

FLAVOUR COMPOSITION

FIELD OF THE INVENTION

The present invention relates generally to the improvement of wholesome ingredients used in food and beverage products. In particular, the present invention relates to the use of bran to enhance the value of food and beverage products and impart appealing organoleptic properties, such as an improved flavour. Specifically, the present invention provides methods involving thermal treatment of bran, such as grain bran, for the preparation of flavour compositions having properties delaying the development of rancid off-notes in a food product, as well as uses of thus prepared compositions in food and beverage products.

BACKGROUND OF THE INVENTION

Bran, the outer layer of cereal grain, is particularly rich in dietary fibres and essential fatty acids. Bran also contains starch, protein, vitamins, dietary minerals, but also phytic acid, which is an anti-nutrient that prevents nutrient absorption. The high oil content of some brans makes them subject to rancidity.

Table 1: Composition of bran of different cereal origins (*i.a.* "Bran", Wikipedia, 2016; "Corn chemistry and technology", Watson and Ramstad ed., 1987).

Nutrient (% by weight)	Barley	Corn	Oat	Rice	Rye	Wheat
Carbohydrates (except starch)	70-80	80-90	16-34	18-23	50-70	45-50
Starch	8-11	5-10	18-45	18-30	12-15	13-18
Protein	11-15	7-10	13-20	15-18	8-9	15-18
Fat	1-2	0.9-3	6-11	18-23	4-5	4-5

Bran is an inexpensive waste material or by-product from various cereal-processing industries. For example, corn bran is a by-product from industrial production of corn flour and corn starch (Kamboj and Rana, 2014). Currently, bran is mainly used as an animal feed.

Nevertheless, bran is known for its high nutritional value. It is a source of essential whole grain elements (e.g. dietary fibre, phenolic compounds) and of important flavour precursors (e.g. five carbon monosaccharides) that can be of high interest for further use in the food industry. For instance, bran is an interesting ingredient due to the presence of dietary

fibre polysaccharides, including arabinoxylans. Arabinoxylan chains are made of 1,4-linked xylose units. Xylose units may be substituted with 2-, 3- or 2,3-linked arabinose residues.

Use of bran by the food industry is currently very limited. Some commercial food producers use bran as a filler in their foods to reduce the caloric value of snack foods. Bran is sometimes used to enrich breads (notably muffins) and breakfast cereals, especially for the benefit of those wishing to increase their intake in dietary fibre. Rice bran in particular finds many uses in traditional Japanese cuisine also for pickling, fermentation etc. In Romania and Moldova, fermented wheat bran is traditionally used when preparing borş soup. In Mexico, the ancient practice of “nixtamalization”, a sort of cooking in alkaline conditions, maximized the benefit of maize, which was the staple food of the area.

The especially high dietary fibre and phenolic acid contents of corn bran in particular would position it as a first choice raw material. Yet at present its usage as dietary fibre source in food is still limited as it remains mostly used for cattle feed, although valorisation for fuel ethanol and pharmaceutical additives has been described. Regarding health benefits, corn bran can be categorized as traditional insoluble dietary fibre with high bulking effect, as opposed to soluble fibre, more extensively fermented in the gut. Direct corn bran supplementation in foods to increase nutritional value has been assessed through preparation and evaluation of cakes, cupcakes, muffins and bread. Manufacture of acceptable products using extrusion and blending corn bran co-products (corn bran, defatted germ and gluten) with corn semolina up to 10% was reported (Sharma et al., 2012). Yet, as usually observed when adding fibre, sensory properties of products obtained are generally less appealing.

To make the most of corn bran as health beneficial food ingredient, the current route is rather to modify its structure or to extract valuable compounds from it, as Xylanase Modified corn Fibre (XMF) (Hu et al., 2010, 2008, 2008). Yet, both extensive presence of intra- and extra-molecular cross-linkage and presence of complex side chains renders corn bran cell wall structure difficult to access for enzymes. Release of hemicellulose fragments (*i.e.* (arabino)-xylo-oligosaccharides) from corn bran by either chemical (alkali, acidic or methanolic solutions) or physical treatments is a more popular approach. Corn bran may as well be valorised as a texture modifier. Much work has been done on extraction of Corn Fibre Gum (CFG), which appears to be an effective replacer of arabic gum as emulsifying agent (Yadav et al., 2007; 2007). Thermochemical production of xylo-oligosaccharides from wood and plant sources is another well-documented field of work and is usually accomplished by steam, dilute

mineral acids, or dilute alkaline solutions. Single-step production by reaction with steam or water through hydronium-catalysed degradation of xylans is known as microwave-assisted auto-hydrolysis and is an alternative to enzymatic treatments. None of these approaches is however targeted for food applications.

5 Extrusion could be considered as a relevant technique to incorporate dietary fibre-rich components such as corn bran, yet increasing dietary fibre levels while maintaining palatability of products is always challenging. Increasing dietary fibre concentration in formulations has almost invariably been found to reduce expansion volume of extruded food. Resulting products are dense, tough and non-crispy (Pai et al., 2009). While insoluble dietary
10 fibers will often reduce expansion and lead to texture less preferred by consumers, soluble dietary fibers (e.g. inulin, polydextrose, pectins) seem to have more moderate effects on texture of extrudates (Robin et al., 2012). They, however, represent a less cost-effective solution than insoluble dietary fibers which are more readily available and still underevaluated in human nutrition.

15 Poor functionality of insoluble dietary fibre-rich material such as corn bran could be overcome through modification of their characteristics before their incorporation into food. More generally, any process which can increase the technological versatility of dietary fibres could have a significant impact on their use in the food industry (Redgwell and Fischer, 2005).

Extrusion cooking has been considered as an approach to modify the functionality of
20 dietary fibres. US 4,500,558 reports a modification of functional properties of corn bran by applying high temperature and high shear during extrusion. As another option, insoluble dietary fibre may also be physically (e.g. milling or microwave treatment) or chemically (e.g. alkaline or acid treatment) treated prior to extrusion.

US 2003/0104103 discloses how to reduce unpopular bitter taste associated with bran.
25 The procedure is based on acidifying bran with an acid (pH 4-6) and treating with low levels of ozone to oxidize native bitter constituent, ferulic acid, preferably to vanillin to provide bran having a better flavour. It is claimed that during treatment concentration of ferulic acid decreases, whereas concentration of vanillin increases, both by factor of at least 50%.

Further patent documents relating to the background of the invention are:
30 US 4,435,430, which relates to a process for producing an all-natural, enzyme-saccharified cereal derived from whole grain, WO 2014/149810 disclosing continuous processes for improving the flavour and texture of bran and germ components, and EP 1 393 634

concerning processing of oat grain for conversion to a food product and improved methods to provide a toast flavour in oat groats through enhancement of Maillard reactions.

WO 2012/126972 describes a method for providing a whole grain cereal based extract, the method being based on grinding, hydrolysis of the macromolecular elements and
5 separating of the insoluble fraction, which then undergoes a second grinding and/or enzymatic modification obtaining a fraction having improved suspension properties. This fraction is then incorporated into the soluble fraction thus obtaining the whole grain cereal based extract.

WO 2006/127922 refers to stabilized whole grain corn flour having extended storage
10 stability and modified functional properties as well as to methods of making the same. WO 2007/011685 discloses wet milling of a slurry from whole grain rice and wheat to release all the protein, fat, fibre, and starch components, resulting in a slurry that can be heated to gelatinize the starch and the subsequent product can be dried. The heated slurry can be treated by enzymatic hydrolysis via the process of liquefaction and optionally saccharification
15 to produce whole grain rice milk products having diverse carbohydrate compositions.

WO 2016/091952 concerns a process for preparing a wet-treated bran product, as well as the wet-treated bran product as such. The process provides a wet-treated bran product having a small particle size and improved expansion properties.

US 2015/0359232 A1 discloses a wheat bran processed product which has a degree of
20 gelatinization of 45% to 100%, a lipid content of 3.8 mass % or less, a moisture content of 2.5 mass% or more and a grain size of 0.1 mm or more. The product is obtained by heating a wheat bran slurry to a temperature from 60°C to 150°C. The wheat bran slurry contains 100 to 300 parts by mass of water per 100 parts by mass of wheat bran. Preferably, the wheat bran is defatted by an overnight treatment in hexane. It is not clear from the disclosure which process
25 parameter contributes to the target gelatinisation and lipid content.

KR 2017/0077880 A discloses an extrusion process applied to a mixture comprising bran and 15% moisture at 100°C. The extruded bran is mixed with 10 times its weight of water, then it is then heated at 121°C for 1 hour under high pressure. After cooling, the slurry is hydrolysed with a cellulase.

30 WO 2014/149810 A1 discloses a process to improve bran and germ flavour. NL 6613978 A discloses a method for the production of wheat bran with a nutty taste and

smell, as well as a process for the preparation of bread. WO 2017/064172 A1 discloses a method of manufacturing a textured food product.

Ramezanzadeh et al (1999) report the effect of microwave heating, packaging and storage temperature on the production of free fatty acids in rice bran. Ertaş (2015) report the effect of wheat bran stabilisation methods on nutritional and physico-mechanical characteristics of cookies.

Rose DJ and Inglett GE (2010) report the production of feruloylated arabinoxylo-oligosaccharides (AXOS) from maize bran by microwave-assisted hydrolysis. This article does not discuss the preparation of flavoured compositions.

Rose DJ et al. (2010) review the utilisation of corn bran and corn fibre in the production of potentially higher-value food components. Corn bran and corn fibre contain potentially useful components that may be harvested through physical, chemical or enzymatic means for the production of food ingredients or additives, including corn fibre oil, corn fibre gum, cellulosic fibre gels, xylo-oligosaccharides and ferulic acid. Components of corn bran and corn fibre may also be converted to food chemicals such as vanillin and xylitol.

However, the potential of bran to become a useful and commercially valuable food ingredient has not been fully exploited yet. Namely, its potential for flavouring and shelf-life has not yet been exploited.

It would therefore be desirable to provide methods of valorisation of bran.

Any reference to prior art documents in this specification is not to be considered an admission that such prior art is widely known or forms part of the common general knowledge in the field.

SUMMARY OF THE INVENTION

The object of the present invention is to improve the state of the art, and in particular to provide compositions and methods that overcome the problems of the prior art and address the needs described above, or at least to provide a useful alternative. Hence, methods of treating bran are provided, thus preparing flavour compositions and compositions having properties delaying the development of rancid off-notes in a food product susceptible to rancidification.

The inventors were surprised to see that the object of the present invention could be achieved by the subject matter of the independent claims. The dependent claims further develop the idea of the present invention.

Accordingly, a first aspect of the invention relates to a method of preparing a flavour composition comprising the steps of:

- (a) providing a bran slurry comprising, or consisting of, bran and water, wherein the bran slurry comprises from 1% to 40% by weight of bran and has a water content of at least 20%, preferably at least 25%, by weight, based upon the weight of the bran slurry;
- (b) subjecting the bran slurry provided in step (a) to a heat treatment at a temperature of from 155°C to 200°C for a holding time of 5 to 180 min; and
- (c) obtaining the heat-treated bran slurry produced in step (b) as the flavour composition.

The method according to the first aspect may further comprise the steps of (d) filtering the heat-treated bran slurry to obtain a filtrate and residual solids, optionally drying the filtrate and/or the residual solids, optionally comminuting the dried filtrate and/or the dried residual solids.

Alternatively, the method according to the first aspect may further comprise the steps of (e) drying the heat-treated bran slurry to obtain a dried heat-treated bran, and optionally comminuting the dried heat-treated bran.

In an embodiment, the bran is provided in the form of ground bran. For instance, the bran is bran from cereals selected from the group of barley, corn, millet, oat, rice, rye, sorghum, spelt or wheat, or from pseudo-cereals selected from the group of buckwheat or quinoa.

In an embodiment, the heat treatment is performed by microwave treatment, autoclaving, direct steam injection, treatment in a tubular heat exchanger, or treatment in a scrape surface heat exchanger.

A second aspect of the invention relates to method of manufacturing a flavoured food product comprising the steps of:

- (a) providing a flavour composition obtainable or obtained by a method according to the first aspect;
- (b) blending said flavour composition and a food matrix;

(c) optionally subjecting the blend provided in step (b) to a food processing treatment selected from the group of extrusion-cooking, drying, roller-drying, spray-drying, baking, retorting, or toasting; and

(d) obtaining the flavoured food product.

5 A third aspect of the invention relates to a flavour composition comprising heat-treated bran obtainable or obtained by a method according to the first aspect, the flavour composition having properties delaying the development of rancid off-notes in a food product susceptible to rancidification.

10 In an embodiment, the flavour composition comprises an increased amount of a flavour compound selected from the group consisting of 2,3-butanedione, 2-acetylthiazole, guaiacol, 4-vinylguaiacol, vanillin, furfural, 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone, 2-acetyl-1-pyrroline, 2,3,5-trimethylpyrazine, 2-furylmethanethiol and 2-methyl-3-furanthiol, as compared to a composition comprising non-heat-treated bran.

15 In an embodiment, the flavour composition exhibits a flavour note selected from the group consisting of caramel, toasty, biscuity, vanilla-like, smoky, meaty, savoury, and spicy.

Another aspect of the invention relates to the use of a flavour composition according to the third aspect as a flavour ingredient in a food product.

20 Another aspect of the invention relates to the use of a flavour composition according to the third aspect as an ingredient for delaying the development of rancid off-notes in a food product susceptible to rancidification.

Another aspect of the invention relates to a method of flavouring and/or of delaying the development of rancid off-flavours in a food product, said method comprising using a flavour composition according to the third aspect.

25 Another aspect of the invention relates to a food product comprising a flavour composition according to the third aspect. In an embodiment, the food product is obtainable or obtained by the method according to the second aspect. For instance, the food product, or flavoured food product, is a roller-dried cereal product, a wafer, or an extruded product.

30 These and other aspects, features and advantages of the invention will become more apparent to those skilled in the art from the detailed description of embodiments of the invention, in connection with the attached drawings.

DETAILED DESCRIPTION OF THE INVENTION

The invention provides methods for valorisation of cereal bran by heat treatment. The inventors believe that the heat treatment may result in partial hydrolysis of the bran, which may develop antioxidative properties and may liberate some precursors, which may then become available for further reactions resulting in the formation of aroma compounds. The heat-treated bran exhibits several benefits, in particular (1) improved flavour properties, and (2) properties delaying the development of rancid off-notes.

Beyond taste, safety and nutrition, product labelling has become of key importance for many consumers, who often report preferring natural products and assume that products based on natural ingredients without additives are healthier (Cheung et al. 2016). There has indeed been a considerable increase in the demand for clarification regarding food additives (Carocho et al. 2014). In an inverse proportion, trust in food agencies has declined and consumers are more concerned about the food they eat. Various surveys indicate that consumers are alarmed about food additives and do not feel well informed regarding their role in food, despite their necessity (Carocho et al. 2014). There is thus now a generalized tendency to prefer products, ingredients and additives of natural origin (Paradiso et al. 2008). Behind the request for naturality, consumers seem to be seeking labels that do not display names of compounds that resemble chemical or synthetic names (Carocho et al. 2014).

There is currently a common trend, driven by consumer perception, to eliminate or replace artificial flavouring ingredients. In these circumstances, flavouring may be achieved either with natural flavours or ideally by application of "clean label" solutions. The latter and preferred alternative consists in generating flavour during the processing only from staple ingredients. Moreover, the use of flavourings is restricted in several product categories such as for example the field of infant nutrition. The creation of intense and desirable flavour that is preferred by the consumers without using a flavouring ingredient is made possible by the present invention.

Apart from flavours, the negative perception of consumers has extended to other functional ingredients such as antioxidants. Antioxidants are necessary to prevent the development of rancid off-notes in a food product, especially food products that contain components or ingredients susceptible to oxidation. Alternative solutions are thus needed to provide food ingredients with properties delaying the development of rancid off-notes in a food product susceptible to rancidification.

Replacing commercial antioxidants with thermally-treated bran could also bring cost savings thanks to the valorisation of a by-product, which, up to now, requires a separate handling. Finally, due to valorisation of bran during food processing, the methods of the invention contribute to improve environmental sustainability and to reduce the environmental footprint associated with the production a food product.

Definitions

The terms “heat treatment” or “thermal treatment” shall be considered as having the same meaning.

The term “flavour composition” refers to a composition which exhibits at least one flavour note and/or which contains at least one flavour compound. A “flavour compound”, herein also referred to as “aroma compound” or “odorant”, is detectable by technical or analytical means. A “flavour note” relates to a flavour composition or compound, and is detectable by sensory evaluation, e.g. by sniffing or tasting. A “flavour ingredient” refers to a flavour composition or to one or more flavour compounds.

The expression “properties delaying the development of rancid off-notes”, as used herein, refers to the properties of some food ingredients to improve the preservation of the food into which they are incorporated, and it relates to the improvement of food shelf-life or food shelf-stability. Thus, a composition or a compound having “properties delaying the development of rancid off-notes” is considered to qualify as a food preservative or food preserving ingredient, in particular for preventing or delaying lipid oxidation. Lipid oxidation may result in the development of rancid off-notes. The development of rancid off-notes in a food product may be designated as “rancidification” of the food product.

The term “a soluble fraction of heat-treated bran” is also referred to herein as “liquor” or “filtrate”.

The term “non-heat-treated bran”, herein also referred to as “native bran” or “original bran” refers to bran as obtained from a supplier and which has not been subjected to a thermal treatment. Also, native bran has not been subjected to a chemical treatment such as defatting with a solvent.

The term “food product”, as used herein, refers to foods and beverages suitable for consumption by humans, as well as feed products (“feed”) suitable for consumption by animals, in particular by pets such as cats and dogs.

As mentioned above, an aspect of the invention is a method of preparing a flavour composition. This method uses wet heating to treat bran. For instance, wet heating of the bran is performed by autoclaving in a batch reactor, microwave heating, heating in a tubular heat exchanger or in a scrape surface heat exchanger. In particular, the present invention uses
5 two procedures, i.e. microwave treatment and autoclaving, for treating bran.

The bran is a crop grain bran. The bran may be a cereal grain bran or a pseudocereal grain bran. Preferably, the bran is native bran. Examples of cereal grain bran include barley bran, corn bran, millet bran, oat bran, rice bran, rye bran, sorghum bran, spelt bran or wheat bran. Examples of pseudocereal grain bran include buckwheat bran or quinoa bran. Mixes of
10 cereal grain brans, of pseudocereal grain brans, or of cereal and pseudocereal grain brans may be envisaged. Preferably, the bran is a cereal grain bran selected from the group comprising, or consisting of, barley bran, corn bran, millet bran, oat bran, rice bran, rye bran, sorghum bran, spelt bran or wheat bran. More preferably, the bran is a cereal grain bran selected from the group comprising, or consisting of, wheat bran, corn bran, barley bran or spelt bran. In
15 another preferred embodiment, the bran is a cereal grain bran selected from the group comprising, or consisting of, buckwheat bran or quinoa bran. Most preferably, the bran is corn bran, wheat bran or buckwheat bran. The choice of a specific crop grain bran may depend on the flavour notes that may be obtained from a given crop grain bran. Examples of flavour notes obtained from various brans are given in the examples below.

In an embodiment, the bran slurry comprises from 1% to 40% by weight of bran, preferably from 1% to 35% by weight of bran. The bran slurry may comprise from 5% to 40% by weight of bran, preferably from 5% to 35% by weight of bran. Preferably, the bran slurry comprises from 1% to 30% by weight of bran, more preferably from 5% to 30% by weight, more preferably from 8% to 30% by weight, even more preferably from 8% to 27% by weight.
20 Most preferably, the bran slurry comprises 10% to 30% by weight of bran.

In an embodiment, the bran slurry comprises a water content of at least 20%, 25% 30%, 40%, 50%, 60%, 70%, 80%, or 90% by weight, based upon the weight of the bran slurry.

In an embodiment, the bran slurry comprises a water content of from 20% to 99% by weight, preferably 25% to 95% by weight, preferably of from 30% to 95% by weight, more
30 preferably of from 40% to 90% by weight, even more preferably of from 50% to 80% by weight, based upon the weight of the bran slurry.

In an embodiment, the bran slurry comprises a water content of from 80% to 99% by weight, preferably of from 85% to 95% by weight, more preferably of from 88% to 92% by weight, even more preferably of from 89% to 91% by weight, based upon the weight of the bran slurry.

5 Hence, in a preferred embodiment, the bran slurry comprises at least 50% by weight of water and from 10% to 20% by weight of bran. In another preferred embodiment, the bran slurry comprises from 20% to 50% by weight of water and from 30% to 40% by weight of bran, based upon the weight of the bran slurry. In an embodiment, the bran slurry consists essentially of bran and water.

10 In another embodiment, the bran slurry comprises at least 50% by weight of water and from 10% to 20% by weight of bran and additional ingredients such as fats, protein sources, or carbohydrates. In another preferred embodiment, the bran slurry comprises from 20% to 50% by weight of water and from 30% to 40% by weight of bran, based upon the weight of the bran slurry, with additional ingredients such as fats, protein sources, or carbohydrates.

15 The bran slurry may be prepared by mixing bran and water, and the optional additional ingredients mentioned above. Preferably, the bran may be milled, ground or micronized. Preferably, the bran is ground bran. The particle size of milled or ground bran is about 500 μm . For instance, the particle size of milled or ground bran ranges from 50 μm to 800 μm . The particle size of micronized bran is below 10 μm , such as below 1 μm . Ground bran may be
20 used directly in the preparation of the bran slurry. Alternatively, native bran may be used in the preparation of a bran slurry. In such a case, the bran slurry may undergo a milling step in order to mill or grind the native bran. A milling step may also be used when the bran starting material is ground bran. This may be useful to further reduce the particle size of the ground bran.

25 After the provision of a bran slurry, the bran slurry is subjected to a heat treatment. In an embodiment, the heat treatment is performed at a temperature of from 155°C to 200°C for a holding time of 5 to 180 min, preferably of from 155°C to 200°C for 5 to 30 min, more preferably of from 155°C to 195°C for 5 to 20 min, or of from 175°C to 200°C for 5 to 15 min, even more preferably of from 160°C to 190°C for 5 to 15 min, or of 160°C to 175°C for 5 to 15
30 min, or of from 180°C to 200°C for 5 to 10 min. Most preferably, the heat treatment is performed at a temperature of 180°C for a holding time of 10 min, or of 160°C for 5 to 10 min, or of 200°C for 5 min.

The inventors have found that a heat treatment of a bran slurry as defined above, at 155°C to 200°C for a holding time of 5 to 180 min, provides a flavour ingredient which is also suitable to reduce the development of rancid off-notes in food products susceptible to rancidification. This is shown in the examples.

5 Alternatively, the heat treatment may be performed by direct steam injection, by treatment in a tubular heat exchanger, or by treatment in a scrape surface heat exchanger.

 In an embodiment, the heat treatment is a microwave treatment, preferably performed at a temperature of 180°C for 10 min. Optionally, the microwave treatment also involves a 15 to 25 minutes heating period, to reach 180°C to 200°C, starting from a
10 temperature of 15°C to 30°C. For instance, the microwave treatment may be performed at 1,600 watts at 100% power of the microwave reactor, preferably at a power corresponding to 100% power of a MARS (Microwave Assisted Reaction Vessel, CEM) microwave laboratory reactor. Optionally, the microwave treatment produces a pressure inside a vessel containing the bran slurry of about 7.5 bar. It is believed that apart from the heating effect, microwave
15 radiation has also a physical impact on bran components that facilitates its hydrolysis.

 In an alternative embodiment, the heat treatment is performed by autoclaving preferably performed at a temperature of 160°C for 5 to 10 min. Optionally, the autoclaving treatment involves a heating period of 15 to 150 min to reach 160°C, starting from a temperature of 15°C to 30°C. The heating period depends, i.e., on the type of equipment, on
20 the batch size and on the total solids of the bran slurry.

 In an embodiment, the heat treatment is performed in a closed vessel. This allows for building up pressure and for heating to a temperature beyond the boiling point of water. When the heat treatment is performed batch wise, the bran slurry may be placed in a closed treatment vessel. When the heat treatment is performed continuously, the continuous heat-
25 treatment device comprises pressure control devices to ensure that the heat treatment is performed under pressure.

 The heat-treated bran slurry, preferably prepared by microwave treatment or by autoclaving, may be subjected to further processing before being used in the preparation of flavoured food products. The heat-treated bran slurry may be filtered to obtain a soluble
30 fraction of heat-treated bran slurry or liquor (filtrate) on the one hand, and residual solids on the other hand. Preferably, the heat-treated bran slurry is cooled down to ambient temperature prior to being filtered. The filtrate and the residual solids retain specific flavour

notes. The filtrate comprises mainly soluble components. The residual solids comprise mainly non-soluble components.

The heat-treated bran slurry or the filtrate may be concentrated. The filtrate and the residual solids may both be dried. Alternatively, the heat-treated bran slurry itself may be
5 dried. Drying may be performed, for instance, by vacuum drying, roller-drying, or spray drying. The appropriate drying method can be selected by the skilled person. The dried heat-treated bran slurry, the dried filtrate or the dried residual solids may then be comminuted, milled or ground to lower particle size.

In another embodiment, the heat-treated bran slurry, the filtrate, or the residual solids
10 may be freeze-dried. In another embodiment, the heat-treated bran slurry, the filtrate, or the residual solids may be frozen and stored at low temperatures, such as at -80°C. Optionally, the freeze-dried or the frozen heat-treated bran slurry, filtrate or residual solids may be milled or ground, to reduce particle size.

The heat-treated bran slurry, the filtrate and the residual solids, dried or not dried,
15 may be used as a flavour composition, for flavouring foods or beverages. The heat-treated bran slurry, the filtrate and the residual solids, exhibit flavour notes that were not present in the bran slurry prior to the heat treatment.

Another aspect of the invention is a method of manufacturing a flavoured food product. The method comprises a step of providing a flavour composition as described above,
20 i.e. obtainable or obtained by the method of preparing a flavour composition according to the first aspect of the invention. Hence, the flavour composition may be a heat-treated bran slurry, a filtrate made from it, or the residual solids obtained after filtration of the heat-treated bran slurry.

An aspect of the invention also includes the use of a heat-treated bran slurry as a
25 flavour ingredient in a food product. A filtrate obtained by filtering said heat-treated bran slurry, or the residual solids obtained by filtering said heat-treated bran slurry, may also be used as a flavour ingredient in a food product.

Examples of foods include, without being limited to, culinary aids, cereal products, bakery products, dairy products, dairy-like products, snacks, confectionery products, or
30 sauces. Examples of cereal products include, without being limited to, infant cereals, breakfast cereals, confectionery cereals, porridge preparations, wafers, doughs, batters for ready-to-cook cakes, ice-cream cones, biscuits, cakes, or breads. Examples of bakery products include,

without being limited to, bread, doughs, viennoiserie, and the like. Examples of confectionery products include, without being limited to, chocolate, chocolate bars, or wafer bars. Examples of dairy products include, without being limited to, chilled desserts, fermented milk products such as yoghurt, non-fermented milk products, dessert mousses, milk powders, concentrated
5 milks, evaporated milks, or creams. Examples of beverages include, without being limited to, powdered beverages, such as soluble powdered beverages, soluble coffee beverages, soluble cocoa beverages, soluble malted beverages, or ready-to-drink beverages, such as cereal shakes, milk shakes, coffee shakes, cocoa beverages, dairy beverages, plant-based beverages, or sport drinks. Examples of feed products include, without being limited to, dry feed products
10 and wet feed products, which may be based on vegetables, cereals, or animal proteins. Preferably, the food product is a wafer, a roller-dried cereal product or an extruded cereal product.

In an embodiment, the food product comprises at least 25% dry weight of plant-based material, such as materials selected from cereals, pseudo-cereals, legumes and pulses. The
15 food product may also comprise mixes of such plant-based materials.

A first step in the preparation of a flavoured food product comprises blending the flavour composition and a food matrix. Examples of food matrices include dry mixes, doughs, solutions or dispersions in a water-based or a fat-based liquid, or emulsions. Usually, food matrices comprise the standard ingredients useful for preparing a food product. For instance,
20 the food matrices may be a dairy composition, a cereal composition, a vegetable composition, a protein composition, such as meat- or fish-based compositions, or a fat-based composition. The food matrix may be dry, fat or wet.

When the dried flavour composition is blended with a dry food matrix, a flavoured dry mix may be obtained directly. For instance, a flavoured ready-to-use cereal product may be
25 obtained by mixing ready-to-use cereals, such a roller-dried cereals, with the dried flavour composition.

Optionally, the blend of the flavour composition with the food matrix is subjected to further food processing treatment(s), such as, without being limited to, extrusion-cooking, drying, roller-drying, spray-drying, baking, retorting, or toasting. Preferably, the food
30 processing treatment is roller-drying, extrusion cooking or baking. These are well-known food processing technologies.

For instance, the flavour composition is blended with flour. The flour may be whole grain flour, refined flour, pseudo-cereal flour, or flour from other vegetables such as legumes (grains of Fabaceae). The ratio of (dry) heat-treated bran to (whole grain) flour may range from 1:5 (w/w) to 1:3 (w/w). Preferably, the ratio of (dry) heat-treated bran to (whole grain) flour is 1:4 (w/w). The blend may then be mixed with water, or other liquids, and subjected to standard processing of doughs.

In an embodiment, the food processing treatment is extrusion-cooking. Preferably, the extrusion-cooking is performed at a temperature of from 100°C to 150°C, preferably of from 120°C to 140°C, more preferably of from 125°C to 135°C. The extrusion cooking may be performed at 22% humidity, 130°C and 400 rpm, preferably at rpm corresponding to 400 rpm of a laboratory extruder Eurolab 16 (Thermo Fischer).

In another embodiment, the food processing treatment is wafer-baking. Preferably, wafer-baking is performed at a temperature of from 120°C to 170°C, more preferably at a temperature of 160°C for 110 s.

In another embodiment, the food processing treatment is wet-mixing. The heat-treated bran slurry, or preferably the concentrated filtrate, or the dried heat-treated bran slurry, or the dried filtrate, or the dried residual solids, may be mixed in a standard process for preparing a ready-to-drink beverage.

In another embodiment, the food processing treatment is dry-mixing. The dried heat-treated bran slurry, or the dried filtrate, or the dried residual solids, may be mixed in a standard process for preparing a powdered soluble beverage, or an infant cereal composition.

Another aspect of the invention is a flavour composition comprising heat-treated bran obtainable or obtained by a method according to the first aspect. As will be explained below, the flavour composition comprising heat-treated bran has properties delaying the development of rancid off-notes in a food product susceptible to rancidification. Therefore, the flavour composition may be used to increase the shelf-life of a food product. The flavour composition comprising heat-treated bran may be considered as a natural food preservative.

In an embodiment, the flavour composition comprising heat-treated bran exhibits a flavour note, preferably a distinct flavour note, selected from the group consisting of caramel, sweet, vanilla-like, toasty, biscuity, smoky, meaty, savoury, and spicy. Preferably, the flavour-

notes are detectable by sniffing or tasting, more preferably are identified by the aid of a trained assessor.

In an embodiment, the flavour composition comprising heat-treated bran exhibits caramel, sweet, vanilla-like, toasty, biscuity, smoky, and spicy flavour notes, preferably distinct flavour notes. In another embodiment, the flavour composition comprising heat-treated bran exhibits meaty and savoury flavour notes, preferably distinct flavour notes. The flavour notes depend on the bran origin. For instance, heat-treated corn bran may exhibit caramel, sweet, vanilla-like, clove-like flavour notes. Also for instance, heat-treated wheat bran may exhibit biscuity, vanilla-like, toasty flavour notes. Also for instance, heat-treated buckwheat bran may exhibit meaty and savoury flavour notes.

In an embodiment, the flavour composition comprising heat-treated bran exhibits an enhanced flavour note selected from the group consisting of caramel, sweet, vanilla-like, toasty, biscuity, smoky, and spicy, as compared to a composition comprising non-heat-treated bran. In another embodiment, the flavour composition comprising heat-treated bran exhibits an enhanced flavour note selected from the group consisting of meaty and savoury flavour notes, as compared to a composition comprising non-heat-treated bran.

In an embodiment, the flavour composition comprising heat-treated bran exhibits enhanced flavour notes of caramel, sweet, vanilla-like, toasty, biscuity, smoky, and spicy, as compared to a composition comprising non-heat-treated bran. In another embodiment, the flavour composition comprising heat-treated bran exhibits enhanced flavour notes of meaty and savoury flavour notes, as compared to a composition comprising non-heat-treated bran.

In an embodiment, the flavour composition comprising heat-treated bran comprises a flavour compound selected from the group consisting of 2,3-butanedione, 2-acetylthiazole, guaiacol, 4-vinylguaiacol, vanillin, furfural, 4-hydroxy-2,5-dimethyl-3(2H)-furanone, 2-acetyl-1-pyrroline, 2,3,5-trimethylpyrazine, 2-furylmethanethiol and 2-methyl-3-furanthiol.

In an embodiment, the flavour composition comprising heat-treated bran comprises the flavour compounds 2,3-butanedione, 2-acetylthiazole, guaiacol, 4-vinylguaiacol, vanillin, furfural, 4-hydroxy-2,5-dimethyl-3(2H)-furanone, 2-acetyl-1-pyrroline, 2,3,5-trimethylpyrazine, 2-furylmethanethiol and 2-methyl-3-furanthiol.

The flavour compounds are detectable and/or quantifiable using common laboratory methods, e.g. solid phase micro extraction, gas chromatography, tandem mass spectrometry, HS-SPME-GC/MS/MS, stable isotope dilution assay (SIDA).

In an embodiment, the flavour composition comprising heat-treated bran comprises increased amounts of a flavour compound selected from the group consisting of 2,3-butanedione, 2-acetylthiazole, guaiacol, 4-vinylguaiacol, vanillin, furfural, 4-hydroxy-2,5-dimethyl-3(2H)-furanone, 2-acetyl-1-pyrroline, 2,3,5-trimethylpyrazine, 2-furylmethanethiol and 2-methyl-3-furanthiol, as compared to a composition comprising non-heat-treated or (native) bran.

In an embodiment, the flavour composition comprising heat-treated bran comprises an increased amount of a flavour compound selected from 4-hydroxy-2,5-dimethyl-3(2H)-furanone and/or 2-acetyl-1-pyrroline, as compared to a composition comprising non-heat-treated bran, when heat treatment is performed by autoclaving.

In an embodiment, the flavour composition comprising heat-treated bran comprises increased amounts of vanillin as compared to a composition comprising non-heat-treated bran. Preferably, the amounts of vanillin are increased by at least 5-fold, 10-fold, 15-fold, or 20-fold as compared to a composition comprising non-thermally treated bran or native bran.

After storage at 40°C for an extended period of time, e.g. 30 to 40 weeks, preferably after storage for 33 weeks, the flavour composition comprising heat-treated bran does not comprise increased amounts of hexanal. Preferably, the amounts of hexanal are lower by at least 20-fold, 30-fold, or 40-fold as compared to a composition comprising non-heat-treated or native bran. Most preferably, the amounts of hexanal are lower by about 40-fold, e.g. 36-fold as compared to a composition comprising non-heat-treated or native bran.

The flavour composition comprising heat-treated bran has improved sensory characteristics. In particular, the flavour composition comprising heat-treated bran has properties delaying the development of rancid off-notes in a food product susceptible to rancidification. For instance, after storage, e.g. at 40°C for an extended period of time such as 30 to 40 weeks, an extrudate containing native corn bran is characterized by a strong rancid aroma, which is not present in an extrudate comprising heat-treated bran according to the present invention.

Therefore, the flavour composition obtainable by, or obtained by, a method according to the first aspect of the invention, may be used as an ingredient for delaying the development of rancid off-notes in a food product susceptible to rancidification. This may improve the shelf-life of a food composition comprising such a flavour composition.

The invention will be illustrated further by means of the following examples taking into account the accompanying figures in which:

Figure 1 shows the relative concentration of odorants (%) in corn bran after microwave-assisted heat-treatment in MARS reactor (original corn bran set at 100%).

5 Figure 2 shows the relative concentration of odorants (%) in extrudate containing thermally-treated corn bran prepared by microwave-assisted heat-treatment in MARS reactor; concentration of odorants in extrudate containing original corn bran set as 100%.

10 Figure 3 shows the relative concentration of odorants (%) in Wafer C prepared with the soluble fraction of heat-treated corn bran; concentration of odorants in Wafer B prepared with extract of native corn bran set as 100%.

Figure 4 shows the relative concentration of odorants (%) in heat treated wheat bran prepared in a laboratory autoclave as compared to native wheat bran set as 100%.

15 Figure 5 shows the relative concentration of odorants (%) in Wafer A prepared with heat treated wheat bran expressed to the concentration of odorants in Wafer B prepared with native (non-treated) wheat bran set as 100%.

20 Figure 6 shows the evolution of pentane concentration along an accelerated shelf-life test, in a reference product (A), a product containing non-heat-treated bran (B) and a product containing heat-treated bran (C). The product is a roller-dried milky porridge composition. More details are found in Example 11.

The following examples are for illustrative purposes only and are not intended to limit the scope of the present invention.

EXAMPLES

25 Example 1: Analytical methodology

The following analytical methods were applied in the analysis of the samples described further in the examples.

Quantitative analysis of aroma compounds

30 The content of aroma compounds (Table 2) was determined using Head Space Solid Phase Micro Extraction in combination with Gas Chromatography and tandem Mass Spectrometry (HS-SPME-GC/MS/MS). Quantification was accomplished by Stable Isotope

Dilution Assay (SIDA) for all odorants except 2-furylmethanethiol and 2-methyl-3-furanthiol that were determined using [$^2\text{H}_4$]-furfural as an internal standard. Concentrations of both thiols were calculated using the response factors that were determined with pure reference compounds.

5 The sample ($1\text{ g} \pm 0.002\text{ g}$) was weighted into a 20 mL headspace vial. Ultrapure water (10 mL) and methanol solution of internal standards (20 μL) were added together with a magnetic stir bar. For the determination of 2-furylmethanethiol and 2-methyl-3-furanthiol, 500 mg cysteine was also added to the mixture in order to release both thiols from the disulphide bounding to the matrix. The vial was closed with a screw cap and the mixture was
10 homogenized by means of a vortex agitator for 5 s and then stirred for 15 min using a magnetic stirrer. The mixture was then centrifuged at 4000 rpm for 3 min and an aliquot of supernatant (2 mL) was transferred into a new 20 mL headspace vial and analysed by HS-SPME-GC/MS/MS. Each sample was prepared in duplicates by two independent work-ups.

For HS-SPME, the incubation (5 min) and extraction (30 min) were performed at 70°C .
15 DVB-CAR-PDMS fibre of 2 cm (Supelco) was used for the extraction under agitator speed of 500 rpm. The fibre was injected into a GC-MS/MS instrument and aroma compounds were desorbed in split mode (ratio 5:1) at 250°C for 5 min.

For GC/MS/MS, an Agilent 7890A gas chromatograph and Agilent 7000 triple quadrupole mass spectrometer with chemical ionization source (CI) were used. Methane was
20 used as a reactant gas. Gas chromatographic separations were achieved on a DB-624-UI column 60 m x 0.25 mm i.d., film thickness 1.4 μm (J&W Scientific). The temperature program of the oven started at 50°C ; the temperature raised by $5^\circ\text{C}/\text{min}$ to 200°C and then by $30^\circ\text{C}/\text{min}$ to 250°C and maintained constant for 10 min. Helium was used as a carrier gas with a constant flow of 1.0 mL/min.

25 The analytes were identified by comparing their retention times and fragmentation patterns with corresponding standards. The concentrations were calculated from the abundances (peak areas) of the ions selected for the analytes and the internal standards and from the amounts of added internal standards. The quantities of the internal standards were adjusted to obtain a peak area ratio of analyte/standard between 0.2 and 5. The ions
30 (transitions) used for the quantification by stable isotope dilution assay are listed together with applied collision energies in [Table 2](#).

Table 2: Selected ions used for the quantification of aroma compounds by means of stable isotope dilution assays

Compound [trivial name (IUPAC name)]	Precursor ion (m/z)	Product ion (m/z)	Collision energy (V)
2,3-butanedione (butane-2,3-dione)	87	59	15
[¹³ C ₄]-2,3-butanedione	91	62	15
2-acetyl-1-thiazole	128	86	35
[¹³ C ₂]-2-acetyl-1-thiazole	130	86	35
guaiacol (2-methoxyphenol)	125	110	15
[² H ₃]-guaiacol	128	110	15
4-vinylguaiacol (4-ethenyl-2-methoxyphenol)	151	91	20
[² H ₃]-4-vinylguaiacol	154	91	20
vanillin (4-hydroxy-3-methoxybenzaldehyde)	153	137	25
[² H ₃]-vanillin	156	137	25
hexanal	83	55	10
[² H ₃]-hexanal	86	57	10
furfural (furan-2-carbaldehyde)	97	69	14
[² H ₄]-furfural	101	73	14
2,3,5-trimethylpyrazine	123	42	35
[² H ₃]-2,3,5-trimethylpyrazine	126	42	35
4-hydroxy-2,5-dimethyl-3(2H)-furanone (HDMF)	129	43	20
[¹³ C ₂]-4-hydroxy-2,5-dimethyl-3(2H)-furanone	131	45	20
2-acetyl-1-pyrroline	112	70	20
[¹³ C ₂]-2-acetyl-1-pyrroline	14	70	20
furfuryl thiol (2-furylmethanethiol)	114	81	5
2-methyl-3-furanthiol	114	71	15

Analysis of pentane

- 5 Pentane is an autoxidation product of linoleic acid (C-18:2) that is formed by homolytic β -scission of corresponding 13-hydroperoxide. The scission on the other side of 13-hydroperoxide provides hexanal that is also often used a marker of lipid oxidation. While hexanal is a reactive aldehyde and may undergo different reactions with matrix components,

pentane is a stable hydrocarbon that is cumulated over the storage and thus indicates well the extent of lipid oxidation.

Pentane was determined in the headspace of sealed aluminium cans using an internally design prototype (Nestlé PTC Orbe) coupling a sampler and gas chromatography analysis using flamme ionisation as detection system (GC-FID, Perkin Elmer Clarus 500). Quantification was done using pentane gas as standard.

Example 2: Heat-treatment of corn bran in laboratory microwave reactor (MARS)

Approximately 2.5 g of corn bran were weighted in Teflon vessels of 50 mL capacity. 25 mL of water were added and the preparation was thoroughly mixed before set up of cells in the microwave laboratory reactor (MARS). Typically, for one batch of thermally-treated corn bran, 12 cells were prepared and one to two batches were run per day. MARS parameters were set-up to 180°C, 10 min and 1600 watts at 100% power. A 20 min-ramp was used to reach 180°C. Pressure inside the Teflon vessels reached about 7.5 bar. All extracts obtained per batch (i.e. 12 cells) were pooled and stored at -80°C before freeze-drying. A total of 9 batches of freeze-dried heat-treated corn bran were obtained and freeze dried powders were finally combined and milled on a sieve to reduce particle size. Approximately 250 g of ingredient were thus obtained.

A significant difference in aroma was detected between thermally-treated and native corn bran. Aroma of thermally-treated corn bran exhibited distinct caramel, sweet, vanilla-like, smoky, and spicy notes, as compared to native corn bran that was perceived rather bland with typical hey/straw-like and raw cereal notes.

Analysis of aroma compounds revealed that the content of the following odorants was significantly higher in thermally treated corn bran as compared to original corn bran (concentration factors given in brackets): 2,3-butanedione (buttery, 24), 2-acetylthiazole (roasty/popcorn, 30), guaiacol (smoky, sweet, 73), 4-vinylguaiacol (clove-like, 52), vanillin (vanilla-like, 19), furfural (caramel/bready, 404), 2,3,5-trimethylpyrazine (earthy/nutty, 45). Figure 1 shows the relative concentration of odorants (%) in thermally-treated corn bran as compared to original corn bran set arbitrary as 100%.

Heat treatment in microwave reactor thus resulted in a corn bran ingredient with improved aroma profile.

Example 3: Use of thermally-treated corn bran in extrusion

Freeze dried thermally-treated corn bran prepared as described in Example 2 was mixed with whole grain corn flour in ratio 1:5 (w/w) and was extruded using laboratory extruder Eurolab 16 (Thermo Fischer) at 22% humidity, 130°C and 400 rpm. A reference extrudate was prepared analogously employing native (non- treated) corn bran instead of thermally treated corn bran.

The extrudates were evaluated by sniffing by 10 assessors. Aroma of extrudate containing thermally treated corn bran was significantly improved as compared to aroma of extrudate containing original corn bran. Aroma of extrudate containing thermally treated corn bran exhibited caramel, sweet, vanilla-like, smoky, and spicy notes, while aroma of extrudate containing native corn bran was rather bland with typical raw cereal notes.

Analysis of aroma compounds revealed that the content of the following odorants was significantly higher in extrudate with heat-treated corn bran as compared to extrudate with original corn bran (concentration factors given in brackets): 2,3-butanedione (buttery, 12), 2-acetylthiazole (roasty/popcorn, 3), guaiacol (smoky, sweet 26), 4-vinylguaiacol (clove-like, 18), vanillin (vanilla-like, 9), furfural (caramel/bready, 71), 2,3,5-trimethylpyrazine (earthy/nutty, 16). Figure 2 shows relative concentration of odorants (%) in extrudate containing thermally treated corn bran as compared to extrudate containing original corn bran set arbitrary as 100%.

Example 4: Shelf-life test with extrudate containing thermally treated corn bran

Cereal extrudates prepared as described in Example 3 were subjected to accelerated shelf-life study. 1 g of milled extrudate containing either native corn bran (reference) or thermally treated corn bran were placed into 20 mL headspace vial. The vial was closed with a screw cap and stored in an oven at 40°C for 33 weeks.

The samples after the storage were evaluated by sniffing by 10 assessors. Strong rancid aroma was detected in the extrudate containing native corn bran (reference) while no rancid notes were perceived in the extrudate containing thermally treated corn bran.

The results from the sniffing were corroborated by quantitative analysis of hexanal (marker of lipid oxidation). Hexanal content in extrudate prepared with native corn bran (86.31 mg/kg) was increased 36-fold as compared to extrudate with thermally treated corn bran (2.43 mg/kg).

Example 5: Heat-treatment of corn bran in laboratory autoclave (LABMAX) and preparation of corn bran extract

25 g of corn bran was dispersed in 250 mL of boiling water and transferred to double-jacket heated laboratory autoclave (LabMax, Mettler Toledo). The mixture was heated up to 160°C during 140 min then held for 10 min and cooled down to ambient temperature (30 min). Non-soluble residues were filtered out using a paper filter and a Büchner funnel to obtain a liquor (soluble fraction).

A corn bran extract (reference) was prepared by stirring of 25 g native corn bran in 250 mL water at ambient temperature for one hour using a magnetic stirrer. The extract was filtered using a paper filter and a Büchner funnel to obtain a soluble fraction.

Aroma of soluble fraction of treated corn bran (liquor) and corn bran extract was evaluated by sniffing (10 assessors). Aroma of corn bran liquor was classified far stronger and much more pleasant as compared to aroma of corn bran extract. Aroma of corn bran liquor exhibited caramel, sweet, vanilla-like, smoky, and spicy notes, while aroma of corn bran extract was rather bland with some greenish and raw cereal notes.

Example 6: Use of soluble fraction of thermally treated corn bran and corn bran extract in wafer baking

Soluble fraction of thermally treated corn bran (liquor) and soluble fraction of corn bran extract prepared as described in Example 5 were applied in wafers in order to evaluate their potential to modulate flavour. Standard wafer without any addition of corn bran was also prepared. Batters were prepared with the following formulation (Table 3):

Table 3

Ingredient	Wafer A (g/batter)	Wafer B (g/batter)	Wafer C (g/batter)
Wheat flour	48	48	48
Corn bran extract (soluble fraction)		25	
Soluble fraction of thermally treated corn bran			25
Water	50	25	25
Fat	1.8	1.8	1.8
Sodium bicarbonate	0.12	0.12	0.12

Salt	0.12	0.12	0.12
Total	100.0	100.0	100.0

Wafers (9-11 g each) were prepared by baking at 160°C for 110 s using laboratory equipment for production of wafer sheets (Hebenstreit).

Sensory evaluation (tasting) revealed no significant flavour differences between reference wafer (A) and wafer containing corn bran extract (B); flavour of both wafers was described as bland with raw cereal notes. On the other hand, flavour of wafer containing liquor from treated corn bran (C) was described as significantly improved, exhibiting caramel, sweet, burnt sugar, smoky, clove, and spicy attributes. Wafer containing corn bran liquor was also found crispier as compared to reference wafer or wafer with corn bran extract.

Wafers were grinded using a coffee grinder (Tristar) and concentrations of selected odorants were determined. Analysis of aroma compounds revealed that the content of the following odorants was significantly higher in wafer with heat-treated corn bran (C) than in wafer with corn bran extract (B) (concentration ratio in brackets): 2,3-butanedione (buttery, 3), guaiacol (smoky, sweet 58), 4-vinylguaiacol (clove-like, 26), vanillin (vanilla-like, 190), furfural (caramel/bready, 182). Figure 3 shows the relative concentration of odorants (%) in Wafer C expressed to the concentration of odorants in Wafer B set arbitrary as 100 %.

Example 7: Heat-treatment of wheat bran in laboratory autoclave

400 g of wheat bran was mixed with 1,6 L of boiling water and transferred to a laboratory autoclave (Versoclave type 3E/ 3L, Büchiglasuster) with jacket pre-heated at 80°C. The mixture was heated under stirring up to 160°C during 30 min, held for 5 min and then cooled down to ambient temperature in 1 hour. The mixture was then transferred into two metal trays, frozen over night at -80°C and freeze-dried. Obtained cake was pulverized into powder using a kitchen blender with blades (Thermomix).

Aroma of the powder was evaluated by sniffing (10 assessors) and compared with aroma of the native wheat bran. Aroma of the treated wheat bran was classified far stronger and much more pleasant as compared to aroma of the native bran. Aroma of the treated bran exhibited caramel, biscuity, and spicy notes, while aroma of the native bran was rather bland with some greenish and raw cereal, straw-like notes.

Analysis revealed that the content of the following odorants was significantly higher in the treated bran as compared to the native wheat bran (increase factors given in brackets): 2,3-butanedione (buttery, 49), 2-acetyl-1-thiazole (roasty/popcorn, 820), guaiacol (smoky/sweet, 39), 4-vinylguaiacol (clove-like, 17), furfural (caramel/bready, 67), 4-hydroxy-
5 2,5-dimethyl-3(2H)-furanone (HDMF, caramel, 435), 2-acetyl-1-pyrroline (popcorn, 177). Figure 4 shows the relative concentration of odorants (%) in treated wheat bran as compared to native wheat bran set arbitrary as 100%.

Example 8: Treatment of buckwheat bran in laboratory autoclave

10 Buckwheat bran was treated in the laboratory autoclave and freeze-dried analogously as the wheat bran described in Example 7. The aroma of the obtained powder was evaluated by sniffing (10 assessors) and compared with that of the native buckwheat bran. Aroma of the treated bran was classified far stronger as compared to aroma of the native bran. Aroma of the treated bran exhibited distinct meaty, savoury, and sulphury notes, while aroma of the
15 native bran was rather bland with some greenish and raw cereal, straw-like notes.

The analysis revealed that the content of following odorants was significantly higher in the treated bran as compared to the native bran (increase factors given in brackets): 2,3- butanedione (buttery, 35), 2-acetyl-1-thiazole (roasty/popcorn, 206), guaiacol (smoky/sweet, 26), 4-vinylguaiacol (clove-like, 29), furfural (caramel/bready, 66), 4 hydroxy-
20 2,5-dimethyl-3(2H)-furanone (HDMF, caramel, 6380). In addition, high amounts of sulphury odorants 2-furylmethanethiol (2114 ppb, coffee-like) and 2-methyl-3-furanthiol (698 ppb, meaty) were detected in the treated bran, while both odorants were below the limit of detection in the native bran. Both thiols are especially potent odorants that have one of the lowest aroma thresholds of any food odorants. Their elevated amounts certainly determines
25 the aroma signature of the treated bran.

Example 9: Use of thermally treated wheat bran for preparation of wafers

The treated wheat bran prepared as described in Example 7 was applied in wafer preparation (Wafer D) in order to evaluate its potential to modulate flavour. A reference wafer
30 (Wafer E) with addition of the native (non-treated) wheat bran was also prepared as well as a control wafer (Wafer F) without bran and based on refined wheat four. Batters were prepared with the following formulation:

Table 5

Ingredient	Wafer D (g/batter)	Wafer E (g/batter)	Wafer F (g/batter)
Refined wheat flour	61.05	61.05	<u>71.82</u>
Thermally-treated wheat bran	10.77		
Wheat bran		10.77	
Water	75.00	75.00	<u>75.00</u>
Fat	3.00	3.00	<u>3.00</u>
Sodium bicarbonate	0.18	0.18	<u>0.18</u>
Total	150.0	150.0	<u>150.0</u>

The ratio between the treated wheat bran and the refined wheat flour was intentionally set to 15% and 85% in order to reflect natural proportion of bran in whole grain flour. Wafers (9-11 g each) were prepared by baking at 160°C for 110 s using laboratory equipment for production of wafer sheets (Hebenstreit).

Sensory evaluation (tasting by 10 assessors) revealed improved flavour of Wafer D prepared with the treated wheat bran. The flavour of Wafer D was classified more intense and more pleasant with caramel, biscuity, and spicy notes as compared to the flavour of Wafer E prepared with the native (non-treated) bran that was rather bland with typical note of whole grain cereals.

The wafers were ground using a coffee grinder (Tristar) and concentrations of selected odorants were determined. The analysis revealed that the content of the following odorants was significantly higher in Wafer D than in Wafer E (increase factors given in brackets): 2,3-butanedione (buttery, 4), guaiacol (smoky, sweet 5), 4-vinylguaiacol (clove-like, 25), vanillin (vanilla-like, 10), furfural (caramel/bready, 7), 2-acetyl-1-thiazole (popcorn, 9), 2-acetyl-1-pyrroline (popcorn, 3), 4-hydroxy-2,5-dimethyl-3(2H)-furanone (HDMF, caramel, 26). Figure 5 shows the relative concentration of odorants (%) in Wafer D expressed to the concentration of odorants in Wafer E set arbitrary as 100 %.

The ground wafers were subjected to an accelerated storage test. 5 g of ground wafer in 50 mL closed Pyrex bottle was stored in an oven at 40°C for 6 months. After storage, aroma was evaluated by 10 assessors. The aroma of Wafer E prepared with the original bran exhibited a distinct rancid off-note, while the aroma of Wafer D prepared with the treated

bran remained pleasant with caramel, biscuity, and spicy notes. The control Wafer F, only made of refined flour also exhibited a distinct rancid off-note. This shows that the addition of bran was not the only driver to rancidity generation. To corroborate the different extent of rancidification, hexanal (marker of lipid oxidation) was determined in all samples. The amount of hexanal in Wafer D prepared with the treated bran (225 ppb) was 4 folds lower than in Wafer E prepared with the original bran (955 ppb) and up to 30 folds lower than in Wafer F with refined flour only (1763 ppb).

Use of the treated wheat bran in the wafer preparation thus resulted in a wafer with improved flavour and delayed development of rancid off-flavours during the storage.

10

Example 10: Treatment of wheat bran in a tubular heat exchanger

A treated wheat bran slurry was produced at pilot scale by mixing native wheat bran and water at 80 °C using a ring layer mixer (AVA drynamix) and applying a ratio of 26 parts of bran for 64 parts of water. The bran slurry was then treated with water vapour to reach 160 °C and maintained at this temperature during 18 minutes (1090 seconds) by flowing in series of heat-jacketed tubes (Tubular Heat Exchanger, Nestlé PTC Orbe). Steam was then released by flashing and the slurry collected once cooled below 60°C. The product was kept at 4°C for 24 hours before incorporation in a full cereal-based recipe.

Example 11: Use of the bran of example 10 in a roller dried milky porridge recipe

The development of rancid off-notes in a cereal-based roller-dried milky porridge recipe was evaluated during an accelerated storage test. Wheat-based roller dried products were prepared using either 15 % of native bran (Product B) or 15 % of treated bran from Example 11 (Product C), on a dry basis, and compared with the reference recipe (Product A) not containing bran but containing di-calcium phosphate as preservative. The following formulations were applied (Table 6). The values are dry weight percent.

Table 6

Ingredient	Product A (% dry weight)	Product B (% dry weight)	Product C (% dry weight)
Refined wheat flour	50	43	43
Sugar	20	20	20
Whole milk powder	7	7	7

Soluble maize fibre	7		
Thermally-treated wheat bran			14
Wheat bran		14	
Di-Calcium phosphate	0.05		
Salt	0.06	0.06	0.06
Water	103	103	103
Total	187	187	187

The slurry was dried on a bi-cylinder roller dryer (ANDRYTZ Gouda) at 184 °C and 3 rpm and the dried film was milled using a 2 mm-sieve. The resulting powder was finally mixed with whole milk powder at a 87 :13 cereal powder : milk powder ratio.

- 5 Sensory evaluation (tasting by 10 assessors) revealed more intense cereal flavour of Product C prepared with the treated wheat bran as compared with Product B. Product C was also evaluated as more “toasty” than product B.

- Products were subjected to an accelerated storage test. 50 g of each product were packed in 1 L tin-cans, sealed and stored in a climatic chamber at 37 °C. Products were
10 analysed every month for pentane (marker of lipid oxidation) and evaluated by sniffing. Sniffing assessment involved scoring the product on a scale from 0 to 10 for rancid notes. A score at 5 or below was considered as not acceptable. Figure 6 shows generation of pentane along the 6 months of storage in sealed tins at 37 °C. Product A (reference product with preservatives) showed moderate yet measurable pentane generation during storage and
15 scored at 5 or below by some assessors as early as after 2 months of storage. Product B with native bran showed the highest pentane generation and scored at 5 or below by some assessors as early as after 3 months of storage. Product C with treated bran showed the smallest pentane generation and was never scored below 5.

- Use of the treated wheat bran in a roller dried milky porridge recipe thus resulted in
20 more intense and toasty flavour and delayed development of rancid off-flavours during the storage. Reference Product A showed development of rancidity markers (pentane and sniffing assessment), even without addition of bran to the product and despite the presence of a preservative (di calcium phosphate).

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