417
Annals
Agric. Sci.,
Ain Shams Univ.,
Cairo, 54(2), 417- 423, 2009

PRODUCTION OF INULIN AND HIGH-FRUCTOSE SYRUP FROM JERUSALEM ARTICHOKE TUBER (*Helianthus tuberosus* L.)

[32]

Safaa, S. Abozed[1]; A. Abdelrashid[2]; M. El-kalyoubi[2] and K.I. Hamad[1]

1- Food Technology Dept., National Research Center, Dokki, Giza, Egypt
2- Food Science Dept., Faculty of Agric., Ain Shams Univ., Shoubra El-Kheima, Cairo, Egypt

Keywords: *Helianthus tuberosus*, Inulin extraction, Acid hydrolysis, High-fructose syrup

ABSTRACT

Jerusalem artichoke (*Helianthus tuberosus* L.) has been reported to have one of the highest carbohydrates yield. The main carbohydrate component in Jerusalem artichoke tubers is inulin, with high fructose content (about 94%). Inulin is a fructose polymer which has been widely investigated for the production of high-fructose syrup by acid or enzymatic hydrolysis. In this work, a process optimization study to extract of inulin at different temperatures, time and solvent solid ratio were used. The optimal conditions for maximum inulin extraction yield were (68.70 %) at 85°C, 60min and water solid ratios 1:20 (v/w). Precipitation of inulin by four solvents (ethanol, propanol, acetone and acetonitrile) at different ratio 1:1, 2:1, 3:1 and 4:1(solvent: supernatant v/v) were studied. Inulin precipitation by ethanol and acetone were more than that occurred using acetonitrile or propanol. The influence of pH, type of acid, temperature and time on hydrolysis of inulin were investigated. The complete hydrolysis of the inulin was attained at pH 2.0, adjusted with sulphuric acid after 90min at 100°C.

INTRODUCTION

Helianthus tuberosus L. (Asteraceae), a perennial plant commonly known as the Jerusalem artichoke. The tubers of *H. tuberosus* have been utilized not only as a food but also as a raw material in the bioethanol industry for its high content of inulin, a fructan that can be easily hydrolyzed (**Pan *et al* 2009**). Inulin acts as a dietary fiber, contributing to the growth of bifidobacteria and to the improvement of the overall conditions in the human gastrointestinal (**Ritsema and Smeekens, 2003**).

Food and pharmaceutical industries have found applications for inulin in the production of functional foods, nutritional composites and medicines. The most stable form for the commercialization of inulin is the powdered extract. This type of product has more advantages for its greater facility of manipulation, transport, storage and consumption (**Molina *et al* 2005**).

The application of inulin in the food industry, at first, was restricted to the production of drinks similar to coffee, for its bitter taste. However, it was recently discovered that the inulin could act as a substitute for sugar or fat due to its very low caloric value (**Van Loo *et al* 1995**). So, inulin was used as an ingredient in foods with reduced or no sugar and fat, such as chocolates, ice creams and yoghurts, among others also. Also, it was used in low caloric foods, reduced fat levels, and so source of dietary fibers, contributing to the improvement of the gastrointestinal system conditions (**Figueira *et al* 2004**).

Inulin is a polysaccharide, with high fructose content reaching 94% (**Saengthongpinit and Sajjaanantakul, 2005**). High fructose syrup is a sweetener found in numerous foods and beverages (starting from bread to pasta sauces to bacon to beer) as sugar substitute because of its high quality characteristics and stability easy to store

(Received October 15, 2009)
(Accepted October 22 , 2009)

(Zhang *et al* 2004 and Rocha *et al* 2006). High fructose syrup can be produced by the hydrolysis of a natural occurring fructan, inulin, which is a polymer of β-(2→1) linked fructose, to yield 95% fructose. **(Barta, 1993).**

The aim of this investigation was to develop a set of optimum extraction conditions for Jerusalem artichoke tubers in order to improving inulin extraction yield. Based on previous work, temperature, extraction period and solvent: solid ratio were considered to be important factors affecting the extraction yield, and precipitate inulin by different concentrations of ethanol, acetonitril,acetone and propanol. Furthermore, inulin utilization for production of high fructose syrup by some parameters of inulin hydrolysis was tested to obtain the highest reducing sugars yield from the selected tubers.

MATERIALS AND METHODS

Material

Jerusalem artichoke tuber (*Helianthus tuberosus* L.) was purchased from the Agriculture Research Center, Dokki, Giza. , Egypt.

Methods

Jerusalem artichoke tubers were washed with tap water and any deteriorated parts were removed, than the tubers were sliced in dividedly to the reasonable thickness by a conventional food slicing machine. The sliced tubers were immersed immediately in boiling water for 5 min, following by immediate dipping in cold acetic acid solution (2%) to inhibit polyphenoloxidase activity **Tchone *et al* (2005)**. After that the slices of tuber were dried in electronic air oven (SHEL LAB 1370FX SHELDON MANUFACTURI Ng, 1NC) at 60-70°C until samples reached constant weight.

Chemical composition

Proximate chemical compositions were determined in dried powdered tubers according to **A.O.A.C. (2000).** Inulin was measured by using the method of **Witon and Witon (1958).**

Inulin extraction

The extraction of inulin was carried out using hot water according to **Lingyun *et al* (2007)** method. The dried powdered tubers were mixed with water at different percentage of powder tubers / water ratio i.e., 1:2.5, 1:5, 1:10, 1:15 and 1:20 (W/V) at different temperatures 65, 75, 85 and 95°C as well as for different periods 40, 50, 60 and 70 min.

Inulin precipitate

Inulin was precipitated according to **Ku *et al* (2003)** method. Four different solvents (ethanol, propanol, acetone and acetonitrile) were used at different ratios of solvent to test sample as follows (1:1, 2:1, 3:1 and 4:1, v/v). The inulin powder was analyzed from moisture, ash, fat, and reducing sugar as the method described in the **A.O.A.C. (2000).**

High fructose syrup production

Sulphuric and hydrochloric acids were used to adjust the pH of the inulin extract to 2.0, 2.5 and 3.0 for each acid. The samples were heated at 60° C, 80°C and 100°C. The effect of hydrolysis was followed by assaying reducing sugars increase after 60 and 90 min for each pH, acid and temperature, however, reducing sugars were estimated according to **A.O.A.C. (2000).**The product was decolorized with activated granular charcoal, after that the liquid was passed through Dowex 66 and concentrated by rotary evaporation to produced high fructose syrup.

Statistically analysis

The mean values of the all obtained results were statistically analysis. Evaluated by one-way analysis of variance (ANOVA) and least significant difference at 0.05 % level of probability using PC–Stat Version IA procedures **(PC- Stat, 1985).**

RESULTS AND DISCUSSION

Chemical composition

Proximate chemical composition of Jerusalem artichoke tubers are summarizes on dry weigh basis as shown in **Table (1)**. The tuber contained 6.17, 6.20, 0.58, 5.33, 2.72, 79.01 and 72.18 % moisture, protein, fat, ash, crude fiber, total carbohydrate and inulin content, respectively. These indicate that a Jerusalem artichoke tuber was found to be higher content of total carbohydrate and a good source for inulin **(Barta, 1993).**

Table 1. Proximate chemical composition of Jerusalem artichoke tuber (g/100g on dry weight basis %)

Constituents (%)	Jerusalem artichoke tuber
Moisture	6.17
Crud protein	6.20
Crude fat	0.58
Ash	5.33
Crude fiber	2.72
*Total carbohydrate	79.01
Inulin	72.18

*Calculated by different

Inulin extract

The effect of using different extraction methods on % of inulin are shown in **Table (2)**. From the obtained data, it could be noticed that, the percentage of extractable inulin was significantly increased as a function of increasing solvent to sample ratio when sample were statistically analyzed at $p \leq 0.05$.

The extraction conditions to recover the highest yield of inulin were 1:15 for 70 min at 75°C which produced 63.80 % of inulin, while the extraction yield at ratio 1: 20 for 60 min at 85°C was 68.71% of inulin. On the other hand, the extraction process using ratio 1:10 for 60 min at 95 °C produced 67.77% of inulin. Previous data showed that their proportion relation between extract of inulin and extraction period, temperature and water solid ratio which are agree with **Leite *et al* (2007)**. The optimum conditions for maximizing inulin extraction yield (68.71 %) were at natural pH for 60min. at 85°C and water solid ratio of 1:20 (v/w).

Inulin precipitate

The weigh percent of inulin (g/100g) precipitated using the four presentation agents at different ratios are listed in **Table (3)**. In general, the weight percent of precipitation by ethanol was the highest one among other solvents and the inulin precipitate increased significantly with increasing solvent ratio. In addition, the table cleared that ethanol and acetone were the best agents to precipitate the inulin from the tuber, however, the precipitation percents were 70.25 and 68.87, respectively. On this regard, **Ku *et al* (2003)** reported that acetone is the potent solvent for precipitation of inulin.

Chemical composition of precipitated inulin

Table (4) showed that the chemical composition of Inulin precipitate was 3.77, 1.59, 0.39, 95.12 and 1.67 % of moisture, ash, fat, inulin and reducing sugar content respectively these results are an agreement with that observed by **Saengthongpinit and Sajjaanantakul (2005)**.

High fructose syrup production

In this manner, the acid hydrolysis was carried out using different four parameters: acid, pH, temperature and hydrolysis period, **Table (5)**. From this table it had been shown that the content of reducing sugars was increased with time increase at different acid, pH values and temperatures. Furthermore, obtained results proved that pH value of 2.0 adjusted with sulphuric acid gave the best results after 60 or 90 min. reaching 81.68 and 83.54 %, respectively, of reducing sugars at 100°C compared with hydrochloric acid at the same conditions as shown in **Table (5)**.

The presented results have indicated that the highest of inulin percentage were observed by acid hydrolysis at pH 2.0 and 100°C for 90 min. However, efficiency of inulin conversation to reducing sugars were obtained using the sulphuric acid as a hydrolyzing agent **(Figure 1)** these evidences are in agreement with that obtained by **Szambelan and Nowak (2006)**. After filtration, the product was decolorized with activated granular charcoal, neutralized to pH 6.5-7.0 by passage through anion exchange resin (Dowex 66), and concentrated to 78.4 % soluble solids by evaporation to produce high fructose syrup.

Table 2. Effect of using different solid / water ratio, and extraction period on the inulin yield at different temperatures

Solid/water ratio (w/v)	Extraction periods (min)			
	40	50	60	70
	65°C			
1:2.5	22.08 [d]	27.17 [d]	33.88 [c]	37.54 [c]
1: 5	23.81 [bc]	31.14 [c]	37.92 [c]	39.38 [bc]
1:10	25.95 [ab]	33.27 [b]	38.15 [b]	40.29 [bc]
1:15	28.08 [b]	34.49 [b]	43.65 [a]	43.96 [a]
1:20	34.19 [a]	38.45 [a]	42.73 [a]	41.51 [ab]
	75°C			
1:2.5	42.12[c]	47.01[c]	49.14[c]	52.52[d]
1: 5	43.35[c]	49.14[bc]	51.29[bc]	57.69[c]
1:10	46.09[b]	51.28[ab]	53.39[ab]	61.97[ab]
1:15	50.48[a]	54.03[a]	56.17[a]	63.80[a]
1:20	52.19[a]	52.19[ab]	54.64[a]	60.74[b]
	85°C			
1:2.5	60.74[b]	61.97[d]	61.36[c]	62.88[c]
1: 5	61.05[b]	64.10[c]	65.93[b]	67.15[ab]
1:10	62.88[ab]	67.46[a]	67.16[ab]	68.07±[a]
1:15	64.40[a]	66.54[ab]	68.38[a]	66.85[ab]
1:20	64.10[a]	65.02[bc]	68.71[ab]	65.02[bc]
	95°C			
1:2.5	61.36[c]	62.27[c]	60.13[b]	61.05[c]
1: 5	62.27[bc]	63.19[c]	66.54[a]	65.93[ab]
1:10	64.71[a]	65.69[bc]	67.77[a]	67.46[a]
1:15	63.79[ab]	67.15[a]	67.15[a]	65.93[ab]
1:20	61.97[bc]	65.93 [bc]	65.63[a]	64.10[bc]

Each value was an average of three determinations
Means which are not significantly different are followed by the same number in column, Significance level = 0.05)

Table 3. Mean values of weight percent of inulins precipitated using four agents at different solvent / extract ratios

Solvent / extract ratio	Inulin weight %(g/100g)			
	Ethanol	Propanol	Acetone	Acetonitril
1:1	28.34 [a]	17.49 [d]	24.03 [b]	21.15[c]
2:1	47.58 [a]	33.25 [d]	45.93 [b]	41.20 [c]
3:1	62.93 [a]	48.82 [d]	60.71 [b]	54.70 [c]
4:1	70.25 [a]	62. 82 [d]	68.87 [b]	65.21 [c]

Each value was an average of three determinations
Means within a row showing the same small letter are not significantly different ($P \geq 0.05$)

Table 4. The Chemical composition of precipitated inulin (g/100g on dry weight basis %)

Constituents (%)	Proximate composition
Moisture	3.77
Ash	1.59
Fat	0.39
Inulin	95.12
Reducing sugar	1.67

Each value was an average of three determinations

Table 5. The reducing sugars content (%) in inulin extract, after hydrolysis with different acids for 60 and 90 min at different temperature

pH	acid	Hydrolysis temperatures		
		60°C	80°C	100°C
		Extraction period for 60 min		
2.0	Sulphuric	57.51[d]	73.73[a]	81.68[a]
	Hydrochloric	41.85[c]	52.92[c]	71.94[b]
2.5	Sulphuric	55.39[b]	63.22[d]	66.46[c]
	Hydrochloric	40.39[c]	52.36[c]	65.04[c]
3.0	Sulphuric	39.64[cd]	51. 24[c]	53.26[d]
	Hydrochloric	35.47[d]	47.66[d]	51.47[e]
	Extraction period for 90 min			
2.0	Sulphuric	65.51[a]	76.19[a]	83.54[a]
	Hydrochloric	64.73[ab]	64.89[c]	72.50[b]
2.5	Sulphuric	61.73[b]	69.43[b]	72.23[b]
	Hydrochloric	59.24[c]	62.77[d]	62.09[c]
3.0	Sulphuric	48.34[d]	47.26[f]	62.83[c]
	Hydrochloric	42.67[e]	48.20[e]	53.82[d]

Each value was an average of three determinations
Means witch are not significantly different are followed by the same number in column, (Significance level = 0.05)

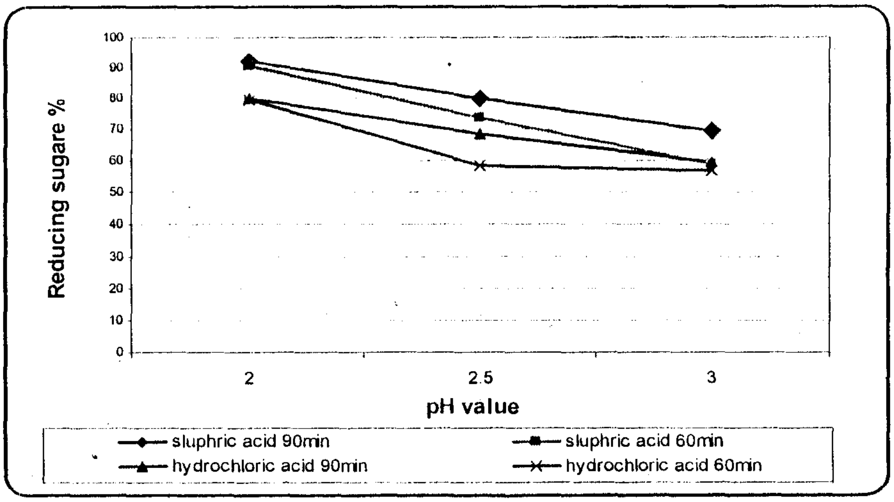

Figure 1. Effect of pH values and different acids on inulin hydrolysis at 100°C

REFERENCES

A.O.A.C. (2000). Association of Official Analytical Chemists. **Official Methods of Analysis 17th Ed. Gaithersburg, Maryland, USA.**

Barta, J. (1993). Jerusalem artichoke as a multipurpose raw material for food products of high fructose or inulin content, **pp. 323-339**. *In*. Fuchs, A.ed. **Inulin and Inulin-containing Crops**. Elsevier, Amsterdam.

Figueira,M. G.; J.K. Park; R.P.F. Brod and L.S. Honorio (2004). Evaluation of desorption isotherms,drying rates and inulin concentration of chicory root (*cichorium intybus* L.) with and without enzymatic inactivation. **J. Food Engin. 63: 273-280.**

Ku, Y.; O. Jansen; J.C. Oles; Z.E. Lazar and I.J. Rader (2003). Precipitation of inulins and oligoglucoses by ethanol and other solvents. **Food Chem. 81: 125–132.**

Leite, J.T.C.; F.E. X. Mürra; P. Martinelli; I.M.F. Dal and K.J. Park (2007). Optimization of a physical concentration process for inulin. **J. Food Engin. 80: 832-838.**

Lingyun, W.; W. Jianhua; Z. Xiaodong; T. Da; Y. Yalin; C. Chenggang; F. Tianhua and Z. Fan (2007). Studies on the extracting technical conditions of inulin from Jerusalem artichoke tuber. **J. Food Engin. 79: 1087-1093.**

Molina, D.L.; M.D.N. Martınez; F.R. Melgarej; A.N.P. Hiner; S. Chazarra and J.N.R.G. Lo´pez (2005). Molecular properties and prebiotic effect of inulin obtained from artichoke (*Cynara scolymus* L.). **Phytochemistry. 66: 1476–1484.**

Pan, L.; M.R. Sindon; A.H. Kennedy; H. Chai; L.E. Watson; T.L. Graham and A.D. Kinghorn (2009). Bioactive constituents of *Helianthus tuberosus* (Jerusalem artichoke). **Phytochemistry Letters; 2: 15–18.**

PC-Stat, (1985). Version IA, Copyright 1985 - The University of Georgia,USA.

Ritsema, T. and S. Smeekens (2003). Fructans: beneficial for plants and humans. Current **Opinion in Plant Biology, 6: 223–230.**

Rocha, J.R.; R. Catana.; B.S. Ferreira; J.M.S. Cabral and P. Fernandes (2006). Design and characterisation of an enzyme system for inulin hydrolysis .**Food Chem. 95: 77–82.**

Saengthongpinit, W. and T. Sajjaanantakul (2005). Influence of harvest time and storage temperature on characteristics of inulin from Jerusalem artichoke (*Helianthus tuberosus* L.) tubers. **Posth. Biology Techn. 37: 93-100.**

Szambelan, K. and J. Nowak (2006). Acidc and enzymatic hydrolysis of jerusalem artichoke (*Helianthus tuberosus* L.) Tubers for further ethanol production. **Food Sci. Techn. 9(4): 38-41.**

Tchone, M.; G. Barwald and C. Meier (2005). Polyphenoloxidas in jerusalem artichoke (*Helianthus tuberosus* L.). **Biritsh. Food J. 107(9): 693-701.**

Van Loo, J.; P. Coussment; L. Leenheer; H. Hoebregs and G. Smits (1995). On the presence of inulin and oligofructose as natural ingredients in the western diet. **Critical Reviews in Food Sci. Nutr. 35(6): 525–552.**

Witon, A.L. and K.B. Witon (1958). The Analysis of Food. pp. 857-859. John Wiley and Sons, Inc., London.

Zhang, Y.; K. Hidajat and A.K. Ray (2004). Optimal design and operation of SMB bioreactor: production of high fructose syrup by isomerization of glucose. **Biochem. Engin. J. 21: 111–121.**

٤٢٣
حوليـــات العلـــوم الزراعية
جامعة عين شمس ، القاهرة
مجلد(٥٤)، عدد (٢)، ٤١٧-٤٢٣، ٢٠٠٩

إنتاج الإنيولين وشراب الفركتوز عالى التركيز من درنات الطرطوفة

[٣٢]

صفاء سيد ابوزيد أحمد[١] - علاء عبد الرشيد محمد[٢]- ممدوح حلمى عثمان القليوبى[٢] - كمال الشناوى ابراهيم حمد[١]

١- قسـم الصناعـات الغذائيـة - المركـز القومـي للبحـوث - الدقى- الجيزه- مصر
٢- قسم علوم الاغذية- كلية الزراعه - جامعة عين شمس- شبرا الخيمة- القاهرة- مصر

الموجـــز

الطرطوفة تعد من أحد أفراد العائلة المركبة التى تتميز بتخزينها العالى للكربوهيـدرات متمثـل فـى الإنيولين الذى يتميز بارتفاع محتواه مـن الفركتـوز حيث يصل الى ٩٤% , الإنيولين عبارة عن سلسلة مستقيمة مكونة من وحدات من البيتا فركتوز وتنتهى السلسلة بوحدة الجلوكـوز. تـم دراسـة التركيـب الكيماوى لدرنات الطرطوفة حيث أظهرت النتـائج إرتفاع محتوى الدرنات من الكربوهيدات الكلية وكذلك الإنيولين حيـث وصـلت ٧٩,٠١ و ٧٢,١٨ علـى التوالى . وتم دراسة تاثير كلاً من درجة الحـرارة والوقت وكذلك نسبة الماء الى درنات الطرطوفة على كفاءة عملية الإستخلاص وذلك لتعين أنسب الظروف الملائمة لإسـتخلاص الإنيـولين حيـث وجـد أن الإستخلاص على ٨٥°م لمدة ٦٠ دقيقة أعطى أعلى إنتاج للإنيولين المستخلص الذى وصلت نسبته الى ٦٨,٧١ % وكذلك تم ترسيب الإنيولين بـأربع مـذيبات مختلفة وهى الإيثانول و البروبـانول و الأسـيتون و الأسيتونيترل حيث كان أفضل هذه المذيبات الإيثانول يليه الأسيتون ثم الأسيتونيترل وأخيراً البروبانول عند نسبة ٤:١. وقد وجـد إمكانيـه تحليـل مـستخلص الإنيولين بالحامض إلى شـراب الفركتـوز عـالى التركيز ٧٨,٤% مواد صلبة كلية وذلـك عنـد (pH) ٢ وحرارة ١٠٠ °م و لمدة ٩٠ دقيقة .

تحكيـم: أ.د رمضان محمد محمود
أ.د زكريا صالح الشامى