



Research article

Sorption behavior and isosteric heat of maize-millet based protein enriched extruded product

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ARTICLE INFO

Keywords:

Equilibrium moisture content
Extruded product
Net isosteric heat
Relative humidity
Temperature condition
Water activity

ABSTRACT

Moisture sorption behaviour and isosteric heat of sorption are essential to be familiar with the stability of products, various storage condition and selection of packaging materials. Sorption behavior of extruded product was done at 30, 40, 50 and 60 °C temperatures under 11–92% relative humidity. The product was developed from an optimized blend of maize, defatted soy, finger millet and yam at an optimum condition of 14% (wb) feed moisture, 110 °C barrel temperature and 301 rpm screw speed. The net isosteric heat of sorption of the extruded product was determined by using Clausius-Clapeyron equation. The equilibrium moisture content (EMC) was found to be raised with the rise in water activity (a_w) in particular temperature. The highest EMC 30.05% (db) was recorded at 0.92 a_w and 30 °C temperature, whereas the lowest EMC 2.46% (db) at 0.11 a_w and 60 °C temperature. The isotherms exhibits curves of Type-II for four temperature conditions. The EMC data were fitted to six sorption models and GAB model was found to be best fitted among them. The net isosteric heat of sorption varied between 16.711 and 3.242 kJ/mol within 5–30% (db) of sample moisture content.

1. Introduction

Knowledge of moisture sorption behavior of food products or extruded snacks is most imperative in the field of food engineering and technology to solve various technical difficulties like design, development and optimizing the processes, selection of materials for packing, prediction of storage-life, etc (Gal, 1987). Sorption characteristics explain the connection of equilibrium moisture content versus equilibrium relative humidity for a product at particular pressure and temperature. Research on temperature dependent isotherms, heat of sorption, mathematical modeling and their pragmatic and semi pragmatic relationships, showing the adsorption behavior of various food materials have also been depicted in published journals. There have been 23 sorption equations fitted to moisture sorption data of dairy, food, snacks and bakery products exist in the previous literature reported by Chirife and Iglesias (1978). The energy required for drying and dehydration of any food material can be estimated by the application of net isosteric heat of sorption. The bound water inside the food product is the amount of water that exhibited the net isosteric heat of sorption, which is equal to the pure water's latent heat of vaporization (Sormoli and Langrish, 2015).

Now a day's consumers are very much interested to consume ready-to-eat breakfast foods, mainly due to their easy availability, fast life, convenience, changes into living status, rapid urbanization, taste, colour, appearance, texture and all a health conscious. In food industry, inexpensive cereal based ingredients are used for development of extruded products through extrusion cooking. The food processing area of the universe possesses potentially good influence in these types of products to acquire R-T-E snacks with a variety of nutritional configuration, including digestive starch and protein rich products for growing children, pregnant women and sports person (Brennan et al., 2013). It is a very powerful exercise to enhance the nutritional values of extruded product by incorporation locally available raw ingredients and has potential to utilize by-products for the production of new commercial products. Flour of finger millet contains high amount of calcium, phosphorus, iron, fiber as well as vitamins. It is also useful for diabetic patients, which is a slower glycemic index. Defatted soy flour is an excellent source of plant protein and also contributes functional, nutritional and healthier effects to the human beings by lowering cholesterol level, obesity, etc (Novotni et al., 2009). Moisture sorption behaviour had also been studied for extruded product developed from Oat-fenugreek by Wani and Kumar (2016); for

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Received 15 December 2020; Received in revised form 4 February 2021; Accepted 1 April 2021

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Corn-lentil based extruded products by Lazou and Krokida (2011) and for the product developed from Corn-grit by Potente et al. (2006).

Extruded foods are very important product in terms of manufacturing, packing, handling, transporting and storability. The evaluation of equilibrium moisture content of extruded product is necessary to awareness with the drying and storage behavior. Moisture content is the most affecting factors for changes in the quality of extrudates in the processing and storage study. The purpose of the present research was to study the sorption isotherm characteristics of extruded foods prepared from a mixture of maize-millet-defatted soy and to compare with six sorption models for determination of more accurate result for forecasting of data. Determination of the net isosteric heat of sorption was also carried out.

2. Materials and methods

2.1. Materials and product development

The product was prepared from an optimized blend composition of maize, finger millet, defatted soy and elephant foot yam (Sahu and Patel, 2020a). The blending of flour proportions was performed to achieve the optimum percentage of protein in the blend to produce proteinacious extruded products and blending was performed by using D-optimal mixture design (Design Expert version 10.0.5.0). Extrusion cooking was performed by using a laboratory model of twin-screw extruder co-rotating type (BTPL, Kolkata, India) (Figure 1). The feed rate of the feeder (24 rpm) and speed of cutting knife (22 rpm) were maintained constant. Die diameter of extruder was selected as 3 mm. The combination of process parameters, feed moisture content 14% (wb), barrel temperature 110 °C and screw speed 301 rpm was adopted for production of extruded product.

2.2. Determination of sorption isotherms

Sorption isotherm behavior of the developed product at 30, 40, 50 and 60 °C temperatures in the water activity range from 0.11 to 0.92 were studied to show the variation of moisture content with respect to changes in water activity. 5 ml toluene in glass dish was placed in

desiccators at higher water activity conditions to check mold development (Labuza, 1984). Drying of extruded product samples was performed at 70 °C for one day before placing it for equilibration into the trial range of water activity (Wani & Kumar, 2016; Sahu and Patel, 2020b). The salt solutions were placed in desiccators to attain the required water activities (Greenspan, 1977). The different salt solution produces diverse water activity under different temperature conditions is shown in Table 1.

The static gravimetric method was employed to study the moisture sorption behavior as described by Rahman and Sablani (2009) and Lazou and Krokida (2011). The sorption apparatus (desiccators) filled with different saturated salt solution was equilibrated at respective temperatures using temperature controlled chamber for four days before placing the samples for actual study. Precisely weighed quantity of extruded product samples (nearly 2 g) were taken in the petri dishes and positioned in the desiccators, which were held at seven different water activity conditions. Finally, this apparatus was consigned into the chamber of temperature controller at a particular temperature. Triplicate samples in each of the desiccators were maintained during the study and their mean was used to keep away from investigational error. Similar experiments were repeated for each temperature conditions. The weight of each sample was documented regularly in the period of 2–3 days until the samples accomplished the equilibrium. The equilibrium condition was adjudged by taking the three consecutive observations, which was less than or equal to 0.001 g. The moisture present in the sample was found out by using a method described by Sahu and Patel (2020b). The graph of equilibrium moisture content (EMC) versus equilibrium relative humidity (water activity) for particular temperature condition was plotted which indicates the graph of sorption isotherm.

2.3. Mathematical moisture sorption model

The six different sorption isotherm models to express the sorption isotherm behavior at four investigating ranges of temperature were selected as presented in Table 2 (Sahu and Patel, 2020b). These six sorption models are shown in Table 2 in the form of expression (Eq. (1) to Eq. (6)). The method of regression analysis, which minimizes the residual sum of square, was exercised to calculate the model parameters within

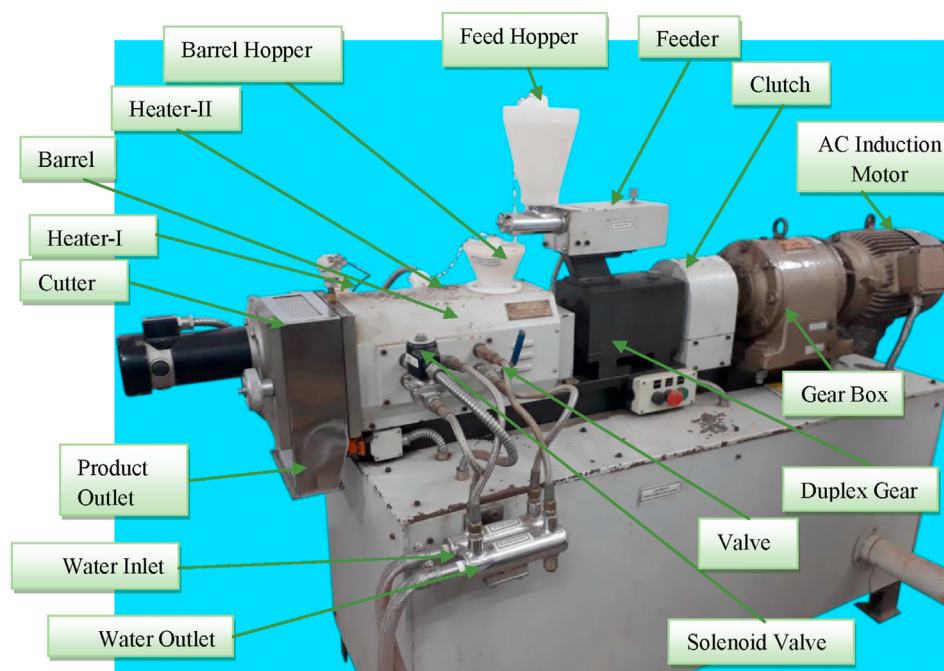


Figure 1. BTPL Lab Model (EB-10) Twin screw co-rotating extruder.

Table 1. Various salts and their corresponding relative humidity at different temperatures.

Salts	Relative humidity, %			
	30 °C	40 °C	50 °C	60 °C
Lithium chloride	11.3	11.2	11.1	11.0
Magnesium Chloride	32.4	31.6	30.5	29.3
Potassium carbonate	43.2	40.0	38.5	37.1
Potassium iodide	67.9	66.1	64.5	63.1
Sodium chloride	75.1	74.7	74.4	74.5
Potassium chloride	83.6	82.3	81.2	80.3
Potassium nitrate	92.3	89.0	84.8	82.8

Table 2. Moisture sorption models applied to analyze data of extruded product.

Model	Mathematical expression	Equation
GAB (Quirijns et al., 2005)	$M = \frac{M_0 C k a_w}{(1 - k a_w)[1 - k a_w + C k a_w]}$	(1)
BET (Brunauer et al., 1938)	$M = \frac{M_0 C a_w}{(1 - a_w) + (C - 1)(1 - a_w) \cdot a_w}$	(2)
Smith (Smith, 1947)	$M = a + b \log(1 - a_w)$	(3)
Caurie (Caurie, 1970)	$M = \exp(a + b a_w)$	(4)
Oswin (Oswin, 1946)	$M = a \left(\frac{a_w}{1 - a_w} \right)^b$	(5)
Iglesias and Chirife (Iglesias and Chirife, 1982)	$M = a + b \left(\frac{a_w}{1 - a_w} \right)$	(6)

M - equilibrium moisture content, % (db), a_w - water activity, a and b - constants, M_0 - monolayer moisture content, % (db), C - Guggenheim constant, and k - constant related to total heat of sorption of multilayer.

the range of water activity (0.11–0.92). The sorption values were evaluated by fitting into the models and the resulting constants were obtained.

2.4. Statistical analysis

The sorption model for better prediction of equilibrium moisture content was found out on the basis of various goodness of fit parameter like coefficient of determination (R^2), percentage error of root mean square (%RMSE), adjusted R^2 and proportional percentage deviation modulus (%P). The following equations (Eqs. (7), (8), and (9)) were used to calculate the goodness of fit variables as suggested by Sahu and Patel (2020b).

$$\%RMSE = \sqrt{\frac{1}{N} \left[\sum_{i=1}^N \left(\frac{Y - Y'}{Y} \right)^2 \right]} \times 100 \quad (7)$$

$$P (\%) = \frac{100}{N} \left[\sum_{i=1}^N \left(\frac{Y - Y'}{Y} \right) \right] \quad (8)$$

$$\text{Adjusted } R^2 = 1 - \frac{\sum_{i=1}^N (Y - Y')^2 (N - 1)}{\sum_{i=1}^N (Y - Y'')^2 (N - M)} \quad (9)$$

where, Y = investigational EMC (%), Y' = expected EMC (%), Y'' = mean value of investigational EMC (%), N = number of observations and M = number of coefficients in the model.

The most appropriate model was selected on the basis of the highest R^2 and adjusted R^2 as well minimum errors (% RMSE and %P) accounted. The values of % RMSE and % P should lie below 10% to be accepted as a good fit (Jena and Das, 2012).

2.5. Determination of net isosteric heat of sorption

The net isosteric heat of sorption was determined by the generalized equation of Clausius-Clapeyron (Eq. (10)) for any food material (Labuza, 1984). At any known water content, the water activity (a_w) value was increased with an increase in temperature. Thus, predicted water activity (a_w) is a function of temperature. The graph of log of water activity versus the inverse of the absolute temperature ($1/T$) gives a straight line at particular condition of moisture content is called isoster. The slope of the isoster was used to calculate the net isosteric heat, which is equal to (q_{st}/R).

$$\ln \left(\frac{a_{w2}}{a_{w1}} \right) = \frac{q_{st}}{R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \quad (10)$$

where, q_{st} is the net isosteric heat of sorption (kJ mol^{-1}), R is the universal gas constant ($8.314 \text{ JK}^{-1} \text{ mol}^{-1}$), a_{w1} is water activity at temperature T_1 and a_{w2} is water activity at temperature T_2

The relationship used to determine the isosteric heat of sorption (Q_{st}) was given in Eq. (11) below.

$$Q_{st} = q_{st} + \Delta H_v \quad (11)$$

where, ΔH_v is latent heat of vaporization of pure water.

3. Results and discussion

3.1. Isotherm behavior

Moisture sorption isotherm of a snack product for four different investigating temperatures of 30, 40, 50 and 60 °C were plotted between the EMC data and water activity (a_w) as exposed in Figure 2. It can be observed that sorption isotherm vary with the temperature conditions of the extruded product. The extruded product exhibits the curves of type-II for all the temperature conditions which is similar to most of the dried food materials. Lazou and Krokida (2011) has also been communicated the similar type of findings for extruded snack samples. The trend of isotherms further depicted that EMC value increased with the increase in water activity at particular temperature condition, which might have been the hydrophilic nature of starch and protein available in the extruded product (Figure 2). The decrease in EMC with the boosting in temperature at fixed water activity condition was observed. This variation was owing to the reduction in the number of dynamic polar sites for attraction of water molecules at higher temperature. The same result was obtained by the previous findings of Sawhney et al. (2011) for the dried acid casein obtained from buffalo whole milk and Wani and Kumar (2016) for the extruded product made from composite flour of rice and corn with the incorporation of green pea and fenugreek powder and Varghese et al. (2014) for beverage mix from freeze dried whey-grape.

3.2. Moisture sorption modeling

The moisture sorption data were employed to fit in the six different sorption models using mathematical expression as presented in Eqs. (1–6). The isotherm model parameters and goodness of fit parameters such as the coefficient of determination (R^2), adjusted R^2 , percentage error for root mean square (% RMSE) and proportional percentage mean error (%P) for the models are given in Table 3. The least value of % RMSE for predicted data and data obtained from experiment resulted better accuracy of the models. The value of percentage RMSE below 10% is considered to be adequate for acceptability of the isotherm model (Koc, Yilmazer, Balkir and Ertekin, 2010). A model is also measured to be appropriate if the value of P is less than 10% (Jena and Das, 2012; Mousa et al., 2014).

The results in Table 3 indicated that the GAB model has been emerged to be more accurate to express the investigational data over the whole range of EMC and ERH of the extruded product with lowest % RMSE and %P values compared to other models. The % RMSE and %P values were ranged from

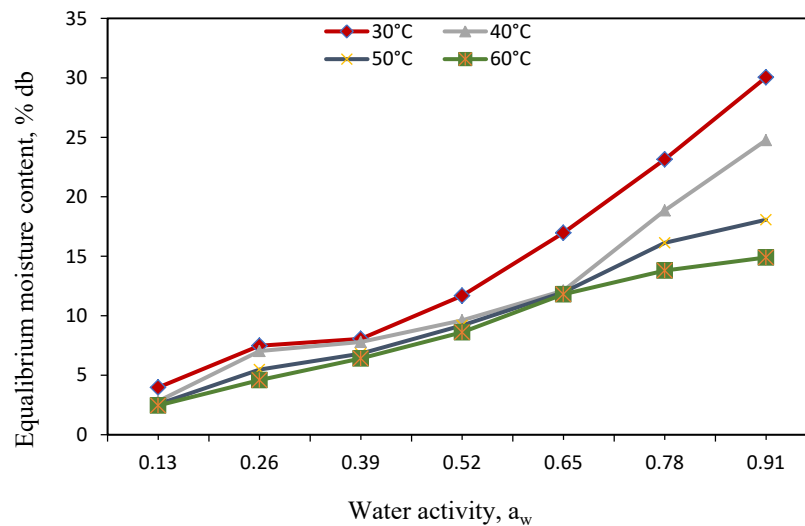


Figure 2. Sorption isotherms of extruded product at different temperatures.

Table 3. Constants and monolayer moisture content of sorption models as well as statistical accuracy values for sorption isotherms of extruded product at four different temperature conditions.

Model	Temperature, °C	%RMSE	P (%)	R ²	Adj R ²	M ₀	C	k	a	b
GAB	30	7.95	0.23	0.93	0.97	5.33	22.11	0.899		
	40	14.65	0.23	0.98	0.90	5.29	12.25	0.853		
	50	7.55	0.08	0.99	0.97	5.18	9.08	0.834		
	60	5.76	0.03	0.98	0.99	5.13	8.27	0.805		
Smith	30	11.81	0.87	0.96	0.97				2.18	10.80
	40	17.01	0.31	0.98	0.92				1.76	9.54
	50	10.14	0.77	0.98	0.97				1.94	8.19
	60	9.70	1.48	0.97	0.98				2.09	7.19
Oswin	30	9.09	0.24	0.94	0.97				9.83	0.45
	40	13.63	1.89	0.97	0.95				8.44	0.48
	50	7.55	0.60	0.99	0.98				7.63	0.49
	60	6.32	0.07	0.98	0.99				7.09	0.48
Caurie	30	38.32	6.98	0.76	0.39				-1.55	475.80
	40	29.48	4.54	0.78	0.45				-1.69	442.88
	50	27.04	5.54	0.78	0.60				-1.84	455.32
	60	26.65	3.19	0.79	0.68				-1.95	474.94
BET	30	26.46	6.96	0.96	0.94	3.63	29.92			
	40	20.12	5.71	0.89	0.88	3.51	31.21			
	50	19.04	6.22	0.95	0.95	3.46	83.30			
	60	20.73	11.90	0.94	0.96	3.42	36.73			
Iglesias & Chirife	30	36.65	11.79	0.76	0.87				7.30	2.13
	40	33.41	8.04	0.87	0.95				4.88	2.58
	50	27.48	6.20	0.94	0.96				4.03	2.66
	60	26.25	6.16	0.95	0.95				3.77	2.47

M₀ = monolayer moisture content, C = Guggenheim constant, k = constant related to total heat of sorption of multilayer, a and b = constant.

5.76 to 14.65% and 0.03–0.23%, respectively for GAB model. The %RMSE and %P values for the model of Oswin, Smith, BET, Iglesias & Chirife and Caurie are also presented in Table 3. The high value of R² (0.93–0.99) and adjusted R² (0.90–0.99) for the GAB, Smith and Oswin models had shown its high adequacy for extruded products. The lowest P value of GAB model compared to other models for predicted EMC indicated its best consideration for the sorption model of the extruded product. Therefore, the GAB model was found to be the most superior model to express the sorption characteristics amongst all the six models used to fit the sorption data. The GAB model is commonly used and is considered as the best suitable model for sorption behavior in food science.

3.3. Monolayer moisture contents

The quantity of moisture detained forcefully and restricted to a specific position on the surface of food is known as monolayer moisture content (M₀). The importance of M₀ on a food product is that, lower than the value of M₀, the existing moisture does not acquire any type of deteriorative effect either as solvent or as one of the substrates. The GAB and BET models are utilized for the estimation of monolayer moisture content (M₀) of food products. The other models do not take part of the monolayer moisture content into account and unable to predict. The M₀ and GAB constants C, and k have also been determined for the present

case and depicted in Table 3. The value of the monolayer moisture of extruded product was determined to 5.33% (db) and 3.63% (db), respectively for GAB and BET equations at 30 °C temperature and their corresponding values for 60 °C temperature were 5.13% (db) and 3.42% (db), respectively.

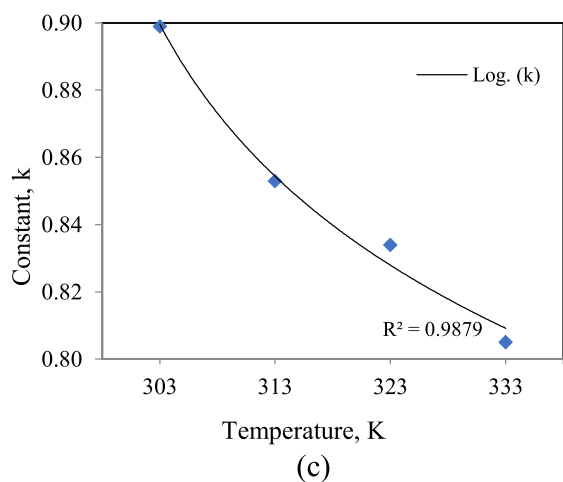
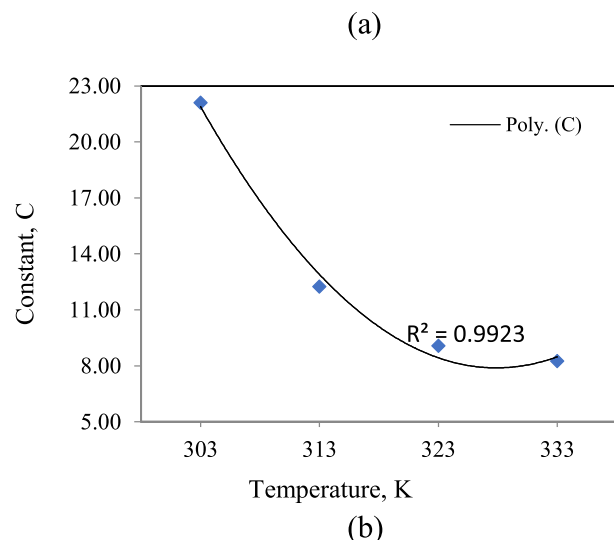
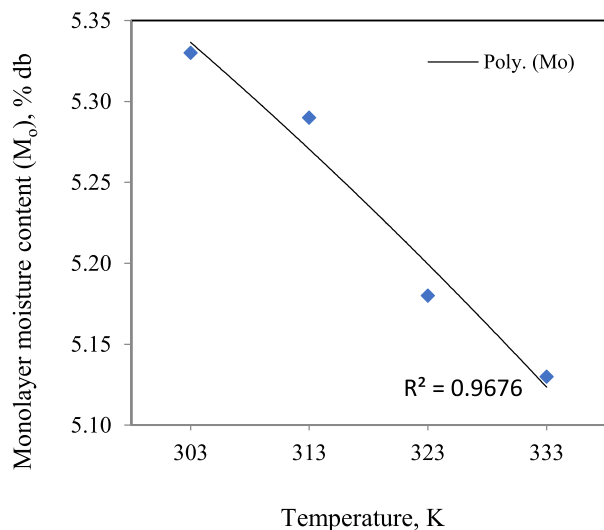


Figure 3. (a) Variation in M_0 with absolute temperature (T). (b) Variation in constant C with absolute temperature (T). (c) Variation in constant k with absolute temperature (T).

A plot between monolayer moisture (M_0) versus absolute temperature (T) is depicted in Figure 3a. The M_0 values followed the decreasing trend as the temperature increased. Similar trends of behavior had also been demonstrated of other dried food materials by earlier researchers (Iglesias and Chirife, 1976; Ayranci and Duman, 2005). This might be due to reduced accessibility of hydrophilic site and less capacity of polymers to make hydrogen bonding (Quirijns et al., 2005; Mousa et al., 2014). The connection between monolayer moisture content (M_0) and absolute temperature has also been developed through regression equation. The following form (Eq. (12)) was obtained to describe the relationship with a very high coefficient of determination ($R^2 = 0.97$) which ensures the accurate prediction of monolayer moisture of the prepared extrudate.

$$Y = -0.002x^2 - 0.058x + 5.397 \quad R^2 = 0.97 \quad (12)$$

where, Y is monolayer moisture (% db) and x is absolute temperature (K).

3.4. GAB constant C and k

The GAB constants C, and k indicate the interaction energies between food and water molecules in the product. The change in constant C with respect to variation in temperature is presented in Figure 3b. The value of C followed the decreasing trend with the increase in temperature. The relationship between constant C and the absolute temperature obtained by regression analysis is given below (Eq. (13)). As the value of the coefficient of determination is high, nearing to 1, this can successfully be used for predication of this constant C.

$$Y = 2.262x^2 - 15.78x + 35.41 \quad R^2 = 0.99 \quad (13)$$

where, Y is constant C and x is absolute temperature (K).

Similarly, the variation in GAB constant k with absolute temperature is revealed in Figure 3c. This figure shows that the constant k decreases linearly with the positive increase in temperature. The relationship between k and temperature (T) is also presented in the regression equation (Eq. (14)). It is worth mentioning here that the value of k being less than 1, the sample contained only bond water and the free moisture was absent (Quirijns et al., 2005).

$$Y = -0.06 \ln(x) + 0.899 \quad R^2 = 0.99 \quad (14)$$

where, Y is constant k and x is absolute temperature (K).

3.5. Isostatic heat of sorption

Water activity at two or more temperatures is necessary for estimation of sorption energy requirement. The net isosteric heat of sorption has strongly associated with water content and it decreases with the increase in water content. By plotting the graph between $-\ln a_w$ versus $1/T$

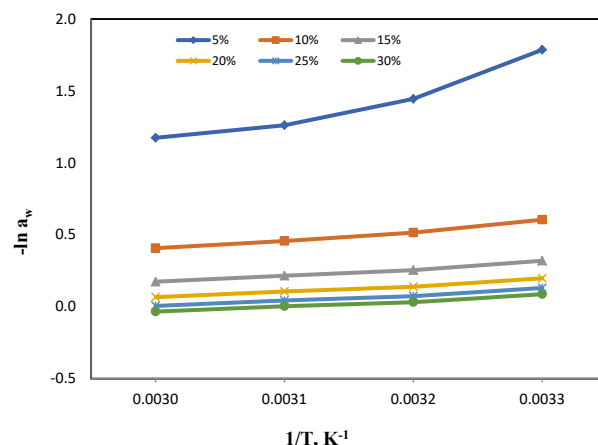


Figure 4. Sorption isosters of extruded product at different water content.

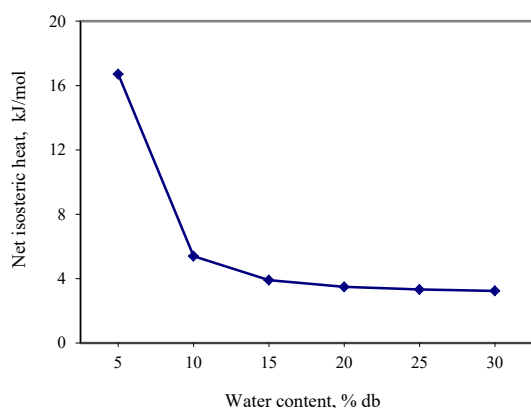


Figure 5. Changes in net isosteric heat of sorption with water content.

(absolute temperature) at particular water content was obtained in the form of straight line called isosters (Figure 4). The net isosteric heat of sorption was found out by determining the slope of each isosters from the graph. The decreasing trends in the slope of isosters occur through the increase in moisture content of the extruded product. Figure 5 depicted that the decreasing trend in net isosteric heat of sorption with the increase in water content. The maximum value of net isosteric heat at minimum water content (5%) was recorded to be 16.711 kJ/mol and minimum 3.242 kJ/mol at 30% water content. It can be seen from Figure 5 that isosteric heat initially decreased sharply up to 15% (db) water content beyond which the further decrease was found to be very slowly. The rapid increase in isosteric heat of sorption at low water content may be because of rapid exposure of extremely active polar sites of food surface, which are surrounded by water molecules providing a monomolecular coat. Once the hydrophilic sites are not accessible anymore, the linkage acquires with the less active site giving lower heat of sorption (Quirijns et al., 2005). It is also evident in Figure 5 that the net isosteric heat of sorption becomes constant after 20% water content of the product, which indicated that the water molecules perform as free water in the product as liquid form. The net isosteric heat of sorption was observed to be fine judgment of the interface of water vapour with the food substrate. This is a good marker on the microbiological, chemical and physical constancy of food products (McMinn and Magee, 2003).

4. Conclusions

The sorption isotherm for maize-finger millet based soy fortified extruded products at 30, 40, 50 and 60 °C temperatures were established to be the typical curves of type II. Water sorption isotherm of the product was found to be temperature dependent. The value of EMC decreased at constant water activity with the rise in temperature condition. EMC increased with the increase in water activity (a_w) under fixed temperature conditions. The GAB model was more appropriately fitted and established to envisage the sorption data adequately over the experimental water activity conditions. Monolayer moisture content (M_0) at temperatures of 30 and 60 °C was recorded to be 5.33 and 5.13% (db), respectively, for the product according to GAB equation which emerged to be in good condition. The M_0 values were observed to be in decreasing trend with the temperature. The net isosteric heat of sorption of extruded product at 5% (db) moisture content was found to be 16.711 kJ/mol.

Declarations

Author contribution statement

Sahu Chandrachas: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Patel Shadanan, Khokhar Dharmendra: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Funding statement

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Data availability statement

The authors do not have permission to share data.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

Acknowledgements

The entire author's delivers thanks and acknowledged to Indira Gandhi Agricultural University, Raipur (CG), DSVCKV, Durg and AICRP (PHT), ICAR, New Delhi for providing facilities for the research work and mathematical analysis during research.

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