

# Kinetics, Optimization and Proximate Analysis of Drying Moringa oleifera Seeds in a Tray Dryer

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## **Abstract**

This study examined the drying behaviour, optimum drying conditions and proximate analysis of *Moringa oleifera* seeds in a tray dryer. Thirteen (13) experimental data sets were generated using Box-Behnken design of design expert at varying temperature, contact time and fan speed on response surface methodology. Data generated from the experiment were fitted into existing models to determine the best fit using Excel Solver. Experimental data was used to simulate mathematical model developed using Box-Behnken design with the objective of minimizing the moisture content of the moringa seed within the lower and upper bound of 25-50°C (drying temperature), 30-100 mins (contact time) and 0.5-2.5 m/secs (fan speed). It was shown that the moisture ratio decreases with increasing drying time in all the plots with equilibrium moisture content being achieved within the range of 60-90 minutes at different operating conditions. However, the experiments with temperature (50°C) and fan speed (2.5 m/s) exhibited lowest moisture ratio with varying time. Also, the drying rate decreased continuously throughout the drying period in all the graphs with no constant rate period at any of the operating conditions. Wang and Singh model best described the drying behaviour of the *Moringa oleifera* seeds in a tray dryer with R<sup>2</sup> and χ<sup>2</sup> values of 0.9991 and 0.00017 respectively at drying temperature of 50°C, fan speed of 0.5 m/s and contact time of 65 min. The moringa seeds that were dried at temperature 37.5°C, contact time 65 mins and fan speed of 1.5 m/s have moisture content of 17.71% with highest crude carbohydrate of 75.49%. A minimum moisture content of 16.7390% was obtained at drying temperature of 25°C, contact time of 75 mins and fan speed of 2.5 m/s using the developed optimization model that gives an excellent prediction with  $R^2$  value of 0.9918.

**Keywords:** Optimization; Kinetics; Drying; Proximate analysis; Tray dryer; *Moringa oleifera*

## **Introduction**

Drying is a [mass transfer](http://en.wikipedia.org/wiki/Mass_transfer) process consisting of the removal of water or another [solvent](http://en.wikipedia.org/wiki/Solvent) by [evaporation](http://en.wikipedia.org/wiki/Evaporation) from a [solid](http://en.wikipedia.org/wiki/Solid), [semi-solid](http://en.wikipedia.org/wiki/Semi-solid) or [liquid](http://en.wikipedia.org/wiki/Liquid) and the oldest known common unit operation in chemical engineering [1,2]. Its process can either be natural (made by the sun or wind) or artificial (which needs power supply) and is usually conducted by vaporizing water in the product and supplying latent heat of vaporization. Factors that affect drying rate are vapour pressure, temperature, relative humidity, air circulation, nature of wet feed and the way it is introduced into the dryer and a host of other environmental factors [3,4]. Common domestic and industrial dryers classified according to design and operating features are tray dryers, tunnel dryers, drum dryers, fluidized bed dryers, spray dryers, flash dryers, rotary dryers, belt dryers, vacuum dryers and freeze dryers [5]. The most common type for drying of leaves and seeds is the tray dryer which can be classified based on mode of operation as batch or continuous; and position and movement of trays as stationary or moving [6].

*Moringa oleifera*, commonly called a miracle tree due to its unique features and multipurpose ability, belongs to a monogenetic family called the *Moringaceae* [7-9]. It is a short, slender, deciduous and perennial tree with cosmopolitan tropical drought tolerance characteristics [10]. It has numerous benefits ranging from medicinal, nutritional domestic to industrial [8,11,12]. Thus, efforts need to be made to make the seeds be well preserved while still retaining the nutrients contained in them for future purpose. Recently, Ali et al. [13] investigated drying kinetics and colour analysis of *Moringa oleifera*  leaves and was shown that oven drying at 40°C reveled optimum colour values. Aremu et al. [14] examined the effects of some drying methods on nutritional characteristics of moringa (*Moringa oleifera*)

seeds and was shown that more nutrients were retained in cabinettray drying method (in terms of protein and fat) compared to other drying methods. It has also been shown that commercial processing of *M. oleifera* leaves could be improved by using microwave drying method with higher total phenolic content, radical scavenging activity and quercetin as the drying time was reducing [15]. Also, Verma model had been shown to be the best for describing the drying kinetics of *Moringa oleifera* leaves [16]. Tray drying method had been proven to be the best method of dehydrating moringa leaves due to its ability to retain moringa leaves nutritional content as compared to sun, shadow and cabinet drying methods [17].

In this study, the optimum conditions for drying parameters of *Moringa oleifera* seed in a tray dryer were determined using Box-Behnken design for drying factors (temperature, contact time and fan speed) on response surface methodology. Thirteen (13) experimental data sets were generated for samples of moringa seed using design expert. Proximate analysis was done for the samples after drying to determine their crude fat, crude protein, carbohydrate, crude fibre and ash content. Data generated from the experiment were fitted into existing models to determine the best fit using Excel Solver. An empirical model, developed using Box-Behnken design, was simulated

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using the experimental data and was then optimized by minimizing the moisture content of the moringa seed. The drying parameters were optimized within the lower and upper bound of 25-50°C (drying temperature), 30-100 mins (contact time) and 0.5-2.5 m/secs (fan speed).

## **Materials and Methods**

## **Sample collection and preparation**

Fresh and matured moringa pods were harvested from *Moringa oleifera* farm of Afe Babalola University, Ado-Ekiti, Ekiti State, Nigeria located on longitudes 5°18'05.78''E and latitudes 7°36'09.59''N. The seeds, removed from the pods and the outer shell, were then cut into smaller particle sizes of diameter  $\leq$  5 mm.

#### **Experimental design and optimization**

Design expert was used to reduce the number of experiments to be conducted. Box Behnken design was chosen with three factors of temperature (T, °C), contact time ( $t_c$ , mins) and speed of fan ( $v_p$ m/s). Moisture content (%) is the output from the response surface methodology used. The optimization tool of the design expert together with the developed model of the system was utilized to obtain the optimum parameters required for appropriate drying of the moringa seed samples. The objective function of the optimization was to minimize the moisture content contained in the dried moringa seeds within the set range of the lower and upper bounds of manipulated variables.

## **Drying of seeds**

The *Moringa oleifera* seeds (≤ 5 mm) were dried using a computer controlled Armfield tray dryer (Armfield UOP8 MKII). The tray dryer, having dimension of 1.4 m  $\times$  2.95 m  $\times$  0.75 m, is made up of three (3) trays with an area 254 mm  $\times$  304.8 mm for each. For each of the experiment, 150 g was weighed and separated into three portions on an average of 50 g in each tray to increase surface area for drying process to occur. After each experiment, total weight of sample against time was taken.

#### **Proximate analysis**

After using the experimental design approach, thirteen (13) samples were examined for proximate analysis to determine percentages of crude fat, crude protein, crude carbohydrate, crude fibre, ash and residual moisture content.

**Moisture content:** This was carried out using a moisture analyser. Two grams of the sample was placed in the equipment at 105°C and readings were taken after 10 minutes. The moisture ratio is calculated thus:

% Ash Content = 
$$
\frac{W_{\alpha} - W_c}{W_s} \times 100\%
$$
 (1)

where  $M_t$ =Moisture content at time t, g

 $M$ <sub>e</sub>=Equilibrium moisture content, g

 $M_{_o}$ =Initial moisture content, g

**Ash content:** An analytical balance was used to weigh a dried crucible dish and 2 g of the sample was placed. The dish plus the sample was placed in a muffle furnace at 600°C for 3 hours. After 3 hours, the sample plus crucible dish was removed from the muffle furnace and placed immediately in a desiccator. It was then weighed. The ash

content was calculated using:

% Ash Content = 
$$
\frac{W_{cs} - W_c}{W_s} \times 100\%
$$
 (1)  
where  $W_{cs}$ =Weight of dried crucible plus sample, g

*W*<sub>c</sub>=Weight of dried empty crucible, g

*W*<sub>s</sub>=Weight of sample, g

**Crude protein:** The amount of crude protein in the sample was determined using Kjeldahl method which involves conversion of nitrogenous compounds to ammonium sulphate by digesting the sample with concentrated  $H_2SO_4$  in the presence of selenium. Two grams of the sample was put into the digestion flask and half of selenium based catalyst tablet and 25 ml of concentrated  $\rm{H_2SO_4}$  were added. The flask was thoroughly shaken to ensure uniformity and then placed on a heating mantle until the resulting solution was clear after which it was cooled to room temperature of 25°C. The digested solution plus distilled water was filled up in a 100-ml volumetric flask. Two drops of mixed indicator were added into 25 ml of 2% boric acid, pipetted into a 250-ml conical flask and the contents were placed under a condenser such that the tip of the condenser was completely immersed in the solution. 10 ml of the digested sample was measured and poured into the decomposition flask with the addition of an excess 40% (about 15-20 ml). To drive the liberated ammonia into the collection flask (a conical flask containing 25 ml of 2% boric acid), steam was forced through the decomposition chamber by shutting the stop cock on the steam trap outlet. The boric acid changed to bluish green as soon as it came into contact with ammonia and distillation was continued for 5 minutes. The receiving flask was lowered so that the condenser tip was just above the liquid which was washed with little distilled water and distillation continued for another 30 seconds. Twenty (20 ml) of the distilled sample was pipetted into a 250-ml conical flask and titrated with 0.0501N HCl solution. The acid was added until the solution was pink. The endpoint of the titration was identified as the first colour change.

Assuming a correction factor of 6.25, the crude protein is calculated using [18,19]:

*% Crude Protein=6.25 × %Nitrogen* (2)

$$
\%Nitrogen = \frac{(V_s - V_b) \times C_A \times 0.014 \times D_{sd}}{W_s \times V_d} \times 100\tag{3}
$$

*s d* where  $V_{s}$ =Titration reading of sample, ml

 $V_b$ =Blank titration reading, ml

 $C_A$ =Concentration of acid=0.0501 mol/dm<sup>3</sup>

*D<sub>s</sub>*=Sample dilution after digestion, ml

 $W_s$ =Weight of sample, g

 $V_d$ =Volume taken for distillation, ml

0.014=Milli equivalent weight of Nitrogen

**Crude fibre:** Two grams of sample was weighed into a round bottom flask with 50 ml of fibre solution measured and boiled for 1 hour. Filter paper was used to decant using vacuum pump to suck the liquid and wash with distil water till it was clear. The residue was scraped into crucible and oven dried for 24 hrs and weighed, thereafter it was ash in a furnace at 600°C for 2 hours and allow to cool down. It was then weighed. Crude fibre was calculated using:

% Crude Fibre = 
$$
\frac{W_{\text{cos}} - W_{\text{c}}}{W_{\text{cs}} - W_{\text{c}}} \times 100
$$
 (4)

where  $W_c$ =Weight of empty crucible, g

*W<sub>c</sub>*=Weight of empty crucible and sample, g

*W<sub>ca</sub>*=Weight of empty crucible and ashed sample, g

**Crude fat:** Two grams of sample was transferred onto a thimble and small ball of cotton wool was placed into it to prevent sample loss. A measured volume of 150 ml petroleum spirit was added into a dried 250 ml flask. A quickfit condenser was connected to the soxhlet extractor and refluxed for 4 hours on heating mantle. The flask was then removed and evaporated on a steam bath. The flask and fat sample was heated for 30 minutes in an oven at a temperature of 103°C. The flask and content was cooled to room temperature of 25°C in a desiccator. They were then weighed accurately to determine weight of fat collected. Weight of fat collected was determined by using:

% Crude Fat = 
$$
\frac{W_{ts} - W_{dst}}{W_{dst} - W_t} \times 100
$$
 (5)

where  $W_{\kappa}$ =Weight of thimble and sample, g

*W<sub>de</sub>*=Weight of defatted sample and thimble after drying, g

 $W_t$ =Weight of thimble, g

**Crude carbohydrate:** The percent of crude carbohydrate in sample is determined using:

*% Crude Carbohydrate*=100%−[*%Moisture Content+%Ash Content+%Crude Protein+%Crude Fibre+%Crude Fat*] (6)

#### **Mathematical modelling for data fitness**

The experimental data obtained were fitted into different models presented in Table 1 to determine the one that best describes the experimental process. The data fitness was performed using excel solver for the determination of constants and coefficient in the models together with the regression analysis.

The reduced chi-square is determined thus:

$$
\chi^2 = \frac{\sum_{i=1}^{N} \left( MR_{\text{exp},i} - MR_{\text{pred},i} \right)^2}{N - n}
$$
\n
$$
(7)
$$

where *MR*<sub>exp,i</sub>=experimental moisture ratio for experiment I, g

*MR*<sub>pred</sub><sub>i</sub>=predicted moisture ratio for experiment I, g

*N*=total number of observations

*n*=total number of constants in the examined model

The room mean square error (RMSE) is determined using:

$$
RMSE = \left[\frac{1}{N} \sum_{i=1}^{N} \left(MR_{\text{exp},i} - MR_{\text{pred},i}\right)^{2}\right]^{\frac{1}{2}}
$$
(8)

The drying rate is calculated using:

$$
DR = \frac{M_{t+dt} - M_t}{dt} \tag{9}
$$

where  $M_{t+dt}$ =moisture content at time t+dt, g

 $M_t$ =moisture content at time t, g

t=drying time, mins.

## **Results and Discussion**

## **Design of experiment**

The result obtained for the experimental design of drying of *Moringa oleifera* seeds in a tray dryer using Box Behnken procedure is

presented in Table 2. The parameters used to determine the optimum drying conditions were all within the set ranges (contact time: 30-120 mins, temperature: 25-50°C and fan speed: 0.5-1.5 m/sec). All the experimental runs vary at different operating conditions. However, it was observed that same drying conditions were generated for the third, fifth and thirteenth experimental runs. This results from random sampling of the procedure used. The values for the raw sample were set at zero because it was not subjected to drying for comparative proximate analysis purpose with dried samples.

#### **Drying behaviour of** *Moringa oleifera* **seed in tray dryer**

The moisture ratio and drying rate were calculated using equations 1 and 9 respectively. Based on the result obtained from the design of experiment, the contact time are in three categories of 30, 65 and 100 minutes, thus the results obtained for the moisture ratio and drying rates were plotted on this basis. Figures 1-3 show graphs of variation of moisture ratio with varying drying temperatures and fan speed at contact time of 30, 65 and 100 minutes respectively. The moisture ratio decreases with increasing drying time in all the plots. This indicates that the drying operating variables are effective parameters to reduce the moisture content of the *Moringa oleifera* seeds. The continuous decrease in the moisture ratio resulted from the removal of free moisture from the surface [2] and also moisture from the interior of the seeds as a result of the internal mass transfer governed by diffusion [16]. In Figures 1 and 2, the equilibrium moisture content was achieved at 30 and 60 minutes respectively for the plots at different operating conditions. However, in Figure 3, the equilibrium moisture content was achieved within the range of 60-90 minutes for the plots at different operating conditions of drying temperature and fan speed of the dryer. The equilibrium moisture content (18-39%) was significantly lower than the initial moisture content (45-47%) as a result of varying relative

Model name	Model	<b>References</b>
I ewis	$MR=exp(-kt)$	[20]
Page	$MR=exp(-ktn)$	$[21]$
Wang and Singh	$MR=1+at+bt^2$	[22]
Logarithmic	$MR=exp(-kt)+c$	$[23]$
Two-Term exponential	$MR=a \exp(-kt)+(1-a) \exp(-kat)$	$[24]$
Verma et al.	$MR=a$ exp(-kt)+(1-a) exp(-qt)	[25]
Modified page	$MR=exp(-kt)^y$	[26]
<b>Henderson and Pabis</b>	$MR=a$ exp(-kt)	[27]
Magee	$MR = a + kt^{1/2}$	$[28]$

**Table 1:** Mathematical Models for Data Fitting.

<b>Run</b>	Temperature (°C)	<b>Contact Time (mins)</b>	Fan Speed (m/secs)
1	50	65	0.5
$\overline{2}$	37.5	100	0.5
3	37.5	65	1.5
4	25	65	0.5
5	37.5	65	1.5
6	25	30	1.5
7	37.5	30	0.5
8	50	30	1.5
9	50	100	1.5
10	25	100	1.5
11	37.5	100	2.5
12	50	65	2.5
13	37.5	65	1.5
Raw	0	0	0

**Table 2:** Design of Experiment.

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humidity during the drying process in Figures 1-3 [29]. Also, the experiments with temperature (50°C) and fan speed (2.5 m/s) exhibit lowest moisture ratio with varying time which indicates the impact of temperature and fan speed of the dryer as key process variables to the drying of the *Moringa oleifera* seed.

The drying rate as a function of time was also calculated in all the experiments at various operating conditions using equation 9. Figures 4-6 are the drying rate curves at varying temperature and fan speed for contact time of 30, 65 and 100 minutes respectively. In all the graphs, there was initial increase in the drying rate with time which indicates removal of more free moisture at the surface of the seeds and internal diffusion mass transfer occurring in the *Moringa oleifera* seed resulting high heat supply. The drying rate decreased continuously throughout the drying period in all the graphs with no constant rate period at any of the operating conditions. The drying behaviour conforms with examined previous studies on drying behaviour of *Moringa oleifera* leaves [13,16]. Many previous studies on drying of food materials have also reported to have same drying characteristics [30]. The nature of the plots depicted in Figures 4-6 also indicate that the drying parameters (contact time, drying temperature and fan speed) are effective enough to enhance drying in the seed.

### **Mathematical modelling for kinetic analysis**

The results obtained for the moisture ratio in each experiment were fitted into various models presented in Table 1. Equations 7 and 8 were used to calculate the chi-square  $(\chi^2)$  and root mean square error (RMSE). The values for the predicted moisture ratio in the models with their respective constants and coefficients were calculated using excel solver. The coefficient of determination  $(R^2)$  between the experiment and predicted values of moisture ratio was done by regression analysis

on the data analysis of excel solver. It was expected that the model with highest R2 and lowest chi-square best describes the drying of *Moringa oleifera* seeds at such drying condition. Appendix I is the detailed results obtained for the constants and coefficients in the models;  $\mathbb{R}^2$ ,  $\chi^2$ and root mean square error (RMSE) at different operating conditions. Table 3 (extracted from Appendix I) is a summary of the maximum and minimum values of  $\mathbb{R}^2$  and  $\chi^2$  at operating conditions at which they were obtained for the models.

From the results obtained, the  $R^2$  values for the fitted mathematical models were greater than 0.92 except with logarithmic and two-term exponential models that have minimum  $R<sup>2</sup>$  values of 0.7544 and 0.7537 respectively. The highest value of 0.9991 was obtained for  $\mathbb{R}^2$ with minimum  $\chi^2$  value of 0.00017 at drying temperature of 50°C, fan speed of 0.5 m/s and contact time of 65 min using Wang and Singh model. This is an indication that the Wang and Singh model best describes the drying behaviour of moringa oleifera seeds at the obtained operating conditions. However, lewis page, modified page, Henderson and Pabis, and Magee models have maximum  $R^2$  values of ˃0.9881 at drying temperature of 37.5°C, fan speed of 2.5 m/s and contact time of 100 min. This indicates that the operating conditions at which these models have maximum  $R<sup>2</sup>$  values are also favourable for optimum drying of *Moringa oleifera* seeds in a tray dryer. Previous literatures had recommended Page's model to be the most suitable mathematical drying method among all mathematical models [31,32]. In other studies, Verma model was considered to represent the drying behaviour of *Moringa oleifera* leaves in a convective type dryer [16] with similar results reported for air drying of bay leaves [33].

## **Proximate analysis**

The proximate analysis of each sample of *Moringa oleifera* seed



**Table 3:** Coefficient of determination  $(R^2)$  and  $\chi^2$  at different Operating Conditions.

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(Table 4) was executed to check their nutritional contents after drying in the tray dryer. All the examined seeds contained measurable amount of crude fat, crude ash, crude protein, crude fibre, crude carbohydrate and moisture content as also reported in previous literatures [19,34-36]. The moringa seeds dried at temperature 25°C, contact time 65 mins and fan speed of 0.5 m/s has the highest moisture content of 38.49% with lowest crude carbohydrate of 53.43%. while the moringa seeds that were dried at temperature 37.5°C, contact time 65 mins and fan speed of 1.5 m/s has moisture content of 17.71% with highest crude carbohydrate of 75.49%. The variance in the values of parameters evaluated resulted from the operating conditions at which the moringa seeds were dried. The drying operating conditions of temperature 37.5°C, contact time 65 mins and fan speed of 1.5 m/s enhanced reduced moisture content and high crude carbohydrate. In all the experiments, crude carbohydrate has the largest percentage of the moringa seeds which is in contrary to many studies where crude protein takes the largest percentage of the moringa seeds [37].

# **Optimization of drying parameters**

The empirical model developed using Box-Behnken design to minimize the moisture content of the moringa seed within the lower and upper bounds of 25-50°C (drying temperature), 30-100 mins (contact time) and 0.5-2.5 m/secs (fan speed) is given as equation 10. A minimum moisture content of 16.7390% was obtained at drying temperature of 25°C, contact time of 75 mins and fan speed of 2.5 m/s using MATLAB optimization toolbox for the generated model. Table 5 shows the values of experimental and simulated moisture contents at different experimental runs using the developed model. The regression analysis performed shows an excellent prediction with  $R<sup>2</sup>$  value of 0.9918.

 $MC = 85.88 - (0.893 \times T) - (0.557 \times t_c) - (17.72 \times v_f) + (0.00305 \times T^2) + (0.004736 \times t_c^2) + (1.076 v_f^2)$  $-(0.00295 \times Tx_t) + (0.3173 \times Tx_y) - (0.0395 \times t \times y_f)$ <sup>−</sup>(0.00295 × T x t<sub>c</sub><sup>}</sup>) + (0.03173 × T x t<sub>c</sub><sup>}</sup>) + (0.03173 × T x t<sub>c</sub><sup>}</sup>) + (0.00295 × T x t





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 **Table 4:** Proximate Analysis of Dried *Moringa oleifera* Seeds.



 **Table 5:** Simulated Moisture Content using the Developed Model.



## **Conclusion**

The drying behaviour, optimum drying conditions and proximate analysis of *Moringa oleifera* seeds in a tray dryer have been examined. The moisture ratio decreases as the drying time progresses in all the plots while the equilibrium moisture content was achieved within the range of 60-90 minutes at different operating conditions. The drying rate decreased continuously throughout the drying period in all the graphs with no constant rate period while Wang and Singh model best described the drying behaviour of the *Moringa oleifera* seeds in a tray dryer with  $R^2$  and  $\chi^2$  values of 0.9991 and 0.00017 respectively at drying temperature of 50°C, fan speed of 0.5 m/s and contact time of 65 min. The moringa seeds that were dried at temperature 37.5°C, contact time 65 mins and fan speed of 1.5 m/s have moisture content of 17.71% with highest crude carbohydrate of 75.49%. A minimum moisture content of 16.7390% was obtained at drying temperature of 25°C, contact time of 75 mins and fan speed of 2.5 m/s using the developed model. shows using the developed model. The regression analysis performed between the values of experimental and simulated moisture contents at different experimental runs shows an excellent prediction with  $R^2$ value of 0.9918.

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