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RESEARCH ARTICLE

Comparative study of the drying characteristics and kinetics of Utasi (*gongronema latifolium*) and Odusa (*piper guineense)* leaves

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ABSTRACT

Drying characteristics and kinetics of Utasi (gongronema latifolium) and Odusa (piper guineense) leaves were investigated in laboratory scale Oven dryer, at a temperature range of 40, 50, 60 and 70oC for utasi leaves and odusa leaves. The thickness of 0.27mm and 0.29mm were obtained for utasi leaves (Gongronema latifolium) and odusa leaves (Piper genus) The measured thickness were 0.27 mm and 0.29 mm for Utasi and odusa respectively with average initial moisture content of 83% (w.b) for Utasi and 80% (w.b) for udusa at 105oC for 30 minutes as determined by oven drying. A proximate analysis conducted on the fresh and dried leaves indicates significant different on te composition; such as moisture, protein, fat, crude fibre, ash and carbohydrate. The results from the drying process showed decrease in moisture ratio with increasing drying time while drying become faster with increase in temperature. Drying process took place only in the falling rate period for utasi and odusa leaves. The Page model showed a better fit than the Newton's and modified page model which could be used to describe a drying process system. Page model represented the thin layer drying of utasi and odusa leaves, compared to the other models used at 40oC, 50oC and 70oC while Newton model represented the thin layer drying of utasi and odusa leaves at 60oC. The values of calculated effective diffusivity ranged from 1.86 x 10-10 to 2.66 x 10-10 for utasi leaves and 1.39 x 10-10 to 6.29 x 10-10 for odusa leaves. Temperature dependence of the diffusivity coefficients was described by an Arrhenius-type relationship. The activation energy for moisture diffusion was found to be 11.535kJ/mol and 45.168kJ/mol for utasi and odusa leaves respectively.

Keywords: Drying, Spices, Moisture Ratio, Moisture Diffusivity, Kinetics

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INTRODUCTION

Spices are common plant materials that are used as food additives to enhance aroma and flavour (Souza et al., 2005, Okwonkwo and Ogu, 2014). They possess medicinal properties such as antimicrobial activity, antioxidants, etcetera. Like fruits and vegetables, they are difficult to preserve due to high moisture content and tender texture. Drying technique is one of the earliest techniques used in removing moisture by means of evaporation process (Afolabi and Garry, 2014), caused by heated air convection forces so that the shelf life of the material can be prolonged significantly. Drying technique also influence other characteristics such as palatability of the food, flavour, aroma, viscosity, hardness, microbial spoilage, enzymatic activity (Izli et al., 2017) and increase the concentration of nutrients (Doymaz, 2005, Famurewa, and Olumofin, 2015).

The Piper Guineense is made up of about 1050 species of tropical shrubs, lianas, and small trees, many of which are important as spices, flavouring agents and medicines Piper guineense (Ashanti pepper) is an erect herbaceous climbing liana native to tropical Africa, ranging from Guinea to Kenya and south of Zambia (Owolabi et al., 2013).

Gongronema latifolium is a perennial edible plant with soft and pliable stem. It is widely used in the West African sub-region for a number of medicinal and nutritional purposes. It belongs to the family of asclepiadaceae (Imo et al., 2015). Gongronema latifolium is widespread in tropical Africa. It is found in rainforest, deciduous and secondary forests, and also in mangrove and disturbed roadside forest, from sea-level up to 900 m altitude. It is known in different part of Nigeria, Ghana and Senegal (Edim et al., 2012). They are sharp-bitter and sweet and widely used as a leafy vegetable and as a spice for sauces, soups and salads. The leaves which have a peppery taste, are pale greenish colour when fresh and darker green when frozen or dried (Besong et al., 2016). Gongronema latifolium is a good source of protein, minerals and vitamins (Balogun et al., 2016). Previous researches have shown that the leaves are suitable for use in food production due to their high amino acid contents (Osuagwu et al., 2013). The leaves can be eaten fresh, dried and used as local powdery spice or as vegetable for food preparations such as unripe plantain porridge, white soup, sauces and salads in which they add a bitter-sweet flavor. There is high concentration of vitamins (A, C, E, and niacin) in the leaves of Gongronema latifolium (Balogun et al., 2016).

In the Literature, there are many studies on drying of spices such as Ginger (Zingiber officinale) (Loha et al., 2012), coffee berry (Correa et al., 2006), Bitter leave (vernonia amygdalina) and Scent leave (ocimum gratissimum), (Gumus and Banigo, 2015) and the phytochemical and mineral composition of Piper Guineense and Gongronema Latifolium respectively. However the drying of the Piper Guineense and Gongronema latifolium leaves for kinetic studies in literature is scarce. Therefore, the drying of these leaves will preserve them for longer period of time and make them available and affordable at off season (Doymaz, 2011) while the drying kinetic data will be useful for design purposes.

2.0 MATERIALS AND METHODOLOGY

2.1 MATERIALS

Fresh utasi (gongronema latifolium) and odusa (piper guineense) leaves were obtained from the market in Use-offot village, the utasi and odusa leaves were washed and drained thoroughly using towel. The thickness of utasi and odusa leaves was measured using a vernier caliper

2.2 METHODS

2.2.1 Determination of initial moisture content The average moisture content of utasi and odusa leaves was 83% (w.b) and 80% (w.b) at 105oC for 30 minutes as determined by oven drying. Experiments were performed at 40oC, 50oC, 60oC and 70oC for Utasi and Odusa leaves respectively. Values of weight loss were taken at an interval of 30 minutes until there was constant value, no more weight loss. Calculation of average moisture content was done using the formula in equation 1.

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$$\text{Average moisture \%} = \frac{W2 - W3}{W2 - W1} \times 100 \quad 1$$

W1 = weight of empty pan
W2 = weight of pan + sample before drying
W3 = weight of pan + sample dried to constant weight

2.2.2 PROXIMATE ANALYSIS

The chemical composition of the samples was determined using the standard methods of Analysis of Association of Official Chemists (AOAC), 1995. Moisture, crude fibre, crude protein, crude lipid, ash and carbohydrate contents in the samples was analyzed.

2.2.3 DETERMINATION MOISTURE CONTENT

A clean crucible was dried in an oven at 110°C, cooled in a desiccator and Weighed (W1). Two grams (2g) of finely ground sample was weighed into the previously labeled crucible and reweighed (W2). The crucible containing the sample was dried in an oven to constant Weight (W3). The percentage moisture content was calculated thus:

$$\%\, Moisture\ content = \frac{W2 - W3}{W3 - W1} x\ 100 \quad 2$$

2.2.4 DETERMINATION OF CRUDE FIBRE

Two grams (2 g) of the sample was weighed into a round bottom flask, 100 cm3, 0.25 M sulphuric acid solution was added and the mixture boiled under reflux for 30 min in a hot plate. The hot solution was quickly filtered under suction. The insoluble matter was washed several times with hot water until it was acid free. It was then transferred into the flask and 100cm3 of hot 0.31 M Sodium Hydroxide solution was added, the mixture boiled under reflux for 30 min and filtered under suction. The residue was washed with boiling water until it was base free, dried to constant weight in an oven at 100°C, cooled in a desiccator and weighed (W1). The weighed sample (W1) was then incinerated in a muffle furnace at 550°C for 2 hours, cooled in a desiccator and reweighed (W2).

Calculation: The loss in weight on incineration = W1- W2

$$\%\, Crude\ fibre = \frac{W1 - W2}{Weight\ of\ original\ sample} x\ 100 \quad 3$$

2.2.5 DETERMINATION OF CRUDE PROTEIN

One gram (1g) of the ground sample was weighed into a beaker. 1.5g of sodium sulphate (Na2SO4), 1.5g of Copper Sulphate (CuSO4) and 10ml of concentrated sulphuric acid (H2SO4) were added to the sample in the beaker. The beaker was placed tilted to an angle in the digester, boiled and allowed to stand until the solution is bluish. The solution was allowed to cool while 90ml of distilled water was added to the digested sample and the volume was made- up to 100ml using 100ml volumetric flask. 20 ml of the digested solution and 20ml of 40% sodium hydroxide solution was added. The solution was distilled under 10ml saturated boric acid. The distillate was titrated with 0.1M hydrochloric acid (HCl) using drops of mixed indicator until the solution changes from blue to reddish brown. The crude protein content was calculated using the Equation 4 and 5.

$$\text{Nitrogen in sample (\%)} = \frac{titre - blank \times 0.01401 \times 0.1}{weight\ of\ sample} \quad 4$$

Crude protein (%) = nitrogen sample x 6.25 5

2.2.6 DETERMINATION OF CRUDE LIPID

The leaves were extracted with 300cm3 of petroleum ether at 40-60°C using Soxhlet extraction unit. for 6 h. The solvent was recovered and the oil dried in an oven set at 70°C for 1 h. The round bottom flask and oil was then Weighed

DOI: https://doi.org./10.24163/ijart/2017/6(2):7-19

(W2). The lipid content was calculated using equation 6

$$\%\ Crude\ Lipid\ content = \frac{W2-W3}{W3-W1} x\ 100 \quad 6$$

2.2.7 DETERMINATION OF TOTAL ASH

A known weight of the leave was placed in and crucible a place in a furnace at 550°C for 8 hours to form ash. The crucible was removed and put in desiccator to cool and the weight taken (w3). The percentage ash content was calculated using equation 7.

$$\%\ Ash\ content = \frac{W3-W1}{W2-W1} x\ 100 \quad 7$$

2.2.8 Total carbohydrate content

Available carbohydrate content in the sample was calculated as the difference obtained after subtracting the lipid, ash and fibre values from the total dry matter using equation 8.

% carbohydrate = 100 – (a + b + c + d) 8

Where a = percentage of crude protein
b = percentage of crude lipid
c = percentage of ash content
d = percentage of crude fiber.

2.3 Drying characteristics

The moisture ratio MR of the samples during drying can be determined using equation 9

$$MR = \frac{M-M_e}{M_o-M_e} \quad 9$$

Where M is the mean moisture content, Mo is the initial moisture content and Me is the equilibrium moisture content. Equation 9 is known as experimental moisture ratio

2.4 Mathematical Models and Statistical Analysis

The moisture ratio data obtained from equation 9 was fitted into the three thin layer drying models in equations 10-13 in order to select the best model that can predict the drying characteristics (ElKhodiry et al., 2015 and Gumus and Banigo, 2015).

$$MR = \frac{M-Me}{Mo-Me} = Exp(-kt) \quad (\text{Newton's Model}) \quad 10$$

$$MR = \frac{M-Me}{Mo-Me} = a \cdot \text{Exp}(-kt) \quad (\text{Henderson and Pabis}) \quad 11$$

$$MR = \frac{M-Me}{Mo-Me} = Exp(-kt^n) \quad (\text{Page model}) \quad 12$$

$$MR = \frac{M-Me}{Mo-Me} = Exp(-kt)^n \quad (\text{Modified Page model}) \quad 13$$

Where; M is Moisture ratio at time t, Me is Equilibrium Moisture ratio, Mo is Initial Moisture ratio. k is slope (for Newton and Henderson's Model), k is Exponential of intercept, n is slope, a is intercept and t is drying time. Also, equation 9 is the experimental moisture ratio, (MRexpi) while equations 10-13 are the predicted moisture ratios (MRprei) from the models

2.5 ESTIMATION OF STATISTICAL ERRORS

The fitness of experimental data was evaluated using statistical analysis. The Statistical parameters such as: reduced sum square error (SSE), Root Mean Square Error (RMSE) and reduced Chi-square error (x2) were estimated using the following mathematical expressions in equations 14-16. The x2 values for all sample thickness at all temperatures for each model was estimated by substituting the MRexpi and MRprei into equation 14. Also, the SSE values were obtained by substituting MRexpi and MRprei into equation 15. Furthermore, the RMSE values were obtained by substituting MRexpi and MRprei into equation 16.

$$x2 = \frac{\sum_{i=1}^{N}(MRexpi-MRprei)}{N-Z} \quad 14$$

$$SSE = \frac{1}{N}\sum_{i=1}^{N}(MR_{expi} - MR_{prei})_2 \quad 15$$

DOI: https://doi.org./10.24163/ijart/2017/6(2):7-19

$$RMSE=\sqrt{\left[\frac{1}{N}\sum_{i=1}^{N}(MR_{expi}-MR_{prei})\right]} \quad 16$$

Where;

$R2$ = coefficient of determination
$\chi 2$ = reduced chi-square
SSE = reduced sum square error
RMSE = root mean square error
MR$expi$ is the experimental moisture ratio,
MRprei, is the moisture ratio predicted from the model equation,
N is the number of data points, and
Z is the number of constants in the model equation.

The best fit was decided for highest value of R2 and minimum value of X2, SSE and RMSE.

2.6 ESTIMATION OF EFFECTIVE MOISTURE DIFFUSIVITY

The effective moisture diffusivities at different temperatures were calculated by applying Fick's second law of diffusion, simplified into equations 17 and 18 (ElKhodiry et al., 2015). This law has been widely accepted to describe the falling rate period of different agricultural products.

$$MR=\frac{8}{\pi^2}e^{-\pi^2 D_{eff}\,t/4L^2} \quad 17$$

$$\ln MR=\ln\left(\frac{8}{\pi^2}\right)\frac{\pi^2 D_{eff}\,t}{4L^2}) \quad 18$$

Where t is the drying time (s), $Deff$ is the effective diffusivity (m2/s), and L is half thickness of the leaves (m)

The effective diffusivities for the samples were determined by plotting ln(MR) vs drying time in equation 19 to give a straight line with slope (k) as presented in equation 19.

$$k=(\frac{\pi^2 D_{eff}}{4L^2}) \quad 19$$

2.7 Drying Kinetics

The temperature dependency of the effective moisture diffusivity of the samples was investigated using the temperature dependent Arrhenius equation (20). The Deff values obtained from equation 20 for all samples were inserted into equation 20.

$$D_{eff}=D_o\exp\left(-\frac{Ea}{RT}\right) \quad 20$$

Where;

$D0$=pre-exponential factor or the diffusivity at infinite temperature (m2/s),
Ea = activation energy (kJ mol−1),
R = universal gas constant (0.008314 kJmol−1K−1), and
T =absolute temperature (K).

Where Do is the pre-exponential factor, Ea is the activation energy, T is the temperature for drying and R is the universal gas constants. The activation energies and the exponential factors can be obtained by plotting ln(Deff) against 1/T.

3.0 RESULTS AND DISCUSSION

3.1 Proximate Analysis for Odusa (piper guineense) and Utasi (gongronema latifolium) leaves

The proximate composition of odusa (piper guineense) and utasi (gongronema latifolium) leaves presented in Table 1 shows that the leaves contain moisture, ash, crude fibre, fat, crude protein and carbohydrate. From the result, it is shown that carbohydrate and protein contents are higher in dried utasi leaves (40.25g and 28.64g) than in odusa leaf (39.02g and 16.86g),While ash and crude fibre are higher in dried odusa leaves (13.81g and 16.89g) than in utasi leaves (9.15g and 7.30g) and moderate content of fat as compared to fresh leaves.

DOI: https://doi.org./10.24163/ijart/2017/6(2):7-19

Table -1: Proximate composition of odusa (piper guineense) and utasi (gongronema latifolium) leaves

Samples	Nutrients %					
	Moisture	Ash	Crude Fibre	Fat	Crude protein	Carbohydrates
Fresh Odusa	80.0	1.05	2.51	1.30	6.33	8.81
Dried Odusa	10.92	13.81	16.89	2.50	16.86	39.02
Fresh Utasi	83.0	0.62	1.32	1.84	5.72	7.50
Dried Utasi	11.08	9.15	7.30	3.58	28.64	40.25

3.0 DRYING CURVES

Low temperatures drying were used to analysis the drying characteristics of Utasi (gongronema latifolium and Odusa (piper guineense) leaves. The relationship between moisture ratio and drying time were presented in the Figures 1 and 2 below for the drying of Utasi and Odusa leaves at the drying temperatures. It was observed that the moisture ratio decreases continuously with increasing time. The moisture ratio decreases faster at 70oC followed by 60oC, 50oC and 40oC which almost having the same values. The temperature at 40oC takes a longer time to reduce the moisture content to lower limit. As can be seen, it takes 90 min, 120min, 150min and 180min to reach steady moisture ratio at temperatures of 70oC, 60oC, 50oC and 40oC respectively. The results show that all drying took place in falling rate period which is an indication that internal mass transfer occurred by diffusion. Figure 1 below shows the result of drying experiment of utasi (gongronema latifolium) leaves at different temperatures of 40, 50, 60 and 70oC at 30 minutes interval. From the result above, it is observed that at higher temperature the sample dry faster than at lower temperature. It takes 90minutes for a sample with initial moisture contents of 1.05g to attain constant weight (equilibrium moisture content) while it takes 180minutes for a sample of 1.10g to attain constant weight. Similar observation is also seen in Figure 2, highlighting the drying experiment of piper guineense leaves.

DOI: https://doi.org./10.24163/ijart/2017/6(2):7-19

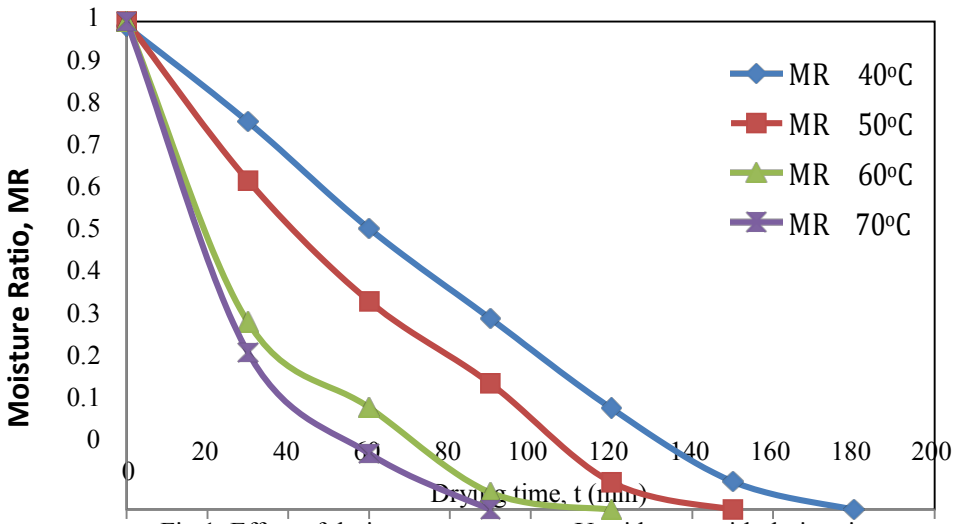

Fig 1: Effect of drying temperature on Utasi leaves with drying time.

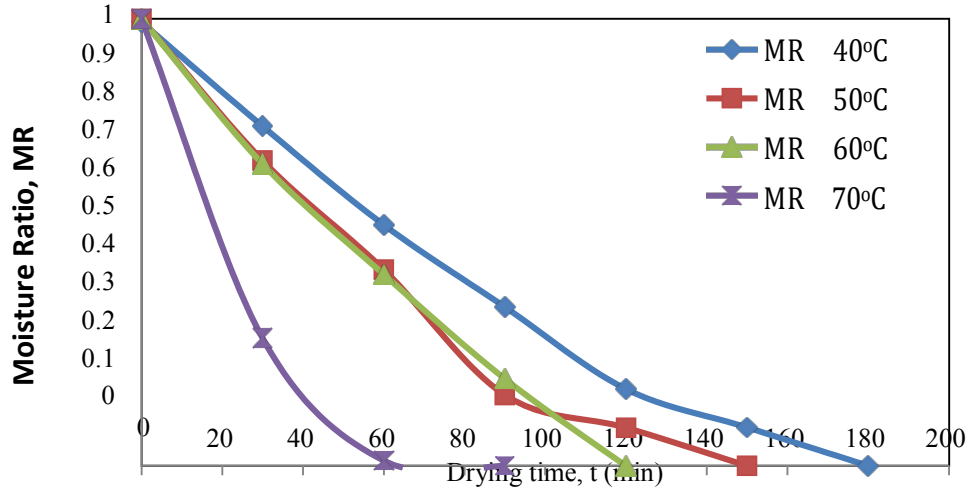

Fig 2: Effect of drying temperature on Odusa leaves with drying time.

At a higher temperature of 70oC, the time was drastically reduced compared to that at 60oC, 50oC and 40oC as can be seen in Figures 1 and 2, it takes only 30min for moisture ratio to reduce to minimum value at higher temperatures for both Utasi and Odusa leaves. At lower temperatures, the trend was different; it takes longer time to constant moisture ratio. The results were generally in agreement with literature studies on drying of basil leaves (Kadam et al., 2011), and drying characteristics and kinetics of bitter leaves and scent leaves by Gumus and Banigo (2015).

3.1 MATHEMATICAL MODEL FOR FITTING DRYING CURVE

Drying curves were fitted with four moisture ratio models, namely, Newton's model, Henderson and Pabis model, Page model and the Modified Page model. The coefficient of correlation and results of statistical analyses are listed in Table 2 with the coefficient of correlation ranging from

DOI: https://doi.org./10.24163/ijart/2017/6(2):7-19

0.797 to 1.000. The analysis indicates that for Utasi and Odusa leaves, Page model has highest values of R2 with lowest values of X2, SSE and RMSE except for the temperature of 60 oC. At 60 oC, drying experimental data fitted very well to Newton models with the coefficient of correlation of 0.994 and 0.966 for Utasi and Odusa leaves respectively. The Page model represented the thin layer drying of utasi and odusa leaves, compared to the other models used.

Table -2: Statistical analysis of fitted mathematical models to thin layer drying data of Utasi and Odusa leaves at different temperatures.

Name of Model	Sample	Temp oC	R2	X2	SSE	RMSE	a	K	n
Newton MR= Exp(-kt)	Utasi Leaf	40	0.962	0.0033	0.0031	0.2147		0.012	
		50	0,978	0.0014	0.0013	0.1477		0.016	
		60	0.994	0.0002	0.0002	0.0648		0.030	
		70	0.998	0.0001	0.0001	0.0351		0.040	
	Odusa leaf	40	0.976	0.0021	0.0019	0.1701		0.013	
		50	0.977	0.0015	0.0014	0.1559		0.017	
		60	0.966	0.0012	0.0012	0.1659		0.018	
		70	0.995	0.0002	0.0002	0.0636		0.000	
Henderson and Pabis $MR=a\cdot Exp(-kt)$	Utasi Leaf	40	0.957	0.0032	0.0516	0.0526	1.058	0.013	
		50	0.976	0.0014	0.0014	0.0347	1.033	0.013	
		60	0.994	0.0002	0.0002	0.0132	0.998	0.030	
		70	0.998	0.0001	0.0001	0.0066	1.001	0.038	
	Odusa leaf	40	0.972	0.0020	0.0018	0.0419	1.045	0.013	
		50	0.974	0.0015	0.0013	0.0363	1.040	0.017	
		60	0.962	0.0012	0.0011	0.0329	1.036	0.017	
		70	0.995	0.0002	0.0001	0.0020	1.121	0.001	
Page MR= Exp(-ktn)	Utasi Leaf	40	0.993	0.0005	0.0004	0.0211		0.001	1.592
		50	0.991	0.0005	0.0004	0.0210		0.003	1.369
		60	0.994	0.0002	0.0002	0.0132		0.032	0.990
		70	0.998	0.0001	0.0001	0.0064		0.030	1.063
	Odusa leaf	40	0.995	0.0004	0.0003	0.0174		0.001	1.447
		50	0.994	0.0003	0.0003	0.0164		0.002	1.460
		60	0.797	0.0353	0.324	0.0799		0.122	0.249
		70	1.000	0.0001	0.0001	0.0017		0.001	0.240
Modified page MR= Exp(-(kt)n)	Utasi Leaf	40	0.962	0.0035	0.0032	0.0553		0.003	3.862
		50	0.978	0.0015	0.0013	0.0358		0.049	0.333
		60	0.994	0.0002	0.0002	0.0132		0.122	0.249
		70	0.998	0.0001	0.0001	0.0066		0.180	0.218
	Odusa leaf	40	0.976	0.0022	0.0019	0.0439		0.003	3.978
		50	0.977	0.0016	0.0014	0.0378		0.050	0.333
		60	0.966	0.0013	0.0011	0.0339		0.074	0.229
		70	0.995	0.0002	0.0000	0.0120		0.000	0.240

DOI: https://doi.org./10.24163/ijart/2017/6(2):7-19

3.4 EFFECTIVE MOISTURE DIFFUSIVITY

Values of effective moisture diffusivity, Deff and coefficient of correlation, R2 calculated from equations 18 and 19 are presented in Table 3. The effective moisture diffusivity ranges from 1.86 x 10-10 to 2.66 x 10-10 for utasi leaves and 1.39 x 10- 10 to 6.29 x 10-10 for odusa leaves. The effective moisture diffusivity increased with an increase in drying air temperature for drying of utasi and odusa leaves. The maximum R2 value was 0.9992 for Utasi leaves drying at 70oC and minimum of 0.8956 at 40oC compared to odusa leaves at the same temperatures with values of 0.9408 and 0.9616. The results from the present work is in accordance to results reported by Bennamoun and Belhamri (2006), Azimi, et al. (2012), ElKhodiry et al. (2015) that the general range of effective moisture diffusivity of agricultural products is between 10-8 to 10-11 m2/s.

Table -3: Moisture diffusivity and its linear equation

Samples	Temperature (oC)	Deff (m2/s)	R2	Linear Equation
Utasi leave	40	1.86 x 10-10	0.8956	y = -0.0178x + 0.3056
	50	1.96 x 10-10	0.9025	y = -0.0224x + 0.2479
	60	2.43 x 10-10	0.9512	y = -0.0356x + 0.1316
	70	2.66 x 10-10	0.9992	y = -0.0361x - 0.0173
Odusa leave	40	1.39 x 10-10	0.9616	y = -0.0163x + 0.1974
	50	2.04 x 10-10	0.9686	y = -0.0213x + 0.175
	60	3.52 x 10-10	0.971	y = -0.0179x + 0.0854
	70	6.29 x 10-10	0.9408	y = -0.0738x + 0.3209

3.5 DRYING KINETICS

Activation energy is the minimum amount of kinetic energy required for a reaction to take place. An increase in temperature brings about a corresponding increase in activation energy. From the slope of the straight lines in Figures 3 and 4, the activation energies were found to be 11.535kJ/mol and 45.168kJ/mol for utasi and odusa leaves respectively and are presented in Table 4.

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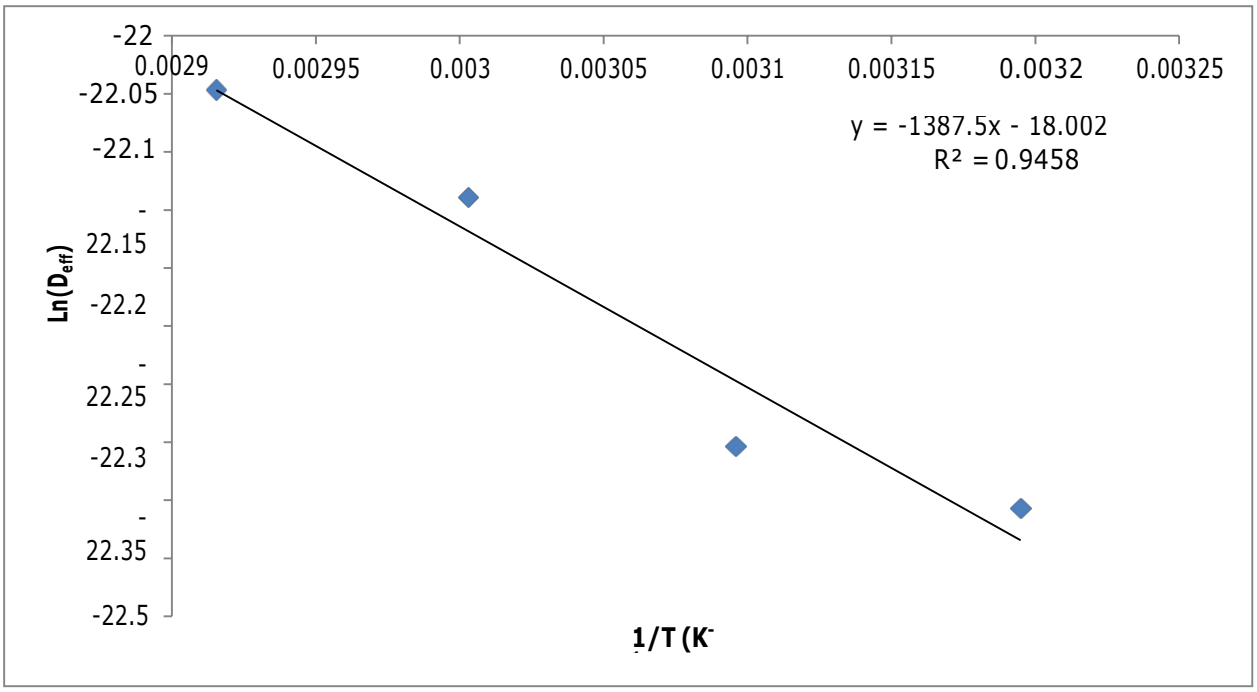

Fig 3: Effect of drying temperature on the effective diffusivity for utasi leaves.

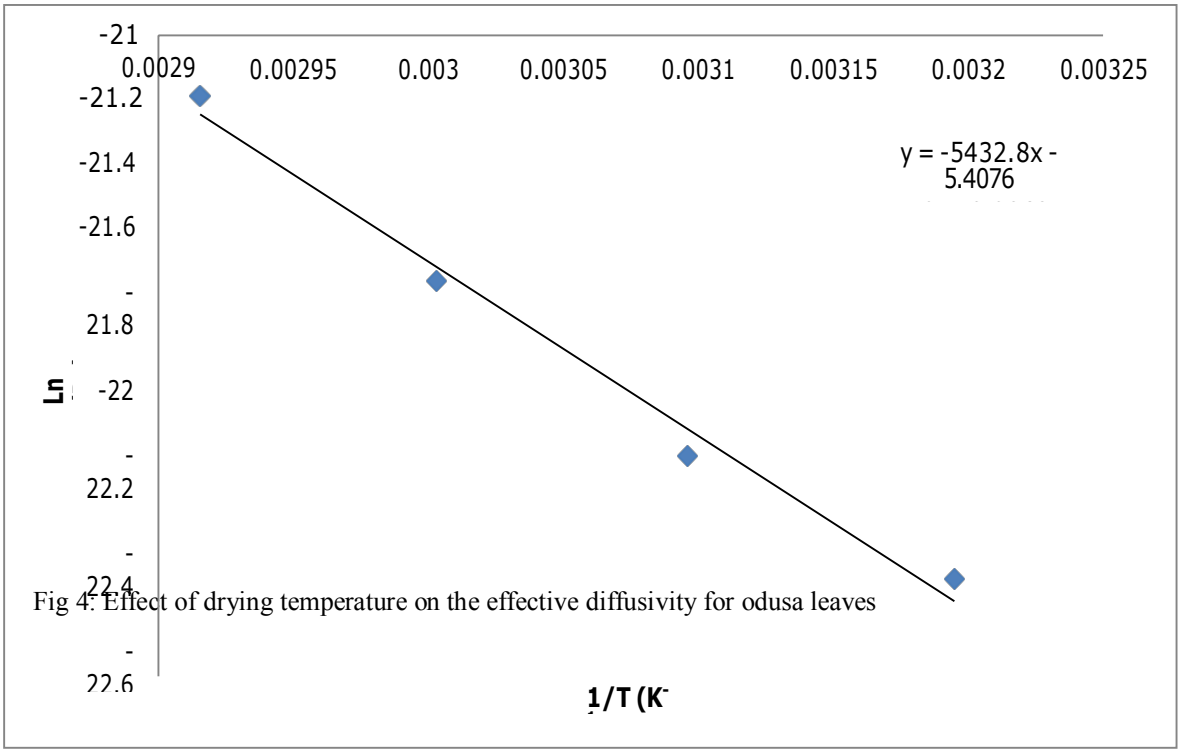

Fig 4: Effect of drying temperature on the effective diffusivity for odusa leaves

DOI: https://doi.org./10.24163/ijart/2017/6(2):7-19

Table -4: Activation Energies of Utasi and Odusa leaves at moderate drying temperature

Sample	Ea (kJ/mol)	Do (m2/s)	R2	Linear Equation
Utasi leaf	11.535kJ	1.5206 x 10-8	0.9458	y = -1387.5x - 18.002
Odusa leaf	45.168	4.48 x 10-3	0.9868	y = -5432.8x - 5.4076

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CONCLUSION

Drying characteristics and drying kinetics of utasi leaves and odusa leaves have been investigated at atemperature 40oC, 50oC, 60oC and 70oC. The results show that an increase in drying temperature decreases the drying time and drying process took place only in the falling rate period for utasi and odusa leaves. The Page model represented the thin layer drying of utasi and odusa leaves, compared to the other models used at 40oC, 50oC and 70oC while Newton model represented the thin layer drying of utasi and odusa leaves at 60oC. The values of calculated effective diffusivity ranged from 1.86 x 10-10 to
2.66 x 10-10 for utasi leaves and 1.39 x 10-10 to
6.29 x 10-10 for odusa leaves. Temperature dependence of the diffusivity coefficients was described by an Arrhenius-type relationship. The activation energy for moisture diffusion was found to be 11.535kJ/mol and 45.168kJ/mol for utasi and odusa leaves respectively. The parameters obtained could be used for designing of drying process

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