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(54) **PROCESS FOR SEPARATING POLYESTER FROM OTHER MATERIALS**

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See application file for complete search history.

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(57) **ABSTRACT**

The present invention is directed to a process for separating polyester, and particularly polyethylene terephthalate, from various contaminants and impurities. In general, the process includes the steps of mixing materials containing polyester with an alkaline composition in a mixer. The mixer imparts sufficient energy to the slurry to provide substantially complete, even coating of the polyester containing materials with the alkaline composition and cause saponification of the outer surface of the polyester with the alkaline composition. After reaction in the mixer, the mixture is heated causing some of the impurities to be chemically modified into a more separable form. After heating, the mixture can be washed with water and the polyester can be easily separated from the remaining impurities. Through the process of the present invention, polyester can be separated and recovered from waste materials such as those containing polyvinyl chloride and aluminum. The process is also effective at removing coatings adhered to the polyester and at removing entrained organic and inorganic compounds contained within the polyester.

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PROCESS FOR SEPARATING POLYESTER FROM OTHER MATERIALS

CROSS REFERENCE TO RELATED APPLICATION

This application claims priority to an earlier filed U.S. provisional patent application Ser. No. 60/299,273 filed Jun. 19, 2001 entitled, "Process for Separating Polyester from other Materials."

BACKGROUND OF THE INVENTION

Polyesters are polymeric materials made from the esterification of polybasic organic acids with polyhydric acids. Perhaps the most commonly made and used polyester is polyethylene terephthalate (PET), which can be manufactured by reacting terephthalic acid with ethylene glycol.

Polyesters are currently being used in increasing amounts in various applications. For instance, polyesters are commonly used to make all types of containers such as beverage and food containers, photographic films, X-ray films, magnetic recording tapes, electrical insulation, surgical aids such as synthetic arteries, fabrics and other textile products, and other numerous items.

Because polyesters can be remelted and reformed, many efforts are underway to recycle as much polyester as possible after use. Before polyesters can be recycled, however, it is necessary to separate the post-consumer polyesters from other products and materials that may be found mixed with or coupled with the polyester. Unfortunately, many problems have been encountered in attempting to separate polyester from other waste materials. In particular, many prior art processes are not capable of efficiently or economically recovering polyester when a significant amount of impurities and contaminants are present. Most prior art processes for separating polyesters from other materials have been limited to floatation separation techniques and mechanical recovery processes.

In floatation separation techniques, polyesters are separated from other materials based on density differences. For instance, materials containing polyester can be combined with water, in which polyester is known to sink. The less dense materials, which float in water, can thus be easily separated from the submerged polyester. This procedure is relatively simple and very effective in separating polyesters from specific low density impurities. Floatation separation techniques, however, cannot be used if the polyester is found in combination with materials that sink in water or that have densities comparable to that of polyester.

For instance, post consumer polyester is typically mixed with polyvinyl chloride (PVC) and aluminum, which are not water buoyant. In fact, PVC has a density that is very similar to the density of PET and is often misidentified as PET. Both aluminum and PVC must be separated from polyester before it can be reused. In particular, if PET and PVC are remelted together, hydrochloric acid gases are produced which destroy the properties of the resulting plastic material.

In the past, in order to separate PET from PVC using a floatation separation technique in a specific gravity bath, others have attempted to modify the surface of the PVC so that the PVC will float in an aerated aqueous medium. For instance, in U.S. Pat. No. 5,234,110 to Kobler and U.S. Pat. No. 5,120,768 to Sisson various processes for separating PET from PVC flakes are disclosed. In these processes, the

when placed in an aqueous medium. In order for these processes to be efficient, however, the PVC flakes must have a high surface area to volume ratio. Consequently, the above processes are deficient in separating PVC chips from PET when the PVC chips have a large interior volume.

Besides failing to separate polyesters from heavier-than-water impurities, floatation separation techniques also fail to remove coatings that are commonly adhered to polyester. For example, polyester containers are commonly coated with vapor barrier coatings, saran coatings, and/or inks.

Mechanical recovery processes as used herein are washing processes used to strip specific binder and adhesive layers off polyester films without substantial reaction occurring between the polyester and the wash solution. For example, U.S. Pat. Nos. 5,286,463 and 5,366,998 both to Schwartz, Jr., both of which are incorporated herein by reference thereto, disclose a composition and process for removing adhesives, particularly polyvinylidene halide and polyvinyl halide based resins, from polyester films, such as photographic films. In one embodiment, the polyester films are mixed with a reducing sugar and a base to remove the adhesive polymeric resin from the film. An acid is then added to precipitate the resin, which can then be separated from the polyester film.

U.S. Pat. No. 4,602,046 to Buser et al. discloses a method for the recovery of polyester from scrap material such as photographic film having a polyester base and at least one layer of macromolecular organic polymer. Specifically, scrap material is cut or chopped into small individual pieces or flakes and treated in a caustic alkaline solution at a solids level of at least 25% by volume and under conditions of high shear. The organic polymer coating material is removed from the polyester flakes. The polyester flakes are then separated from the polymer coating material by filtration or centrifugation, rinsed in water, and dried. The recovered polyester flakes can be used as a feed stock for making films, bottles or other polyester articles. A method and apparatus for recovering silver and plastic from used film is also disclosed in U.S. Pat. No. 4,392,889 to Grout. In this method, the used film is first passed through a bath preferably comprising a hot caustic solution for precipitating silver layered on the film. The film then passes through a second bath of hot caustic until an adhesive sheet disposed on the film has been dissolved. Typically, the adhesive sheet is made of polyvinylidene chloride, which adheres the silver to the film. After a second caustic bath, the film is dried and available for use.

Other processes for recovering polyester from photographic films are disclosed in U.S. Pat. No. 3,928,253 to Thornton et al., U.S. Pat. No. 3,652,466 to Hittel et al., U.S. Pat. No. 3,647,422 to Wainer, and U.S. Pat. No. 3,873,314 to Woo et al.

As shown above, mechanical recovery processes have generally been limited to use with photographic films. In recycling the photographic films, silver is also recovered making the processes economically viable. Mechanical recovery processes, although very successful at removing the emulsion-type coatings found on photographic films, have generally not been successful in removing other types of coatings from polyesters. For instance, most of these processes are not capable of efficiently removing some of the vapor barrier coatings and inks that are applied to polyesters.

Other contaminants that are generally not removable from polyesters using floatation separation techniques and mechanical recovery processes as described above are

toluene, pesticides and other compounds that are absorbed by polyesters when placed in contact therewith. If the entrained organic and inorganic compounds are not substantially removed from the polyester materials during recycling, the recycled polyesters cannot be used as food containers or as beverage containers.

Because of the above noted deficiencies in prior art processes, large amounts of recyclable polyesters are being scrapped and loaded into landfills or are being incinerated. Unfortunately, not only is the polyester not being reused, but the polyester materials are creating a waste management and disposal problem.

Recently, the focus of recovering polyester from the waste stream has changed from mechanical washing processes to chemically converting the polyester into usable chemical components. For instance, in U.S. Pat. No. 5,958,987, 6,147,129, and 6,197,838, all to Schwartz, Jr., which are incorporated herein by reference thereto, processes for recycling polyesters in which a portion of the polyesters are reduced to their original chemical reactants are disclosed. The processes include the steps of combining the polyester materials with an alkaline composition to form a mixture. The mixture is heated to a temperature sufficient to convert the surface of the polyester to an alkaline salt of a polybasic organic acid and a polyol.

The above described patents represent great advancements in the art. The process of the present invention is directed to further improvements in processes for recycling polyesters.

SUMMARY OF THE INVENTION

In general, the process of the present invention is directed to a process for separating a polyester substrate from various contaminants and impurities. For example, the process of the present invention can be used to release various contaminants from a polyester substrate to which the contaminants have been coupled, i.e. either adhered or entrained contaminants. Additionally, the process of the present invention can facilitate the separation of polyester from other contaminants which can be mixed with polyester in the waste stream; aluminum and polyvinyl chloride, for example.

The process of the present invention generally includes mixing materials containing polyester with an alkaline composition to form a slurry. The slurry can then be mixed in a high energy mixer which can not only substantially and evenly coat the materials with the alkaline composition, but can also impart sufficient energy so as to promote reaction between the materials and the alkaline composition which can enable separation of the polyester from various contaminants and impurities. For example, the slurry can be mixed in a high energy plow mixer. In one embodiment, the mixer can operate at a Froude number greater than about 4.2 to promote the reaction. Specifically, the mixer can operate at a Froude number greater than about 6.6. In one embodiment, the mixer can operate at a Froude number greater than about 9.5.

In general, the alkaline composition can be a metal hydroxide solution. For example, the metal hydroxide can be sodium hydroxide, calcium hydroxide, potassium hydroxide, lithium hydroxide, magnesium hydroxide, or mixtures thereof. In one embodiment, the alkaline composition can be formed of only sodium hydroxide and water. For example, the alkaline composition can be sodium hydroxide and water in a 1:1 ratio.

the polyester as possible and yet still promote separation of the polyester from any contaminants or impurities. For example, the alkaline composition can be combined with the polyester materials in an amount of less than about 10% by weight of the polyester materials. Specifically, the alkaline composition can be combined with the polyester materials in a stoichiometric amount sufficient to react with less than about 20% of the polyester. More specifically, the alkaline composition can be combined with the polyester materials in a stoichiometric amount sufficient to react with less than about 10% of the polyester.

The reaction process can substantially exhaust the metal hydroxide in the mixer. For example, the metal hydroxide remaining in the slurry after the mixing and reaction process can generally be less than about 1% of the weight of the slurry. Specifically, the metal hydroxide remaining in the slurry after the mixing and reaction process can generally be less than about 0.5% of the weight of the slurry. More specifically, the metal hydroxide remaining in the slurry after the mixing and reaction process can generally be less than about 0.1% of the weight of the slurry.

If desired, the slurry can be heated after the reaction in the mixer is substantially complete. For example, the slurry can be heated to a first temperature, such as, for example, a temperature of between about 120° C. and about 170° C., to dry the slurry and produce a dry product, and then heated to a second temperature, such as, for example a temperature of between about 200° C. and about 240° C., which can further degrade the contaminants and make them easier to separate from the polyester substrate.

In one embodiment, the polyester containing materials can include contaminants which are coupled to the polyester substrate, such as contaminants which are entrained in the polyester or contaminants which are adhered to the surface of the polyester. In this embodiment, the alkaline composition can react with the materials during mixing and cause saponification of a portion of the polyester which can release the contaminants from the surface of the polyester substrate.

Alternatively, the polyester containing materials can include impurities or contaminants which are mixed with the polyester, though not necessarily coupled to the polyester, such as polyvinyl chloride or aluminum materials, for example. In this embodiment, the alkaline composition can react with the contaminant and cause them to be altered into a form which is more easily separable from the polyester. For example, polyvinyl chloride can be dechlorinated by the alkaline composition in which form it can be easily separated from the polyester substrate.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

The present invention is generally directed to a process for recovering and separating polyesters from various contaminants and impurities. For instance, through the process of the present invention, various contaminants can be released from a polyester substrate such as various coatings including vapor barrier coatings, inks, and saran coatings as well as other contaminants which are entrained within the outer surface of the polyester substrate, such as various volatile organic and inorganic contaminants. During the process, the polyester can be partially saponified but largely remain in polymer form and the contaminants can be physically released from the polyester substrate.

The process is also directed to the separation and recovery

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the process, the impurities can be converted into a form which is more easily separable from the polyester substrate.

As used herein, a polyester is defined as an esterification or reaction product between a polybasic organic acid and a polyol. It is believed that any known polyester or copolyester may be used in the process of the present invention. The process of the present invention is particularly directed to a class of polyesters referred to herein as polyol polyterephthalates, in which terephthalic acid serves as the polybasic organic acid.

As used herein, a polybasic organic acid refers to any organic acid having two or more carboxyl groups (—COOH). Most polyesters are derived from dibasic acids or, in other words, from dicarboxylic acids. Polybasic acids can have a linear or a cyclic conformation. Examples of linear polybasic acids that can be used to make polyesters include the aliphatic dicarboxylic acids. In particular the aliphatic dicarboxylic acids having up to ten carbon atoms in their chains can be used. These acids include adipic acid, glutaric acid, succinic acid, malonic acid, oxalic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, maleic acid, and fumaric acid.

Cyclic polybasic organic acids, on the other hand, include the carbocyclic dicarboxylic acids. These acids are known as phthalic acid, isophthalic acid, and terephthalic acid. In particular, terephthalic acid is used to make polyethylene terephthalate, which is perhaps the most commercially available polyester.

As described above, a polybasic organic acid is combined with a polyol to produce a polyester. Polyols are compounds that contain at least two hydroxyl groups. Many polyesters are synthesized using a polyol that contains two hydroxyl groups, which are referred to as diols. Diols are normally prepared from an alkene by the net addition of two hydroxy groups to the double carbon bond in a method known as hydroxylation. Polyols are commonly referred to as glycols and polyhydric alcohols. Examples of polyols used to make polyesters include ethylene glycol, propylene glycol, butylene glycol, and cyclohexane dimethanol.

For exemplary purposes, the following table contains a nonexhaustive list of commercially available polyesters that may be recovered and recycled according to the present invention. For each polyester, the corresponding polybasic organic acid and polyol are provided.

POLYESTER	POLYBASIC ORGANIC ACID	DIOL
Polyethylene Terephthalate	Terephthalic Acid	Ethylene Glycol
Polybutylene Terephthalate	Terephthalic Acid	Butylene Glycol
PETG Copolyester	Terephthalic Acid	Cyclohexane-dimethanol and Ethylene Glycol
PBTG Copolyester	Terephthalic Acid	Cyclohexane-dimethanol and Butylene Glycol
Polycyclohexane-dimethanol Terephthalate	Terephthalic Acid	Cyclohexane-dimethanol
PEN Polyester	Napthalene Dicarboxylic Acid	Ethylene Glycol

In general, the process of the present invention includes

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mixer. The mixer chosen is one which can not only provide substantially complete and even coating of the polyester materials by the alkaline solution, but also can impart sufficient energy to cause a portion of the polyester to saponify, or, in other words, to hydrolyze. During saponification, various coatings that may be adhered to the polyester and/or other contaminants which may be entrained within the surface of the polyester can be released from the polyester. The energy provided from the mixer can also promote reaction between the alkaline solution and other impurities which can be mixed in with the polyester in the slurry, such as polyvinyl chloride or aluminum, for example, such that the impurities can be converted to another form, one which is more easily separable from the polyester substrate. After the reaction within the mixer, the slurry can then be heated, usually in a two-step heating process.

The process of the present invention can run continuously or can be set up as a batch system. Practically any material containing a polyester can be processed by the present invention including but not limited to, for example, beverage and food containers, photographic and X-ray films, recording tapes, insulation fibers, textile fibers and other products. Preferably, the polyester materials are recovered from the solid waste stream, thus alleviating many environmental concerns and disposal problems. The present invention is particularly directed to recycling food containers and beverage containers made from PET. Through the process of the present invention, polyesters can be separated, recovered and reused from post consumer waste, even when the polyesters are found mixed with polyvinyl chloride or aluminum, adhered to various coatings, or entrained with various organic and inorganic compounds. Such materials are currently being disposed of in landfills or are being incinerated after use due to a lack of an economical process that will recover the polyester.

Prior to being contacted with the alkaline composition, the materials containing the polyester can be, if desired, chopped or ground into a particular size. Sizing of the materials is done solely for the purpose of facilitating handling. Generally speaking, the larger the size of the materials and the less surface area to volume ratio, the less saponification of the polyester will occur later in the process. Consequently, smaller dimensions should be avoided and the size of the materials should be left as large as practicable. However, it should be understood that all different sizes and shapes of material may be used within the process of the present invention and no one size or shape is required.

Also prior to being combined with the alkaline composition, the materials containing the polyester can be immersed in water or some other fluid in order to separate the less dense or lighter materials from the heavier materials containing the polyester. More particularly, it is known that polyester sinks in water while paper products and other polymers, such as polyolefins, are water buoyant. Thus, the lighter materials can be easily separated from the heavier materials when contacted with a fluid. Subjecting the materials to a sink/float separation step prior to contacting the materials with the alkaline composition not only can reduce the quantity of materials being processed but also can clean the surface of the materials prior to further processing.

After being sized and subjected to a sink/float separation, if desired, the polyester containing materials can be combined and mixed with an alkaline composition to form a slurry, or mixture. Preferably, an alkaline solution can be combined with the materials so as to coat the material

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