PROCESS FOR MANUFACTURE OF A POLYETHYLENE TEREPTHALATE IN FINELY DIVIDED FORM


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3 Claims. (Cl. 260—75)

This invention relates to synthetic polyesters and more particularly it relates to a process for the manufacture of synthetic polyesters in a finely divided form.

The addition of water to solutions of synthetic polyesters in water miscible solvents results in precipitation of the synthetic polyester in a form which is not finely divided, it is chiefly fibrous in character. It is difficult to convert such a fibrous precipitate into a finely divided form by milling. Processes which have hitherto been used for the production of synthetic polyesters in a finely divided form involve heating the polyesters with organic solvents at high temperatures and subsequently cooling the solution. Such processes tend to degrade and discolor the synthetic polyester. Furthermore it is difficult to control such processes in such a way as to produce particles of uniform size.

According to the invention there is provided a process for the manufacture of synthetic polyesters in a finely divided form which comprises adding a dilute aqueous solution of a halogenated lower fatty acid to a solution of the synthetic polyester in a halogenated lower fatty acid or in a concentrated aqueous solution of a halogenated lower fatty acid and isolating the finely divided synthetic polyester which is precipitated.

As an example of a synthetic polyester which may be used in the process of the invention there may be mentioned polyethylene terephthalate.

When a concentrated aqueous solution of a halogenated lower fatty acid is used for dissolving the polyester this should contain not less than 80% by weight of the halogenated lower fatty acid and preferably not less than 90% of the halogenated lower fatty acid. The dilute aqueous solution of the halogenated lower fatty acid used for precipitating the polyester should contain not more than 50% by weight of the halogenated lower fatty acid and preferably between 20% and 40% by weight of the halogenated lower fatty acid.

The amount of the dilute aqueous solution of the halogenated lower fatty acid required for precipitating the synthetic polyester from the solution in the halogenated lower fatty acid is such as to give a mixed aqueous solution of the halogenated lower fatty acid in which the synthetic polyester is not appreciably soluble. The most satisfactory results are obtained when the mixed aqueous solution of the halogenated lower fatty acid contains between 50% and 65% by weight of the halogenated lower fatty acid.

The process of the invention is conveniently carried out at about 30° C. (although temperatures up to 50° C. may be used if desired) by dissolving a synthetic polyester in a halogenated lower fatty acid or in a concentrated aqueous solution of a halogenated lower fatty acid containing not less than 80% by weight of a halogenated lower fatty acid, adding such an amount of a dilute aqueous solution of a halogenated lower fatty acid, containing between 20% and 40% by weight of a halogenated lower fatty acid, that the resulting mixed solution contains between 50% and 65% by weight of the lower fatty acid, filtering off the finely divided synthetic polyester which is precipitated, washing with water and, if desired, drying the product.

As examples of suitable halogenated lower fatty acids which may be used in the process of the invention there may be mentioned dichloracetic acid, trichloracetic acid, chloroformic acid and dichloropropionic acid. The halogenated lower fatty acid used need not be pure, and may, for example contain a small amount of the corresponding lower fatty acid. If desired mixtures of the halogenated lower fatty acids may be used, for example dichloracetic acid containing monochloro and dichloroacetic acid, the said mixture being obtained by chlorinating acetic acid. Such mixtures may also contain minor amounts of the parent lower fatty acid.

It is preferred to use those halogenated lower fatty acids or mixtures thereof which are liquid at ordinary temperatures, but halogenated lower fatty acids or mixtures thereof which are solid at ordinary temperatures may be used by warming the said acids until they are in a liquid state.

The finely divided synthetic polyester obtained by the process of the invention consists of small particles of uniform size, and when dry the product may be used as a moulding powder. It may also be used, preferably before drying, for incorporation with pigments. Pigment mixtures so obtained are of value for the mass colouration of synthetic polyesters and polyamides and may also be used for the preparation of coating compositions for application to paper or textiles or for the colouration of polymeric materials suitable for moulding or extrusion.

The process of the invention does not produce any appreciable deterioration in the colour of the synthetic polyester nor result in any marked degradation of the synthetic polyester.

The invention is illustrated but not limited by the following examples in which the parts and percentages are by weight:

Example 1

To a solution of 60 parts of polyethyleneterephthalate in 780 parts of 80% trifluoroacetic acid at 30° C. there are added with stirring 1025 parts of a dilute aqueous solution of dichloracetic acid containing 21% of dichloroacetic acid. The mixture is stirred for 2½ hours, then allowed to stand for 16 hours and the polyethyleneterephthalate which is precipitated in the form of fine particles is filtered off and washed with 10,000 parts of water.

The aqueous paste, which contains 15% of solids, is then dried at between 50° and 100° C. Polyethylene terephthalate is obtained in the form of a white powder of small particle size.

Example 2

To a solution of 60 parts of polyethyleneterephthalate in a mixture containing 78 parts of acetic acid, 102 parts of trichloroacetic acid and 600 parts dichloroacetic acid at 30° C. there are added with stirring 1025 parts of a dilute aqueous solution of acetic acid containing 3.1% of acetic acid, 4% trichloroacetic acid and 23.9% dichloroacetic acid. The mixture is stirred for 2½ hours, then allowed to stand for 16 hours and the polyethyleneterephthalate which is precipitated in the form of fine particles is filtered off and washed with 10,000 parts of water.

The aqueous paste, which contains approximately 15% of solids is suitable for milling with pigments to produce pigment compositions which may be used in the mass colouration of polyethylene terephthalate.

Example 3

To a solution of 60 parts of polyethyleneterephthalate in 780 parts of 80% trichloroacetic acid at 20° C. there
are added with stirring 1200 parts of a dilute aqueous solution of trifluoroacetic acid containing 20% of trifluoroacetic acid. The mixture is stirred for 2½ hours, then allowed to stand for 16 hours and the polyethylene terephthalate which is precipitated in the form of fine particles is filtered off and washed with 10,000 parts of water.

The aqueous paste is suitable for milling with pigments to produce pigment compositions which may be used in the mass colouration of polyethylene terephthalate.

What we claim is:

1. A process for the manufacture of polyethylene terephthalate in a finely divided particulate form which comprises adding a dilute aqueous solution of a halogenated acetic acid containing between 20% and 40% by weight of the halogenated acetic acid to a solution of polyethylene terephthalate in a solvent selected from the group of solvents consisting of halogenated acetic acids and concentrated aqueous solutions of halogenated acetic acids containing not less than 80% by weight of halogenated acetic acid, said dilute aqueous solution being added to said polyethylene terephthalate solution in an amount so that the resulting mixture contains between 50% and 65% by weight of halogenated acetic acid, and thereafter isolating the finely divided particulate polyethylene terephthalate which is precipitated.

2. A process for the manufacture of polyethylene terephthalate in a finely divided particulate form which comprises adding a dilute aqueous solution of a halogenated acetic acid containing between 20% and 40% by weight of the halogenated acetic acid to a solution of polyethylene terephthalate in a solvent selected from the group of solvents consisting of halogenated acetic acids and concentrated aqueous solutions of halogenated acetic acids containing not less than 90% by weight of halogenated acetic acids, said dilute aqueous solution being added to said polyethylene terephthalate solution in an amount so that the resulting mixture contains between 50% and 65% by weight of halogenated acetic acid, and thereafter isolating the finely divided polyethylene terephthalate which is precipitated.

3. Process according to claim 1 wherein the halogenated acetic acid is a mixture obtained by chlorinating acetic acid.

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