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(54) Title: PLANT AND METHOD FOR DRYING AND SOLID STATE POLYCONDENSING OF POLYMERIC MATERIAL

(57) Abstract: A plant (1) for drying and solid state polycondensing a moisture-containing polymeric material (M) in granular form, comprising a conduit (2) for feeding the material to be treated in a longitudinal direction (L), at least one treatment zone (7) for treating the polymeric material, means (6) for blowing an inert gas into the conduit, radiating means (8) for emitting an alternating electromagnetic field in the radio-frequency band and comprising a plurality of applicators (9) located in correspondence of said treatment zone and external to the conduit in longitudinally offset positions; said applicators are connected to the terminals of an electromagnetic wave generator (10) and comprise pairs of opposed radiating elements (11, 11') which are adapted to generate an alternating electromagnetic field in the conduit; the pairs of radiating elements defining magnetic dipoles with opposite polarities along the conduit. Further claimed is a method of drying and solid state polycondensing a polymeric material in granular form obtained by polycondensation using the plant.

FIG. 1
PLANT AND METHOD FOR DRYING AND SOLID STATE POLYCONDENSING OF POLYMERIC MATERIAL

Field of the invention

[0001] The present invention generally finds application in the field of treatment of polymeric materials, and particularly relates to a plant for drying and solid state polycondensing a polymeric material in granular form.

[0002] The invention further relates to a treatment method for drying and solid state polycondensing the aforementioned polymeric material.

Background art

[0003] Certain virgin and/or recycled polymeric materials, as used for molding plastic materials, are known to require treatment in appropriate plants for drying, upgrade and possibly crystallization.

[0004] Particularly, the materials selected for this purpose are polymers obtained by polycondensation reactions and selected from the group comprising polycondensates in general, such as: polyamides, elastomeric polyesters and PET and the term "upgrade" as used hereinafter is intended to designate the process of forming larger polymeric chains by repeated condensation reactions which occur in the solid state, to increase the molecular weight of polymers and, as a result, the inherent viscosity of the material.

[0005] Treatment plants generally use a plurality of treatment working stations in which a hot air stream flows through the polymeric materials to promote heating thereof to predetermined temperatures.

[0006] A first drawback of treatment plants is that the hot air that is used for thermally treating polymers causes an oxidation reaction which alters the structural or aesthetic characteristics of the material.

[0007] Furthermore, the material is heated using a plurality of distinct thermal stations cascaded along a product feed line, and this eventually increases the complexity and overall size of the plant.

[0008] In order to at least partially obviate the above mentioned drawbacks, a number of plants have been developed for thermal treatment of polymers by application of an electromagnetic field, generally in the radio-
frequency (RF) band.

[0009] These plants are particularly suitable for treating polymerized-by-condensation materials, with temperature being controlled either by adjusting the electric parameters of the electromagnetic field, particularly frequency and/or power, or by adjusting the field application time.

[0010] Thermal treatment of polymers by a RF electromagnetic field has the effect of removing the water that is naturally contained in the material to increase its molecular weight and provide a high-strength, high-viscosity polymer.

[0011] US6316518 discloses a plant for thermal treatment of polymers, e.g. polyesters, polyamides and polyurethanes, comprising a feed conduit for the polymeric material to be treated and two treatment stations along the feed conduit.

[0012] Each of the stations comprises an applicator for emitting a radio-frequency electromagnetic field of predetermined power, which is adapted to heat the polymeric material to a temperature ranging from 190°C to 205°C.

[0013] Particularly, the electromagnetic field generated by the electrodes has a frequency of about 40 MHz and its application time is of about 30 minutes per kilogram of material being treated.

[0014] Furthermore, the plant comprises means for blowing nitrogen into the treatment stations, in order to remove moisture that forms during the material polymerization reactions.

[0015] A first drawback of this arrangement is that the electromagnetic field generated by applicators is not uniform and cannot ensure optimal heating of the polymeric material that passes through the conduit.

[0016] This drawback hinders the drying and solid state polycondensing processes, whereby the polymeric materials being treated are found to have a very low molecular weight.

[0017] A further drawback is that, since the electromagnetic flux lines are transverse relative to the feed of the material in the conduit, the flow rate of the material shall be very low to ensure treatment of the polymeric material that is being fed therein, which can be obtained by reducing the diameter of
the conduit.

[0018] This drawback will further increase the overall times required for thermal treatment of the polymeric material.

[0019] Furthermore, due to the particular conformation of the electromagnetic field being used, this plant has a very low thermal efficiency and a very high overall consumption.

Technical Problem

[0020] In light of the prior art, the technical problem addressed by the present invention consists in providing thermal treatment of a polymeric material in granular form, obtained by polycondensation, to promote uniform drying and solid state polycondensing thereof, in a very short time and with high thermal efficiency.

Disclosure of the invention

[0021] The general object of the present invention is to solve the above discussed technical problem by obviating the drawbacks of the prior art.

[0022] A particular object is to provide a plant for drying and solid state polycondensing a polymeric material in granular form, that is highly efficient and relatively cost-effective.

[0023] Another particular object of the present invention is to provide a plant as mentioned above, that can ensure uniform thermal treatment of the polymeric material.

[0024] A further object of the present invention is to provide a plant for drying and solid state polycondensing a polymeric material that ensures a high thermal efficiency.

[0025] Another object of the present invention is to provide a plant as mentioned above that has a simple structure and can be easily managed.

[0026] Yet another object of the present invention is to provide a method of drying and solid state polycondensing a polymeric material that has very short overall treatment times.

[0027] These and other objects, as better explained hereafter, are fulfilled by a plant for drying and solid state polycondensing a polymeric material in granular form obtained by polycondensation as defined in claim 1, which
comprises a feed conduit for the material to be treated, means for blowing an inert gas into the conduit, and a plurality of radiating means for emitting an alternating electromagnetic field in the radio-frequency band.

[0028] The radiating means comprise a plurality of applicators arranged along and external to the conduit at respective treatment stations, each applicator comprising a pair of opposed radiating elements for generating a radio-frequency electromagnetic field in the conduit, with field lines at least partially parallel to the feed direction of the material.

[0029] In a further aspect, the invention relates to a method of drying and solid state polycondensing such polymeric material, as defined in claim 11.

[0030] Advantageous embodiments of the invention will be defined in the dependent claims.

**Brief Description of the Drawing**

[0031] Further characteristics and advantages of the invention will be more apparent upon reading of the detailed description of a preferred, non-exclusive embodiment of a plant and a method for drying and solid state polycondensing a polymeric material in granular form according to the invention, which is described as a non-limiting example with the help of the annexed drawings, in which:

- FIG. 1 is a schematic perspective view of the plant for drying and solid state polycondensing a solid polymeric material of the invention according to a first embodiment;
  - FIG. 2 is a sectional side view of a first detail of Fig. 2;
  - FIG. 3 is a schematic perspective view of the plant for drying and solid state polycondensing the polymeric material according to a second embodiment;
  - FIG. 4 is a schematic perspective view of the plant for drying and solid state polycondensing the polymeric material according to a third embodiment;
- FIGS. 5 to 7 are sectional side views of the plant in its three embodiments, comprising the flux lines of the electromagnetic fields that have been generated;
FIG. 8 is a block diagram of the method of drying and solid state polycondensing the solid polymeric material according to the invention.

Detailed description of a preferred embodiment

[0032] Referring to the aforementioned figures, a plant for drying and solid state polycondensing a moisture-containing polymeric material M is shown and generally designated by numeral 1.

[0033] The materials that can be treated by the plant 1 belong to the group of the polymers that can polymerize by condensation polymerization, such as polyamides and polyesters.

[0034] Particularly, as shown in Table I below, the polymers that exhibit a highly reduced inherent moisture upon drying and a considerably increased viscosity upon solid state polycondensing are polyamide 6 as obtained by ring-opening polycondensation, elastomeric polyesters obtained by polycondensation of an ester of a dicarboxylic acid and a soft segment such as PTMG, PET and other similar polymers.

[0035] The polymeric material M to be treated may further be in granular form, which will increase the exchange surface for thermal treatment.

[0036] In a preferred embodiment of the invention, the plant 1 comprises a feed conduit 2 for feeding the material M to be treated in a predetermined longitudinal direction L, and located between an inlet station and an outlet station, not shown.

[0037] Advantageously, as shown in FIG. 1, the conduit 2 may comprise a wall 3 made of a dielectric material and may extend in a substantially vertical direction L to facilitate natural gravity feed of the material M.

[0038] Alternatively, means 4 may be provided for forced feeding of the material M, which are located in the conduit 2 and are selected from the group comprising augers or Archimedean screws, not shown, or the belt conveyors 5, as shown in FIGS. 3 and 4.

[0039] Furthermore, the conduit 2 may have an inside diameter ranging from 20 mm to 60 mm, preferably of about 40 mm, affording a feed flow of polymeric material ranging from 4 to 11 kg/h.

[0040] According to a further embodiment of the invention, not shown, the
conduit 2 may comprise a plurality of substantially radial wings therein, arranged in the longitudinal direction, to cause mixing of the polymeric material M while it is being fed.

[0041] Conveniently, loading means, not shown, may be provided at the inlet station, for introducing the polymeric material M, which means may comprise a hopper with a discharge passage level with the inlet station, and means for controlling the flow rate of material into the conduit.

[0042] Blower means 6 may be also provided for blowing an inert gas into the conduit 2 to facilitate removal of the moisture that has come out of the polymeric material M upon drying.

[0043] The inert gas may be selected from the group comprising argon and nitrogen and the conduit 2, the loading station and the discharge station must be obviously isolated from the external environment, for a controlled atmosphere to be maintained therein.

[0044] Therefore, as the material M is being fed in the conduit 2, the moisture removed therefrom will not be reintroduced into the polymers, and the reaction kinetics of condensation processes will not be reversed.

[0045] The plant 1 further comprises at least one treatment zone 7 situated along the conduit 2 and radiating means 8 for emitting an alternating electromagnetic field in the radio-frequency band, for thermal treatment of the polymeric material M, such that it can be dried and polycondensed, as best shown in FIGS. 1 to 4.

[0046] The electromagnetic field may have a fixed frequency ranging from 5 MHz to 50 MHz, preferably from 25 MHz to 29 MHz, more preferably of about 27.12 MHz.

[0047] In addition to affording effective drying and solid state polycondensing of the polymeric material M, these frequency values provide higher safety as compared with plants that use different electromagnetic fields, e.g. in the microwave range.

[0048] The radiating means 8 comprise a plurality of applicators 9, located in correspondence of the treatment zone 7 outside the conduit 2 and in longitudinally offset positions, and are connected to the terminals of an
electromagnetic wave generator 10 whose power ranges from 10 kV/m to 20 kV/m.

[0049] Furthermore, the generator 10 shall be of such a size as to be able to generate an alternating current of adjustable amplitude, to thereby obtain such electromagnetic field values.

[0050] According to a peculiar aspect of the invention, the applicators comprise pairs of opposed radiating elements 11, 11', which are adapted to generate an alternating electromagnetic field in the conduit 2, with field lines F at least partially parallel to the direction of feed L of the material M and defining magnetic dipoles with opposite polarities along the conduit 2, as best shown in FIGS. 5 to 7.

[0051] Thus, the electromagnetic field so generated is able to uniformly treat the polymeric material M that is being fed along the conduit 2, with a homogeneous temperature distribution, thereby considerably reducing inherent moisture and increasing the viscosity of the material in very short times.

[0052] In a first embodiment of the invention, as shown in FIG. 2, the radiating elements 11 consist of rings 12 of conductive material, extending substantially perpendicular to the axis L of the conduit 2.

[0053] Particularly, the rings 12 are divided into a first series 13 of rings 12 connected in parallel with one of the terminals 14 of the generator 10 and a second series 15 of rings 12 connected to the other terminal 16 of the generator 10.

[0054] The rings 12 of each series 13, 15 are alternately arranged along the longitudinal extent of the conduit 2 to form respective pairs 11, 11' of radiating elements with opposite polarities.

[0055] In a second embodiment of the invention, as shown in FIG. 3 and preferably used in combination with a belt conveyor 5 as a feed means, the radiating elements 11 consist of two series 17, 18 of bars 19 made of an electrically conductive material and extending transverse to the conduit 2.

[0056] The two series 17, 18 are placed at the transversely opposite sides 20, 21 of the conduit 2 and are connected to first 14 and second 16 terminals
of the generator 10 respectively, such that the pairs 11, 11’ of radiating elements are alternately arranged in diametrically and longitudinally offset positions.

[0057] In a third embodiment of the invention, as shown in FIG. 4, the radiating elements 11 consist of longitudinally offset pairs 22 of bars 19 made of a conductive material, which are placed transverse to the conduit 2 in diametrically opposite positions.

[0058] In this embodiment, the bars 19 include a first series of pairs 22 connected, in parallel to each other, to a terminal 14 of the generator 10 and a second series of pairs 22’ connected, in parallel to each other, to the other terminal 16 of the generator 10.

[0059] Furthermore, the pairs 22, 22’ of bars 19 with opposite polarities are alternated and in longitudinally offset positions to define the pairs of radiating elements 11, 11’ with opposite polarities.

[0060] In a further aspect, as shown in FIG. 8, the invention provides a method of drying and solid state polycondensing a polymeric material M in granular form obtained by polycondensation, using the aforementioned plant 1, and comprising a step a) of providing a predetermined amount of the polymeric material M to be treated and a step b) of introducing the polymeric material M into the conduit 2.

[0061] These steps are followed by a step c) of feeding the polymeric material M along the conduit 2 to the treatment zone 7, a step d) of actuating the generator 10 and generating a radio-frequency electromagnetic field using the radiating elements 11, for thermal treatment of the material M and finally a step e) of blowing an inert gas into the conduit 2 to facilitate solid state polycondensing of the polymeric material M, in combination with the electromagnetic field.

[0062] The radiating elements 11 are designed to generate an alternating electromagnetic field in the conduit 2, with field lines F at least partially parallel to the feed direction L of the material M, and having magnetic dipoles with opposite polarities between each radiating element 11 and the one 11’ next to it.
The treatment zone 7 is maintained at such a temperature as to cause the material M to be heated to a temperature ranging from 120°C to 180°C, whereas the feed flow is adjusted for the material M to be kept within the conduit 2 for a time ranging from 30 s to 7 minutes, with an average flow rate ranging from 4 to 11 kg/h.

The following table shows the treatment conditions for the polymeric material M and the drying and solid state polycondensing results obtained for each sample.

<table>
<thead>
<tr>
<th>Product</th>
<th>Flow rate (kg/h)</th>
<th>Initial moisture (%)</th>
<th>Final moisture (%)</th>
<th>Initial temperature (°C)</th>
<th>Final temperature (°C)</th>
<th>Initial viscosity</th>
<th>Final viscosity</th>
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</thead>
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<tr>
<td>A1 - Polyamide 6</td>
<td>10</td>
<td>2.0</td>
<td>0.5</td>
<td>23°C</td>
<td>140°C</td>
<td>2.4</td>
<td>2.4</td>
</tr>
<tr>
<td>A2 - Polyamide 6</td>
<td>8</td>
<td>2.0</td>
<td>0.003</td>
<td>23°C</td>
<td>150°C</td>
<td>2.4</td>
<td>2.45</td>
</tr>
<tr>
<td>A3 - Polyamide 6</td>
<td>8</td>
<td>2.0</td>
<td>0.002</td>
<td>23°C</td>
<td>180°C</td>
<td>2.4</td>
<td>3.1</td>
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</table>

<table>
<thead>
<tr>
<th>B1 Elastomeric polyester</th>
<th>5</th>
<th>0.7</th>
<th>0.005</th>
<th>23°C</th>
<th>120°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>B2 Elastomeric polyester</td>
<td>5</td>
<td>0.7</td>
<td>0.003</td>
<td>23°C</td>
<td>130°C</td>
</tr>
<tr>
<td>C1 - PET</td>
<td>10</td>
<td>2.5</td>
<td>0.5</td>
<td>23°C</td>
<td>130°C</td>
</tr>
<tr>
<td>C2 - PET</td>
<td>8</td>
<td>2.5</td>
<td>0.003</td>
<td>23°C</td>
<td>150°C</td>
</tr>
<tr>
<td>C3 - PET</td>
<td>8</td>
<td>2.5</td>
<td>0.002</td>
<td>23°C</td>
<td>180°C</td>
</tr>
</tbody>
</table>

It shall be noted that, for all the samples being treated, the plant can ensure a considerable reduction of inherent moisture, while limiting the final temperature of the material.

It shall be further noted that, for the samples A3 and C3 that were
blown with nitrogen within the conduit, a significant increase of viscosity is observed, which indicates that solid state polycondensing has occurred.

[0067] The above disclosure shows that the plant and method of the invention fulfill the intended objects and particularly meets the requirement of affording quick and effective curing of the polymeric material.

[0068] The plant and method of the invention are susceptible to a number of changes or variants, within the inventive concept disclosed in the appended claims. All the details thereof may be replaced by other technically equivalent parts, and the materials may vary depending on different needs, without departure from the scope of the invention.

[0069] While the plant and method have been described with particular reference to the accompanying figures, the numerals referred to in the disclosure and claims are only used for the sake of a better intelligibility of the invention and shall not be intended to limit the claimed scope in any manner.

Industrial applicability

[0070] The present invention may find application in industry, because it can be produced on an industrial scale in polymer processing and recycling factories.
CLAMS

1. A plant (1) for drying and solid state polycondensing a moisture-containing polymeric material (M) in granular form, which plant (1) comprises:
   - a conduit (2) for feeding the material (M) to be treated in a longitudinal direction (L);
   - at least one treatment zone (7) for treating the polymeric material (M), which is located along said conduit (2);
   - blower means (6) for blowing an inert gas into said conduit (2);
   - radiating means (8) for emitting an alternating electromagnetic field in the radio-frequency band, for thermal treatment of the polymeric material (M), such that it can be dried and upgraded;

wherein said radiating means (8) comprise a plurality of applicators (9) located in correspondence of said treatment zone (7) and external to said conduit (2), in longitudinally offset positions, said applicators (8) being connected to the terminals of an electromagnetic wave generator (10);

characterized in that said applicators (9) comprise pairs of opposed radiating elements (11, 11') which are adapted to generate an alternating electromagnetic field in said conduit (2), with field lines (F) at least partially parallel to the direction of feed (L) of the material (M), wherein said pairs of radiating elements (11, 11') define magnetic dipoles with opposite polarities along said conduit (2).

2. A plant as claimed in claim 1, characterized in that said radiating elements (11, 11') consist of rings (12) made of a conductive material, and substantially perpendicular to the axis (L) of said conduit (2).

3. A plant as claimed in claim 2, characterized in that said rings (12) comprise a first series (13) of rings (12) connected in parallel to one of the terminals (14) of said generator (10) and a second series (15) of rings (12) connected in parallel to the other terminal (16) of said generator (10), said series (13, 15) being in alternate arrangement.

4. A plant as claimed in claim 1, characterized in that said radiating elements (11, 11') consist of pairs (22) of bars (19) made of an electrically conductive material, and extending substantially transverse to said conduit.
5. A plant as claimed in claim 4, characterized in that said pairs (22) of bars (19) comprise a first series of pairs (22) connected, in parallel to each other, to one terminal (14) of said generator (10) and a second series of pairs (22') connected, in parallel to each other, to the other terminal (16) of said generator (10), said pairs (22, 22) of bars (19) being in alternate arrangement and in longitudinally offset positions.

6. A plant as claimed in claim 1, characterized in that said radiating elements (11) consist of bars (19) made of an electrically conductive material, placed in alternate arrangement at one of the transversely opposite sides (20, 21) of said conduit (2) and in longitudinally offset relationship.

7. A plant as claimed in claim 6, characterized in that said bars (19) comprise a first series (17) of bars connected, in parallel to each other, to one terminal (14) of said generator (10) and a second series (18) of bars connected, in parallel to each other, to the other terminal (16) of said generator (10), said pairs (11, 11') of bars (19) being in alternate arrangement and in diametrically and longitudinally offset positions.

8. A plant as claimed in claim 1, characterized in that said conduit (2) comprises a side wall (3) made of a dielectric material and extending in a substantially vertical direction (L) to facilitate natural gravity feed of the material (M), said conduit (2) having an inside diameter ranging from 20 mm to 60 mm, preferably of about 40 mm.

9. A plant as claimed in claim 1, characterized in that it comprises means (4) for forced feeding of the material (M), located within said conduit (2), wherein said forced feed means (4) are selected from the group comprising augers, Archimedean screws and belt conveyors (5).

10. A plant as claimed in claim 1, characterized in that said conduit (2) comprises a plurality of substantially radial wings therein, arranged in the longitudinal direction, to cause mixing of the polymeric material (M) while it is being fed.

11. A method of drying and solid state polycondensing a polymeric material (M) in granular form obtained by polycondensation, using the plant
(1), as claimed in one or more of claims 1 to 10, which method comprises the steps of:

a) providing a predetermined amount of a polymeric material \((M)\) to be treated;

b) introducing said predetermined amount of the polymeric material \((M)\) to be treated into said conduit \((2)\);

c) feeding the material \((M)\) along said conduit to said at least one treatment zone \((7)\) equipped with a plurality of radiating elements \((1 1, 11')\);

d) actuating said generator \((10)\) and generating a radio-frequency electromagnetic field onto the polymeric material \((M)\) using said at least one applicator \((9)\) for thermal treatment of the polymeric material \((M)\);

e) blowing an inert gas into said conduit \((2)\) to facilitate solid state polycondensing of the polymeric material \((M)\), in combination with said electromagnetic field;

wherein said radiating elements \((1 1, 11')\) are designed to generate an alternating electromagnetic field in said conduit \((2)\), with field lines \((F)\) at least partially parallel to the feed direction \((L)\) of the material \((M)\), wherein said alternating magnetic field has magnetic dipoles with opposite polarities between each radiating element \((1 1)\) and the one \((1 1')\) next to it; and

wherein said treatment zone \((7)\) is maintained at an average temperature ranging from 120°C to 180°C, and

wherein the feed flow of the polymeric material \((M)\) is adjusted to keep the material \((M)\) to be treated within said conduit \((2)\) for a time ranging from 30 sec to 7 minutes with an average flow rate of the polymeric material ranging from 4 to 11 kg/h.
a) 
\[\rightarrow\] 
b) 
\[\rightarrow\] 
c) 
\[\rightarrow\] 
d) 
\[\rightarrow\] 
e) 

**FIG. 8**
**INTERNATIONAL SEARCH REPORT**

**International application No**: PCT/IB2016/05 1446

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**A. CLASSIFICATION OF SUBJECT MATTER**

| INV. | B29B9/16 | B29B13/08 | BG1J8/00 | B01J8/08 | B01J19/12
|------|-----------|-----------|----------|----------|----------
| ADD. | B29C35/08 |

According to International Patent Classification (IPC) or to both national classification and IPC

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**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

- B29B
- BG1J
- B29C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
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Further documents are listed in the continuation of Box C.

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**Date of the actual completion of the international search**: 8 June 2016

**Date of mailing of the international search report**: 23/06/2016

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**Name and mailing address of the ISA/European Patent Office, P.B. 5818 Patentlaan 2 NL-2330 HM Hilversum Tel. (+31-70) 340-2040, Fax (+31-70) 340-3016**

**Authorized officer**: Bruno l d, Axel
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<td>A</td>
<td>DE 10 2005 043526 A (SCHÖELLER PET RECYCLING GMBH [DE])</td>
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