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ALKALI TREATMENT OF AGAVE AND MANILA HEMP FIBERS TO PRODUCE FLEXIBLE FIBERS

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This invention relates to a process for the improvement of the properties of agave fibers and manila hemp fibers wherein the same are rendered flexible without substantially diminishing their original strength. More particularly the invention relates to the treatment of such fibers in their raw state, i. e., before their separation from the bundles in which they grow with solutions of aluminum and/or zinc oxide or hydroxide in solutions of alkali metal hydroxide whereby the fibers are rendered suitable or more satisfactory for the production of rope or cloth for a number of articles of commerce, such as bags or sacks.

As produced by known processes, the raw fibers of agave, such as sisal and manila hemp, are stiff in character and possess such slight flexibility that their employment for a number of potential industrial applications is restricted. In contrast with articles from the more flexible jute fibers, articles from sisal fibers for example such as bags and sacks, are not satisfactory for practical use on account of their low flexibility. The raw sisal individual fibers are fairly large in diameter and the fibers are firmly held together in bundles in their natural condition.

Heretofore attempts have been made to increase the flexibility of such fibers by treating them with acids, chlorides, alkalies, soap or oils. In order to make these chemicals sufficiently active it has been necessary that they be employed at high concentrations or at high temperatures or both, but under such conditions the tensile strength of the fibers is considerably reduced and there is an appreciable loss of material in the operation. The treatment of the fibers with oils, on the other hand, has resulted in insufficiently altered fibers.

It has also heretofore been proposed that various vegetable fibers including sisal and hemp fibers are chemically treated by first bleaching the same and then subjecting them to the action of a solution of a mixture of a metal chloride and sodium hydroxide. By this treatment in which adverse effects result from the bleaching treatment, fibers resembling wool are said to be obtained, but due to the fairly high concentrations of sodium hydroxide required, a great reduction in tensile strength has occurred. At the same time a substantial loss of material has been unavoidable.

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An object of the present invention is to produce flexible, high tensile strength fibers from agave and manila hemp fibrous masses which are admirably suited for spinning and weaving into various manufactured products by a process operable at low cost.

Broadly considered the present invention involves treating agave and manila hemp fibers (fibrous masses) with alkali metal hydroxide solutions containing either the oxide or hydroxide of aluminum or of zinc or both metals. Heat is generally necessary to accelerate the reaction. The alkali concentration and temperature may be at any degree or elevation which does not destroy the tensile strength of the fibers. When high temperatures are employed, lower alkali concentrations are required.

In its preferred mode of operation, the process involves treating the raw fibers (fibrous masses) with any of the above-mentioned solutions in which the alkali metal hydroxide is at a relatively low concentration, for example less than 15% (calculated as NaOH), at an elevated temperature of from 60 to 100° C., the time of the treatment under these conditions being about 1 to 2 hours. In a less preferred procedure, the treatment is carried out with a more concentrated alkaline solution at an ordinary or slightly elevated temperature, in which operation the contact of the chemicals with the reagents is relatively long, involving several hours or a day or more. Even superatmospheric pressures and temperatures over 100° C. can be used provided the strength of the alkaline solution and the time of treatment are restricted such that the fibers are not weakened to any substantial degree. In general, however, the use of superatmospheric pressures and temperatures over 100° C. has no specific advantage.

The concentration of the solutions, the temperature and the time employed in the operation of the process of the present invention are correlated in such manner that the binding or incrusting substances between the fibers, such as pectins and lignins are effectively dissolved while at the same time the individual fibers themselves are not appreciably attacked. In any specific operation of the process or in the treatment of any certain fiber, the optimum concentration, temperature and duration of treatment can be readily ascertained. In a preferred embodiment operated at higher temperatures, the concentration of the alkali hydroxide in the solution calculated as NaOH preferably amounts to from about 3 to 10%. Solutions of other alkali metals than sodium may likewise be employed.

In all operations of the process, the quantity of the metal compound preferably should not be less than an amount equivalent to 0.5% of zinc oxide or to 0.5% of aluminium oxide, calculated with reference to the total amount of the solution. The maximum quantity which can be used is determined by the solubility of the metal ion in the alkali metal hydroxide solution employed. The content of aluminum oxide in the solution is preferably at least 2%.

Excellent results may be obtained by the use of alkali metal hydroxide solutions containing both zinc oxide and aluminum oxide, for example solutions containing from 1 to 3% of zinc oxide and 2 to 6% of aluminum oxide. When alkali hydroxide solutions of this concentration are employed, the temperature of the treatment is preferably maintained higher than 60° C. and most desirably between 80° and 100° C. Under such concentrations and temperatures, requiring the treatment over a short period of from only 1 to 2 hours, the loss or material is very small, while the fiber at the same time retains its great tensile strength.

After the treatment of the fiber masses with the alkaline solution of the metal compounds, the resulting fibers are squeezed to remove excess solution and then washed, preferably first with water and then subsequently with a dilute acid of either inorganic or organic nature, such as hydrochloric acid or acetic acid, after which the fibers are again washed with water, finally, the fibers are dried and in such condition they are then mechanically processed to provide the same in condition for their intended use.

In accordance with an important embodiment of the overall treatment to provide fibers of best quality, the chemically treated fibers, either before or after the mechanical processing, are treated with a flexibility imparting oil, either a mineral oil or a non-mineral oil, preferably in the form of an emulsion. The effect of the oil on the treated fibers is surprisingly high in greatly improving the flexibility. Examples of suitable oils are light lubricating oils such as spindle oil, rapeseed oil, cottonseed oil, castor oil, fish oil. The treatment of the agave and manila hemp fibers according to the process of the invention is so effective that it is even possible first to manufacture a cloth or the articles desired, for example ropes, bags or sacks, from the fibers and then to subject the cloth or the articles to the process hereinbefore described, including a treatment with a flexibilizing oil.

In the following examples, set forth merely to illustrate the invention, the amounts of the fibers and reagents employed are given in parts by weight.

Example 1

A quantity of 25 parts of henequen fibers was introduced into a sodium hydroxide solution (8%) containing 1.3% of zinc oxide, and the mass was heated for a period of one hour at 90° C. The fibrous mass obtained was then squeezed out as thoroughly as possible, washed with water, then treated with a hydrochloric acid solution (1%), again washed with water and finally dried. The resulting fibers were then subjected to mechanical processing. The fibers obtained had high tensile strength and during the treatment practically no loss of material occurred.

Before the obtained fibers are spun, it is preferable to treat the same with oil in accordance with known procedures, as by emersing the same in an emulsion of rapeseed oil or other conventional oil in water. After this oiling treatment, the fibers are squeezed out and dried. As a result, flexible water-repellent fibers are obtained.

Example 2

Ten parts of sisal are soaked for 16 hours at about 20° C. in a solution of 9 parts of zinc oxide and 27 parts of sodium hydroxide in 64 parts of water. Then the excess solution is removed as thoroughly as possible by squeezing and the sisal fibers are washed successively with water, hydrochloric acid (1%) and water. Finally, the fibers are dried after which they are suitable for mechanical processing. The resulting fibers are preferably oiled as described in Example 1 before being processed.

Example 3

Twenty-one parts of raw sisal fibers are soaked for 24 hours at about 20° C. in a solution of 6 parts of aluminum hydroxide and 6 parts of zinc oxide in 200 parts of sodium hydroxide solution having a sodium hydroxide content of 18%. After this treatment, the fibrous mass is squeezed out as thoroughly as possible and washed successively with water, with a 5% aqueous solution of acetic acid and with water. Finally, the fibers are subjected to any suitable oiling treatment before being subjected to mechanical processing.

Example 4

Eight parts of manila hemp are soaked for 24 hours at about 20° C. in the solution left after the treatment of the sisal fibers employed in the process of Example 2. Thereupon the hemp mass is squeezed out, washed with water, treated with a 1% solution of hydrochloric acid, washed and dried, after which the fibers are oiled in the manner hereinbefore described.

Example 5

Forty-eight parts of manila hemp are soaked for 16 hours at 60° C. in an aqueous solution composed of 24.5 parts of sodium hydroxide, 31 parts by weight of aluminum oxide and 500 parts of water. The resulting hemp mass is then further processed by the procedure described in Example 1.

Example 6

Twenty-five parts of Java sisal are treated for 90 minutes at a temperature of 95° C. with a 10% sodium hydroxide solution in which 3% of aluminum oxide and 1.3% of zinc oxide have been dissolved. The fibrous material obtained is then squeezed out, washed, rinsed with a 1% solution of hydrochloric acid, washed once more and finally oiled with an emulsion of mineral oil.

Example 7

A cloth made of henequen fibres was soaked for about 60 minutes in the solution of Example 1 at a temperature of about 90° C. After being de-alkalized and dried as described hereinbefore the cloth was flexibilized in a known way in an emulsion of spindle oil in water. The cloth thus treated is very suitable for being made into sacks of a great flexibility.

In the same way ready made sacks or bags consisting of henequen or sisalfibers were treated, whereby the articles obtained a high flexibility.

The process of the present invention has the outstanding advantage that the treatment conditions may readily be so chosen that the undesired binding components of the fibrous mass, such as pectins and lignins, can be dissolved out without the fibers themselves being chemically attacked to any substantial degree. As a result, the substances cross-linking or binding the fibers together disappear and the fibers retain substantially their original length and tensile strength. The process also has the advantage that the loss of material ordinarily is very small.

The process of the present invention also has the advantage that it is not necessary to prebleach the fibers. Hence the cost and detrimental effects of the bleaching operation are avoided.

The herein before disclosed oil treatment has a surprisingly beneficial effect in improving the flexibility of the fibers. The raw fibers are practically not improved at all by the oil treatment and hence the novel treatment step of the present invention alters the fibers physically or chemically in such manner that the oil treatment can become effective.

It should be understood that the present invention is not limited, except as herein indicated, to the specific compounds and conditions of treatment disclosed, but that it extends to all equivalents which will occur to those skilled in the art upon consideration of the tenor of the specification and the scope of the claims appended hereto.

We claim:

1. A process for the production of strong, flexible, cellulose fibers from raw agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses with an aqueous alkali metal hydroxide solution of about 3–15% concentration (calculated as sodium hydroxide) containing dissolved therein at least 0.5% (calculated as oxide) of at least one compound of the group consisting of aluminum and zinc oxides and hydroxides until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby the strong, flexible, cellulose fibers are obtained.

2. A process for the production of strong, flexible, cellulose fibers from raw agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses with an aqueous alkali metal hydroxide solution of about 3–15% concentration (calculated as sodium hydroxide) containing dissolved therein at least 0.5% (calculated as oxide) of at least one compound of the group consisting of aluminum and zinc oxides and hydroxides until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, drying the fibers and subjecting the same to mechanical processing whereby strong, flexible, cellulose fibers in condition for spinning are obtained.

3. A process for the production of strong, flexible, cellulose fibers from raw agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, con-

tacting the said fibrous masses with an aqueous alkali metal hydroxide solution of about 3–15% concentration (calculated as sodium hydroxide) containing dissolved therein at least 0.5% (calculated as oxide) of at least one compound of the group consisting of aluminum and zinc oxides and hydroxides until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, drying the fibers and applying thereto a flexibility imparting oil whereby strong, water-repellent fibers of increased flexibility are obtained.

4. A process for the removal of lignins and other natural substances binding the cellulose fibers together in raw agave and manila hemp fibrous masses whereby strong, flexible fibers are obtained which comprises, submerging said raw fibrous masses in an aqueous alkali metal hydroxide solution of about 3–15% concentration (calculated as sodium hydroxide) containing dissolved therein at least 0.5% (calculated as oxide) of at least one compound of the group consisting of aluminum and zinc oxides and hydroxides and heating the solution containing the fibrous masses until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby strong, flexible, cellulose fibers are obtained.

5. A process for the production of strong, flexible, cellulose fibers from raw agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses with an aqueous sodium hydroxide solution of 3–10% concentration containing dissolved therein at least 0.5% (calculated as oxide) of at least one compound of the group consisting of aluminum and zinc oxides and hydroxides until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby strong, flexible, cellulose fibers are obtained.

6. A process for the production of strong, flexible cellulose fibers from raw agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses with an aqueous alkali metal hydroxide solution of about 3–15% concentration (calculated as sodium hydroxide) containing dissolved therein at least 0.5% (calculated as oxide) of at least one compound of the group consisting of aluminum and zinc oxides and hydroxides until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, drying the fibers, applying thereto a flexibility-imparting oil, and subjecting the fibers to mechanical processing whereby strong, flexible, cellulose fibers in condition for springing are obtained.

7. A process for the production of strong, flexible, cellulose fibers from agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses with an

aqueous alkali metal hydroxide solution of about 3–15% concentration (calculated as sodium hydroxide) containing dissolved therein at least 0.5% of zinc oxide until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby strong, flexible, cellulose fibers are obtained.

8. A process for the production of strong, flexible, cellulose fibers from agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses with an aqueous alkali metal hydroxide solution of about 3–15% concentration (calculated as sodium hydroxide) containing dissolved therein at least 0.5% aluminum oxide until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby strong, flexible, cellulose fibers are obtained.

9. A process for the production of strong, flexible, cellulose fibers from agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses with an aqueous alkali metal hydroxide solution of about 3–15% concentration (calculated as sodium hydroxide) containing dissolved therein at least 0.5% of both zinc oxide and aluminum oxide until the said binding substances are substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby the strong, flexible, cellulose fibers are obtained.

10. A process for the production of strong, flexible, cellulose fibers from agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises contacting the said fibrous masses at a temperature in excess of about 60° C. with an aqueous alkali metal hydroxide solution of about 3–15% concentration (calculated as sodium hydroxide) containing dissolved therein at least 0.5% (calculated as oxide) of at least one compound of the group consisting of aluminum and zinc oxides and hydroxides until the said binding substances are substantialy dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby strong, flexible, cellulose fibers are obtained.

11. A process for the production of strong, flexible, cellulose fibers from agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses at a temperature between about 80° and 100° C. with an aqueous sodium hydroxide solution of 3–10% concentration containing dissolved therein at least 0.5% (calculated as oxide) of at least one compound of the group consisting of aluminum and zinc oxides and hydroxides until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby strong, flexible, cellulose fibers are obtained.

12. A process for the production of strong, flexible, cellulose fibers from agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses at a temperature of 60–100° C. with an aqueous sodium hydroxide solution of about 3 to 10% concentration containing dissolved therein at least 0.5% of zinc oxide until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby strong, flexible, cellulose fibers are obtained.

13. A process for the production of strong, flexible, cellulose fibers from agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses at a temperature of 60–100° C. with an aqueous sodium hydroxide solution of about 3 to 10% concentration containing dissolved therein at least 0.5% (calculated as oxide) of at least one compound of the group consisting of aluminum and zinc oxides and hydroxides until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, removing the solution containing the dissolved substances from the fibers, drying the fibers and applying thereto a flexibility-imparting oil whereby strong, water-repellent fibers of increased flexibility are obtained.

14. A process for the production of strong, flexible, cellulose fibers from agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses with an aqueous sodium hydroxide solution of about 3 to 10% concentration containing dissolved therein 2–6% aluminum oxide and 1–3% zinc oxide until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby strong, flexible, cellulose fibers are obtained.

15. A process for the production of strong, flexible, cellulose fibers from agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses with an aqueous sodium hydroxide solution of about 10% concentration containing dissolved therein about 3% aluminum oxide and about 1.3% zinc oxide until the said binding substances are substantially dissolved out while leaving the cellulose fibers substantially free from attack, and removing the solution containing the dissolved binding substances from the fibers, whereby the strong, flexible, cellulose fibers are obtained.

16. A process for the production of strong, flexible, cellulose fibers from agave and manila hemp fibrous masses the individual fibers of which are bound together with lignin and other natural binding substances which comprises, contacting the said fibrous masses at a temperature of 60–100° C. with an aqueous alkali metal hydroxide solution of about 3 to 10% concentration-

tion, (calculated as sodium hydroxide) containing dissolved therein at least 0.5% (calculated as oxide) of at least one compound of the group consisting of aluminum and zinc oxides and hydroxides for a period of from 1 to 2 hours whereupon the said binding substances will be substantially dissolved out and the cellulose fibers left in a substantially free from attack form, and removing the solution containing the dissolved binding substances from the fibers, whereby strong, flexible, cellulose fibers are obtained.

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