



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<p>(21) International Application Number: PCT/NL00/00165</p> <p>(22) International Filing Date: 10 March 2000 (10.03.00)</p> <p>(30) Priority Data: 99200796.3 15 March 1999 (15.03.99) EP</p> <p>(71) Applicant (<i>for all designated States except US</i>): COÖPERATIEVE VERKOOP- EN PRODUCTIEV- ERENIGING VAN AARDAPPELMEEL EN DERIVATEN AVEBE B.A. [NL/NL]; Beneden Oosterdiep 27, NL-9641 JA Veendam (NL).</p> <p>(72) Inventors; and (75) Inventors/Applicants (<i>for US only</i>): SIEPEL, Ugo [NL/NL]; Grote Vaartlaan 53, NL-9642 PB Veendam (NL). BUWALDA, Pieter, Lykle [NL/NL]; Mondriaanstraat 32, NL-9718 MJ Groningen (NL).</p> <p>(74) Agent: OTTEVANGERS, S., U.; Vereenidge Octrooibureaux, Nieuwe Parklaan 97, NL-2587 BN The Hague (NL).</p>	<p>(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b> <i>With international search report.</i></p>	
<p>(54) Title: INGREDIENTS FOR EXPANDED FOODS</p>		
<p>(57) Abstract</p> <p>The invention relates to a composition for use in preparing an expanded foodstuff such as snacks, said composition comprising at least a non-cereal amylopectin starch. It is a further object of the present invention to provide a method for obtaining an expanded foodstuff having improved expansion characteristics. Said composition, such as dough, at least comprising a non-cereal amylopectin starch, such as that isolated from potato tubers or tapioca. The preparation is carried out by heating at least part of the composition to a temperature above its glass transition temperature and letting it cool to below said glass transition temperature.</p>		

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Title: Ingredients for expanded foods.

The invention relates to expansion of foodstuff, in particular to the production of expanded food items such as snacks.

Expanded foods are well-known to every consumer. Their highly aerated texture is appreciated in toast, snacks, crackers and a multitude of other products that are often eaten snack wise. In general, expansion of a certain product is achieved by heating a composition such as a dough to a temperature above its glass transition temperature and then expanding it, for example by applying a gas which blows the plasticised mass to a foam. This foam is subsequently brought to below its glass transition temperature whereby the expanded dough settles resulting in a stable, glassy or crispy and expanded foam.

Perhaps the best known example of such an expanded product is popcorn. The corn is heated well above the glass transition temperature of the starch. At this temperature water boils vigorously and the resulting steam blows the corn kernel to a foam (the popping). Since water is a very efficient plasticiser for starches, the release of water results in a sharp increase in the glass transition temperature. As a result the popcorn is frozen into its well-appreciated tender and crispy form.

Apart from flavour, texture is the second most important criterion in determining the acceptability and attractiveness of a food product. For many products the crispiness and extent of expansion mainly govern the sensation of texture. Examples of expanded products are extruded snacks, crackers, cookies, coated nuts, Japanese style snacks, some types of confectionery, dry roasted nuts, chip-like products, etc. In general, such snacks are best appreciated when they are most expanded, i.e. contain the most air and are most brittle. Often such snacks are also described as being light and crispy.

As already mentioned glass transition temperature and the influence of water on this temperature can govern the expansion. All starches have their own typical glass transition

at a fixed moisture content as is described in J-.L. Jane et al, "Effects of starch chemical structures on gelatinisation and pasting properties" in *Zywnosc Technologiczna* Jakosc, 4(17) 63-71, Cracow 1998. Apart from these factors the viscosity and the visco-elasticity are also important factors in governing the foaming properties of plasticised materials.

Degrees of crispiness and expansion are often achieved by varying process conditions and formula aspects. For example in snack preparations often a mixture of starches, flour and a (most times limited) amount of water is prepared. A process step is carried out such as extrusion, sheeting or coating in order to bring the dry or semi-dry mix, dough or batter into a certain shape or condition. Final process steps can be drying, baking or frying, through which expansion is obtained as described above.

Yet another typical example of expanded foodstuff are puffed snacks. Farinaceous materials are formed into pellets with a distinct shape. Heating the pellets for instance through frying or with a puffing gun results in the desired snack. Also, fabricated corn or potato chips are an important factor in the market. In U.S. Patent 3,576,647 a process is described in which the preparation of chip-like expanded products is described. In U.S. Patent 5,500,240 the application of pregelatinised waxy maize starch for the production of (potato) chip like products is described. The machinability of the transient dough is rather good, resulting in low fat chip-like products. No remarks with respect to expansion are made. However, the application of cereal starches in potato or other non-cereal snacks may lead to off taste products.

For foodstuff, it is in general desirable that a starch be bland or neutral in flavour. The starches generally having the most neutral taste are non-cereal, such as tuber- or root-type, starches, such as potato or tapioca, when compared to starches such as corn, wheat, rice, sorghum, waxy maize and waxy sorghum, which, when incorporated into food, give some undesirable flavour (peculiar to the starch) to the food. These off-flavours have been described by some individuals as 'woody', 'corny', 'starchey', 'bitey' or 'chalkey', and these

flavours often come out most poignant after heat treatment, amongst others because cereal starches contain considerable amounts of lipids and proteins, when compared to non-cereal starches.

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It is an object of the present invention to provide expanded foodstuff, and methods of obtaining these, having improved expansion characteristics, containing more air or being even more light and/or crispy over comparable expanded foodstuffs that have been traditionally on the market. It is a further object of the present invention to provide expanded foodstuff wherein the starch used has a unpronounced or neutral taste.

The invention provides a method for obtaining an expanded foodstuff having improved expansion characteristics comprising preparing a composition, such as a dough, coating mix, pre-mix, etc, at least comprising a non-cereal amylopectin starch, heating at least part of said composition to a temperature above its glass transition temperature, i.e. expanding said heated composition and letting it cool to below said glass transition temperature. As referred to herein, non-cereal amylopectin starch is a starch isolated from non-cereals, such as potato tubers or tapioca and having an amylopectin content typically more than 90 wt.%, preferably of at least 95 wt.%, and preferably at least 98 wt.%, or even at least 99 wt.%, based on dry substance.

Due the presence of the non-cereal amylopectin starch, preferably a starch having an amylopectin content of at least 90 weight percent based on dry substance of said starch in said composition, an improved expansion is obtained. In a preferred embodiment, a method according to the invention is provided wherein said starch is derived from a potato.

The invention also provides a composition for use in preparing an expanded foodstuff, said composition at least comprising a non-cereal amylopectin starch. Such a composition is for example a dough, (tempura or fritter) batter, mix or ready-to-use pre-mix to which for example only water need to be added for preparing a dough or batter, said composition

optionally containing other starches, carbohydrates, fats, proteins, flavours, salts, or other food components. In a preferred embodiment a composition according to the invention is provided wherein said non-cereal amylopectine starch has an amylopectin content of at least 90 weight percent based on dry substance. It is preferred that at least 10%, more preferably at least 25% of the starch fraction in said expanded products comprises non-cereal amylopectine starch, be it native non-cereal amylopectin starch or a non-cereal amylopectin starch derivative, to provide for the expansion characteristics desired for the type of expanded food product.

The invention also provides an expanded foodstuff comprising a non-cereal amylopectin starch. Examples of such foodstuff as provided by the invention are extruded snacks, crackers, cookies, doughnuts, chip-like products, Japanese style snacks, some types of confectionery, or coated foodstuff such as coated nuts, dry roasted nuts, fritter or tempura type products, etc., or food coatings, such as snack coatings, bread coatings, French fries coatings, and pre-mixes therefor, comprising a non-cereal amylopectin starch, particularly those comprising starch or starch granules containing more than 90 or 95%, and usually more than 98% of amylopectin.

The present invention in particular relates to expanded products in which amylopectin starch or derivatives thereof are applied as to impart expansion properties. It has been provided by the invention that use of non-cereal amylopectin starch, and non-cereal amylopectin starch derivatives for the preparation of expanded foodstuffs induces unexpected high expansion when compared to other starches. Most starches typically consist of granules in which two types of glucose polymers are present. These are amylose (15-35 wt.% on dry substance) and amylopectin (65-85 wt.% on dry substance). Amylose consists of unbranched or slightly branched molecules having an average degree of polymerisation of 100 to 5000, depending on the starch type. Amylopectin consists of very large, highly branched molecules having an average degree of polymerisation of 1,000,000 or more. The commercially most important starch types (maize starch, potato starch, wheat starch and tapioca starch) contain 15 to 30 wt.% amylose. For some cereal types, such as barley,

maize, millet, wheat, milo, rice and sorghum, varieties are known of which the starch granules nearly completely consist of amylopectin. Calculated as weight percent on dry substance (wt.%), these starch granules contain more than 95%, and usually more than 98% of amylopectin. The amylose content of these cereal starch granules is therefor less than 5%, and usually less than 2%. The above cereal varieties are also referred to as waxy cereal grains, and the amylopectin starch granules isolated therefrom as waxy cereal starches.

In contrast with the different cereals, starch granules of non-cereal starches, such as root and tuber varieties, that (nearly) exclusively consist of amylopectin are traditionally not known in nature. For instance, potato starch granules isolated from potato tubers usually contain about 20% amylose and 80 % amylopectin. During the past 10 years, however, successful efforts have been made to cultivate by genetic modification non-cereals, such as potato plants which, for example in the potato tubers, form starch granules consisting for more than 95 wt.% of amylopectin. It has even been found feasible to produce potato tubers comprising substantially only amylopectin.

In the formation of starch granules, different enzymes are catalytically active. Of these enzymes, the granule bound starch synthase (GBSS) is involved in the formation of amylose. The presence of the GBSS enzyme depends an the activity of genes encoding for said GBSS enzyme. Elimination or inhibition of the expression of these specific genes results in the production of the GBSS enzyme being prevented or limited. The elimination of these genes can be realised by genetic modification of potato plant material or by selection for plants having a recessive mutation, preferably in homozygous form, of said gene. An example of the latter is the amylose-free mutant of the potato (amf) of which the starch substantially only contains amylopectin through a recessive mutation in the GBSS gene. This mutation technique is described in, inter alia, J.H.M. Hovenkamp-Hermelink et al., "Isolation of amylose-free starch mutant of the potato (*Solanum tuberosum* L.)", *Theor. Appl. Gent.*, (1987), 75:217-221" and E. Jacobsen et. al., "Introduction of an amylose-free (amf), mutant- into

breeding of cultivated potato, *Solanum tuberosum* L.,  
Euphytica, (1991), : 53:247-253.

Elimination or inhibition of the expression of the GBSS  
gene in the plant is also possible by using so-called antisense  
5 inhibition. Genetic modification of for example potato is  
described in R.G.F. Visser et al., "Inhibition of the  
expression of the gene for granule-bound starch synthase in  
potato by antisense constructs", Mol. Gen. Genet., (1991),  
225:289-296. By using genetic modification, it has been found  
10 possible to cultivate and breed non-cereal roots and tubers,  
for instance potato, yam, or cassava (Patent South Africa  
97/4383), of which the starch granules. contain little or no  
amylose. As referred to herein, non-cereal amylopectin starch  
is the starch isolated from non-cereals such as potato tubers  
15 or tapioca and having an amylopectin content typically more  
than 90 wt.%, preferably of at least 95 wt.%, and preferably at  
least 98 wt.%, or even at least 99 wt.%, based on dry  
substance.

Regarding production possibilities and properties, there  
20 are significant differences between amylopectin potato starch  
on the one hand, and the waxy cereal starches on the other  
hand. This particularly applies to waxy maize starch, which is  
commercially by far the most important waxy cereal starch. The  
cultivation of waxy maize, suitable for the production of waxy  
25 maize starch is not commercially feasible in countries having a  
cold or temperate climate, such as The Netherlands, Belgium,  
England, Germany, Poland, Sweden and Denmark. The climate in  
these countries, however, is suitable for the cultivation of  
potatoes. Tapioca starch, obtained from cassava, may be  
30 produced in countries having a warm and moist climate, such as  
is found in regions of South East Asia and South America.  
The composition and properties of root and tuber starch, such  
as amylopectin potato starch and amylopectin tapioca starch,  
differs from those of the waxy cereal starches. Amylopectin  
35 potato starch, for example has a much lower content of lipids  
and proteins than the waxy cereal starches. Problems regarding  
off taste, odour and foaming, which, because of the lipids  
and/or proteins, may occur when using waxy cereal starch  
products (native and modified), do not occur, or occur to a

much lesser degree when using corresponding amylopectin potato starch products.

Furthermore, in contrast to the waxy cereal starches, amylopectin potato starch contains chemically bound phosphate groups. As a result, amylopectin potato starch products in a dissolved state have a distinct polyelectrolyte character. In the present invention non-cereal amylopectin starch and non-cereal amylopectin starch derivatives are applied in expanded food products. In relation to the same products based on other starches, the products based on non-cereal amylopectin starch show very good expansion properties. In some cases unmodified non-cereal amylopectin starch, such as for example derived from potato, even outperforms traditionally applied modified waxy cereal starch derivatives.

In one embodiment of the present invention unmodified, native non-cereal amylopectin starch, preferably derived from potato, is used in expanded products. The unmodified starch may or may not be pregelatinised by drum drying, spray cooking, spray drying, extrusion or heating in aqueous alcohol.

In a further embodiment the application of non-cereal amylopectin starch derivatives in expanded products is provided. The starch derivative may or may not be a cold water swellable derivative obtained using the methods described above.

The starch derivative may be a cross-linked starch, wherein said cross-linking is achieved by using sodium trimetaphosphate, phosphorus oxytrichloride or adipic anhydride, using for example a method known in the art. These cross-linking agents are most suitable for use in the food industry but others can also be contemplated.

The starch may be stabilised by treatment with acetic anhydride, vinyl acetate, or comparable agents. The starch may be stabilised by hydroxypropylation. Stabilisation by hydroxyalkylation of starch is, for example obtained with reagents containing a halohydrin, or an epoxide group as reactive site. The addition of hydroxypropyl groups is generally performed in aqueous suspensions of starch using propylene oxide, under alkaline conditions. The starch may also be derivatised by a combination of cross-linking and

stabilisation. Cross-bonding and/or stabilising reagents are in general reacted with starch under alkaline conditions. Suitable alkali materials are: sodium hydroxide, potassium hydroxide, ammonium hydroxide, magnesium hydroxide, sodium carbonate and trisodiumphosphate. Preferred are the alkali metal hydroxides and carbonates, most preferred are sodium hydroxide and sodium carbonate. Sometimes salts are added as to prevent swelling under alkaline reaction conditions. Preferred are sodium chloride and sodium sulphate.

The invention is further described in the detailed description and the examples therein without limiting the invention.

#### Detailed description

#### Example 1

#### Method for nut coating

#### Preparation of dry mix

A mixture is prepared of a native starch, here 400 g native potato starch, and a cold water swellable (pregelatinised) starch, here 200 g cold water swellable adipate acetate based on waxy maize starch. Herewith a dough coating is prepared of:

400 g starch mixture

80 g powdered sugar

10 g salt

10 g glutamate

water

#### Processing

Coat 400 g shelled peanuts in an Erweka AR 400 rotating coating pan (30 rpm, inclination of about 30°)

## Pre-coating

During this experiment the nuts are kept rotating. 8 g of water is sprayed on the nuts. The nuts are dusted with a malto  
5 dextrin such as AVEBE's Paselli SA 2, Paselli MD 10, Paselli MD6 and again 10 g of water are sprayed on. Immediately afterwards 65 g of coating mix is dusted on the nuts.

## Main coating

10

The main coating is performed in several steps according to a scheme for example as below.

step	spray with water (in g)	dust with coating mix (in g)
1	15	25
2	15	40
3	20	60
4	10	40
5	10	40
6	20	40
7	10	50
8	20	40

## 15 Final coating and processing steps

The nuts are dusted with 15 g of coating mix. Rotating is maintained for about 10 minutes.

## 20 Frying

The coated nuts were fried in oil at 155-160 °C for 5 minutes.

The cooled nuts were evaluated for organoleptic features. The  
25 volume of 200 g of coated nuts was measured in 1 L cylinder. The results are summarised in table 1, where example 1 is compared with examples 2 - 4.

## Example 2

In example 2 potato starch was exchanged for amylopectin potato starch. The results are summarised in table 1.

## Example 3.

Example 3 is a repetition of example 2 except that the cold swellable adipate/acetate based on waxy maize starch has been replaced by a none modified cold swellable waxy maize starch. The results are summarised in table 1.

## Example 4.

Example 4 is a repetition of example 3 except that the cold swellable waxy maize starch has been replaced by a cold swellable amylopectin potato starch. The results are summarised in table 1.

Table 1.

Example nr	native starch	cold swellable starch		Crisp	Expansion	Volume ml
		starch	modification			
1	PS	WMS	Adipate/acetate	4	6	550
2	APS	WMS	Adipate/acetate	6	7	620
3	APS	WMS	none	5	6	520
4	APS	APS	none	8	8	650

Several conclusions can be derived from examples 1 - 4 and Table 1.

- changing the native starch from normal potato starch to amylopectin potato starch improves expansion and crispiness.

- upon changing the cold water swellable starch from modified waxy maize starch to unmodified waxy maize starch expansion and crispiness go down
- changing the cold water swellable starch from unmodified or  
5 modified waxy maize to unmodified amylopectin potato starch improves the expansion and crispiness.
- it is possible to avoid the off-taste brought in by the waxy maize starch by replacing it with a non-cereal amylopectin starch.

## 10 Example 5

### Method

A dough was prepared of 14.4 kg of whole potato flakes (Rixona, German flakes), 5.80 kg of pregelatinised potato starch (with a  
15 moisture content of 9.3%), small components (0.39 kg of salt, 0.28 kg of sodium bicarbonate, 0.11 kg of citric acid, 0.14 kg of acid sodium pyrophosphate, 0.19 kg of sunflower oil, 0.11 kg lecithin) and 14.5 kg of water. The blend of dry ingredients is mixed for 30 seconds on low speed (52 rpm) in a high speed  
20 mixer. Oil and lecithin are added and mixed in for 2 minutes at high speed (104 rpm). Water is added and mixing is continued for 2.5 minutes. The dough is left to rest for 60 minutes. A biscuit line is fed with dough and the thickness of the dough is brought down to 0.6 cm. Round pieces are cut and dockered.  
25 The products are baked in a continuous oven with two zones (front: 215 °C, back: 185 °C) for 2.8 minutes.

The final products were evaluated for expansion, hardness in bite and crispiness. The results are summarised in table 2.

### 30 Expansion measurement

The weight of the amount used to fill a 2 l measuring cylinder with baked snacks was determined. The results were expressed as the volume which is occupied by 200 g.

Example 6.

In example 6 the instant starch was derived from waxy maize  
5 starch. It proved very hard to prepare a workable dough from  
this starch. The results are summarised in table 2. It show  
clearly that waxy maize starch induces less expansion in this  
application.

10 Example 7.

In example 7 the instant starch is a medium cross-linked potato  
starch derivatives. This example clearly shows that regular  
potato starch derivatives give low expansion values. The  
15 product is very hard also, which leads to a low appreciation.

Example 8.

In example 8 the instant starch is a low cross-bonded (STMP)  
20 amylopectin potato starch derivative. The results clearly show  
that this product has very good expansion characteristics and  
good crispiness.

Example 9.

25 In example 9 the instant starch is a medium cross-bonded  
amylopectin potato starch adipate/acetate. It can be seen that  
the food characteristics are still satisfactorily but less than  
the product from example 8.

30

Example 10.

In example 10 the starch derivative dosage is raised 50 % as  
35 compared to example. The resulting product has very good  
characteristics.

Example 11.

Example 11 is a repetition of example 7 except that a 40 %  
 extra dosage of water has been added. This example shows that  
 5 it is possible to obtain higher expansion values with regular  
 starch derivatives, although the expansion is still not as high  
 as with amylopectin potato starch derivatives and the final  
 product is harder. A 40 % extra dosage of water is however in  
 most cases not appreciated. When frying follows extrusion the  
 10 fat will deteriorate more rapidly and in other processes more  
 energy has to be applied in order to get the same dry crispy  
 product.

Table 2.

Example nr	5	6	7	8	9	10	11
instant starch	APS	WMS	PS	APS	APS	APS	PS
modification	none	none	crossl.	crossl.	crossl. stab	crossl. stab.	crossl.
Level			medium	low	medium	medium	medium
Reagent			STMP	STMP	adip/ac	adip/ac	STMP
						*	**
Expansion ml	2410	1880	1610	2530	2170	2550	2100
hardness	2	2	4.5	2	2	2	3.5
crispiness	5	5	1	5	5	5	5

15 STMP= sodium trimetaphosphate, adip/ac = adipate/acetate

\*= 1,5 fold dosage of starch, \*\*= 40 % extra water dosage

CLAIMS

1. A method for obtaining an expanded foodstuff having improved expansion characteristics comprising preparing a composition at least comprising a non-cereal amylopectin starch, heating at least part of said composition to a  
5 temperature above its glass transition temperature and letting it cool to below said glass transition temperature.
2. A method according to claim 1 wherein said composition is a dough.
3. A method according to claim 1 or 2 wherein said starch  
10 has an amylopectin content of at least 90 weight percent based on dry substance.
4. A method according to anyone of claims 1 to 3 wherein said starch is derived from a potato.
5. A composition for use in preparing an expanded  
15 foodstuff, said composition at least comprising a non-cereal amylopectin starch.
6. A composition according to claim 5 wherein said starch has an amylopectin content of at least 95 weight percent based on dry substance.
- 20 7. A composition according to claim 5 or 6 wherein said starch is derived from a potato.
8. A composition according to anyone of claims 5 to 7 wherein said starch is cross-linked.
9. A composition according to anyone of claims 5 to 8  
25 wherein said starch is stabilised.
10. An expanded foodstuff at least comprising a non-cereal amylopectin starch.
11. An expanded foodstuff according to claim 10 wherein said starch has an amylopectin content of at least 95 weight  
30 percent based on dry substance.
12. An expanded foodstuff according to claim 10 or 11 wherein said starch is derived from a potato.
13. Use of a non-cereal amylopectin starch for the preparation of an expanded foodstuff.

14. Use according to claim 13 wherein said starch is modified.
15. Use according to claim 13 or 14 wherein said foodstuff is a snack.
- 5 16. Use according to anyone of claims 13 to 15 wherein said foodstuff comprises a coating.

INTERNATIONAL SEARCH REPORT

International application No  
PCT/NL 00/00165

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7 A23L1/0522 A23L1/164 A23L1/217

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 A23L

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)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4 634 596 A (EASTMAN JAMES E.) 6 January 1987 (1987-01-06)	1, 5, 10, 13
A	HOVENKAMP-HERMELINK J H M ET AL: "ISOLATION OF AN AMYLOSE-FREE STARCH MUTANT OF THE POTATO (SOLANUM TUBEROSUM L.)" THEORETICAL AND APPLIED GENETICS, vol. 75, no. 1, 1 December 1987 (1987-12-01), pages 217-221, XP000610709 ISSN: 0040-5752 cited in the application	
A	EP 0 565 386 A (UNILEVER PLC.) 13 October 1993 (1993-10-13)	5, 10, 13
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Further documents are listed in the continuation of box C.  Patent family members are listed in annex.

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Date of the actual completion of the international search <b>5 May 2000</b>	Date of mailing of the international search report <b>12/05/2000</b>
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