

Method for separating a cereal material into fractions that are respectively enriched with beta-glucan, proteins, lipids and starch

Field of the invention

5 The present invention relates to methods for separating a cereal material, in particular oats or barley, comprising de-oiling by extraction using alcohol and dry fractionation. The invention also relates to the products (fractions) obtained by these methods.

Technical background

10 Beta-glucans are natural polysaccharides present in various organisms, notably plants (such as oats and barley), fungi (such as shiitake and reishi) and certain algae and bacteria. They are molecules made up of glucose, linked together by β -glycosidic bonds.

 Beta-glucans are known for their biological and functional properties beneficial for health. They are often considered as soluble dietary fibers and have been studied for their
15 potential immunomodulatory, antioxidant, cholesterol lowering and anti-tumor benefits. Beta-glucans are therefore used as functional ingredients in dietary supplements, health foods and cosmetic products.

 Oats and barley as well as the co-products derived from these cereals, originating from malting, brewing and plant-based dairies, are known to contain high levels of beta-
20 glucan. Among the main cereals (wheat, rye, oats and barley), oats and barley are those whose grains contain the largest amounts of beta-glucans. Oat and barley grains consist of 4 main components:

- The lemmas or awns (also called "hull" in the present text): this is the fibrous shell that surrounds the whole grain. The operation consisting of removing this
25 part is called "de-awning" or "dehulling". The grain freed of these hulls is called groats.
- The bran: this is the protective envelope (outer layer) of the seed. This part is very rich in fiber and beta-glucan.
- The kernel: this is the heart of the seed without its protective envelope. The
30 operation that consists of removing the envelope of the grain is called dehulling. The ground kernel is known as flour.
- The endosperm: this is the main part of the kernel. It contains carbohydrates in the form of starch and proteins.
- The germ: this is the smallest part of the kernel. It mainly contains the fats of the
35 grain.

The oat beta-glucans are found in the endosperm and its envelope. It is therefore necessary to dehull and fractionate the whole grain to produce commercial bran enriched in beta-glucans. By using technology and certain processing conditions, it is possible to obtain a natural oat bran with a high content of dietary fiber (such as up to 44%) and of beta-glucans (up to 22%). In hull-less barley, beta-glucans are distributed more uniformly in the grain; consequently, even the refined products such as barley flour contain beta-glucans.

The products and co-products that result from the use of oats and barley, notably in malting, brewing, distillation, plant-based dairy and milling, are also rich in beta-glucans.

Diets comprising the soluble fibers of certain foods that have a low content of saturated fatty acids and cholesterol can reduce the risk of coronary heart disease. Recent studies have made it possible to determine that beta-glucan is the physiologically active component responsible for the cholesterol lowering effect observed with oat consumption. The health claim according to which regular consumption of beta-glucan reduces cholesterol levels and the risks of coronary heart disease is currently authorized by the FDA (Food and Drug Administration) of the United States and by the EFSA (the European Food Safety Authority).

Beta-glucans are linear, unbranched, non-starchy polysaccharides composed of glucose molecules linked by $\beta(1\rightarrow4)$ and $\beta(1\rightarrow3)$ bonds, and thus have physical properties of water solubility and gelation. It is thought that the ability of beta-glucans to form highly viscous solutions at relatively low concentrations in the human intestine is a key factor in the health benefits of these substances. The cholesterol lowering effect of soluble beta-glucan fibers is believed to be attributable to several mechanisms of action: increased excretion of bile acids and cholesterol, reduced insulin secretion (leading to reduced cholesterol synthesis), production of short-chain fatty acids (which could inhibit cholesterol biosynthesis), reduced fat absorption rates, and inhibition of pancreatic lipase or reduced gastric lipase activity.

It has also been shown that cereal beta-glucans reduce the glycemic response, which could help improve metabolic regulation in diabetics.

Cereals, and in particular barley and oats (comprising the products and co-products resulting from the use of these 2 cereals), contain other constituents that also have significant nutritional and functional characteristics such as proteins, starch and oil (lipids). All these compounds find applications in food as well as in pharmaceuticals or cosmetics.

Moreover, in Commission Implementing Regulation (EU) No 828/2014 of 30 July 2014, the European Union recognizes that *“most people with intolerance to gluten can include oats in their diet without adverse effect on their health. This is an issue of ongoing*

study and investigation by the scientific community". Oat proteins are therefore likely to constitute a significant alternative to wheat proteins traditionally used in food.

The use of different fractionation technologies to recover beta-glucans and proteins contained in cereal grains, notably oats or barley, as well as in the products and co-products resulting from the use of these 2 cereals, is unfortunately almost impossible due to a high fat content in the grains and the products resulting from their use. Thus, the average lipid content in oat groats varies between 4 and 8% of the dry matter weight, causing sticking to the mill walls in the case of direct grinding of the grain or groats.

In order to be able to fractionate oat or barley grain or the products and co-products resulting from their use, a preliminary de-oiling step is necessary. The historical process is extraction with hexane. For example, the article by Wu et al., "*Enriched Protein- and β -Glucan Fractions from High Protein Oats by Air Classification*", Cereal Chemistry, 1995, vol.72, no.1, p.132-134, describes a process for obtaining fractions enriched in proteins and beta-glucans from oats, in which the oats are first dehulled in a pin mill, the recovered groats are then defatted using hexane, and the delipidated groats are then ground and then subjected to air classification.

However, the use of this solvent has several major drawbacks. First, it is produced from petroleum, resulting in high greenhouse gas emissions (for example, 620 kg CO₂eq/t). In addition, its degree of toxicity is significant and, after extraction, hexane residues are always present in the cereal subjected to treatment.

More recently, hexane has been replaced by supercritical CO₂ as the extraction solvent. The main advantage of using this solvent is that it leaves no toxic residues in the powder, but this new technology remains very energy-intensive and expensive, and requires substantial investments.

For example, document WO 2008/096044 describes a process for producing beta-glucan, protein, starch and lipid concentrates from oats, comprising a step of extracting oats using a supercritical fluid, a dry grinding step of the delipidated fraction, a step of separating the obtained fraction into a fraction enriched in starch and a fraction enriched in beta-glucan, and a step of separation by sieving or by air classification of the starch-enriched fraction to obtain 3 fractions: a fraction comprising a protein concentrate, one comprising a beta-glucan concentrate and a fraction comprising a starch concentrate.

The article by Sibakov et al., "*Lipid removal enhances separation of oat grain cell wall material from starch and protein*", Journal of Cereal Science, 2011, vol.54, no.1, p.104-109, describes a process comprising grinding oat groats, extracting lipids based on supercritical CO₂, followed by grinding and then a first step of air classification of the delipidated oats. The fraction of larger particles obtained is subjected to grinding and a second step of air

classification to obtain a fraction enriched in beta-glucans. The fraction of finer particles obtained at the end of the first air-classification step is also subjected to another air-classification step to obtain a fraction enriched in proteins and a fraction enriched in starch.

Furthermore, the cereal beta-glucan extraction processes currently used may lead to certain negative effects that can alter their properties and their functionality. For example, they may alter their native structure, which can reduce their ability to form a viscous gel in the intestinal tract. This loss of structure may result in a reduction of their beneficial effects, such as cholesterol reduction and blood-glucose regulation.

There is therefore a real need to develop a process for obtaining fractions enriched in oil (or lipids), starch, protein and beta-glucan from cereal, respectively, leading to low denaturation of the components to be isolated and in particular of beta-glucans and proteins, and having reduced greenhouse gas emissions.

Summary of the invention

The invention relates first to a method for separating a cereal material, comprising the following steps:

- providing at least one cereal material comprising starch, proteins, lipids and beta-glucans;
- treating the cereal material by solid/liquid extraction with an alcohol solution comprising at least 50% by volume of alcohol, so as to obtain a solid fraction depleted in lipids and a liquid fraction enriched in lipids;
- grinding the solid fraction depleted in lipids;
- separating the particles of the ground lipid-depleted fraction according to their size, so as to obtain a fraction enriched in beta-glucans and a fraction depleted in beta-glucans;
- grinding the fraction depleted in beta-glucans; and
- treating the ground fraction depleted in beta-glucans by air classification, so as to obtain a fraction enriched in proteins and a fraction enriched in starch.

In embodiments, the cereal material is an oat and/or barley cereal material.

In embodiments, the cereal material is at least one cereal grain and/or at least one product from the malting, malt-extract manufacturing, brewing, cereal distillation, plant-based dairy and/or milling industries.

In embodiments, the alcohol solution is a mixture of alcohol and at least one solvent, the solvent preferably being chosen from the group consisting of water, 2-methyloxolane and mixtures thereof, more preferably the alcohol solution consists of a mixture of alcohol and water.

In embodiments, the alcohol solution comprises from 50 to 90% by volume of alcohol.

In embodiments, the alcohol of the alcohol solution is recycled alcohol.

In embodiments, the alcohol of the alcohol solution is ethanol.

5 In embodiments, grinding the fraction depleted in beta-glucans comprises micronizing the fraction depleted in beta-glucans.

In embodiments, separating the particles of the ground lipid-depleted fraction according to their size is carried out by sieving.

10 In embodiments, the method further comprises a step of separating the alcohol from the liquid fraction enriched in lipids, preferably by distillation, the method preferably further comprising a step of recycling the separated alcohol for the solid/liquid extraction step.

In embodiments, the method further comprises, before the step of treating the cereal material by solid/liquid extraction, a step of grinding and/or flattening the cereal material so as to obtain a ground and/or flattened fraction, the provided cereal material being preferably a cereal grain.

15 In embodiments, the cereal material is a cereal grain comprising a hull, the method further comprising a step of dehulling the grain prior to the step of grinding and/or flattening the cereal material.

20 In embodiments, the fraction enriched in beta-glucans comprises a beta-glucan amount greater than or equal to 10% by weight, preferably from 10 to 40% by weight, more preferably from 15 to 25% by weight, relative to the total dry matter weight of the fraction.

In embodiments, the starch-enriched fraction comprises a carbohydrate amount greater than or equal to 40% by weight, preferably from 40 to 90% by weight, more preferably from 50 to 85% by weight, relative to the total dry matter weight of the fraction.

25 In embodiments, the protein-enriched fraction comprises a protein amount greater than or equal to 15% by weight, preferably from 15 to 35% by weight, more preferably from 15 to 30% by weight, even more preferably from 20 to 30% by weight, relative to the total dry matter weight of the fraction.

30 The present invention makes it possible to meet the need expressed above. More particularly, it provides an improved method for separating a cereal material, making possible the fractionation of four components of interest (oil, beta-glucans, starch, proteins) that is simple, effective, more economical, that leads to low denaturation (transformation) of the fractionated components, notably beta-glucans and proteins, that is environmentally friendly, with lower water consumption and reduced greenhouse gas emissions, and generating a minimum of effluents that are easy to recycle.

35 This is achieved by using the combination of a de-oiling step by extraction using a specific solvent, namely an alcohol solution comprising at least 50% by volume of alcohol,

and a dry fractionation process, using one or more grinding steps, and an air classification step.

In particularly advantageous embodiments in which the alcohol used for the de-oiling, in particular ethanol, is recycled to be reused in the extraction step, the method according to the invention makes it possible to further reduce greenhouse gas emissions as well as solvent consumption and effluent generation.

Brief description of the figures

Figure 1 shows a flowchart of an example method according to the invention.

Detailed description

The invention is now described in more detail and in a non-limiting manner in the description that follows.

Unless indicated otherwise, all percentages are by weight.

In the present text, the quantities indicated for a given species can apply to that species according to all its definitions (as mentioned in the present text), including the narrower definitions.

In the present text, “fraction enriched in a substance X” obtained after a certain treatment means a fraction in which the ratio of molar proportions of substance X to total dry matter is greater than that of the fraction before treatment.

In the present text, the terms “lipids”, “oil” and “fats” have the same meaning and are used interchangeably.

The invention relates to a method for separating a cereal material, in particular to obtain a lipid-enriched fraction, a beta-glucan enriched fraction, a starch-enriched fraction and a protein-enriched fraction. In the context of the present invention, “cereal material” means any material based on or derived from at least one cereal, in any possible form (such as whole, ground, powdery, flattened, etc.). More particularly, it may be the cereal itself (raw), any part thereof, or any material obtained after processing, transformation or use of the cereal.

The method according to the invention involves a step of de-oiling grains by extraction using an alcohol solution comprising at least 50% by volume of alcohol, followed by fractionation of the cereal material by dry process. “Dry process” means any operation, such as grinding or air classification, carried out without using water.

Cereals belong to the Poaceae family. They produce grains, which comprise starch, proteins, lipids and soluble fibers, in particular beta-glucans. In general, cereal grains

comprise at least one kernel surrounded by an envelope. However, in the present text, the term “grain” may generally designate the total (whole) grain as well as any part of the grain (such as the grain freed of its lemmas, or the kernel, etc.), unless indicated otherwise.

Within the scope of the present invention, the starting cereal material may be or may be derived from any suitable cereal. However, in a particularly preferred manner, this cereal is chosen from the group consisting of oats, barley and mixtures thereof.

In the present invention, any suitable cereal material may be used, comprising starch, proteins, lipids and beta-glucans.

Thus, advantageously, the starting cereal material used in the invention may be a cereal grain (in particular an oat and/or barley grain). The dehulled oat grain (i.e. the grain from which the lemmas have been removed) is called groats. The dehulled barley grain is called pearled barley. In the present text, the terms “oat grain” and “barley grain” mean both the total grain and any part of the grain (such as oat groats or pearled barley), unless indicated otherwise. Oat and barley grains have the advantage of being rich in beta-glucan (they may comprise from 3 to 7% by weight for oats and from 3 to 11% by weight for barley). Preferably, a cereal grain is used as the starting raw material, in particular an oat and/or barley grain, comprising a hull and a groat.

Alternatively, or additionally, it is advantageously possible to use any product, as the starting cereal material, which results from the use or transformation of a cereal (in particular oats or barley), notably in fermentation and distillation processes for the production of germinated cereal (in particular malt), for the production of plant-based beverages or for the production of flours. More particularly, products usable in the method according to the invention are:

- the co-products of malting and the manufacture of malt extracts: during the barley germination process, rootlets (small roots) form. After drying, the grains are processed to remove the rootlets as well as the grain envelope. These 2 products are rich in proteins and beta-glucans.
- the co-products of brewing and/or cereal distillation: after the fermentation step, the “must” is filtered in order to remove all solid residues originating from the cereal grain. This substrate is rich in proteins and beta-glucans.
- the co-products of plant-based dairies: during the production of the plant-based beverage, a filtration or decantation operation is carried out to remove the solid residues originating from the cereal grain. These residues are called okara. These residues contain a high level of proteins and beta-glucans.
- milling products.

When the cereal material is a cereal grain, and more particularly an oat and/or barley grain, the method according to the invention advantageously comprises a step of removing the hull (or dehulling) from the cereal grains (in particular oats and/or barley). Indeed, the hull is composed mainly of poorly edible insoluble fibers. In addition, most contaminants of the oat or barley seed are found in the hull; its removal thus reduces the risks of contamination of the prepared fractions.

Preferably, the dehulling is mechanical dehulling, carried out by abrasion, compression, impact, shearing, for example, or by any other appropriate mechanical action. More preferably, the dehulling is carried out by abrasion.

The dehulling can be carried out using any suitable device, notably using one of the mechanical forces mentioned above, abrasion in particular. Advantageously, the dehulling is carried out by means of a de-awner. De-awners operate according to the principle of a friction polishing machine. They can be used to remove awns, separate double grains and treat the surface of cereal grains. According to one example of a dehulling process by means of a de-awner, a feed screw conveys the cereals into the working chamber where a rotor in a perforated sleeve subjects the product being treated to mechanical forces by friction. The friction intensity can be adjusted, by an adjustable counterweight, for example, or by adjustable springs at the machine outlet. The detached elements that are separated from the treated product (the grain), such as awns or impurities adhering to the surface, can exit the machine through a separate suctioned outlet.

Using a de-awner to carry out dehulling is advantageous since the de-awner cleans the grain surface at the same time as it detaches the hull from the grain by mechanical stress. In addition, this processing step improves the flow properties of the grains.

The cereal material may be subjected to a grinding and/or flattening step, and a ground and/or flattened fraction is then collected. This step is carried out in particular when the starting cereal material is a cereal grain. When the method comprises a dehulling step as described above, the grinding and/or flattening step is preferably carried out after the dehulling (the cereal material subjected to this grinding and/or flattening step then being a dehulled grain). This grinding and/or flattening step may be omitted, notably when the starting cereal material is in a powder or flattened form, such as in embodiments in which the starting cereal material is a product derived from the use of a cereal. Conversely, when the cereal material is not in a powder or flattened form, the method according to the invention preferably comprises this grinding and/or flattening step of said cereal material.

The grinding may be carried out by means of any type of grinder, notably any mechanical grinder using compression, impact, shearing or any other appropriate mechanical action. Preferably, the particles obtained after grinding have a median volume

diameter (D50) greater than or equal to 500 μm (the D50 can be measured according to the NF ISO 13320-1 standard). This grinding step may be carried out regardless of the cereal material (notably cereal grain and/or product derived from the use of a cereal).

5 The flattening may be carried out using a cereal flaker, preferably using the mechanical force of compression. This flattening step is preferably carried out when the cereal material used is a cereal grain.

10 The cereal material (in embodiments, the ground and/or flattened fraction obtained after grinding and/or flattening) is subjected to a solid/liquid extraction carried out using an alcohol solution. The alcohol solution according to the invention comprises at least 50% by volume of alcohol. The alcohol solution may be the alcohol itself (it then comprising 100% by volume of alcohol). However, it is preferably a mixture comprising at least one alcohol and at least one solvent. The solvent is preferably chosen from the group consisting of water, 2-methyloxolane and mixtures thereof. More preferably, the alcohol solution is an aqueous alcohol solution, i.e. a solution comprising water and alcohol. In an even more preferred manner, the aqueous ethanol solution consists of water and alcohol. Advantageously, the alcohol solution comprises less than 10% by volume of supercritical CO_2 and preferably of supercritical fluid, preferably less than 5% by volume, more preferably less than 2% by volume; more preferably the alcohol solution is free of supercritical CO_2 , more particularly free of supercritical fluid.

20 The alcohol solution preferably comprises from 50 to 90% by volume of alcohol (relative to the total volume of the alcohol solution used to perform the extraction), more preferably from 60 to 90% by volume, more preferably from 70 to 90% by volume, even more preferably from 80 to 90% by volume. In embodiments, the alcohol solution comprises from 50 to 60% of alcohol, or from 60 to 70%, or from 70 to 80%, or from 80 to 90%, by volume.

The alcohol is preferably chosen from the group consisting of ethanol, methanol, pentanol, isoamyl alcohol and mixtures thereof. In a particularly preferred manner, the alcohol is ethanol. In other words, the alcohol solution used according to the invention is preferably an ethanol solution.

30 The extraction is advantageously carried out by bringing the cereal material (the ground and/or flattened fraction, for example) into contact with the alcohol solution (preferably ethanol) in a reactor, preferably under agitation. Preferably, the cereal material (the ground and/or flattened fraction, for example) is brought into contact with the alcohol solution (preferably ethanol) at a mass ratio of 1:3 to 1:8, more preferably at a mass ratio of 1:4 to 1:7 (such as at a mass ratio of 1:3 to 1:4, or 1:4 to 1:5, or 1:5 to 1:6, or 1:6 to 1:7, or 1:7 to 1:8). Contact advantageously takes place for a duration of 15 to 120 min, preferably

from 20 to 60 min. Preferably, the cereal material particles are then separated from the alcohol solution by filtration, such as on a 10 µm mesh cloth.

In a particularly advantageous manner, when the alcohol solution is an ethanol solution, the ethanol used in the ethanol solution is produced by fermentation from a substrate as described in patent application FR 2211884, filed on 11 November 2022 and entitled "*Process for preparing a substrate for fermentation*". In particular, the fermentation substrate can be prepared by a process comprising the following steps: providing at least one legume seed comprising starch and proteins; micronizing said at least one seed so as to obtain a micronized fraction; purifying the micronized fraction (preferably by air classification) so as to collect a fraction enriched in starch and depleted in protein; mixing the fraction enriched in starch and depleted in protein with a liquid so as to form a starch fluid; hydrolyzing the starch by mixing water vapor with the starch fluid (preferably carried out using a direct steam injection device in continuous mode) so as to obtain a hydrolyzed starch fluid; and preferably introducing into the hydrolyzed starch fluid one or more glucosidase and/or saccharide enzymes. Using such a process to prepare the fermentation substrate for ethanol production has the advantage of a relatively low greenhouse gas emission rate, which can be below 400 kg CO₂ eq/t of ethanol.

Preferably, the alcohol (preferably ethanol) used to prepare the alcohol solution is recycled alcohol, i.e. it has already been previously used in a treatment process, preferably in an extraction process, more preferably in an extraction step of a method according to the invention.

The extraction step according to the invention makes it possible to extract at least a portion of the lipids from the cereal material in the alcohol solution (preferably ethanol). Thus, at the end of the extraction step and preferably of the filtration step, a liquid fraction enriched in lipids and a solid fraction depleted in lipids (also called oil cake) are obtained.

The oil cake is advantageously subjected to a drying step. Preferably, it is dried in a vacuum oven, preferably at a temperature of 30-50°C. Preferably, the oil cake is dried for a duration of 2 to 10 hours.

The extraction step may be repeated one or more times on the oil cake, preferably after drying the oil cake. In other words, the method according to the invention may comprise one, two or more extraction steps using an alcohol solution (preferably ethanol).

The liquid fraction enriched in lipids (comprising the alcohol/fat mixture) is preferably subjected to a purification step in order to separate the alcohol (preferably ethanol) from the lipid enriched fraction. More preferably, the alcohol (preferably ethanol) is separated by distillation. In particular, the liquid fraction enriched in lipids may be distilled at a temperature of 30-70°C, preferably in a tray column under vacuum. The oil is recovered at the bottom of

the column (forming a purified lipid-enriched fraction) while the alcohol (preferably ethanol) is collected at the top of the column.

In a particularly advantageous manner, the alcohol (preferably ethanol) recovered after separation from the liquid fraction enriched in lipids is recycled, and is more particularly reused for a new extraction cycle of the method according to the invention.

The method according to the invention comprises a step of grinding the solid fraction depleted in lipids (oil cake), preferably dried, then a step of separating the particles obtained as a function of their size. These steps aim to separate the beta-glucan fraction from the starch and protein fractions. For grinding, any suitable type of grinder may be used, notably any grinder using abrasion, compression, impact, shearing or any other mechanical action. In particular, a pendulum mill using compression may be used. At the end of grinding, a mixture of envelope fragments (essentially fibers) (also called bran in the present text) and kernel powder (also called flour in the present text) is obtained.

The step of separating the particles obtained at the end of grinding is preferably carried out by sieving. In particular, the particles may be separated by passing through a suitable mesh sieve making it possible to separate the bran from the flour. For example, particles smaller than a size between 200 and 600 μm (smaller than a size of between 200 and 250 μm , for example, or between 250 and 300 μm , or between 300 and 350 μm , or between 350 and 400 μm , or between 400 and 500 μm , or between 500 and 600 μm) may be separated from bran of a size greater than or equal to such a size. A mesh sieve of between 200 and 600 μm may be used in particular (such as between 200 and 250 μm , or between 250 and 300 μm , or between 300 and 350 μm , or between 350 and 400 μm , or between 400 and 500 μm , or between 500 and 600 μm). The bran separated during this step (i.e. the particles of a size greater than or equal to the size mentioned above, more particularly the particles of a size greater than or equal to the mesh of the sieve used, thus the particles retained by the sieve) corresponds to a fraction enriched in beta-glucans; the flour obtained after separation (i.e. the particles of a size smaller than the size mentioned above, more particularly the particles of a size smaller than the mesh of the sieve used, thus the particles passing through the sieve) corresponds to a fraction depleted in beta-glucans.

Preferably, the beta-glucan enriched fraction comprises a carbohydrate amount greater than or equal to 40% by weight, preferably an amount of 40 to 90% by weight, more preferably 40 to 80% by weight, more preferably 45 to 70% by weight (relative to the total dry matter weight of the fraction). In particular, the carbohydrate amount in the beta-glucan enriched fraction may comprise 40 to 50% by weight, or 50 to 60% by weight, or 60 to 65% by weight, or 65 to 70% by weight, or 70 to 75% by weight, or 75 to 80% by weight, or 80

to 90% by weight, relative to the total dry matter weight of the fraction. The carbohydrate amount can be measured according to the ISO 15914 standard.

Preferably, the beta-glucan enriched fraction comprises a protein amount less than or equal to 30% by weight, preferably 0.5 to 30% by weight, more preferably 3 to 20% by weight, more preferably 5 to 15% by weight (relative to the total dry matter weight of the fraction). In embodiments, the protein amount in the beta-glucan enriched fraction, relative to the total dry matter weight of the fraction, can be from 0.5 to 3% by weight, or 3 to 5% by weight, or 5 to 7% by weight, or 7 to 10% by weight, or 10 to 12% by weight, or 12 to 15% by weight, or 15 to 20% by weight, or 20 to 30% by weight. The protein amount can be measured according to the ISO 5983-2 standard (Kjeldahl method).

Preferably, the beta-glucan enriched fraction comprises a beta-glucan amount greater than or equal to 10% by weight, preferably 10 to 40% by weight, more preferably 15 to 25% by weight, relative to the total dry matter weight of the fraction; in particular the fraction may comprise a beta-glucan amount of 10 to 13% by weight, or 13 to 15% by weight, or 15 to 17% by weight, or 17 to 20% by weight, or 20 to 22% by weight, or 22 to 25% by weight, or 25 to 30% by weight, or 30 to 35% by weight, or 35 to 40% by weight, relative to the total dry matter weight of the fraction. The beta-glucan amount can be measured according to the AOAC 995.16 method.

Preferably, the beta-glucan enriched fraction comprises a fat (lipid) amount less than or equal to 5% by weight, preferably 0.5 to 5% by weight, more preferably 0.5 to 3% by weight, relative to the total dry matter weight of the fraction; in particular the fraction may comprise a fat amount of 0.5 to 1% by weight, or 1 to 2% by weight, or 2 to 3% by weight, or 3 to 4% by weight, or 4 to 5% by weight, relative to the total dry matter weight of the fraction. The fat amount can be measured according to the AOAC 922.06 method.

The beta-glucan-depleted fraction (flour) obtained after separation of the beta-glucan enriched fraction is subjected to a grinding step, and a ground fraction is then collected.

In a particularly advantageous manner, this grinding comprises, or is, micronization, preferably by a dry process. The ground fraction obtained is then a micronized fraction.

The micronization step makes it possible to dissociate the proteins from the starch granules to make their subsequent separation possible.

"Micronization" means a grinding that makes it possible to obtain particles with a median volume diameter of less than 100 μm , preferably less than 50 μm , more preferably less than 30 μm . The median volume diameter (D50) of the particles can be measured according to the NF ISO 13320-1 standard.

The micronization can be carried out by any suitable grinder (notably any grinder using mechanical forces of abrasion, compression, impact or shearing). For example, an impact mill may be used.

5 The micronization is very preferably carried out at ambient temperature (i.e. between 15 and 30°C).

The micronization has the additional advantage of being low in greenhouse gas emissions, with it preferably being carried out at ambient temperature and by a dry process.

10 The method according to the invention comprises a step of separating the starch granules from the proteins of the ground, beta-glucan depleted fraction, preferably further micronized.

At the end of the separation, a starch-enriched fraction, preferably a starch-enriched and protein-depleted fraction, is collected. A protein-enriched fraction, preferably a protein-enriched and starch-depleted fraction, is also recovered. A “starch-enriched and protein-depleted fraction” means a fraction in which the protein/starch molar proportion ratio (on dry matter) is greater than that of the fraction subjected to purification. A “protein-enriched and starch-depleted fraction” means a fraction in which the protein/starch molar proportion ratio (on dry matter) is greater than that of the fraction subjected to purification.

20 Given the difference in size between the proteins and the starch granules (D50 of about 1 to 5 µm for proteins and about 10 to 30 µm for starch granules) and in density, a separation based on a difference in particle size, density or weight can advantageously be used. In the method according to the invention, the separation is air classification. “Air classification” means any separation technology that uses a gas jet (preferably air) carrying along at least part of the particles to be separated. More preferably, the separation is cyclonic separation. It can be carried out by means of a cyclone, advantageously associated with a selector. A “selector” means any variable-speed rotating element equipped with radial blades installed in one part (preferably the upper part) of a cyclonic separator. This equipment makes it possible to increase the particle separation efficiency as a function of their density. Using an air classification apparatus makes it possible to recover the lightest particles, carried along by the gas flow, at one end of the apparatus (protein-enriched fraction), while heavier particles are collected at another end (starch-enriched fraction). In other words, at the end of air classification, a fraction of lighter particles corresponding to the protein-enriched fraction and a fraction of heavier particles corresponding to the starch-enriched fraction are obtained.

35 The air classification is preferably carried out using a selector operating at a rotational speed of between 7,000 and 14,000 rpm, more preferably between 8,000 and 13,000 rpm, more preferably between 9,000 and 12,000 rpm. The gas flow (preferably air) passing

through the selector preferably has a flow rate of between 40 and 200 m³/h, more preferably between 60 and 150 m³/h, more preferably between 80 and 120 m³/h.

Preferably, the starch-enriched fraction comprises a carbohydrate amount greater than or equal to 40% by weight, preferably 40 to 90% by weight, more preferably 50 to 85% by weight, more preferably 60 to 82% by weight (relative to the total dry matter weight of the fraction). In particular, the carbohydrate amount in the recovered starch-enriched fraction may comprise 40 to 50% by weight, or 50 to 60% by weight, or 60 to 65% by weight, or 65 to 70% by weight, or 70 to 75% by weight, or 75 to 80% by weight, or 80 to 90% by weight, relative to the total dry matter weight of the fraction.

Preferably, the starch-enriched fraction comprises a protein amount of less than or equal to 15% by weight, preferably 0.5 to 15% by weight, more preferably 3 to 15% by weight, more preferably 8 to 15% by weight (relative to the total dry matter weight of the fraction). In embodiments, the protein amount in the recovered starch-enriched fraction, relative to the total dry matter weight of the fraction, may be 0.5 to 3% by weight, or 3 to 5% by weight, or 5 to 7% by weight, or 7 to 10% by weight, or 10 to 12% by weight, or 12 to 15% by weight.

Preferably, the starch-enriched fraction comprises a fiber amount of less than or equal to 10% by weight, preferably 0.5 to 10% by weight, more preferably 1 to 8% by weight, relative to the total dry matter weight of the fraction; in particular the fraction may comprise a fiber amount of 0.5 to 2% by weight, or 2 to 4% by weight, or 4 to 6% by weight, or 6 to 8% by weight, or 8 to 10% by weight, relative to the total dry matter weight of the fraction. "Fiber" means all plant polymeric molecules, soluble or insoluble, other than starch and starch fragments. In particular, fibers comprise cellulose, hemicellulose, lignin, beta-glucans and pectin. The fiber amount can be measured according to the AOAC 2011.25 method.

Preferably, the collected starch-enriched fraction comprises a fat (lipid) amount less than or equal to 5% by weight, preferably 0.5 to 5% by weight, more preferably 0.5 to 3% by weight, relative to the total dry matter weight of the fraction; in particular the fraction may comprise a fat amount of 0.5 to 1% by weight, or 1 to 2% by weight, or 2 to 3% by weight, or 3 to 4% by weight, or 4 to 5% by weight, relative to the total dry matter weight of the fraction.

Preferably, the protein-enriched fraction comprises a protein amount greater than or equal to 15% by weight, preferably 15 to 35% by weight, more preferably 15 to 30% by weight, more preferably 20 to 30% by weight (relative to the total dry matter weight of the fraction). In embodiments, the protein amount in the recovered protein-enriched fraction can be, relative to the total dry matter weight of the fraction, 15 to 18% by weight, or 18 to 22%

by weight, or 22 to 25% by weight, or 25 to 28% by weight, or 28 to 32% by weight, or 32 to 35% by weight.

Preferably, the protein-enriched fraction comprises a carbohydrate amount less than or equal to 75% by weight, preferably 0.5 to 70% by weight, more preferably 20 to 70% by weight, more preferably 40 to 70% by weight (relative to the total dry matter weight of the fraction). In embodiments, the carbohydrate amount in the recovered protein-enriched fraction can be, relative to the total dry matter weight of the fraction, 0.5 to 20% by weight, or 20 to 30% by weight, 30 to 40% by weight, or 40 to 50% by weight, or 50 to 60% by weight, or 60 to 70% by weight, or 70 to 75% by weight.

Preferably, the protein-enriched fraction comprise a fiber amount less than or equal to 10% by weight, preferably 0.75 to 10% by weight, more preferably 1 to 8% by weight, relative to the total dry matter weight of the fraction; in particular the fraction may comprise a fiber amount of 0.5 to 2% by weight, or 2 to 4% by weight, or 4 to 6% by weight, or 6 to 8% by weight, or 8 to 10% by weight, relative to the total dry matter weight of the fraction.

Preferably, the collected protein fraction comprises a fat (lipid) amount less than or equal to 5% by weight, preferably 0.5 to 5% by weight, more preferably 0.5 to 3% by weight, relative to the total dry matter weight of the fraction; in particular, the fraction may comprise a fat amount of 0.5 to 1% by weight, or 1 to 2% by weight, or 2 to 3% by weight, or 3 to 4% by weight, or 4 to 5% by weight, relative to the total dry matter weight of the fraction.

The carbohydrate, protein, fiber and fat amounts can be determined as described above.

An example method according to the invention is shown in Figure 1. In this example, a cereal material 1, in particular oats and/or barley, preferably undergoes a first grinding 2 and a ground fraction 3 is obtained. This ground fraction 3 is subjected to an extraction treatment 4 using an alcohol solution, preferably an ethanol solution. At the end of this extraction 4, a liquid fraction 5 enriched in lipids comprising an alcohol/lipid mixture and a solid fraction 6 depleted in lipids are collected. The lipid-depleted solid fraction 6 is then subjected to a second grinding step 7. The recovered fraction 8 corresponds to a mixture of flour (ground kernel) and bran. A step 9 of separating the particles of fraction 8, as a function of their size, in particular by sieving, is then carried out in order to separate the flour from the bran. Two fractions are collected: the fraction of larger particles corresponds to bran and to a beta-glucan enriched fraction 10, and the fraction of finer particles corresponds to flour and to a beta-glucan-depleted fraction 11. The beta-glucan-depleted fraction 11 is subjected to a grinding step 12, preferably micronization, followed by a particle separation step 13 by air classification. Two fractions are then obtained: a fraction of lighter

particles corresponding to a protein-enriched fraction 14 and a fraction of heavier particles corresponding to a starch-enriched fraction 15.

The invention also relates to a fraction obtained or obtainable by a separation method as described above.

5 In particular, the invention also relates to a lipid-enriched fraction obtained or obtainable by a separation method as described above.

The invention also relates to a beta-glucan enriched fraction obtained or obtainable by a separation method as described above.

10 The invention also relates to a protein-enriched fraction obtained or obtainable by a separation method as described above.

The invention also relates to a starch-enriched fraction obtained or obtainable by a separation method as described above.

The lipid-, beta-glucan-, protein- or starch-enriched fractions may be as described above.

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Examples

The following examples illustrate the invention, without limiting it.

In the examples, the following analysis and measurement methods were used:

- 20 - Measurement of protein amount: the Kjeldahl method using a FOSS titrator according to the ISO 5983-2 standard;
- Measurement of lipid amount: the AOAC 922.06 method;
- Measurement of carbohydrate (starch) amount: the method according to the ISO 15914 standard;
- Measurement of total fiber amount: the AOAC 2011.25 method;
- 25 - Measurement of beta-glucan amount: the AOAC 995.16 method.

Example 1: Production of a beta-glucan enriched fraction

25 kg of coarsely ground oat groats were introduced into a Nutsche-type reactor with a capacity of 250 liters.

30 The reactor was equipped with a 10 µm filter at the bottom of the vessel.

The composition of the oat groats introduced into the reactor is given in the table below (as weight percentage).

[Table 1]

Moisture	12.0%
Proteins	11.1%
Fats	5.8%
Carbohydrates	58.9%
Fibers	10.8%
Ash	1.4%

134 kg (170 liters) of dehydrated ethanol were mixed with 30 kg of water in order to obtain an aqueous ethanol solution with an ethanol concentration of 85% v/v. The ethanol solution was prepared at ambient temperature.

The ethanol solution was introduced into the reactor, with the mass ratio of ethanol solution to groats being 6.6:1. The groats/ethanol solution mixture was kept agitated for 45 minutes at ambient temperature.

After this extraction time, the liquid was drained through the 10 μm filter under a constant pressure of 2 bar. The oil cake was then dried in a vacuum oven at a temperature of 45°C until all traces of ethanol disappeared.

The ethanol solution containing the fats was distilled in a tray column configured to obtain ethanol at 96% v/v at the top of the column. This alcohol can thus be recycled in a new extraction.

Oat oil was recovered at the bottom of the column.

A second extraction was carried out on the dried oil cake under the same conditions as the first (i.e. 45 minutes of contact time under agitation with 200 liters of ethanol at 85% v/v in water).

The fat extraction yield is 91% (by weight). A small amount of flour is present in the oil obtained, this flour resulting from the passage of particles smaller than 10 μm through the filter of the extraction reactor.

The lipid balance of the extraction operations is shown in the table below:

[Table 2]

	Quantity (kg)	Fats (% by weight)	Fat quantity (kg)	Oil yield of the extraction (%)
Groats	25	5.8	1.45	
Oil collected after the first extraction			1.13	77.9
Oil collected after the second extraction			0.19	91.0
De-oiled flour obtained after the first extraction	23.87	1.3	0.31	
De-oiled flour obtained after the second extraction	23.68	0.5	0.12	

The de-oiled flour was then ground according to the following process. The bran (outer fibrous envelope of the oat) was separated using a Buhler MLU 202 mill in soft-wheat configuration and a 250 μ m sieve. The bran fraction obtained at the end of this separation is a beta-glucan enriched fraction; the white flour fraction remaining after separation of the bran corresponds to a beta-glucan-depleted fraction.

The mass balance of the separation operation is summarized in the table below:

[Table 3]

	Mass balance (% by weight)
Bran fraction	24.7
White flour fraction	73.8
Losses	1.5

The losses are due to a decrease in moisture and to some traces of powders remaining on the walls of the powder-conveying ducts.

The fiber content balance given in the table below (as weight percentage relative to the total dry matter weight (DM)) indicates an almost threefold increase in the beta-glucan content in the beta-glucan enriched fraction (bran) as compared with the flour obtained after extraction, for a mass yield of the bran fraction of 24.7%.

[Table 4]

	Total fiber content (TDF) (%/DM)	Beta-glucan content (%/DM)
De-oiled oat flour	10.6	5.1
Bran fraction	26.3	14.9
White flour fraction	5.3	2.2

Example 2: Production of a de-oiled oat flour

25 kg of coarsely ground oat groats were introduced into a Nutsche-type reactor with a capacity of 250 liters. The reactor was equipped with a 10 µm filter at the bottom of the vessel.

The groats are as described in Example 1.

A single extraction was carried out under the same conditions as in Example 1 (i.e. 45 minutes contact time, under agitation, with 200 liters of ethanol at 85% v/v in water, with a mass ratio of aqueous ethanol solution to groats of 6.6).

The oil was separated from ethanol by distillation, as described in Example 1. The fat extraction yield is 82.8% by weight. A small amount of flour is present in the oil obtained (particles smaller than 10 µm).

The lipid balance of the extraction operation is shown in the table below:

[Table 5]

	Quantity (kg)	Fats (% by weight)	Fat quantity (kg)	Oil yield of the extraction (%)
Groats	25	5.8	1.45	
Oil collected after extraction			1.20	82.8
De-oiled flour	23.8	1.0	0.24	

Example 3: Production of a de-oiled oat flour

25 kg of coarsely ground oat groats were introduced into a Nutsche-type reactor with a capacity of 250 liters. The reactor was equipped with a 10 µm filter at the bottom of the vessel.

The groats are as described in Example 1.

100.6 kg (127.3 liters) of dehydrated ethanol were mixed with 22.5 kg of water in order to obtain an aqueous ethanol solution with an alcohol concentration of 85% v/v. The ethanol solution was prepared at ambient temperature.

The ethanol solution was introduced into the reactor, with the mass ratio of ethanol solution to groats being 4.9:1. The groats/ethanol solution mixture was kept agitated for 45 minutes at ambient temperature.

After this extraction time, the liquid was drained through the 10 μ m filter. The operation was carried out under a constant pressure of 2 bar. The oil cake was then dried in a vacuum oven at a temperature of 45°C until all traces of ethanol disappeared.

The ethanol solution containing the fats was distilled in a tray column configured to obtain ethanol at 96% v/v at the top of the column. This alcohol can be recycled to perform a new extraction.

Oat oil was recovered at the bottom of the column.

The fat extraction yield is 80.7% by weight. A small amount of flour is present in the oil obtained.

The lipid balance of the extraction operation is shown in the table below:

[Table 6]

	Quantity (kg)	Fats (% by weight)	Fat quantity (kg)	Oil yield of the extraction (%)
Groats	25	5.8	1.45	
Oil collected after extraction			1.17	80.7
De-oiled flour	23.83	1.1	0.26	

Example 4: Production of a de-oiled oat flour

25 kg of coarsely ground oat groats were introduced into a Nutsche-type reactor with a capacity of 250 liters. The reactor was equipped with a 10 μ m filter at the bottom of the vessel.

The groats are as described in Example 1.

74 kg (93.7 liters) of dehydrated ethanol were mixed with 18.6 kg of water in order to obtain an aqueous ethanol solution with an alcohol concentration of 83% v/v. The ethanol solution was prepared at ambient temperature.

The ethanol solution was introduced into the reactor, the mass ratio of ethanol solution to groats being 3.7:1. The groats/ethanol solution mixture was kept agitated for 30 minutes at ambient temperature.

After this extraction time, the liquid was drained through the 10 μm filter. The operation was carried out under a constant pressure of 2 bar. The oil cake was then dried in a vacuum oven at a temperature of 45°C until all traces of ethanol disappeared.

The ethanol solution containing the fats was distilled in a tray column configured to obtain alcohol at 96% v/v. This alcohol can be recycled to perform a new extraction.

The oat oil was recovered at the bottom of the column.

The fat extraction yield is 77.2% by weight. A small amount of flour was found in the oil obtained.

The lipid balance of the extraction operation is shown in the table below:

[Table 7]

	Quantity (kg)	Fats (% by weight)	Fat quantity (kg)	Oil yield of the extraction (%)
Groats	25	5.8	1.45	
Oil collected after extraction			1.12	77.2
De-oiled flour	23.88	1.3	0.31	

Example 5: Production of a protein-enriched fraction and a starch-enriched fraction

The white flour fraction (oat flour without bran) obtained in Example 1 was micronized by means of a grinder then subjected to cyclonic air classification (turbo-separation). The lighter protein-enriched fraction (called protein fraction) was collected at the top of the cyclone while the heavier starch-enriched fraction (called starch fraction) was collected at the bottom of the cyclone. Details of the equipment are given below:

– ZPS mill (70ZPS+70 ATP, Hosokawa-Alpine®):

- mill speed: 14,000 rpm;
- baffle orientation: facing
- air flow rate: 100 m³/h;
- feed: 105 rpm;
- selector speed: 3,800 rpm.

– 70 ATP turbo-separator (Hosokawa-Alpine®):

- air flow rate: 100 m³/h;
- feed: 105 rpm;

- selector speed: 9,000 rpm.

The mass balance of the operation is summarized in the table below:

[Table 8]

	Mass balance (% by weight)
Protein fraction	30.3
Starch fraction	66.8
Losses	2.9

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The losses are due to a decrease in moisture and to some traces of powders remaining on the walls of the powder-conveying ducts.

The protein and carbohydrate balance given in the table below (as weight percentage relative to the total dry matter weight (DM)) indicates a 35% increase in protein content in the protein fraction compared with the white flour fraction, for a mass yield of the fraction of 30.3%.

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[Table 9]

	Protein content (%/DM)	Carbohydrate content (%/DM)
White flour fraction	11.8	78.0
Protein fraction	15.9	74.0
Starch fraction	10.1	79.8

Example 6: Production of a protein-enriched fraction and a starch-enriched fraction

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The white flour fraction (oat flour without bran) obtained in Example 1 was micronized by means of a grinder then subjected to cyclonic air classification (turbo-separation). The lighter protein fraction was collected at the top of the cyclone while the heavier starch fraction was collected at the bottom of the cyclone. The equipment and operating conditions used to carry out these steps are the same as those indicated in Example 5, except for the selector speed of the turbo-separator, which was 12,000 rpm.

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The mass balance of the operation is summarized in the table below:

[Table 10]

	Mass balance (% by weight)
Protein fraction	12.0

Starch fraction	85.9
Losses	2.1

The losses are due to a decrease in moisture and to some traces of powders remaining on the walls of the powder-conveying ducts.

- 5 The protein and carbohydrate balance given in the table below (as weight percentage relative to the total dry matter weight (DM)) shows that the protein content has doubled in the protein fraction compared with the white flour fraction, for a mass yield of the fraction of 12.0%.

[Table 11]

	Protein content (%/DM)	Carbohydrate content (%/DM)
White flour fraction	11.8	78.0
Protein fraction	23.4	68.3
Starch fraction	10.3	79.3

Claims

1. A method for separating a cereal material, comprising the following steps:

 - providing at least one cereal material (1) comprising starch, proteins, lipids and beta-glucans;
 - treating the cereal material by solid/liquid extraction (4) with an alcohol solution comprising at least 50% by volume of alcohol, so as to obtain a solid fraction depleted in lipids (6) and a liquid fraction enriched in lipids (5);
 - grinding (7) the solid fraction depleted in lipids (6);
 - separating (9) the particles of the ground lipid-depleted fraction (8) according to their size, so as to obtain a fraction enriched in beta-glucans (10) and a fraction depleted in beta-glucans (11);
 - grinding (12) the fraction depleted in beta-glucans (11); and
 - treating the ground fraction depleted in beta-glucans by air classification (13), so as to obtain a fraction enriched in proteins (14) and a fraction enriched in starch (15).
2. The method according to claim 1, wherein the cereal material (1) is an oat and/or barley cereal material.
3. The method according to claim 1 or 2, wherein the cereal material (1) is at least one cereal grain and/or at least one product from the malting, malt extract manufacturing, brewing, cereal distillation, plant-based dairy and/or milling industries.
4. The method according to any one of claims 1 to 3, wherein the alcohol solution is a mixture of alcohol and at least one solvent, the solvent preferably being chosen from the group consisting of water, 2-methyloxolane and mixtures thereof, more preferably the alcohol solution consists of a mixture of alcohol and water.
5. The method according to any one of claims 1 to 4, wherein the alcohol solution comprises from 50 to 90% by volume of alcohol.
6. The method according to any one of claims 1 to 5, wherein the alcohol of the alcohol solution is recycled alcohol.
7. The method according to any one of claims 1 to 6, wherein the alcohol of the alcohol solution is ethanol.

8. The method according to any one of claims 1 to 7, wherein grinding (12) the fraction depleted in beta-glucans (10) comprises micronizing the fraction depleted in beta-glucans (10).

9. The method according to any one of claims 1 to 8, wherein separating (9) the particles of the ground lipid-depleted fraction (8) according to their size is carried out by sieving.

10. The method according to any one of claims 1 to 9, further comprising a step of separating the alcohol from the liquid fraction enriched in lipids, preferably by distillation, the method preferably further comprising a step of recycling the separated alcohol for the solid/liquid extraction step (4).

11. The method according to any one of claims 1 to 10, further comprising, before the step of treating the cereal material by solid/liquid extraction, a step of grinding (2) and/or flattening the cereal material so as to obtain a ground (3) and/or flattened fraction, the provided cereal material (1) being preferably a cereal grain.

12. The method according to claim 11, wherein the cereal material (1) is a cereal grain comprising a hull, the method further comprising a step of dehulling the grain prior to the step of grinding (2) and/or flattening the cereal material (1).

13. The method according to any one of claims 1 to 12, wherein the fraction enriched in beta-glucans (11) comprises a beta-glucan amount greater than or equal to 10% by weight, preferably from 10 to 40% by weight, more preferably from 15 to 25% by weight, relative to the total dry matter weight of the fraction.

14. The method according to any one of claims 1 to 13, wherein the starch-enriched fraction (15) comprises a carbohydrate amount greater than or equal to 40% by weight, preferably from 40 to 90% by weight, more preferably from 50 to 85% by weight, relative to the total dry matter weight of the fraction.

15. The method according to any one of claims 1 to 14, wherein the protein-enriched fraction (14) comprises a protein amount greater than or equal to 15% by weight, preferably from 15 to 35% by weight, more preferably from 15 to 30% by weight, even more preferably from 20 to 30% by weight, relative to the total dry matter weight of the fraction.