Drying Kinetics and Thin Layer Modelling of *Clerodendrum Volubile* (Marugbo) Leaves

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***Abstract*—The drying kinetics of *Clerodendrum Volubile* leaves was investigated at different temperature of 50, 60 and 70ºC in oven dryer. The weight loss with time was recorded and moisture ratio was computed and fitted into different eleven thin-layer drying models. The result showed that moisture ratio reduces with time for all drying temperatures. The drying rate was observed to increase with temperature peaking at 0.185, 0.117 and 0.059 g H2O/g dry solid.min at 70, 60 and 50ºC respectively. Drying occurred in falling rate period and no constant rate period was observed. The approximation of diffusion model was observed to give the best fit model for the drying process with highest coefficient of determination (0.9985), lowest sum of square errors (0.0032), reduced chi square (0.00012) and root mean square error (0.0107) occurring at 50 ºC. The effective diffusivity for Marugbo drying increases with temperature from 3.65×10^{-12} to 1.28×10^{-11} m2/s. The Arrhenius equation also described the temperature dependence of diffusivity with activation energy of 57.74 kJ/mol.**

***Index Terms*—*Clerodendrum Volubile*, drying kinetics, moisture ratio, thin-layer models.**

I. Introduction

Clerodendrum volubile, locally known in South Western Nigeria as Marugbo or Eweta is a climbing green shrub that belongs to family Lamiaceae (Verbenaceae) [1]. Its leaves are used in traditional African medicine because it possesses antimicrobial, antioxidant, antinociceptive and anti-inflammatory properties [2]. It also has potential in cancer treatment because its extracts have the ability to scavenge reactive oxygen species [3], [4]. *Marugbo* leaves are also commonly consumed as soup medicine for sedation and managing arthritis, rheumatism, and any form of swellings [5]. Its flavonoids aid renal arsenic detoxification [6], analgesic activities [7] cardio-protective properties and anti-diabetic properties [8], [9]. Marugbo soup, commonly eaten among *Ilaje*s in Ondo state, Nigeria is locally prepared by blending with *efinrin* (African Basil) leaves, pepper and cooked with tilapia fish or meats and served with *pupuru* (smoked cassava flour) or pounded yam. The leaves can be used fresh or dried; they are typically used after being completely sun dried, which gives the soup a very dark green to black colouring. Marugbo leaves are low in fat (6 %), high in carbohydrate (44.69 %), protein (13.6 %) and fibre (11.66 %).It contains dietary minerals such as calcium (30.19 mg/100g), potassium (27.69 mg/100g), magnesium (27.11 mg/100g), phosphorus (27.61 mg/100g) with no lead and high essential amino acids such as leucine and arginine which is good for infants [1].

Drying involves removal of moisture from food products up to a level that prevents microbial growth thereby enhancing storage, shelf life, packaging and transportation [10]. Previous studies on *Marugbo* leaves has been limited to its medicinal potentials [2], [3], [5]–[9], [11] and nutritional values [1], [12]. Quantitative understanding of the fundamental drying mechanisms and thin layer equations is crucial for understanding of the heat and mass transfer equations phenomena, process design, quality control, product handling and energy savings [13]. The main objectives of this study are (i) to evaluate effect of drying temperature on drying characteristics Marugbo leaves (ii) to determine drying model that best fit the drying process (iii) to compute effective moisture diffusivity and activation energy of samples. Data generated from the study would be very useful for equipment design and process development.

II. Methodology

A. Sample Collection

Fresh *Clerodendrum volubile* leaves were collected from swampy area in University of Lagos where it grows as wild plant. No permission is required for collection of the plant as it is not listed as endangered species [14]-[16] and it is usually grown by the locals and sold in the market. The collected leaves were rinsed to remove dirt, soils and impurities on the leaves. The wet leaves were shaken in air and spread out to drain out excess water. Samples were identified and authenticated by Dr Nodza George at University of Lagos Herbarium, Lagos, Nigeria with voucher number LUH9495.

B. Drying of Marugbo Leaves

The moisture content was determined by drying at 135ºC for 2hrs according to AOAC procedure [17]. The initial moisture content was 78% w. b. and drying experiments were carried out at 50, 60 and 70ºC using an oven dryer. The oven dryer was preconditioned for about 40 minutes before loading the sample [13]. Samples of 10 g were distributed on the tray before drying in the oven. The weight was measured after every 10 minutes with a ± 0.01g precision digital weighing scale, up to a constant weight in three consecutive readings which was taken as the equilibrium moisture content. Each experiment was performed three times and the average was used in the drying analysis.

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C. Mathematical Modelling

The moisture content, M on dry basis was determined from the measured weight loss using Equation (1)

$$M = \frac{m_t - m_b}{m_b} \quad (1)$$

m_t is the mass at time t and m_b is the bone-dry solid.

The amount of moisture remaining at any time to the initial moisture content on dry basis is represented by the moisture ratio *(MR)* which was calculated from Equation (2)

$$MR = \frac{M_t - M_e}{M_o - M_e} \quad (2)$$

The rate of drying was calculated from Equation (3) [18]

$$DR = \frac{M_{t+dt} - M_t}{d_t} \quad (3)$$

Eleven existing thin layer models in Table I were fitted into experimental data to determine the best model for the drying kinetics through nonlinear regression analysis by the General reduced Gradient (GRG) Nonlinear Method using Excel solver.

TABLE I: STANDARD THIN LAYER DRYING MODELS

Model Name	Model
Lewis [19]	$MR = \text{Exp}(-kt)$
Wang and Singh [20]	$MR = at^2 + bt + 1$
Page [21]	$MR = \text{Exp}(-kt^n)$
Modified Page [22]	$MR = \text{Exp}(-kt)^n$
Henderson and Pabis [23]	$MR = a\,\text{Exp}(-kt)$
Logarithmic [24]	$MR = a\,\text{Exp}(-kt) + c$
Verma *etal* [25]	$MR = a\,\text{Exp}(-kt) + (1-a)\,\text{Exp}(-gt)$
Midilli *etal.* [26]	$MR = a\,\text{Exp}(-kt^n) + bt$
Two term exponentials [27]	$MR = a\,\text{Exp}(-kt) + (1-a)\,\text{Exp}(-akt)$
Two term [25]	$MR = a\,\text{Exp}(-k_o t) + b\,\text{Exp}(-k_1 t)$
Approximation of diffusion [28]	$MR = a\,\text{Exp}(-kt) + (1-a)\,\text{Exp}(-bkt)$

D. Effective Diffusivity and Activation Energy

Moisture removal in drying of leaves is mainly by diffusion, hence Fick's second law of diffusion was employed in the estimation of effective diffusion coefficient of Marugbo leaves during drying process. Assuming a uniform initial moisture distribution, constant moisture diffusivity and negligible shrinkage, the solution of Fick's law for infinite slab geometry is as given in Eq. (4) [29],

$$MR = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\text{Exp}\left(-\frac{(2n+1)^2\pi^2 Dt}{4L^2}\right) \quad (4)$$

D is the effective diffusivity (m^2/s), t is the time (s), L is the thickness of the sample and n = positive integer 1, 2, 3…

For a long drying period, the first term of the series in Eq. (4) is considered [30], yielding

$$MR = \frac{8}{\pi^2}\text{Exp}\left(-\frac{\pi^2 Dt}{4L^2}\right) \quad (5)$$

The effective diffusivity is determined from the slope of the graph of *In MR* against *t*.

$$\text{slope } m = \frac{\pi^2 D}{4L^2} \quad (6)$$

The effect of drying temperature on the effective moisture diffusivity and the activation energy is determined from Arrhenius equation [31];

$$D = D_o Exp\left(-\frac{E_a}{RT}\right) \quad (7)$$

D is the effective diffusivity (m^2/s), D_o is Arrhenius pre-exponential factor (m^2/s), E_a is the activation energy (kJ/mol), R is the ideal gas constant (8.3143 J/mol K) and T (K) is the drying temperature.

The graph of *In D* against reciprocal of drying temperature (1/T) gives a slope of the activation energy E_a/R.

E. Statistical Analysis

The most suitable thin layer model was determined using coefficient of determination (R^2), chi square (χ^2), sum square errors (SSE) and root mean square error (RMSE). R^2 was determined from regression analysis carried out with excel data analysis. Reduced Chi square (χ^2), SSE and RMSE are determined from equation (8), (9) and (10) respectively

$$\chi^2 = \frac{\sum_{i=1}^{n}\left(MR_{\text{Exp}} - MR_{\text{model}}\right)^2}{N - z} \quad (8)$$

$$\text{SSE} = \sum_{i=1}^{n}\left(MR_{\text{Exp}} - MR_{\text{model}}\right)^2 \quad (9)$$

$$\text{RSME} = \sqrt{\frac{\sum_{i=1}^{n}\left(MR_{\text{Exp}} - MR_{\text{model}}\right)^2}{N}} \quad (10)$$

$MR_{experimental}$ is the moisture ratio from drying experiment computed from equation (2). MR_{model} is the predicted moisture ratio from thin layer drying model. N is the number of data points collected and z is the number of constants in the thin layer model. The most suitable thin layer model has higher values of $R^2$with lower values of χ^2, SSE and RMSE [13], [23], [25], [28], [31]-[34].

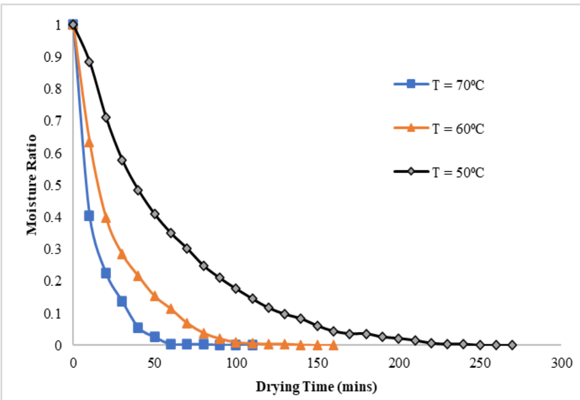

Fig. 1. Variation of moisture ratio with time.

III. RESULTS AND DISCUSSION

A. Drying Characteristics of Marugbo Leaves

The variation of moisture ratio with time at different drying temperature of 50, 60 and 70°C shows a decreasing trend as shown in Fig. 1. Drying time was 50, 150 and 250 minutes for 70, 60 and 50°C drying temperature respectively. Shorter drying time at 70°C is as a result of larger heat transfer driving force as compared with lower temperatures as reported in previous works [31], [35].

The drying rates for the three drying temperatures are shown in Fig. 2. It was observed to increase with temperature and reach very high peak at 0.185, 0.117 and 0.059 g H_2O/g dry solid. min at 70, 60 and 50°C respectively because of high temperature gradient which makes more evaporation to take place [31]. Drying rate becomes higher as temperatures increases due to the increased vapour pressure of water as evaporation occurs from the regions near the surface [36]. The rates then decreases as drying progressed due to internal moisture diffusion process as also observed in previous studies on drying [31], [37]. Hence, increasing the drying temperature causes increases in rate of drying and reduction in the drying time.

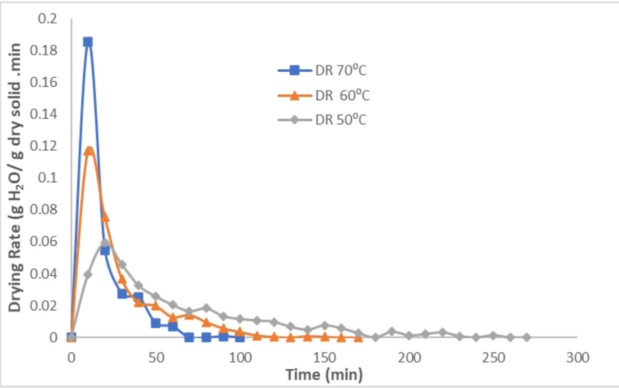

Fig. 2. Drying rate variation with time at different temperature.

The drying curves of Marugbo leaves show that there was absence of constant drying rate period and drying process occurred in falling rate region only as shown in Fig. 3. This agrees with previous studies [13], [18], [35], [36]. During the falling rate period, moisture transport is by mechanism of diffusion and there is increase in mass and heat transfer resistance through the material [18], [21].

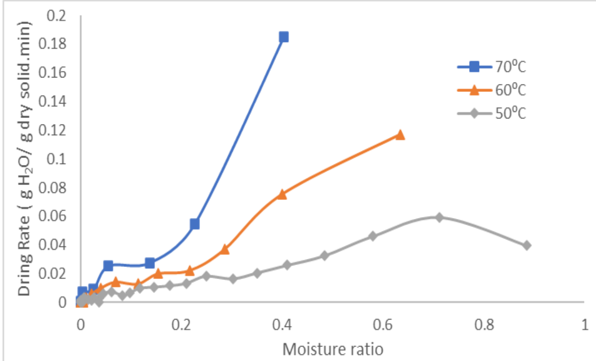

Fig. 3. Drying rate curve at different temperature.

B. *Evaluation of Thin Layer Models*

The estimated kinetic constants, coefficient of regression (R^2), reduced chi- square(χ^2), sum square errors (SSE) and root mean square error (RMSE)for eleven different thin-layer drying models used to describe the drying process of *Marugbo* leaves are presented in Table II.

The model with highest R^2 but lowest χ^2, SSE and RMSE gives the best fit for the drying process. Comparison of R^2, χ^2, SSE and RMSE for all three temperatures showed that the Approximation of diffusion's model and oven drying at 50 °C gave better fit than other drying temperatures with R^2 = 0.9985, SSE = 0.0032, χ^2= 0.00012 and RMSE = 0.0107. Also, for all drying temperatures, the Wang and Singh Model is the least fit for the drying prediction.

The model validation of the experimental moisture ratio shows a good fit as depicts in Figure 4. The predicted values banded closely around the experimental curve showing the suitability of the models for predicting the drying characteristics of *Marugbo* leaves.

TABLE II: RESULTS OF DRYING KINETIC CONSTANTS FOR DIFFERENT THIN LAYER MODELS FOR MARUGBO LEAVES

Temp.	Model Name	Kinetic constants	R^2	SSE	X^2	RMSE
70°C	Page	n = 1.1545, k = 0.0479	0.9874	0.0135	0.00052	0.0220
	Modified Page	n = 1.1546, k = 0.0719	0.9874	0.0135	0.00052	0.0220
	Henderson and Pabis	a = 1.127, k = 0.0915	0.9919	0.0256	0.00099	0.0302
	Lewis	k = 0.0894	0.9929	0.0092	0.00034	0.0181
	Logarithmic	a = 1.2021, k = 0.0999, c = 0.0014	0.9871	0.0539	0.00216	0.0439
	Vermal et al	a = 2.8283, k = 0.1052, g = 0.1329	0.9874	0.0135	0.00054	0.0219
	Wang and Singh	a = -0.0289, b = 0.0002	0.8750	0.2710	0.01042	0.0984
	Midilli et al	n = 0.931, k = 0.1407, b = 3.119E-05, a = 1.3589	0.9732	0.1460	0.00608	0.0722
	Two term exponentials	a = 1.00, k = 0.0894	0.9929	0.0092	0.00035	0.0181
	Two term	a = 0.5635, k_o = 0.0915, b = 0.5635, k_1 = 0.0915	0.9919	0.0256	0.00107	0.0303
	Approximation of diffusion	a = 0.9999, k = 0.0944, b = -0.5378	0.9899	0.0132	0.00053	0.0217
60°C	Page	n = 1.1432, k = 0.0230	0.9895	0.0204	0.00127	0.0336
	Modified Page	n = 1.1432, k = 0.0369	0.9895	0.0204	0.00127	0.0336
	Henderson and Pabis	a = 1.163, k = 0.0466	0.9935	0.0444	0.00278	0.0497
	Lewis	k = 0.0452	0.9948	0.0090	0.00053	0.0223
	Logarithmic	a =1.1027, k = 0.0446, c = -0.00074	0.9952	0.0222	0.00148	0.0351
	Vermal et al	a = 3.5977, k= 0.0541, g = 0.0634	0.9905	0.0179	0.00119	0.0315
	Wang and Singh	a = -0.01598, b = 6.328E-05	0.8965	0.3948	0.02467	0.1481
	Midilli et al	n = 1.0514, k= 0.0351, b = -2.748E-06, a = 1.0456	0.9949	0.0142	0.00102	0.0281
	Two term exponentials	a = 1.00, k = 0.0452	0.9948	0.0090	0.00056	0.0223
	Two term	a = 0.5899, k_o= 0.0466, b = 0.5731, k_1 = 0.0466	0.9935	0.0444	0.00317	0.0497
	Approximation of diffusion	a = 1.0222, k = 0.0420, b = 0.4604	0.9962	0.0057	0.00038	0.0178
50°C	Page	n = 1.3132, k = 0.0041	0.9895	0.0520	0.00199	0.0431
	Modified Page	n = 1.3129, k = 0.0152	0.9896	0.0518	0.00199	0.0430
	Henderson and Pabis	a = 1.3375, k = 0.0232	0.9834	0.1843	0.00709	0.0811

Lewis	k = 0.02174	0.9896	0.0592	0.00219	0.0460
Logarithmic	a =1.0689, k = 0.0180, c = -0.01187	0.9986	0.0063	0.00025	0.0150
Vermal et al	a = 12.884, k = 0.0296, g = 0.03153	0.9904	0.0430	0.00172	0.0392
Wang and Singh	a = -0.00896, b = 2.00734E-05	0.9545	0.2838	0.01092	0.1007
Midilli et al	n = 1.05199, k = 0.01414, b = -3.666E-05, a= 1.01665	0.9989	0.0037	0.00015	0.0115
Two term exponentials	a = 1.00, k = 0.02174	0.9896	0.0592	0.00228	0.0460
Two term	a = 0.6687, k_o = 0.02318, b = 0.6687, k_1 = 0.02318	0.9834	0.1843	0.00768	0.0811
Approximation of diffusion	a = -2.1378, k = 0.0119, b = 1.1275	0.9985	0.0032	0.00012	0.0107

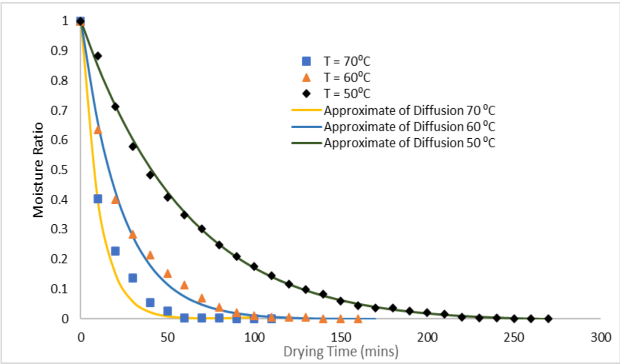

Fig. 4. Comparison between experimental data and best fit thin layer model.

C. Effective Diffusivity and Activation Energy

The effective diffusivity which indicates the speed at which moisture diffuses out of the sample was determined from the slope of the graph of *In MR* against *t* at different temperature is presented in Fig. 5.

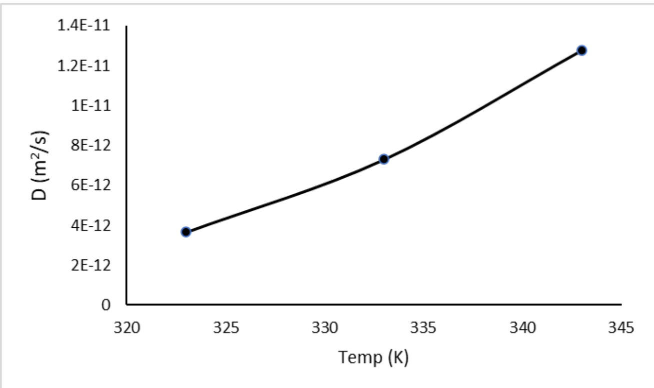

Fig. 5. Effective diffusivity of Marugbo leaves at different temperature.

The diffusivity increases with temperature and ranges from 3.65×10^{-12} to 1.28×10^{-11} m^2/s Although there is no record of effective diffusivity of Marugbo leaves during drying process but these values are within reported values for a typical savoury (6.76×10^{-12} - 1.57×10^{-10} m^2/s) [31] and medicinal leaves ($5.55\times 10^{-12}$to 1.17×10^{-11}) [13], [32]. Also, similar direct dependence of diffusivity on drying temperature has been recorded in the previous studies on drying of leaves [13], [23], [28], [31]-[33], [36].

The minimum energy required for diffusion (activation energy) was computed to be 57.74 kJ/mol. from the slope of the graph of *In D* against reciprocal of drying temperature (1/T) as shown in Fig. 6.

The activation energy value of Marugbo leaves is greater than that recorded for savoury leave (42.07 kJ/mol.) [31], lower than medicinal leaves such as *pachystachya* leaves (64.53 kJ/mol.) [34], fever leaves (80.78kJ/mol.) [13] but within the range of 12.7–110 kJ/mol. recorded for food materials [31]. There is also strong dependence of diffusivity on temperature with R^2 = 0.9981 and the Arrhenius equation for Marugbo drying is presented in equation (11).

$$D = 8.06\times10^{-3}\ \text{Exp}\left(-\frac{6944.9}{T}\right) \qquad (11)$$

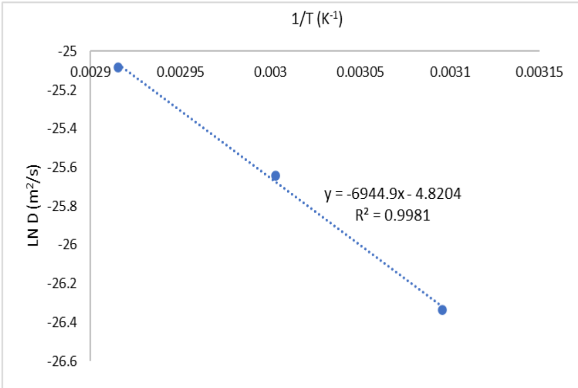

Fig. 6. Arrhenius representation of the effect of temperature on diffusivity.

IV. Conclusion

The drying kinetics of Marugbo leaves was studied in an oven dryer at different temperatures of 50, 60 and 70ºC. Moisture ratios calculated from weight loss with time obtained from the drying experiment were fitted in eleven thin-layer drying models. The observed results showed decrease in drying time with increase in temperature while drying rate increases with increase in temperature. These are general observations in drying studies of food and agricultural materials. Drying process occurs at falling rate period at all temperatures with no constant rate period. Approximation of diffusion model which gave the least sum of square errors, root mean square error and reduced chi square and higher coefficient of determination was considered to be the best model for predicting drying of Marugbo leaves. The effective diffusivity for Marugbo drying increases with temperature from 3.65×10^{-12} at 50ºC to 1.28×10^{-11} m^2/s at 70ºC. The Arrhenius equation also describe the temperature dependence of diffusivity with activation energy of 57.74 kJ/mol. This research work was carried out during raining season which made it difficult to carry out sun drying for comparison with oven drying experimental results. Therefore, comparison of drying kinetics of oven drying with drying kinetics of sun drying of Marugbo leaves is a future research work is this area of study. This will give opportunity for evaluating effect of the two drying processes on nutritional values of dried Marugbo leaves.

Conflict of Interest

The authors declare no conflict of interest.

Authors' Contributions

A. A designed, performed the experimental study and analyzed the data. He was a major contributor in writing. G. O. was responsible for writing the introduction and review the literature. She is also responsible for sample collection and data recording. I. O. participated in discussion and interpretation of data. He also reviewed and edited the article. O. I. served as supervisor and correct the manuscript. All authors read and approved the final manuscript.

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