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Effect of hot-air drying on the physicochemical properties of kaffir lime leaves (*Citrus hystrix*)

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Abstract

The effect of hot-air drying namely drying time (3-15 h), drying temperature (40-80°C) and loading capacity (0.5-2.0 kg/m^2) on the physicochemical characteristics of kaffir lime leaves was optimized using Response Surface Methodology. Twenty treatments were assigned based on the second-order CCD including 6 center points, 6 axial points and 8 factorial points. The quality of dried kaffir lime leaves was evaluated by determining moisture content, water activity, texture (brittleness) and Hunter L, a, b values. The results indicated that the most significant ($p<0.05$) hot-air drying conditions that affect the physicochemical properties of dried kaffir lime leaves were the main effect of drying time and drying temperature as well as the quadratic effects of the independent variables. The overall optimum conditions that resulted in desirable dried kaffir lime leaves, were achieved when the drying time was 4.9 h, the drying temperature, 60°C and loading capacity 1.4 kg/m^2.

Key words: Kaffir lime leaves, hot-air drying, physicochemical properties, response surface methodology, optimization, herbs and spices.

Introduction

Kaffir lime (*Citrus hystrix*) is a perennial tropical plant, widely grown in South East Asia [1] for leaves and fruits as food. The lime fruit is wrinkled, pear shaped and dark green turning to yellow on ripening. The leaves are dark green and glossy, usually grow in pairs and look like figure of eight [2]. Usually, both fresh and dried kaffir lime fruits and leaves are rich with strong, pungent and lime-lemon aroma [3] and contain 0.08% essential oil. The leaf contains essential oil (about 80%) such as citronellol, citral, nerol and limonene. These essential oils are able to act as antioxidant and function as anticancer [3, 4]. Kaffir lime leaves play an important role in Thailand, Indonesian and Malaysian cooking. The lime fruits and leaves are mainly used as flavouring in both savoury and sweet food besides having a pleasant lemon smell [2]. The aroma and flavour from the leaves give a distinctive taste to chicken and fish dishes [1]. In Thailand dishes, the leaves are used for imparting aroma in soups, salads, curries and stir-fried dishes. Furthermore, Malays used it as tonics, medicine and hair shampoo. In Thailand, citrus juices have also been included in ointments and as natural bleach.

The leafy spices are highly perishable in nature and therefore have very short shelf life. Usually, they deteriorate rapidly after harvesting which leads to loss of flavour and quality. So, there is a need to preserve the product quality and also to prevent the spoilage of the product during storage with an application of drying technology. Food drying is a traditional way of food preservation, and it has also been used for the production of special foods and food ingredients [5]. The term drying always refers to the removal of a relatively small amount of moisture from a solid or nearly solid material by evaporation [6], which promises microbial stability and guarantees longer expected shelf-life of the product[7]. Most of the drying methods used application of heat on the products to remove moisture. Moreover, good drying technique can enhance the quality of the product significantly [8]. However, different drying conditions and techniques can create diverse food structure [9, 10]. Previous studies revealed that numerous methods of drying have been applied to food materials, especially herbs and spices. Hot air drying is still the most widely used method to produce dried products, because of their lower cost. In addition, air drying of aromatic herbs can be an effective method of preservation that inhibits growth of microorganism and delays some biochemical reactions in the final product [11].

Numerous studies have been conducted on the effects of drying conditions on different plants. Balladin and Headly [12] evaluated selected constituents of solar dried thyme and compared the selected constituents with oven and microwave methods. Demir *et al.* [13] determined optimum conventional drying conditions for bay leaves. The conditions studied included colour, shape, drying time and essential oil. Ozcan *et al.* [14] observed the effect of oven and sun drying on the mineral contents of basil herbs. Mohamed *et al.* [15] investigated the effects of drying air temperature and air flow rate on the drying kinetics of *Citrus aurantium* leaves. Doymaz *et al.* [16] determined the effect of drying air temperature on the drying time and drying characteristics of dill and parsley. Sakhale *et al.* [17] studied the effect of magnesium oxide on curry leaves dried under different drying methods such as sun drying, shade drying and tray drying and the leaves were subsequently analyzed for their nutritional and organoleptic qualities. Derya and Ozcan [18] determined sun, oven and microwave drying characteristics of rosemary leaves and compared traditional sun drying and conventional oven drying methods to the microwave drying method. Therdthai and Zhou [19] determined the characteristics of microwave-assisted vacuum drying of mint leaves in comparison with conventional hot air drying and their effects on the colour and structure of the dried leaves. However, there is very little published work on dried kaffir lime leaves.

Response Surface Methodology (RSM) is the most popular tool used to determine the optimum levels of two or more treatment variables and the aim is to optimize the responses. RSM applications have been performed to model food ingredients [20, 21], optimize food process variables [22, 23] and to optimize orange beverage emulsion [24]. Besides that, RSM can be used for the determination of kinetic constants and investigate enzyme stability and kinetics [25, 26]. RSM has important applications in the design, development and also formulation of new products, as well as in the improvement of existing product design. It defines the effect of independent variables, alone or in combination, on the processes. In addition to analyzing the effects of independent variables, this experimental methodology generates a mathematical model which describes the chemical or biochemical processes [21].

Thus, this study aimed to examine the effect of hot-air drying conditions on the physicochemical characteristics of kaffir lime leaves (*C. hystrix*), and to find the optimum conditions for drying and production of high quality dried material that could be used for a high food grade spicing material.

Materials and Methods

Fresh kaffir lime (*Citrus hystrix*) leaf samples were purchased from the same local supplier to avoid variation as reported by Juhari *et al.* [48]. The samples were separated from stalks and stem and washed thoroughly under running water to remove dirt, and weighed. The excess water was drained and kept in frozen storage at -60°C. Prior to drying experiment, the samples were taken out of storage and thawed at room temperature. The samples were dried under different drying conditions. After drying, the samples were placed into moisture impermeable plastic bags and stored at room temperature until further analysis. Each sample was evaluated for its physicochemical properties.

Drying equipment: A programmable oven with 400 W (Drying Oven Model SMA-112, Japan) was used. The drying oven is equipped with a temperature control function that allows the user to select the required drying temperature and also to adjust the time of processing. The hot-air drying experiments were performed using a drying tray (52 cm x 41 cm). Air was circulated from above and the bottom of the perforated drying tray.

Drying procedure: Fresh kaffir lime (*Citrus hystrix*) leaf samples with different loading capacities (0.5-2.0 kg/m^2) were dried in a drying oven (Smoke Master Model SMA-112, Japan) under different drying temperatures (40-80°C) and drying time (3-15 h) and prepared for optimization procedure based on a central composite design (CCD) (Table 1). The term of loading capacity can be defined as a specified amount of weight per square foot that is allowed to be placed on a given platform (tray). Drying procedures were carried out as reported by Juhari *et al.* [48]. During drying process, samples were turned over every 1 hour for evenly distribution of heat among samples. The oven was switched on 1 hour before the drying process to equilibrate the temperature.

Moisture content: The moisture content of samples was determined (in triplicate) using hot-air oven method at 105°C for constant weight [17]. Prior to moisture content analysis, samples were ground into coarse powder and mixed thoroughly. Crucibles and lids were dried in oven (Memmert Model 500 ULM, Schwabach, Germany) at 105°C for constant weight. Crucibles were weighed without lid after attained room temperature. Samples (2 g) were placed in crucible and dried in the oven at 105°C for constant weight. After that, the crucibles were transferred directly to the desiccators and cooled in the desiccators. Soon after attaining room temperature, the weight of crucible was measured. The loss on drying (LD) was reported as the moisture content (%)[27]:

$$\%\ (\text{w/w})\ \text{LD} = \%\ (\text{w/w})\ \text{moisture} = \frac{100 \times \text{wt loss on drying (g)}}{\text{Wt test portion}}$$

Water activity: Water activity of dried samples were measured using a water activity meter (Aqualab Series 3 TE, Decagon Device, Inc, Pullman, WA, USA) as reported by Yousif *et al.* [11]. Initially, the water activity meter was warmed up before measurement. The chamber of this device was opened and the samples were filled into the cup to ¾ levels before it was returned into the chamber and sealed. The results were read on the meter screen [28].

Texture analysis: Textures of dried herbs were evaluated using instrumental method. A TA.XT2 i plus Texture Analyser (Stable Micro Systems, Godalming, UK) was used for force/displacement measurement with a 5 kg load cell, using a 2 mm cylinder spherical stainless probe SMS (P/2) to measure the force required to penetrate an individual dried kaffir lime leaf sample placed on the centre of corresponding platform with 10 mm diameter. The test settings were test speed 0.5 mm/s, trigger force 10 g, travel distance of the probe 8 mm. The values of rupture strength and brittleness reported were the mean of 20 measurements.

Colour analysis: All dried herbs were analyzed using Hunter Lab colorimeter (Hunter Lab D65 Spectrocolorimeter Ultrascan PRO, Reston, VA) as reported by Yousif *et al.* [11]. Five g of each treatment sample was ground (in triplicate) for 10 s to produce a powder of a uniform colour. The samples were transferred to a 10 cm glass cuvette, and read by a Hunter Lab Scan II Spectrocolorimeter. The instrument, equipped with a D_{65} illuminant and 2° observer optical position, was calibrated using a black plate and standard white plate ($X = 79.8$, $Y = 84.67$, $Z = 91.23$). The results were expressed as Hunter Lab *L* (whiteness/darkness), *a* (red/green), and *b* (yellow/blue) values on the screen of colorimeter and recorded. Each reading gave three different coordinates *L, a* and *b*.

Experimental design and data analyses: The effect of three independent variables, X_1 (drying time), X_2 (drying temperature) and X_3 (loading capacity), on six response variables (Y_1-Y_6, namely moisture content, water activity, texture, colour-L, colour-a and colour-b) were evaluated by using RSM. A central composite design (CCD) (Table 1) was employed (1) to study the main and combined effects of main drying conditions on the physicochemical properties of dried kaffir lime leaves, (2) to create models between the variables; and (3) to use these variables to optimize the drying conditions for the production of high quality food grade spicing material. As shown in Table 2, 20 treatments were assigned based on the second-order CCD with three independent variables. The three independent variable ranges studied for hot-air oven drying were: time (3-15 hours), temperature (40-80°C) and loading capacity (0.5-2.0 kg/m^2) as shown in Table 1. The experiments were randomized in order to minimize the effects of unexplained variability in the actual responses due to extraneous factors. The center points were repeated six times to calculate the repeatability of the method [29]. The matrix of the CCD including the values corresponding to the levels of factors and the treatments is shown in Table 2. As shown in Table 2, the arrangement of CCD presented was in such a way that it will allow the development of the appropriate empirical equations [30, 31].

Statistical analyses: Analysis of variance (ANOVA) and regression surface analysis was conducted to (1) determine regression coefficients and statistical significance of model terms; and (2) to fit the mathematical models to the experimental data, aiming at an overall optimal region for the response variables[31]. The generalized response surface model for describing the variation in response variables is given below:

$$Y = \beta_o + \Sigma\beta_i x_i + \Sigma\beta_{ii} x^2_i + \Sigma\beta_{ij} x_i x_j \quad (1)$$

where Y is the response value predicted by the model; β_o is an offset value; β_i, β_{ii} and β_{ij} are main (linear), quadratic and interaction regression coefficients, respectively. The adequacy of the models was determined using model analysis, lack-of-fit test and coefficient of determination (R^2) analysis [32]. It is suggested that for a good fitness of a response model, R^2 should be at least 0.80 [33]. Atkinson and Donev [34] indicated that the corresponding variables were more significant *(p<0.05)* if the absolute t value becomes larger and p-value becomes smaller. The terms statistically found non-significant (p>0.05) was dropped from the initial models and the experimental data was refitted only to significant (p<0.05) independent variable effects in order to obtain the final reduced model. It should be noted that some variables were kept in the reduced model despite non-significance. For instance, linear term containing the variables was also kept in the model if a quadratic or interaction term containing the variable was significant (p<0.05) [31]. The experimental design matrix, data analysis and optimization procedure was carried out using Minitab v. 14 statistical package (Minitab Inc, PA, USA).

Table 1. Levels of independent variable for hot-air oven drying established according to the central composite design (CCD).

Variable	Independent variable levels				
Independent variables	Low	Center	High	Axial (-α)	Axial (+α)
Drying time (hours)	3	9	15	-1.09	19.09
Drying temperature (°C)	40	60	80	26.36	93.64
Loading capacity (kg/m^2)	0.5	1.25	2.0	-0.01	2.51

Optimization procedure: In this study, both multiple graphical and numerical optimization procedures were applied to determine the optimum different hot-air drying conditions on the physicochemical properties of dried kaffir lime leaves. For graphical optimization procedure, the final reduced models were expressed as three-dimensional (3D) response surface plot to better visualize the interaction effect of main drying conditions on the physicochemical properties of dried kaffir lime leaves. The 3D plots were drawn by keeping one variable constant at the center point and varying the other two variables within the experimental range in order to show how each response variable related to two continuous design variables. For numerical multiple

Table 2. The matrix of central composite design (CCD) and experimental data obtained for the response variables studied (Y_{1-6}) (mean ±SD).

		Independent variables			Response variables					
Treatment runs	Blocks	Drying time (hours) (X_I)	Drying temperature (°C) (X_2)	Loading capacity (kg/m^2) (X_3)	Moisture content (%) (Y_1)	Water activity (Aw) (Y_2)	Texture (brittleness) (kg/mm) (Y_3)	Colour (powder - coarsely ground form)		
								L (Y_4)	a (Y_5)	b (Y_6)
1	1	9.00	40.00	-0.01	13.44 ± 0.29	0.24 ± 0.02	6.59 ± 0.44	43.69 ± 1.11	-1.58 ± 0.14	10.22 ± 0.55
2	1	19.09	60.00	1.25	4.69 ± 0.08	0.39 ± 0.02	7.11 ± 0.65	43.88 ± 2.19	0.60 ± 0.03	11.80 ± 0.36
3(C)	1	9.00	60.00	1.25	5.57 ± 0.34	0.37 ± 0.02	6.65 ± 0.45	46.38 ± 0.09	0.61 ± 0.02	13.61 ± 0.14
4	1	15.00	80.00	0.50	4.62 ± 0.19	0.46 ± 0.02	6.85 ± 0.64	44.20 ± 0.40	-0.56 ± 0.05	13.03 ± 0.55
5	1	-1.09	60.00	1.25	51.70 ± 0.21	0.98 ± 0.00	4.32 ± 0.56	40.54 ± 0.80	-0.55 ± 0.08	8.72 ± 0.63
6	1	3.00	80.00	0.50	7.12 ± 0.05	0.58 ± 0.01	7.05 ± 1.09	49.14 ± 0.11	0.65 ± 0.02	12.15 ± 0.06
7	2	3.00	40.00	2.00	46.14 ± 0.34	0.97 ± 0.00	4.99 ± 0.20	44.66 ± 0.19	-2.09 ± 0.02	11.20 ± 0.27
8	2	15.00	40.00	0.50	23.00 ± 0.12	0.85 ± 0.00	4.50 ± 0.53	45.64 ± 0.48	0.44 ± 0.03	11.04 ± 0.27
9(C)	2	15.00	40.00	1.25	5.79 ± 0.07	0.36 ± 0.01	6.90 ± 0.55	44.43 ± 0.31	1.00 ± 0.04	12.85 ± 0.09
10	2	15.00	40.00	2.00	36.77 ± 0.07	0.96 ± 0.01	4.74 ± 0.45	37.68 ± 2.09	0.39 ± 0.06	10.22 ± 0.26
11	2	3.00	40.00	0.50	57.73 ± 0.78	0.99 ± 0.01	4.25 ± 0.44	42.52 ± 0.37	-1.65 ± 0.06	10.92 ± 0.15
12	2	3.00	80.00	2.00	5.29 ± 0.18	0.44 ± 0.04	7.40 ± 1.00	47.44 ± 0.50	0.22 ± 0.06	12.66 ± 0.42
13(C)	3	9.00	60.00	1.25	5.57 ± 0.34	0.36 ± 0.02	6.65 ± 0.45	46.38 ± 0.09	0.61 ± 0.02	13.61 ± 0.14
14	3	9.00	93.64	1.25	4.14 ± 0.02	0.40 ± 0.03	7.69 ± 0.58	43.35 ± 0.97	0.63 ± 0.01	11.00 ± 0.17
15	3	9.00	60.00	2.51	14.27 ± 0.39	0.38 ± 0.02	7.04 ± 0.40	44.62 ± 0.23	-1.85 ± 0.02	10.62 ± 0.14
16(C)	3	9.00	60.00	1.25	5.57 ± 0.34	0.37 ± 0.02	6.65 ± 0.45	46.38 ± 0.09	0.61 ± 0.02	13.61 ± 0.14
17(C)	3	9.00	60.00	1.25	5.79 ± 0.07	0.36 ± 0.01	6.90 ± 0.55	44.43 ± 0.31	1.00 ± 0.04	12.85 ± 0.09
18	3	15.00	80.00	2.00	5.67 ± 0.11	0.25 ± 0.02	6.82 ± 0.47	47.31 ± 0.20	-0.80 ± 0.03	13.31 ± 0.14
19	3	9.00	26.36	1.25	54.37 ± 0.09	0.98 ± 0.00	4.47 ± 0.61	45.09 ± 0.15	-4.14 ± 0.11	12.09 ± 0.14
20(C)	3	9.00	60.00	1.25	5.79 ± 0.07	0.36 ± 0.00	6.90 ± 0.55	44.43 ± 0.31	1.00 ± 0.04	12.85 ± 0.09

(C), Center point.

optimizations, the response optimizer was carried out by using the Minitab software for determining the exact optimum level of independent variables leading to individual and overall response goals. Response optimizer allows us to compromise a single response or a set of responses. This numerical response optimization allows us to interactively change the input variable settings to perform sensitive analyses and possibly improve the initial solution [24].

Verification of models: Both practical and theoretical methods can be applied for validation. For practical method, the experimental data were run again by using optimum point obtained by each factor. The model is validated practically if obtained data are close to predicted data. For theoretical method, the experimental data were compared with predicted values in order to verify the adequacy of final reduced models by using 2-sample t-test. Close agreement and no significant difference must exist between the experimental data and predicted values [24].

Table 3. Regression summary and analysis of variance for moisture content, water activity, texture, colour L, colour a and colour b in uncoded form of process variable.

	Responses					
Regresssion coefficient	Moisture content	Water activity	Texture	Colour, L	Colour, a	Colour, b
b_o	181.049	2.746	0.804	41.666	-14.108	8.506
b_1	-7.840	-0.086	0.268	0.635	0.477	0.450
b_2	-3.544	-0.053	0.149	0.026	0.317	-
b_3	-	-	-	-1.928	2.969	3.207
b_1^2	0.205	0.004	-0.012	-0.023	-	-0.021
b_2^2	0.019	-0.0004	-0.001	-0.0003	-0.002	-
b_3^2	-	-	-	-0.257	-1.251	-1.247
b_{12}	0.044	-	-	-0.001	-0.007	-
b_{13}	-	-	-	-0.147	-	-
b_{23}	-	-	-	0.060	-	-
R^2	0.928	0.892	0.840	0.315	0.808	0.469
R^2(adj)	0.902	0.863	0.798	0.000	0.720	0.328
Regression (P value)	0.000*	0.000*	0.000*	0.836†	0.000*	0.039*
Lack of fit (F value)	3.440	5.750	17.380	13.250	18.080	0.910
Lack of fit (P value)	0.055†	0.010*	0.000*	0.007*	0.003*	0.493†

b' ~ The estimated regression coefficient for the main linear effect; b3 The estimated regression coefficient for quadratic effect; b4 The estimated regression coefficient for the interaction effects: (1) drying time; (2) drying temperature; and (3) loading capacity. *Significant (*P*<0.05). †Not significant (*P*>0.05).

Results and Discussion

Fitting the response surface models: In this study, multiple regression analyses were carried out using response surface analysis to (1) determine regression coefficients and statistical significance of model terms and fitting (2) the mathematical models to the experimental data, aiming at and overall optimal region for the response variables [31]. Lasekan and Abbas [35] demonstrated that the response surface analysis allows the development of an empirical relationship where each response (Y_i) was assessed as a function of time (X_1), temperature (X_2) and loading capacity (X_3) and predicted as the sum of constant (b_o), three first-order effects (linear terms in X_1, X_2 and X3), three interaction effects (interactive terms in X_1X_2, X_1X_3 and X_2X_3) and three second-order effects (quadratic terms in X_1^2, X_2^2 and X_3^2). The estimated regression coefficient of response surface models with the corresponding R^2 values and lack of fit test are reported in Table 3. The R^2 values for these response variables were between 0.315 and 0.928. It can be seen from Table 3 that the regression models for the response variables were significant by the test at the 5% confidence level ($p<0.05$). The results exhibited that the final reduced models were significantly ($p<0.05$) fitted for all response variables studied, namely moisture content, water activity, texture, colour-L and colour-a as well as colour-b (Table 3). There was not significant ($p>0.05$) lack of fit for the regression models fitted for all responses. These results suggested that this model adequately fits the data.

In order to assess the goodness of fit, ANOVA was used to analyze the results obtained. Only terms found significant ($p<0.05$) were included in the final reduced model. Eq. 2 to 7 showed that the models obtained predicting for the response variables explained the main quadratic and interaction effects of factors affecting the response variables. As shown in Table 3, the sign and magnitude of the coefficients indicate the effect of the variables on the responses. As mentioned by Montgomery *et al.* [30] and Lasekan *et al.* [20], a negative coefficient means a decrease in response when the level of the variable is increased, whereas a positive coefficient indicates an increase in the response. Furthermore, a significant interaction suggests that the level of one of the interactive variables may increase while that of the other may decrease for a constant value of the response. The lack of fit, which is used to measure the fitness of models, exhibited no significant F value ($p>0.05$) in terms of the response variable studied. The results, as shown in Table 3, indicated that the models were accurate for predicting those response variations. The following response surface models, Eq. 2 to 7 were fitted to the response variable (Y_i) and three independent variables (X_1, X_2 and X_3):

Moisture content:

$$Y_1 = 181.049 - 7.840X_1 - 3.544X_2 + 0.205X_1^2 + 0.019X_2^2 + 0.044X_1X^2 \quad (2)$$

Water activity:

$$Y_2 = 2.746 - 0.086X_1 - 0.053X_2 + 0.004X_1^2 + 0.0004X_2^2 \quad (3)$$

Texture:

$$Y_3 = 0.804 + 0.268X_1 + 0.149X_2 - 0.012X_1^2 - 0.001X_2^2 \quad (4)$$

Colour, L:

$$Y_4 = 41.666 + 0.635X_1 + 0.026X_2 - 1.928X_3 - 0.023X_1^2 - 0.0003X_2^2 - 0.257X_3^2 - 0.001X_1X_2 - 0.147X_1X_3 - 0.060X_2X_3 \quad (5)$$

Colour, a:

$$Y_5 = -14.108 + 0.477X_1 + 0.317X_2 + 2.969X_3 - 0.002X_2^2 - 1.251X_3^2 - 0.007X_1X_2 \quad (6)$$

Colour, b:

$$Y_6 = 8.506 + 0.450X_1 + 3.207X_3 - 0.021X_1^2 - 1.247X_3^2 \quad (7)$$

Effect of different drying variables on physicochemical characteristics (moisture content, water activity, texture and colour) of kaffir lime leaves (Citrus hysrix)

Moisture content: As shown in Table 4, the moisture content (Y_1) has significantly ($p<0.05$) affected by the main effect of the drying temperature and time as well as their quadratic effects. Also, the interaction effect of drying temperature and time was significant ($p<0.05$). The results indicated that the quadratic and main effects of drying temperature and time had the most significant ($p<0.05$) effects on the moisture content of the dried kaffir lime leaves. The interaction effect of independent variable also exhibited significant ($p<0.05$) effect on the moisture content. Also, the results showed that the regression model for the response variable was significant by the F-test at the 5% confidence level ($p<0.05$). The R^2 value (0.928) for the response variable ensures a satisfactory fitness of the regression model to the experimental data (Table 3).

To aid visualization, the 3D response surface plot for the moisture content during drying is shown in Fig. 1a. The individual optimum region led to a minimum moisture level ($Y_1 = 10.70 \pm 0.18$ dry basis) predicted to be obtained at 4.9 h of drying time, 60°C drying temperature and 1.4 kg/m^2 loading capacity. These results were in agreement with spice qualities and specifications listed by Muggeridge *et al.* [36] where the moisture content (after drying) that is required for spices or similar products in the market is 12% maximum. Fig. 1a shows the surface plot, were the moisture concentration decreases as the drying time and drying temperature were increased, respectively. The reason for this is because of the wide ranges in the drying time from 2.45 to 20 hours [17]. Moreover, this behavior may be caused by excitation of molecules, where molecules are in increased state of excitation increase when temperature increased, thus rising their distance reduces the attractive force between them [37]. These findings are consistent with Doymaz [38], Abalone *et al.* [39] and Vega *et al.* [40] who found that drying temperature had significant ($p<0.05$) negative effect on moisture content for hot-air drying of okra, amaranth seeds and aloe vera, respectively.

Water activity: Similarly, the water activity (Y_2) was significantly ($p<0.05$) affected by both the main effect of the drying time and

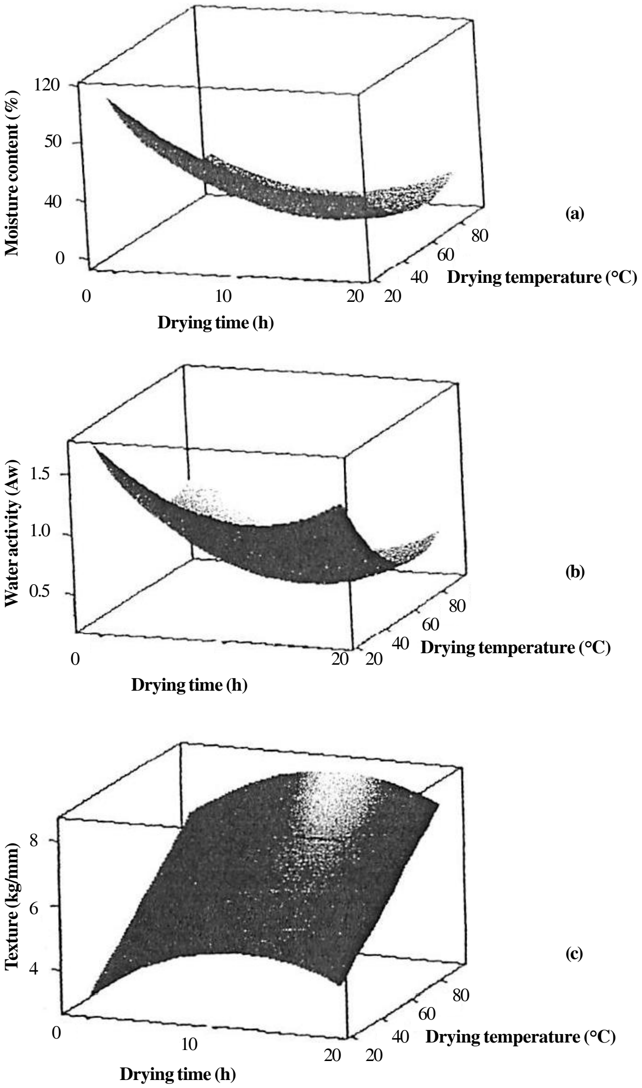

Figure 1. Effect of hot-air drying conditions (drying time and drying temperature) on the moisture content, water activity and texture of dried kaffir lime leaves.

Table 4. ANOVA and regression coefficients of the first- and second-order polynomial models.

	Main effects			Quadratic effects			Interaction effects		
Variables	X_1	X_2	X_3	X_1^2	X_2^2	X_3^2	X_1X_2	X_1X_3	X_2X_3
Moisture content									
P value	0.000*	0.000*	0.170†	0.000*	0.000*	0.116†	0.025*	0.107†	0.856†
F ratio	41.796	50.666	2.187	26.389	29.041	2.969	6.933	3.136	0.035
Water activity									
P value	0.000*	0.000*	0.856†	0.000*	0.000*	0.863†	-	-	-
F ratio	31.203	35.094	0.035	22.052	22.620	0.031	-	-	-
Texture									
P value	0.003*	0.004*	0.497†	0.008*	0.043*	0.687†	-	-	-
F ratio	13.162	12.222	0.489	9.986	5.054	0.170	-	-	-
Colour L									
P value	0.386†	0.920†	0.742†	0.294†	0.875†	0.851†	0.884†	0.527†	0.391†
F ratio	0.823	0.011	0.114	1.228	0.026	0.037	0.023	0.430	0.805
Colour a									
P value	0.030*	0.001*	0.100*	0.657†	0.007*	0.008*	0.015*	0.806†	0.939†
F ratio	6.396	19.528	3.291	0.209	11.479	11.036	8.703	0.064	0.001
Colour b									
P value	0.008*	0.174†	0.023*	0.015*	0.252†	0.021*	-	-	-
F ratio	9.891	2.065	6.595	7.935	1.440	6.838	-	-	-

Values of P value and F ratio less than 0.050 indicate model terms are significant. Values of P value and F ratio greater than 0.100 indicate model terms are not significant. *Significant ($P<0.05$). †Not significant ($P>0.05$).

temperature as well as their quadratic effects. However, there was no interaction effect between the independent variables. The results of the regression model for the response variable was significant by the F-test at the 5% confidence level ($p<0.05$). Also, the R^2 value (0.892) for the response variable ensures a satisfactory fitness of the regression model to the experimental data.

A 3D response surface plot for the effect of drying conditions on water activity is shown in Fig. 1b. The water activity is shown to decrease with increase in drying time and temperature. The individual optimum region led to be a minimum water activity ($Y_2 = 0.57\pm0.18$) obtained at 4.9 h drying time, 60°C drying temperature and 1.4 kg/m² loading capacity. The illustration changes of a_w showed relationship with moisture content. Reducing the a_w and moisture content would extend the shelf life and inhibit microorganism. These results were in agreement with previous studies [41].

Texture: The results of this study indicated that texture (brittleness) (Y_3) of dried kaffir lime leaves was significantly ($p<0.05$) affected by both main effect of drying time, drying temperature and their quadratic effects, respectively (Table 4). A 3D surface plot displays the effect of drying time and drying temperature on the texture (Fig. 1c). The individual optimum region led to a maximum texture ($Y_3 = 6.54 \pm 0.65$) which was predicted to be obtained at the drying time of 4.9 h, shows drying temperature, 60°C and loading capacity, 1.4 kg/m², respectively. Fig. 1c shows an increase in texture with an increase in drying time and drying temperature. The higher texture values mean the samples are more brittle after undergoing different drying conditions as compared to the lower texture values (Table 2).

The texture of kaffir lime leaves that have been dried at low temperature (40°C) was softer than that of kaffir lime leaves that was dried at high temperature (80°C). Besides that, it took a longer time to fracture the dried samples because during drying, liquid diffuses to the surface of the kaffir lime leaves from the interior and carries solute with it. It can be observed that the samples that have been dried at higher temperature resulted in very dry skin compared to low temperature drying. These findings further support the idea of Lin *et al.* [42] who showed that as the moisture evaporates, solutes concentrate and precipitate, leaving a hard and dry skin.

Vegetable tissues undergo some degree of shrinkage during drying process. This statement is supported by Brennan *et al.* [43] who showed the fact that usually in the early stage of drying, at low rates, the amount of shrinkage bears a simple relationship to the amount of moisture removed. Towards the end of drying, shrinkage is slowly reduced so that the final size and shape of the material is fixed before drying is completed. Other researchers also reported that shrinkage of foodstuffs during drying may influence their drying rates due to the changes in drying surface area and the setting up of pressure gradients within the material [44].

Colour (L, a and b): To assess the colour of dried kaffir lime leaves, the value of *L* (lightness), -*a* (greenness) and *b* (yellowness) were investigated. Both the quadratic and the main terms showed significant ($p<0.05$) effect on colour-*b* and colour-*a* values while for colour-*L* value, neither the main effect nor the quadratic effect showed any significant ($p>0.05$) influence (Table 4). The results revealed that both main effect of drying time and drying temperature and their quadratic effects had no significant ($p>0.05$) influence on colour-*L*. Also, the interaction effect of the independent variables had no effect on colour-*L*. The individual region led to maximum values for colour-*L*, colour-*a* and colour-*b* ($Y_4 = 46.90 \pm 0.67$), ($Y_5 = -0.89 \pm 0.17$), ($Y_6 = 11.28 \pm 0.46$) predicted to be obtained at 4.9 h of drying time, 60°C drying temperature and 1.4 kg/m² loading capacity.

Preferred colours are those closest to the original colour of fresh kaffir lime leaves. The original colour of fresh kaffir lime leaves was *L* (40.32 ± 0.40), *a* (-2.43 ± 0.03), *b* (10.65 ± 0.29). However, the findings of the current study do not support the previous research. The results showed that drying caused a decrease in lightness (*L* value). Higher temperature in hot-air drying resulted in a darker colour (lower *L* value) of dried kaffir lime leaves. This result is in agreement with data reported by Therdthai and Zou [19] who demonstrated that the lightness of dried mint leaves was decreased after hot-air drying. This may be because of the discolouration during drying which may also be related to nonenzymatic browning [45] and longer time of drying. Moreover, the degree of colour change also depends on drying temperature, drying time and oxygen level [46].

Also, there were positive significant ($p<0.05$) effects of drying time and drying temperature on colour-*b* and colour-*a* of dried kaffir lime leaves (Table 4). Fig. 2 displays the effects of hot-air drying conditions (drying time, drying temperature and loading capacity) on colour-*L* of dried kaffir lime leaves. It can be seen that colour-L values tend to increase with increase in drying time, drying temperature and loading capacity.

On the other hand, the 3D surface plot of the effect of drying conditions on colour-a value showed that the drying time and drying temperature increased colour-*a* values (Fig. 3a-c). Moreover, increase in loading capacity initially led to an increase in colour-*a*. With futher increase in the loading capacity, the values of colour-*a* decreased significantly.

Furthermore, only drying time and loading capacity had a significant ($p<0.05$) effect on colour-*b* of dried kaffir lime leaves (Table 4) and to visualize this, a 3D response thus surface plot created for the effect of hot-air drying conditions on the colour-*b* of kaffir lime leaves is shown in Fig. 3d. It seems colour-*b* values have curvature effect where colour-*b* increased initially with increased drying time and loading capacity followed by gradual decrease. Previous researches reported that this value increased fast for air-dried samples, as drying goes beyond 70°C, yellowness (*b* value) of dehydrated materials is strongly affected by temperature and air relative humidity [47].

Optimization and validation procedures: Both numerical and multiple graphical optimizations were employed to determine the exact optimum point of different hot-air drying conditions on the physicochemical properties of dried kaffir lime leaves leading to desirable response goals. As mentioned by Mirhosseini *et al.*[24], for graphical optimization procedure, the reduced models were illustrated as three-dimensional (3D) response surface plot to better visualize the interaction effect of main drying conditions on the physicochemical properties of dried kaffir lime leaves. The optimum drying process performed at 4.9 h, 60°C and 1.4 kg/m² was proposed to provide dried kaffir lime leaves with optimum quality. The

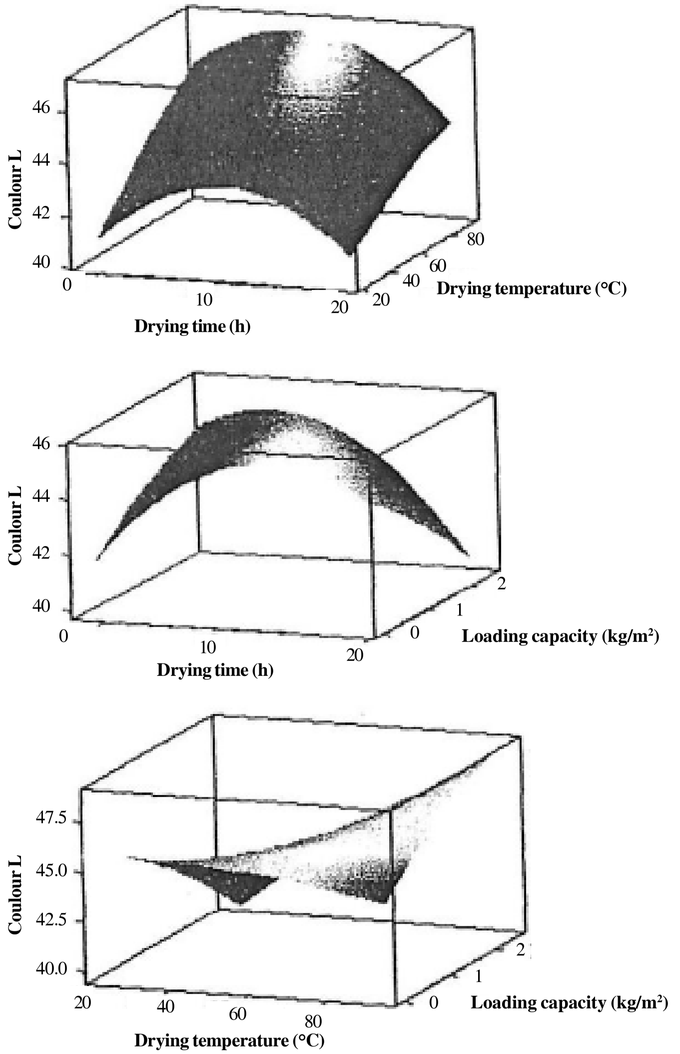

Figure 2. Effect of hot-air drying conditions (drying time, drying temperature and loading capacity) on the colour-*L* of dried kaffir lime leaves,

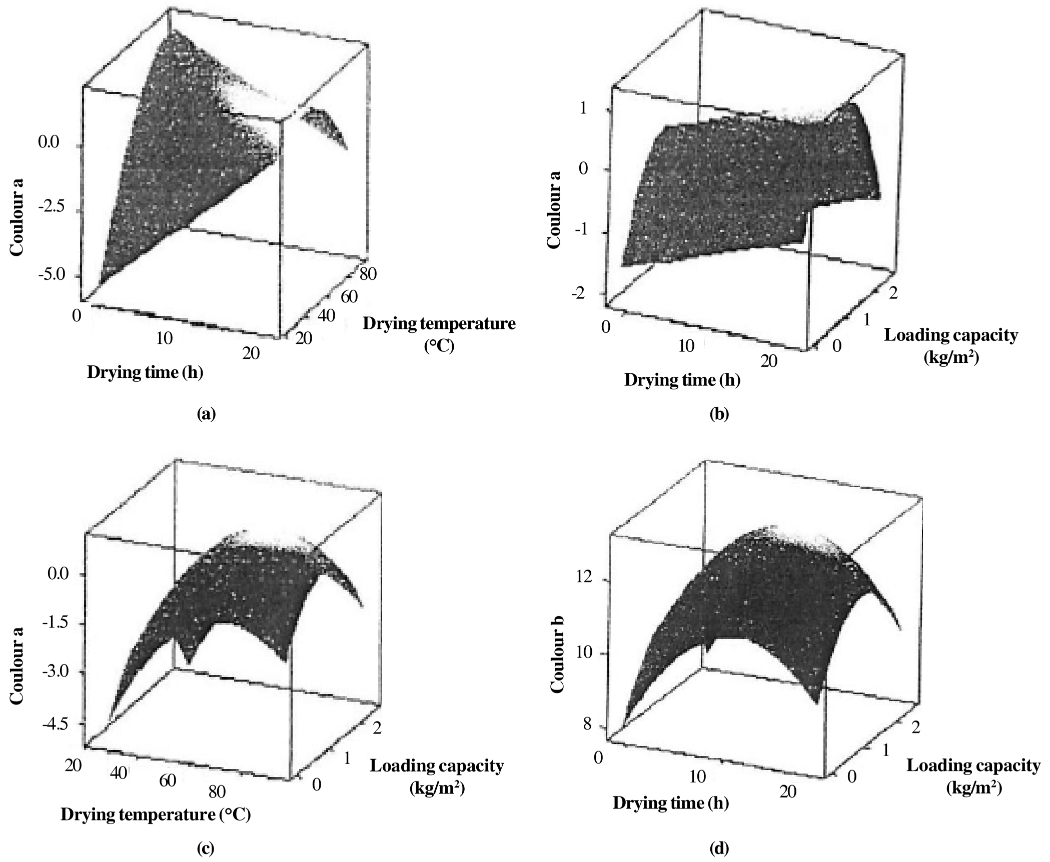

Figure 3. Effect of hot-air drying conditions (drying time, drying temperature and loading capacity) on the colour-*a* and colour-*b* of dried kaffir lime leaves.

Table 5. Experimental and predicted values for the response variables studied.

	Moisture content			Water activity			Texture			Colour L			Colour a			Colour b		
Run	Y_o	Y_1	Y_o-Y_1	Y_o	Y_1	Y_o-Y_1	Y_o	Y_1	Y_o-Y_1	Y_o	Y_1	Y_o-Y_1	Y_o	Y_1	Y_o-Y_1	Y_o	Y_1	Y_o-Y_1
1	5.67	2.52	3.15	0.25	0.31	-0.06	6.82	7.37	-0.55	47.31	44.98	2.33	-0.8	-0.61	-0.19	13.31	11.95	1.359
2	46.14	53.86	-7.74	0.97	0.97	0.00	4.99	4.60	0.39	44.66	42.83	1.83	-2.09	-2.77	0.68	11.20	11.09	0.11
3	7.12	10.37	-3.25	0.58	0.53	0.05	7.05	6.8	0.25	49.14	44.37	4.77	0.65	0.70	-0.05	12.15	10.96	1.19
4	5.57	7.83	-2.26	0.37	0.36	0.01	6.65	6.74	-0.09	46.38	45.36	1.02	0.61	0.70	-0.09	13.61	12.92	0.70
5	4.14	1.98	2.16	0.40	0.39	0.01	7.69	7.71	-0.02	43.35	46.82	-3.47	0.63	-0.05	0.68	11.00	12.92	-1.92
6	5.79	7.83	-2.04	0.36	0.36	0.00	6.90	6.74	0.16	44.43	45.36	-0.93	1.00	0.70	0.30	12.85	12.92	-0.07
7	14.27	7.83	6.45	0.38	0.36	0.02	7.04	6.74	0.30	44.62	44.60	0.02	-1.85	-1.49	-0.36	10.62	11.04	-0.42
8	51.70	44.11	7.59	0.98	0.93	0.05	4.32	5.01	-0.69	40.54	43.41	-2.87	-0.55	0.17	-0.72	8.72	10.05	-1.33
9	4.69	13.25	-8.56	0.39	0.57	-0.18	7.11	5.97	1.14	43.88	42.60	1.28	0.60	1.22	-0.62	11.80	11.49	0.31
10	13.44	7.83	5.62	0.24	0.36	-0.12	6.59	6.74	-0.15	43.69	45.30	-1.61	-1.58	-1.09	-0.49	10.22	10.82	-0.60
11	4.62	2.52	2.10	0.46	0.31	0.15	6.85	7.37	-0.52	44.20	44.91	-0.71	-0.56	-0.38	-0.18	13.03	11.82	1.21
12	36.77	25.03	11.74	0.96	0.76	0.21	4.74	5.17	-0.43	37.68	41.32	-3.64	0.39	-0.44	0.83	10.22	11.95	-1.73
13	5.29	10.37	-5.08	0.44	0.53	-0.09	7.40	6.80	0.60	47.44	47.09	0.36	0.22	0.46	-0.24	12.66	11.09	1.57
14	5.57	7.83	-2.26	0.36	0.31	0.15	6.65	6.74	-0.09	46.38	45.36	1.02	0.61	0.70	-0.09	13.61	12.915	0.695
15	23.00	25.03	-2.03	0.85	0.76	0.21	4.50	5.17	-0.67	45.64	44.87	0.77	0.44	-0.27	0.65	11.04	11.82	-0.78
16	5.57	7.83	-2.26	0.37	0.36	0.00	6.65	6.74	-0.09	46.38	45.36	1.02	0.61	0.70	-0.09	13.61	12.92	0.70
17	5.79	7.83	-2.04	0.36	0.360	0.00	6.90	6.74	0.16	44.43	45.36	-0.93	1.00	0.70	0.30	12.85	12.92	-0.70
18	57.73	53.88	3.85	0.99	0.97	0.02	4.25	4.60	-0.35	42.52	43.73	-1.21	-1.65	-2.53	0.88	10.92	10.96	-0.04
19	54.37	57.50	-3.13	0.98	1.13	-0.15	4.47	4.00	0.47	45.09	43.21	1.88	-4.14	-2.62	-1.52	12.09	12.92	-0.83
20	5.79	7.83	-2.04	0.36	0.36	0.00	6.90	6.74	0.16	44.43	45.36	-0.93	1.00	0.70	0.30	12.85	12.92	-0.07

(Yo) Experimental value, (Y1) predicted value, (YoY1) residue. a No significant (P > 0.05) difference between experimental and predicted value.

expected response values for moisture content, water activity, texture (brittleness), colour-*L*, colour-*a* and colour-*b* were 10.70, 0.57, 6.54, 46.90, -0.89 and 11.28, respectively.

The adequacy of the response surface equations were checked by the comparison of the experimental and fitted values predicted by the response regression models. The experimental and predicted values are given in Table 5. No significant (p>0.05) difference was found between the values. The experimental response values were shown to be in agreement with the predicted ones. Closeness between the experiment and predicted values confirmed the adequacy of the corresponding response surface models employed for describing the differences obtained in the dried kaffir lime leaves as a function of drying conditions.

Conclusions

The present study was designed to optimize the optimum different drying conditions leading to desirable quality of physicochemical characteristic of dried kaffir lime leaves using RSM. The response surface analysis showed significant (p<0.05) relationship between the independent variables and the response variables, with R^2 values, ranging from 0.315 to 0.928. The optimization results indicated that the best response, within the range studied was reached when the drying time was 4.9 h, the drying temperature, 60°C and loading capacity, 1.4 kg/m^2, respectively. Under the optimum condition, the corresponding predicted response values for moisture content, water activity, texture (brittleness), colour-*L*, colour-*a* and colour-*b* were found to be 10.70, 0.57, 6.54, 46.90, -0.89 and 11.28, respectively.

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