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Adsorption isotherm and heat of sorption of osmotically pretreated and air-dried pineapple slices.

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Abstract Slabs $(1 \times 1.5 \times 5 \text{ cm})$ of pineapple were immersed in sucrose solutions of 52°B, 60°B and 68°B (w/w) at 25 °C for 12 h. Osmosed pineapple slices were subsequently air-dried at 60 °C for 72 h. Adsorption isotherms of osmo-dried pineapple slices were determined at 20 °C and 40 °C, using the gravimetric-static method. Isotherms were modeled using Brunnauer, Emmet and Teller (BET) and Guggenheim Anderson de Boer (GAB) models. Heat of moisture adsorptions were calculated from adsorption data using the Clausius-Clapeyron equation. Adsorption isotherms of osmo-oven-dried pineapple followed the characteristic type III (J-shape) isotherms. Equilibrium moisture contents at constant water activity decreased with increasing temperature, but crossing of the isotherms occurred at $a_{\rm w}$ ~ 0.86. GAB and BET monolayer moisture decreased with increased temperature. The net isosteric heat increased with decreased moisture content, and decreased with increased sucrose pretreatment concentration of pineapple slices.

Keywords Osmotic pretreatment · Air-drying · Pineapple slices · Heat of sorption · Adsorption isotherms.

Introduction

Osmotic dehydration has been applied to many fruits such as apple, banana, kiwifruit [1, 2, 3, 4] and pineapple [5]. Osmotic dehydration is a water removal process in which a cellular material, such as that of fruit and vegetables, is immersed in a concentrated sugar or salt solution [6]. Partial dehydration by immersion of slices of fruits or vegetables in hypertonic solutions of sugar or salt basically creates two simultaneous mass transfers [1, 7] namely: water outflow from product to solution, and solute transfer from solution to product. Solids uptake by fruit pieces makes it possible to infuse the desired amount of solute of nutritional interest, or sensory quality improver into the product. Thus, osmotic dehydration enhances a formulation and modification of food pieces with minimal damage to their integrity. Complementary treatments such as hot air, or vacuum drying may be applied to previously osmosed fruit or vegetable to produce an intermediate moisture food product. Intermediate moisture fruit products are developed to preserve quality characteristics such as colour, flavour, appearance, and texture as close as possible to their fresh counterparts. Moreover, intermediate moisture foods are expected to be stable under storage and marketing. Thus, it is important to derive adequate information in this regard.

Moisture sorption isotherms are useful thermodynamic tools for determining interactions of water and food substances, and providing information on food processing operations such as drying, mixing, packaging and storage [8]. Sorption data can also be used for selecting appropriate storage conditions and packaging systems to optimize or maximize retention of aroma, colour, texture, nutrients and biological stability [9, 10, 11]. Heat of sorption is useful when assessing energy requirements for drying [12]. The isosteric heat sorption is a measurement of the energy or intermolecular bonding between water molecules and absorbing surfaces [13]. Thus, the objectives of this work are to establish the adsorption isotherms of osmotically pretreated and air-dried (osmo-dried) pineapple, and to estimate the isosteric heat sorption.

Materials and Methods

Mature green pineapples were purchased from the Oje fruit market in Ibadan, western part of Nigeria. Pineapples were kept at 25 °C until they were ripe. Sucrose (table sugar) was obtained from Bodija market, Ibadan, Nigeria. Sucrose solutions were prepared by dissolving known weights of sucrose in hot (98 °C) distilled water to produce 52°C, 60°B, and 68°B (w/w). The solutions were then allowed to cool to 25 °C. Ripe pineapple fruits were washed and peeled. After the core was removed, slices/slabs (1 cm×1.5 cm ×5 cm) were manually cut from the pineapple with very sharp

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knives. Pineapple slices were packed into wire meshes and immersed in sucrose solutions maintained at 25 °C in a water bath. A fruit:solution ratio of 1:20 was maintained to avoid significant changes in the solution concentration during the 12 h immersion. After osmotic treatment, samples were removed from the solutions, quickly rinsed with distilled water and gently blotted with tissue to remove surface solution. Some samples were removed to determine the solids gain during osmotic pretreatment, according to the method of Panagiotu et al.[4]. Osmosed pineapple slices were subsequently dried in a hot-air Gallenkamp (Model OV-160) oven at 60 °C for 72 h, to remove all the free moisture present in the osmosed pineapple slices prior to the sorption experiment.

Adsorption isotherm determination. Adsorption isotherms of osmosed and oven-dried (osmo-dried) pineapple slices was conducted using a gravimetric-static method. Sulphuric acid solutions were prepared at different concentrations to give the required relative humidities [14, 15]. Osmo-oven-dried pineapple slices were kept in desiccators maintained at different relative humidities between 10 and 90%, and at 20 °C and 40 °C. A few drops of formalin were added to a cotton wool placed inside each dessicator to prevent mould growth. Samples were weighed regularly until equilibrium was attained after three equal consecutive readings. Equilibrium was reached in about 14-18 days depending on relative humidity and temperature. After equilibrium, moisture contents of equilibrated samples were determined according to the method of Sankat et al.[1]. Experiments were carried out in triplicate. The mean of the equilibrium moisture content was plotted against water activity (a_w) i.e. ERH/100.

Experiments data was fitted into Guggenlein Anderson deBoer (GAB) and Brunnaner-Emmet-Teller (BET) models. GAB and BET model parameters were calculated using a non-linear regression program developed by Professor T.P. Labuza, University of Minnesota, Minneapolis, and USA.

Results and discussion

As expected, equilibrium moisture content increased with increased water activity (a_w) in all osmo-dried pineapple slices. Fig. 1 shows the effect of pretreatment solution concentration on adsorption isotherms of preosmosed and air-dried pineapple slices at 20 °C. Equilibrium moisture content was reduced in the low and intermediate a_w range, but increased rapidly at high a_w range. Adsorption isotherms of osmo-dried pineapple showed type III (J-type) isotherms characteristic of products with high sugar content. This agreed with the classification of Labuza et al. [16]. Adsorption isotherms of osmo-dried pineapple agreed well with those of dried apricot, fig, raisin, prunes, currants [17, 18, 19] and blueberries [12]. Generally, equilibrium moisture contents decreased with increasing pretreament solution concentration. The solids gain in pineapple slices immersed in 52°B, 60°B and 68°B were 0.0459, 0.0505 and 0.0732 g solids/g initial mass, respectively. Higher solids gain occurred with increasing pretreament sucrose solution concentration. The increased solids in pineapple slices with increasing pretreament solution concentration prior to air-drying depressed equilibrium moisture content. The same trend was obtained for osmo-oven-dried products equilibrated at 40 °C (Fig. 2).

Equilibrium moisture content should increase with increasing temperature [20]. A similar trend was obtained in osmo-oven-dried pineapple slices until inversion of



Fig. 1 Effect of sucrose concentration on adsorption isotherm of air-dried (60 $^{\circ}$ C) pineapple at 20 $^{\circ}$ C



Fig. 2 Effect of sucrose pretreatment concentration on adsorption isotherm of air-dried (60 $^{\circ}$ C) pineapple at 40 $^{\circ}$ C

temperature occurred at $a_{\rm w} \sim 0.86$. Figure 3 shows the effect of temperature on adsorption isotherms of pineapple slices preosmosed in 52°B and subsequently air-dried at 60 °C. As shown, crossing of the 20 °C and 40 °C isotherms occurred at $a_{\rm w} \sim 0.86$. The same trend was observed in pineapple slices preosmosed in 60°B and 68°B sucrose solutions and subsequently air-dried at 60 °C

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Fig. 3 Effect of temperature on adsorption isotherm of air-dried (60 °C) pineapple preosmosed in 52°B sucrose solution



Fig. 4 Effect of temperature on adsorption isotherm of air-dried (60 $^{\circ}$ C) pineapple slices preosmosed in 60 $^{\circ}$ B sucrose solution at 25 $^{\circ}$ C

(Fig. 4 and Fig. 5). For all the products, moisture content was greater at 40 °C than at 20 °C above this inversion point. Crossing of the 20 °C and 40 °C isotherms at $a_{w} \sim 0.86$ was an indication of variation in moisture content versus water activity, and dissolution of sugar, in this water activity range [18]. This inversion of the usual dependence of water sorption isotherms with temperature may be due to an endothermic dissolution of sugar at high water activities and moisture contents [19, 21], so that more water was being held by the food products at high temperatures [18]. The inversion is then a relevant factor



Fig. 5 Effect of temperature on adsorption isotherm of air-dried (60 $^{\circ}$ C) pineapple slices preosmosed in 68°B sucrose solution at 25 $^{\circ}$ C



Fig. 6 Effect of osmotic pretreatment on isosteric heat of sorption of oven-dried (60 $^{\circ}$ C) pineapple slices

opposing the normal trend for an increased microbial activity with temperature [22].

Calculated net isosteric heat of sorption increased with decreasing moisture content. Figure 6 shows the variation of the net isosteric heat of sorption with moisture content of osmo-dried pineapple slices. Net isosteric heat of sorption was high at low moisture contents, but decreased to zero or negative values as moisture content increased. The observed positive value of net isosteric heat of sorption at low moisture contents is supposedly due to an easy physical sorption of water molecules forming a mono-

 Table 1
 Effect of pretreatment concentration and equilibrium temperature on the GAB and BET parameters of osmo-oven-dried pineapple

 slices
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Pretreatment solution concentration (°B)	Temperature (°C)	GAB parameters			BET parameters	
		Monolayer (gH ₂ O/g solid)	C_1	C_2	Monolayer (gH ₂ O/g solid)	C_1
52	20	0.0736	0.98290	61.9189	0.0570	0.09318
60	20	0.0705	0.98561	73.9973	0.0557	0.09206
68	20	0.0679	0.98089	55.3653	0.0554	0.08856
52	40	0.0619	0.97349	40.4649	0.0529	0.08770
60	40	0.0588	0.96754	33.1172	0.0514	0.08529
68	40	0.0557	0.96186	28.2484	0.0495	0.082387

molecular layer [19]. On the other hand, the negative values of isosteric heat of sorption which occured between 0.62 and 0.70 g water/g dry solids, especially in pineapple slices preosmosed in 60°B and 68°B sucrose solutions, have been explained by the contribution of the endothermic dissolution of sugars in the sorbed water. This agreed with the conclusions of Saravacos et al.[21] and Lim et al.[12] on sultana raisins and freeze-dried blueberries. The initially high net sorption heat value, and its decrease to approach the latent heat of vapourization of pure water (ΔH_{st} =0) as moisture content increased show that the heat evolved during sorption of the first few layers of water molecules was substantially more than the heat of condensation of free water [12].

Table 1 shows the summarized GAB and BET parameters for osmo-dried pineapple at 20 °C and 40 °C. The GAB monolayer moisture was consistently greater than the BET monolayer moisture content. Both GAB and BET monolayers decreased with increasing temperature. The temperature dependence of the monolayer value has been linked [23] to a reduction in sorption active sites as a result of physicochemical changes induced by temperature [24]. Moreover, both GAB and BET monolayers decreased with increasing pretreatment concentration of sucrose solution. Increased solid gains with increased pretreatment osmotic solution concentration in osmo-dried pineapple reduced the monolayer moisture content.

In conclusion, adsorption isotherms of osmo-air dried pineapple slices showed a type III (J-shaped) isotherm characteristic of high sugar foods. Equilibrium moisture content increased with temperature up to $a_w \sim 0.86$. The inversion of the usual dependence of water sorption isotherms with temperature occurred at $a_w \sim 0.86$. Calculated net isosteric heat of sorption increased with increasing moisture content. Calculated GAB and BET monolayer moisture (M_m) contents decreased with increasing temperature.

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