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Chapter

Sorption Isotherms and Some Functional Properties of Cowpea Varieties Flour

Issoufou Amadou

Abstract

In sub-Saharan, preservation of processed cowpea flour remained a challenge, and there are no standard isotherm conditions for drying cowpea flour. This study aims to define the optimum isotherm conditions for cowpea flour and assess their functional properties. Adsorption isotherms of three varieties of cowpea at temperatures 30, 40, and 50°C and in each case with six different applications depending on the constant relative humidity of the medium were executed. Water and oil absorption capacities including swelling index were determined. Results show that water content at equilibrium is inversely proportional to the temperature, and at the same temperature, the water content increases when water activity augments. The adsorption isotherms are of type II according to the fitted BET and GAB models. The absorption capacities ranged from 1.06 ± 0.01 , 1.08 ± 0.02 , and 1.09 ± 0.01 (mL/g), respectively, for CS133, CS032, and control. However, the swelling index was significantly separated ($P < 0.05$). The adsorption isotherm curve of the sample CS032 at 50°C shows a stronger correlation ($R^2 = 0.9274$) than the other varieties regardless of the mathematical isotherm model used. It can be concluded that depending on some functional properties of cowpea variety flour, these varieties seemed to behave separately *vis-a-vis* their sorption isotherm.

Keywords: adsorption isotherm, cowpea varieties flour, GAB and BET model, swelling index

1. Introduction

The *Vigna unguiculata* (L) Walp, Cowpea, is a legume of genus *Vigna*, family *Fabaceae*, subfamily *Faboideae* [1, 2]. Although, cowpea plants are grown globally, it is suggested that they originated from Africa [3, 4]. Currently, West Africa is the major producer of cowpeas globally, of which 80% is from Central Africa [5]. Similar to beans, cowpeas are highly nutritious, hence a source of food and income globally. Cowpeas contain 17–42% protein, 1.4% fat, and 35–61% carbohydrate [6]. Compared to cereals, cowpeas are 2–3 times richer in amino acids, such as lysine, thiamine and riboflavin, and

carbohydrate; which make it also an energetic food [7]. Nowadays, cowpea farming has reached a level of important yielding in the area of its potential production. However, to make this important production profitable to the farmers, processing into cowpea flour as such is necessary, to improve the quality and profitability. Indeed, to stabilize cowpea flour, to make it competitive to other imported flour (cereals; flour), there was a need to study the drying conditions of cowpea flour [8]. Cowpeas have been a nutritious legume and the new trend in developing countries such as Sub-Saharan Africa representing about 95% of world cowpea production [9].

Drying food product such as cowpea flour is an essential step to evaluate its hygroscopic character, which indicates the affinity that the food product may have with its surrounding environment. Sorption isotherms represent the interrelation between the activity of water and the water content of food at a constant temperature. The most used technique to preserve the quality of food is to reduce the water activity to a sufficiently low level. Obtaining the sorption isotherm is essential in determining the moisture level at which microbial growth and mycotoxin production is inhibited during storage [10–12]; in addition, it predicts the speed and intensity of chemical and enzymatic reactions [11]. Drying and storage of industrial or artisanal processing operations required the know-how of the nature of water-substrate interactions or sorption isotherms [13]. When exposed to high temperatures, the functional properties of cowpea flours are affected [14–16]. This further affects the downstream processing of cowpea flour for food products. Examples of cowpea flour food products include spaghetti, couscous, porridge, infant donuts, dough, and infant flour as a dietary supplement [6, 17].

Biochemical components of cowpea varieties are expected to impact the properties of its sample, in which the hydrophilic behavior of protein has an effect on water-holding capacity. Then, the most important energy reserve, the carbohydrates, have also significant influence on the physicochemical properties of flour from cowpea varieties [1, 18]. Furthermore, the critical factors that influence the functional properties on downstream processing of food-legume products include oil and water absorption capacity [6]. The influence on the functional properties varies according to the biochemical properties of the legume, the phenomenon is likely to be even more distinct in cowpeas, especially from diverse varieties [11]. Therefore, after drying, it is relevant to assess the cowpea flour hydrophilicity (the affinity for water) and the sorption isotherm conditions (the temperature at which the flour absorbs water). The consequences of not assessing these parameters are that when the flour is not well dried, the downstream process is affected. Furthermore, the presence of moisture favors bacterial and fungal (such as aflatoxin) growth [10–12]. However, until now, the optimum isotherm conditions for drying cowpea flour are not known. The present work aimed to study the conditions of sorption isotherm for drying cowpea flour. The sorption isotherm conditions and their effect on functional properties were examined against flour from three varieties of cowpea plants. The findings will be vital in establishing a standard protocol for preserving cowpea flour, which will improve the processing of cowpea-based food products.

2. Materials and methods

2.1 Materials

Three cowpea dried seed samples were used in the study of which two seed samples (CS133 and CS032) were obtained from Dan Dicko Dankoulodo University of

Maradi (UDDM), Cowpea Square research project, Niger, whereas the control sample was obtained from the Maradi city, Niger.

2.2 Methods

2.2.1 Preparation of samples

First, the cowpea seed samples were sorted to remove foreign matter. Thereafter the seed samples were soaked in potassium hydroxide solution for 24 hours, dehulled, and dried at $90 \pm 2^\circ\text{C}$ for four (4) hours, followed by milling. The resulting flours were sifted using a $250\ \mu\text{m}$ sieve. The sifted flours were collected in an air-free polyethylene bag, sealed, and stored for subsequent experiments.

2.2.2 Physico-functional properties

The moisture content of the samples was carried out by the methods of AOAC [19]. Both for oil and water (OAC/WAC) absorption capacities of seed samples, flours (1 g) were mixed into 10 mL, centrifuged, and then stirred with *Balanites aegyptiaca* oil or distilled water, respectively. Slurries were centrifuged at $3000 \times g$ for 10 min (80–2 15/20 mL Electronic Lab Centrifuge Machine, Jiangsu, China), and the oil or water released after centrifugation was massed and expressed as (mL/g and g/g) OAC/WAC capacities, respectively, according to Sofi et al. [20] with some modifications.

The swelling index was obtained by taking a 1 g of cowpea flour sample in pre-weighed centrifuge tubes with distilled water (10 mL), vortexed; then incubated at room temperature on the shelf for 24 hours. The noted marked volume after 24 hours was considered as the total volume of flour sample and expressed as followed:

$$SV = \frac{TV}{W_i} \quad (1)$$

SV: swelling index (g/cm^3).

TV: Total volume (g).

Pi: Initial sample weight (g).

2.2.3 Determination of sorption isotherms

Equilibrium moisture content (EMC) by the static gravimetric method was used to evaluate moisture desorption isotherms at different water activity levels. In brief, cowpea flour samples (in triplicate) were placed in a desiccator, each containing a 250 mL solution of sulfuric acid at different relative humidity (10–90%) and preserved at the temperatures of 30, 40, and 50°C [21]. Periodically, samples were weighed precisely until no significant variation in weight was detected or till equilibrium was reached [22]. The EMC was calculated considering samples' initial moisture contents as described in the following:

$$EMC = \frac{w_f - w_i + \left[\frac{\%H_2O}{100} w_i \right]}{w_i \left[\frac{100 - \%H_2O}{100} \right]} \quad (2)$$

Models	Equations	Parameters
GAB [23]	$\frac{EMC}{M_m} = \frac{ABa_w}{[1-Ba_w](1-Ba_w+ABa_w)}$ <p>Rearranged equation into the second-degree polynomial form</p> $\frac{a_w}{EMC} = A_1a_w^2 + A_2a_w + A_3$	$A_1 = [B/Mm] [(1/A) - 1]$ $A_2 = (1/Mm) [1 - (2/A)]$ $A_3 = 1/(MmAB)$ a_w = water activity A, A_1, A_2, A_3, B, C = constants EMC = equilibrium moisture content Mm = GAB monolayer
BET [24]	$\frac{a_w}{EMC(1-a_w)} = \frac{1}{M_oC} + \frac{C-1}{M_oC}a_w$	M_o = BET monolayer value C = constant for a given water sorbent system a_w = water activity EMC = equilibrium moisture content

Table 1.
Sorption isotherms models equations.

Where:
 w_f = final weight of sample.
 w_i = initial weight of sample.
 $\%H_2O$ = initial moisture content.

2.2.4 Sorption equations

To smooth the model of adsorption and/or desorption curves, empirical models that can describe the relationship between water equilibrium content, relative humidity, and temperature exist. Indeed, both the GAB (Guggenheim–Anderson–De Boer) and BET (Brunauer, Emmett, and Teller) models are the best formulas used to determine the monolayer of a food, applicable for water activities between 0.05 and 0.95. The GAB monolayer value can be estimated using linear or non-linear methods. The difference in percentage in EMC between the duplicate samples was on average < 5% when the average of the two values was taken. The BET value is found by plotting $a_w/(1-a_w)EMC$ versus a_w and using the intercept to cover the monolayer. Both GAB and BET sorption equations were used to analyze the sorption isotherm data (Table 1).

2.2.5 Statistical analysis

Data were obtained in triplicate. One-way analysis of variance (ANOVA) was performed, and significant differences in mean values were evaluated by Tukey HSD multiple range test at ($P < 0.05$) using SPSS version 17.0 (SPSS, Chicago, IL, USA). Microsoft Excel 2013 was used to carry out the regression analysis of water activity as a function of increasing relative humidity and constant temperature of cowpea varieties flour.

3. Results and discussion

The cowpea grains processing technologies used by the local population of the sub-Saharan region are less efficient. The use of heterogeneous and unsuitable packaging, as well as traditional drying and preservation methods, constitutes constraints for this

activity to flourish [16, 25]. Indeed, drying influences, in particular, the water content and consequently some physicochemical, rheological, organoleptic, and functional characteristics of the food product. The moisture content of the three cowpea varieties flour samples varies from 07.52 to 08.28%, as presented in **Table 2**. The oil absorption capacities of cowpea varieties flour are found to be 1.06 ± 0.01 , 1.08 ± 0.02 , and 1.09 ± 0.01 , respectively, for CS133, CS032, and control sample mL/g. The water-holding capacity and the swelling index were revealed to be significantly dependent ($P < 0.05$) from one to another. Thus, the results showed that the water absorption capacity ranged, respectively, for CS133, CS032, and control from 1.01 ± 0.17 , 0.83 ± 0.10 , and 0.69 ± 0.11 g/g. The swelling index data varied to 4.10 ± 0.29 , 3.78 ± 0.02 , and 3.67 ± 0.24 g/cm³ for CS133, CS032, and control (**Table 2**).

This can be explained that flour which absorbs less water absorbs more oil and vice versa. These results corroborate with those of Naiker [6]. Moreover, a high-water content of flour could promote chemical and enzymatic reactions, the development of microorganisms leading to the deterioration of the product quality. In addition, the water content of food products plays a key role in their preservation [4, 26]. Thus, it appears essential to determine the minimum water content that can promote the preservation of cowpea varieties flour. The ability of a food to absorb more water can also be attributed to its content of protein and carbohydrates with free hydrophilic residues. The result shows that CS133 flour swells more than other varieties; this could be explained by the fact that flour that absorbs more water content swells better indeed. Flour with a good swelling index could be of good quality and used in pastry [11, 27]. Furthermore, a quality flour stability depends also on its drying conditions and preservation [17, 28].

Isotherms are particularly important for determining the minimum water content of a food product. Two mathematical models (GAB and BET) were used to obtain equilibrium moisture content (EMC). The data of EMC of cowpea varieties flour obtained experimentally at the temperatures of 30, 40, and 50°C, and for each of the relative humidity or the water, activities are presented in **Table 3**.

However, the average EMC values are used for the representations of adsorption isotherms by samples of cowpea varieties flours. The GAB model happened to be the most adequate model for the smoothing of the adsorption isotherms of cowpea varieties flour in this study, contrary to the findings where BET model was best smooth of sorption isotherms in the study on the dehydrated beef made in Nigeria [12]. The different adsorption curves of the cowpea varieties flour related to temperature, and different varieties are shown in **Figure 1**.

Practically, the hygroscopic equilibrium was observed around 2 weeks later, and it was found that the greater the water activity (a_w) is maintained in the medium, the easier it is for the water content to be determined. Therefore, this shows the influences of relative humidity and temperature on the drying of each of the cowpea

Cowpea varieties	Moisture content (%)	Oil adsorption capacity (mL/g)	Water adsorption capacity (g/g)	Swelling index (g/cm ³)
CS133	$9.92 \pm 0.03a$	$1.06 \pm 0.01a$	$1.01 \pm 0.17a$	$4.10 \pm 0.29a$
CS032	$7.96 \pm 0.05a$	$1.08 \pm 0.02a$	$0.83 \pm 0.10a$	$3.78 \pm 0.02b$
Control	$6.97 \pm 0.13a$	$1.09 \pm 0.01a$	$0.69 \pm 0.11a$	$3.67 \pm 0.24c$

Table 2.
Physico-functional properties of cowpea varieties flour.

Temperature (°C)	Relative humidity (%)	a _w	EMC		
			CS133	CS032	Control
30	10	0.1	1.09	1.54	1.92
	30	0.3	1.74	1.91	2.09
	50	0.5	2.27	2.59	3.58
	70	0.7	3.84	4.58	4.44
	80	0.8	4.15	5.1	5.43
	90	0.9	9.28	11.28	10.51
40	10	0.1	1.26	1.42	1.21
	30	0.3	1.75	2.27	2.35
	50	0.5	3.19	2.37	2.46
	70	0.7	4.62	4.12	4.09
	80	0.8	9.11	4.89	4.67
	90	0.9	10.25	11.4	10.69
50	10	0.1	1.29	1.34	1.37
	30	0.3	2.2	2.42	2.24
	50	0.5	3.18	4.45	3.28
	70	0.7	3.35	5.97	4.19
	80	0.8	4.29	11.3	5.33
	90	0.9	11.8	21.26	12.75

Table 3.
Equilibrium moisture content (EMC) of cowpea varieties flour as a function of water activity (a_w).

variety flour. Likewise, previous research on food products has demonstrated that the sorption isotherm curves are represented in a sigmoidal shape [12, 28]. In fact, it can be noted that only the adsorption isotherm curve of the sample CS032 at 50°C showed a strong correlation different from other samples at different temperatures (**Figure 2**) with $R^2 = 0.9274$ and $R^2 = 0.8226$ respectively for GAB and BET models.

It can be seen that the more the temperature increases, the less noticeable the difference in the drying of the inter-varietal cowpea flour is. In this line, the sorption isotherms are of type II, that is the sigmoidal sorption isotherms, in which the curves are concave upwards, taking into account the existence of multilayers at the internal surface of cowpea flour material. Generally, the increase in temperature induces the decrease in EMC, leading to an increase in a_w for constant water content. For lower a_w, the EMC is also low, compared to levels when the a_w approaches one (1) [29, 30]. Knowingly, the EMC decreases with increasing temperature and for the same value of relative humidity (RH) leads to the endothermic reaction [31]. It was observed for the samples CS133 and control for the temperatures of 30 to 50°C that the water content decreases when the temperature increases within the interval values of a_w between 0.37 and 0.78%, of which most of the curves are found around 0.8 a_w. Indeed, this decrease can be caused by the increase in the heat of absorption in the case of high temperatures, which makes it possible to reduce the EMC [32, 33]. Benseddik et al. and Ferradji and Matallah [26, 34] stipulated that they observed changes in sorption

Figure 1.
Adsorption isotherms of cowpea varieties flour by temperatures 30(a), 40(b) and 50(c) °C).

Figure 2.
Adsorption isotherms curves of CS032 flour at 50°C smoothed by BET and GAB.

isotherms with an increase in temperature is relative to the composition of the products in starch content and its solubility with an increase in thermal agitation within the samples [35]. Though, at high temperatures, the state of excitation is stronger and favors the reduction of the forces of attraction of molecules among them (**Figure 3**).

Moreover, the processing of flour regardless of the variety is considered to depend on its end-use; as result, in certain products, the more the sugars dissolve, the more mobility and the availability of water are reduced. In addition, the ability of water adsorption can also be attributed to its content of protein. Moreover, for products

Figure 3.
Adsorption isotherms with temperature differences on the cowpea varieties flour (CS133, CS032 and control).

with low sugar content such as flour, the curves do not intersect, likewise, the work by Koko et al. [4]. The flour of cowpea varieties is not an exception of this trend, whereas the physicochemical properties further affect their drying conditions.

4. Conclusion

It can be concluded that the flour of cowpea varieties absorbs differently the water and oil, and the swelling index significantly. It was a fact that flour, which absorbs more water content, swells better, as far as the sorption isotherms of cowpea varieties flour were carried out at different temperatures range of 30, 40, and 50°C. The data showed a sigmoidal shape, characteristic of type II isotherms, whether determined by the GAB or BET models. It was found that the GAB model allows the most important relative squared errors. Thus, the GAB model was the most adequate model for the moisture absorption isotherms on the flours of the three varieties of cowpea studied. For lower a_w , the EMC was also low, compared to levels when the a_w approaches one (1). It was noted that the more the surrounding temperature increases, the more the water content of the product decreases. A significant linear interaction was revealed to better describe the variation within the EMC range considered in this work. Thus, depending on the rate of swelling of these varieties, it can be deduced that the CS032 cowpea variety flour seemed to behave better than its counterpart varieties. More such findings are needed to favor these legume development programs for expanding preferable varieties for targeted applications.

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Author contributions

Issoufou AMADOU designed, supervised lab technicians for the experiments, wrote and proofread the article.

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Conflicts of interest

The author declares no conflict of interest.

Abbreviations

a _w	Water activity
BET	Brunauer, Emmett, and Teller
CS133 and CS032	Names of cowpea varieties for CowpeaSquare
EMC	Equilibrium moisture content
GAB	Guggenheim - Anderson - De Boer
UDDM	Université Dan Dicko Dankoulodo de Maradi

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