Drying characteristics and nutritional quality of Roselle (*Hibiscus sabdariffa* L.) leaves and calyces during infrared drying

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Abstract: Roselle (*Hibiscus sabdariffa* L.) is a rich medicinal plant, therefore, the effects of infrared drying on the drying characteristics, colour and nutritional quality (vitamin C and carotenoid contents) of Roselle (*Hibiscus sabdariffa* L.) leaves and calyces were investigated. Roselle leaves and calyces were subjected to infrared drying at temperature of 80°C and 110°C and infrared power of 1000W. The experimental data obtained during infrared drying were subjected to five drying models namely, Newton, Page, Wang and Singh, Henderson and Pabis, and Logarithm models. The results showed that the drying time for Roselle leaves and calyces were less than 60 and 80 min, respectively. Page model best described the drying behavior of Roselle leaves and calyces during infrared drying. The moisture diffusivity of Roselle leaves and calyces ranged from 7.49×10^{-11} to 1.16×10^{-10} m^2 s^{-1} and 6.13×10^{-10} to 6.91×10^{-10} m^2 s^{-1}, respectively. As a result of the short drying time, the vitamin C and carotenoid content of Roselle leaves and calyces were significantly preserved. However, the infrared temperature led to a high total colour change of the samples, particularly at 110°C. This work therefore showed that infrared drying is a good drying technique that could be adopted by the food industries involved in the drying of Roselle leaves and calyces.

Keywords: Infrared drying, carotenoid, moisture diffusivity, Roselle (*Hibiscus sabdariffa* L.), vitamin C.

Citation: Oladejo, A. O., M. Sanctus, A. F. Alonge, P. J. Etim, C. J. Etti, M. G. Akpan, O. M. Nkem, and R. Banjo. 2024. Drying characteristics and nutritional quality of Roselle (*Hibiscus sabdariffa* L.) leaves and calyces during infrared drying. Agricultural Engineering International: CIGR Journal, 26(4): 169-178.

1 Introduction

Roselle (*Hibiscus sabdariffa* L.) plants are very rich in bioactive compounds like anthocyanin, carotenoids, vitamin C, and minerals and have been used medicinally to treat high blood pressure, diabetes, inflammation and obesity (Ajiboye et al., 2011; Juhari et al., 2021). The red calyx can also be converted to colouring agent which can be used in the food industries (Ansari, 2013; Ismail et al., 2008). Roselle plants originated from Africa and is now widely grown in India, Sri Lanka, Thailand, Malaysia and Indonesia (Adesokan et al., 2013; Ansari, 2013) and consumed all over the world. The morphological structure of the plants shows that it is made of leaves,

Received date: 2023-12-21 **Accepted date:** 2024-10-01

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calyces, seeds, stems and roots and it has been observed that higher percentage of the bioactive compounds are located in the leaves and calyces (Ansari, 2013; Ismail et al., 2008). As a result, the leafy and calyx parts of the plants are usually consumed as tea and beverage (Adesokan et al., 2013).

Drying has been applied in the processing of Roselle leaves and calyces as tea and to also improve the shelf life of the product. Sun drying has been widely used in the processing of freshly harvested leaves and calyces. This brings a negative resultant on the hygiene, nutritional and structural quality of the sun-dried Roselle leaves and calyces (Amoasah et al., 2018; Hahn et al., 2011). Due to the low quality of the sun-dried leaves and calyces, it cannot be exported to the US and the European countries. In order to address this problem, many drying methods have been explored. Low-cost thermodynamic solar drying was developed by (Meza-Jiménez et al., 2009) to dry Roselle calyx to moisture content of 12% (wet basis) and the drying time was 4.5 h. Zaman et al. (2017) also subjected Roselle calyces to oven drying and reported that some of the physicochemical properties were preserved compared to sun-drying. Freeze-drying has also been employed in the drying of Roselle Calyces and it was reported that freeze drying preserved the structure, and nutritional content of the Roselle calyces significantly (Juhari et al., 2021). Recently, Oladejo et al., (2023) investigated the effect of ultrasound pretreatments of Roselle leaves during oven drying and found out that the ultrasound pretreated leaves had shorter drying time and better quality compared to the untreated samples.

However, there is a need to explore other drying methods that will give optimal product quality. Infrared drying is a good drying technology that has been used in the drying of some agricultural products and has yielded positive results in terms of its ease of operation, eco-friendliness, fast drying rate and product quality (Chen et al., 2017; Pawar and Pratape, 2017). Infrared is an electromagnetic wave with a wavelength in the range of 0.75 to 1000 μm. During infrared drying, the food product absorbs the infrared rays and this increases the temperature within the food thus leading to high moisture diffusivity from the food to the surrounding heated air (Chen et al., 2017; Moon et al., 2014). This enhances rapid drying of the food product (Delfiya et al., 2022). Application of infrared drying to agricultural products has increased in recent times. For example, infrared drying was used for the drying of carrot slices (Chen et al., 2017; Kocabiyik and Tezer, 2009), sea cucumber (Moon et al., 2014), banana slices (Shi et al., 2020) and rice (Wang et al., 2017).

The application of infrared drying method to the drying of Roselle leaves and calyces have not been employed by the researchers to the best of our knowledge. Therefore, the aim of this work was to investigate the possibility of using infrared radiation for the drying of Roselle leaves and calyces and its effect on the nutritional quality of the products.

2 Material and methods

2.1 Materials

The fresh leaves and calyces of Roselle plants were harvested from the research farm of the University. The Roselle leaves and calyces were rinsed inside the water to remove the dirt and were drained. The average batch mass per experiment for Roselle leaves and calyces was 5 g each. The average thickness of the Roselle leaves and calyces used for the experiment was 0.55 and 2.0 mm, respectively. The average initial moisture content of the Roselle leaves and calyces were 89.90% and 81.70% (wet basis), respectively using AOAC (2000) method.

2.2 Infrared drying

The drying of Roselle leaves and calyces took place inside a laboratory infrared dryer built in our department (Figure 1). Roselle leaves of 5 g were placed at a distance of 200 mm below the infrared source. The infrared power used for the experiment was 1000 W at infrared temperature of 80°C and 110°C, respectively. During infrared drying, the moisture content was determined at regular intervals of 5 min until constant mass was attained. The same procedure was repeated for Roselle calyces. All experiments were carried out in triplicates.

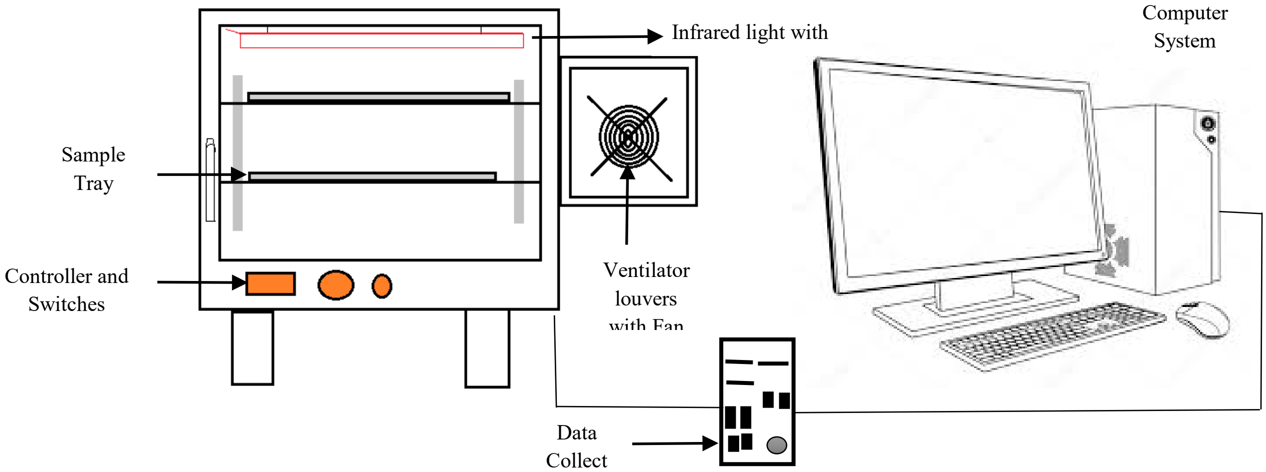

Figure 1 Schematic diagram of a laboratory infrared dryer

2.3 Effective moisture diffusivity and drying rate

To determine the effective moisture diffusivity of the Roselle leaves and calyces during infrared drying, Fick's second law of diffusion was used to analyze the experimental data:

$$MR=\frac{M_t-M_e}{M_o-M_e}=\frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp[\frac{-(2n+1)^2\pi^2D_{eff}t}{4L^2}] \quad (1)$$

Where, MR is the moisture ratio (dimensionless), M_t is the moisture content (g g^{-1}, dry basis) at any time t (min), M_o is the initial moisture content (g g^{-1}, dry basis), M_e is the equilibrium moisture content (g g^{-1}, dry basis), D_{eff} is the effective moisture diffusivity (m^2 s^{-1}) and L is the half-thickness of an infinite slab-shaped sample (m), n is an integer number from 0 to infinity corresponding to terms in the equation.

As a result of long drying time, Equation (1) becomes:

$$Ln(MR)=Ln(\frac{8}{\pi^2})-(\frac{\pi^2D_{eff}}{4L^2}t) \quad (2)$$

The moisture diffusivity was then determined from the slope of graph of Ln(MR) versus drying time:

$$Slope=\frac{\pi^2D_{eff}}{4L^2} \quad (3)$$

The drying rate was determined by using:

$$DR=\frac{M_t-M_{t+\Delta t}}{\Delta t} \quad (4)$$

Where, M_t and $M_{\Delta t}$ are the moisture content at time t and t+Δt, respectively.

2.4 Drying models

The experimental data obtained during infrared drying were subject to five drying models (Table 1) namely, Newton, Page, Wang and Singh, Henderson and Pabis, and Logarithm models. The models were validated using coefficient of determination (R^2), reduced chi-square (X^2 X^2), sum square error (*SSE*) and root mean square error (*RMSE*):

Coefficient of determination:

$$R^2=\frac{\sum_{i=1}^{N}(MR_{exp}-MR_{pre})^2}{\sum_{i=1}^{N}(MR_{pre}-MR_{exp})^2} \quad (5)$$

Reduced chi-square:

$$X_c^2=\frac{\sum_{i=1}^{\bar{N}}(MR_{exp}-MR_{pre})^2}{\bar{N}-Z} \quad (6)$$

Sum of square error:

$$SSE=\frac{1}{N}\sum_{i=1}^{N}(MR_{pre,i}-MR_{exp,i})^2 \quad (7)$$

Root mean square error:

$$RMSE=[\frac{1}{\bar{N}}\sum_{i=1}^{\bar{N}}(MR_{exp}-MR_{pre})^2]^{\frac{1}{2}} \quad (8)$$

Where, MR_{exp} is the experimental values, MR_{pre} is the predicted values, $\bar{N}$ is the number of observations, Z is the number of constants and X_c^2 is the Chi-square. The best model selected was based on the highest R^2; and the least X_c^2, SSE and RMSE values.

2.5 Colour determination

The colour measurement of the fresh and dried Roselle leaves and calyces was carried out using a colorimeter (CS-10, CHNSPEC Technology Co. Ltd, Zhejiang, China). The values of L^* (degree of lightness), a^* (degree of darkness) and b^* (degree of yellowness) of the dried samples were determined from the colorimeter. The total colour difference *ΔE* was determined using:

$$\Delta E = \sqrt{(L_0 - L^*)^2 + (a_0 - a^*)^2 + (b_0 - b^*)^2} \quad (9)$$

The colour parameters with the nought subscript (L_0, a_0 and b_0) represent the colour values of the fresh Roselle leaves/calyces.

Table 1 Thin layer mathematical models for drying

S/N	Model Name	Model	References
1	Newton Model	*MR=exp(-kt)*	(Lopez-Quirogaet et al., 2020)
2	Page Model	*MR=exp(-kt^n)*	(Onwude et al., 2018)
3	Wang and Singh	*MR=1+αt+bt²*	(Chen et al., 2020)
4	Henderson and Pabis Model	*MR=α×exp(-kt)+c*	(Szadzińska et al., 2020)
5	Logarithm	*MR=α×exp(-kt)+c*	(Aradwad et al., 2022)

Note: a, b, c and k are model constants.

2.6 Determination of carotenoid content

The carotenoid of the fresh and dried Roselle leaves and calyces were determined according to the method of Rodriguez-Amaya and Kimura (2004). Then, the carotenoid content was calculated using:

$$Total\ carotenoid\ content\ \left(\mu g\ g^{-1}\right) = \frac{A \times V \times 10^4}{A_{1cm}^{1\%} \times P} \quad (10)$$

where, *A* is the absorbance, *V* is the volume of the extract (mL). $A_{1cm}^{1\%} = 2592$ (Beta carotene absorption coefficient in petroleum ether); *P* = sample weight (g).

2.7 Determination of vitamin C

The modified method of Satpathy et al. (2021) was used for the measurement of vitamin C in the fresh and dried Roselle leaves and calyces.

2.8 Statistical analysis

Non-linear regression, analysis of variance (ANOVA) and comparison of means by Tukey's Posthoc test were carried out using statistical software, SPSS version 20 (SPSS Inc., Chicago, Ill., U.S.A.) and the graphing was done using Microsoft Excel (2013) software.

3 Results and discussion

3.1 Effects of infrared drying on the drying kinetics of Roselle calyces and leaves

The moisture ratio of Roselle calyces and leaves dried at 80°C and 110°C are shown in Figure 2. The moisture ratio shows that generally for all samples, the drying process followed a falling rate period. This means that the drying of the samples was diffusion controlled. It is observed that the Roselle leaves dried faster than the calyces. This may be due to the fact that Roselle leaves are thinner (0.5 mm) than the calyces (2.0 mm). It has been reported that the size (thickness) of a sample affects the moisture ratio during drying (Correia et al., 2015). The thinner a sample is, the faster the drying process (Onwude et al., 2016). Figure 2 also shows that the samples that had the shortest drying time were Roselle leaves dried at 110°C, and longest drying time was Roselle calyces dried at 80°C. This observation also confirms that apart from the size of a sample, temperature also affects the moisture ratio of the sample during drying (Moon et al., 2014; Shi et al., 2020). Higher temperature leads to increased movement of moisture leaving the samples to the heated surrounding area. Liu et al. (2015) similarly reported a decrease in the moisture content of Flos Lonicerae as the far infrared temperature increased when it was subjected to infrared drying.

The drying rates versus the moisture ratio of infrared dried samples are shown in Figure 3. It is shown that at the initial moisture content of all the samples and in the first few minutes of drying, there was a sharp rise in the drying rate. This could be due to the surface moisture on the samples which evaporated unhindered as the drying began. Similar observation was reported for apple slices (Aradwad et al., 2022), carrot slices (Kocabiyik and Tezer, 2009). However, as the drying progressed, there was a fall in the drying rates of all the samples. This was attributed to the increased internal resistance to the movement of moisture as the structure of the samples began to collapse, shrink and formation of crust on the surface of the samples. The effects of temperature on the drying rates can also be seen in Figure 3. Roselle leaves and calyces dried at 80ºC had a lower drying rate compared to those dried at 110ºC. The high temperature gradient caused by the high radiation temperature led to increased internal heating of moisture thus resulting in an accelerated movement of moisture out of the samples (Chen et al., 2017).

3.2 Effects of infrared drying on the effective moisture diffusivity of Roselle calyces and leaves

Table 2 shows the values of the effective moisture diffusivity of Roselle calyces and leaves. The values obtained in this work for Roselle calyces were in the range reported by (Tajudin et al., 2019) and likewise the effective moisture diffusivity of Roselle leaves obtained in this work were similar to the values reported by Oladejo et al. (2023). As shown in Table 2, the Roselle calyces had higher effective moisture diffusivity than the Roselle leaves at 80ºC and 110ºC. This may be due to the fact that the structure of the calyces was more porous than that of the leaves, thus allowing faster movement of moisture from the calyces. Another reason could be that the Roselle leaves got dried faster leading to crust formation which slowed down the moisture movement out of the leaves as the drying progressed. The effect of temperature can also be observed from Table 2. Higher radiation temperature led to increased moisture diffusivity for both Roselle calyces and leaves.

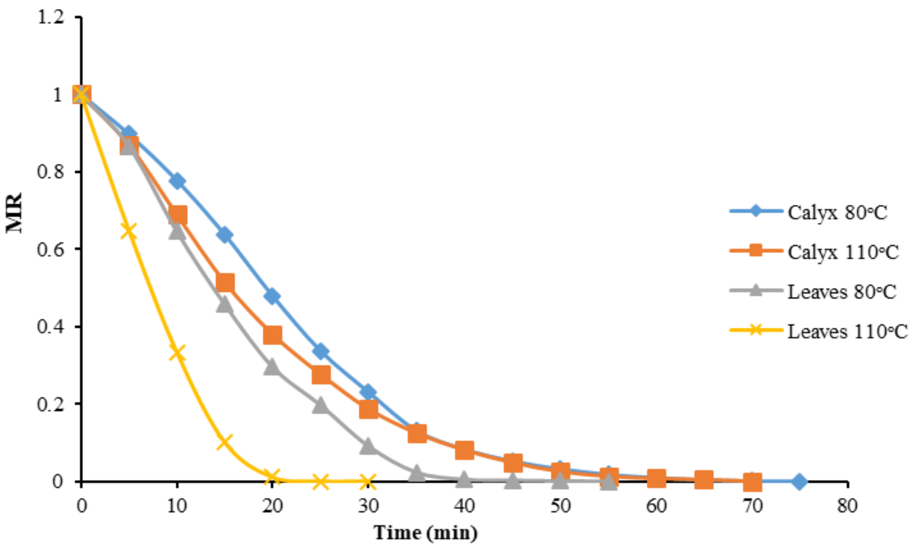

Figure 2 Moisture ratio (MR) of infrared dried Roselle calyces and leaves

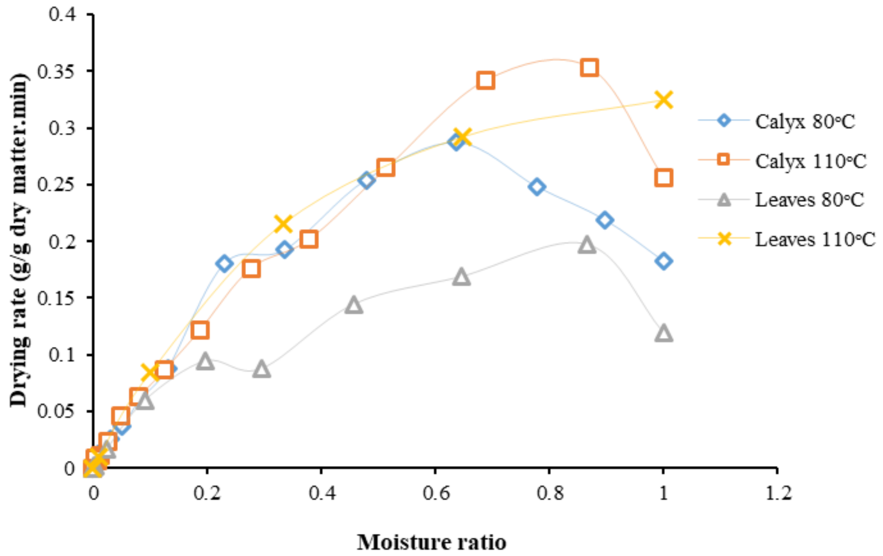

Figure 3 Drying rates of infrared dried Roselle calyces and leaves

Table 2 Effect of infrared drying on the moisture diffusivity of Roselle calyces and leaves

Samples	Moisture diffusivity (m^2s^{-1})	R^2
Roselle calyces dried at 80°C	6.13×10^{-10}	0.9124
Roselle calyces dried at 110°C	6.91×10^{-10}	0.9069
Roselle leaves dried at 80°C	7.49×10^{-11}	0.9116
Roselle leaves dried at 110°C	1.16×10^{-10}	0.8959

The higher radiation temperature penetrated faster and deeper on the samples than the lower temperature, and this led to an increase in the movement of molecules trying to escape from the samples. Similar observation was reported by Ashtiani et al. (2017), when they investigated the drying behavior of peppermint leaves subjected to hot air and infrared drying.

3.3 Drying models for Roselle leaves and calyces subjected to infrared drying

Tables 3 and 4 showed the results of the drying modelling describing the behavior of Roselle leaves and calyces during infrared drying. The best model was selected based on the highest R^2, lowest X^2 and lowest *RMSE*. In this work, page model was found to be most suitable for describing the drying behavior of Roselle leaves and calyces during infrared drying. Similarly, page models have been found to describe the infrared drying behavior of carrot slices (Chen et al., 2017), onion slices (Sharma et al., 2005) and paddy (Das et al., 2009).

3.4 Effects of infrared drying on the vitamin C and carotenoid content of Roselle leaves

Table 5 contains information about the nutritional values of fresh and treated Roselle leaves. The average value of vitamin C of the fresh Roselle leaves was 10.0388 mg/100g. This value was close to the value reported by Oladejo et al. (2023) in a previous experiment. It is shown in Table 5 that the Vitamin C contents of Roselle leaves subjected to infrared drying had a higher vitamin C value than the fresh samples. Particularly the Roselle leaves dried at 80°C had significantly higher amount of vitamin C than the fresh samples. This could be due to the fact that the Roselle leaves were subjected to a high temperature but short drying time. The short drying time helped to retain the vitamin C contents from getting lost. Fang et al. (2009) reported an increase in the amount of vitamin C of Chinese jujube when drying temperature increased from 50°C to 70°C. Similar trends were observed for the carotenoid content of Roselle leaves, where the samples dried at 80°C and 110°C were higher than the fresh samples.

3.5 Effects of infrared drying on the vitamin C and carotenoid content of Roselle calyces

The values of vitamin C and carotenoid content for fresh and dried Roselle leaves and calyces are shown in Table 6. The values of vitamin C obtained in this work were close to the values reported by Ansari (2013) and Tajudin et al. (2019). Although there was no significant difference between the fresh and dried samples, the samples subjected to the infrared drying at 80°C and 110°C showed higher values than the fresh samples. This implies that infrared drying of Roselle calyces at higher temperature and short time can help to retain the vitamin C of the Roselle calyces. The decreased moisture content in the calyces during infrared drying led to an increase in the concentration of vitamin C.

Table 6 showed that there was no significant difference ($p>0.05$) between the carotenoid content of the fresh Roselle calyces and dried Roselle calyces. This showed that infrared drying of Roselle calyces did not lead to significant destruction of carotenoid content. It was also observed by Tayyab et al. (2022) that there was an increase in the amount of total carotenoid of sweet potato slices compared with the fresh samples as the temperature increased from 60°C to 80°C during infrared drying. The short drying period favored high retention of carotenoid during infrared drying.

Table 3 Parameters and regression coefficient of drying models applied to the drying kinetics of Roselle leaves

Infrared temperature (℃)	Model name	Coefficient of determination (R^2)	Reduced chi-square (X^2)	Sum square error (*SSE*)	Root mean square error (*RSME*)
80	Newton	0.971	0.0053	0.0049	0.0699662
	Logarithm	0.971	0.0020	0.0018	0.0427137
	Henderson and Pabis	0.971	0.0042	0.0038	0.0619437
	Page	0.971	0.0002	0.0002	0.0133176
	Wang and Singh	0.971	0.0008	0.0007	0.0268898
110	Newton	0.971	0.0041	0.0035	0.0596
	Logarithm	0.971	0.0019	0.0017	0.0406
	Henderson and Pabis	0.971	0.0038	0.0033	0.05741
	Page	0.971	0.0003	0.0002	0.01536
	Wang and Singh	0.971	0.0004	0.0004	0.01909

Table 4 Parameters and regression coefficient of drying models applied to the drying kinetics of Roselle calyces

Infrared temperature (°C)	Model name	Coefficient of determination (R^2)	Reduced chi-square (X^2)	Sum square error (*SSE*)	Root mean square error (*RSME*)
80	Newton	0.971	0.0131	0.0123	0.1108935
	Logarithm	0.971	0.0025	0.0023	0.0482447
	Henderson and Pabis	0.971	0.0045	0.0042	0.064788
	Page	0.971	0.0001	0.0001	0.0099687
	Wang and Singh	0.971	0.0014	0.0013	0.0356796
110	Newton	0.971	0.0026	0.0024	0.0489002
	Logarithm	0.971	0.0009	0.0008	0.283355
	Henderson and Pabis	0.971	0.0018	0.0017	0.0415048
	Page	0.971	0.0000	0.0000	0.045088
	Wang and Singh	0.971	0.0006	0.0006	0.0236941

Table 5 Vitamin C and carotenoid content of Roselle leaves dried with infrared dryer at 80°C and 110°C

Samples	Vitamin C (mg 100g^{-1})	Carotenoid (µg^{-1}) (µ g^{-1})
Fresh Roselle leaves	10.0388±3.84^{a}	87.0628±0.06^{a}
Roselle leaves dried at 80°C	18.9639±3.28^{b}	89.0561±0.06^{b}
Roselle leaves dried at 110°C	18.2578±5.44^{ab}	88.9918±0.06^{b}

Note: Means± standard deviation. Means with different superscripts on the same column are significantly different (p<0.05).

Table 6 Vitamin C and carotenoid content of Roselle calyces dried with infrared dryer at 80°C and 110°C

Samples	Vitamin C (mg 100g^{-1})	Carotenoid (µg^{-1})
Fresh Roselle calyx	12.69±7.38^{a}	31.44±0.45^{a}
Roselle calyx dried at 80°C	16.79±2.20^{a}	33.50±0.50^{a}
Roselle calyx dried at 110°C	22.84±5.96^{a}	33.78±2.11^{a}

Note: Means± standard deviation. Means with different superscripts on the same column are significantly different (p<0.05).

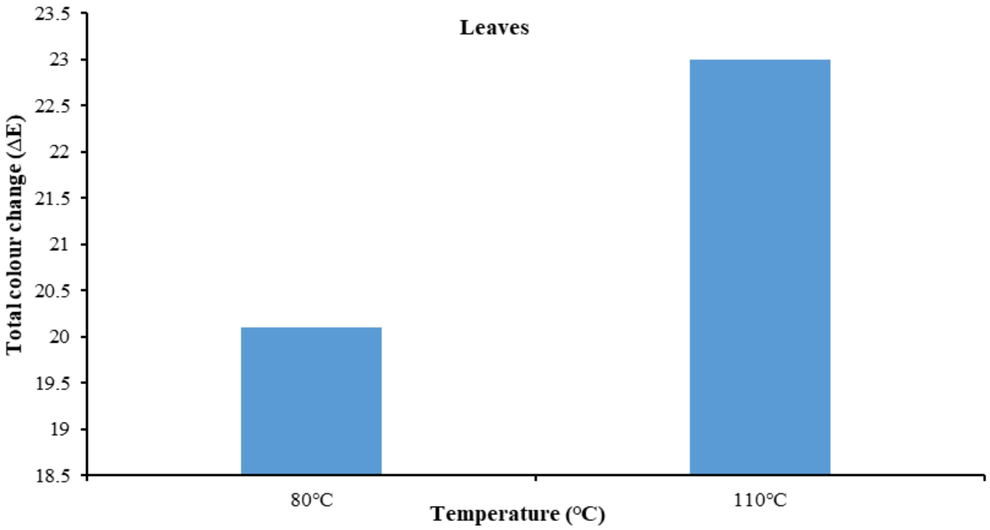

Figure 4 Total colour change of Roselle leaves as affected by infrared drying temperature

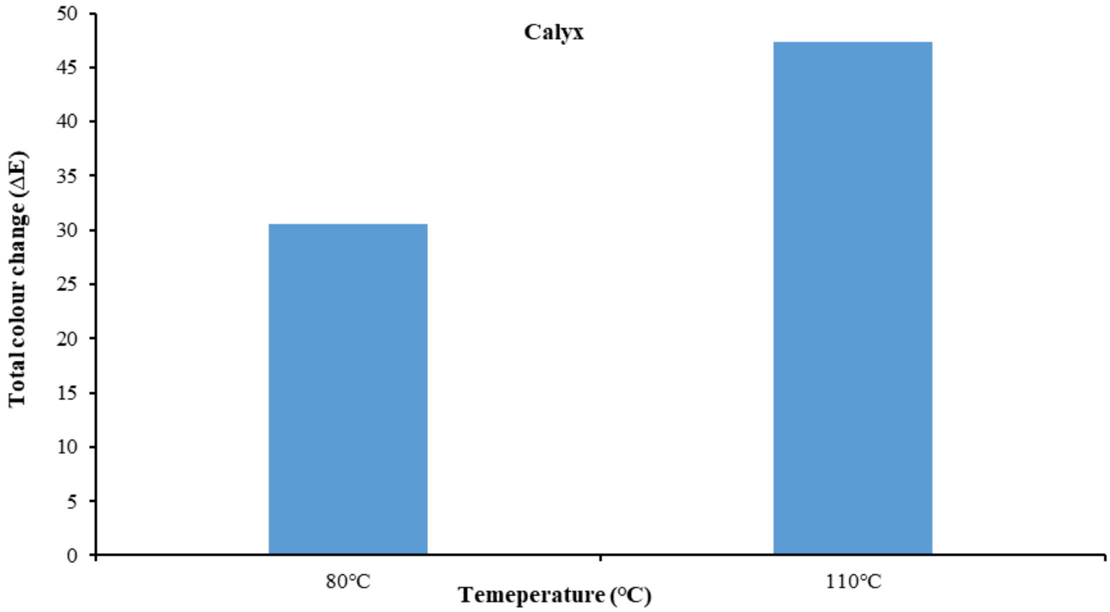

Figure 5 Total colour change of Roselle calyces as affected by infrared drying temperature

3.6 Effects of infrared drying on the colour of Roselle leaves and calyces

Figures 4 and 5 showed the effects of infrared drying temperature on the total colour change of Roselle leaves and calyces, respectively. It was very clear for both the Roselle leaves and calyces that temperature had a significant effect on the total colour change of the samples. The higher the temperature, the greater the total colour change of the samples. High Infrared drying temperature could lead to degradation of colour of Roselle leaves and calyces as a result of enzymatic and non-enzymatic browning that occurred during drying. Ning and Han (2013) also reported an increase in total colour change of Taegeuk ginseng (*Panax ginseng* C.A. Meyer) when the temperature increased from 45°C to 65°C during infrared drying.

4 Conclusion

Investigation into the applicability of infrared drying of Roselle leaves and calyces was carried out in this work at infrared temperatures of 80°C and 110°C and infrared power of 1000 W. The drying time for Roselle leaves and calyces were less than 60 and 80 min, respectively. The moisture diffusivity of the Roselle calyces was found to be higher than that of Roselle leaves. Furthermore, infrared drying of Roselle leaves and calyces were found to have positive effects on vitamin C and carotenoid content of Roselle leaves and calyces. But infrared drying of both Roselle leaves and calyces showed higher total colour change at temperature of 110°C.

Therefore, this work showed that infrared drying of Roselle leaves and calyces at temperature of 80°C is recommended for short drying time and retention of nutrients.

Acknowledgement

This work was supported by the Nigeria TETFund National Research Fund (NRF) Intervention (TETFund/DR&D/CE/NRF/STI/14/VOL1).

Conflict of Interest

The authors declare no conflict of interests.

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