Analysis of Indigestible Dextrin

Introduction

Dietary fiber is a general term for indigestible food components that are difficult to digest by human digestive enzymes. Dietary fiber has preventive effects on dyslipidemia, constipation, obesity, and diabetes, as well as on arteriosclerosis by regulating lipid metabolism. Its many physiological functions have been attracting people's interests.

There are two types of dietary fibers: insoluble and water-soluble. Insoluble dietary fibers are mainly the structural substance of plant cell walls. Water-soluble dietary fibers are found inside plant cells, and are storage substances and secretions which dissolve in water to become gel-like substances. Table 1 lists typical dietary fibers.

Table 1 List of common dietary fibers

Water-soluble dietary fibers	Insoluble dietary fibers
Pectin	Cellulose
Glucomannan	Hemicellulose
Sodium alginate	Lignin
Guar gum	Agar
Chondrotin sulfate	Chitin
Low-molecular-weight alginic acid	
Indigestible dextrin	
Polydextrose	
Inulin	

Water-soluble dietary fibers are further categorized into two types: high-molecular-weight water-soluble dietary fibers and low-molecular-weight water-soluble dietary fibers. High-molecular-weight water-soluble dietary fiber is a component conventionally referred to as water-soluble dietary fiber. Low-molecular-weight water-soluble dietary fiber refers to indigestible oligosaccharides that are listed in the Standard Tables of Food Composition in Japan (Supplement 2018 edition).

In Japan, indigestible dextrin is approved to label as a Food for Specified Health Uses (FOSHU). This labeling system was started in 1991. The Japanese Director-General of the Consumer Affairs Agency approves certain food products to label their effects.

For nutritional labeling of dietary fiber, the enzyme-gravimetric method (Prosky method) has been used. With this method, samples are treated by enzymatic reaction, precipitated using about 80% methanol, and filtered. The residual part contains the dietary fiber, protein, and ash. Prosky method works well for the measurement of high-molecular-weight water-soluble dietary fiber, but not for the low-molecular-weight water-soluble dietary fiber. An alternative is to use enzyme-HPLC method, especially for samples containing a large amount of low-molecular-weight water-soluble dietary fiber. The enzyme-HPLC method used in this application was performed following the FOSHU standards. It enrolls a series of sample pretreatment and an HPLC analysis. The determined dietary fiber fraction peak area and the glycerin internal standard peak area are compared to obtain their ratio. The following introduces the analysis of indigestible dextrin in food using the said method.

Methods

1. Sample

Three commercial indigestible dextrin-containing supplements were analyzed in this study (Two powder products and one beverage). Those products were pretreated following the FOSHU standards method. The detailed pretreatment procedure is summarized in Figure 1.

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Enzyme Treatment

Dissolve the sample in 0.08 M phosophate buffer (Injection sample (a))
(powder sample 1 g + 50 mL phosphate buffer,
beverage sample 5 mL+ 45 mL phosphate buffer)
↓
Add 0.2 mL of α-amylase
↓
Let it react at pH 6.3 and 90 ℃, with shaking for 30 minutes
↓
After cooling, add 0.2 mL of protease and amyloglucosidase
↓
Let it react at pH 6.3 and 60 ℃, with shaking for 30 minutes
↓
Heat at 100 ℃ for 10 minutes
↓
After cooling, add glycerin (diluted 10 times)*1
(powder sample + 5 mL, beverage sample + 3 mL)
↓
Make up to 100 mL with water
Obtained is the enzyme-treated solution (Injection sample (b))

Purification

Enzyme-treated solution 50 mL
↓
Pass through an ion exchange resin*2 at 50 mL/h
↓
Pass through approximately 150 mL of water
(to obtain 200 mL of effluent)
↓
Concentrate using a rotary evaporator to
20 mL (powder sample) and 10 mL (beverage sample)
↓
Filter using a 0.45 μm membrane filter
↓
Obtained is the purified solution (Injection sample (c))

*1 Glycerin is an internal standard
*2 Ion Exchange Resin used was a mixture of Amberlite™ IRA-67 and Amberlite™ 200CT (DuPont™) by mixing 25 mL each of resin.

Fig. 1 Sample pretreatment flow for indigestible dextrin

2. Calculation

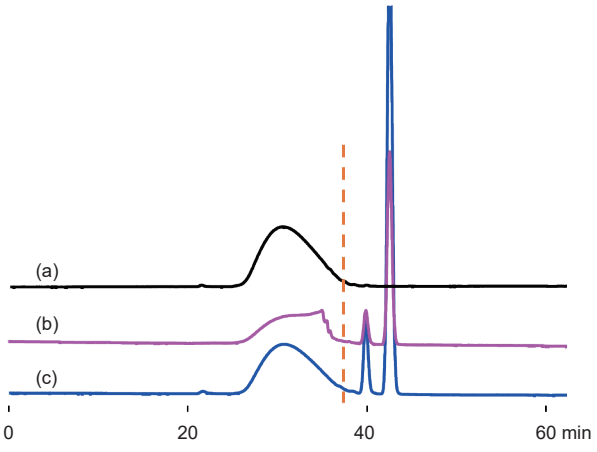
The formula below described in FOSHU standards, in the section for “indigestible dextrin” (measured as a dietary fiber) was used.

Dietary fiber components content (%) = (peak areas of dietary fiber/peak area of glycerin) x f1* x (weight of glycerin internal standard (mg)/weight of sample (mg)) x 100

*f1: Glycerin and glucose peak area sensitivity ratio = 0.82

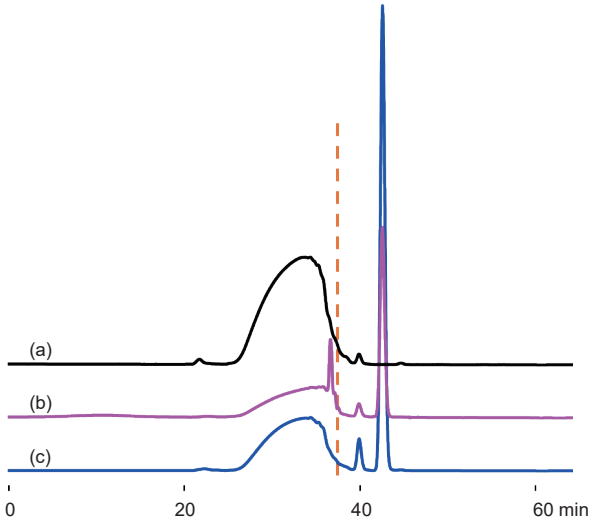
Results

Three commercially available indigestible dextrin-containing products were analyzed after the pretreatment described in Fig.1. For each product, three injection samples were prepared: (a) untreated (the sample dissolved in phosphate buffer), (b) enzyme treated (the sample dissolved in phosphate buffer and performed enzyme treatment), and (c) purified sample (the enzyme-treated sample processed with ion exchange resin and concentrated). Figures 2 and 3 show the analysis results of two indigestible dextrin-containing powder samples, and Fig. 4 shows the analysis results of the indigestible dextrin-containing beverage. Chromatograms of three injection samples were adjusted to the same y-axis scaling.



Sample : Indigestible dextrin (powder sample A)
(a) untreated, (b) enzyme treated, (c) purified, 20 μL
Column : Shodex OHpak LB-G 6B (6.0 mm I.D. x 100 mm,13 μm)
+ OHpak LB-802.5 × 2 (8.0 mm I.D. x 300 mm, 6 μm)
Eluent : H_2O
Flow rate : 0.5 mL/min
Detector : RI
Column temp.: 80 ºC

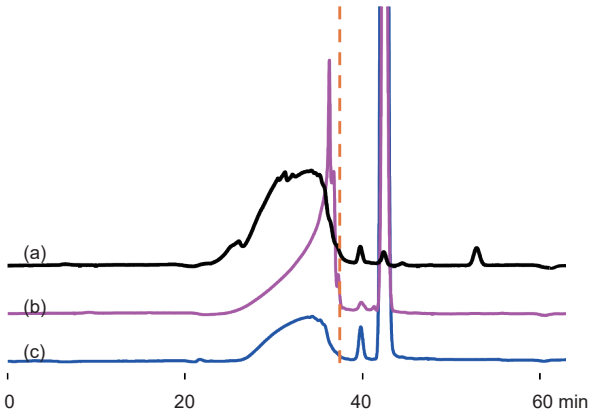
Fig. 2 Chromatograms of insoluble dextrin (powder sample A)



Sample : Indigestible dextrin (powder sample B)
(a) untreated, (b) enzyme treated, (c) purified, 20 µL

Refer to Fig. 2 for the analytical conditions

Fig. 3 Chromatograms of insoluble dextrin (powder sample B)



Sample : Indigestible dextrin (beverage sample)
(a) untreated, (b) enzyme treated, (c) purified, 20 µL

Refer to Fig. 2 for the analytical conditions

Fig. 4 Chromatograms of insoluble dextrin (beverage sample)

According to the Standard Tables of Food Composition in Japan, the low-molecular-weight water-soluble dietary fibers are considered as compounds elutes before maltotriose at around 38 minutes (dotted line). The peak at 40 minutes is glucose and the peak at 41.5 minutes is glycerin added as an internal standard. Although the commercial products contain many components, the measurements of indigestible dextrin was not influenced from the presence of other components.

Using the obtained results (purified sample) for each product, the content of indigestible dextrin was determined using the formula mentioned in 2.

Sample	Content
Powder Sample A	67.5%
Powder Sample B	55.5%
Beverage Sample	1.1%

The manufacturers of two powder products do not mention about their dextrin contents. Meanwhile the indigestible dextrin content of the beverage product mentioned was 5 g/480 mL (about 1.04%). It was close to the value obtained from the measurement.

Conclusions

An enzyme-HPLC method, according to the FOSHU standards, was used to analyze commercially available indigestible dextrin containing products. The method was feasible extracting and analyzing the indigestible dextrin. Analysis of indigestible dextrin requires the column oven setting to be at 80°C. Since Shodex OHpak LB-802.5 can be used with a maximum temperature of 80°C, the column was suitable for this method.

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