Pitfall in the determination of the dietary fibre content and nutritional value of food products

Kommer Brunt

Carbohydrate Department, Eurofins Food, Leeuwarderstraatweg, Heerenveen, The Netherlands

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Correspondence:
Kommer Brunt, Carbohydrate Department, Eurofins Food, Leeuwarderstraatweg 129, 8441 PK Heerenveen, The Netherlands.
FAX: +31(0)88 831 01 00
Email: k.brunt@eurofins.nl

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Abstract

Dietary fibre and nutritional value of food in a broad variety of matrices are frequently requested analyses. In the situation that the analysing laboratory is not informed about the addition of specific ingredients such as low-molar weight dietary fibre ingredients/prebiotics (e.g. inulin/FOS, GOS, resistant maltodextrins) for the fortification purposes of the dietary fibre content in the product, erroneous results can be obtained. In this paper two case studies dealing with this problem are described. The case studies concern firstly a local industrial bakery who tried to develop a healthier product with an increased dietary fibre content and a lower nutritional value and secondly a candy manufacturer.

Introduction

It is generally recognized that dietary fibre is an essential part of the human diet. In our western world the daily intake of dietary fibre is considerably less than the recommended intake of 14.2 g/1000 kcal energy intake for adults (Institute of Medicine, 2002). In practice this means an advised daily intake of about 35 g dietary fibre for a man for an energy intake of 2500 kcal and of about 28 g dietary fibre for woman for a daily energy intake of 2000 kcal.

Dietary fibre is strongly associated with the natural constituents in food. However, apart from the dietary fibres which are naturally present in our food, nowadays a whole range of a new generation of industrially prepared low-molar weight dietary fibres (prebiotics) products are commercially available.

Food manufacturers are continuously improving their food products in order to produce healthier products. In the last decade numerous new products were launched globally with statements regarding their high-(dietary) fibre contents. Mostly they included cereal and/or bakery products, energy bars and breakfast cereals but it also involved products in the category of beverages and dairy products. Beneficial effects of dietary fibres are related in various ways to their composition (Health Council of The Netherlands, 2006). Fibres in food are linked to reduced risks of constipation, cardiovascular diseases, obesity, diabetes and certain cancers. Fibre consumption has also been related to factors contributing to well-being, including satiety. With growing concerns about diabetes and obesity in our affluent western society, it is expected that the efforts of the food industry to launch new products with increased dietary fibre content and decreased nutritional value will further increase.

Presently food products are fortified with different dietary fibre ingredients, ranging from classical natural high-molar weight (HMW) dietary fibres [e.g. cereal based β-glucans, hemicelluloses and pentosans, and fruit-based pectins, resistant starches (RSs)] to low-molar weight (LMW) soluble dietary fibres/prebiotics (e.g. resistant maltodextrins, inulin/fructose-oligosaccharides (FOS), galacto-oligosaccharides (GOS), and polydextrose).

Beneficial effects of dietary fibre are related in various ways to their composition. LMW dietary fibre, prebiotics, will enhance the growth of different microorganism in the intestinal track. HMW soluble dietary fibre constituents will contribute to the viscosity in the intestinal tract and will slow down the glucose up-take by the body. Insoluble (HMW) dietary fibre will remain mostly undigested and contribute to the bulking properties and reduce constipation. They also can bind cholesterol moieties as well as their bile acid derivatives and increase in that way the excretion of cholesterol (Lairon, 2004).

Based on physicochemical properties such as solubility and molar weight, dietary fibres can be subdivided into different groups (Figure 1), being HMW and LMW dietary fibre. Although RS basically belongs to the HMW dietary fibre, it is given in this scheme its own group, partly for analytical reasons and partly because of its special character (Sajilata *et al.*, 2006). Based on chemical and physical characteristics. RS is classified into four different types:

- RS1: starch that is physically inaccessible to digestive enzymes (e.g. due to the presence of intact cell walls in grains, seeds, leaves)
- RS2: native RS granules (e.g. raw potatoes and green bananas)
- RS3: retrograded starch in processed food and food products (cooked and cooled potatoes, bread, corn flakes)
- RS4: chemically modified starches

Methods for the determination of resistant starches in food and food products have been developed by Englyst *et al.*, (1992), and improved by among others Goni *et al.* (1996) and McCleary & Monaghan (2002).

Different analytical protocols (AOAC 985.29, AOAC 991.43, AOAC. 2001.03 and AOAC 2002.02) are available for the different classes of dietary fibres (Figure 2). Until now, the golden standard has been the AOAC 985.29 protocol for the determination of the total (soluble and insoluble) HMW dietary fibre. However, RS is just partly measured applying this method or applying the extended AOAC 991.43 protocol for discriminating between soluble and insoluble HMW dietary fibre. The RS1, RS2, and RS4 type resistant starches are not 'seen' by these two commonly applied methods. Only the retrograded RS3 type resistant starch will be measured with the AOAC 985.29 and 991.43 methods.

For the different LMW dietary fibres or prebiotica, different dedicated analytical tests (AOAC 997.08, AOAC 999.03, AOAC 2000.11 and AOAC 2002.02) are available (Figure 3).

There are a lot of different methods described in the literature for the analytical characterization of the non-digestible dietary fibre carbohydrates (Sanders & Brunt,

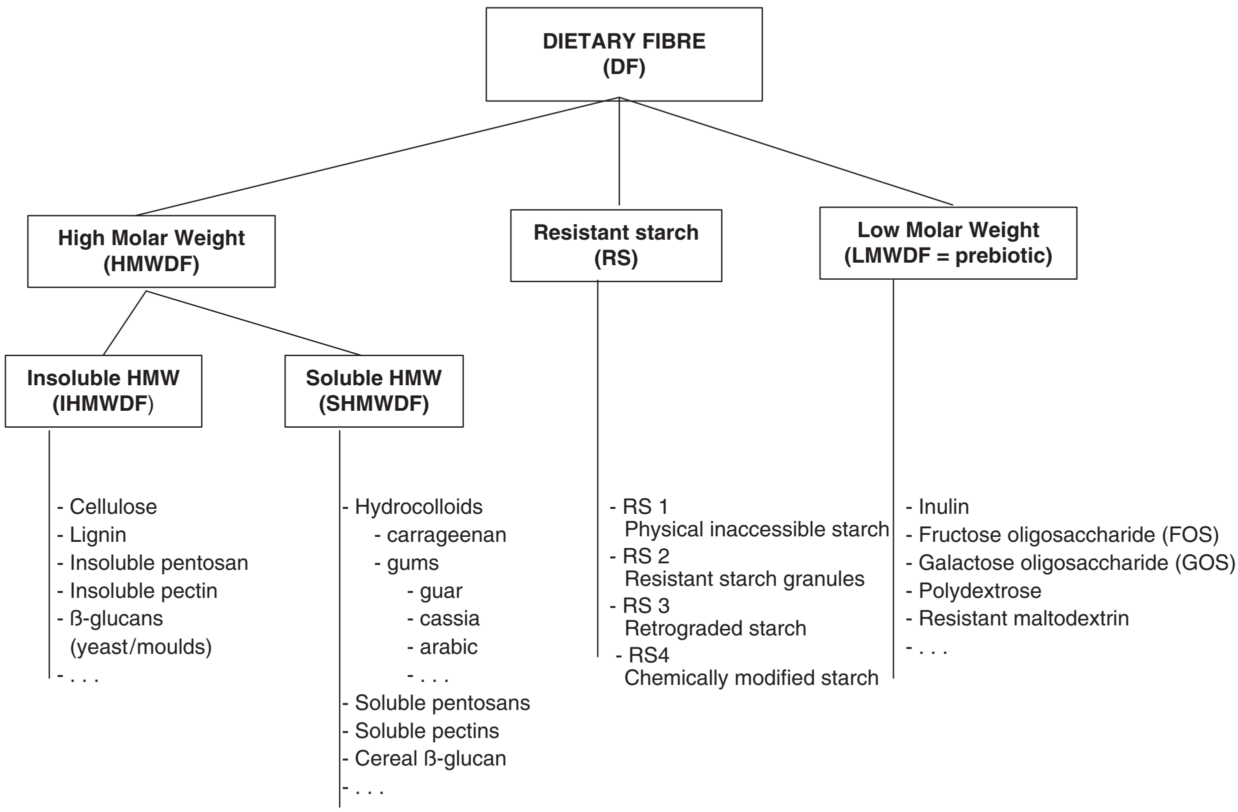

Figure 1 Subdivision of dietary fibre constituents in different groups.

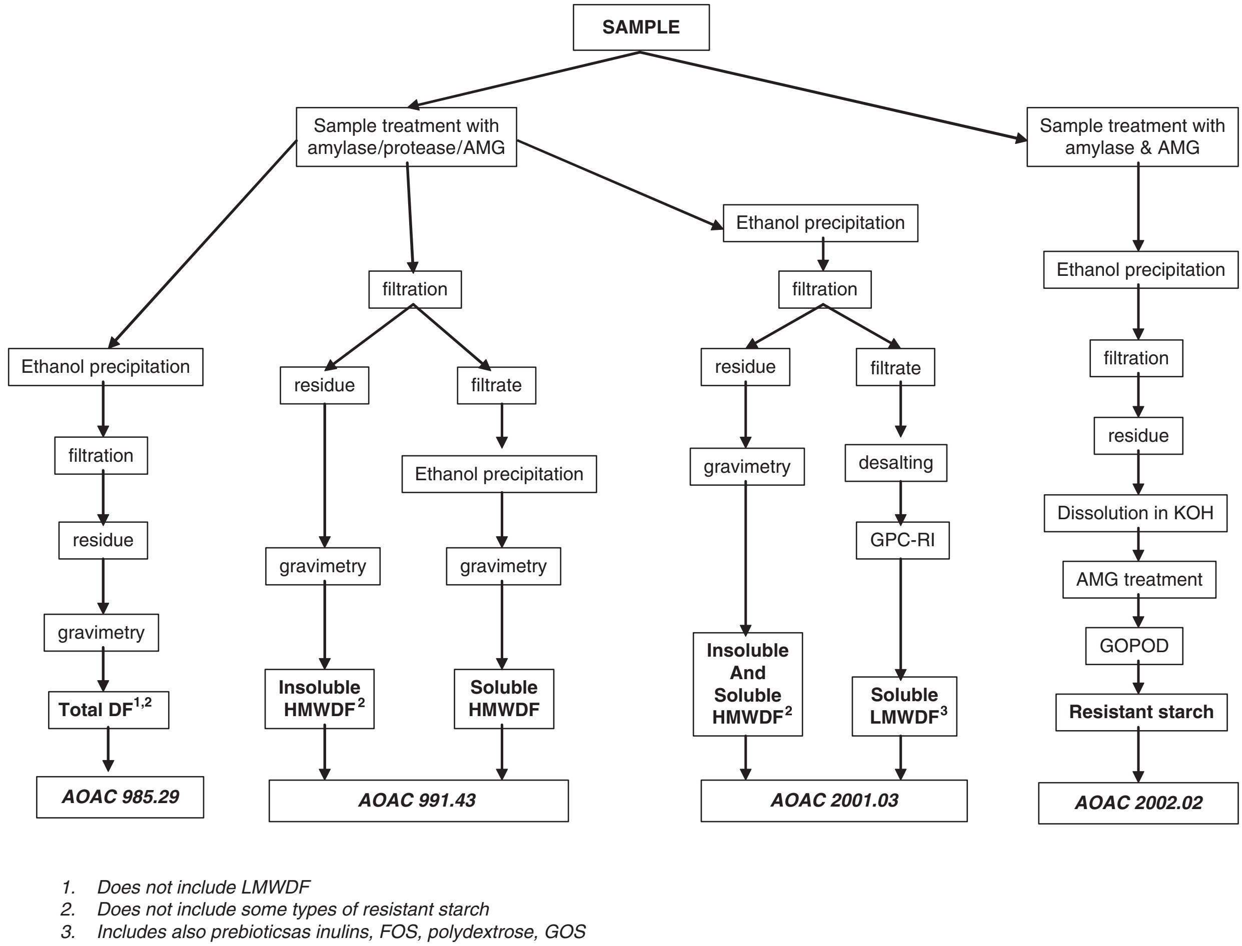

Figure 2 The different AOAC standard protocols for the measurement of the various classes of dietary fibres.

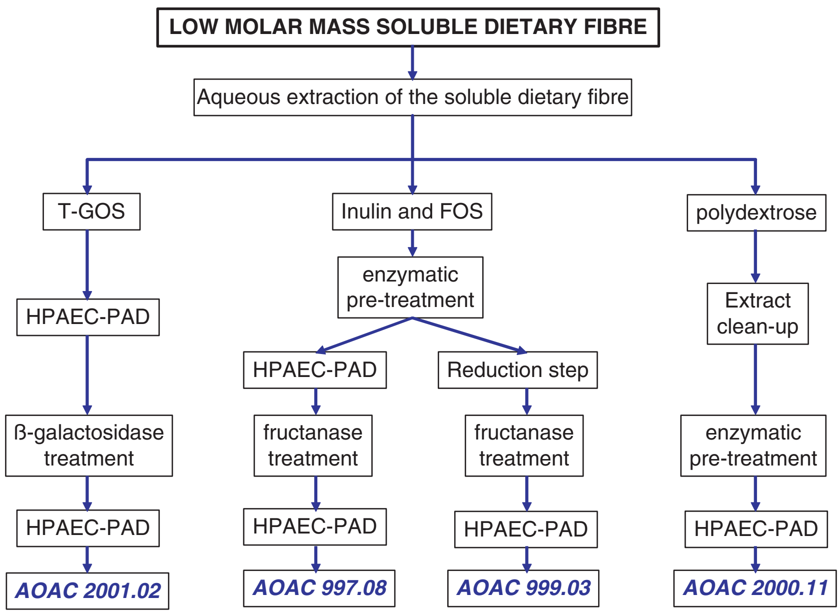

Figure 3 The AOAC standard protocols for the measurement of the various prebiotics or LMW dietary fibres.

2004). This diversity in tests for the determination of the (specific) fibre content in food products can be difficult for food manufacturers to understand. From our experience it is clear that mistakes are made in requesting the appropriate (combination of) tests for the determination of the total dietary fibre content and the nutritive value of food products. Also the reservation in communicating confidential information concerning the applied ingredients and the (new developed) formulations of manufactured products that have to be analysed, easily results in erroneous requests for analytical tests, ending with the delivery of incorrect results.

In this paper a frequently occurring incorrect and/or incomplete request for analytical support and its consequences are discussed. It concerns two typical examples of the application of a prebiotic in a food formulation.

Case study 1: Inulin containing fruit-filled biscuits

A local industrial bakery requests Eurofins Food several times a year for the standard total dietary fibre analyses (AOAC 985.29) and determinations of the nutritive value of his products. It mostly concerned raw materials and finished products – different types of (fruit-filled) biscuits and cookies. Some time ago we were asked to determine the total dietary fibre content and the nutritive value in a series of bakery products applying the same tests as before. The samples received looked quite normal and were comparable with samples we have received before for the same tests. Up to that time the quality department of the bakery was content with the reported results.

As had been the practice the AOAC 985.29 method was applied for the determination of the total dietary fibre content in the bakery products. The nutritive value was calculated, based on the measured product composition (moisture, fat, protein, total digestible carbohydrates, and dietary fibre) of the sample. The total digestible carbohydrates were measured by applying firstly the enzymatic hydrolyses of the starch and malto-oligosaccharides, followed by a weak acidic hydrolysis in order to hydrolyse the sucrose. Then the total sugar content in the hydrolysate was quantified by the Luff–Schoorl reduction method (NEN, 3571). The nutritive value is calculated using the following conversion factors (EU Council Directive): carbohydrates 17 kJ g^{-1} or 4 kcal, polyols 10 kJ g^{-1} or 2.4 kcal, protein 17 kJ g^{-1} or 4 kcal, fat 37 kJ g^{-1} or 9 kcal.

The reported results are summarized in Table 1.

The quality department of the bakery was not content at all with these results; they had made a serious effort to develop a healthier fruit-filled biscuit with a decreased nutritive value and enriched with dietary fibre. Due to their reluctance to communicate confidential information to the testing laboratory concerning the applied ingredients and the formulation of the manufactured fruit-filled biscuit, they requested inappropriate analytical tests. Knowing the formulation and the applied ingredients, the quality department of the bakery stated that the established dietary fibre content was much too low and the digestible carbohydrate content too high.

In discussion with the customer it became clear that about 10% of the prebiotic inulin was added in order to enrich the biscuits with LMW dietary fibre. We explained that inulin is not quantified with the classical AOAC 985.29 method and that therefore this test resulted in a much too low dietary fibre content. They also did not realize that due to the weak acidic hydrolysis necessary for converting the sucrose into its monosaccharides, the inulin was hydrolysed too into its monosaccharides, resulting in an erroneous too high total digestible carbohydrate content.

Table 1 Reported dietary fibre content and nutritive value of the fruit filled biscuit

Food constituent	Analytical method	Content (% w/w)	Nutritive value (kJ or kcal)
Moisture	Oven drying	9.7	
Protein	Kjeldahl	4.9	83 or 20
Fat	Soxhlet	11.5	426 or 104
Sugars	High-performance anion exchange chromatography		
Glucose		9.1	
Fructose		16.0	
Sucrose		3.7	
Lactose		1.5	
Maltose		0.1	
Total digestable carbohydrates	Enzymatic and weak acid hydrolyses +Luff-Schoorl	64.7	1100 or 259
HMW dietary fibre	AOAC 985.29	2.9	
Total nutritive value			1609 or 383

With this new information in mind some additional analyses were done. Firstly, the effect of inulin on the AOAC 985.29 test result was checked by the analysis of a biscuit sample spiked with an extra 7.0% inulin (Orafti, Raftilose P95). In spite of the added extra 7% inulin, the total dietary fibre content, measured with the AOAC 985.29 protocol, was 3.1%. The difference of 0.2% absolute compared with the previous result (Table 1) is within the experimental error of the total dietary fibre determination, indicating that the added inulin does not affect the results of the AOAC 985.29 total (HMW) dietary fibre content. And secondly, the inulin content in the sample was measured in with the AOAC 999.03 protocol. The established inulin content of 9.7% (w/w) was in good agreement with the expected value that the biscuit should contain about 10% inulin (recovery about 97%). The total digestible carbohydrate content was corrected for the (hydrolysed) inulin content (= 180/162 × inulin content), resulting in a content of 53.9% instead of the erroneously measured 64.7%. The results of the re-analyses and the recalculations are summarized in Table 2.

In summarizing, due to analyses with inappropriate methods, the established nutritive value was erroneously about 13% too high (383 instead of 340 kcal) and the total dietary fibre content much too low (2.9 instead of 12.6%).

Case study 2: Galacto-oligosaccharide (GOS) containing candy

A candy manufacturer requests Eurofins for the AOAC 985.29 total dietary fibre determination in different types of candy. Just as in the above case study with the bakery, the candy manufacturer did not communicate any information concerning the applied ingredients. Afterwards it appeared that one of the candy samples was an experimental candy product containing about 15% GOS. The manufacturer, without any analytical dietary fibre knowledge, did not realize that the GOS will not be quantified by applying just the ‘standard’ AOAC 985.29 total dietary fibre determination.

The AOAC 985.29 total dietary fibre analysis resulted in an erroneous much too low dietary fibre content of 2.0%. First an additional AOAC 2001.02 GOS analysis was done on the sample, which resulted in a GOS content of 14.2%. This was in good agreement with the expected 15%. Secondly the sample was spiked with an extra 10% GOS and analysed again with the classical total dietary fibre AOAC 985.29 protocol. This resulted in a total dietary fibre content of 2.1%, which is in good agreement with the previously obtained erroneous result of 2.0%, confirming our statement that the AOAC 985.29 protocol does not measures LMW dietary fibres.

Developed questionnaire for dietary fibre analyses

At present food products are often fortified with different dietary fibre ingredients, ranging from classical/natural HMW dietary fibres (e.g. cereal-based β-glucans, fruit-based pectins, resistant starches) to LMW dietary fibres/prebiotics (e.g. inulins, fructose-oligosaccharides, galactose-oligosaccharides, polydextroses, resistant maltodextrins). The necessity of re-analyzing samples should be kept to a minimum. Firstly, it is costly for the laboratories and secondly, it

Table 2 Results of re-analysis of the dietary fibre content and re-calculation of the nutritive value of the fruit filled biscuit

Food constituent	Analytical method	Content (% w/w)	Nutritive value (kJ or kcal)
Moisture	Oven drying	9.7	
Protein	Kjeldahl	4.9	83 or 20
Fat	Soxhlet	11.5	426 or 104
Sugars	High performance anion exchange chromatography		
Glucose		9.1	
Fructose		16.0	
Sucrose		3.7	
Lactose		1.5	
Maltose		0.1	
Total ‘digestable’ carbohydrates	Enzymatic and weak acid hydrolyses +Luff-Schoorl	64.7	
HMW dietary fibre	AOAC 985.29	2.9	
Inulin (LMW DF)	AOAC 999.03	9.7	
Total Dietary Fibre (HMW+LMW)		2.9+9.7 = 12.6	
Inulin corrected total digestible carbohydrate	Water correction for weak acidic inulin hydrolysis	64.7–10.8 = 53.9	916 or 216
Total nutritive value			1425 or 340

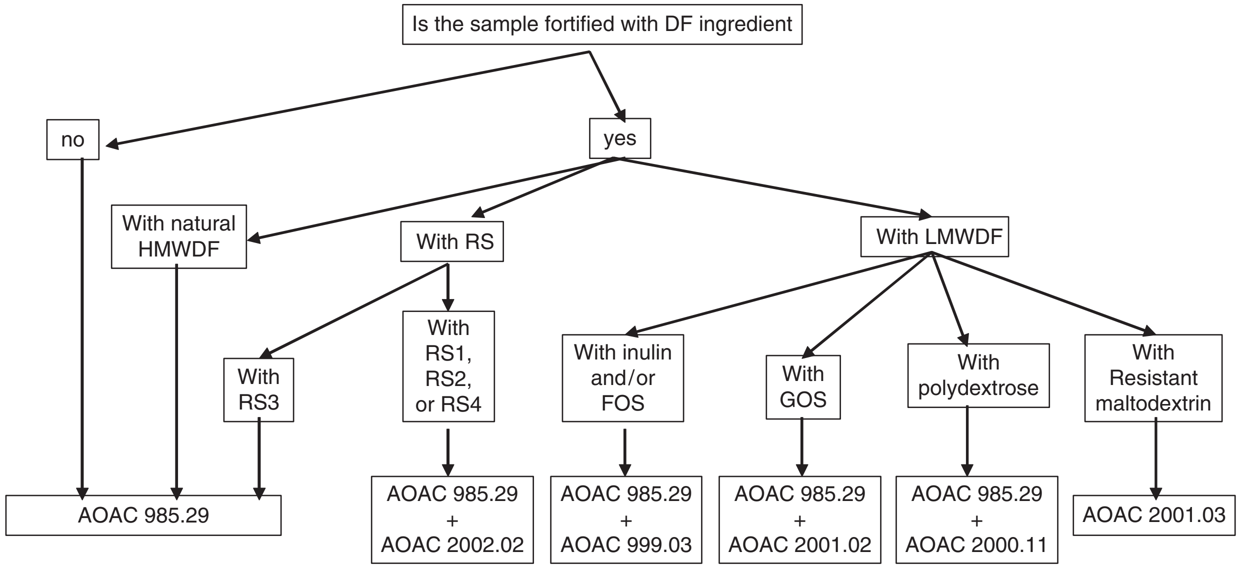

Figure 4 Questionnaire scheme for choosing analytical method(s) for correct determination of the dietary fibre content.

undermines the confidence in the laboratory results and expertise. Therefore Eurofins Food has developed a helpful scheme in order to facilitate the decision-making process as to which dedicated specific dietary fibre tests should be applied for a correct determination of the dietary fibre content in dietary fibre enriched food products (Figure 4) to be sure that the correct analyses/tests are requested for the broad variety of dietary, fibres which can be present in food products.

Since the developed questionnaire has been in use, considerably less errors are made in the dietary fibre analysis requests, resulting in a significant decrease in complaints when dealing with dietary fibre analyses and a more efficient use of the laboratory.

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