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

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
18 December 2003 (18.12.2003)

PCT

(10) International Publication Number
WO 03/104314 A1

(51) International Patent Classification[7]: C08J 11/04 // C08L 67/02

(21) International Application Number: PCT/CZ02/00055

(22) International Filing Date: 16 October 2002 (16.10.2002)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
PV 2002-1799 23 May 2002 (23.05.2002) CZ

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(81) Designated States *(national)*: CA, GE, HR, HU, IL, JP, KR, LT, LV, NO, PL, RO, SI, TN, UA, US, YU.

(84) Designated States *(regional)*: Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR).

**Published:**
— *with international search report*

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

WO 03/104314 A1

(54) Title: METHOD FOR TREATMENT OF POLYESTER MATERIALS

(57) Abstract: A Method for treatment of polyester materials A method for treatment of polyester materials specifically such as used polyester containers by which the waste mixture is crushed, washed, dried and melt to obtain a source of polyester material characterized in that the mixture of crushed and dried waste polyester material is supplied by 4 to 6 % by weight based on the total of the waste mixture of a modifying agent elected from the group comprising in combination a polysiloxane and a plasticizer elected from the group comprising phthalates, or in combination silazanes and silanes.

# Method for treatment of polyester materials

## Technical field

The invention relates to a method for treatment of polyester materials preferably such as waste polyester containers wherein the waste mixture is crushed, washed, dried and melt to obtain a recovered source of polyester material. Said waste includes mostly the materials based on polyethylene terephthalate generally indicated also as PET and known from the packing technology for example as so called PET bottles. The final product to be used as recycled source of polyester material is mostly supplied in the form of granulates.

## Description of prior art

Polyester materials represent well known and widely used group of sound materials with proved hygienic properties used for example in packaging, picture or reproduction technology etc.. Probably, the most widely used polyester material is polyethylene terephthalate (hereinafter referred to also as PET) and the used PET containers are the most common waste PET material to be recovered.

As ecologically most desirable method for disposal of used polyester containers is the recycling thereof with the effort to recover materials of adequate quality that may be used for the original purpose. The method of recovery treatment of said waste materials, for example used PET containers include several steps such as waste crushing, washing and subsequent drying the crushed material and melting and processing thereof to obtain granulate, which is the source polyester material for manufacturing new products - such packing, using extruders or injection molding machines. The manufacture of granulate is accompanied by undesirable phenomena such as oxidation and hydrolysis of polyester macromolecules what on the other hand may result in excess amount of rejects in processing such recycled source material or in final products of lesser quality. The unfavorable features of final products may be yellow tinge of moldings, enhanced penetrability for $CO_2$ of the recycled packing and moreover, increased contents of low molecular fractions including aldehyde in the recycled source of polyester material. Such fractions then pass from the raw material into the packing product and deteriorate its quality.

To avoid such difficulties and to secure production of high quality food packing materials it was desirable to use other special methods that nevertheless mostly appeared to be complicated and in each case bringing about higher production cost.

For example, in the recycling process according to US patents 5 395 858 and 5 580 905 polyesters are converted into the original chemical compounds. Polyester materials are mixed with an alkaline mixture and converted at higher temperatures to alkaline salts and then saponified. By this procedure various impurities and organic and inorganic compounds contained in the waste are removed. Nevertheless, this method requires rather complicated technology and high operating costs.

The Czech patent application PV 2001-926 describes a process for purifying crushed polyester material by heating it up in a water-free medium of alkaline mixture after having been first dried, in which process the polyester material is partially saponified without being melt what enables its subsequent physical clarifying and removing of impurities. This method includes of course a multi-stage process that is like in the previous case based on the application of alkaline materials in the form of alkaline hydroxides.

The object of the invention is to provide a method that would lead to a source of polyester material recovered from polyester waste that has been recycled specifically by crushing and following treatment of PET beverage containers, which material may be subsequently used for manufacture of high quality food package without the necessity to make use of complicated and expensive methods in production of recycled material.

Summary of invention

The substantial feature of the method for treatment of polyester materials, specifically such as used polyester containers, according to the invention by which the waste mixture is crushed, washed, dried and melt to obtain a source of polyester material and which should eliminate certain problems of the prior art methods is that the mixture of crushed and dried waste polyester material is supplied by 4 to 6 % by weight based on the total of the waste mixture of a modifying agent elected from the group comprising in combination a polysiloxane and a plasticizer elected from the group comprising phthalates, or in combination silazanes and silanes.

According to another feature of the invention the mixture of crushed and dried waste polyester material is supplied by 4 to 6 % by weight based on the total of the waste mixture of a modifying agent elected from the group comprising in combination polyhydrosiloxane and a plasticizer elected from the group comprising dioctylphthalate, dinonylphthalate, dibutylsebacinate or in combination hexamethyldisilazane and tetraethoxysilane.

According to still another feature of the invention the modifying agent contains 20 to 30 % by weight of liquid polyhydrosiloxane and 70 to 80 % by weight of a plasticizer elected from the group comprising dioctylphthalate, dinonylphthalate, dibutylsebacinate or 55 to 65 % by weight of hexamethyldisilazane and 35 to 45 % by weight of tetraethoxysilane.

The modifying agent may be added to the waste mixture in a reactor - mixer at temperature of 130 ± 5 °C whereupon the waste mixture is subjected to stirring for 50 to 100 minutes.

Advantageously, the waste mixture is further processed at 240 to 250 °C to obtain a source of polyester material in the form of granulate.

As the result of using the method according to the invention the recycled waste polyester shows higher resistance of the source polyester against material destruction caused by oxidation and hydrolysis, while the other basic material parameters corresponding to the standard polyester are retained. This includes the removal of undesirable harmful fractions and lessening the penetration of gases with final products. Another advantage is that the entire production time is in comparison with similar processes reduced. The source of polyester material obtained by using the method according to the invention is contrary to common known types of such materials characterized by enhanced elasticity while maintaining its desired strength and further by its higher heat stability and higher crystallization temperature amounting up to 110 °C.

Detailed description of invention

Example 1

10 kg of waste polyester bottles was crushed in a crusher rinsed in a washer and the waste mixture so obtained was dried in a dryer. The dried waste mixture was put into a reactor - mixer and heated up to 130 ± 5 °C. At this temperature 400 g of a modifying agent was added to the reactor. The modifying agent consisted of 25 % by weight of liquid polyhydrosiloxane and 75 % by weight of dioctylphthalate as plasticizer. Subsequently the mixture was stirred at this temperature for about 60 minutes. The received waste mixture was then processed at temperature of 240 to 250 °C in an extruder to produce the granulate. The resulting granulate was of dark gray color and characterized by high brightness.

Example 2

10 kg of waste polyester containers was crushed in a crusher, rinsed in a washer and the waste mixture so obtained was dried in a dryer. The waste mixture was put into a reactor - mixer and heated up to 130 ± 5 °C. At this temperature 400 g of a modifying agent was added to the reactor. The composition of the modifying agent was as follows: 60 % by weight of hexamethyldisilazene and 40 % by weight of tetraethoxysilane. Subsequently, the mixture was stirred at this temperature for about 60 minutes. The waste mixture received was then processed at temperature of 240 to 250 °C in an extruder to produce granulate. The resulting granulate was clear and characterized by high brightness.

In comparison with the conventional products the recovered source of polyester materials have shown high elasticity while maintaining the required strength, higher heat stability and higher crystallization temperature - up to 110 °C.

Industrial applicability

The method according to the invention may be used in the process of recycling polyester waste including without limitation so called PET bottles to receive a standard quality source material preferably usable for manufacture of food containers.

## CLAIMS

1. A method for treatment of polyester materials specifically such as used polyester containers by which the waste mixture is crushed, washed, dried and melt to obtain a source of polyester material characterized in that the mixture of crushed and dried waste polyester material is supplied by 4 to 6 % by weight based on the total of the waste mixture of a modifying agent elected from the group comprising in combination a polysiloxane and a plasticizer elected from the group comprising phthalates, or in combination silazanes and silanes.

2. A method for treatment of polyester materials in accordance with claim 1 characterized in that the mixture of crushed and dried waste polyester material is supplied by 4 to 6 % by weight based on the total of the waste mixture of a modifying agent elected from the group comprising in combination polyhydrosiloxane and a plasticizer elected from the group comprising dioctylphthalate, dinonylphthalate, dibutylsebacinate or in combination hexamethyldisilazane and tetraethoxysilane.

3. A method for treatment of polyester materials in accordance with claim 1 characterized in that the modifying agent contains 20 to 30 % by weight of liquid polyhydrosiloxane and 70 to 80 % by weight of a plasticizer elected from the group comprising dioctylphthalate, dinonylphthalate, dibutylsebacinate or 55 to 65 % by weight of hexamethyldisilazane and 35 to 45 % by weight of tetraethoxysilane.

4. A method for treatment of polyester materials in accordance with claim 1 characterized in that the modifying agent is added to the waste mixture in the reactor - mixer at temperature of 130 ± 5 °C whereupon the waste mixture is subjected to stirring for 50 to 100 minutes.

5. A method for treatment of polyester materials in accordance with claim 3 characterized in that the waste mixture is further processed at 240 to 250 °C to obtain a source of polyester material in the form of granulate.

# INTERNATIONAL SEARCH REPORT

International Application No
PCT/CZ 02/00055

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08J11/04 //C08L67:02

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)
IPC 7 C08J C08K

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category ° | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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| A | EP 0 794 216 A (PHENIPLASTICS S A)<br>10 September 1997 (1997-09-10)<br>claims 1-4 | 1-5 |

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

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| Date of the actual completion of the international search | Date of mailing of the international search report |
|---|---|
| 29 April 2003 | 09/05/2003 |

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

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/CZ 02/00055

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