

THE EFFECT OF pH, TEMPERATURE AND HEATING TIME ON INULIN CHEMICAL STABILITY

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Background. Inulin is a storage carbohydrate found in many plants especially in chicory root, Jerusalem artichoke and dahlia tuber. It is a prebiotic with many functional properties. In earlier research concerning chemical stability of inulin, the effect of pH on rheological properties of inulin gels was mainly analysed. In these studies, the effect of time, temperature and pH on inulin chemical stability was not analysed profoundly especially considering the inulin concentrations unable to form gel structure. Thus, the aim of this work was to study the effect of the above mentioned factors on inulin chemical stability in water solution.

Material and methods. 5% (w/w) inulin solutions at pH 1-12 were heated at 20, 40, 60, 80 and 100°C for 5-60 min. After the neutralisation the content of reducing sugar was analysed according to Miller's method (1959) with 3,5-dinitrosalicylic acid.

Results. The conducted studies showed that inulin chemical stability at $\text{pH} \leq 4$ decreased with an increase of heating time and temperature. In a neutral and basic environment inulin was chemically stable regardless of heating time and temperature.

Conclusions. Inulin application in food systems may be limited in acidic products especially when heated above 60°C during the production process. However, in products at $\text{pH} \geq 5$, the degradation of this fructan does not occur even at thermal processing.

Key words: inulin, hydrolysis, temperature, pH

INTRODUCTION

Inulin is a polyfructan industrially produced from chicory, though as a storage carbohydrate it is also found in big amounts in Jerusalem artichoke and dahlia tubers. Inulin is a chain of fructose molecules connected by β -(2-1) glycosidic bonds, terminated by one glucose molecule. The number of fructose molecules in an inulin chain varies from few to many tens [Niness 1999].

Nowadays inulin may be purchased in several different forms. The most common are: native inulin with an average degree of polymerization (DP) of 10-12, inulin containing short chain fractions (with DP from 2 to 10) and a high performance (HP) inulin with an average DP no less than 23.

Recent big interest in inulin results from its prebiotic properties. Inulin because of its chemical structure is not decomposed by human digestive enzymes however, it is a perfect carbon source for health-promoting prebiotic bacteria. As not hydrolysed in human gastrointestinal tract, inulin is ranked as a soluble fibre [Gibson and Roberfroid 1995].

Besides pro-healthy properties inulin has miscellaneous functional properties. One of the most important is its fat mimetic property which is successfully used in the production of mayonnaise [Bortnowska and Makiewicz 2006], margarine [Rooyackers et al. 1994], processed meats [Florowski et al. 2010, Hadron et al. 2007] or yogurts with decreased calorificity and full fat taste [Güven et al. 2005]. Inulin was also applied in bread [Brien et al. 2003], pasta [Brennan et al. 2004] and ice-cream [El-Nagar et al. 2002] production.

Recently, the effect of environment reaction on inulin chemical stability has been analysed in a few studies [Florkowska et al. 2004, Kim et al. 2001, Glibowski and Wasko 2008]. Authors of these studies focused mostly on the effect of pH on rheological properties of the gels formed by inulin. Inulin can form a gel when its concentration is above 15% (w/w). In the quoted studies authors concluded that in the acidic environment gelation properties of inulin were deteriorated. However, the effect of time, temperature and pH on inulin chemical stability was not analysed profoundly, especially considering the inulin concentrations unable to form gel structure. Thus, the aim of this work was to study the effect of the above mentioned factors on inulin chemical stability in water solutions.

MATERIAL AND METHODS

The studied material was inulin Frutafit® TEX! kindly delivered by Sensus Operations C.V. (Roosendaal, The Netherlands). All other chemicals like sodium hydroxide, hydrochloric acid, 3,5-dinitrosalicylic acid (DNS), potassium sodium tartrate, sodium sulphate, phenol and fructose were of analytical grade supplied by POCH Katowice SA (Katowice, Poland).

5% (w/w) inulin solutions were prepared by mixing inulin with distilled water (20°C) using a MS 11HS magnetic stirrer (Wigo, Piastów, Poland). The pH of the inulin solutions was subsequently adjusted to 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11 and 12 using a 1M HCl, 0.1M HCl, 1M NaOH or 0.1M NaOH. Afterwards the solutions were poured to test-tubes and heated in a water bath for 5, 10, 15, 20, 30, 40, 50 or 60 min. The solutions with pH from 1 to 7 were heated at 20, 40, 60, 80 and 100°C while the solutions with pH from 8 to 12 were heated only at 100°C. For reduction sugar analysis, the samples were neutralized immediately after heating using a 0.1 or 1M HCl, or NaOH solution and afterwards cooled to ambient temperature in the running tap water.

The Miller method [1959] was used for the quantitative analysis of the reducing sugar in inulin solutions. Adequately diluted samples were reacted with DNS acid. The intensity of developed colour was measured using a spectrophotometer (Spekol 11, Carl Zeiss Jena). Fructose was used for the establishment of a standard curve. For the ana-

lytical purpose the reducing sugar amount was expressed as reducing sugar percentage share in total sugar (inulin) content.

For the statistical purposes and figures clarity the mean values were drawn from the heating times. The data were analysed by the Statistical Analysis System (SAS Enterprise Guide 3.0.3.414) using the ANOVA procedure for analysis of variance and Student-Newman-Keuls t-test for ranking the means.

RESULTS AND DISCUSSION

Heating 5% inulin solution in a strong acidic environment (pH 1-3) caused intensive hydrolysis (Fig. 1-3). The amount of unbound reducing sugar increased with heating time and temperature. In the environment with the lowest analysed pH, hydrolysis proceeded quite intensely even at not very high temperature (40°C). The higher temperature was necessary to release more than 60% reducing sugar when the acidity decreased.

The inulin breakdown occurred when the temperature reached 80°C at pH 4 (Fig. 4) however, even after 55 min heating at boiling water bath, only 20% reduction sugar was released.

Studying the gelation properties of inulin Kim et al. [2001] showed an impairment of a gel structure for 25% inulin gels produced at pH 3 and lower. Florkowska et al. [2004] noticed weaker gelation for 20% inulin solution at pH 4, whilst 25% inulin gel had compact structure. Very similar effect on the texture noticed Glibowski and Wasko [2008]. Reduction sugar analysis revealed its significant increase in the gels formed at pH lower or equal 3, which explained the decrease in gel hardness [Kim et al. 2001, Glibowski and Wasko 2008]. The inulin breakdown occurred in an acidic environment (pH < 4) which caused loosening of the gel structure.

The content of reduction sugar was very similar in a slightly acidic and neutral environment – at pH 5, 6 and 7 – regardless of time and temperature of thermal treatment (Fig. 5-7). This trend was maintained at an alkaline environment. Heating the samples

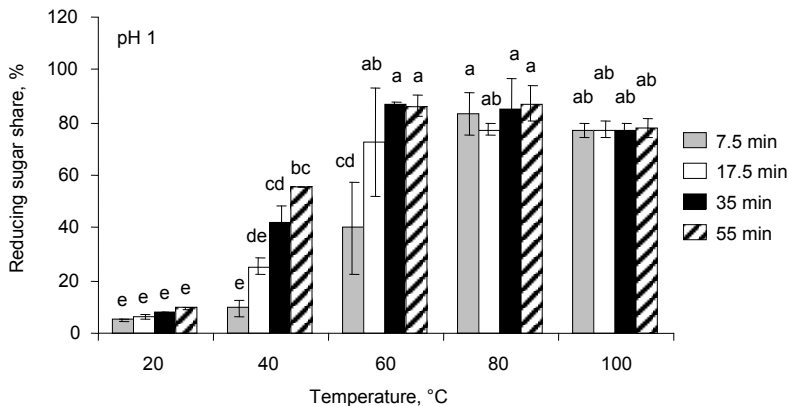


Fig. 1. Percentage share of reducing sugar in total sugar content as affected by heating time and temperature at pH 1. Data are presented as means \pm standard deviation. Means with different letters are significantly different, $p \leq 0.05$

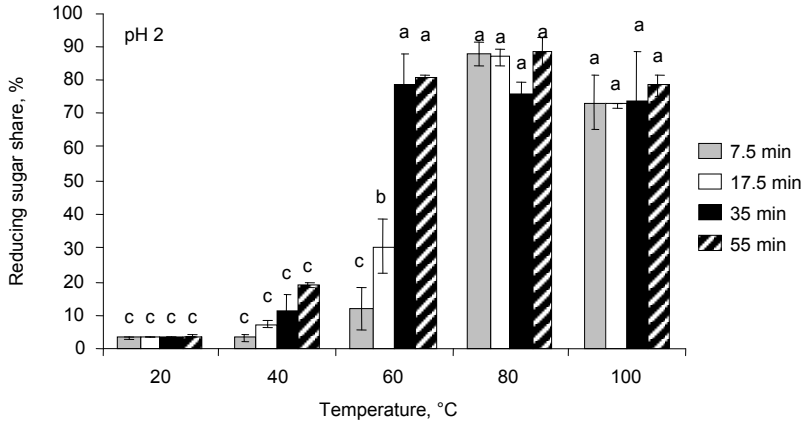


Fig. 2. Percentage share of reducing sugar in total sugar content as affected by heating time and temperature at pH 2. Data are presented as means \pm standard deviation. Means with different letters are significantly different, $p \leq 0.05$

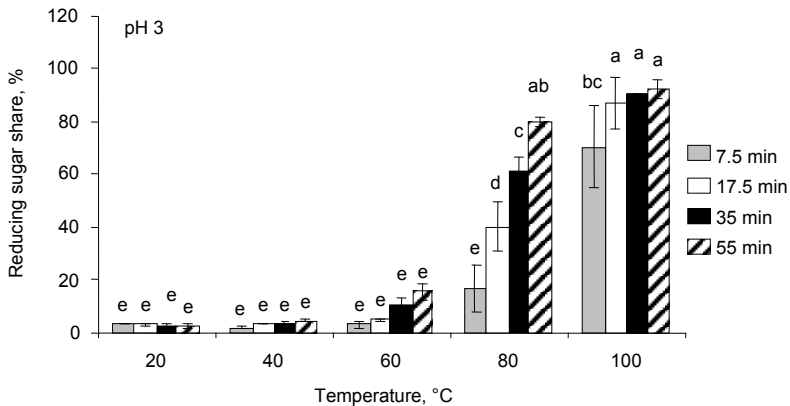


Fig. 3. Percentage share of reducing sugar in total sugar content as affected by heating time and temperature at pH 3. Data are presented as means \pm standard deviation. Means with different letters are significantly different, $p \leq 0.05$

at 100°C even for 55 min at pH in the range of 8-12 did not cause the change in the content of reduction sugar (Fig. 8). The analysed reduction sugar content at 2-3% level at pH higher than 5 resulted probably from a natural presence of simple sugars in the used inulin powder.

Florkowska et al. [2004] affirmed stability of 20% inulin gels formed at pH > 5. Only the sample with 15% inulin concentration did not form a gel with the complete structure but this was probably caused by too low concentration to form three dimension network. The analysis of reducing sugar by two independent researchers groups showed

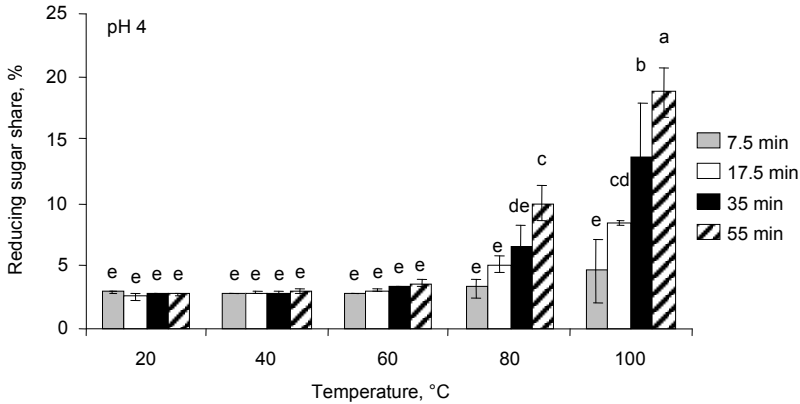


Fig. 4. Percentage share of reducing sugar in total sugar content as affected by heating time and temperature at pH 4. Data are presented as means \pm standard deviation. Means with different letters are significantly different, $p \leq 0.05$

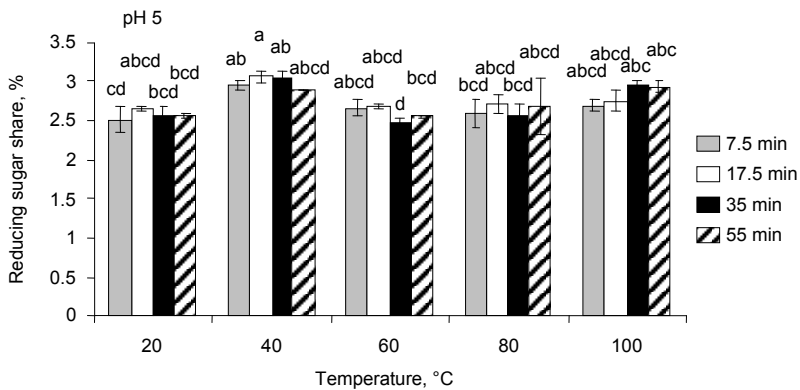


Fig. 5. Percentage share of reducing sugar in total sugar content as affected by heating time and temperature at pH 5. Data are presented as means \pm standard deviation. Means with different letters are significantly different, $p \leq 0.05$

low reducing sugar concentrations in the gels formed at little acidic, neutral and alkaline pH [Kim et al. 2001, Glibowski and Wasko 2008].

In contrast to the results obtained in this work, Kim et al. [2001] noticed a little increase in reducing sugar concentration due to heating temperature increase for the samples heated at neutral pH. As the temperature increase caused considerable decrease in inulin gelling properties they supposed that it resulted from inulin molecules breakdown into shorter chains, which was indirectly confirmed by the simple sugar concentration increase. These results did not corroborate with Glibowski and Wasko [2008] studies. Applying HPLC analysis Glibowski and Wasko [2008] showed that inulin is chemically stable even at high temperatures in the environment close to neutral pH, whereas

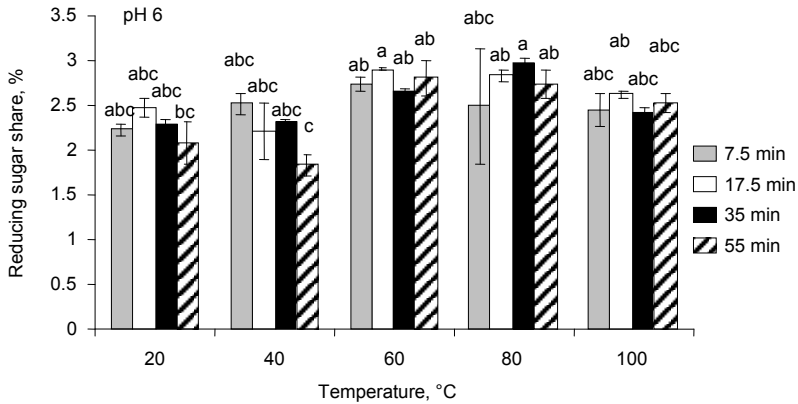


Fig. 6. Percentage share of reducing sugar in total sugar content as affected by heating time and temperature at pH 6. Data are presented as means \pm standard deviation. Means with different letters are significantly different, $p \leq 0.05$

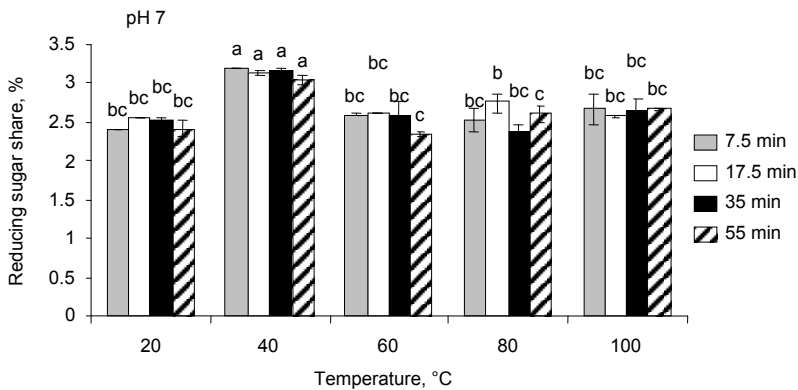


Fig. 7. Percentage share of reducing sugar in total sugar content as affected by heating time and temperature at pH 7. Data are presented as means \pm standard deviation. Means with different letters are significantly different, $p \leq 0.05$

the observed deterioration in inulin gelation properties resulted from crystal character of the inulin gel structure. The inulin gel structure depended on the presence of the crystal seeds which were dissolved at high temperatures.

Generally, it can be concluded from this study, that inulin can be applied in food systems especially as a fat substitute. Food which contains considerable amount of fat with pH lower than 4 is rare. The pH of the most acidic food (e.g. yoghurt or kefir) in which inulin is used as a fat substitute is never lower than 4. What is more important, this acidity appears during maturation i.e. maximum at 43–45°C in case of yoghurt maturation. In this temperature range, considerable increase in simple sugar concentration was observed at pH 3, thus in an environment with 10 times higher hydronium ion concentration.

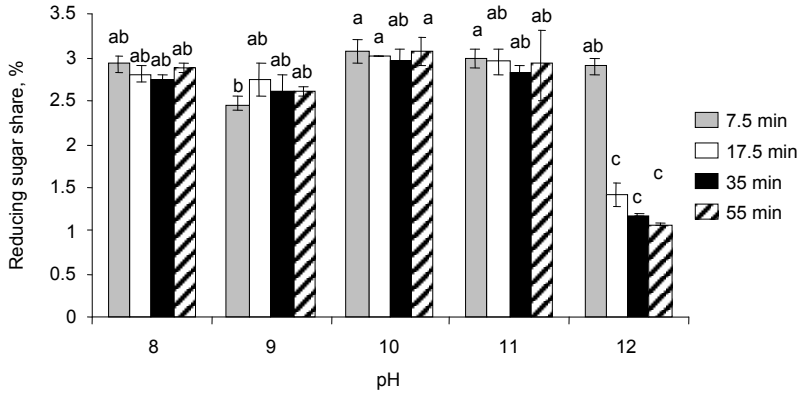


Fig. 8. Percentage share of reducing sugar in total sugar content as affected by heating time at 100°C in an alkaline environment. Data are presented as means \pm standard deviation. Means with different letters are significantly different, $p \leq 0.05$

CONCLUSIONS

1. Inulin chemical stability decreases in an acidic environment at $\text{pH} \leq 4$ due to the heating time and temperature increase.
2. In a neutral and alkaline environment inulin is chemically stable independently of pH, heating time and temperature.
3. Inulin can have limited applications in acidic foods ($\text{pH} \leq 4$), especially heated at temperatures above 60°C.
4. Inulin degradation does not take place in the products with $\text{pH} \geq 5$ heated up to 100°C.

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WPLYW ODCZYNU ŚRODOWISKA, TEMPERATURY I CZASU OGRZEWANIA NA STABILNOŚĆ CHEMICZNĄ INULINY

Wstęp. Inulina jest zapasowym węglowodanem obecnym w dużych ilościach w cykorii, dali i topinamburze. Wykazuje cechy prebiotyczne oraz ma wiele właściwości funkcjonalnych. W dotychczasowych badaniach nad stabilnością chemiczną inuliny skupiano się przede wszystkim na wpływie odczynu środowiska na cechy reologiczne tworzonych przez inulinę żeli. W pracach nie badano szczegółowo wpływu czasu, temperatury i odczynu środowiska na stabilność chemiczną inuliny w roztworach o stężeniach uniemożliwiających jej żelowanie. Dlatego celem pracy było zbadanie wpływu wymienionych czynników na stabilność chemiczną inuliny w roztworach wodnych.

Materiały i metody. W celu przeprowadzenia analiz 5-procentowe (w/w) roztwory inuliny o pH od 1 do 12 ogrzewano w temperaturze 20, 40, 60, 80 i 100°C, przez okres od 5 do 60 min. Po zobojętnieniu oznaczano poziom cukrów redukujących metodą Millera z użyciem kwasu 3,5-dwunitrosalicylowego.

Wyniki. Przeprowadzone badania wykazały, że stabilność chemiczna inuliny w środowisku kwaśnym, w $\text{pH} \leq 4$, zmniejszyła się wraz ze wzrostem temperatury i czasu ogrzewania. W środowisku obojętnym i zasadowym inulina jest stabilna niezależnie od pH, temperatury i czasu ogrzewania.

Wnioski. Zastosowanie inuliny w żywności może być ograniczone w produktach kwaśnych i poddawanych ogrzewaniu w temperaturach powyżej 60°C, a w produktach o $\text{pH} \geq 5$, nawet podlegających obróbce termicznej do 100°C, nie zachodzi degradacja tego fruktanu.

Słowa kluczowe: inulina, hydroliza, temperatura, odczyn środowiska

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