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(54) Title: ENHANCEMENT OF THE FERMENTABILITY OF CARBOHYDRATE SUBSTRATES BY CHROMATOGRAPHIC PURIFICATION

(57) Abstract: A process for the production of ethanol by fermentation, comprising the steps of: providing an aqueous feedstock comprising dissolved carbohydrates, dissolved salts, and dissolved amino acids; separating the feedstock by chromatography into a raffinate fraction enriched in the salts and a fermentation fraction enriched in the carbohydrates and the amino acids; and fermenting the fermentation fraction to convert the carbohydrate to ethanol. Preferably, the feedstock is a sucrose-containing stream obtained as a by-product in the extraction or purification of sucrose from plant sources.



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ENHANCEMENT OF THE FERMENTABILITY OF CARBOHYDRATE
SUBSTRATES BY CHROMATOGRAPHIC PURIFICATION

The present invention relates to methods for the production of ethanol by fermentation, and
5 in particular to methods for the provision of improved fermentation feed materials. -

Ethanol is gaining wide popularity as a fuel, particularly when mixed with petrol (gasoline)
to form a mixture known as gasohol. Automobiles can run on gasohol containing up to
about 10 volume percent ethanol without requiring engine modifications. Ethanol is also
10 widely used as a chemical solvent and as a raw material in the manufacture of drugs,
plastics, lacquers, polishes, perfumes and the like.

Ethanol is derived primarily from the fermentation of mash, usually from starch and/or
sugars of natural origin. Natural fermentation is capable of producing an alcohol/water
15 product mixture containing up to about 12 volume percent ethanol. Subsequently, the
ethanol is separated from the ethanol/water product mixture. Separation is carried out at
least initially by distillation (the ethanol/water system exhibits an azeotrope at 90%
ethanol).

20 US-A-5820687 describes a process for producing ethanol from cellulosic starting
materials, in particular rice straw. The cellulose is hydrolysed with sulfuric acid in two
stages, and the resulting solution containing acids and sugars is added to an ion exchange
separating unit containing a cross-linked polystyrene ion exchange resin. The sugar is
adsorbed onto the resin bed, and the acid is eluted as an acid stream containing less than
25 2% sugar. The thus-separated sugar is eluted and fermented.

WO95/17517 describes a process for the production of ethanol from commercial waste
streams containing cellulosic material. The process may comprise an ion exchange step to
remove heavy metal ions that can inhibit fermentation.

30

It would be desirable to provide feedstocks for ethanol production from low-cost sources,
wherein the feedstocks undergo fast fermentation and wherein the ease of separation of the
ethanol after fermentation is enhanced.

One source of carbohydrate for ethanol fermentation is the byproducts from sugar production. Sugar is produced from both sugar beet and sugar cane.

- 5 Sugar is extracted from sugar beet by diffusion. The conventional manufacturing process starts by cleaning the sugar beet with water and slicing it into thin slices called cossettes. The slicing machines work in a similar manner to a kitchen grater. The cossettes they produce have a "V" cross section, which ensures that the largest possible surface area is exposed to maximise the extraction of sugar. The cossettes are mixed with hot water at
10 around 70°C and the sugar passes from the plant cells into the surrounding water. Then the vegetable material is mechanically pressed to extract as much remaining sugar as possible. The pressed vegetable material is used to produce animal feed. The liquid resulting from the diffusion process is dark in colour and is called raw juice.
- 15 The raw juice is purified by carbonatation. This involves mixing the juice with milk of lime (aqueous calcium hydroxide) and adding carbon dioxide. During this process, the carbon dioxide and the milk of lime combine to produce calcium carbonate, which precipitates out taking most of the impurities from the raw juice with it. This precipitated lime contains important trace elements and is used as soil improving agent. The liquid
20 resulting from the carbonatation process is pale yellow and is called thin juice.

The thin juice, while much purer than the raw juice, is still relatively low in sugar content. Therefore the thin juice is concentrated by a multi-effect evaporation process, where water is boiled off to increase the solid content of the juice from about 15% in thin juice to about
25 65%. The resulting concentrated juice, known as thick juice, is passed through filters.

The thick juice is then purified by crystallisation. The crystallisation process conventionally takes place in vacuum pans, in which the thick juice is boiled at lower temperatures under vacuum. When the juice reaches a predetermined concentration it is
30 seeded with tiny sugar crystals, which provide the nucleus for larger crystals to form and grow. When the crystals reached the desired size, the process is stopped and the resultant mixture of sugar crystals and syrup is spun in centrifuges to separate the mixture. The sugar crystals are washed, dried and cooled to yield a white sugar.

Some sugar remains in the separated syrup. Thus, the crystallisation step is repeated with the separated syrup to produce so-called raw sugar. The process is then repeated a third time to produce so-called final sugar and molasses. The colour of the sugar produced
5 increases with each successive crystallisation step. The raw and final sugar of the second and third step are generally not clean enough to be sold and thus they are reintroduced into the purification process by re-dissolving into the thick juice. However, reintroducing the final sugar of the third step is often problematic as, due to the combination of temperature and long residence times in the crystallisation pans, it is often intensely coloured.

10

It is known to use molasses from the crystallisation of sugar in either the cane or the beet processes as a feedstock for subsequent fermentation processes. The molasses may undergo further treatments before fermentation is carried out. For example, JP-A-1168275 describes treating molasses by dilution, heating, adjusting the pH with a mineral acid, and
15 separation of a precipitated impurity fraction, prior to carrying out fermentation on the molasses. EP-A-0756011 describes carrying out a chromatographic step on molasses to separate it into a sugar-rich fraction and a low-sugar fraction, and then mixing the low-sugar fraction with further molasses to produce a fermentation feedstock. GB-A-2152057 describes a feedstock for the production of glutamic acid. The feedstock is produced from
20 molasses by the steps of inverting the molasses, followed by passing the inverted molasses through an ion exchange column to obtain a sugar-containing eluate fraction suitable for use as the feedstock.

The present invention provides a process for the production of ethanol by fermentation,
25 comprising the steps of: providing an aqueous feedstock comprising dissolved carbohydrates, dissolved salts, and dissolved amino acids; separating the feedstock by chromatography into a raffinate fraction enriched in the salts and a fermentation fraction enriched in the carbohydrates and the amino acids; and fermenting the fermentation fraction to convert the carbohydrate to ethanol.

30

Suitably, the aqueous feedstock is a by-product of sugar manufacture. That is to say, a by-product of the extraction and purification of sucrose from natural sources such as sugar cane and sugar beet. Preferably, the feedstock is a by-product of the extraction and

purification of sucrose from sugar beet. In such cases the aqueous feedstock frequently comprises dissolved betaine, and suitably (but not necessarily) the chromatographic separating step further produces a betaine fraction containing a major portion of the betaine.

5

The feedstock may be a thick juice as hereinbefore described, or it may be a liquor fraction from the first or second crystallisation stages, or it may be a molasses from a third crystallisation stage. These feedstocks contain, respectively, increasing amounts of coloured organic impurities and salts, which are thought to interfere with the production of ethanol by fermentation, and with the separation of ethanol after fermentation. Accordingly, the chromatography conditions are preferably selected such that these impurities are substantially absent from the second fraction produced in the chromatographic separation step.

15 Preferably, the feedstock used in the process of the present invention is the liquor from the second stage of sucrose crystallisation from thick juice, in particular thick beet juice. In these embodiments, the process of the present invention further comprises the steps of:

- (i) crystallising a sucrose thick juice to yield white sugar and syrup;
- (ii) crystallising the said syrup to yield raw sugar and a liquor;

20 wherein the liquor is used (optionally with dilution or concentration) as the aqueous feedstock for the chromatographic separation.

It is unusual to use only two crystallisation stages in the production of crystalline sucrose. However, it has been found that by limiting the number of successive crystallisation steps of the thick juice to two, preferably with recycle of the sugar from step (ii), the colour of the white sugar obtained by the first crystallisation step is improved. This is a further benefit of the processes according to the present invention.

In certain preferred processes according to the present invention, the feedstock has the following composition:

total dissolved solids from about 20 to about 70 wt %, preferably from about 40 to about 60wt %;

dissolved carbohydrate from about 50 to about 90 wt% on dry substance, preferably from about 55% to about 75 wt % on dry substance;

dissolved salts (or ash) from about 5 to about 25wt.% on dry substance, preferably from about 10% to about 20wt.% on dry substance;

5 nitrogenous compounds: from about 2 to about 15 wt.% on dry substance, preferably from about 10% to about 12wt.%; and

other organic impurities: from about 1% to about 10wt.% on dry substance, preferably from about 5% to about 8 wt.% on dry substance.

10 The dissolved carbohydrate preferably comprises a dissolved monosaccharide or disaccharide. Suitably, at least 90% of the dissolved carbohydrate is monosaccharide or disaccharide, and preferably it is sucrose. A particularly preferred feedstock is the liquor obtained from the second crystallisation step carried out on a sugar beet thick juice. The carbohydrate content of the feedstock is measured by HPLC using an Aminex 87K column
15 (BioRad Laboratories Inc) with refractive index detection

The dissolved salts present in the feedstock are detrimental to the fermentation of sucrose into ethanol, and removal of these salts in the chromatography step allows more rapid fermentation to higher ethanol concentrations. The wt.% salt (ash) values herein are as
20 measured after heating to 600°C in air for 4 hours. Approximate values may also be determined by measuring conductivity. The conductimetric salt measurement compares the conductivity of the solution with a standard salt solution (potassium chloride). This gives a good approximation of the total ash content.

25 The nitrogenous compounds in the feedstock may include, but are not limited to, betaine, amino acids and nucleosides. The term "nitrogenous compounds" does not include inorganic nitrates or ammonia. The principal amino acids present in the feedstock are Lysine, Alanine, Glycine, Valine, Serine, Leucine, Isoleucine, and Tyrosine. They are important nutrients for the fermentation of the carbohydrates to ethanol, and it is an
30 advantage of the present invention that the chromatography conditions are selected to substantially retain these nutrients, and preferably also other nutrients such as nucleosides (uridine, adenosine, guanosine) , in the fermentation fraction.

The other organic impurities in the feedstock include in particular higher molecular weight compounds and colour impurities, such as those found in molasses. These are preferably removed into the raffinate fraction.

- 5 Detailed discussion of the components of the byproduct streams from sugar manufacture that can be used in the practice of the present invention may be found for example in Sugar Technology – Beet and Cane Sugar Manufacture, Van der Poel, Schiweck & Schwartz, Bartens 1998, isbn 3-87040-065-x., the entire content of which is incorporated herein by reference.

10

As already noted, the fermentation fraction is enriched in the carbohydrate and amino acids. That is to say, the concentration of carbohydrate and amino acids in the fermentation fraction on a dry solids basis is higher than in the feedstock, preferably at least 20% higher, more preferably at least 50% higher. Suitably, a major fraction of the carbohydrates and amino acids in the feedstock are present in the fermentation fraction. By major fraction is meant more than 50% of these components originally present in the feedstock, preferably more than 75% of these components present in the feedstock. The concentration of these components in the fermentation fraction is preferably at least double the concentration of the components in the feedstock, more preferably at least three times the concentration of the components in the feedstock.. Conversely, the concentration of the salts and colour components in the fermentation fraction preferably is less than 50%, more preferably less than 25% of the concentration of these components in the feedstock.

The raffinate fraction is enriched in the salts. That is to say, the concentration of the salts in the raffinate fraction on a dry solids basis is higher than in the feedstock, preferably at least 20% higher, more preferably at least 50% higher. Suitably, a major fraction of the salts in the feedstock are present in the raffinate fraction. By major fraction is meant more than 50% of these components originally present in the feedstock, preferably more than 75% of these components present in the feedstock. The concentration of these components in the raffinate fraction is preferably at least double the concentration of the components in the feedstock, more preferably at least three times the concentration of the components in the feedstock.. Conversely, the concentration of the carbohydrates in the raffinate fraction

preferably is less than 50%, more preferably less than 25% of the concentration of these components in the feedstock.

For example, the fermentation fraction may have the following composition: total
5 dissolved solids from 15 to 30 wt %, preferably 20 –25 wt%; dissolved carbohydrate from
about 85 to about 98 wt.% on dry solids (DS), preferably from about 92 to about 96 wt.%
on DS; dissolved salts [or ash] from about 0.5 to about 5 wt.% on DS, preferably from
about 0.5 to about 2 wt.% on DS; amino acids from about 0.5 to about 5 wt.% on DS,
preferably from about 1 to about 3 wt.% on DS; other organic impurities from about 0.5 to
10 about 3 wt.% on DS, preferably from about 1 to about 2 wt.% on DS.

The application of chromatography to the separation of sugar containing streams is
described in a number of patent applications, including EP-A-0054544, EP-A-0345511,
WO96/10650, WO94/17213, and WO97/45185, the entire contents of which are
15 incorporated herein by reference and will not be discussed further. The chromatographic
separation may be carried out on an ion exchange resin (cationic or anionic), a size
exclusion resin, an affinity chromatography bed. Preferably, the separation is carried out
on a monovalent cation exchange resin (i.e. an anionic resin having a monovalent cation or
 H^+ initially bound thereto). Suitable resins are discussed in the above references.

20

The chromatographic separation step may conveniently be carried out by a simple batch
method, or by a simulated moving bed (SMB) method. The simulated moving bed method
may be sequential or continuous or comprise a combination of a continuous and a
sequential method. In a continuous simulated moving bed process, all fluid streams
25 typically flow continuously. The continuous simulated moving bed process has been
disclosed for example in US-A-2985589. The streams are: the supply of feed solution and
eluent, the circulating of the liquid mixture, and the withdrawal of products. The flow rate
for these flows may be adjusted in accordance with the separation goals (yield, purity,
capacity). Typically 8 to 20 partial packed beds are combined into a loop. The eluent and
30 feed supply and product withdrawal points are shifted cyclically in the downstream
direction in chromatographic bed. On account of the supply of eluent and feed solution, the
withdrawal of products, and the flow through the chromatographic bed, a dry solids profile
is formed in the chromatographic bed. Constituents having a lower migration rate in the

chromatographic material are concentrated in the back slope of the separation profile, i.e. dry solids profile, while constituents having a higher migration rate are concentrated in the front slope. The points of introduction of the feed solution and eluent and the withdrawal points of the product or products are shifted cyclically at substantially the same rate at which the dry solids profile moves in the chromatographic bed. The eluent and feed supply and product withdrawal points are shifted cyclically by using feed and product valves located along the chromatographic bed, typically at the upstream and downstream end of each partial packed bed. If product fractions of very high purity are desired, short cycle times and multiple partial packed beds must be employed, in which case the apparatus has the requisite valves and feed and withdrawal equipment.

In the sequential simulated moving bed process, some of the fluid streams do not flow continuously. The streams are: the supply of feed solution and eluent, the circulating of the liquid mixture, and the withdrawal of products (eluting phase; two to four or more products). The flow rate and the volumes of the different feeds and product fractions may be adjusted in accordance with the separation goals. The process commonly comprises three basic phases: feeding, elution and circulation. During the feeding phase, a feed solution, and possibly also an eluent during a simultaneous eluting phase, is introduced into predetermined partial packed beds, and simultaneously a product fraction or fractions are withdrawn. During the eluting phase, eluent is introduced into a predetermined partial packed bed or predetermined partial packed beds, and during these phases two, three or even four product fractions are withdrawn. During the circulating phase, no feed solution or eluent is supplied to the partial packed beds and no products are withdrawn.

The method of the present invention may comprise either continuous or sequential, or a combination of a continuous and a sequential process. Suitably, the SMB method comprises two consecutive SMB loops as described in EP-A-0764219. Suitably, the SMB method is adapted to produce three fractions as described in EP-A-0345511 and EP-A-0764219. In particularly preferred processes, the feedstock is a byproduct of the production of sucrose from sugar beet, and the SMB chromatographic separation divides the feedstock into a fermentation fraction containing the sucrose and amino acids, a raffinate fraction containing the salts and colour impurities, and a betaine fraction. SMB has advantages that include continuous or quasi-continuous operation and minimal dilution

of the product streams by eluant. Preferably, the total eluant added in the chromatographic separation is less than about 50% of the volume of the feedstock, more preferably less than about 15% of the feedstock. The chromatography thereby has the additional advantage of concentrating the sugars and nutrients such as amino acids and nucleosides in the
5 fermentation fraction.

In certain embodiments, the separating step of the present invention may comprise separating the feedstock into a separate sugar-rich fraction, in addition to said fermentation fraction and said raffinate fraction. This sugar fraction has low color and can be recycled
10 to the crystallisation stages of the process to increase effective sugar recovery. This also allows the ratio of sugar to amino acids in the fermentation fraction to be further optimised.

Specific embodiments of the present invention will now be described further, by way of example, with reference to the accompanying drawings, in which:

15

Fig. 1 shows a block diagram of the chromatographic separation process used in an embodiment of the invention; and

Fig. 2 shows a graph of the measured concentrations of various constituents against chromatography fraction number for separation of a beet sugar liquor on an ionic
20 chromatography resin in the potassium form.

Example 1

A sugar beet thick juice was prepared as described above. The juice was subjected to a
25 two-step crystallisation, with recycle of the crystalline sucrose from the second crystallisation to the first crystallisation stage. This gave a very white sugar from the first crystallisation, and a liquor from the second crystallisation containing residual sucrose and the various non-sucrose impurities including amino acids, salts, colour impurities and betaine. The sugar content of the liquor was about 75% on dry solids basis, and the total
30 solids concentration was about 70-80%

The liquor was fed to the two-stage chromatographic separation process shown schematically in Fig.1. The liquor F is fed initially to a homogenizer 1 where it is diluted

with a recycled dilute fraction 9,10 from the separators. The dilute fraction 9,10 consists mainly of water, together with some salts and sucrose to a total solids content of about 20%. The diluted feed stream 4, The sugar content of the liquor was about 75% on dry solids basis, and the total solids concentration was about 50-60%, is then fed to a SMB chromatographic separation system 2 constructed in accordance with EP-A-0345511 and comprising six columns filled with an a strongly acidic ion-exchange resin such as PUROLITE PCR 651 (available from the Purolite Company).

The output from the chromatographic separator 2 is shown schematically in Fig. 2. This shows the composition of samples 1 to 28 of a complete SMB cycle, as measured at point A shown schematically in Fig.1. Samples 1-3 and 27-28 are the raffinate fraction 6, consisting mainly of the colour impurities and salts. Samples 4-7 and 17-20 are complex mixtures that are recycled through a loop 11 internally within the SMB separation. Samples 8-10 are dilute fraction 10 (about 10% total dry solids), and are recycled to the homogenizer 1 as diluent for the liquor F. Samples 11-16 are the fermentation fraction 5 containing the major part of the sucrose. It has now been found that important nutrients including amino acids and nucleosides (Not shown in Fig. 2) run with the sucrose on the chromatography column, and are concentrated in the fermentation fraction 5. This reduces the amount of nutrients that need to be added in the fermentation step.

20

Finally, samples 21-26 of the separation profile shown in Fig.2 are the betaine fraction 12. This undergoes further purification in a second SMB separator 3 of six columns, as shown in Fig. 1 and as described in detail in EP-A-0764219, the entire content of which is incorporated herein by reference. The further separation results in a further dilute fraction 9 which is recycled to the homogenizer 1, a raffinate fraction 8 which may be combined with the raffinate fraction 6 from the first SMB loop, and a betaine fraction 7. The combined raffinate fractions 6,8 and the final betaine fraction 7 each contain only 18-19% sucrose on dry solids basis.

The fermentation fraction 5 contains about 95% sucrose on dry solids basis, and the concentration of the sucrose is about 23g/100g. The remainder of the solids in the fermentation fraction are mainly nutrients such as amino acids and nucleosides. This composition is very suitable for fermentation to ethanol.

The fermentation fraction gives improved fermentation performance compared to conventional molasses. The fermentation fraction contains a substantial amount of nutrients such as amino acids and nucleosides, whereby the fermentation can be carried out
5 substantially or completely without addition of nitrogenous nutrients to the fermentation fraction. Furthermore, higher gravity fermentations are possible and hence higher ethanol content can be achieved in the fermentation broth.

The above examples have been described for the purpose of illustration only. Many other
10 examples falling within the scope of the accompanying claims will be apparent to the skilled reader.

CLAIMS

1. A process for the production of ethanol by fermentation, comprising the steps of:
providing an aqueous feedstock comprising dissolved carbohydrates, dissolved
5 salts, and dissolved amino acids;
separating the feedstock by chromatography into a raffinate fraction enriched in the
salts and a fermentation fraction enriched in the carbohydrates and the amino acids; and
fermenting the fermentation fraction to convert the carbohydrates to ethanol.
- 10 2. A process according to claim 1, wherein the aqueous feedstock is a byproduct of
sugar manufacture.
3. A process according to claim 1 or 2, wherein the aqueous feedstock contains
dissolved betaine.
- 15 4. A process according to claim 3, wherein the chromatographic separating step
further produces a betaine fraction containing a major portion of the betaine.
5. A process according to any preceding claim, wherein the feedstock further
20 comprises coloured organic impurities, and a major portion of these colour impurities are
concentrated in the raffinate fraction produced in the chromatographic separation step.
6. A process according to any preceding claim, wherein the feedstock has the
following composition;
25 total dissolved solids from about 20 to about 70 wt %, preferably from about 40 to
60wt %;
dissolved carbohydrate from about 50 to about 90wt% on dry substance, preferably
from about 55 to about 75 wt % on dry substance;
dissolved salts (ash) from about 5 to about 25wt.%, on dry substance, preferably
30 from about 10 to about 20 wt.% on dry substance; and
nitrogenous compounds: from about 2 to about 15 wt.% on dry substance,
preferably from about 10 to about 12 wt.% on dry substance; and

other organic impurities: from about 1 to about 10 wt.% on dry substance, preferably from about 5 to about 8 wt.% on dry substance.

7. A process according to any preceding claim, wherein the fermentation fraction has
5 the following composition:

total dissolved solids from about 15 to about 30 wt % preferably about 20 to about 25 wt%;

dissolved carbohydrate from about 85 to about 98 wt.% on dry substance, preferably from about 92 to about 96 wt.% on dry substance;

10 dissolved salts [or ash] from about .5 to about 5 wt.% on dry substance, preferably from about 0.5 to about 2 wt.% on dry substance;

amino acids from about 0.5 to about 5 wt.% on dry substance, preferably from about 1 to about 3 wt.% on dry substance; and

other organic impurities from about 0.5 to about 3 wt.% on dry substance,
15 preferably from about 1 to about 2 wt.% on dry substance.

8. A process according to any preceding claim, wherein the chromatographic separation step is carried out by a simulated moving bed (SMB) method.

20 9. A process according to claim 8, wherein the SMB method comprises two consecutive SMB loops.

10. A process according to claim 8 or 9, wherein the feedstock is a byproduct of the production of sucrose from sugar beet, and the SMB chromatographic separation divides
25 the feedstock into a fermentation fraction containing the sucrose and amino acids, a raffinate fraction containing the salts and colour impurities, and a betaine fraction.

11. A process according to any preceding claim, further comprising the steps of:
(i) crystallising a sucrose thick juice to yield white sugar and syrup; and
30 (ii) crystallising the said syrup to yield raw sugar and a liquor;
wherein the said liquor is used (optionally with dilution or concentration) as the aqueous feedstock for the chromatographic separation.

12. A process according to any preceding claim, wherein the raffinate fraction contains a major fraction of the salts present in said feedstock and the fermentation fraction contains a major fraction of the carbohydrates and of the amino acids present in said
5 feedstock.

13. A process according to any preceding claim, wherein the step of separating comprises separating the feedstock into a sugar-rich fraction, in addition to said fermentation fraction and said raffinate fraction.
10

14. A process according to any preceding claim, wherein the fermentation is carried out substantially without addition of nitrogenous nutrients to the fermentation fraction.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB2005/002970

A. CLASSIFICATION OF SUBJECT MATTER IPC 7 C12P7/06 C13J1/06				
According to International Patent Classification (IPC) or to both national classification and IPC				
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC 7 C13J C12P				
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched				
Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal, WPI Data, BIOSIS, MEDLINE, EMBASE, CHEM ABS Data				
C. DOCUMENTS CONSIDERED TO BE RELEVANT				
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.		
Y	GB 1 209 562 A (AB CHEMATUR) 21 October 1970 (1970-10-21) page 1, left-hand column, line 9 - line 12 page 1, right-hand column, line 87 - page 2, left-hand column, line 1	1-14		
Y	EP 0 764 219 B (CULTOR OY; DANISCO FINLAND OY) 21 November 2001 (2001-11-21) cited in the application paragraph '0010!; examples	1-14		
A	WO 2004/002938 A (FINNFEEDS FINLAND OY; PAANANEN, HANNU; HEIKKILAE, HEIKKI; PUUPPO, OUTI) 8 January 2004 (2004-01-08) page 6, line 4 - line 21 page 10, line 4 - line 12 ----- -/--	1-14		
☒ Further documents are listed in the continuation of box C. ☒ Patent family members are listed in annex.				
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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB2005/002970

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