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Research Article

Kinetics of β-Carotene Breakdown and Moisture Sorption Behavior of Yellow Cassava Flour during Storage

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β-Carotene is an important plant pigment with high vitamin A activity. The kinetics of β-carotene degradation and moisture sorption behavior of yellow cassava flour produced by different drying techniques was investigated during storage. The β-carotene degradation kinetics were described using a first-order kinetic model, while the moisture adsorption data was fitted to five mathematical equations using nonlinear regression. During storage, the reaction rate constant for β-carotene degradation, which increased with increasing temperature, ranged from 0.0045 to 0.0396, 0.0029 to 0.0309, and 0.0025 to 0.0349 per month for flour produced by solar drying, hot air oven drying, and drum drying, respectively. Flour produced by solar drying had the highest activation energy of 124.2 kJ/mol, whereas drum drying had the lowest activation energy of 85.8 kJ/mol. All the yellow cassava flours showed a type II sigmoidal sorption curve in which the equilibrium moisture content increased with increasing water activity. The GAB model was the best for describing the moisture sorption behavior of the product, with a predicted monolayer moisture ranging from 2.8 (for drum drying) to 8.0 g water/100 g of flour (for solar drying). Findings from this study are vital for establishing the packaging and storage requirements of yellow cassava flour.

1. Introduction

Carotenoids are a group of compounds responsible for the red-orange-yellow pigmentation in plants. These compounds have nutritional and health significance, as their consumption is known to be beneficial. Among this group of plant pigments, β-carotene has the highest vitamin A activity, and its nutritional and health benefits include the prevention of night blindness and chronic diseases, and maintaining immunity against diseases [1]. It is also used as a food colorant, and its antioxidant potential is leveraged in preventing rancidity in food oils [2]. However, its highly unsaturated structure makes it sensitive to thermal stress as it undergoes oxidation and/or isomerization in the presence of heat, light, and oxygen [3]. Ultimately, these factors influence the conformation and stability of β-carotene in food.

Cassava is an indispensable food crop which provides nourishment for thousands of people globally. Due to its low level of important micronutrients, yellow cultivars containing significant levels of carotenoids have been developed through breeding [4]. Consumption of yellow cassava has shown promising results in improving the vitamin A status of children in sub-Saharan Africa [5]. Cassava flour is a semi-finished product and an important starting material for processing a diversity of cassava-based products. A critical consideration in the use of yellow cassava flour relates to its β-carotene stability during processing and storage [6, 7]. The loss of β-carotene in yellow cassava flour may lead to defects in color and aroma and ultimately reduce its nutritional benefits.

The degradation of β-carotene in food may be described by kinetic studies, which provide useful information for predicting and controlling the rate of degradation and changes in product quality. Several studies describing the kinetics of carotenoids breakdown during processing or storage have been documented for different commodities [8–12]. Many of these studies concluded that the breakdown of β-carotene follows a first-order kinetic reaction ([13–15]; Bollinedi et al., 2022). Previous studies involving β-carotene-rich cassava revealed the dependence of carotenoid loss on processing technique, the final product, and packaging or storage conditions [16–21]. For example, in Eyinla et al. [16], a retention of 10–29% was reported for *gari* from yellow cassava fermented from one to three days, while Chavez et al. [17] recorded 38-72% retention after drying and 18-32% after four weeks of storing oven- and solar-dried yellow cassava. These studies mainly focused on only carotenoid retention or breakdown during processing without examining the kinetics associated with its degradation during storage of these products.

Moisture content and water activity are essential in determining the storage quality of food. During storage, the dynamics of moisture affect the physical and biochemical changes that occur. Indeed, the influence of moisture on reactions is quite complicated, with somewhat contrasting outcomes. For instance, whereas moisture enhances the mobility of reactants and intensifies oxidation, it may also dilute enzymes and catalysts and slow down these reactions. Moisture sorption isotherm relates the equilibrium moisture content of food to its water activity. This is a useful thermodynamic tool for predicting the relative stability of food products and establishing packaging needs and storage conditions.

Given that carotenoids are prone to degradation on exposure to oxygen, heat, and light, it is reasonable to anticipate significant losses during storage of yellow cassava flour. Ekpa et al. [22] suggest that packaging material and storage conditions directly affect the stability of β-carotene in products during storage. Elucidating the moisture sorption behavior and stability of β-carotene during storage is of nutritional and commercial interest, since this would inform the selection of appropriate packaging, storage, and handling conditions [6]. Therefore, the objective of this study was to evaluate the moisture sorption and degradation kinetics of β-carotene during storage of yellow cassava flour.

2. Materials and Methods

2.1. Processing and Sample Preparation. Yellow-fleshed cassava was obtained from demonstration plots of the CSIR-SARI and transported to the laboratory for processing. The cassava roots were washed and manually peeled with a stainless steel knife. The parenchyma was washed again before being sliced (3 mm) with a mechanical slicer. The slices were steam-blanched in a pressure cooker at 110°C for 5 min at 0.1 bar.

2.2. Drying. The steam-blanched samples were allowed to cool to room temperature before spreading in a single layer on stainless steel drying trays and loaded into a tunnel solar dryer. The average drying temperature in the dryer was 52°C. For air-oven drying, thinly spread samples were dried at 65°C after heating the dryer (Gallenkamp Hot Box 2, Gallenkamp, Sanyo/Weiss, UK) to attain a steady temperature. At the end of the drying process, the slices were allowed to cool to room temperature before milling into flour (300 μm) with a laboratory mill (Waring 8420, Torrington, USA). The flour was sealed in transparent polypropylene pouches and held at room temperature (RH 75%).

The steam-blanched samples were mashed, while still hot, before drum drying on a pilot-scale single drum dryer (ANDRITZ Gouda, The Netherlands), equipped with four applicator rollers. The drum was preheated to stabilize the temperature at 120°C before introducing the yellow cassava mash to the drum surface while rotating at 10 rpm. After traveling about ¾ of the drum's circumference, the dried yellow cassava flakes were scraped off with the doctor's blade and place in a stainless-steel collector. The dry flakes were allowed to cool to room temperature, milled, and sealed in polypropylene pouches. The drying conditions for air oven and drum drying were based on an initial optimization step (data not shown) to ensure higher carotene retention.

2.3. Flour Storage Studies

2.3.1. Sample Storage. Yellow cassava flour (250 g) made from either of the three drying techniques was packaged into clear, flexible polypropylene pouches. Samples were sealed with an impulse sealer and stored at 25, 35, and 45°C for three months in a climate control chamber (Memmert ICH 260 C, Memmert GmbH, Germany) at a relative humidity of 75%. Samples were drawn fortnightly for the first month, and subsequently, monthly for the rest of the storage period for β-carotene analysis.

2.3.2. Determination of β-Carotene. The β-carotene content of the yellow cassava flour was determined by the method of Dutta et al. [9] with some modifications. Five grams of sample was suspended in a mixture of 15 mL isopropyl alcohol and 5 mL hexane and stirred for 1 min. The volume of the mixture was adjusted with distilled water in a 125-mL amber separation funnel and allowed to stand for 30 min before filtering (Whatman, No. 4). The filtrate was transferred into an amber volumetric flask from which aliquots were manually dispensed into a cuvette, and its absorbance was determined at 450 nm with a UV-Vis spectrophotometer (T80, PG Instruments, Leicestershire, UK). A standard curve was plotted using a β-carotene standard (>95% pure, Sigma Aldrich), from which the concentration of β-carotene was determined and used to quantify the amount of β-carotene in the sample. Retention was expressed as a percentage of the ratio of the β-carotene content after drying to the β-carotene content before drying [23].

2.3.3. β-Carotene Degradation Kinetics. β-Carotene degradation in food has been shown to follow a first-order kinetic reaction [10, 24] of the linear form

$$\ln(C/C_0) = -kt, \quad (1)$$

where C is the β-carotene level at time t. C_o is the amount of β-carotene at time zero. k is a rate constant and t is the storage period (months) [12]. The Arrhenius equation was used to illustrate the dependence of β-carotene breakdown on temperature as

$$k = k_0 \exp\left(-\mathrm{E}/RT\right), \tag{2}$$

where k is the reaction rate constant. k_o is frequency factor (min^{-1}). E is the activation energy (kJ/mol). R is the universal gas constant (8.314 J/mol.K) and T is the absolute temperature (K).

The effect of increasing temperature on the reaction rate was assessed using Q_{10}

$$Q_{10} = \left(\frac{k_2}{k_1}\right)^{10/(T_2-T_1)}, \tag{3}$$

where k_1 and k_2 are the reaction rate constants at two successive temperatures, T_1 and T_2 (°C), respectively.

2.3.4. Effect of Packaging Material on β-Carotene Content. To study the impact of packaging material on the breakdown of β-carotene in the yellow cassava flour during storage, 250 g of drum-, hot air-, or solar-dried flour was packaged in either a polypropylene bag (76.6% light transmittance) or a paper-polyethylene laminate (0.1% light transmittance), sealed and monitored for 8 months at room temperature (28°C, RH of 75%). On a monthly basis, samples were analyzed for β-carotene.

2.3.5. Moisture Sorption Behavior of Yellow Cassava Flour. The equilibrium moisture content (EMC) of the samples was determined following the gravimetric method described by Rosa et al. [25] with slight modifications. The experiments were setup in seven hermetically closed jars, each filled to a quarter of its height with a given concentration (Table 1) of sulfuric acid solution to maintain a water activity of 0.1 to 0.9. The water activity of the acid solutions was verified using an electronic water activity meter (Rotronic HygroLab, Bassersdrof, Germany). One gram of yellow cassava flour sample was weighed into a plastic weighing boat and carefully hung from a cotton thread from the cap of the jar to an appreciable height above the acid solution to prevent contact. The weight of the flour samples was measured at three-day intervals until a constant weight was attained. At the point of equilibrium, the moisture content, expressed as g H_20/100 g of flour, was determined as the equilibrium moisture content.

2.4. Statistical Analysis

2.4.1. Modeling Sorption Data. The water sorption data were fit to the linear forms of five mathematical models (Table 2) using nonlinear regression (Statgraphics Centurion 19.1). These models are relatively simple, applicable to a wide range of water activity at specific temperatures, and have proved adequate in characterizing the moisture sorption behavior of many foods [27]. The goodness of fit of each model was evaluated using the coefficient of determination (R^2) and root mean square error (RMSE). Models with a high R^2, and low RMSE were considered to better describe the experimental data.

Table 1: Water activity of sulfuric acid solution at 30°C [26].

Concentration (%)	Water activity
15	0.9245
25	0.8252
35	0.6693
40	0.5711
50	0.3574
55	0.2563
65	0.0972

Table 2: Moisture sorption isotherm models.

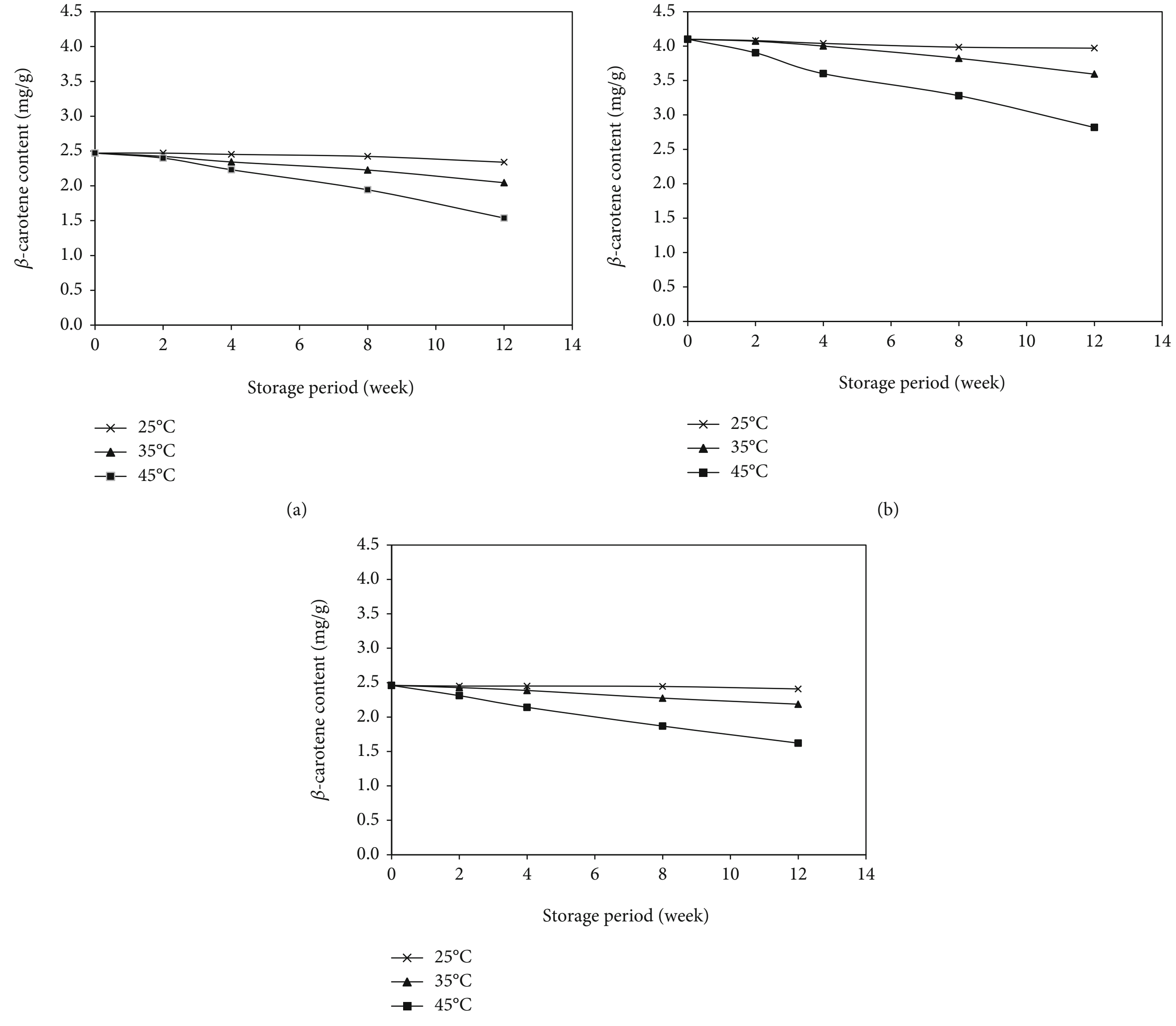
Model	Expression
GAB	$M = \frac{X_m \mathrm{Ck} a_w}{(1 - k a_w)(1 - k a_w + \mathrm{Ck} a_w)}$
BET	$M = \frac{X_m C a_w}{(1 - a_w)(1 + a_w(C - 1))}$
Oswin	$M = \frac{a \times a_w}{(1 - a_w)^b}$
Smith	$M = A + \mathrm{B}\log(1 - a_w)$
Iglesias & Chirife	$M = A + B\left(\frac{a_w}{1 - a_w}\right)$

where M is the equilibrium moisture content. X_m is the monolayer moisture content. C is the constant related to the heat of sorption of the monolayer. k is the constant related to the total heat of sorption. A, a, and B are the model constants.

3. Results and Discussion

3.1. Kinetics of β-Carotene Degradation during Storage of Yellow Cassava Flour. Results of the study showed a gradual reduction in β-carotene levels during storage, irrespective of storage temperature. However, higher temperature caused faster breakdown in flour β-carotene levels (Figure 1), similar to observations in previous studies [10]. The results emphasize the need to store pigmented flours at lower temperatures to reduce the rate of carotenoid degradation, as suggested by the findings of previous studies [22, 28]. Indeed, storage at -20°C has been documented to virtually stop the degradation of carotenoids in biofortified maize flour [29].

The β-carotene degradation during storage of the flours was described using the first-order kinetic model, which showed adequate fit with an R^2 ranging from 0.8965 to 0.9997 (Table 3). The β-carotene breakdown was influenced by temperature, as reflected in the k values, which increased with increasing storage temperature from 25 to 45°C. At all the storage temperatures, comparatively higher k values were observed for drum-dried samples, indicating a higher degradation rate among these flours compared to flours processed by the other drying methods. Two reasons related to the flour's structural characteristics may account for this observation. Firstly, the particles of drum-dried flours were



(a)

(b)

(c)

Figure 1: Breakdown of β-carotene in (a) drum-, (b) hot air oven-, and (c) solar-dried yellow cassava flour at different storage temperatures.

Table 3: Kinetic parameters of β-carotene degradation during storage of drum-, hot air-, and solar–dried yellow cassava flour.

Sample	Temperature (°C)	k (month^{-1})	R^2	Q_{10}	E_a (kJ/mol)
Drum	25	0.0045	0.8965	3.47	85.78
	35	0.0156	0.9848	2.54	
	45	0.0396	0.9683		
Hot air	25	0.0029	0.9462	3.86	93.32
	35	0.0112	0.9699	2.76	
	45	0.0309	0.9927		
Solar	25	0.0025	0.9347	4.04	124.21
	35	0.0101	0.9936	3.46	
	45	0.0349	0.9997		

k is the reaction rate constant. R^2 is the coefficient of regression. Q_{10} is the temperature coefficient. E_a is the activation energy.

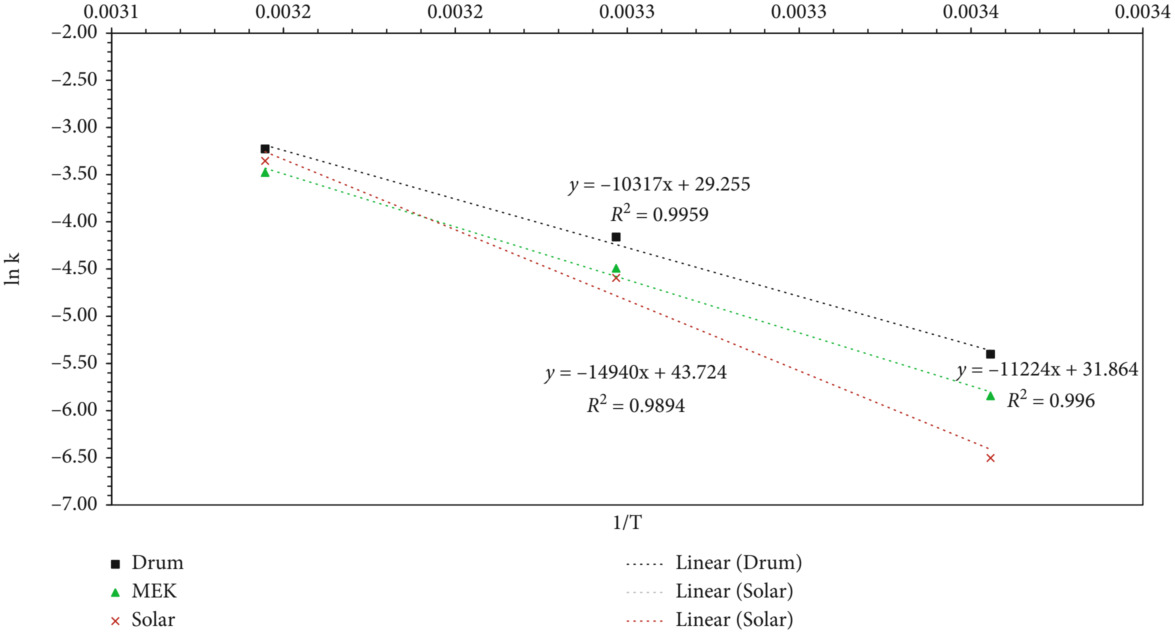


Figure 2: β-carotene degradation rate constant as a function of temperature.

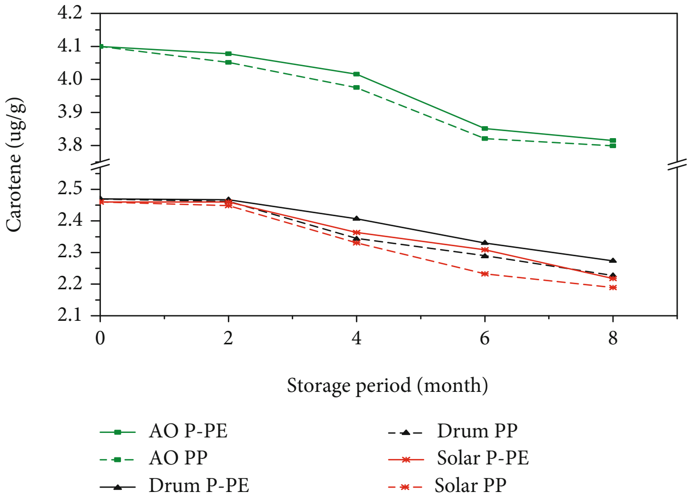


Figure 3: β-Carotene breakdown in yellow cassava flour as a function of time (months) and packaging material at room temperature.

finer (results not shown) compared to flours dried by the other methods, therefore exposing a wider area to degradation. Secondly, the porous nature of drum-dried flours enhanced its contact with oxygen, while the bulkier and apparently compact nature of flours dried by hot air oven or solar may have limited the extent to which they interacted with oxygen [24].

In accordance with the Arrhenius equation, for a 10 °C rise in storage temperature, the rate of β-carotene breakdown in cassava flour increased markedly. For instance, increasing the storage temperature from 25 to 35°C caused a more than a threefold increase in the breakdown rate. However, an increase from 35 to 45°C recorded lower Q_{10} values (<3). Moreover, higher Q_{10} values were recorded in solar-dried flours compared to the others. The reason for this may not be readily ascertained. However, it is possible that degradation of the yellow color pigment may have already been initiated or even advanced because of excessive exposure to sunlight and oxygen during solar drying. The activation energy for β-carotene breakdown during storage ranged between about 86 and 124 kJ/mol for yellow cassava flour produced by drum drying and solar drying (Table 3). The higher activation energy in drum-dried samples suggests these samples are the most sensitive to changes in temperature during storage [6]. The flat geometry of drum-dried flours may expose a wider surface area for β-carotene degradation compared to the other flours which were largely composed of spherical particles. The activation energy for β-carotene breakdown obtained in this study were comparable to Bechoff et al. [6], who reported a reaction rate of 0.0093–

0.0405 day^{-1} for the degradation of β-carotene in OFSP at a storage temperature of 20 to 40°C.

Figure 2 presents the effect of temperature on the degradation rate constant (ln k). The coefficients of determination were greater than 0.98, an indication that the Arrhenius equation was adequate in predicting the dependence of the degradation of cassava flour β-carotene levels on storage temperature.

3.2. Impact of Storage Material on β-Carotene Loss during Storage. Studies have shown that β-carotene is degraded during the storage of carotenoid-rich products [6, 22]. These losses may be influenced by several factors, including temperature and storage conditions. The impact of different packaging materials, polypropylene and paper-polyethylene laminate, on the β-carotene content of the flours in storage was investigated. At the start of storage, β-carotene is ranged from 2.46 mg/g for solar dried flours to 4.1 mg/g for hot air-dried flours. During storage at room temperature (28°C), all the flour samples, regardless of drying method or storage material, showed a gradual decrease in β-carotene levels (Figure 3). The decrease was steeper after "month 2" of storage. At the end of the storage period, β-carotene ranged from 2.19 to 3.81 mg/g for solar-dried flours in polypropylene and hot air oven-dried flours in paper-polyethylene laminates.

There was a comparatively lower β-carotene loss in flours stored in the paper-polyethylene laminate compared to the polypropylene material. For instance, at the end of the storage period, an 11.0% reduction was recorded in solar-dried flour packaged in polypropylene pouches compared to 9.8% for the same flour packaged in paper-polyethylene laminate (Figure 4). This was anticipated since the polypropylene pouches were transparent ($T = 76.6\%$). On the other hand, the paper-polyethylene laminate, being an opaque laminate of brown paper and polyethylene ($T = 0.1\%$), may have reduced the incidence of light on β-carotene breakdown. Additionally, the laminate most likely provided better air barrier property compared to the polypropylene material. This suggests a better retention ability of paper-polyethylene laminates compared to transparent polypropylene pouches as packaging material options for yellow cassava flour.

Oxygen, temperature, and light are critical factors influencing the degradation of pigments in food. That notwithstanding, Bechoff et al. [6] suggest that oxygen has a greater impact than light. Ekpa et al. [22] explained that together with temperature, the presence of oxygen may trigger the autooxidation of β-carotene, leading to considerable losses during storage. This may equally account for the degradation of β-carotene in the paper-polyethylene laminates, even though it had a light transmittance of nearly zero. In previous studies involving biofortified maize, lower carotenoid retention was recorded in flour stored in laminated paper bags compared to aluminium bags or double-sealed polyethylene bags [22] and aluminium pouches containing an oxygen scavenger [24]. The results of the present study also agree with Singh et al. [30] who reported a reduction in β-carotene levels of dehydrated carrot products in different packaging materials. Chavez et al. [17] noted that the efficient vacuum sealing, which maintains the vacuum during storage may reduce β-carotene losses. The findings emphasize the importance of keeping β-carotene-rich powders in packaging materials which effectively exclude light and oxygen. These may include the use of metalized laminates or double packaging in plastic pouches and paper carton boxes.

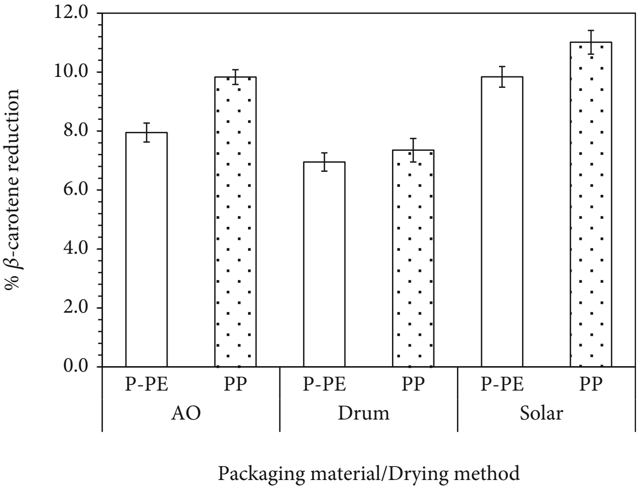


FIGURE 4: Percent reduction of β-carotene in AO-, drum-, and solar-dried flours in different packaging materials after 8 months of storage. P-PE: paper polyethylene laminate; PP: polypropylene pouch.

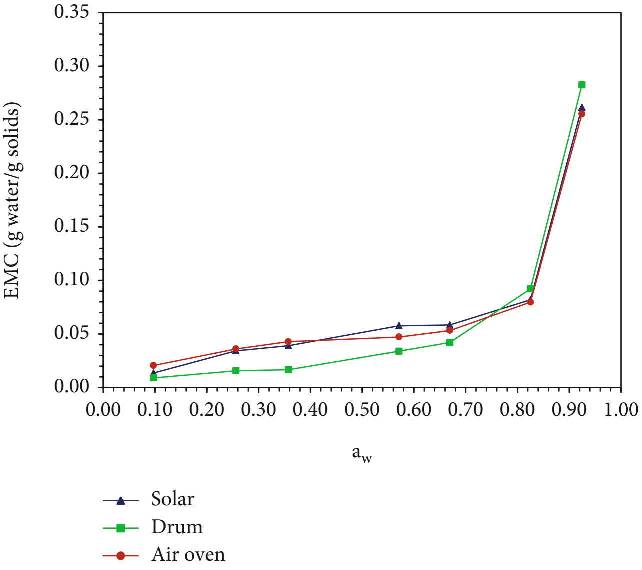


FIGURE 5: Adsorption isotherms of yellow cassava flour produced by different drying methods.

3.3. Moisture Sorption Behavior of Cassava Flour. The relationship between moisture content and water activity in flours provides useful information about product hygroscopicity and stability. It is also a useful tool for establishing packaging materials and storage conditions [31]. As

Table 4: Parameter estimates for moisture sorption modelling of yellow cassava flour produced by different drying methods.

Model	Model parameters	Drying method Solar	 Air oven	 Drum
GAB	X_m (g H_2O/100 g solid)	7.995	7.080	2.76
	C	0.011	0.012	0.200
	K	0.98	0.98	0.95
	R^2	*0.9927*	*0.9803*	*0.9975*
	χ^2_{red}	5.10×10^{-5}	1.29×10^{-4}	2.45×10^{-5}
BET	X_m (g H_2O/100 g solid)	1.836	3.116	1.253
	C	0.027	0.015	0.213
	R^2	0.9165	0.9097	0.9696
	χ^2_{red}	5.86×10^{-4}	5.92×10^{-4}	2.93×10^{-4}
Oswin	a	0.033	0.029	0.010
	b	0.818	0.858	1.328
	R^2	0.9252	0.9156	0.9894
	χ^2_{red}	5.2×10^{-4}	5.54×10^{-4}	1.02×10^{-4}
Smith	A	-0.010	-0.007	-0.035
	B	-0.198	-0.189	-0.227
	R^2	0.7936	0.7619	0.7484
	χ^2_{red}	1.45×10^{-3}	1.56×10^{-3}	2.42×10^{-3}
Iglesias & Chirife	A	0.020	0.020	0.028
	B	0.019	0.018	0.022
	R^2	0.9657	0.9658	0.9704
	χ^2_{red}	2.41×10^{-4}	2.24×10^{-4}	2.86×10^{-4}

X_m: monolayer moisture content; C: constant related to heat of sorption of the monolayer; k: constant related to the total heat of sorption; A, a, B, and b: model constants; χ^2_{red}: reduced chi-square.

expected, the isotherms of the yellow cassava flours displayed an increase in equilibrium moisture content (EMC) with increasing water activity (Figure 5). According to the GAB classification, the isotherms followed the classical type II sigmoid curve, which is characteristic of many starchy foods [32]. Initially, there was a relatively gentle rise in EMC from a_w 0.1 up to nearly 0.7. The lower level of this region ($< a_w$ 0.2) is characterized by the monolayer moisture content, where water is held by strong hydrophilic bonds at polar sites in the flour. This region gradually transitions into a zone of multilayer of water, where water is relatively held loosely by hydrogen bonds and is partly available for chemical reactions. A gentle increase in moisture was observed around a_w 0.6, with a corresponding EMC of nearly 0.05 g water/g solid. This observation suggests that practically, the most conducive moisture content for ensuring longer shelf stability of the yellow cassava flours is about 5%. Beyond a_w 0.7, there was a steep increase in EMC, reflecting the condensation of water in the granular interstices and dissolution of soluble food components. A comparison of the sorption curves showed drum-dried flours to have a steeper increase in EMC within this region. This observation suggests that the drum-dried flours absorb the most moisture and will, therefore, be the least stable within this region. Moisture sorption behavior similar to that of the yellow-fleshed cassava flour has been reported for cassava [33], modified cassava starch [34], modified cassava flour [32], and cassava flour [35].

Using nonlinear regression, the experimental moisture sorption data were fitted to five models commonly used to describe the moisture sorption behavior of food. The curve-fitting analysis showed the GAB model to be the best to describe the moisture sorption behavior of the yellow cassava flours over a water activity range of 0.1–0.9. Without an exception, this model had the highest R^2 (0.9803-0.9975) and the lowest χ^2_{red} $(1.1 - 8.5 \times 10^{-5})$ for all the drying methods used (Table 4). Previous studies involving cassava [32, 35], potato, and cereal-based extruded snack [36] also found the GAB to best describe their moisture sorption characteristics.

The monolayer moisture content of the dry flours was predicted by the GAB and BET models. This describes a single layer of water strongly bound at specific binding sites on the surface of the material, cannot be frozen and is unavailable as a solvent for chemical reactions or as a plasticizer [37]. Dried foods are most stable at their monolayer moisture content. The monolayer capacity estimated using the GAB model were higher (1.3–8.0 gH_2O/100 g solid) than those calculated with the BET model (1.2–5.4 gH_2O/100 g solid). Conversely, the constant C, which is associated with the interaction energies between food and water, was higher in BET than GAB. These discrepancies occur because, whereas the GAB model considers sorbed water in the multilayer zone, the BET focuses on surface adsorption in the first layer [38]. The drum-dried flours had lower monolayer moisture compared to flours produced by air oven drying or solar drying. The difference in monolayer moisture content may be attributed to heat-induced physicochemical changes that reduce the number of water binding sites during drying [39]. Another explanation may be that drum drying was more efficient in moisture removal from the flour because of the thermodynamics of this drying technique. The monolayer values obtained in this study were comparable to 5.4-7.3 g H_2O/100 g reported by Carmo and Pena [40] and 4.4-7.3 g H_2O/100 g for cassava starch (Ayala-Aponte [41] but lower than 7.9 and 9.0 g H_2O/100 g for cassava flour [35].

The constant "k" from the GAB model was lower in the drum-dried flours (0.91–0.95) than flours produced by solar and air oven drying, which were comparable (0.92–0.98). This suggests that, compared to pure water, the drum drying induces a less structured state of water in the layer following the monolayer [42]. The Iglesias and Cherife, Oswin, and BET models also showed a good fit with the experimental data ($R^2 > 0.9$). Among the five, Smith's model was the least suitable (with $R^2 < 0.9$) for describing the moisture sorption behavior of the yellow cassava flours.

4. Conclusion

Results of the study showed the loss of β-carotene to occur during the storage of yellow cassava flours, with the kinetics revealing a strong dependence of β-carotene degradation storage on temperature. The degradation constant, k, ranged between 2.9×10^{-3} and 3.1×10^{-2} month^{-1} and was higher for flours produced by drum drying compared to air oven and solar drying. Activation energies obtained in the study suggest that flours produced by drum drying were the most sensitive to temperature changes during storage. The flours' monolayer moisture content correspondingly ranged from 2.7 to 8.0 and 1.3 to 1.8 gH_2O/100 g solid for the GAB and BET models, respectively. The air oven samples had a comparatively lower monolayer moisture content, suggesting better stability during storage. These results are relevant for both household and industrial processing and storage of yellow cassava flour.

Data Availability

All the data supporting the results of this study are included in the manuscript.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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