of the dried product. The interaction of microwave energy and food products causes internal heat generation. The rapidly alternating electromagnetic field produces intraparticle collisions in the material, and the translational kinetic energy is converted into heat. Microwave heating in a drying system may adversely affect product quality due to non-uniform temperature distribution and difficulty in controlling final temperature of product at low moisture content (Ibrahim *et al*., 2012) [15]. Table 6 gives the nutritional value of dried dill leaves in comparison of fresh dill leaves. The variation in chlorophyll, protein, calcium, iron and ascorbic acid between fresh and dried leaves was 7.12 %, 2.58%, 3.33%, 6.68% and 5.88% respectively.

Table 6: Nutritional Value of Dill Leaves in dry basis

Parameter	Fresh dill leaves	Dried dill leaves
Chlorophyll content (mg/100g)	1.22	1.13
Protein (g/100g)	28.089	27.363
Calcium (mg/100g)	300.00	290.00
Iron (mg/100g)	22.00	20.53
Ascorbic acid(mg/100g)	85.00	73.00

Conclusion

Microwave drying using single power level is not suitable for drying dill leaves as it causes burning effect before it reaches to desired moisture content at high microwave powers whereas at low microwave power, the prolonged drying causes large variation in colour (ΔE), which indicates destruction of micronutrients during microwave power drying. Intermittent and Stepwise Decreasing Microwave Power Drying (ISDMD) of dill leaves at decreasing microwave power levels (900W, 720W, 540W, 360W and 180W) and by allowing drying for specified ON-time (75 s) at each microwave power level and by allowing OFF-time (36 s) between two successive ON-time periods at varied and decreasing microwave power level, the desired moisture content with minimum variation in colour (ΔE) was achieved in case of dill leaves considered for experimentation. The total drying time required was 555s. The final moisture content of 5.76 %(db) with 8.28 ΔE was achieved. The variation in chlorophyll, protein, calcium, iron and ascorbic acid between fresh and dried leaves was 7.12 %, 2.58%, 3.33%, 6.68% and 5.88% respectively.

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