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(54) Title: FEEDSTUFFS ADDITIVES CONTAINING L-LYSINE WITH IMPROVED ABRASION RESISTANCE, AND PROCESS FOR THEIR PRODUCTION

(57) Abstract: The invention relates to a shaped, in particular granulated feedstuffs additive containing L-lysine and treated with additives, preferably oils, having improved abrasion resistance, which optionally contains constituents from the fermentation broth and biomass, and a process for the production of this product.

Feedstuffs Additives containing L-lysine with Improved Abrasion Resistance, and Process for their Production

Subject Matter of the Invention

The invention relates to a shaped, in particular granulated feedstuffs additive containing L-lysine and treated with additives, preferably oils, having an improved abrasion resistance, which optionally contains constituents from the fermentation broth and biomass, and a process for the production of this product.

Prior Art

An animal feedstuffs additive based on fermentation broth is known from EP 0 809 940 B1 (US 5,840,358), which is obtained in granulated form in a fluidised bed.

This product represents a significant advance as regards its properties compared to the products known at that time.

However, certain problems still remain as regards the dust formed by abrasion during the further processing. In order to ensure that feedstuffs additives containing carrier materials are free of dust, it was proposed to spray a mineral oil-containing aerosol in an amount of 0.25 to 2 wt.% onto dust-containing material, 75% of which has a particle size of < 400 µ (GB 2 293 304 A).

An agglomeration of the powder is thereby achieved, which then consists in an amount of at least 75% of particles having a size of 400 to 1000 µ.

An essential constituent of these mixtures are inorganic carrier materials, which may comprise up to 96 % of the total.

The above specification does not give any details of the dust formed after transportation and storage.

The active substances that are used in the specification are pure substances that do not contain any constituents from a fermentation broth.

Object of the Invention

It is known that there is a need for granulated products containing L-lysine such as occur when using fermentation broths, that have an improved handling ability. This concerns in particular an improvement in the abrasion resistance, which is manifested in a reduced dust formation after transportation, storage in silos, and processing in the feedstuffs-producing operation, for example using conveyor belts.

Description of the Invention

The invention provides substantially dust-free shaped animal feedstuffs additives having improved abrasion resistance, based on fermentation broth and containing L-lysine and preferably the major proportion of the further constituents of the fermentation broth, the biomass produced during the fermentation being contained in an amount of ≥ 0 to 100%, characterised in that the animal feedstuffs additives

1.1. contain L-lysine in a concentration of 30 to 90 wt.%, in particular 40 to 70 %, referred to the total amount,

1.2 preferably ≥ 97 wt.% of the animal feedstuffs additive, in particular ≥ 98 % of the feedstuffs additive has a mean particle size between > 0.1 and 1.8 mm, and

1.3 contain on the surface an added additive in an amount of 0.02 to 2.0 wt.%, in particular 0.02 to 1.0 wt.%, referred to the total amount of the feedstuffs additive.

A proportion of 0.2 to 1.0 wt.%, in particular 0.2 to 0.6 wt.%, is particularly preferred.

The expression "based on fermentation broth" means that the starting material is a broth containing L-lysine, produced in a known way by fermentation (e.g. EP 0 533 039 B1). This contains in general as a further essential constituent the biomass formed during the fermentation, consisting of the amino acid-forming microorganisms.

This broth can be separated before the shaping stage, but is generally contained in an amount of up to 100 % in the shaped or granulated product.

The expression "on the surface" implies that the added liquid diffuses, even if only to a slight extent, into regions beneath the surface.

"Substantially dust-free" means that the proportion of particles of size < 100 µm is in the range > 0 up to 1 %, preferably up to 0.5 wt.%, and that the dust value is from 0.1 to 5.6, in particular 0.3 to 2.5.

More than 97 wt.% of the product, in particular ≥ 98 % and particularly preferably 99 % of the feedstuffs additive has a mean particle size preferably between 0.1 and 1.8 mm.

Further preferred constituents of the animal feedstuffs additive (referred to the total amount) include, in addition to the optionally contained biomass derived from the fermentation broth:

1. L-lysine in an amount of 30 to 90 wt.%, in particular 40 to 70 wt.%; if the L-lysine content produced in the fermentation is not sufficient, the desired value is adjusted by adding for example L-lysine HCl.

2. Protein content of 0.5 to 20 %, in particular 0.5 to 10 %, particularly preferably 0.5 to 7 %.

3. Content of carboxylic acid that is originally contained in the product of at most 13 %, carboxylic acids with fewer than 8 carbon atoms making up at most 10 % of the weight.

4. Fats and oils from the original product (optionally biomass and dissolved fractions from the fermentation broth), making up at most 6% of the weight.

The product does not contain inorganic carrier materials such as are described for example in GB 2 293 304 A.

Granules produced according to EP 0 809 940 B1 constitute preferred starting products for the feedstuffs additives that can be produced according to the invention.

In particular the granules have a mean particle size between 0.1 and 1.8 mm (≥ 97 %), preferably ≥ 95 wt.% of the particles having a particle size in the range between 0.3 and 1.8 mm. In a particularly preferred variant the particle size is in the range between 0.3 and 1.5 mm (≥ 95 %).

These granules are preferably obtained by spraying fermentation broths containing L-lysine in fluidised bed granulation drying units.

However, granules or shaped animal feedstuffs additives produced in another way and that contain lysine may also be used (e.g. EP 0 615 693 B1).

The dust fraction (particles < 100 μ) of the starting material is ideally < 3 wt.%. This value should not however be regarded as critical. Amounts of for example up to 10 wt.% may also be used without very fine dust having to be separated beforehand. Since very fine dust amounts of < 3 % are very difficult to determine gravimetrically, an optical test was developed.

Analysis method for the optical detection of the dust content

50 g of the product fall 0.8 metre in a closed chamber having the dimensions 320(W) x 210(D) x 950(H) mm. When the product falls to the floor the dust whirls up into the air space. The attenuation of the light beam in the head space of the apparatus is measured as a function of time, and the maximum value (%) is recorded. A halogen lamp (not a monochromatic light source) serves as light source. This measurement may be repeated after 30 seconds to obtain the 30-second value.

At values of less than 5 the product appears to be virtually optically dust free. If the dust value is above 20, the very fine dust fraction in the product can also be determined in a simple manner gravimetrically according to the prior art.

The dust content of the untreated product drastically increases under mechanical stress, such as occurs for example when the product is conveyed. In the case of pneumatic transportation over 40 to 120 m the untreated product may have a 100 % greater dust fraction and a dust value that is three times as large compared to the treated product (see Table 1). These figures also apply to other types of mechanical conveyance (see Fig. 1).

The values are those that were measured after combining the product with the finely particulate fractions that are conventionally trapped in filters. The additionally recorded dust fraction consists especially of 10 to ca. 90 µm, in particular 10 to ca. 50 µm large, granule fragments that have flaked off, and is identical as regards composition to the desired product. The product according to the invention is preferably substantially round and is compact in appearance.

The very fine dust fraction (< 100 µ) after conveyance over 40, 80 or 120 m by various methods (dense flow, strand, pneumatic) is < 1 wt.%, preferably < 0.5 wt.%, if products according to the invention are used having a dust value of ≤ ca. 1.

Dust values of ≤ ca. 11, preferably ≤ ca. 6, are found in particular with conveying lengths of 40 or 80 m.

In dense flow conveyance air speeds of 1.5 to 4 m/sec and product/air ratios of 30 to 80 are generally employed. In strand conveyance the ranges are 5 to 15 m/sec and a product/air ratio of 6 to 20. The values for pneumatic conveyance are 17 to 30 m/sec and a product/air ratio of 1 to 5.5.

The constituents of a feedstuffs additive based on fermentation broth are known *per se* to the person skilled in the art.

These constituents on the one hand optionally comprise the total amount or parts of the biomass (≥ 0) that is formed during the fermentation. Alternatively the fermentation broth contains, apart from L-lysine, dissolved constituents that are derived on the one hand from the nutrient medium, or also contains compounds separated from the micro-organism that is employed.

The bulk density of the shaped, in particular granulated feedstuffs additive is generally in the range from 600 to 900 kg/m^3, in particular 650 to 850 kg/m^3.

After the addition according to the invention of the aforementioned additives in an amount of 0.1 to 2.0 %, an increase in the bulk density was observed (see Table 5). Mineral oils, vegetable oils or mixtures of vegetable oils may be used as oils (additives). These include in particular oils that are liquid at room temperature, such as soybean oil, olive oil, soybean oil/lecithin mixtures

or other edible oils, as long as they do not alter the character of the animal feedstuffs additives.

Silicone oils, polyethylene glycols or hydroxyethylcellulose in aqueous solution are also suitable as additives.

Oil-water emulsions are also suitable in order to obtain the product according to the invention.

Furthermore complex by-products obtained from the sugar and starch industry such as corn steep liquor may be used as additives, and in particular also oily by-products, for example in the form of phospholipid fractions, formed in the extraction and hydrolysis of maize starch.

The liquid treatment agent is applied to the particles at a temperature of 10 to 100°C, preferably 20 to 60°C.

Although the feedstuffs additive to be treated has, due to its derivation from the fermentation, an analysable oil fraction, only the surface treatment with the aforementioned additives, in particular oils, leads to the improved abrasion resistance of the product.

It is also possible to distinguish clearly the treated product from the starting products optically under the microscope by the shape of the surface, the treated product after mechanical stress, for example on a conveyor belt, having significantly smaller dust fractions that the untreated product.

At the same time the originally feared clumping of the particles due to the additional oil content does not occur on prolonged storage, with the result that the flow behaviour too is not impaired.

The product according to the invention has after mechanical stress a significantly narrower grain size distribution that the untreated granulated animal

feedstuffs additive. Under mechanical stress the initial granules are destroyed, to some extent with dust formation, and the grain spectrum is broadened. This is manifested for example during storage in silos in the form of an undesirable demixing of the stored product.

This means that the dust fractions and dust values vary considerably in the known granules, depending on the sampling site.

Corresponding incalculable dust loads occur on emptying silos, when such demixed products are being loaded.

This problem does not occur with the product according to the invention.

The invention also provides a process for the production of a feedstuffs additive containing lysine based on fermentation broth and having an improved abrasion resistance, which process is characterised in that a shaped, in particular granulated animal feedstuffs additive containing 30 to 90 wt.%, in particular 40 to 70 wt.% of L-lysine, with a mean particle size preferably in the range from > 0.1 to ≤ 1.8 mm, is sprayed with an additive, the aforementioned additives, preferably oils, being metered in in an amount of 0.02 to 2 wt.% referred to the animal feedstuffs additive that is used.

Amounts of 0.2 to 1 wt.%, in particular amounts of 0.1 to 0.6 wt.%, are preferred.

The starting product contains the major proportion of the further constituents of the fermentation broth, ≥ 0 to 100 % of the biomass produced during the fermentation being contained.

The product according to the invention is then obtained.

An improved abrasion resistance is achieved by the addition of these small amounts and in addition the very

fine dust fraction that is present becomes bound. The largely dust-free product that is thereby formed and the optimised free-flowing behaviour lead to an improved handling of the product. This stability is found to be unchanged in long-term storage tests and under storage at high temperatures. The additive may be added batchwise or continuously. In order to achieve a uniform distribution of the additive in the product, it is recommended to feed the additive through one or more nozzles.

Mechanical or pneumatic mixers may be used as mixing equipment:

- vessels rotating about one or more axes, such as rotary tube mixers, tumble dryers, double-cone dryers,
- vessels with fixed or moving mixing tools such as turbulent flow mixers, ploughshare mixers, paddle-screw mixers,
- airmix mixers.

Furthermore, the additive may also be sprayed in during other process stages and mixed with the animal feedstuffs additive, such as for example

- in the bed of a fluidised bed dryer,
- in a pneumatically operating product conveyor line,
- in a screw-driven product conveyor line,
- in a product silo with mechanical or pneumatic mixing.

The product obtained has, despite the liquids added in minor amounts and the hydrophilic nature of the feedstuffs additive, a homogeneous distribution of these liquids. No agglomerates or lumps are formed.

Example 1

Production

100 kg of an L-lysine-containing feedstuffs additive (granules) produced according to EP 0 809 940 B1 were added to a 300 l capacity Lödige ploughshare mixer (60 % filling) and the mixer was set to a speed of 150 rpm. 0.5 kg of soybean oil was then added through a hollow-cone nozzle having a 1.1 mm bore. The dust content was analysed after a mixing time of 60 seconds.

Dust value before oil addition: 9.0

Dust value after oil addition: 0.1

It can be seen that the very fine dust fraction is bound to the granule particles.

A granular material containing L-lysine, the biomass and constituents from the fermentation was used as starting material. The material contains at least 46.8 % lysine and has a bulk density of 600 to 800 kg/m^3.

This product was also used in the further examples.

The bulk density was measured according to DIN 1060, except that the feedstuffs additive continuously falls through the funnel into the bulk material vessel. It is not collected in the funnel, which is then emptied into the bulk material vessel after opening a cap.

Example 2: Influence of the oil treatment on the dust content under pneumatic conveyance

Product	**Time**	**Conveying Length**	**Dust Amount in the Filter after in each case 40 m**	**Ratio of Dust in the Filter to Total Amount of Product**	**Dust Value**
	Min	**m**	**g**	**%**	**-**
without oil		0			1
	3.16	40	148	0.4	11
	3.08	80	122	0.72	23.6
	2.87	120	236	1.36	38.9
			506	1.36	
with 0.5 % oil		0			0.2
	3.66	40	50	0.11	2.75
	3.66	80	90	0.32	6
	3.66	120	90	0.52	10.5
			230	0.52	

Table 1

The measurements show a significantly reduced dust formation on account of the improved abrasion resistance after the treatment of the animal feedstuffs granules with oil (soybean oil).

Both products were previously practically dust-free in order to avoid falsification of the results by any dust contents already present.

Example 3

Influence of various oils on the dust content

Test	**Dust Value**
Without oil	5.6
+0.5% mineral oil, low viscosity	2.5
+0.52% mineral oil, viscous	1.4
+0.55% silicone oil, industrial	1.6
+0.52% olive oil, native	1.5

Table 3

Example 4

Use of various oils

The starting product according to Example 1 was used in an amount of 400 g, and was stirred with a blade mixer (60 rpm) and sprayed with the various oils.

Table 4

Experiment	Additive	Amount (%)	Temp. (°C)	Time (min.)	Dust Value (-)
1st pattern	without				20.9
A	AU10	1.38	50	10	0.2
B	AU10	1	60	5	0.1
C	AU20	0.55	60	5	1.3
D	AU30	0.563	40	5	0.7
E	edible oil	0.5	60	5	3.7
F	PEG400	0.75	60	5	0.6
G	CSL	0.875	60	5	5.3
2nd pattern	without				17.7
A	soybean oil	0.825	60	5	0.7
B	soybean oil	1.28	60	5	0.1
C	crude soybean oil	0.33	60	5	0.8

It can be seen that there is a significant reduction in the dust content when using various oils and additives.

Abbreviations used:

PEG400: polyethylene glycol

CSL: corn steep liquor

AU: lecithin/soybean oil mixtures
(AU: acetone-insoluble fraction)

1) AU 10	16.1g lecithin(AU 62) 83.9g soybean oil
2) AU 20	32.3g lecithin(AU 62) 67.7g soybean oil
3) AU 30	48.4g lecithin(AU 62) 51.6g soybean oil

Example 5

Properties of the treated product

Oil addition [%]	-	+ 0.1	+ 0.2	+ 0.3	+ 0.5
Flowability	2	2	2	2	2
Bulk density	760 kg/m^3	770 kg/m^3	770 kg/m^3	790 kg/m^3	810 kg/m^3
Dust value	6	2	0.6	0.3	0.1
Water uptake 1 hr / 40 °C / 75%	+ 5 %	+ 4 %	+ 4 %	+ 4 %	+ 4 %
Water uptake 4 hrs / 40 °C / 75%	+ 13 %	+ 12 %	+ 14 %	+ 12 %	+ 12 %

Table 5. Oil was sprayed (60°C) onto the animal feedstuffs additive (see Example 1) in a 150 l capacity ribbon mixer (28 rpm, t = 4 min).

Example 6

Influence of the addition of oil in various forms of transportation, on the abrasion

An animal feedstuffs additive as described in Example 1 was conveyed under various conditions matching practical applications.

The conveyance involved dense flow, strand and pneumatic conveyance with conveying lengths of 40 to 120 m. These are operated at different air speeds and product/air ratios.

The following were selected in the present case:

	Air Speed (m/sec)	**Product/Air Ratio (μ)**
Dense flow conveyance	2.2	55
Strand conveyance	7.3	10.6
Pneumatic conveyance	24.0	3.3

The value for the dust includes the dust deposited in the filter

The influence of the treatment of the granulated starting product, in this case with soybean oil, on the abrasion can clearly be seen, which is measured as the dust value after mechanical treatment over various conveying lengths compared to the starting product (Fig. 1).

Example 7

Demixing behaviour in the silo

Untreated granulated animal feedstuffs additive and additive treated with 0.5 wt.% soybean oil were in each case discharged from a silo into 1000 kg sacks.

A sample was taken from every tenth sack and the dust content was measured; the maximum value as well as the value after 30 seconds' settling time were measured.

A bandwidth of the dust value ranging from 10.1 to 21.7 is found in the untreated product (Table 6).

The dust value for the treated animal feedstuffs additive is between 1.7 and 4.1, with a mean value of 2.9 (Table 7).

The mean value of 1.6 for the dust content after 30 seconds also points to the extremely low very fine dust content in the treated product.

Fig. 2 shows the distribution of the dust values of Example 7. The number of sacks having the corresponding dust value is given under "Frequency". The untreated product does not exhibit a standard distribution but instead a broad scatter of the dust value. This means that batches with significantly varying, increased dust fractions occur within any one lot. This accords with the empirical result that the dust fraction of product fractions contained in a silo cannot be predicted.

On account of its improved abrasion behaviour the treated product has only a very low dust fraction and thus a narrow particle spectrum. The particle distribution in a silo expressed as the dust value has the form of a standard distribution, with a very small standard deviation. This is confirmation of the fact that, after

treatment of the product, unpredictable "dusty fractions" no longer have to be expected when emptying a silo.

Sample	Dust Value	Dust Value after 30 sec
1	21.2	14.6
2	11.5	9.8
3	10.1	7.7
4	16.5	14.3
5	13.0	10.6
6	17.1	10.8
7	21.7	17.5
8	16.3	14.1
9	19.1	13.3
10	14.5	11.4
11	20.0	14.2
12	20.8	13.9
13	15.4	10.1

Min.	10.1	7.7
Max.	21.7	17.5
Average	16.7	12.5
Standard deviation	3.76	2.65

Table 6: Untreated product

Sample	Dust Value	Dust Value after 30 sec
1	2.3	1.4
2	1.8	0.9
3	1.7	1.1
4	3.5	1.4
5	4.1	1.7
6	3.7	2.3
7	3.3	1.7
8	2.9	1.5
9	2.7	1.7
10	3.1	1.9
11	3.4	1.7
12	2.6	1.7

Min.	1.7	0.9
Max.	4.1	2.3
Average	2.9	1.6
Standard deviation	0.74	0.36

Table 7: Product treated with soybean oil

Patent Claims

1. A substantially dust-free animal feedstuffs additive based on fermentation broth having improved abrasion resistance, containing L-lysine and preferably the majority of the further constituents of the fermentation broth, the biomass produced during the fermentation being contained in an amount of ≥ 0 to 100 %, wherein the animal feedstuffs additive

   1.1 contains L-lysine in a concentration of 30 to 90 wt.% referred to the total amount,

   1.2 an amount of preferably ≥ 97 %, in particular ≥ 98 %, has a mean particle size of > 0.1 to 1.8 mm, and

   1.3 contains on the surface a proportion of added additive, in particular oil, in an amount of 0.02 to 2 wt.% referred to the total amount of the feedstuffs additive.

2. An animal feedstuffs additive as claimed in claim 1, wherein it contains on the surface, as additive, one or more oils selected from the group comprising mineral oil, vegetable oils, soybean oil, olive oil, soya/lecithin mixtures, edible oils, mixtures of vegetable oils.

3. An animal feedstuffs additive as claimed in claim 1, wherein it contains on the surface one or more of the additives selected from the group comprising silicone oils, polyethylene glycols or hydroxyethylcellulose.

4. An animal feedstuffs additive as claimed in claim 1, wherein it contains byproducts from the sugar and starch industry, in particular CSL or oily compounds.

5. An animal feedstuffs additive as claimed in claim 1, wherein it contains 0.2 to 1.0 wt.% of added additive.

6. An animal feedstuffs additive as claimed in claim 1, wherein after pneumatic conveyance over 40, 80 or 120 m it has a dust fraction of < 1 wt.% and a dust value of < ca. 10.

7. An animal feedstuffs additive as claimed in claim 1, with a bulk density in the range between 600 to 950 kg/m^3, in particular 650 to 900 kg/m^3.

8. A process for the production of a substantially dust-free feedstuffs additive containing L-lysine based on fermentation broth, containing L-lysine and the major proportion of the further constituents of the fermentation broth, the fermentation biomass being contained in an amount of ≥ 0 to 100 %, wherein a shaped, in particular granulated animal feedstuffs additive containing 30 to 90 wt.% of lysine is sprayed with an additive, the additive being metered in in an amount of 0.02 to 2 wt.% referred to the amount of animal feedstuffs additive used.

9. A process as claimed in claim 8, wherein an animal feedstuffs additive having a mean particle size of 0.1 to ≤ 1.8 mm is used, 0.1 to 10 wt.% having a particle size of < 100 µ.

10. A process as claimed in claim 8, wherein as additive there is used one or more oils selected from the

group comprising mineral oil, vegetable oils, soybean oil, olive oil, soya/lecithin mixtures, edible oils, mixtures of vegetable oils.

11. A process as claimed in claim 8, wherein one or more additives selected from the group comprising silicone oils, polyethylene glycols or hydroxyethylcellulose (in aqueous solution) are used.

12. A process as claimed in claim 7, wherein the feedstuffs additive is sprayed in a moved state.

13. A process as claimed in claim 8, wherein the feedstuffs additive is sprayed in a mechanical or pneumatic mixer.

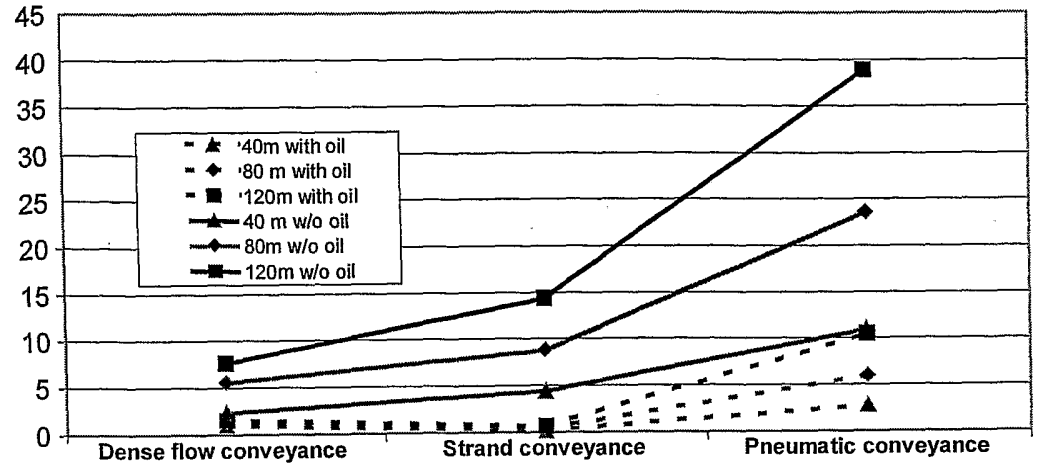
45
40
35
30
25
20
15
10
5
0
'40m with oil
'80 m with oil
'120m with oil
40 m w/o oil
80m w/o oil
120m w/o oil
Dense flow conveyance
Strand conveyance
Pneumatic conveyance

Fig. 1

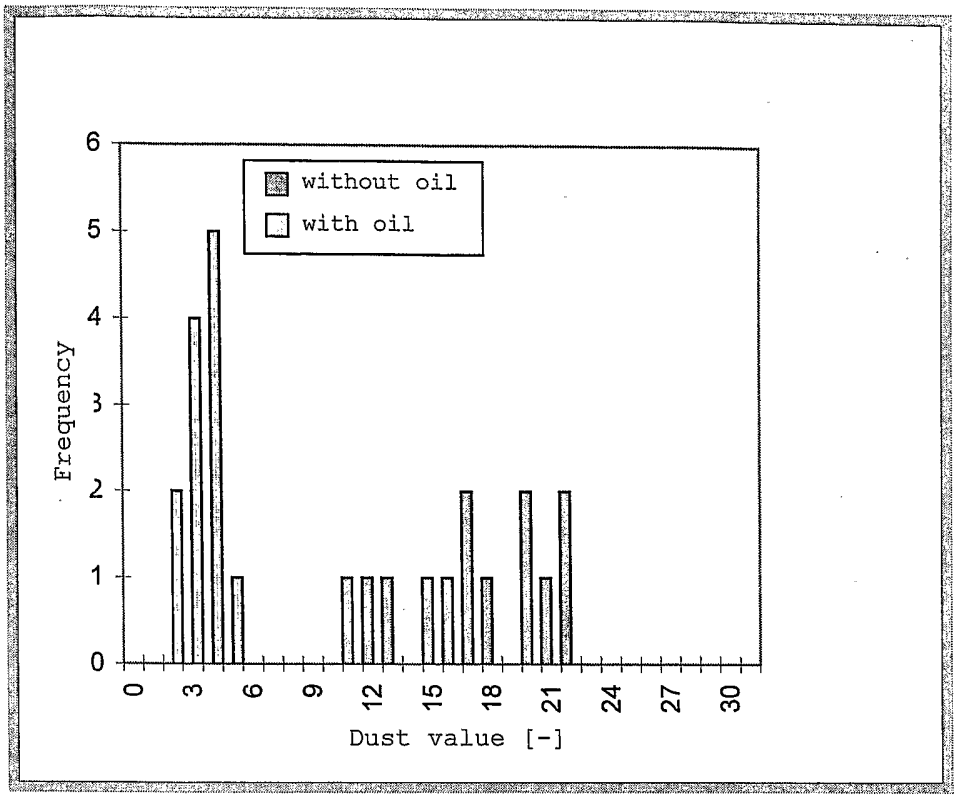
without oil
with oil
Frequency
Dust value [-]
0
1
2
3
4
5
6
0
3
6
9
12
15
18
21
24
27
30

Fig. 2

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 03/13200

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A23K1/00 A23K1/16

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 A23K

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, PAJ, BIOSIS, FSTA

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 743 016 A (AJINOMOTO KK) 20 November 1996 (1996-11-20) page 3, line 30-41 page 4, line 10-29,57,58 page 5, line 1,2 example 1 page 16, line 35,36 claim 1	1,4,5,7
A	page 3, line 3-8; claim 7	8-13
Y	US 5 840 358 A (SCHUETTE RUEDIGER ET AL) 24 November 1998 (1998-11-24) cited in the application column 3, line 20-62 column 5, line 30-50 column 6, line 15-17 column 10, line 31-67 examples 1-5	1,2,5, 7-10,12, 13

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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"A" document defining the general state of the art which is not considered to be of particular relevance

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the International search	Date of mailing of the international search report
12 March 2004	19/03/2004

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Couzy, F

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 03/13200

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 615 693 A (DEGUSSA) 21 September 1994 (1994-09-21) cited in the application page 2, line 55 -page 3, line 18 page 3, line 46-48 page 4, line 8,54 page 6, line 21-24 examples 1-3	1,2,5, 7-10,12, 13
Y	US 5 431 933 A (BINDER WOLFRAM ET AL) 11 July 1995 (1995-07-11) column 3, line 35 -column 4, line 68 example 3	1,2,5, 7-10,12, 13
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A	US 5 486 363 A (BETZ ROLAND ET AL) 23 January 1996 (1996-01-23) column 1, line 7-18,59-67	1-13

Form PCT/ISA/210 (continuation of second sheet) (July 1992)

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

Claims Nos.: 6

Present claim 6 relate to a product defined by reference to a desirable characteristic or property, namely to have certain dust fraction and dust values properties after having been conveyed in a pneumatic system. As this is not a standard nor standardized test, and since the values obtained will depend very much upon the test itself, that claim lacks clarity (Article 6 PCT) to such an extend as to render a meaningful search over the whole of the claimed scope impossible. Consequently, the search has not been carried out for that claim.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/EP 03/13200

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☒ Claims Nos.: 6
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
see FURTHER INFORMATION sheet PCT/ISA/210

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
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