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(54) METHOD FOR PREPARING POLYCARBONATE POWDER

(71) We, IDEMITSU KOSAN COMPANY LIMITED, a company organized and existing under the laws of Japan of No. 1-1, 3-chome, Marunouchi, Chiyoda-ku, Tokyo, Japan do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 The present invention relates to a method for continuously producing polycarbonate powder.

Prior art processes require a corresponding apparatus for each unit process such as concentration of polycarbonate solution, powdering, grinding and drying, so that they possessed such defects as difficulty in performing processes in a short period of time and moreover difficulty in preparing good quality powder, with complete elimination of solvent or moisture.

It is an object of this invention to provide an improved process for producing polycarbonate powder from polycarbonate solution in a continuous manner.

The present invention provides a process for the continuous production of polycarbonate powder which comprises heating polycarbonate solution in a receptacle and simultaneously repeatedly subjecting the solution to to-and-fro agitation provided by rotation of one or more shafts provided in the receptacle and having blades mounted thereon, each blade being set at an angle to the axis of rotation of the shaft on which it is mounted, and there being a first set of said blades arranged to direct the solution in a first direction and a second set of said blades arranged to direct the solution in an opposite direction.

Preferably said polycarbonate solution is selected from a solution of polycarbonate prepared by using 4,4' dioxydiphenylalkane and a solution of copolymer of 4,4'-dioxydiphenylalkane and halogenated 4,4'-dioxydiphenylalkane, 4,4'-dioxydiphenyl sulfone or halogenated 4,4'-dioxydiphenyl sulfone.

Preferably solvent used for dissolving said polycarbonate is selected from tetrachloroethane, trichloroethane, dichloroethane, trichloroethylene, dichloroethylene, chloroform, methylene chloride, dioxane, tetrahydrofuran, chlorobenzene, toluene, ortho-, meta- or para-chlorobenzene and mixtures thereof.

Preferably concentration of polycarbonate in the solution is 5 to 40 weight percent.

A process according to the invention preferably also comprises the steps of concentrating polycarbonate solution, powdering polycarbonate, grinding polycarbonate and drying polycarbonate, in a single processing zone provided by said receptacle.

A process as set forth in any one of the last five immediately preceding paragraphs may be carried out in an apparatus comprising an elongate receptacle having an inlet provided at or adjacent one end of the receptacle and an outlet provided at or adjacent the other end of the receptacle, said one or more shafts being mounted for rotation in the receptacle, the first set of blades being arranged to direct said solution towards the outlet, and the second set of blades being arranged to direct said solution away from the outlet whereby to mix the solution, and means for heating the solution in the receptacle.

In an apparatus as set forth in the last preceding paragraph two shafts may be provided in spaced parallel relationship to one another, each shaft being provided with a first set of blades and a second set of blades, with a blade of the first set of blades paired with a blade of the second set thereof.

In an apparatus as set forth in the last preceding paragraph the two shafts may be arranged so that the blades thereof are in complementary relationship to one another.

In an apparatus as set forth in any one of the last three immediately preceding paragraphs the blades adjacent the inlet may be predominantly blades of the first set. In an alternative form of apparatus as set forth in the last preceding paragraph but two the two shafts may be provided in spaced parallel relationship to one another, one shaft being provided with said first set of blades and the other shaft being provided with the second set of blades.

In an apparatus as set forth in the last preceding paragraph preferably the first and second sets of blades are each provided by one or more continuous blades extending helically along the respective shafts.

In an apparatus as set forth in any one of the last four immediately preceding paragraphs the two shafts may be so positioned that the blade or blades of each shaft serve to clean the blade or blades of the other shaft.

In an apparatus as set forth in any one of the last five immediately preceding paragraphs preferably the means for heating the solution comprises a jacket surrounding at least a part of the receptacle through which jacket heated gases or liquids can be passed.

There now follows a detailed description, to be read with reference to the accompanying drawings of a process and an apparatus for use in carrying out a method according to the present invention; it is to be clearly understood that these have been selected for description to illustrate the invention by way of example and not by way of limitation.

In the drawings:

Figure 1 illustrates a side view of an apparatus for use in carrying out a method according to this invention;

Figure 2 illustrates a sectional view from the front of the apparatus shown in Figure 1.

Figure 3 illustrates a schematic developmental view showing positions of blades on two shafts of the apparatus; and

Figure 4 is a perspective view of a part of the two shafts of the apparatus.

In carrying out a method of producing polycarbonate powder from polycarbonate solution, a continuous process for preparing polycarbonate powder according to the invention is provided as follows; all the processes are performed in a single zone where concentration of polycarbonate solution and powdering, grinding and drying of polycarbonate are achieved by mixing polycarbonate solution with the application of heat and simultaneously agitating the solution with a back and forth action with the aid of blades mounted on rotatable shafts, with each blade set at an angle to the axis of the shaft on which it is mounted.

A process according to the present

invention can be carried out advantageously using an apparatus comprising a horizontal receptacle having a jacketed base in which two shafts are set rotating in opposite directions to each other and are set with helically positioned blades used for causing flow of the solution back and forth within the receptacle, the jacketed base being provided for heating the solution and having an inlet for supplying polycarbonate solution into the receptacle at one end and an outlet for discharging polycarbonate powder from the receptacle at the other end. Two parallel rotatable shafts are mounted in the receptacle for rotation in opposed directions relative to each other, each shaft being provided with helically-positioned arcuate blades which adjacent the inlet end of the receptacle are so positioned as to direct solution along the receptacle towards the outlet end, and along the remainder of their length are so positioned that they cause flow of solution in both directions.

The apparatus described above is designed to transfer the fed materials through the receptacle using the afore-mentioned blades which direct the materials to and fro and thus cause forced mixing of the fed materials and discharge the product by extrusion from the outlet.

An example of such an apparatus is explained by referring to the drawings. In Figures 1 and 2, 1 indicates a receptacle which has a heating jacket forming with the receptacle bottom a jacketed base 3. Two shafts 4, 4' are mounted for rotation in the receptacle 1 adjacent the base with end portions of the shafts projecting from the ends of the receptacle. Arcuate blades 5, 5' which are described in detail later are fixed to the shafts 4, 4'. The heating jacket 2 is provided with an inlet 6 and an outlet 7 for admission and discharge respectively of a heating medium and, for the receptacle 1, an outlet 8 is provided for discharging solvent vapor.

The blades 5, 5' attached to the shafts 4, 4' respectively comprise as described above, first sets of blades 5a, 5a' for directing fed materials from the inlet 9 towards an outlet 10 and second sets of blades 5b, 5b' for directing the fed materials towards the inlet 9. Figure 3 is a schematic view of the blades attached to the shafts 4, 4' and the view is taken along the outer peripheral edges of the blades; the blades attached to the shaft 4 being indicated by solid line and the blades attached to the shaft 4' being indicated by dotted line which is developed inversely. Of those indicated by solid line or dotted line, those with a right-hand ascent are the blades for directing materials towards the outlet and those with a left-hand ascent are those for directing the materials towards the inlet.



As is shown in Figure 3, the blades on both shafts adjacent the inlet 9 are positioned to direct materials predominantly towards the outlet 10, as indicated by section I of Figure 3 which indicates the portion of the shafts adjacent the inlet. On the remainder of the two shafts as indicated by the section II of Figure 3, the blades are generally arranged to form pairs of oppositely directed blades.

As the result of the blade arrangements described above, in the section I of the receptacle all the materials are urged towards the outlet 10 whilst in the section II the simultaneous action of to and fro movement is operated, with the overall resultant flow towards the outlet 10. In the section I it is sometimes desirable to mount oppositely directed blades at appropriate positions in order to prevent holding of polycarbonate. However these blades are not essential.

In section II sufficient mixing of the polycarbonate solution fed thereto is provided, and evaporation of solvent and powdering of polycarbonate is promoted. In addition, grinding and drying of powder produced are also performed therein.

In the view shown in Figure 3, the oppositely directed blades in the section II are generally arranged to form pairs, as aforesaid, thus to complement each other through the section. However provided that almost the same numbers of oppositely directed blades are mounted in complementary relationship, the blades may be attached to the two shafts at any part of the section II. Furthermore in order to achieve this relationship, the blades for directing materials towards the outlet can be mounted on one shaft whilst the oppositely directed blades can be attached to the other shaft. Moreover, it is possible to attach oppositely directed blades alternately to one shaft and to attach similarly positioned blades to the other shaft but in complementary relationship to the blades on the first shaft.

Blades on the two shafts can be continuous, or they can as shown be provided by numerous small blades. A preferable arrangement of blades is that in which an angle subtended by each blade at the axis of its shaft is 30° to 150° and on the other shaft a similar arrangement of blades to the above is made with the blades on the two shafts in complementary relationship.

As a result of the arrangement described above, sufficient mixing is provided, and self-cleaning (prevention of adhesion) can also be provided by positioning the shafts sufficiently closely that the blades of each clean the other.

In addition, it is even more preferable to

mount blades for directing materials back towards the inlet on specific positions of the shaft. Such an arrangement provides for obtaining uniform grain size of powder even when the feeding rate of polycarbonate solution may vary. Furthermore, holding time of powder in the apparatus can be controlled and thus the mixing action can also be controlled.

As described above, the blades may be attached to the shafts continuously along the periphery thereof or by division into many small parts. That is, the angle subtended by each blade around the axis of rotation of its shaft may be more than 360° or within the range 90° to 360° and the number of blades may be as many as desired.

The angle between the general plane of each blade and a plane perpendicular to the axis of its shaft (lead angle) is preferably within the range 5° to 30° .

Spacing between the blades and the walls of the receptacle is set as small as possible (less than 20 mm) and is more preferably set at less than 10 mm.

The shafts are preferably rotated at a rotational speed of between 20 rpm and 150 rpm.

Figure 4 is a view of a part of the shafts within the section II in Figure 3. The shape resembles the threads of a right and left-handed screw which is made by cutting threads of left and right-hand on the same shaft, and as shown in Figure 3, blades for directing materials in opposed directions form a pair and are in close relationship to each other (e.g. 5a:5a' and 5b:5b').

As described above, the polycarbonate solution fed from an inlet 9 is directed towards the outlet 10 in the section I and then in the section II it is repeatedly directed back and forth but moves generally towards the outlet 10 by being pushed with materials introduced through the inlet. During this process, sufficient concentration of the solution and powdering of polycarbonate, grinding and drying processes are performed and polycarbonate powder is provided continuously from the outlet 10.

The polycarbonate solution which can be processed by carrying out a process according to the present invention is selected from, for example, a solution of polycarbonate as set out hereinbefore.

Solvents used for dissolving the polymer include tetrachloroethane, trichloroethane, dichloroethane, trichloroethylene, dichloroethylene, chloroform, methylene chloride, dioxane, tetrahydrofuran, chlorobenzene, toluene, ortho-, meta- or parachlorobenzene and mixtures of them. Polycarbonate is added to make its concentration 5 to 40 weight per cent (wt%) in these solvents.

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In carrying out a process according to the present invention, precipitants and water are not necessary and thus drying of powder is very easily accomplished. In addition, this process provides many advantages as follows: little depolymerization during subsequent extrusion of the polycarbonate, good working environment avoiding evolution of fine dust which may cause dust explosion; obtaining of powder of high bulk density suitable for extrusion and making an excellent dispersion of additives.

The present invention will now be described in detail by reference to the following example.

Example

Polycarbonate of bisphenol A was prepared by the phosgene method, and methylene chloride solution of polycarbonate having molecular weight of 27,300 was obtained. In said solution, the concentration of the polymer was 7 wt%, which was concentrated to 28 wt% by heating and evaporating the solvent. Using this polycarbonate solution, polycarbonate powder was prepared by the use of the following apparatus according to the present invention.

Into a receptacle 1 of 216 mm width and of 1050 mm length having a heating jacket 2 and jacketed base 3, two shafts 4,4' each of 60 mm in diameter were placed horizontally and on each of said shafts blades whose outer curved periphery was of 118 mm external diameter, were mounted. All the apparatus was made of stainless steel. Arrangement of the blades was as shown schematically in Figure 3.

The apparatus was heated by passing steam at 150°C through the jacket 2. Continuous feeding of polycarbonate solution at 62 kg/hr was made while rotating the shafts towards each other (as viewed from above) at 60 rpm. The polycarbonate was held in the receptacle for 12 minutes on average and subsequently was discharged from the outlet 10 as a powder.

Average grain size of polycarbonate powder thus obtained was 1,700 microns and contents of powder less than 200 mesh was less than 0.3 wt% and no clumps were observed. Bulk density was 0.62 g/cc and the methylene chloride content in the powder was 18 wt%. Any alternation in grain size, bulk density of polycarbonate powder and methylene chloride content was not observed when the feeding rate of polycarbonate solution was changed from 56 kg/hr to 68 kg/hr.

Polycarbonate powder thus obtained was dried for 4 hr at 120°C and the methylene chloride content was decreased to less than 0.5 wt% and then mixed well with octyldi-phenylphosphite (0.05 wt%) as an anti-

oxidant. The mixture was introduced into the hopper of an extruder with vent having a barrel of 65 mm in diameter and length to diameter ratio of 22 and was made into pellets after melting at a maximum temperature of 290°C. No discoloration of the pellets was observed and the antioxidant was distributed uniformly. Upon introducing the powder into a hopper, no dusting occurred and thus no difficulty of operation was observed. The molecular weight of the pellets was 27,100 and therefore depolymerization during the pelleting processes was negligible.

Polycarbonate powder was made into pellets using the same extruder with vent as described above without drying the powder, but the pellets thus obtained were uniform in grain size and had no discoloration and therefore these pellets suffered nothing by comparison with those obtained after a drying process.

WHAT WE CLAIM IS:—

1. A process for the continuous production of polycarbonate powder which comprises heating polycarbonate solution in a receptacle and simultaneously repeatedly subjecting the solution to to-and-fro agitation provided by rotation of one or more shafts provided in the receptacle and having blades mounted thereon, each blade being set at an angle to the axis of rotation of the shaft on which it is mounted, and there being a first set of said blades arranged to direct the solution in a first direction and a second set of said blades arranged to direct the solution in an opposite direction.

2. A process according to Claim 1, wherein said polycarbonate solution is selected from a solution of polycarbonate prepared by using 4,4'-dioxydiphenylalkane and a solution of copolymer of 4,4'-dioxydiphenylalkane and halogenated 4,4'-dioxydiphenylalkane, 4,4'-dioxydiphenyl sulfone or halogenated 4,4'-dioxydiphenyl sulfone.

3. A process according to either one of claims 1 and 2 wherein solvent used for dissolving said polycarbonate is selected from tetrachloroethane, trichloroethane, dichloroethane, trichloroethylene, dichloroethylene, chloroform, methylene chloride, dioxane, tetrahydrofuran, chlorobenzene, toluene, ortho-, meta- or para-chlorobenzene and mixtures thereof.

4. A process according to any one of claims 1 to 3 wherein concentration of polycarbonate in the solution is 5 to 40 weight percent.

5. A process according to claim 1 which comprises the steps of concentrating polycarbonate solution, powdering polycarbonate, grinding polycarbonate and drying polycarbonate, in a single processing zone provided by said receptacle.

(12)



In carrying out a process according to the present invention, precipitants and water are not necessary and thus drying of powder is very easily accomplished. In addition, this process provides many advantages as follows: little depolymerization during subsequent extrusion of the polycarbonate, good working environment avoiding evolution of fine dust which may cause dust explosion; obtaining of powder of high bulk density suitable for extrusion and making an excellent dispersion of additives.

The present invention will now be described in detail by reference to the following example.

Example

Polycarbonate of bisphenol A was prepared by the phosgene method, and methylene chloride solution of polycarbonate having molecular weight of 27,300 was obtained. In said solution, the concentration of the polymer was 7 wt%, which was concentrated to 28 wt% by heating and evaporating the solvent. Using this polycarbonate solution, polycarbonate powder was prepared by the use of the following apparatus according to the present invention.

Into a receptacle 1 of 216 mm width and of 1050 mm length having a heating jacket 2 and jacketed base 3, two shafts 4,4' each of 60 mm in diameter were placed horizontally and on each of said shafts blades whose outer curved periphery was of 118 mm external diameter, were mounted. All the apparatus was made of stainless steel. Arrangement of the blades was as shown schematically in Figure 3.

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Polycarbonate powder thus obtained was dried for 4 hr at 120°C and the methylene chloride content was decreased to less than 0.5 wt%, and then mixed well with octyldi-phenylphosphite (0.05 wt%) as an anti-

oxidant. The mixture was introduced into the hopper of an extruder with vent having a barrel of 65 mm in diameter and length to diameter ratio of 22 and was made into pellets after melting at a maximum temperature of 290°C. No discoloration of the pellets was observed and the antioxidant was distributed uniformly. Upon introducing the powder into a hopper, no dusting occurred and thus no difficulty of operation was observed. The molecular weight of the pellets was 27,100 and therefore depolymerization during the pelleting processes was negligible.

Polycarbonate powder was made into pellets using the same extruder with vent as described above without drying the powder, but the pellets thus obtained were uniform in grain size and had no discoloration and therefore these pellets suffered nothing by comparison with those obtained after a drying process.

WHAT WE CLAIM IS:—

1. A process for the continuous production of polycarbonate powder which comprises heating polycarbonate solution in a receptacle and simultaneously repeatedly subjecting the solution to to-and-fro agitation provided by rotation of one or more shafts provided in the receptacle and having blades mounted thereon, each blade being set at an angle to the axis of rotation of the shaft on which it is mounted, and there being a first set of said blades arranged to direct the solution in a first direction and a second set of said blades arranged to direct the solution in an opposite direction.

2. A process according to Claim 1, wherein said polycarbonate solution is selected from a solution of polycarbonate prepared by using 4,4'-dioxydiphenylalkane and a solution of copolymer of 4,4'-dioxydiphenylalkane and halogenated 4,4'-dioxydiphenylalkane, 4,4'-dioxydiphenyl sulfone or halogenated 4,4'-dioxydiphenyl sulfone.

3. A process according to either one of claims 1 and 2 wherein solvent used for dissolving said polycarbonate is selected from tetrachloroethane, trichloroethane, dichloroethane, trichloroethylene, dichloroethylene, chloroform, methylene chloride, dioxane, tetrahydrofuran, chlorobenzene, toluene, ortho-, meta- or para-chlorobenzene and mixtures thereof.

4. A process according to any one of claims 1 to 3 wherein concentration of polycarbonate in the solution is 5 to 40 weight percent.

5. A process according to claim 1 which comprises the steps of concentrating polycarbonate solution, powdering polycarbonate, grinding polycarbonate and drying polycarbonate, in a single processing zone provided by said receptacle.



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- 5 6. A process according to claim 1 substantially as hereinbefore described with reference to the Example.
7. A process according to any one of the preceding claims when carried out in an apparatus comprising an elongate receptacle having an inlet provided at or adjacent one end of the receptacle and an outlet provided at or adjacent the other end of the receptacle, said one or more shafts being mounted for rotation in the receptacle, the first set of blades being arranged to direct said solution towards the outlet, and the second set of blades being arranged to direct said solution away from the outlet whereby to mix the solution, and means for heating the solution in the receptacle.
- 10 8. A process according to claim 7 wherein two shafts are provided in spaced parallel relationship to one another, each shaft being provided with a said first set of blades and a said second set of blades, with a blade of the first set of blades paired with a blade of the second set thereof.
- 15 9. A process according to claim 8 wherein the two shafts are arranged so that the blades thereof are in complementary relationship to one another.
- 20 10. A process according to any one of claims 7 to 9 wherein the blades adjacent the inlet are predominantly blades of the first set.
11. A process according to claim 7 wherein two shafts are provided in spaced parallel relationship to one another, one shaft being provided with said first set of blades and the other shaft being provided with the second set of blades.
12. A process according to claim 11 wherein the first and second sets of blades are each substituted by one or more continuous blades extending helically along the respective shafts.
13. A process according to any one of claims 8 to 12 wherein the two shafts are so positioned that the blade or blades of each shaft serve to clean the blade or blades of the other shaft.
14. A process according to any one of claims 8 to 13 wherein the means for heating the solution comprises a jacket surrounding at least a part of the receptacle through which jacket heated gases or liquids can be passed.
15. A process according to any one of the preceding claims when carried out in an apparatus substantially as hereinbefore described with reference to the accompanying drawings.

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