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METHOD OF SEPARATING POLYETHYLENE (PE) AND POLYPROPYLENE (PP)

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(71) Applicant(s)
KALI UND SALZ AKTIENGESELLSCHAFT

(72) Inventor(s)
INGO STAHL; AXEL HOLLSTEIN; ULRICH KLEINE-KLEFFMANN; IRING GEISLER; ULRICH NEITZEL

(74) Attorney or Agent
CALLINAN LAWRIE , Private Bag 7, KEW VIC 3101

(56) Prior Art Documents
AU 23199/92 B03C 7/00
US 4570861
US 3563375

(57) Claim

1. A process for the separation of a mixture of plastic particles of plastics of a chemically different type which, however, have approximately the same density range by an electrostatic separation process by means of a free-fall separator, whereby the plastic particles are reduced to a particle size of less than 10mm subjected to air at a temperature of 15°C to 50°C, and a relative humidity of 10 to 40%, thereby undergoing triboelectric charging, wherein prior to the triboelectric charging, the plastic particles are subjected to a surface treatment.

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(71) Anmelder *(für alle Bestimmungsstaaten ausser US)*: KALI UND SALZ AKTIENGESELLSCHAFT [DE/DE]; Friedrich-Ebert-Straße 160, Postfach 10 20 29, D-3500 Kassel (DE).

(72) Erfinder; und
(75) Erfinder/Anmelder *(nur für US)* : STAHL, Ingo [DE/DE]; Staufenbergstr. 31, D-3592 Vellmar (DE). HOLLSTEIN, Axel [DE/DE]; Löwenburgstr. 1a, D-3500 Kassel (DE). KLEINE-KLEFFMANN, Ulrich [DE/DE]; Am Wendeberg 25, D-6430 Bad Hersfeld (DE). GEISLER, Iring [DE/DE]; An der Sommerseite 4a, D-6430 Bad Hersfeld (DE). NEITZEL, Ulrich [DE/DE]; Am Donarbrunnen 28, D-3500 Kassel (DE).

(81) Bestimmungsstaaten: AU, BR, CA, CS, HU, JP, KR, PL, RU, US, europäisches Patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LU, MC, NL, SE).

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654578

(54) Title: METHOD OF SEPARATING POLYETHYLENE (PE) AND POLYPROPYLENE (PP)

(54) Bezeichnung: VERFAHREN ZUR TRENNUNG VON POLYETHYLEN (PE) UND POLYPROPYLEN (PP)

(57) Abstract

The invention calls for mixtures of plastics materials, in particular materials of similar density such as polyethylene and polypropylene, to be separated using electrostatic techniques, the mixture being subjected to a surface-treatment operation before being electrostatically charged.

(57) Zusammenfassung

Kunststoffgemenge, insbesondere solche ähnlicher Dichte, wie Polyethylen und Polypropylen, werden auf elektrostatischem Wege getrennt, wobei das Gemenge vor der Aufladung einer Oberflächenbehandlung unterworfen wird.

Method for separation of polyethylene (PE) and polypropylene (PP)

The present invention relates to a method for separation of synthetic material fragments of a mixture of synthetic materials of different chemical types, which possess approximately the same range of densities, for example, polyethylene (PE) and polypropylene (PP) by electrostatic separation using a free-fall separator,

The polyolefines, polyethylene (PE) and polypropylene (PP), belong to the most commonly-used solid synthetic materials. They therefore represent the predominant portion of synthetic plastics materials occurring in waste. The density of PE lies in the range between 0.92 and 0.97 gm/cm^3, and that of PP lies in the range between 0.9 and 0.91 gm/cm^3.

Many utility- and single-use-articles consist of these synthetic plastics materials. An example of this is medicinal single-use syringes. Single-use syringes consist of a cylinder made from polypropylene and a plunger made from polyethylene. After removal of the injection needle, they are thrown away as rubbish and, up till the present time, they have largely been disposed of by burning. The trend in the waste material sector today is towards recovery of the materials. In many hospitals, there are already pilot projects for the collection of synthetic plastics material articles for re-cycling.

The amount of synthetic material in syringes consists of approximately equal parts of PE and PP. There is no particular demand for such mixtures and there is thus little or no profit to be made from them. There are even re-cycling enterprises which demand payment for their removal.

In contrast to this, for re-cyclates of a single type of material there is a profit to be made which is related to the price of the new material and can amount to as much as 60% of the price of new material. As the result of this it is economically attractive to be able to separate such mixtures into their components.

According to the present state of the art, the following possibilities exist for the separation of such types of mixtures of synthetic plastics materials.

1. Sorting by hand This method is employed on a large scale in re-cycling operations for the want of better methods, although it involves very high labour costs and is therefore uneconomical.

2. Sorting by density does not appear to be a very successful method, because PE and PP have similar densities and their separation by means of a water-alcohol mixture having a density of 0.91 has not found any practical application. From this it may be seen that a density separation based on water, because of the densities of the two substances being substantially the same, is not possible.

The separation of mixtures of synthetic materials in a free-fall separator is described in the DE-PS 30 35 649. However, the known procedure cannot be utilised for the separation of the afore-named synthetic plastics materials because, in the triboelectric charging of the mixture of synthetic materials, the different synthetic plastics materials PE and PP are not selectively charged.

The result of this is that, in the passage through the free-fall separator, a very substantial amount of middlings is produced, that is to say, the fragments of synthetic material carry a very small charge which is not sufficient for their deflection in the electrostatic field. Frequently, the charging is quite un-selective.

The objective to be achieved by the invention is therefore to develop a method of the type initially referred to in such a way that, in particular in the separation of PE and PP, a higher degree of purity of each of the synthetic plastics materials involved will be achieved, in which case the amount of middlings obtained will be kept as small as possible.

In accordance with the present invention, there is provided a process for the separation of a mixture of plastic particles of plastics of a chemically different type which, however, have approximately the same density range by an electrostatic separation process by means of a free-fall separator, whereby the plastic particles are reduced to a particle size of less than 10mm subjected to air at a temperature of 15°C to 50°C, and a relative humidity of 10 to 40%, thereby undergoing triboelectric charging, wherein prior to the triboelectric charging, the plastic particles are subjected to a surface treatment.

~~The objective to be achieved by the invention is therefore to develop a method of~~ the type initially referred to in such a way that, in particular in the separation of PE and PP, a higher degree of purity of each of the synthetic plastics materials involved will be achieved, in which case the amount of middlings obtained will be kept as small as possible.

In accordance with the present invention, this objective may be achieved by subjecting the mixture of synthetic materials to a surface treatment before the triboelectric charging. The surface treatment of the mixture of synthetic materials is effected in accordance with an embodiment of the invention by using mineral acid. In another embodiment the mixture of synthetic materials is brought into contact with an alkaline solution. The mineral acid recommended for use is, in particular, dilute hydrochloric acid, in which case the dilution is such that the pH-~~value is approximately 3.~~

The caustic soda solution is preferably diluted to such an extent that a solution of caustic soda is obtained having a pH-value of approximately 10 - 12.

Experiments have shown that a surface treatment with the appropriate substances will yield good results in the free-fall separator, which is evident, in particular, from the high degree of purity of the fractions, and also from the relatively small amount of middlings obtained.

The explanation for this appears to be that the caustic soda solution produces a change in the surface of the synthetic material in such a way that a better triboelectric charging is made possible.

Before the actual surface treatment, the mixture of synthetic materials, preferably comminuted to fragments having a size less than 10 millimetres, preferably less

than 6 millimetres, is freed from foreign substances, such as paper for example, and washed clean with water. In accordance with a special feature of the invention, mineral acid or alkaline solution is added to the cleansing water, during the cleansing process, in which case, however, the degree of dilution, characterised by adjustment to the appropriate pH-value, must be maintained.

After the cleansing process, if necessary with the addition of alkaline solution or mineral acid, and if necessary after the washing of the mixture of synthetic materials with clear water, it is dried in de-watering equipment, for example a centrifuge, to a moisture content of approximately 2% or less.

Subsequently the mixture of synthetic materials is subjected to heat treatment at a temperature in the range from 70 to 100 °C during not less than 5 minutes.

This treatment also serves to alter the surface with the objective of achieving a better triboelectric charging of the individual fragments of synthetic material.

After the temperature treatment, in accordance with another advantageous feature of the invention, fatty acid is added at the rate of 10 - 50 mg/kg of mixture of synthetic materials.

It has been demonstrated that, on the basis of this preliminary treatment and here, in particular on the basis of the preliminary treatment with acid or alkali solution, it is satisfactory if the free-fall separator operates at a field strength maintained at only 2 - 3 KV/cm, in order to promote the deposition of the fragments of synthetic material on the appropriate electrodes. With such a comparatively low field strength, corona discharges, such as could occur at higher field strengths, are avoided, and consequently the possible ignition of the fragments of synthetic material, or a probable dust explosion, is avoided.

The triboelectric charging of the mixture of synthetic materials is introduced into a fluidised-bed drier.

The triboelectric charging itself is effected, after the temperature treatment at a temperature in the range from 15 to 50 °C, preferably from 20 to 35 °C, with a relative humidity of the surrounding air of 10 to 40%, preferably 15 to 20%. The charging itself may be effected in a fluidised-bed drier or in a spiral screw conveyor of sufficient length. The charging may equally well be possible by forwarding the mixture of synthetic materials over a specified distance by pneumatic means.

The method of the invention is explained in the following two examples:

Example 1

Discarded syringes from a hospital were comminuted in a cutting mill. The mixture of fragments obtained had a PP-content of 51.1% and a PE-content of 49.9%. The mixture of fragments was washed, centrifuged and dried in a fluidised-bed drier at 80 °C for a period of 6 minutes and, after cooling, it was charged for a further 3 minutes in the fluidised-bed at 25 °C and 21% relative humidity. However, before the charging in the fluidised-bed, fatty acid to the extent of 50 milligrams /kg of synthetic material was added.

The following results were obtained from the separation:

	Analysis (degree of purity)						Yield (amount)					
Eff. amt. (%)			% PE			% PP			PE (%)			PP (%)		
P	M	N	P	M	N	P	M	N	P	M	N	P	M	N
46,4	7,2	46,4	96,9	55,2	3,9	3,1	47,5	92,1	89,0	7,4	3,6	2,9	6,9	90,2

for explanation:

P = positive electrode

N = negative electrode

M = middlings

From the fragments of the synthetic mixture derived from discarded syringes, outstanding results were obtained after washing, drying at a higher temperature, conditioning with fatty acid and charging at a temperature slightly higher than room temperature.

Example 2

A dried mixture of fragments of PP/PE from comminuted bottles had a PP-content of approximately 57% and a PE-content of approximately 43%. The mixture was first of all treated with 4% caustic soda solution and then washed with water, centrifuged and dried in the air for 20 hours.

The mixture was charged in a fluidised bed for 3 minutes at a temperature of 25 °C and 11% relative humidity and then separated in a free-fall separator.

The following results were obtained from the separation:

	Analysis (degree of purity)						Yield (amount)					
Eff. amt. (%)			% PE			% PP			PE (%)			PP (%)		
P	M	N	P	M	N	P	M	N	P	M	N	P	M	N
41,1	15,8	43,1	88,2	26,7	6,8	11,8	73,3	93,2	83,5	9,7	6,8	8,6	20,4	71,0

It was found that washing with diluted caustic soda solution led to useful results from the separation.

Example 3

Discarded syringes from a hospital were comminuted in a cutting mill and, after different drying procedures, was separated in a free-fall separator.

The mixture of fragments obtained had a PP-content of 57.6% and a PE-content of 42.4%.

a) The mixture of fragments was heated in a fluidised-bed drier at 80 °C for a period of 6 minutes and, after cooling to 30 °C, it was charged for a further 3 minutes in the fluidised-bed at 30 °C.

b) The same mixture was charged for 5 minutes in a fluidised-bed at 30 °C.

c) The same mixture was charged for 5 minutes in a fluidised-bed at 80 °C.

The following results were obtained from the separation:

	Analysis (degree of purity)									Yield (amount)					
	Eff. amt. (%)			% PE			% PP			PE (%)			PP (%)		
	P	M	N	P	M	N	P	M	N	P	M	N	P	M	N
a)	36,5	28,5	35,0	83,2	40,9	14,3	16,8	59,1	85,7	64,6	24,8	10,6	11,6	31,8	56,6
b)	29,1	63,6	7,3	40,8	44,7	40,8	59,2	55,3	59,2	27,4	65,7	6,9	30,3	62,0	7,6
c)	50,9	19,9	26,2	42,5	51,1	46,5	57,5	48,9	53,5	47,7	22,4	29,9	53,6	17,8	28,6

According to this, it is advantageous to dry the mixture of fragments first of all at a relatively high temperature and then charge it at a moderate temperature.

The claims defining the invention are as follows:-

1. A process for the separation of a mixture of plastic particles of plastics of a chemically different type which, however, have approximately the same density range by an electrostatic separation process by means of a free-fall separator, whereby the plastic particles are reduced to a particle size of less than 10mm subjected to air at a temperature of 15°C to 50°C, and a relative humidity of 10 to 40%, thereby undergoing triboelectric charging, wherein prior to the triboelectric charging, the plastic particles are subjected to a surface treatment.

2. The process as claimed in claim 1 whereby the plastic particles are reduced to a particle size of less than 6mm.

3. The process according to claim 1 or 2, wherein for the surface treatment, the plastic particles are contacted with a mineral acid.

4. The process according to claim 1 or 2, wherein for the surface treatment, the plastic particles are contacted with an alkali lye.

5. The process according to any one of the preceding claims, wherein prior to the surface treatment, the mixture of plastic particles is cleaned of foreign substances, including paper, by water.

6. The process according to any one of claims 3 to 5, wherein the mineral acid or alkali lye is added to the cleaning water.

7. The process according to claim 6, wherein after the surface treatment with mineral acid or alkali lye, the mixture of plastic particles is washed with clear water.

8. The process according to anyone of claims 5 to 7, wherein the mixture is dried to a residual water proportion of less than 2% by dehydration aggregates, including a curved screen or centrifuge.

9. The process according to claim 8 wherein after the drying by dehydration aggregates, the mixture of plastic particles is subjected to a temperature treatment at 70 to 100°C over a time period of at least 5 minutes.

10. The process according to claim 9, wherein after the temperature

treatment, 10 to 50 mg fatty acid per kg mixture is added to the mixture.

11. The process according to any one of claims 3 or 7 to 10, wherein diluted hydrochloric acid is used as mineral acid.

12. The process according to claim 11, wherein the dilution of the hydrochloric acid is selected in such a way that a pH of approximately 3 is obtained.

13. The process according to any one of claims 4 or 7 to 10, wherein diluted soda lye is used as alkali lye.

14. The process according to claim 13, wherein the dilution of the soda lye is selected in such a way that a pH of approximately 11 to 12 is obtained.

15. The process according to claim 1, wherein the mixture of plastic particles is passed through a spiral worm.

16. The process according to claim 1, wherein the mixture of plastic particles is conveyed pneumatically.

17. The process according to any one of the preceding claims wherein the mixture of plastic particles is a mixture of polyethylene and polypropylene particles.

DATED this 12th day of September 1994.

KALI UND SALZ AKTIENGESELLSCHAFT

By their Patent Attorneys:

CALLINAN LAWRIE

Jeffrey A. Ryder

ABSTRACT

Mixtures of synthetic materials, in particular those of similar density, such as polyethylene and polypropylene, are separated into their individual components by electrostatic separation, in which case the electrostatic separation is preceded by a surface treatment.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/EP 92/01614

A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl.[5]: B 03 C 7/00; B 03 C 7/12; B 29 B 17/02

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)

Int.Cl.[5]: B 03 C

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 practicable, 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.
Y	DE, A, 3 035 649 (KALI UND SALZ AG) 8 April 1982 cited in the application	1,17
A	see page 6, paragraph 1 - page 7, paragraph 1; claims 1,2	4,11,16
	-.-	
Y	DE, A, 1 758 756 (SOCIETE DE PRODUITS CHIMIQUES D'AUBY) 25 February 1971	1,17
A	see page 2, paragraph 3 - page 3, paragraph 1; claims 1,4 see page 6, paragraph 4 - page 7, paragraph 1	2,10
	-.-	
A	DE, A, 3 227 874 (KALI UND SALZ AG) 26 January 1984 see page 4, paragraph 3; claim 1	1,4,11, 16,17
	-.-	
A	FR, A, 1 505 274 (KALI-FORSCHUNG-ANSTALT G.M.B.H.) 8 December 1967	

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

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

"E" earlier document but published on or after the international filing date

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"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
13 October 1992 (13.10.92)	27 October 1992 (27.10.92)
Name and mailing address of the ISA/ European Patent Office	**Authorized officer**
Facsimile No.	**Telephone No.**

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

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. EP 9201614 SA 62780

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information. 13/10/92

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE-A-3035649	08-04-82	None	
DE-A-1758756	25-02-71	FR-A- 1539876	
		GB-A- 1233764	26-05-71
		NL-A- 6811079	11-02-69
		OA-A- 2861	15-12-70
		US-A- 3563375	16-02-71
DE-A-3227874	26-01-84	US-A- 4570861	18-02-86
FR-A-1505274		None	

INTERNATIONALER RECHERCHENBERICHT

Internationales Aktenzeichen PCT/EP 92/01614

I. KLASSIFIKATION DES ANMELDUNGSGEGENSTANDS (bei mehreren Klassifikationssymbolen sind alle anzugeben)[6]

Nach der Internationalen Patentklassifikation (IPC) oder nach der nationalen Klassifikation und der IPC

Int.Kl. 5 B03C7/00; B03C7/12; B29B17/02

II. RECHERCHIERTE SACHGEBIETE

Recherchierter Mindestprüfstoff [7]

Klassifikationssystem	Klassifikationssymbole
Int.Kl. 5	B03C

Recherchierte nicht zum Mindestprüfstoff gehörende Veröffentlichungen, soweit diese unter die recherchierten Sachgebiete fallen [8]

III. EINSCHLÄGIGE VERÖFFENTLICHUNGEN [9]

Art.*	Kennzeichnung der Veröffentlichung [11], soweit erforderlich unter Angabe der maßgeblichen Teile [12]	Betr. Anspruch Nr. [13]
Y	DE,A,3 035 649 (KALI UND SALZ AG) 8. April 1982 in der Anmeldung erwähnt	1,17
A	siehe Seite 6, Absatz 1 - Seite 7, Absatz 1; Ansprüche 1,2	4,11,16

Y	DE,A,1 758 756 (SOCIÉTÉ DE PRODUITS CHIMIQUES D'AUBY) 25. Februar 1971	1,17
A	siehe Seite 2, Absatz 3 - Seite 3, Absatz 1; Ansprüche 1,4 siehe Seite 6, Absatz 4 - Seite 7, Absatz 1	2,10

A	DE,A,3 227 874 (KALI UND SALZ AG) 26. Januar 1984 siehe Seite 4, Absatz 3; Anspruch 1	1,4,11, 16,17

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* Besondere Kategorien von angegebenen Veröffentlichungen [10]:

"A" Veröffentlichung, die den allgemeinen Stand der Technik definiert, aber nicht als besonders bedeutsam anzusehen ist

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"X" Veröffentlichung von besonderer Bedeutung; die beanspruchte Erfindung kann nicht als neu oder auf erfinderischer Tätigkeit beruhend betrachtet werden

"Y" Veröffentlichung von besonderer Bedeutung; die beanspruchte Erfindung kann nicht als auf erfinderischer Tätigkeit beruhend betrachtet werden, wenn die Veröffentlichung mit einer oder mehreren anderen Veröffentlichungen dieser Kategorie in Verbindung gebracht wird und diese Verbindung für einen Fachmann naheliegend ist

"&" Veröffentlichung, die Mitglied derselben Patentfamilie ist

IV. BESCHEINIGUNG

Datum des Abschlusses der internationalen Recherche	Absendedatum des internationalen Recherchenberichts
13.OKTOBER 1992	27. 10. 92
Internationale Recherchenbehörde	**Unterschrift des bevollmächtigten Bediensteten**
EUROPAISCHES PATENTAMT	DECANNIERE L.

Internationales Aktenzeichen PCT/EP 92/01614

III. EINSCHLAGIGE VEROFFENTLICHUNGEN (Fortsetzung von Blatt 2)

Art *	Kennzeichnung der Veröffentlichung, soweit erforderlich unter Angabe der maßgeblichen Teile	Betr. Anspruch Nr.
A	FR,A,1 505 274 (KALI-FORSCHUNG-ANSTALT G.M.B.H.) 8. Dezember 1967	

ANHANG ZUM INTERNATIONALEN RECHERCHENBERICHT ÜBER DIE INTERNATIONALE PATENTANMELDUNG NR.

EP 9201614
SA 62780

In diesem Anhang sind die Mitglieder der Patentfamilien der im obengenannten internationalen Recherchenbericht angeführten Patentdokumente angegeben.
Die Angaben über die Familienmitglieder entsprechen dem Stand der Datei des Europäischen Patentamts am 13/10/92
Diese Angaben dienen nur zur Unterrichtung und erfolgen ohne Gewähr.

Im Recherchenbericht angeführtes Patentdokument	Datum der Veröffentlichung	Mitglied(er) der Patentfamilie	Datum der Veröffentlichung
DE-A-3035649	08-04-82	Keine	
DE-A-1758756	25-02-71	FR-A- 1539876	
		GB-A- 1233764	26-05-71
		NL-A- 6811079	11-02-69
		OA-A- 2861	15-12-70
		US-A- 3563375	16-02-71
DE-A-3227874	26-01-84	US-A- 4570861	18-02-86
FR-A-1505274		Keine	

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