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Effect of pH on the Acid Hydrolysis of Jerusalem Artichoke Inulin

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ABSTRACT

The effect of pH on the simultaneous dehydration of fructose and hydrolysis of the Jerusalem artichoke inulin was investigated. Between pH 1 and 2 the extent of dehydration of fructose varied linearly with time and pH but was minimal at pH 2. The complete hydrolysis of the Jerusalem artichoke inulin was therefore attained at pH 2 after 2·5 h and at 100 °C with minimum fructose dehydration.

INTRODUCTION

The acid hydrolysis of inulin has been investigated utilizing sulphuric (McGlumphy *et al.*, 1931; Ohira & Kobayasi, 1949; Conti, 1953; Guiraud & Galzy, 1981), hydrochloric (Hoche, 1926; Arsem, 1927*a*; McGlumphy *et al.*, 1931) and some organic (Schering, 1927; Arsem, 1927*b*; Sandkuhl & Hulbach, 1957; Grandel & Neuman, 1958) acids. The conditions of hydrolysis varied, depending on the author (pH, 1–4; temperature, 60–100 °C; hydrolysis time, 5 min to several hours). From these studies it is not easy to identify optimal conditions for the hydrolysis of native inulin because data that characterize the final product are lacking. Drastic conditions (low pH, high temperature) may decrease hydrolysis time and lead to total hydrolysis, but they negatively influence

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the final product yield due to the acid-heat sensitivity of the fructose molecule. It is known (Estford, 1958; Kuster & Temmink, 1977; Fleming & Grootwassink, 1979) that fructose is more easily decomposed at high temperature and in an acidic medium than glucose, but gentle hydrolysis conditions prolong the conversion time and do not accomplish the total hydrolysis (Guiraud & Galzy, 1981). Therefore, the aim of this paper is to investigate the simultaneous effect of pH on the fructose molecule (dehydration) and on the degree of native inulin hydrolysis using hydrochloric acid as a hydrolyzing agent.

MATERIAL AND METHODS

Estimation of fructose sensitivity to pH

Fructose solutions were prepared by placing 2·5 g of D-fructose in a 100-ml volumetric flask and dissolving it in HCl solutions of pH 1, 1·25, 1·50, 1·75, 2, 3 and 4. The solutions were heated at 100 °C for 5 h; 10-ml aliquots were removed at various time intervals, neutralized with NaOH solution and diluted with twice-distilled water in a 25-ml volumetric flask.

Preparation of Jerusalem artichoke extracts

Freshly prepared artichoke tuber portions (100 g) were suspended in 150 ml of hot water (about 90 °C) and disintegrated with an Ultra-Turrax homogenizer. After passing the suspension through a Bücher funnel, the residue was washed with 2 × 150 ml of hot water in a 500-ml volumetric flask.

Hydrolysis procedure

A 100-ml aliquot of artichoke extract was adjusted with diluted HCl (0·1 mol/litre) to the desired pH (2 or 3) and then heated at 100 °C with reflux. Aliquots of 10 ml were removed at various times and neutralised with NaOH (0·1 mol/litre). They were then filtered and diluted with twice-distilled water in a 25-ml volumetric flask. After passing 4 ml of the hydrolysate through a C_{18} Sep Pak (Waters Associates), the first 2 ml were discarded and the next 2 ml were used for chromatographic analysis.

Liquid chromatography

A Waters Associates liquid chromatograph Model ALC/GPC 244, equipped with a model 440 UV detector and a R 401 differential refractometer, was employed. The chromatograms were obtained with a Model M 730 Data Module. The column system consisted of a 3·9 mm inside diameter × 4·0 cm precolumn packed with C_{18} Porasil B and a 3·9 mm inside diameter × 30 cm column packed with μBondapak/carbohydrate packing (Waters Associates). The mobile phase was acetonitrile–water (85:15 v/v). The flow rate was 1·4 ml/min and the column temperature was maintained at 25 °C. For recovery studies of fructose and glucose the external standard method was applied. For fructose stability studies and artichoke extract analysis, 20-μl samples were injected into the unit.

RESULTS AND DISCUSSION

The effect of pH on the dehydration of fructose at 100 °C and atmospheric pressure is illustrated in Fig. 1. The dehydration of fructose is most noticeable below pH 2. The extent of dehydration of fructose, at a given pH, is linear with time and increases with decreasing pH (Fig. 1a). At pH 1, after 5 h of treatment, the initial fructose content (100 %) falls to 48·5 %. Between pH 1 and 2 (Fig. 1b) the fructose content at various times varies linearly with pH. At pH 2 and higher no significant dehydration of the

TABLE 1
The Fructose and Glucose Contents in the Varieties of the Jerusalem Artichoke after Hydrolysis

Variety	*Content in relation to the fresh tuber*		
	Fructose (%)	*Glucose* (%)	*Total* (%)
Bela	11·5	3·80	15·3
Bianca	14·4	3·06	17·5
Topianca	17·4	3·05	20·4
Ozor	17·8	3·51	21·2
Violet de Rennes	18·0	3·32	21·3
Violet Communes	20·4	1·78	22·2

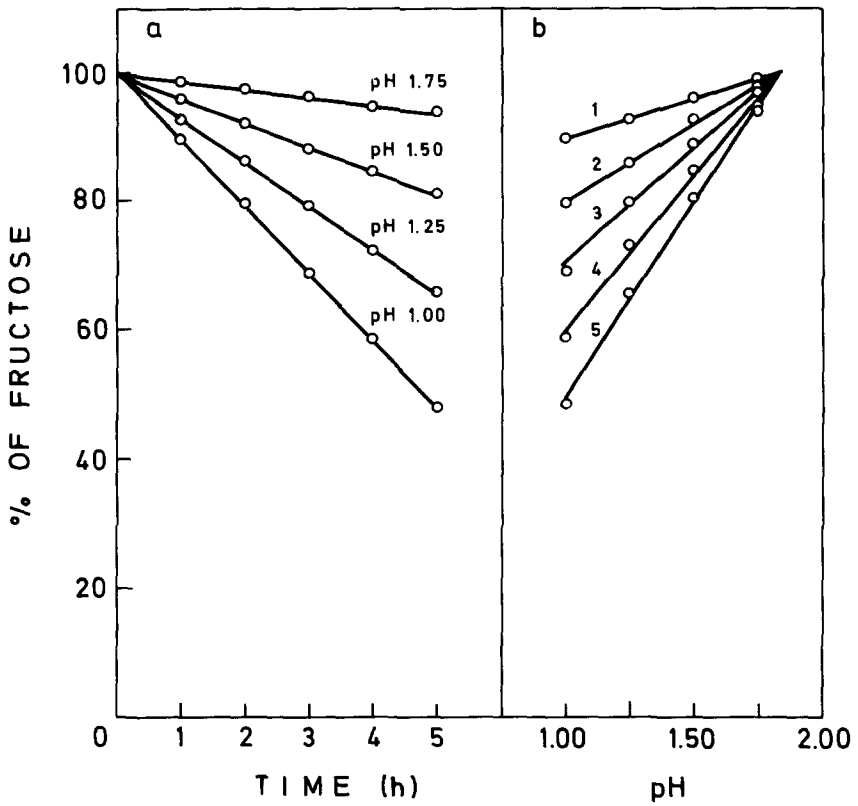

Fig. 1. The effect of pH and time (in hours) on the dehydration of fructose at 100 °C. (a) The relationship between the fructose content and time at defined pH. (b) The relationship between the fructose content and pH at defined time (arabic numbers).

fructose molecule was observed. Therefore, the hydrolysis of artichoke inulin was carried out at pH 2 and 3. At pH 2, 0·5 h was needed for 95 % hydrolysis of artichoke inulin. Guiraud & Galzy (1981), with sulphuric acid as the hydrolyzing agent, obtained about 62 % hydrolysis. Complete hydrolysis was attained after 2·5 h. At pH 3, 5 h were needed for 98·5 % hydrolysis. Guiraud & Galzy (1981) obtained about 80 % hydrolysis with sulphuric acid. Therefore, hydrochloric acid seems to be a more efficient hydrolyzing agent than sulphuric acid. Using optimal conditions (pH, 2; hydrolyzing temperature, 100 °C; hydrolyzing time, 2·5 h), we determined the carbohydrate content of the tubers of several varieties of the Jerusalem artichoke at full maturity (Table 1). These data indicate that the content of carbohydrate in the Jerusalem artichoke broadly varies

(from 15·3% in the Bela variety to 22·2% in the V. Communes variety). Also, the fructose/glucose ratio increases from the Bela to the V. Communes varieties, showing that the polymerization degree of polyfructosan in the Jerusalem artichoke tubers depends on the variety.

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