applied sciences

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Preparation and Characterization of Soluble Dextrin Fibre from Potato Starch Obtained on a Semi-Industrial Scale

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Abstract: Currently, dietary fibre intake is low, which is one of the reasons for the global obesity epidemic and other metabolic disorders. Dietary fibre has many documented health-promoting properties, such as a prebiotic effect, inducing feelings of satiety and reducing postprandial glucose. Therefore, there is an increasing interest in the search for new products rich in dietary fibre. One of the sources of dietary fibre may be resistant dextrins obtained as a result of dextrinization of starch. In this study, soluble dextrin fibre (SDexF) was prepared by heating potato starch in the presence of hydrochloric and citric acids on a semi-industrial scale in the prototype dextriniser. The aim of the study was the optimisation of the preparation of SDexF on a semi-industrial scale and the physicochemical characterisation of the obtained product. Also, the molecular structure of the prepared product was analysed by using SEM and FTIR. The semi-industrial production of SDexF was successfully implemented, achieving approximately 100 times higher product quantities in one process cycle. SDexF was characterised by over 30% total dietary fibre (TDF) content, almost 100% water solubility, low viscosity and no retrogradation tendency. The physicochemical and functional properties of the obtained product indicate the possibility of implementing SDexF to enrich food products.

Keywords: resistant dextrins; dietary fibre; functional food; semi-industrial scale

Citation: Wojcik, M.; Kapusniak, K.; Zarski, A.; Kapusniak, J. Preparation and Characterization of Soluble Dextrin Fibre from Potato Starch Obtained on a Semi-Industrial Scale. *Appl. Sci.* **2024**, *14*, 1438. https://doi.org/10.3390/app14041438

Academic Editor: Leonel Pereira

Received: 19 January 2024
Revised: 7 February 2024
Accepted: 7 February 2024
Published: 9 February 2024

1. Introduction

Nowadays, the majority of countries in the world follow a Western diet, which is rich in refined carbohydrates and saturated fats [1], leading to civilisation diseases such as obesity or type 2 diabetes [2,3]. Moreover, although childhood obesity rates have historically been lower compared to adults, in many countries, the escalation of childhood obesity has exceeded the rate of increase observed in adults [4]. Dietary fibre intake is inversely associated with obesity, while higher consumption is often linked to reduced weight gain, improved satiety and lipid metabolism, and prebiotic effects [5–8]. However, despite the benefits of dietary fibre intake, such as obesity prevention or stimulation of the growth of beneficial intestinal microbiota, its intake is still below the recommended intake levels [9]. Moreover, children do not consume the appropriate amount of products containing large amounts of dietary fibre, such as vegetables or legumes [10]. For this reason, it is necessary to look for new products rich in dietary fibre, especially for children [11,12]. One of the possible solutions may be functional foods, i.e., those enriched with an additional amount of dietary fibre. Functional foods can be defined as foods that offer additional health benefits beyond basic nutrition [13]. The term "functional" refers to the specific health-promoting or disease-preventing properties these foods may possess [14,15].

Dextrinization represents one of the methods to derive dietary fibre products by thermal/enzymatic modification of starch, yielding soluble, indigestible dextrins/maltodextrins [16]. Many health benefits of enzyme-resistant dextrins have been demonstrated so far, including a beneficial effect on the intestinal microbiota, ensuring health of the host [17–19]. In previous studies, resistant dextrins have been obtained by heating potato starch acidified with catalytic amounts of food-grade hydrochloric and citric acids. Soluble dextrin fibre (SDexF) was obtained on a laboratory scale [20–22]. However, due to the launch of the project with the acronym PreSTFibre4Kids, it became necessary to develop and implement a semi-industrial scale in order to obtain the required amount of resistant dextrin. The project assumed examining vegetable and fruit mousses enriched with SDexF from potato starch with prebiotic properties [23], in terms of the prevention of overweight and obesity in children and the reduction of metabolic disorders secondary to obesity. The project was carried out by a consortium consisting of Jan Dlugosz University (Czestochowa, Poland) as leader, Tymbark LLC (Olsztynek, Poland), The Children's Memorial Health Institute (Warsaw, Poland), Lodz University of Technology (Lodz, Poland) and Maria Sklodowska-Curie National Research Institute of Oncology. During the implementation of the project, it was necessary to optimise the production process in the prototype machine and to characterise the obtained product.

The aim of the study was to optimize the production of SDexF on a semi-industrial scale. Moreover, the conducted research will provide information on whether SDexF obtained on a semi-industrial scale can be used to enrich vegetable and fruit products as a dietary fibre source.

2. Materials and Methods

2.1. Materials

Food-grade potato starch was delivered by Pepees SA, Lomza, Poland. Food-grade hydrochloric acid and citric acid were purchased from PCC Rokita, Brzeg Dolny, Poland and Bartex, Czestochowa, Poland. Analytical grade reagents were bought from Sigma-Aldrich, Poznan, Poland. Enzymatic kits for determining total dietary fibre content, residual citric acid and ethanol were purchased from Megazyme, Wicklow, Ireland.

2.2. Preparation of SDexF on a Semi-Industrial Scale

During project implementation, more than 400 kg of SDexF were produced using a prototype machine type UHP-01.00 (Mysak, Poznan, Poland) designed for obtaining indigestible dextrins on a semi-industrial scale. The most important systems of this device included (Figure 1) feeding and mixing of solid raw material (heating chamber with a mixer), liquid dispensing, air and oil heating, dedusting, temperature control, mixing and regulation of the frequency of regeneration and dust ejection.

SDexF was prepared according to patent PL220965 [24]. First, 10 kg of food-grade potato starch was weighed and placed in the mixing chamber of the machine. Then mixing was started and the starch was sprayed with 0.5% (v/v) aqueous food grade hydrochloric acid solution and the same amount of 0.5% (v/v) aqueous food grade citric acid solution to a final concentration of each acid amounting to 0.1% w/w related to dry starch basis (d.s.b). The next step involved pre-drying the acidified starch to a water content below 5% using two different heating methods: hot air at 110 °C and the oil heating jacket at 105 °C. The water content was checked for a sample taken from the heating chamber using a moisture analyser (Radwag, Radom, Poland). After confirming the water content was below 5%, the next step was dextrinization, which was carried out by heating only with the oil heating jacket at 140 °C for 4 h. The product was continuously mechanically mixed throughout the entire process. The obtained SDexF was cooled to room temperature in the desiccator, then ground up and sieved through laboratory stainless steel sieves (pore diameter of 250 µm) to remove the burnt formed during heating. The sieved product was washed with several portions of food-grade ethyl alcohol in order to remove low-molecular starch thermolysis products and citric acid residues. Residual ethyl alcohol was removed from

the final product by evaporation under the following conditions: air drying for 2 h and then drying in a dryer at 60 °C for 48 h (until the smell of ethanol was no longer detected). Initially, in order to evaluate the process of obtaining SDexF on a semi-industrial scale, samples were taken from 4 batches, where each batch consisted of a product obtained from the dextrinization process carried out twice (1 batch = 2 × 7 kg) marked as SDexF1, SDexF2, SDexF3, SDexF4. For these samples, total dietary fibre content (TDF), solubility in water at 20 °C, dextrose equivalent (DE), residual citric acid and residual ethanol were determined. The remaining experiments were evaluated by testing an average of the sample obtained from the mixed SDexF1–SDexF4.

Figure 1. Prototype dextriniser for SDexF production on a semi-industrial scale (Jan Dlugosz University, Czestochowa, Poland, 2020).

2.3. Total Dietary Fibre Content (TDF)

TDF content was determined according to AOAC 2011.25 Method [25]. High molecular weight dietary fibre (HMWDF), comprising insoluble dietary fibre (IDF), dietary fibre soluble in water but precipitated in 78% aqueous ethanol (SDFP), and dietary fibre soluble in water and not precipitated in 78% aqueous ethanol (SDFS), was determined. TDF is determined by calculating the sum of IDF, SDFP, and SDFS. Briefly, the samples were subjected to enzymatic hydrolysis with a mixture of α-amylase and amyloglucosidase enzymes in maleate buffer (pH 6.0) at 37 °C for 16 h. Then, after changing the pH to 8.2, hydrolysis was carried out using protease at 60 °C for 30 min. Next, the hydrolysate was filtered under reduced pressure through a Gooch-type glass crucible with a sintered disc (G-3 porosity) in order to determine the content of insoluble dietary fibre (IDF). Then, ethanol was added to the filtrate, which precipitated water-soluble high-molecular fibre fractions (SDFP). The precipitate was separated from the solution by vacuum filtration through another Gooch glass crucible with a sintered disc. Subsequently, the precipitate on the filters was washed two times with 15 mL portions of 78% ethanol, 95% ethanol and acetone, dried overnight in a 105 °C oven and weighed. Based on the mass of sediments, the content of IDF and SDFP was determined, respectively. The hydrolysate remaining after filtration was concentrated and analysed using high-performance liquid chromatography

Nexera-2 high-performance liquid chromatography (Shimadzu, Kyoto, Japan) for SDFS content. The results were calculated using Mega-Calc Data Calculator (Megazyme).

2.4. Solubility in Water

Solubility in water at 20 °C was measured using Richter's method [26]. In brief, 0.5 g of sample was weighed and suspended in 40 mL of distilled water. The sample was mixed for 30 min by using a magnetic stirrer. Then, the samples were centrifuged and 10 mL of supernatant was pipetted to a weighing cell with known weight and dried at 130 °C until a constant weight was achieved. Solubility in water was calculated from Equation:

$$S = \frac{100 \times 40 \times b}{10 \times a} \ [\%] \quad (1)$$

where: a—sample weight, b—weight of residue after drying, volume of evaporated supernatant (10 mL), and total volume of added water (40 mL).

2.5. Dextrose Equivalent (DE)

The DE of the samples was determined by Schoorl–Regenbogen's method [27]. The samples were weighed in amount of 0.5 g and 10 mL of distilled water was added to them. Samples were mixed on a vortex and placed on a magnetic stirrer for 30 min. After this time, 10 mL of Fehling I solution, 10 mL of Fehling II solution, a solution of the sample and 20 mL of distilled water were added to the conical flasks. The prepared mixture was boiled for 5 min and later cooled down. Then, 10 mL of 30% potassium iodide solution, 10 mL of 25% H_2SO_4 solution and 5 mL of 1% soluble starch solution were added. The content of conical flasks was titrated with 0.1 M sodium thiosulfate. The procedure for blanks was performed analogically using distilled water.

2.6. Quantitive Determination of Residual Citric Acid and Ethanol

The residue of unreacted citric acid in the SDexF was determined by using The Citric Acid Assay Kit (Megazyme, Wicklow, Ireland). In short, 1.8 mL of water, 0.5 mL of buffer (pH 7.5) with sodium azide (0.02%), 0.2 mL nicotinamide adenine dinucleotide with polyvinylpyrrolidone and 0.02 mL L-Malate dehydrogenase/D-lactate dehydrogenase solution were added to 0.2 mL of dissolved samples (5% concentration) in disposable cuvettes. Samples were mixed, and after 4 min, absorbances of the solution were read by using Helios Delta UV-Visible spectrophotometer (Thermo Fisher Scientific, Waltham, MA, USA) at wavelength 340 nm. Next, 0.02 mL citrate lyase lyophilisate solution was added and mixed with samples. Absorbances of the solutions were read at the end of the reaction (~5 min) at the same wavelength as before. The procedure for blanks was performed analogically using distilled water instead of a sample. The residual unreacted citric acid in samples was calculated using Mega-Calc Data Calculator (Megazyme, Wicklow, Ireland).

The residue of ethanol after the washing process in the SDexF was determined by using The Ethanol Assay Kit (Megazyme, Wicklow, Ireland). In brief, 0.1 mL of dissolved samples (0.05% concentration) was placed in disposable cuvettes. In blanks, 0.1 mL of distilled water was added instead of the sample. To each sample, 0.2 mL of buffer (pH 9.0), 0.2 mL of nicotinamide adenine dinucleotide, 0.05 mL of aldehyde dehydrogenase solution and 2 mL distilled water were added. Then, samples were mixed and absorbances of the solutions were read after 2 min by using Helios Delta UV-Visible spectrophotometer (Thermo Fisher Scientific, Waltham, MA, USA) at wavelength 340 nm. Subsequently, alcohol dehydrogenase suspension was added to samples in the amount of 0.02 mL. Samples were mixed again and absorbances of solutions were read at the end of the reaction (~5 min) at the same wavelength as before. The residual ethanol in the samples was calculated using Mega-Calc Data Calculator (Megazyme, Wicklow, Ireland).

2.7. The Nutrient Content and Energy Value

Analyses of fat, including fatty acids, carbohydrates, protein, nitrogen, sodium chloride and total ash content in SDexF were performed in the accredited laboratory NUSCANA—biotechnika laboratoryjna (Poland, accreditation AB 1179). Tests for fat and fatty acid content were carried using the Soxhlet method according to PN-EN ISO 11085:2015-10 [28] and using gas chromatography with a flame ionisation detector, according to PN-EN ISO 12966-1:2015-01/AC:2015-06 [29], PN-EN ISO 12966-2:2017-05 point 5.2 [30], respectively. The carbohydrate content was determined using the Luff–Schoorl method, which is consistent with PN-A-74108:1996 [31]. The protein and nitrogen content were determined with the Kjeldahl method according to PN-EN ISO 20483:2014-02 [32]. For the determination of sodium chloride, the Mohr method was used according to PN-A-74108:1996 [31]. The total ash content was measured using the weight method according to PN-EN ISO 2171:2010 [33]. Additionally, the starch content was determined using the standardised polarimetric method PN-EN ISO 10520:2002 [34]. The principle of determination involved extracting starch from the sample with hydrochloric acid and then measuring the angle of rotation of the plane of polarised light of the resulting solution using a polarimeter (Polarimeter POL-X, OPTIKA SRL, Ponteranica, Italy). The measurement of the angle of rotation was used to calculate a quantitative measure of the starch content in a given mass of samples [35].

Moreover, the energy value was calculated based on the previously mentioned test results in accordance with Regulation (EU) No 1169/2011 of the European Parliament and of the Council of 25 October 2011 (Annex XIV) [36].

2.8. Assessing the Presence of Contaminants

Microbiological analyses and confirmation of the absence of heavy metals were performed in Food, Nutrition and Consumer Products Research Section of the District Sanitary-Epidemiological Station in Czestochowa (Poland, accreditation AB 521). SDexF prepared on a semi-industrial scale was checked for the presence of *Salmonella* spp., *Listeria monocytogenes* and β-glucuronidase-positive *Escherichia coli* according to PN-EN ISO 6579-1:2017-04 [37], PN-EN ISO 11290-2-2017-07 [38] and PN-ISO 16649-2:2004 [39] methods, respectively. Additionally, the obtained product was examined for lead, cadmium and mercury content carried out by Atomic Absorption Spectrometry-Flame Atomic Absorption Spectroscopy [40] and Atomic Absorption Spectrometry-Flame Atomic Absorption Spectroscopy-Cold-vapour Atomic Fluorescence Spectrometry [41].

2.9. Fourier Transform Infrared (FT-IR) Spectroscopy

In order to identify and determine the chemical structure of native starch and SDexF obtained from it on a semi-industrial scale, spectra were recorded using a NEXUS NICOLET FT-IR spectrometer (Madison, WI, USA). Powdered samples were ground in an agate mortar. In the next step, each of them was mixed with an analytical grade potassium bromide (KBr) in a ratio of 1:50 (w/w), and then pressed into a disc using a manual hydraulic press. The spectra were recorded in the wavelength range from 4000 to 400 cm^{-1}, with a resolving power of 4 cm^{-1} and the number of scans was equal to 32. Before the recording, the baseline was adjusted against a KBr background. FTIR spectroscopy for structural studies was performed after each processing cycle (7 cycles in total), on a given batch of material (raw material and product). The spectral analysis concerned averaged samples (from a given batch), and the spectra presented are representative of a single such sample. A single representative overall sample was selected according to the sample preparation order, i.e., primary (individual) sample → aggregate sample → average sample → representative overall sample.

2.10. Scanning Electron Microscopy (SEM)

The morphology of native potato starch and possible changes following its dextrinization on a semi-industrial scale were examined using a Tescan VEGA 3 SBU scanning

electron microscope (Tescan, Brno, Czech Republic). Powder samples were placed on tables without covering them with conductive material. Microscopic observations were carried out under high vacuum conditions and at an accelerating voltage of 3 kV.

2.11. Pasting Properties

The pasting properties of potato starch and SDexF were determined by using a Rapid Visco Analyser (RVA 4500, PerkinElmer, Waltham, MA, USA). Pasting properties were determined by peak viscosity (PV), hot paste viscosity (HPV), breakdown (BD = PV − HPV), final viscosity (FV), and setback (SB = FV − HPV). As a result of heating and cooling of the prepared suspension of SDexF, a pasting curve is obtained, which illustrates viscosity changes during the test. Its appearance depends on the properties and concentration of the tested samples and test profile used. The procedure of the sample preparation included weighing 26 g of distilled water and 1.37 g of potato starch (5% concentration) and 25 g of water and 6.25 g of SDexF (20% concentration) into a measuring cup. The weighed samples were thoroughly mixed with water. For each suspension/solution, measurement was made by using the following temperature profile: heating for 1 min at 25 °C, heating from 1 to 6 min to 95 °C, maintaining the temperature of 95 °C for 6 to 9 min, cooling from 9 to 14 min to 25 °C, maintaining the temperature at 25 °C for the rest of the measurement up to 15 min, and stirring at 960 rpm for the first 10 s and 160 rpm for the rest of the measurement.

2.12. Retrogradation Tendency

Retrogradation tendency was determined by the use of the turbidimetric method [42]. Briefly, 2% slurries (*w*/*w*, d.s.b.) of the potato starch and SDexF with repetitions were prepared. The suspensions/solutions were heated in a boiling water bath for 1 h and then cooled to 25 °C. Subsequently, 1.5 mL was pipetted out from each slurry and placed in disposable cuvettes. The initial turbidity was determined using a Helios Delta UV-Visible spectrophotometer (Thermo Fisher Scientific, USA) by measuring at a wavelength of 640 nm. After the measurement, remaining slurries were stored in the refrigerator at 8 °C. Analogous measurements were made after 1, 3, 5. . ., 21 days of storage, each time gently mixing the slurries after taking them out of the refrigerator and heating them before taking samples for measurement in the water bath at 25 °C.

2.13. Colour Parameters

The colour of SDexF was measured by using Chroma Meter CR-400 (Konica Minolta Sensing, Osaka, Japan). Parameters L*, a*, b* were determined with the CIELab colour space, where L* is luminosity, a* represents green/red colour and b* represents blue/yellow colour. To calculate the colour difference (ΔE), the following Equation was used:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (2)$$

where ΔL*, Δa* and Δb* were the differences in the values of L*, a*, b* between native potato starch and SDexF, respectively. The tests were performed in 10 repetitions for each sample.

2.14. Statistical Analysis

The data were analysed with the Statistica 13.3 software (StatSoft, Tulsa, OK, USA). All assays were performed in triplicate unless otherwise noted, and their results were averaged. All experiments utilised a completely randomised design.

3. Results and Discussion

3.1. Preparation of SDexF on a Semi-Industrial Scale

The application of a semi-industrial scale resulted in a 100-fold increase in the amount of product in one process cycle, from approximately 70 g on a laboratory scale to 7 kg, while reducing the process time. The efficiency of SDexF production process was around

70%. Some of the most important research from the point of view of future applications were performed for individual batches to check the repeatability of the process.

3.2. Total Dietary Fibre Content in SDexF

It is well known that during the dextrinization process, hydrolysis, transglucosidation and repolymerisation occur [43], which can be crucial for increasing the TDF content in starch-based products [44]. Resistant dextrins, including SDexF, can replace some of the refined sugars and cereal flours found in food products, making them more resistant to enzymatic digestion in the human digestive tract [45]. TDF content in SDexF obtained on a semi-industrial scale ranged from 30.07% to 34.47% (Table 1). The samples contained almost exclusively soluble fibre (SDFP and SDFS) and a negligible IDF content, similarly to the resistant dextrins obtained by microwave heating of acidifed potato starch [27]. Average percentage of SDFP and SDFS was 8.09 ± 1.68 and 23.19 ± 2.05, respectively. The SDFP content in SDexF was similar to resistant dextrins obtained by Kapusniak et al., with the reservation that in the gravimetric method, it is not the same procedure [21]. The TDF content of SDexF obtained on a semi-industrial scale was higher than the TDF content of pyrodextrins heated at similar conditions (temperature and time of heating) on a laboratory scale [46–48]. In subsequent studies, the use of a higher temperature could be considered, which potentially increased the TDF content by several to several dozen percent, according to the works of other authors [44,49,50].

Table 1. TDF content of SDexF1-SDexF4 batches.

Sample	IDF [%]	SDFP [%]	SDFS [%]	TDF [%]
SDexF1	0.75 ± 0.03 ^{b}	10.42 ± 0.06 ^{b}	20.84 ± 1.08 ^{a}	32.01 ± 1.17 ^{ab}
SDexF2	0.67 ± 0.02 ^{a}	7.44 ± 0.16 ^{a}	22.60 ± 0.95 ^{ab}	30.07 ± 0.78 ^{a}
SDexF3	0.71 ± 0.01 ^{b}	8.00 ± 1.22 ^{a}	25.76 ± 0.24 ^{c}	34.47 ± 1.46 ^{b}
SDexF4	0.77 ± 0.05 ^{b}	6.48 ± 1.30 ^{a}	23.54 ± 0.02 ^{b}	30.78 ± 1.27 ^{a}

^{a,b,c} Distinct letter superscripts within identical columns denote statistical significance ($p < 0.05$).

3.3. Solubility in Water of SDexF

The starch dextrinization process leads to an increase in the solubility in water of the obtained product, because of disrupting the native granular structure of starch, breaking down the crystalline structure and exposing more sites for water interaction [51]. In general, resistant dextrins have good solubility in water [52,53], which is a significant factor contributing to their functionality as dietary fibres [54]. The solubility in water at 20 °C of four SDexF batches is presented in Table 2. The average solubility in water of SDexF was 97.89 ± 2.12%, where repeatable results were obtained for individual batches. Compared to native potato starch, an almost 100% increase in water solubility was observed for SDexF prepared on a semi-industrial scale. According to Kapusniak et al. [22], the solubility of SDexF obtained on a laboratory scale under the same conditions as SDexF on a semi-industrial scale in this study was lower, which indicates that application of a semi-industrial scale obtains a product that is completely soluble in water. The solubility in water of SDexF was similar to the solubility of pyrodextrins obtained from maize starch [55]. The authors achieved a solubility of over 90%, although using a much higher dextrinization temperature (180 °C) compared to this study. The solubility of SDexF in water increased due to the shortening of the chain lengths (starch depolymerization) because of dextrinization.

Table 2. Solubility in water of SDexF1-SDexF4 batches in comparison to native potato starch.

Sample	Solubility in Water at 20 °C [%]
Native potato starch	0.40 ± 0.34 [a]
SDexF1	95.80 ± 0.85 [b]
SDexF2	96.40 ± 0.14 [b]
SDexF3	99.15 ± 0.85 [c]
SDexF4	100.20 ± 0.49 [c]

[a,b,c] Distinct letter superscripts within identical columns denote statistical significance ($p < 0.05$).

3.4. Dextrose Equivalent (DE) of SDexF

DE is a numerical value corresponding to the degree of hydrolysis or the amount of reducing sugars present in a carbohydrate, typically starch. It indicates the percentage of dextrose (glucose) equivalent to the total reducing sugars present in the substance. A higher DE value signifies a higher degree of hydrolysis and a higher content of simple sugars (mainly glucose) in the product [56]. As shown in Table 3, DE of SDexF varied from 4.48 to 4.57, where the average value was 4.51 ± 0.05.

Table 3. Dextrose equivalent (DE) of native potato starch and SDexF.

Sample	Dextrose Equivalent (DE)
Native potato starch	0.20 ± 0.10 [a]
SDexF1	4.48 ± 0.09 [b]
SDexF2	4.45 ± 0.05 [b]
SDexF3	4.54 ± 0.08 [b]
SDexF4	4.57 ± 0.09 [b]

[a,b] Distinct letter superscripts within identical columns denote statistical significance ($p < 0.05$).

No statistically significant difference was observed between the results for individual batches. DE values were higher for SDexF obtained on a semi-industrial scale compared to SDexF previously obtained on a laboratory scale, where DE was less than 3.00 [22,50]. Generally, higher DE values in SDexF indicate a higher degree of hydrolysis and the presence of more simple sugars. Higher DE values might correspond to higher solubility due to the breakdown of starch molecules into shorter saccharide chains [57]. Greater DE and solubility values for SDexF indicate a stronger depolymerisation of starch [27].

3.5. Quantitive Determination of Citric Acid and Ethanol in SDexF

The quantitative determination of citric acid and ethanol was performed to assess the efficiency of the washing process of SDexF with ethyl alcohol and subsequent drying. The citric acid and ethanol content in SDexF are presented in Table 4. Average residues of citric acid and ethanol in SDexF were 0.006 ± 0.003 g/100 g and 4.40 ± 0.24 g/100 g, respectively. The amount of residual citric acid in the samples was very low and not significantly different, which indicates the repeatability of the applied dextrinization conditions. Furthermore, it confirms the effectiveness of washing SDexF with ethyl alcohol to remove possible residues of citric acid. European law allows the use of citric acid (E330) as a food additive in many food products at quantum satis levels [58]. SDexF is intended to enrich vegetable and fruit mousses designed for consumption by children. Therefore, it is important that the product does not contain ethanol. With the assumed addition of SDexF to the mousse (5 g), the alcohol content will be less than 0.5%, which is below the permitted amount for non-alcoholic drinks [59].

Table 4. Citric acid and ethanol content [g/100 g] in SDexF1-SDexF4 batches.

Sample	Citric Acid Content	Ethanol Content
SDexF1	0.006 ± 0.003 [a]	4.16 ± 0.02 [a]
SDexF2	0.008 ± 0.002 [a]	4.22 ± 0.22 [a]
SDexF3	0.007 ± 0.002 [a]	4.65 ± 0.09 [b]
SDexF4	0.008 ± 0.003 [a]	4.56 ± 0.06 [b]

[a,b] Distinct letter superscripts within identical columns denote statistical significance ($p < 0.05$).

3.6. The Nutrient Content and Energy Value of SDexF

All tested features regarding the nutrient content in SDexF are presented in Table 5 as mean values along with standard deviation. For a complete picture of the nutritional value, the table also includes the average total fibre content (discussed in Section 3.1). The energy value of SDexF was 296.66 kcal/100 g (1238.56 kJ/100 g), which is not a very high amount, considering that SDexF was added in the amount of 5 g to vegetable and fruit mousses, which gives a value of approximately 60 kcal. The starch content in SDexF was 29.06 g/100 g. The amount of fat was below the Limit of Quantification (LOQ), which was 0.04 g/100 g. The fatty acid content, including saturated fatty acids (SFA), monounsaturated fatty acids (MUFA), polyunsaturated fatty acids (PUFA), trans fatty acids (TFA), omega-3 fatty acids and omega-6 fatty acids was below 0.06 g/100 g. The nitrogen content was 0.013 g/100 g and calculated on this basis as protein content was 0.08 g/100 g. These parameters prove that SDexF contains small amounts of fat and protein but contains 55.84 g/100 g carbohydrates. Total ash content was also tested and amounted 0.39 g/100 g. The sodium chloride content was as low as 0.1 g/100 g.

Table 5. Nutrient content and energy value of SDexF.

Tested Parameter	Result ± Uncertainty	LOQ
Energy value	295.66 kcal/100 g 1238.56 kJ/100 g	n.a. (not applicable)
Fat, including:	<LOQ	0.04 g/100 g
- SFA	<0.06 g/100 g	n.a.
- MUFA	<0.06 g/100 g	n.a.
- PUFA	<0.06 g/100 g	n.a.
- TFA	<0.06 g/100 g	n.a.
- omega-3	<0.06 g/100 g	n.a.
- omega-6	<0.06 g/100 g	n.a.
Digestible carbohydrates, including:	55.84 g/100 g	From calculations, based on the test results given in this study
- total sugars	4.49 ± 0.24 g/100 g	0.43 g/100 g
Starch	29.06 ± 2.53 g/100 g	n.a.
Dietary fibre	31.83 ± 1.93/100 g	n.a.
Sodium chloride	0.10 ± 0.01 g/100 g	0.05 g/100 g
Nitrogen	0.013 ± 0.00 g/100 g	0.018 g/100 g
Protein	0.08 ± 0.00 g/100 g	n.a.
Ash	0.39 ± 0.01 g/100 g	0.01 g/100 g

3.7. Assesing the Presence of Contaminants in SDexF

In the SDexF, the content of metals particularly harmful to health was determined due to their high toxicity. The levels of lead, cadmium, and mercury in the samples were below LOQ (Table 6). This means that SDexF does not contain any metals that may impair health and is safe for potential consumers. Additionally, SDexF was microbiologically tested (Table 7). According to the method used, *Salmonella* spp. was not detected in 25 g of preparation. Tests were also performed to detect *Listeria monocytogenes* and β-glucuronidase-positive *Escherichia coli* in SDexF, resulting in both being < 10 CFU/g, which is a lower value than LOQ.

Table 6. Content of selected heavy metals in SDexF.

Contents of Heavy Metals, Including:	Result	LOQ
lead	<0.010 mg/kg	0.010
cadmium	<0.005 mg/kg	0.005
mercury	<0.005 mg/kg	0.005

Table 7. Selected microbiological tests of SDexF.

Presence of Bacteria of the Genus	Result
Salmonella spp.	not detected in 25 g
Listeria monocytogenes	<10 jtk/g
β-glucuronidase-positive *Escherichia coli*	<10 jtk/g

3.8. Fourier Transform Infrared (FT-IR) Spectra of SDexF

In the spectra of potato starch, bands characteristic of polysaccharides were observed, i.e., those occurring in the fingerprint region (wavenumber range 1500–650 cm^{-1}), corresponding mainly to the skeletal vibrations of the glucopyranose ring and occurring at a wavenumber of 1650 cm^{-1}, attributed to the bending vibrations of the H-O-H bonds absorbed by the polymer of water molecules. The FTIR spectrum of starch also showed a very broad band in the range of 3500–3000 cm^{-1}, corresponding to the stretching vibrations of inter- and intramolecular hydrogen bonds of hydroxyl groups. When comparing the spectra of starch before and after dextrinization carried out on a semi-industrial scale, apart from changes in the intensity of the bands, no changes in their position, i.e., shifts resulting from stretching or bending vibrations, were observed (Figure 2).

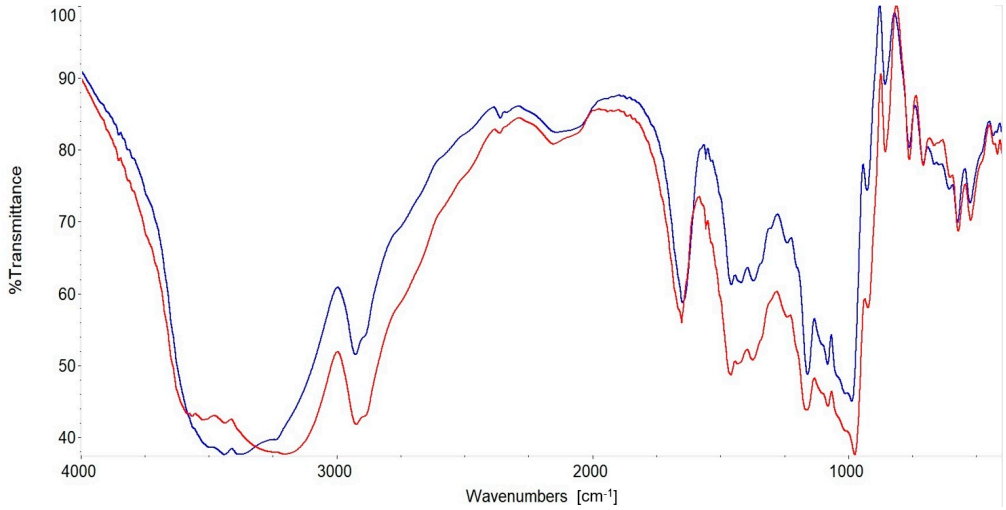

Figure 2. FTIR spectra of native potato starch (blue) and SDexF (red).

The possible differences in the intensity of the bands in starch after dextrinization are related to quantitative changes in relation to specific functional groups, mainly hydroxyl, and thus from the reorganization of hydrogen interactions. Similar results were obtained in another paper concerning structural studies of resistant dextrin [60].

3.9. Scanning Electron Microscopy (SEM) of SDexF

According to SEM images of potato starch, granules of irregular shape (oval or spherical) and sizes (in a wide range from 10 to 80 μm) with clear edges and a smooth surface were observed. Dextrinization led to a partial loss of granularity and obtaining products with a more or less deformed surface, as shown in Figure 3.

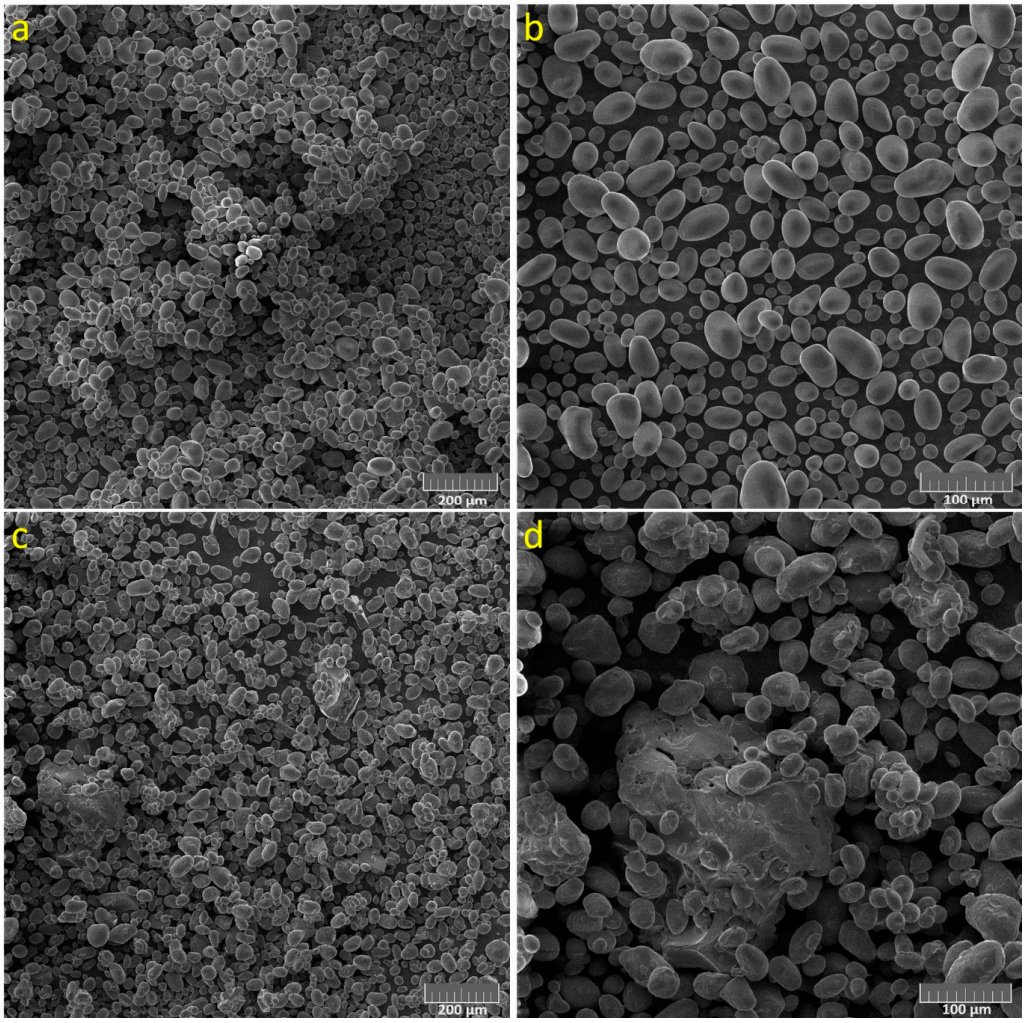

Figure 3. SEM images of native potato starch (**a**,**b**) and SDexF (**c**,**d**) at 200× and 500× magnification (left and right sides, respectively).

The surface deformations of granules indicate the physicochemical factors during dextrinization (acid hydrolysis, endothermic changes). The outer layers of granules, especially amorphous areas, are most susceptible to this type of influence [57]. Moreover, the damaged granules agglutinated to a greater or lesser extent, thus taking on forms that are difficult to identify. The analysis of SEM images confirmed that the morphological changes recorded in the case of non-selective dextrinization were much less noticeable than in the case of, for example, obtaining maltodextrins, where the granular structure is completely destroyed [61].

3.10. Pasting Properties of SDexF

Applied potato starch modification affects interactions with water molecules, impacting solubility while also altering the ability to form gels or impact viscosity during heating. In this study, SDexF obtained on a semi-industrial scale did not show pasting ability, even at a concentration four times higher than native potato starch. All pasting property parameters for SDexF were very low and significantly lower than for starch (Table 8).

Table 8. Pasting property parameters [mPas] of native potato starch (5% concentration) and SDexF (20% concentration).

Sample	PV	HPV	BD = PV − HPV	FV	SB = FV − HPV
Native potato starch	1704 ± 344	1117 ± 52	587 ± 392	1986 ± 114	869 ± 62
SDexF	28 ± 4	13 ± 3	16 ± 2	21 ± 2	7 ± 1

PV—peak viscosity, HPV—hot paste viscosity, BD—breakdown, FV—final viscosity, SB—setback.

Comparing the pasting property curves of starch and SDexF, one of the SDexF batch curves is presented with a potato starch curve in Figure 4. As observed in this research, the inverse relationship between solubility in water and pasting properties of SDexF can be attributed to the structural changes caused by their production process. SDexF was characterized by very low values close to those characteristic of Newtonian fluids [55]. Such low values confirm that SDexF will not thicken the enriched mousse. The results for SDexF obtained on a semi-industrial scale were consistent with those obtained for SDexF prepared on a laboratory scale [22]. In studies by other authors, starches modified by acids and enzymes also showed lower pasting parameters compared to native starches [62,63].

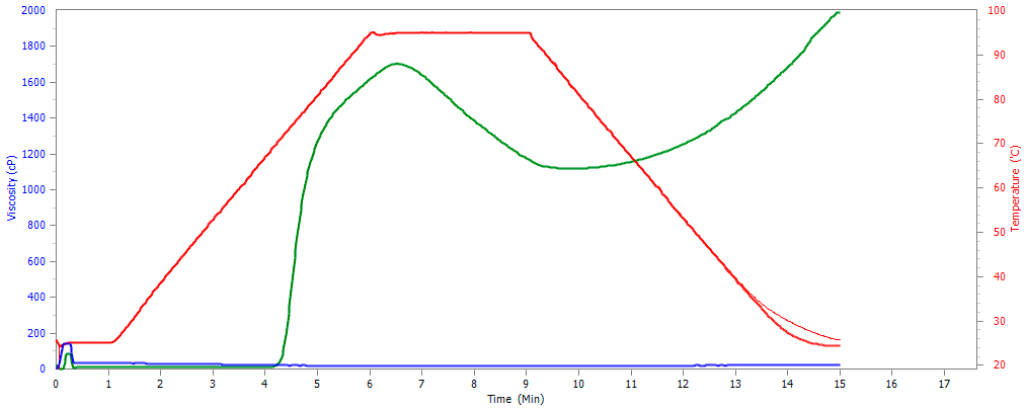

Figure 4. The pasting profile of native potato starch (green; 5% concentration) and SDexF (blue; 20% concentration) and the temperature profile used (red).

3.11. Retrogradation Tendency of SDexF

Starch retrogradation refers to the process where gelatinised starch, which has been heated and swelled in water, undergoes structural changes upon cooling. This cooling leads to the rearrangement and reassociation of amylose and amylopectin molecules within the starch granules, resulting in the formation of a more ordered and crystalline structure [64]. Foods containing retrograded starches may lose their desired taste, moistness, and freshness due to the changes in texture and structure over time, affecting consumer acceptance. Additionally, this process can change the functional properties of the product and cause deterioration of the overall quality [65]. Therefore, it is important that starch modification can inhibit the retrogradation process. To compare the retrogradation ability of potato starch and SDexF, a turbidimetric method was used. In the beginning, on the day of preparing slurries, both SDexF and native starch exhibited low initial turbidity values (Figure 5).

The turbidity of SDexF slightly increased throughout the entire storage period (by 0.021), while native potato starch showed a significant increase in turbidity, especially after the third day of storage. On the last day of storage (21st day), SDexF still had a lower turbidity than native potato starch on the day of preparation. Additionally, an approximately 15-fold increase in the turbidity of native potato starch and an approximately 1.3-fold increase in turbidity of SDexF were observed throughout the study period. Compared to SDexF obtained on a laboratory scale [22], the turbidity values for SDexF were slightly lower. With reference to pasting properties of SDexF, setback viscosity indicates the tendency of starch to retrogradation [66] and in this study, low SB values are associated with a low tendency of retrogradation of SDexF.

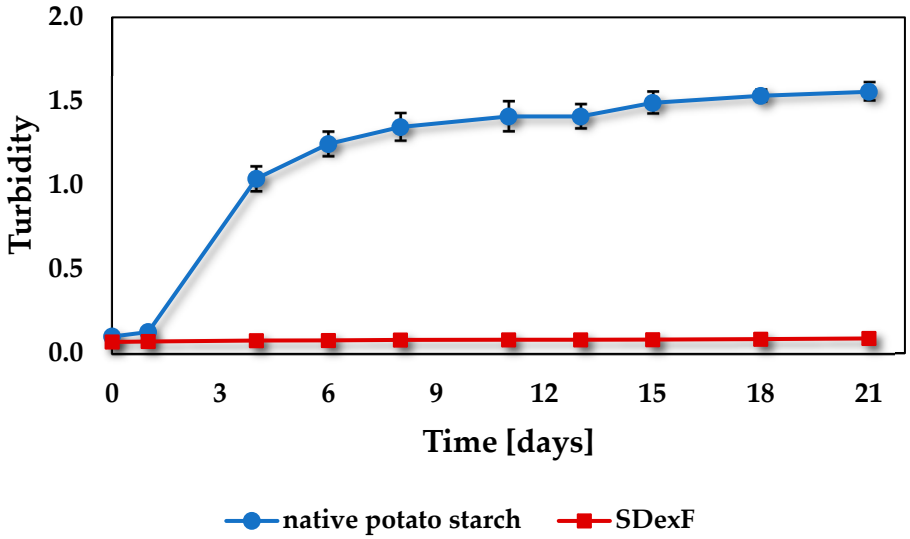

Figure 5. Retrogradation tendency of native potato starch and SDexF measured as turbidity.

3.12. Colour Parameters of SDexF

The colour parameters of native potato starch and SDexF are shown in Table 9. Regarding native potato starch, the a* parameter indicated a minimal share of green (negative value), while the b* parameter indicated the presence of a slightly yellow colouration (positive value). According to the SDexF colour parameters, it was more yellow than native starch because it had a similar negative value of a* parameter, but a much positive value of b* parameter. A comparison of photos of native potato starch and SDexF is shown in Figure 6.

Table 9. Colour parameters of native potato starch and SDexF.

Sample	L*	a*	b*	ΔE
Native potato starch	94.90	−0.19	1.00	-
SDexF	92.11 ± 0.67	−0.54 ± 0.04	10.46 ± 1.73	9.88 ± 1.81

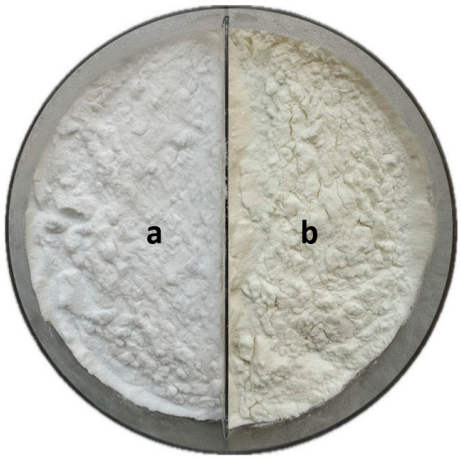

Figure 6. Colour comparison of native potato starch (**a**) and SDexF (**b**).

Although, the L* of SDexF was close to the value obtained for potato starch of native potato starch and it seems that samples had similar, the difference in colour (ΔE) was high and reached almost 10. This value was similar to the ΔE value of pyrodextrins obtained from normal and waxy maize starch obtained by heating at 140 °C in the presence of hydrochloric acid and heated at 140 °C [67,68]. A colour close to white is desired by food

manufacturers because the product should be visually appealing and additives should not negatively affect enriched food product colour.

4. Conclusions

The characterisation of the obtained SDexF indicates the successful application of a semi-industrial scale for dextrin production. SDexF was characterised by parameters and properties allowing its addition to functional products, i.e., vegetable and fruit mousses. SDexF showed practically 100% solubility in water, TDF content of over 30%, low pasting parameters and no tendency to retrogradation. The presented study is an introduction to further innovative research on the production of SDexF on an industrial scale. Further scale-up may enable broader cooperation with the industry and the implementation of the product as a food ingredient, increasing the dietary fibre content. In future research, non-patented conditions for modifying native potato starch could be considered that would increase dietary fibre content and process efficiency from 70% to more.

Author Contributions: Conceptualization, J.K.; methodology, K.K.; formal analysis, M.W.; investigation, M.W., K.K. and A.Z.; data curation, M.W., K.K. and A.Z.; writing–original draft preparation, M.W. and A.Z.; writing—review and editing, K.K. and J.K.; supervision, K.K. and J.K.; funding acquisition, J.K. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by The National Centre for Research and Development, grant number POIR.04.01.02-00-0102/17-00.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: All data are included in the article.

Conflicts of Interest: The authors declare no conflicts of interest.

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