

H. GAMBUŚ<sup>1</sup>, R. GUTTERCH<sup>1</sup>, A. NOWOTNA<sup>1</sup>, W. PRAZNIK<sup>2</sup>

## PHYSICO-CHEMICAL PROPERTIES OF DEFATTED TRITICALE STARCH

### Abstract

The aim of this work was to find out to what degree different defatting conditions viz., solvents and temperature of extraction affect defatting efficiency and physico-chemical defatted starch properties. Triticale starch subjected to 10 h extraction using 75 % n-propanol at 30, 50 and 80°C, as well as mixture of chloroform:methanol:water (in volume proportion 18:6:1) at 30 and 50°C was used for investigation. Our studies confirmed the highest effectiveness of starch defatting with 75 % n-propanol at 80°C.

### Introduction

Lipids in cereal starches have been divided by Morrison [8, 9] into internal starchy lipids, mainly phospholipids called also the true starch lipids, and surface lipids. A small layer of outer lipids on the surface of starch granules has a chemical composition similar to starchy lipids as it contains relatively higher amounts of monoacylic residues than non-starchy lipids and is able to form complexes with amylose [5, 8, 9]. Other outer lipids called non-starchy lipids are mainly triglycerides.

Starchy lipids by forming complexes with amylose determine many physico-chemical starch characteristics [1, 2, 3, 5, 16]. Removal of starchy lipids or a change in their chemical composition may serve therefore as a method of altering these properties [1, 5].

In the process of starch defatting surface or internal lipids can be removed. This depends on the kind of the solvent used [14, 15]. Defatting efficiency is determined by polarity of the solvent and by the temperature of extraction [12, 14]. High extraction efficiency of starchy lipids can be achieved in high temperature, by using n-propanol and water in the volumetric ratio of 3:1 [8, 12]. A standard mixture used for the removal of non-starchy lipids includes chloroform, methanol and water and is efficient even at room temperature [9, 12, 15].

<sup>1</sup> Department of Carbohydrate Technology, Agricultural University, Kraków, Poland

<sup>2</sup> Institute of Chemistry, University for Bodenkultur, Vienna, Austria

Water is an important constituent of all mixtures used for defatting as it enables starch granules to swell and makes solvent penetration within the granule more easy [8].

The objective of the work was an attempt to learn the influence of the quantity and quality of lipid removed from starch granule on the physico-chemical characteristics of defatted starch.

### Material and methods

Triticale starch isolated from a winter variety Ugo served as the investigated material. The starch was defatted during 10 h extraction either with 75 % n-propanol at temperatures 30, 50 and 80°C [6], or with a mixture of chloroform: methanol: water (18:6:1, volumetric ratio) at 30 and 50°C. After fat extraction the solvent was evaporated and the total solids was analysed for crude fat (by dissolving in acetone) and determining sum of carbohydrates with antron reagent, as described in [7]. Defatted starch was also analysed for:

- graininess by using analyser type Analysette 22 made by Fritsch GmBH
- total phosphorus content [4]
- total protein content (N x 5,7) [13]
- the content of amylose [11]
- water binding capacity and solubility in water [13]
- gelatinization characteristic of 8.5 % starch suspension in water in rotation viscosimeter Rheotest 2 [2].

The starches were pictured by scanning electron microscope type Tesla BS-300 (magnification 900 or 1125 x).

### Results and discussion

Analyses of the total solids after solvent evaporation showed that in the process of fat extraction small amounts of protein and carbohydrates were removed regardless of the type of the solvent used and extraction temperatures applied (Table 1). Differences among dry residues resulting from type of the solvent and applied temperatures can be attributed to the amounts of fat removed. The highest dry residue was found when fatty substances were extracted with 75 % n-propanol at 80°C and the residue was highest in crude fat.

Temperature of extraction with 75 % n-propanol affected total phosphorus content of defatted starches, the lowest amounts of the compound were found in starches defatted at 80°C. This proves that at 80°C phospholipids were removed from starch granules most efficiently (Table 2). Additional evidence that starch defatting at 80°C

was the most efficient provide amounts of amylose liberated from its lipids complexes from the insides of starch granules – highest at this temperature.

Compared to starting material, in starches defatted by the mixture of chloroform, methanol and water higher amounts of amylose were found. It seems however that the increased contents of amylose can be attributed to complexes located on the surface of the starch granules. This was concluded from the fact that starches extracted at 30 and 50°C by either of two solvents had similar contents of amylose, whereas it is well known that removal of true starchy lipids requires hot solvent to be used [12]. It seems that the removal of outer starchy lipids, having composition similar to phospholipids, as well as non-starchy lipids (triglycerides) is more efficient with the chloroform, methanol and water mixture than with 75 % n-propanol at 30 or 50°C.

Table 1

Results of chemical analysis performed on total solids (after evaporation of the solvent used for extraction) of Triticale starches isolated from the variety Ugo

Solvent used	Extraction temperature [°C]	Total solids [%]	Fat components [%]	Carbohydrate <sup>x)</sup> [%]	Protein <sup>xx)</sup> [%]
n-propanol	30	0.42	0.12	0.28	0.05
	50	0.54	0.20	0.34	0.07
	80	0.74	0.30	0.30	0.07
chloroform: methanol: water	30	0.39	0.11	0.16	0.06
	50	0.49	0.18	0.27	0.07

<sup>x)</sup> carbohydrate = dissolved in: cold water + autoclave (1 atm, temp. 121°C)

<sup>xx)</sup> protein in naive starch - protein in defatted starch

Table 2

The content of amylose and non-carbohydrate components in Triticale starch samples of Ugo variety

Kind of starch samples	Extraction temperature [°C]	Total phosphorus [%]	Raw protein [%]	Amylose [%]
native		0.047	0.15	26.31
defatted n-propanol	30	0.038	0.11	29.63
	50	0.032	0.09	30.09
	80	0.016	0.08	32.88
defatted chloroform: methanol:water	30	0.32	0.10	29.34
	50	0.024	0.09	30.51

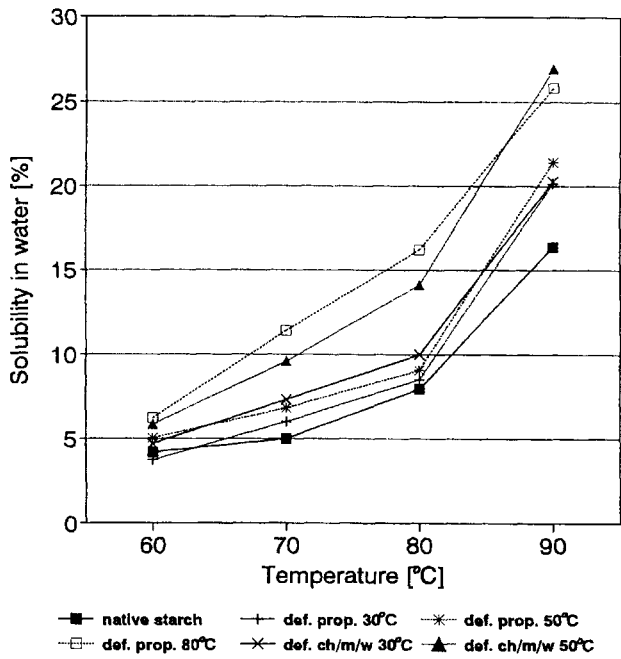


Fig. 1. Solubility in water of starch samples of Ugo variety.

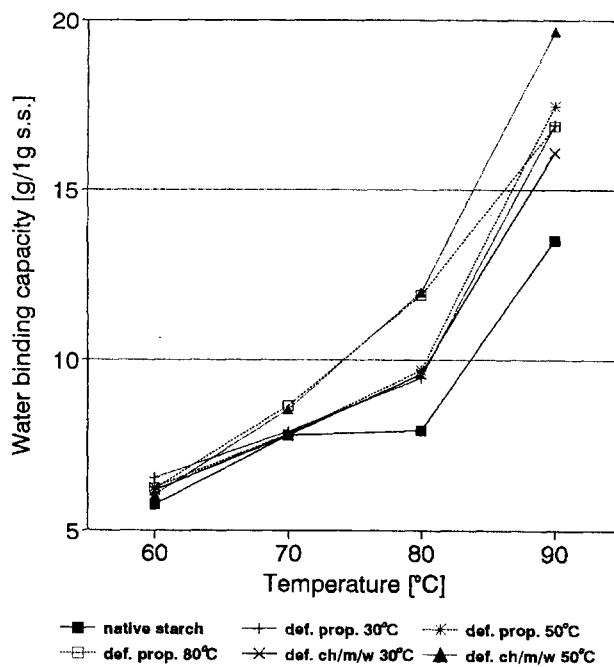


Fig. 2. Water binding capacity of starch samples of Ugo variety.

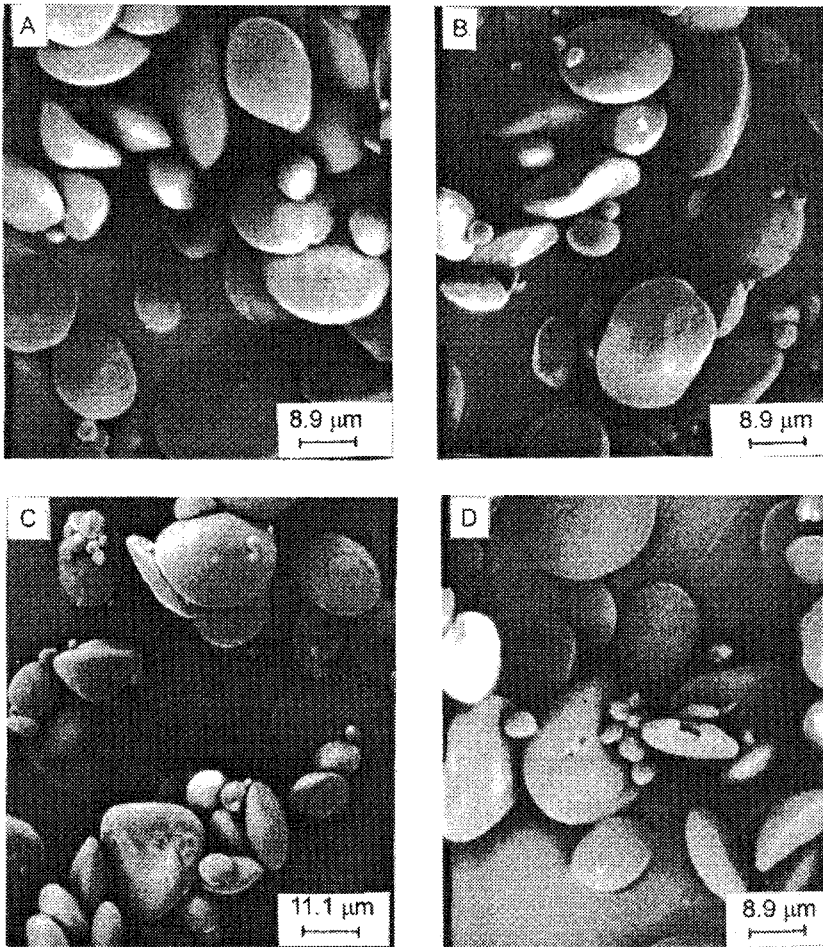


Fig.3. Scanning electron micrograph of granules of Triticale Starch native (A) and defatted in 75% n-propanol at: 30°C (B), 50°C (C) and 80°C (D).

Defatted starch had higher water binding capacity and solubility in water at temperatures 60-90°C than non-defatted starch (Figs. 1, 2). Fat removal from starch with 75 % n-propanol at 80°C caused disappearance of a two-phase character of these processes, what provides just another evidence that at the temperature lipids were released from their complexes with amylose [5, 8, 9, 16]. Lower temperatures of extraction with the same solvent, as well as the mixture of chlorophorm, methanol and water, do not destroy the amylose-lipids complexes and consequently the two-phase character of starch granules swelling is retained (Figs. 1, 2) , in spite of removing some amounts of lipids (Table 2).

Table 3

Pasting characteristic of 8.5 % water suspensions of starch samples from triticale of Ugo variety measured by means of rotation viscosimeter Rheotest 2 using rot as a measuring unit

Kind of starch samples	Pasting temperature [°C]	Maximum viscosity [J.U.]	Viscosity after cooling to 50°C [%]
native	65.0	105.7	111.0
defatted			
- n-propanol	30°C	61.0	98.3
	50°C	61.5	114.7
	80°C	61.5	115.3
- chloroform: methanol: water	30°C	62.0	107.3
	50°C	61.5	116.3

Table 4

Grainines of starch samples from Triticale of Ugo variety

Kind of starch samples	% starch granules < 10µm	
native	11.64	
deffated		
- n-propanol	30°C	10.23
	50°C	9.71
	80°C	11.18
- chloroform: methanol: water:	30°C	11.83
	50°C	11.59

Removal of the outer starchy and non-starchy fats lowered gelatinization temperatures of starches defatted by either of the solvents used (Table 3).

Extraction of lipids from complexes with amylose using 75 % n-propanol at 80°C did not cause any further decrease in gelatinization temperature. Gelatinized defatted starch had always slightly higher maximal viscosity, and viscosity after cooling to 50°C, than gelatinized non-defatted starch. Removal of inner starchy lipids did not cause considerable changes in the values of gelatinization parameters. The influence of amylose-lipid complexes on changes in these parameters was visible only in a less pronounced two-phase character of starch gelatinization when starch was defatted with 75 % n-propanol at 80°C.

Extraction of lipids did not cause any damage to the surface of starch granules as can be seen on the pictures made by the scanning electron microscope (Fig. 3). This process did not cause any changes in graininess of defatted starches either (Table 4).

## Conclusions

1. No visible changes in the appearance of defatted starch granules were observed by the electron scanning microscope.
2. Compared to native starches, defatted starches were characterized by unchanged graininess, lower contents of protein and phosphorus, higher contents of measured amylose, higher water binding capacity and solubility at 60–90°C, higher maximal viscosity of paste formed from water suspensions. Those differences were most noticeable in starches defatted with 75 % n-propanol at 80°C.
3. The research performed confirmed 75 % n-propanol at 80°C to be the most effective defatting agent.
4. Analyses done on the defatted residues showed that in addition to lipids the extraction process removes small amounts of carbohydrates and protein, no matter what the temperature and type of solvent were used.
5. Lipids inside starch granules bound in complexes with amylose were removed by 75 % n-propanol at 80°C.

## REFERENCES

- [1] Eliasson A.C., Larson K., Mieziš Y.: *Starch/Stärke*, **33**, 1981, 231-235.
- [2] Gambuś H., Nowotna A.: *Pol. J. Food Nutr. Sci.*, **1/42**, 1992, 101-107.
- [3] Lorenz K., Collins F., Kulp K.: *Starch/Stärke*, **35**, 1993, 123-129.
- [4] Marsh B.B.: *Biochem Biophys. Acta*, **32**, 1959, 357-359.
- [5] Melvin M.A.: *J. Sci. Food Agric.*, **30**, 1979, 731-738.
- [6] *Methodensammlung der Abteilung Kohlenhydrate des Zentralinstituts für Ernährung der Akademie der Wissenschaften der DDR. Potsdam-Rehbrücke*, 1986, pp. 19.
- [7] Morris D.L.: *Science*, **107**, 1948, 254-255.
- [8] Morrison W. R.: *Starch/Stärke*, **33**, 1981, 408-410.
- [9] Morrison W.R.: *J. of Cereal Sci.*, **8**, 1988, 1-15.
- [10] Morrison W.R., Coventry A.M.: *Starch/Stärke*, **37**, 1985, 83-87.
- [11] Morrison W.R., Laignelet B.: *J. Cereal Science*, **1**, 1983, 9-20.
- [12] Pomeranz Y.: *Modern Cereal Science and Technology*. VCH Publishers, 1986.
- [13] Richter M., Augustat S., Schierbaum F.: *Ausgewählte Methoden der Stärke- chemie*. VEB Fachbuch Verlag, Leipzig, 1969, 111-113, pp. 63-64.
- [14] Rogols S., Green J.E., Hilt M.A.: *Cereal Chemistry*, **46**, 1969, 181-188.
- [15] Vasanthan T., Hoover R.: *Food Chemistry*, **43**, 1992, 19-27.
- [16] Vasanthan T., Hoover R.: *Food Chemistry*, **45**, 1992, 337-347.

**FIZYKO-CHEMICZNE WŁAŚCIWOŚCI ODTŁUSZCZONEJ SKROBI PSZENŻYTNEJ****Streszczenie**

Celem pracy było sprawdzenie w jakim stopniu różne warunki odtłuszczenia tj.: rozpuszczalniki i stosowane temperatury ekstrakcji wpływają na skuteczność odtłuszczenia oraz na fizyko-chemiczne właściwości skrobi odtłuszczonej. Do badań użyto skrobię pszenżytnią, którą poddano procesowi odtłuszczenia przez 10 godzinną ekstrakcję 75 % n-propanolem w temperaturach 30, 50 i 80°C lub mieszaniną chloroformu:metanolu:wody w stosunku 18:6:1 w temperaturach 30 i 50°C. Wykonane badania potwierdziły największą skuteczność w odtłuszczeniu skrobi, 75 % n-propanolu w temperaturze 80°C. ☒