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## (54) Process for producing proteinrich product, fibrous product and/or vegetable oil from brewer's spent grain.

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## Description

This invention relates to a process for producing a protein-rich product and a fibrous product from brewer's spent grain (hereinafter referred to as BSG). The protein-rich product obtained by the present invention is suitable for use as feed for farm animals and cultured fish, and is also expected to find applications as a human food material, e.g. as starting material for the manufacture of seasoning.

Soybean and defatted soybean are widely used to produce protein-rich vegetable products. However, soybean-producing districts are localized in the world, and a problem of short supply of soybean can arise on account of unseasonable weather. In addition, soybean supply in Japan almost completely depends upon imports, with the import price varying depending upon various factors. Hence, protein-rich sources other than soybean, which can be supplied stably at a low price, have been desired.

BSG, which is a by-product in the brewing of beer, contains protein in an amount of as low as about 25% based on dry weight, and accordingly its use has been limited in the past to cattle feed.

In order to increase its protein content, it has been attempted to mill and sieve dry BSG (U.S. Patents 4,377,601 and 4,547,382). In this process, husks become so fine in the grinding step that separation thereof from the protein-containing fraction becomes difficult, and the protein content of the resulting product is still as low as 30 to 40 wt % based on dry weight. Thus, the product of these prior processes is still not satisfactory as a protein source. In addition, the process has another defect that, since raw BSG contains water, a large amount of heat energy is required for drying it. Hence, it is desired to develop a process which provides a protein-rich product containing proteins in an amount equal to or more than that of soybean (containing about 40% of proteins) or defatted, protein-rich product containing proteins in an amount equal to or more than that of defatted soybean (containing about 50% of proteins).

Japanese Unexamined Patent Publication No. 51-129776 discloses a process of extracting BSG using an alkaline aqueous solution of 11-12 in pH at a temperature of 220°F-250°F (104°C-121°C), then precipitating a protein-rich product from the thus-obtained extract by isoelectric precipitation. This process, however, involves decomposition of proteins due to the severe, high-temperature extraction condition, resulting in a decreased yield of proteins and deteriorated quality of the product. Also, the protein-rich product obtained by this process does not contain lipid present in BSG, with the lipid, which is important as feed for farm animals and fish, being wasted. In addition, this process requires a large heat energy for extraction at an elevated temperature. Further, in actual procedures, the alkaline aqueous solution must be added to the BSG, resulting in an increase in temperature of the system and thus a complication of the extracting procedures.

Another process for recovering protein solids from BSG using an alkaline extraction procedure is taught in US-A-3846397.

DE-A-3704651 discloses a process for separating BSG into a high fibre fraction and a lipid fraction, in which a suspension of BSG in water is heated and then introduced into a high pressure homogenizer which pulps the BSG particles. The resulting homogenized product, on separation into its fractions, yields a lipid-containing emulsion layer and a protein-rich sediment layer. However, this protein-rich sediment contains a significant content of pulped husk material from the BSG as a result of the vigorous treatments to which the BSG is subjected, and since this husk material cannot be removed the protein content of the sediment is thereby limited. Moreover, the process involves a heating step which for reasons of cost it would be desirable to avoid.

As a result of intensive investigations to solve the above-described problems, the inventors have found that a protein-rich product can be inexpensively obtained from BSG.

There is provided in accordance with the present invention a process for obtaining a protein-rich product and a fibrous product from brewer's spent grain (BSG) containing germ, husks and a proteinaceous material adhering to the husks, wherein the BSG in a wet state is pressed and then sieved for the separation thereof into a protein-rich product and a fibrous product, characterized in that the BSG in a wet state is passed through a roll mill to press said BSG with simultaneous grinding of the germ and the proteinaceous material, thereby separating said germ and proteinaceous material from said husks, and in that the BSG, after passage through the roll mill, is sieved one or more times, with at least one sieving being in water, whereby there is recovered a protein-rich product containing the germ and proteinaceous material as a minus fraction and the husks as a plus fraction.

In an embodiment of the present invention the sieving is carried out so that the protein-containing product, separated from the husks, is recovered as a fine fraction having a high protein content and a coarse fraction having a relatively lower protein content, and thereafter the protein-containing coarse particulate fraction obtained by the above sieving treatment is extracted with a 0.05 N to 0.15 N aqueous alkaline solution at a temperature of 60 to 100°C for 10 to 40 minutes, in order to obtain a protein-rich

product.

If desired, the protein-rich products obtained by the above-mentioned processes may be extracted in order to produce a vegetable oil and a defatted, protein-rich product.

These processes are described in detail below.

BSG is a saccharification residue of brewer's malt (optionally containing rice, corn grits, corn starch, etc. as secondary starting materials) and is obtained as a by-product in the production of beer. Usually, the BSG is separated from wort in a wet state by means of a solid-liquid separator such as a lauter tub or a mash filter, and has a water content of about 80 wt % and contains about 25 wt % of proteins on dry basis. In the present invention, BSG with a water content of about 70-80 wt % separated from the wort may be used as such, or the BSG may be dewatered or moistened before use. Still further, dried BSG may be moistened so as to be useful as the starting material.

The process of the present invention involves the steps of pressing BSG in a wet state by means of a roll mill and sieving the pressed BSG in water.

The BSG is constituted by husks, germs and other particles, and contents of proteins thereof are about 5 wt %, about 50 wt % and about 50 wt %, respectively. The germs and other particles having a high protein content are bound or stuck to the husks as vegetable tissues. Therefore, if the husks can be removed from the BSG, a protein-rich product can be obtained.

The inventors' investigation has revealed that, when BSG is pressed in a wet state in a roll mill, germs and other particles having a high protein content are separated from husks and, at the same time, the separated germs and the particles are ground, with the husks remaining substantially non-ground and that the thus-obtained pressed BSG can be effectively separated into a protein-containing product and a fibrous (husk) product by sieving the pressed BSG at least once in the presence of water.

In the pressing treatment using a roll mill, BSG in a wet state is pressed and partly ground by the pressing force between the rolls to thereby separate from the husks protein-rich germs and particles which have been bound or stuck thereto with the simultaneous grinding of the germs and particles. The gap between the rolls is suitably 0.05 to 2 mm, preferably 0.1 to 0.3 mm. In the treatment of pressing BSG, it suffices for BSG to have a water content not causing fine grinding of husks, with a water content equal to or greater than 65 wt % being particularly preferable. If the water content is too low, part of the husks are ground to fine particles, which makes it difficult to separate the pressed BSG into husks and a protein-containing product by sieving.

Additionally, BSG before being processed contains, to some extent, a fine particulate, protein-containing product in a separate state from husks. It is, therefore, possible to preliminarily sieve the BSG prior to the pressing treatment to thereby separate and recover this fine particulate, protein-containing product. A sieve to be used in this preliminary sieving is of 20 to 50 meshes, preferably 30 to 35 meshes. This sieving treatment is conducted preferably in the presence of water.

In the present invention, the pressed BSG obtained as mentioned hereinbefore is then sieved one or more times, with at least one sieving being in the presence of water, whereby the BSG is separated into a fibrous fraction comprising husks and a fraction of protein-containing product. As suitable techniques for sieving the pressed BSG in the presence of water, there may be mentioned, for example, a sieving technique wherein BSG is sieved using an apparatus whose sieving part is submerged, a sieving technique wherein previously watered BSG is fed to a sieving apparatus, and a sieving technique wherein BSG is sieved with a sieving apparatus while supplying or spraying water thereto. As the sieving apparatus, a vibrating filter is preferably used.

In the sieving treatment, a fraction of a fibrous product (size: 1 - 5 mm) composed of husks is obtained as a plus sieve. For this purpose, the sieve opening is suitably of 5 - 20 meshes, preferably 10 - 15 meshes. On the other hand, a fraction passing through this sieve comprises both a fraction of relatively fine particles having a higher protein content and relatively coarse particles having a lower protein content than the fine particles. In order to separate the former fraction of more protein-rich product (fraction of fine particles) from the coarser, lower protein content particles a sieving treatment may be further conducted using a sieve of 20 - 50 meshes, preferably 30 - 35 meshes. In this further sieving treatment, the coarse particles product is retained over the sieve, whereas the fine particles product is permitted to pass through the sieve.

However, the sieving treatment employed in the present invention is not particularly limited as to the number of times of sieving, order of sieving treatments, and other conditions, which may be variously varied and properly selected in consideration of clogging of sieve opening, etc. In the present invention, the aforesaid pressing treatment and this sieving treatment are preferably repeated several times (2 to 5 times) for the purpose of effectively separating and recovering the fraction of protein-rich product (fraction of fine particles). For example, a fraction of fibrous product is separated from the pressed BSG by first sieving, the

remaining fraction is again sieved by second sieving to thereby separate it into a fraction of relatively coarse particles and a fraction of relatively fine particles, the fraction of coarse particles is again pressed, and the thus-obtained pressed coarse particles are sieved into a fraction of fine particles and a fraction of coarse particles.

The combination of pressing treatment and sieving treatment described above enables one to obtain a fine particulate protein-rich product (fraction of fine particles) having a protein content of 40 wt % or more (based on dry weight), a protein-containing product (fraction of coarse particles) having a protein content of about 25 wt % ((based on dry weight), and a fibrous product composed of husks. The thus-obtained fine particulate protein-rich product can be used as such or after drying or other treatment, as material for feed or food. For some uses, the product is advantageously defatted to be used as defatted protein-rich product. On the other hand, the coarse particulate protein-containing product can be utilized as feed for cattle, like conventional BSG, or it may be subjected to an alkali-extracting treatment to be described in more detail hereinafter to separate and recover a protein-rich product therefrom, which is preferred. The fibrous product composed of husks can be effectively utilized as feed or fuel, and the combustion ash thereof can be utilized as fertilizer or ceramic material.

The alkali extraction of the coarse particulate protein-containing product (fraction of coarse particles) obtained as described above is conducted by adding a 0.05 N - 0.15 N alkaline aqueous solution to the coarse particulate protein-containing product and conducting the extraction at 60 • - 100 • C, preferably 70 • - 90 • C, for 10 - 40 minutes, preferably 15 - 35 minutes. As the alkali for preparing the alkaline aqueous solution, sodium hydroxide, potassium hydroxide, calcium hydroxide, etc. are usually used.

After the aforesaid extraction using the alkaline aqueous solution, a protein-rich product is recovered by precipitation. For example, an acid is added to the extract immediately after the extraction treatment to adjust pH to 7 - 11, an extraction residue is removed from the extract, and further acid is added to the thus-obtained extract to adjust the pH to 2 to 5, preferably to an isoelectric point of 3 to 4, to thereby precipitate the protein-rich product, which is separated and recovered by a solid-liquid separation method. As the solid-liquid separation method, conventional techniques such as centrifugal separation, filtration separation, etc. may be employed. By this means a product having a protein content of about 50 wt % or above and a lipid content of about 15 wt % or above can be obtained.

In the extraction treatment in accordance with this embodiment of the present invention, a comparatively low extracting temperature of 60 to 100 • C and a short extracting time of 10 to 40 minutes are employed. Employment of such conditions serves to avoid decomposition of the extracted protein-rich product. After completion of the alkali extraction treatment, an acid may immediately be added, if necessary, to the extract to reduce its pH to 7 - 11 to thereby inhibit decomposition of protein with alkali. A protein-rich product may be obtained by supressing decomposition of proteins as described above. This protein-rich product may be utilized as feed or the like in an as-produced form or after treatment such as defatting treatment or drying.

All of the thus-obtained protein-rich products contain lipid but, in some uses, they are advantageously used as defatted products. For such uses, the protein-rich product is extracted with a solvent in a wet state or in a state of being dried at low temperatures (30 - 100 • C) to recover lipid (vegetable oil), followed by removing the solvent. As solvents to be used in this case, there may be used paraffins such as n-hexane, alcohols such as ethanol, and supercritical carbon dioxide gas, etc. The solvent-removing treatment may be conducted by heating the extract residue to a temperature equal to or above the boiling point of the solvent under ambient or reduced pressure, or by a pressure-reducing procedure. Further, removal of the solvent from the extract enables lipid (vegetable oil) to be recovered.

This invention thus can provide a protein-rich product, a fibrous product and vegetable oil with an enhanced value effectively and inexpensively from BSG. The protein-rich product can be advantageously utilized as feed for farm animals or cultured fish or as material for food such as seasoning material, as is the case with conventional soybean protein.

The present invention is illustrated by the Examples which follow.

Example 1

About 10 kg of water was added to 3 kg (dry weight: 672 g) of BSG in a wet state (water content: 77.6 wt %), and the resulting mixture was subjected to preliminary sieving in water using a 35-mesh sieve. A fraction of fine particulate protein-containing product which passed through the sieve was dewatered by centrifugation and recovered in a slurry state. On the other hand, a fraction of coarse particulate protein-containing product remaining over the sieve was pressed flat by means of a roll mill (roll-rotating rate: 100 rpm; roll-to-roll gap: 0.1 mm) to thereby separate particles bound or stuck to husks from the husks and

concurrently grind the particles. Then, this roll mill-treated product was sieved using a 35-mesh sieve to recover a fraction of fine particulate protein-containing product. Then, a fraction of coarse particulate protein-containing product remaining over the sieve was again subjected to roll mill treatment and sieving treatment in the same manner to recover a fraction of fine particulate protein-containing product. Further, a fraction of coarse particulate protein-containing product remaining over the sieve was sieved in water using a 10-mesh sieve to recover 160 g by dry weight of a fibrous product as a fraction composed of only husks remaining over the sieve.

The fine particulate protein-containing product recovered as a slurry in the above-described manner was vacuum dried to obtain 180 g of a dry product. This fine particulate protein-containing product was a protein-rich product having a protein content of 50.8 wt % based on dry weight.

Example 2

500 ml of ethanol was added to 100 g of the protein-rich product obtained in Example 1, followed by extraction treatment at 30 °C for 1 hour. This extraction treatment yielded 83.0 g of defatted protein-rich product (dry product) and 17.0 g of a vegetable oil, with the protein content in the defatted protein-rich product being 62.0 % based on dry weight.

Example 3

About 30 liters of water was added to 10 kg (dry weight: 2.24 kg) of BSG in a wet state (water content: 77.6 wt %), and the resulting mixture was subjected to preliminary sieving in water using a 35-mesh sieve. A fraction of fine particulate protein-containing product passed through the sieve was dewatered by centrifugation and recovered in a slurry state. On the other hand a fraction of coarse particulate protein-containing product remaining over the sieve was pressed by means of a roll mill (roll-rotating rate: 100 rpm; roll-to-roll gap: 0.3 mm) to thereby separate particles bound or stuck to husks from the husks and concurrently grind the particles. Then, this roll mill-treated product was sieved using a 10-mesh sieve to recover a fraction of fibrous product composed of only husks remaining over the sieve, and a fraction of fine particulate and coarse particulate protein-containing product which passed through the sieve. Then, this sieve-passed fraction was sieved in water using a 35-mesh sieve to recover a coarse particulate protein-containing product as a plus sieve and a fine particulate protein-containing product as a minus sieve.

The dry weight of the thus recovered fraction of fine particulate protein-containing product was 522 g, with its protein content being 51.49 % based on dry weight and the protein-recovery ratio being 47.2 %.

On the other hand, the weight (dry weight) of the fraction of the coarse particulate protein-containing product obtained above was 943 g, with its protein content being 25.9 wt %.

Then, 500 ml of water was added to 100 g (in a wet state) of the fraction of coarse particulate protein-containing product, followed by further adding a 2N NaOH aqueous solution to prepare a 0.1 N alkaline aqueous solution as a whole. After stirring the solution at 85 °C for 30 minutes, an extraction residue was removed by centrifugation, and the resulting extract was adjusted to 4.5 in pH with 2N hydrochloric acid. This pH adjustment gave a precipitate, and this precipitate was recovered by centrifugation. The dry weight of this precipitate was 19.0 g, with its protein content being 58.6 wt % and the protein-recovery ratio from the fraction of coarse particulate protein-containing product being 42.9 wt %.

As is described above, 72.2 % of proteins can be recovered from BSG by combining the pressing treatment and the alkali extraction treatment.

The relationship between alkali concentration, extracting period and protein-recovery ratio when conducting the extraction of the coarse particulate protein-containing product by adding 1 liter of a NaOH aqueous solution to 100 g (water content: 75 wt %) was determined and the results are tabulated in Table 1.

Table 1

| Alkali concentration (N) | Protein-recovery Ratio (wt %) | | | | |
|---|---|---|---|---|---|
| | Extracting Period (min.) | | | | |
| | 15 | 30 | 45 | 60 | 120 |
| 0.04 | 42.1 | 41.9 | 38.4 | 41.3 | 39.1 |
| 0.10 | 44.0 | 50.5 | 33.7 | 31.3 | 28.1 |
| 0.20 | 34.6 | 26.8 | 24.9 | 17.8 | 15.6 |
| 0.40 | 7.64 | 6.13 | 5.19 | 5.23 | 2.18 |

Further, the relationship between temperature, alkali concentration, extracting period and protein-recovery ratio and protein content in the case of conducting the extraction of the coarse particulate protein-containing product for 30 minutes by adding 1 liter of a 0.1 N NaOH aqueous solution to 100 g (water content: 75 wt %) was determined and the results are tabulated in Table 2.

Table 2

| Extracting Temp.(°C) | 50 | 70 | 90 | 100 | 120 |
|---|---|---|---|---|---|
| Protein-recovering Ratio (%) | 27.2 | 44.1 | 48.4 | 55.3 | 35.0 |
| Protein Content (%) | 60.2 | 59.8 | 58.6 | 46.6 | 46.1 |

**Claims**

1. A process for producing a protein-rich product and a fibrous product from brewer's spent grain (BSG) containing germ, husks and a proteinaceous material adhering to the husks, wherein the BSG in a wet state is pressed and then sieved for the separation thereof into a protein-rich product and a fibrous product, characterized in that the BSG in a wet state is passed through a roll mill to press said BSG with simultaneous grinding of the germ and the proteinaceous material, thereby separating said germ and proteinaceous material from said husks, and in that the BSG, after passage through the roll mill, is sieved one or more times, with at least one sieving being in water, whereby there is recovered a protein-rich product containing the germ and proteinaceous material as a minus fraction and the husks as a plus fraction.

2. A process according to Claim 1, wherein said pressing treatment is preceded by a preliminary sieving treatment to recover a fine particulate protein-containing product.

3. A process according to Claim 2, wherein said preliminary sieving treatment is carried out in water.

4. A process according to any preceding claim, wherein said sieving treatment following said pressing treatment is carried out so as to recover a fraction of fine particulate protein-containing product and a fraction of coarse particulate protein-containing product.

5. A process according to Claim 4, including the further step of extracting said fraction of a coarse particulate protein-containing product with a 0.05 N to 0.15 N alkaline aqueous solution at a temperature of 60-100 °C for a period of time of 10 to 40 minutes.

6. A process according to Claim 5, which additionally comprises adding an acid to said extracted fraction immediately after the extraction to adjust the pH thereof to 7-10 and to obtain a solid-containing mixture, separating the solids from said mixture, and adding an acid to the solid-free mixture to adjust the pH thereof to 2-5 to thereby precipitate a protein-rich product.

7. A process according to any preceding claim, which additionally comprises extracting protein-containing product recovered from the process with a solvent, to thereby separate said fraction into a defatted

protein and a vegetable oil.

## Patentansprüche

1. Verfahren zur Herstellung eines proteinreichen Produktes und eines faserigen Produktes aus Malztreber (BSG), das Keime, Drusen, und ein an den Drusen anhaftendes proteinhaltiges Material enthält, in dem die BSG in einem nassen Zustand gepresst werden und dann zu deren Trennung in ein proteinreiches Produkt und ein faseriges Produkt gesiebt werden, dadurch gekennzeichnet, dass die BSG in einem nassen Zustand durch eine Walzenmühle gebracht werden, um die BSG mit gleichzeitigem Mahlen der Keime und des proteinhaltigen Materials zu pressen, dabei die Keime und das proteinhaltige Material von den Drusen zu trennen, und dadurch, dass die BSG nach Durchbringung durch die Walzenmühle einmal oder mehrmals gesiebt werden, wobei wenigstens eine Siebung in Wasser stattfindet, wobei dort ein proteinreiches Produkt gewonnen wird, das die Keime und das proteinhaltige Material als einen Minusanteil und die Drusen als einen Plusanteil enthält.

2. Verfahren nach Anspruch 1, in dem die Pressbehandlung von einer vorbereitenden Siebbehandlung vorausgegangen wird, um ein feines, aus Teilchen bestehendes, proteinhaltiges Produkt zu gewinnen.

3. Verfahren nach Anspruch 2, in dem die vorbereitende Siebbehandlung in Wasser durchgeführt wird.

4. Verfahren nach einem der vorhergehenden Ansprüche, in dem die der Pressbehandlung folgende Siebbehandlung durchgeführt wird, um einen Anteil von feinem, aus Teilchen bestehendes, proteinhaltiges Produkt und einen Anteil von grobem, aus Teilchen bestehendes, proteinhaltiges Produkt zu gewinnen.

5. Verfahren nach Anspruch 4, das den weiteren Schritt einschliesst, den Anteil eines groben, aus Teilchen bestehenden, proteinhaltigen Produktes mit einer 0,05 N bis 0,15 N alkalischer wässriger Lösung bei einer Temperatur von 60-100 °C für eine Zeitdauer von 10 bis 40 Minuten zu entziehen.

6. Verfahren nach Anspruch 5, das zusätzlich umfasst, dem entzogenen Anteil nach der Entziehung sofort eine Säure zuzugeben, um dessen pH-Wert auf 7-10 einzustellen, und um eine Mischung zu erhalten, die Feststoffe enthält, die Feststoffe von der Mischung zu trennen, und eine Säure zu der von den Feststoffen befreiten Mischung zu geben, um dessen pH-Wert auf 2-5 einzustellen, um dabei ein proteinreiches Produkt auszufällen.

7. Verfahren nach einem der vorhergehenden Ansprüche, das zusätzlich umfasst, das von dem Verfahren gewonnene proteinhaltige Produkt mit einem Lösungsmittel zu entziehen, um dabei den Anteil in ein entfettetes Protein und ein Pflanzenöl zu trennen.

## Revendications

1. Procédé de production de produit riche en protéines et un produit fibreux à partir de la drêche de brasserie (BSG) contenant le germe, les cosses et une matière protéinique adhérant aux cosses, suivant lequel le BSG à l'état humide est comprimé et criblé pour le séparer en produit riche en protéines et produit fibreux, **caractérisé en ce que** le BSG à l'état humide est passé par un broyeur à rouleaux de façon à comprimer ledit BSG en broyant simultanément le germe et la matière protéinique du BSG, séparant ainsi le germe et la matière protéinique desdites cosses, et en ce que suite au passage par le broyeur à rouleaux le BSG est criblé une ou plusieurs fois, une opération de criblage étant effectuée dans l'eau, permettant la récupération d'un produit riche en protéines contenant le germe et la matière protéinique en fraction passant le crible et les cosses en fraction retenue par le crible.

2. Procédé selon la revendication 1, suivant lequel le traitement de compression est précédé d'un traitement préliminaire de criblage pour récupérer un produit contenant les protéines en particules fines.

3. Procédé selon la revendication 1, suivant lequel le traitement de criblage préliminaire est effectué dans l'eau.

4. Procédé selon l'une ou l'autre des revendications précédentes, dont le traitement de criblage suivant le traitement de compression est effectué de telle façon à récupérer une fraction de produit en particules fines contenant des protéines et une fraction de produit en grosses particules contenant des protéines.

5. Procédé selon la revendication 4, comportant en outre la phase d'extraction de ladite fraction de produit en grosses particules contenant des protéines par une solution alcaline aqueuse de 0,05 à 0,15 N à une température de 60°-100°C pendant 10 à 40 minutes.

6. Procédé selon la revendication 5, comportant en outre une addition d'acide à ladite fraction ainsi extraite immédiatement après son extraction pour en redresser le Ph à 7-10 et pour obtenir un mélange contenant des solides, suivie de la séparation des solides dudit mélange, et l'addition d'acide au mélange sans solides pour en ajuster le Ph à 2-5 pour en précipiter un produit riche en protéines.

7. Procédé selon tout revendication précédente, qui comporte en outre l'extraction de produit contenant des protéines récupéré du procédé avec un solvant, afin d'en séparer ladite fraction en protéine dégraissée et en huile végétale.

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