Sorption characteristics of pectin isolated from Jerusalem Artichoke tubers (*Helianthus tuberosus L.*)

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Abstract

Introduction. The aim of the present study is the isolation of pectin from Jerusalem artichoke tubers (*Helianthus tuberosus L.*) and the analysis of its sorption characteristics

Materials and methods. Research was carried out on the pectin content of the tubers of Jerusalem artichoke plants cultivated in Bulgaria. The polyuronide content (PUC) was determined via the McCready method. The static gravimetric method was used for analysis of the sorption characteristics of pectins.

Results and discussion. The polysaccharide was extracted. The isolated pectins were analyzed in physical terms: the equilibrium sorption isotherms, belonging to type II in Brunauer's classification, were obtained experimentally. The entire isotherm length demonstrated statistically significant hysteresis. The Henderson and Chung-Pfost models provided adequate isotherm description. The pectin content of the three Jerusalem artichoke samples is 14.8, 9.2 and 11.9 % a.d.m., respectively. The monomolecular moisture content of pectin was within the 7.42 – 7.92% dry basis range, its corresponding water activity value – within the 0.14 – 0.16 range.

Conclusion. The results of research are advisable for use in develop of functional food ingredient which is used pectin as a gelling agent and a stabilizer.

Introduction

Pectins are carbohydrates possessing complex composition and structure. They belong to the acidic branched heteropolysaccrahides. Their major chain is constructed by linearly joined with (1→4)-glycosidic bonds α-D-galactopyranosyluronic acid residues, partially esterified with methanol. α-L-rhamnose with (1→2)-bonds is found among them. Various neutral sugars – D-xylose, D-glucose, D-mannose, L-fructose, etc. – occur in the chain branches [10, 17].

Pectin is a common component of the cell walls of all land plants [18]. Among the basic raw materials used in the manufacture of commercial pectin is apple and citrus fruit peel [12].

Pectin extraction is a multi-stage physico-chemical process taking place under the influence of a number of factors, notably temperature, pH and duration [11]. It has been studied by many researchers. El-Nawawi and Shehata [8] have analyzed the factors bearing upon pectin production during its isolation from orange peel, the results showing that the highest yield is achieved with hydrochloric acid as extractant, at 90 °C, pH 1.7 and 120-minute duration of extraction.

Pagán and Ibarz [13] have studied the production and rheological properties of peach pomace pectin demonstrating that the maximum yield is achieved with 70%-nitric acid, 80°C, pH 1.2 and 60-minute duration. After isolating and analyzing waste apple peel pectins, Virk and Sogi [16] have found out that citric acid is a more efficient extractant than hydrochloric acid. Rehmann et al. [14] have extracted mango peel pectin with the help of sulphuric acid, their results testifying to a maximum yield at 80°C, pH 2.5 and 120-minute duration of extraction. Our previous research features a description of Jerusalem artichoke pectins *(Helianthus tuberosus L.)* [1]. Our results manifest that in tuber pectin extraction maximum yield and purity are achieved with ammonium oxalate as extractant, at 85°C and 45-minute extraction.

In food industry, pectin has a long-established application as a functional food ingredient which is used as a gelling agent and a stabilizer. Scientific literature provides data on the physical and physico-chemical properties of this polysaccharide accounting for its functional and technological characteristics [2, 3]. The equilibrium isotherms of food products show the correlation between equilibrium moisture content and water activity at a given temperature. The sorption isotherms of the product reveal the manner in which water is bound to the solid skeleton. Scientific literature suggests a multitude of empirical and theoretical models for sorption isotherm description [5, 7]. Chen and Morey [7] draw the conclusion that there is no universally applicable model. In general, several models are used, the most adequate of which is opted for on the basis of specific criteria. Model evaluation criteria are usually mean relative error (P, %) and standard deviation (SEM) [4]. Quite a few studies prove that the products whose moisture content corresponds to monomolecular moisture can be stored for long periods of time with no changes to their technological properties [6].

The aim of the present study is the isolation of pectin from Jerusalem artichoke tubers (*Helianthus tuberosus L.*) and the analysis of its sorption characteristics.

Materials and methods

1. Raw materials.

This study is based on the analysis of Jerusalem artichoke tubers collected during the technological maturity of the plant (November, 2012) in three Bulgarian regions: the

territory of the city of Stara Zagora, Stara Zagora District (sample №1), the town of Parvomai, Plovdiv District (sample №2) and the town of Vidin, Vidin District (sample №3).

All reagents used in the analysis are p.a.

2. Determination of pectins.

The polyuronide content (PUC) was determined via the McCready method which we used earlier [1], as follows:

2.1 Preparation and washing of the raw material.

10 g preliminarily ground plant matter is weighed, to which 100 cm^3 of a 5 %-solution of hydrochloric acid and 70 %-ethanol is added and the mixture is stirred for 1 h with the help of an electromagnetic stirrer. Afterwards, it is filtered in a Büchner funnel and rinsed with 70 %-ethanol first (until a neutral reaction) and then with 96 %-ethanol. The substance is dried at 50°C.

2.2 Polyuronide Content (PUC) determination.

Two 2-gram samples (with a precision of ± 0.0001 g) of the rinsed material are weighed, to each of which 2.00 g of NaCl and 150 cm^3 of distilled water is added. The samples are stirred with an electromagnetic stirrer for 2 h, after which 50 cm^3 of distilled water is added to each of them. Two check samples are prepared. 4-5 drops of Hinton reagent are added to each sample, which is followed by titration with 0.1 n NaOH. 40 cm^3 of 0.1 n NaOH are added to each sample, the samples are then left undisturbed for 2 h, after which 50 cm^3 of 0.1 n H_2SO_4 is added to each of them. The remainder of the acid is titrated with 0.1 n NaOH.

The PUC (%) of the rinsed plant matter is calculated by the following formula:

$$PUC = \frac{V_1 \cdot F \cdot 0.01761 + V_2 \cdot F \cdot 0.01901}{m} \cdot 100\%$$

V_1 is the volume of NaOH spent in the first titration, cm^3;

V_2 – the volume of NaOH spent in the second titration, cm^3;

F – NaOH factor;

0.01761 – the amount of the non-esterified galacturonic acid residue corresponding to 1 cm^3 of 0.1n NaOH in g;

0.01901 – the amount of the esterified galacturonic acid residue corresponding to 1 cm^3 of 0.1n NaOH in g;

m – sample mass, g.

In order to determine the degree of esterification (DE, %), the following formula is used:

$$DE = \frac{V_2 \cdot F}{V_1 \cdot F + V_2 \cdot F} \cdot 100\%$$

3. Extraction of pectins.

2000 cm^3 of 85-90°C distilled water containing 18.8 g (0.075 mol/l) of ammonium oxalate is poured on 100 g of the rinsed plant material. The extraction of the mixture continues for 45 min, at 85°C, with regular stirring. It is filtered while hot, the filtrate volume is measured and the filtrate is left to cool at room temperature. 20 cm^3 of concentrated hydrochloric acid and an equal volume of 96 %-ethanol are added. The

mixture is stirred well and left undisturbed at room temperature for 2 h. The resultant gel is filtered, rinsed a few times with 70 %-ethanol until the elimination of all chloride ions, then rinsed twice with 96 %-ethanol and dried at 40°C to get constant weight.

4.Analysis of the sorption characteristics of pectins.

We used the static gravimetric method recommended for food products [6]. One-gram samples (with a precision of ± 0.005 g) are weighed in weighing dishes. The latter are placed in hygrostats over saturated solutions of seven salts (LiCl, CH_3COOK, $MgCl_2$, K_2CO_3, NaBr, NaCl, KCl) keeping the water activity of the product in the 0.11 – 0.85 range [9]. The hygrostats undergo tempering in a thermostat at 20 ± 0.1°C. When equilibrium is reached (within 20 - 30 days), sample moisture is determined by weighing, the samples being dried for 24 h at 105°C. The experimentally obtained data are average values of the results achieved after triplicate tests.

In order to describe sorption isotherms, we resorted to the two-parameter Chung-Pfost, Halsey, Oswin, and Henderson models [5, 7]:

Chung-Pfost:

$$\ln(a_W) = -Ae^{-BM} \tag{1}$$

Halsey:

$$a_W = -\exp^{AM^B} \tag{2}$$

Oswin:

$$M = B\left(\frac{a_W}{1-a_W}\right)^C \tag{3}$$

Henderson:

$$\ln(1-a_W) = -AM^B, \tag{4}$$

M is equilibrium moisture content, % dry basis; a_W – water activity, decimal; *A*, *B, C* – constants.

To determine the monomolecular moisture content, the wide-known Brunauer-Emmett-Teller (BET) model was used [15], valid for $a_W < 0{,}5$ [6]:

$$M = \frac{M_m C a_W}{(1-a_W)(1-a_W+Ca_W)}, \tag{5}$$

M_m is the monomolecular moisture, % dry basis; C - constant.

Results and discussion

Table 1 presents the results related to the polyuronide content (PUC) and the degree of esterification (DE) of pectins contained in Jerusalem artichoke tubers (*Helianthus tuberosus L*).

Table 1

Polyuronide content and degree of esterification of pectins in Jerusalem artichoke tubers *(Helianthus tuberosus L).*

№ in turn	PUC, % a.d.m.	DE, %
Sample №1	14.8	60.7
Sample №2	9.2	63.4
Sample №3	11.9	59.8

a.d.m. – absolute dry matter

It is evident that sample №1 is the richest in pectins. Therefore, this sample was chosen to isolate the polysaccharide from in order to determine its sorption characteristics.

The experimentally obtained pectin sorption isotherms are illustrated in Figure 1. It demonstrates that with low water activity the character of the isotherms is typical of monomolecular adsorption whereas with high water activity it is typical of polymolecular adsorption, i.e. the isotherms possess the characteristic S-shape of Brunauer's II type [6]. Such sorption isotherms are common for many colloid capillary porous products and most foods are such products. The hysteresis effect is statistically significant (ά=0.05) along the entire length of the isotherm and the highest for higher water activity values (over 0.6), reaching 2.5% dry basis. With lower water activity, hysteresis values amount to about 1% dry basis on average.

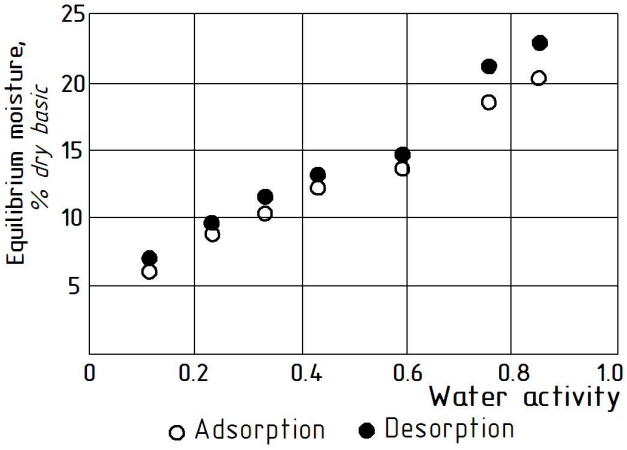

Fig. 1. Equilibrium isotherms of pectin

Table 2

Coefficients of the models (*A*, *B*), mean relative error (*P*,%) and standard deviation (*SEM*) for desorption

Model	*A*	*B*	*P*	*SEM*
Chung-Pfost	6,398093	0,1566	3,36	0,84
Oswin	0,3169	13,85852	4,45	0,94
Halsey	172,2075	-2,1395	7,55	1,66
Henderson	0,001587	2,2702	3,43	0,92

Table 3

Coefficients of the models (*A*, *B*), mean relative error (*P*, %) and standard deviation (*SEM*) for adsorption

Model	*A*	*B*	*P*	SEM
Chung-Pfost	6,86956	0,1793	3,79	0,61
Oswin	0,3155	12,5059	4,94	0,84
Halsey	134,5317	-2,1284	8,34	1,69
Henderson	0,001937	2,2838	2,56	0,58

The coefficients of the linear equations were determined on the basis of the Least Squares method. The coefficient values, mean relative error P and standard deviation SEM of the models from (1) to (4), for desorption and adsorption, respectively, are presented in Tables 2 and 3. The results obtained show that in adsorption the Henderson model is most suitable for sorption isotherm description (the lowest values of P and SEM) while in desorption the most adequate model is that of Chung-Pfost. However, since the differences in the P and SEM values for both models and both processes are minimal, both models can be recommended as equally adequate for the description of pectin sorption isotherms.

In order to calculate the monomolecular moisture content, equation (5) can be transformed into a linear form:

$$\frac{a_W}{M\left(1-a_W\right)}=\frac{1}{MmC}+\frac{\left(1-C\right)a_W}{MmC} \tag{6}$$

On the basis of the slope of the line, using the Least Squares method, one can determine the coefficients of the linear equation (6) and, hence, the monomolecular moisture M_m and the C coefficient. The linear dependence $a_W/\left[M\left(1-a_W\right)\right]=f\left(a_W\right)$, with the experimental data for desorption and adsorption for $a_W < 0.5$, is illustrated in Fig. 2. The monomolecular moisture values obtained and the correlation coefficients are given in Table 4.

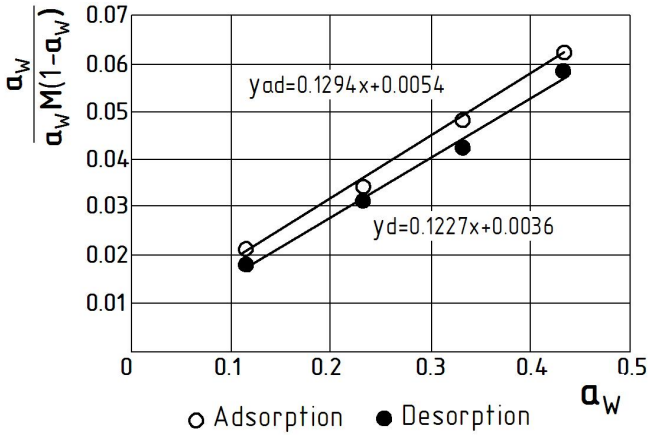

Fig 2. Dependence $a_W/\left[M\left(1-a_W\right)\right]=f\left(a_W\right)$ for pectin

Table 4

Monomolecular moisture values (M_m), correlation coefficients (R^2) and water activities corresponding to the monomolecular moisture content (a_{wm})

Pectin	M_m	R^2	a_{wm}
Desorption	7,92	0,9936	0,144
Adsorption	7,42	0,9958	0,16

The results demonstrate that the monomolecular moisture content of pectin is from 7.42 to 7.92 % dry basis, the hysteresis effect still occurring, while the value for desorption is higher.

The BET model makes it possible to determine the product's water activity at which it is to be stored in order to preserve its monomolecular moisture:

$$a_{wm} = \left(\sqrt{C} - 1\right) / \left(C - 1\right) \tag{7}$$

The results obtained for a_{wm} are given in Table 4. They show that if pectin is to have moisture content approximating monomolecular moisture, it should be stored at water activity within the 0.14 – 0.16 range.

Conclusion

The pectin content of the three Jerusalem artichoke samples is 14.8, 9.2 and 11.9 % a.d.m., respectively. The experimentally obtained equilibrium sorption isotherms of the pectin isolated from sample №1 belong to type II according to Brunauer's classification. The entire isotherm length manifests statistically significant hysteresis. The Henderson and Chung-Pfost models have been found to be adequate for isotherm description. The monomolecular moisture content of pectin is between 7.42 and 7.92 % dry basis, the corresponding water activity being between 0.14 and 0.16.

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