

[54] **METHODS OF RECOVERING WASTE CELLULOSIC FIBERS**

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[56] **References Cited**

UNITED STATES PATENTS

2,832,663 4/1958 Drelich 8/141

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[57] **ABSTRACT**

Methods of recovering waste cellulosic fibers from

mixtures of waste cellulosic fibers, waste polyester and/or acrylic fibers, and synthetic, cross-linked resin materials which comprise: heating a mixture of waste cellulosic fibers, waste polyester and/or acrylic fibers, and synthetic, cross-linked resin materials within the range of from about 212° to about 275°F. for a period of from about ¼ hour to about 5 hours in an aqueous treating solution containing an alkali metal hydroxide and one or more added normally liquid chemical agents such as ketones, alcohols, lactones, and sulfoxides, which initiate the decomposition or solubilization of the waste polyester and/or acrylic fibers and the synthetic, cross-linked resin materials; adding a neutral or alkaline oxidizing agent to the mixture of waste fibers and synthetic, cross-linked resin materials; heating the mixture of waste fibers and synthetic, cross-linked resin materials in the presence of the neutral or alkaline oxidizing agent to complete the decomposition or solubilization of the waste polyester and/or acrylic fibers and the synthetic, cross-linked resin materials; and recovering the waste cellulosic fibers.

14 Claims, No Drawings

METHODS OF RECOVERING WASTE CELLULOSIC FIBERS

The present invention relates to methods of recovering waste cellulosic fibers from mixtures of waste cellulosic fibers, waste polyester and/or acrylic fibers, and synthetic, cross-linked resin materials.

BACKGROUND OF THE INVENTION

In various industries such as the textile, leather, paper, paper products, and like industries, there is a need to recover cellulosic or other fibers which have been coated, saturated, or impregnated with resins. This need to recover cellulosic or other fibers and recycle them grows increasingly important with the ever-growing awareness of our basic ecological requirements.

In the following specification, the present invention will be described in particularity with reference to the recovery of cellulosic or other fibers used in the manufacture of nonwoven fabrics in the textile industry. This, however, is merely illustrative and the broader aspects of the inventive concept are not to be construed as limited thereto.

One of the conventional commercial methods of making nonwoven fabrics is to prepare a relatively flat, fibrous web of several thicknesses or layers primarily of cellulosic fibers which are arranged generally in parallel or carded fashion, or distributed in random haphazard array. Other fibers such as polyester fibers and/or acrylic fibers including modacrylic fibers are frequently included for various purposes in order to obtain special properties and characteristics.

These fibrous layers are then bonded, either in overall fashion or in intermittent print patterns, with an adhesive bonding agent or a synthetic resin, to form an integral, self-sustaining nonwoven fabric. At the end of the manufacturing operation, the nonwoven fabric is usually trimmed to a desired predetermined width, thus creating two relatively narrow edge strips of "trim waste." These strips of trim waste represent only a small proportion of the total finished nonwoven fabric, but ultimately accumulate to very considerable amounts and are well worth recovering and recycling, not only from an ecological viewpoint but also for economic reasons.

One very large class of synthetic resins used for bonding nonwoven fabrics comprises the polymers and copolymers of vinyl esters, of which polyvinyl acetate is presently the most important single member. Fortunately, these polyvinyl ester resins are susceptible to decomposition, solubilization, or saponification treatments with caustic and the fibers bonded thereby are recoverable by known methods.

Another large class of synthetic resins used for bonding nonwoven fabrics comprises the polymers and copolymers of vinyl halides, of which polyvinyl chloride is presently the most important single member. Fortunately, these polyvinyl chloride resins are susceptible to decomposition, solubilization, or saponification treatments with caustic and added ketones or alcohols (see U.S. Pat. No. 2,832,663 which issued Apr. 29, 1958) and the fibers bonded thereby are also recoverable.

In recent years, however, another class of synthetic resins have become very important commercially for bonding nonwoven fabrics. These resins are the self-cross-linkable acrylic resins which, unfortunately, are

not susceptible to known decomposition or solubilization treatments, either with caustic alone or with caustic and added chemicals such as ketones, alcohols, lactones, or sulfoxides.

Specifically, when nonwoven fabric "trim waste" which has been bonded with a well-cured, self-cross-linking acrylic resin is boiled under pressure at elevated temperatures in a dilute caustic-ketone, alcohol, lactone, or sulfoxide solution for a long period of time, the resin is attacked to some degree but is not completely decomposed or made soluble. The resin does break down to some degree but the resulting product is a viscous, slimy jelly which clings tenaciously to the fibers and does not wash out. If dried, the jelly-covered fibers become a tightly bonded unworkable, stiff mass of fibers and resin.

The problem is rendered all the more acute when the fibers used in the manufacturing process comprise a blend or mixture of cellulosic fibers, such as cotton or rayon, and other fibers selected from the group consisting of polyester fibers, acrylic fibers, modacrylic fibers, and blends or mixtures thereof.

It is therefore a principal purpose of the present invention to provide methods of recovering waste cellulosic fibers from mixtures of waste cellulosic fibers, waste polyester and/or acrylic fibers, and synthetic, cross-linked resin materials without degrading the cellulosic fibers to a degree whereby their usefulness for recycling and reuse in textile or other processes for the production of fabrics or other fibrous products is not impaired.

STATEMENT OF THE INVENTION

It has been discovered that such principal purpose and other purposes to be described hereinafter can be accomplished by heating the mixture of waste cellulosic fibers, waste polyester and/or acrylic fibers, and synthetic, cross-linked resin materials within the range of from about 212°F. to about 275°F. for a period of from about ¼ hour to about 5 hours in an aqueous treating solution containing an alkali metal hydroxide and a normally liquid ketone, alcohol, lactone, or sulfoxide, to initiate the decomposition or solubilization of the waste polyester and/or acrylic fibers and the synthetic, cross-linked resin materials; adding a neutral or alkaline oxidizing agent to the mixture of waste cellulosic fibers and partially decomposed or solubilized polyester and/or acrylic fibers and synthetic, cross-linked resin materials; heating the waste fibers and synthetic, cross-linked resin materials in the presence of the neutral or alkaline oxidizing agent to complete the decomposition or solubilization of the polyester and/or acrylic fibers and the synthetic, cross-linked resin; and recovering the waste cellulosic fibers.

GENERAL DESCRIPTION OF THE INVENTION

In the following specification, there are described preferred embodiments of the invention, but it is to be understood that the inventive concept is not to be considered limited to the specific embodiments disclosed except as determined by the scope of the appended claims.

THE FIBERS

The fibers which form the recoverable materials of the present inventive concept are primarily of cellulosic nature, such as cotton or rayon (viscose or regen-

erated cellulose). Other recoverable fibers, however, which are capable of resisting the chemical treatment described herein, without excessive degradation, decomposition, or solubilization, may also be applicable and may be included. Examples of such other applicable chemically more resistant fibers include nylon polyamide 6/6 and 6, the polyolefins such as polyethylene, etc.

The other fibers in the mixture of waste fibers which are not recovered are selected from the group consisting of polyester fibers and acrylic fibers. As used herein, the term polyester fibers includes manufactured fibers in which the fiber-forming substance is any long chain synthetic polymer composed of at least 85 percent per weight of an ester of a dihydric alcohol, such as ethylene glycol, and terephthalic acid. Examples of polyester fibers are "Dacron," "Encron," "Fortrel," "Kodel," etc. These are normally ethylene glycol-terephthalic acid polyesters but in a few special cases are other polyesters, such as "Kodel 211," which is poly-1, 4-cyclohexylene dimethylene terephthalate. The acrylic fibers may have either the normal acrylic form composed of at least 85 percent by weight of acrylonitrile units or may have the modacrylic form composed of less than 85 percent by weight but at least 35 percent by weight of acrylonitrile units. Examples of acrylic fibers are "Acrilan," "Creslan," "Orlon," etc. Examples of modacrylic fibers are "Dynel," "Verel," etc.

THE RESINS

The synthetic resin may be one or more of a relatively large group of synthetic resins well known in industry and may be of a self-cross-linking type or an externally cross-linking type. Specific examples of such synthetic cross-linkable resins include: (1) polymers and copolymers of vinyl halides such as plasticized and unplasticized polyvinyl chloride, polyvinyl chloride-polyvinyl acetate, polyvinyl chloride-methyl acrylate, ethylene-vinyl chloride, etc.; vinylidene polymers and copolymers, such as polyvinylidene chloride, polyvinylidene chloride-vinyl chloride, polyvinylidene chloride-ethyl acrylate, polyvinylidene chloride-vinyl chloride-acrylonitrile, etc.; (2) polymers and copolymers of vinyl esters such as plasticized and unplasticized polyvinyl acetate, ethylene-vinyl acetate, acrylic-vinyl acetate, etc.; (3) polymers and copolymers of the polyacrylic resins such as ethyl acrylate, methyl acrylate, butyl acrylate, ethyl-butyl acrylate, ethyl hexyl acrylate, hydroxyethyl acrylate, dimethyl amino ethyl acrylate, etc.; (4) polymers and copolymers of the polymethacrylic resins such as methyl methacrylate, ethyl methacrylate, isopropyl methacrylate, butyl methacrylate, etc.; (5) nitrogen-containing polymers and copolymers of acrylonitrile, methacrylonitrile, acrylamide, N-isopropyl acrylamide, N-methylol acrylamide, methacrylamide, etc.; (6) copolymers of polyolefinic resins including ethylene-vinyl chloride and ethylene-vinyl acetate which have been listed previously; etc.

These resins may be present either as homopolymers comprising a single repeating monomer unit, or they may be used as copolymers comprising two, three, or more different monomer units which are arranged in random fashion, or in a definite ordered alternating fashion, within the polymer chain. Also included within the inventive concept are the block polymers comprising relatively long blocks of different monomer units in

a polymer chain and graft polymers comprising chains of one monomer attached to the backbone of another polymer chain.

THE INITIAL HEATING STEP

The mixture of waste fibers and the synthetic cross-linked resin materials are initially heated with or without pressure at elevated temperatures within the range of from about 212° (with or without pressure) and 275°F. (with pressure) for a period of from about ¼ hour to about 5 hours in an aqueous treating solution containing an alkali metal hydroxide and a normally liquid organic compound such as a ketone, an alcohol, a lactone, or a sulfoxide.

The alkali metal hydroxide is preferably sodium hydroxide or potassium hydroxide and is present in the aqueous treating solution in a concentration of from about 1¼ percent by weight to about 5 percent by weight and preferably from about 2 percent by weight to about 4 percent by weight.

The normally liquid organic compound is a ketone, an alcohol, a lactone, or a sulfoxide, and includes compounds such as: aliphatic ketones including methyl ethyl ketone, methyl isobutyl ketone, ethyl isopropyl ketone, diethyl ketone, etc.; aliphatic monohydroxy alcohols such as methyl alcohol, ethyl alcohol, normal propyl alcohol, isopropyl alcohol, t-butyl alcohol, isobutyl alcohol, sec-butyl alcohol, n-butyl alcohol, etc.; dihydroxy alcohols such as ethylene glycol, diethylene glycol, propylene glycol, etc.; other aromatic or heterocyclic alcohols such as tetrahydrofurfuryl alcohol, etc.; lactones such as 4-hydroxy butanoic acid lactone (butyrolactone), 4-hydroxy-2-methylene butanoic acid lactone, 4-hydroxy pentanoic acid lactone, 5-hydroxy pentanoic acid lactone, 4-hydroxy hexanoic acid lactone, etc.; sulfoxides such as dimethyl sulfoxide (DMSO); methyl ethyl sulfoxide, diethyl sulfoxide, methyl propyl sulfoxide, etc.

These organic compounds are liquids under normal conditions of room or ambient temperature and normal atmospheric pressure. Their water solubility must be such that they can form aqueous solutions of at least 2 percent by weight. Greater water solubility of ten percent by weight or even water miscibility is preferred. The concentration of the organic compound in the aqueous treating solution is in the range of from about 2 percent or slightly less by weight to about 10 percent by weight.

Further specific details regarding the initial heating of the waste fibers and the synthetic, cross-linked resin materials are to be noted in U.S. Pat. No. 2,832,663 which issued Apr. 29, 1958.

During the initial heating under pressure, the polyester and/or acrylic fibers and the synthetic, cross-linked resin are attacked to some degree but are not sufficiently decomposed so as to be rendered soluble. A viscous, slimy jelly is formed which clings tenaciously to the cellulosic fibers and does not wash out. In cases where the original resin component is a cross-linked polyacrylic ester, the jelly present at this stage is believed to be a high molecular weight, two or three-dimensional cross-linked polymer of sodium polyacrylate.

THE SUBSEQUENT HEATING STEP

The viscous, slimy, practically insoluble jelly is therefore treated further in order to enable a successful separation and recovery of the cellulosic fibers.

The mass of fibers covered with the viscous, slimy jelly is rinsed to remove most of the alkali metal hydroxide and the normally liquid organic compound. Sufficient alkali metal hydroxide is permitted to remain, however, to provide a pH range of at least about 8½ and preferably at least about 9, up to about 11 or even higher, if desired or required.

The next step in the process involves a heat treatment of this fiber mass containing the viscous, slimy jelly in a treating solution containing a neutral or alkaline oxidizing agent such as sodium hypochlorite, sodium perborate, oxygen, hydrogen peroxide, alkali metal peroxides such as sodium peroxide, potassium peroxide, lithium peroxide; alkaline earth metal peroxides such as barium peroxide, calcium peroxide; etc.

The neutral or alkaline oxidizing agent is present in the solution in a concentration of from about 0.1 percent by weight to about 8 percent by weight and preferably from about 0.2 percent by weight to about 4 percent by weight. These limits are, of course, dependent upon the relative activity of the oxidizing agent used. Sodium perborate is relatively less active and requires greater concentrations; sodium hypochlorite is relatively more active and permits lesser concentrations. The upper limits of the concentrations used are naturally dictated by the possibility of damage or degradation to the fibers being recovered.

This heating step is accomplished by simply heating, preferably to the boiling point of the treating solution, and, if necessary, holding the treating solution at the elevated temperature or boiling point for a few minutes. In some instances, it has been found that merely heating to near the atmospheric boiling point is sufficient. Freedom of access of the oxidizing agent to the resin materials being treated is important.

During this heating step, the ratio of the amount of liquid to the amount of fibers should be kept within controlled limits. This ratio must be at least about 4:1 to permit access of the treating agent to the jellied fiber mass. Higher ratios may be employed up to 10:1 or even as high as 25:1 but such higher ratios are not desirable from an economical viewpoint inasmuch as increased amounts of the neutral or alkaline oxidizing agents are required with such larger volumes of water.

This treatment decomposes and breaks down the viscous, slimy jelly into a water-soluble form whereby it is easily removed. It is believed that the oxidizing agent breaks down the chain length of the jelly-like polymer into water or alkali soluble fragments, without damaging the recoverable fibers. The fibers are then washed with water, preferably at room temperature, and dried, forming a loose, fluffy, unbonded, resin-free mass of fibers. Degradation of the fibers is of such a low order that the usefulness of the fibers is not impaired for recycling and re-use in the production of nonwoven, woven, or knitted fabrics by textile processes, or the production of other fibrous products by other manufacturing processes.

The invention will be further illustrated in greater detail by the following specific examples. It should be understood, however, that although these examples may

describe in particular detail some of the more specific features of the invention, they are given primarily for purposes of illustration and the invention in its broader aspects is not to be construed as limited thereto.

EXAMPLE I

One hundred grams of rayon - "Dacron" ethylene glycol-terephthalic acid polyester (80:20 ratio by weight) trim waste fibers produced in the course of the manufacture of nonwoven fabrics comprising several layers of card webs and containing as a bonding agent approximately 20 percent (20 grams) of synthetic self-cross-linked polyethyl acrylate is introduced into a vessel containing 700 grams of water, 35 grams of sodium hydroxide, and 70 grams of tetrahydrofurfuryl alcohol (THFA). The mixture of waste fibers and polyethyl acrylate is heated for a period of 1 hour at a temperature of 250°F. The polyester fibers and the polyethyl acrylate are attacked during this heating step, but are not solubilized. A viscous, slimy jelly is formed which clings tenaciously to the cellulosic fibers and does not wash out.

The "Dacron" polyester fibers are believed to be saponified or hydrolyzed and partially converted into a "semi-soluble" gel or fragmented state. They are not believed to be fully soluble at this time.

The slimy mass of rayon - "Dacron" polyester trim waste fibers and polyethyl acrylate resin is then rinsed in water to remove part of the sodium hydroxide, but sufficient sodium hydroxide remains to provide a pH of 9. While the fibrous mass is still wet, a sufficient amount of hydrogen peroxide is added to provide a concentration of hydrogen peroxide in the total composition of about 1 percent. Intimate contact is provided between the hydrogen peroxide and the slimy mass of jellied fibers. The mixture is then heated to boiling. During this heating, the hydrogen peroxide attacks, decomposes and breaks down the polyester fibers and the jelly-like synthetic resin into a water soluble form. The polyester fibers are decomposed and are easily washed out. The cellulosic fibers are then washed and dried, forming a loose, fluffy, unbonded, resin-free mass of fibers. Degradation of the cellulosic fibers is of such a low order that their usefulness in textile processes for the production of nonwoven, woven or knitted fabrics is not impaired.

EXAMPLE II

The procedures of Example I are followed substantially as set forth therein with the exception that the cross-linked resin is a copolymer of polyethyl acrylate and methyl methacrylate. The results are generally comparable to the results obtained in Example I. The fibers are recovered in loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE III

The procedures of Example I are followed substantially as set forth therein with the exception that the cross-linked resin is a copolymer including butyl acrylate. The results are generally comparable to the results obtained in Example I. The fibers are recovered in loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness

in textile processes for the production of fabrics is not impaired.

EXAMPLE IV

The procedures of Example I are followed substantially as set forth therein with the exception that the cross-linked resin is a copolymer of butyl acrylate and ethyl acrylate. The results are generally comparable to the results obtained in Example I. The fibers are recovered in loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE V

The procedures of Example I are followed substantially as set forth therein with the exception that the tetrahydrofurfuryl alcohol is replaced by normal propyl alcohol. The results are generally comparable to the results obtained in Example I. The fibers are recovered in a loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE VI

The procedures of Example I are followed substantially as set forth therein with the exception that the tetrahydrofurfuryl alcohol is replaced by normal butyl alcohol. The results are generally comparable to the results obtained in Example I. The fibers are recovered in a loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE VII

The procedures of Example I are followed substantially as set forth therein with the exception that the tetrahydrofurfuryl alcohol is replaced by ethylene glycol. The results are generally comparable to the results obtained in Example I. The fibers are recovered in a loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE VIII

The procedures of Example I are followed substantially as set forth therein with the exception that the tetrahydrofurfuryl alcohol is replaced by gamma butyrolactone. The results are generally comparable to the results obtained in Example I. The fibers are recovered in loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE IX

The procedures of Example I are followed substantially as set forth therein with the exception that the tetrahydrofurfuryl alcohol is replaced by dimethyl sulfoxide. The results are generally comparable to the results obtained in Example I. The fibers are recovered in loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness

in textile processes for the production of fabrics is not impaired.

EXAMPLE X

The procedures of Example I are followed substantially as set forth therein with the exception that the tetrahydrofurfuryl alcohol is replaced by methyl ethyl ketone. The results are generally comparable to the results obtained in Example I. The fibers are recovered in loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XI

The procedures of Example I are followed substantially as set forth therein with the exception that the hydrogen peroxide is replaced by sodium peroxide. The results are generally comparable to the results obtained in Example I. The fibers are recovered in a loose, fluffy, unbonded resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XII

The procedures of Example I are followed substantially as set forth therein with the exception that the hydrogen peroxide is replaced by potassium peroxide. The results are generally comparable to the results obtained in Example I. The fibers are recovered in a loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XIII

The procedures of Example I are followed substantially as set forth therein with the exception that the hydrogen peroxide is replaced by sodium perborate. A sufficient amount of sodium perborate is added to provide a concentration of about 3 percent by weight in the total composition. The results are generally comparable to the results obtained in Example I. The fibers are recovered in loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XIV

The procedures of Example I are followed substantially as set forth therein with the exception that the hydrogen peroxide is replaced with sodium hypochlorite. The sodium hypochlorite content is approximately 0.2 percent by weight and the treating cycle is 1 hour at 40°C. The results are generally comparable to the results obtained in Example I. The fibers are recovered in loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XV

The procedures of Example I are followed substantially as set forth therein with respect to the first step of initiating the breakdown of the cross-linked resin to form the practically insoluble jelly or slime on the rayon fibers. These fibers are rinsed to remove most of

the excess caustic, bringing the pH to about 9, and surplus liquid is removed by centrifuging to bring the fiber:liquid ratio down to about 1:10.

Gaseous oxygen is bubbled slowly (10 ml oxygen per minute) through the wet fibrous mass for one hour with excellent access to the jellied fibers while the wet fibrous mass is maintained at a temperature of about 90°C. The treated fibers are rinsed in water, removing the remaining caustic and the degradation products of the polyester fibers and of the resin which are now water-soluble.

The results are generally comparable to the results obtained in Example I. The fibers are recovered in a loose, fluffy, unbonded, resin-free form. Degradation of fibers is of such low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XVI

The procedures of Example XV are followed substantially as set forth therein except that air is bubbled slowly through the wet fibrous mass for three hours. The results are generally comparable and similar to those obtained in Example XV.

EXAMPLE XVII

The procedures of Example I are followed substantially as set forth therein with the exception that the rayon fibers are replaced by cotton fibers. The results are generally comparable to the results obtained in Example I. The cotton fibers are recovered in a loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XVIII

The procedures of Example I are followed substantially as set forth therein with the exception that the "Dacron" polyester fibers are replaced by "Acrilan" acrylic fibers. The results are generally comparable to the results obtained in Example I. The cellulosic fibers are recovered in a loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XIX

The procedures of Example I are followed substantially as set forth therein with the exception that the "Dacron" polyester fibers are replaced by "Dynel" modacrylic fibers. The results are generally comparable to the results obtained in Example I. The cellulosic fibers are recovered in a loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XX

The procedures of Example I are followed substantially as set forth therein with the exception that 80:20 weight ratio of rayon:polyester fibers is changed to (a) 65:35 and (b) 95:5. The results of both procedures are generally comparable to the results obtained in Example I. Degradation of the rayon fibers is of such a very low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XXI

The procedures of Example I are followed substantially as set forth therein with the exception that the fiber mixture comprises 80 percent by weight of cotton fibers, 15 percent by weight of polyester fibers, and 5 percent by weight of polypropylene fibers. The results are generally comparable to the results obtained in Example I. The cotton fibers and the polyolefinic fibers are recovered in a loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XXII

The procedures of Example I are followed substantially as set forth therein with the exception that the rinsing of the slimy mass of trim waste fibers and polyethyl acrylate is such as to provide a pH of (a) 8½, (b) 10, and (c) 11. The results are generally comparable to the results obtained in Example I. The fibers are recovered in loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

EXAMPLE XXIII

The procedures of Example I are followed substantially as set forth therein with the exception that the amount of hydrogen peroxide which is added is changed so that the concentration of hydrogen peroxide in the total composition becomes (a) 1/2 percent, (b) 2 percent, and (c) 3 percent. The results are generally comparable to the results obtained in Example I. The fibers are recovered in loose, fluffy, unbonded, resin-free form. Degradation of the fibers is of such a low order that their usefulness in textile processes for the production of fabrics is not impaired.

Although several specific examples of the inventive concept have been described, the same should not be construed as limited thereby nor to the specific features mentioned therein but to include various other equivalent features as set forth in the claims appended hereto. It is understood that any suitable changes, modifications and variations may be made without departing from the spirit and scope of the invention.

What is claimed is:

1. A method of recovering waste cellulosic fibers from a mixture of waste cellulosic fibers, waste polyester and/or acrylic fibers, and synthetic, cross-linked resin materials which comprises: heating said mixture of waste cellulosic fibers, waste polyester and/or acrylic fibers, and synthetic, cross-linked resin materials within the range of from about 212°F. to about 275°F. for a period of from about ¼ hour to about 5 hours in an aqueous treating solution containing an alkali metal hydroxide and a normally liquid, organic compound selected from the group consisting of ketones, alcohols, lactones, and sulfoxides; adding a neutral or alkaline oxidizing agent to the mixture of waste fibers and synthetic, cross-linked resin materials; heating the mixture of waste fibers and synthetic, cross-linked materials in the presence of said neutral or alkaline oxidizing agent to partially decompose or solubilize the waste polyester and/or acrylic fibers and the

synthetic, cross-linked resin; and recovering the waste cellulosic fibers.

2. A method as defined in claim 1 wherein the oxidizing agent is hydrogen peroxide.

3. A method as defined in claim 1 wherein the oxidizing agent is gaseous oxygen.

4. A method as defined in claim 1 wherein the oxidizing agent is sodium hypochlorite.

5. A method as defined in claim 1 wherein the oxidizing agent is sodium perborate.

6. A method as defined in claim 1 wherein the addition of the oxidizing agent takes place during the heating of the waste fibers and synthetic, cross-linked resin materials.

7. A method as defined in claim 1 wherein the normally liquid organic compound is normal propyl alcohol.

8. A method as defined in claim 1 wherein the nor-

mally liquid organic compound is methyl ethyl ketone.

9. A method as defined in claim 1 wherein the normally liquid organic compound is tetrahydrofurfuryl alcohol.

10. A method as defined in claim 1 wherein the normally liquid organic compound is gamma butyrolactone.

11. A method as defined in claim 1 wherein the normally liquid organic compound is dimethyl sulfoxide.

12. A method as defined in claim 1 wherein the cellulosic fibers are rayon.

13. A method as defined in claim 1 wherein the cellulosic fibers are cotton.

14. A method as defined in claim 1 wherein the polyester fibers are ethylene glycol-terephthalic acid polymers.

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