Patented Apr. 17, 1928. 1,666,083

UNITED STATES PATENT OFFICE.

ALBERT BODMER, OF WATTWIL, SWITZERLAND, ASSIGNOR TO HEBERLEIN PATENT CORPORATION, OF NEW YORK, N. Y., A CORPORATION OF NEW YORK.

VEGETABLE YARN AND FABRIC WITH WOOLLIKE EFFECTS.

No Drawing. Original application filed April 11, 1923, Serial No. 631,374, and in Germany April 15, 1922. Divided and this application filed April 12, 1923. Serial No. 631,745.

The invention relates to vegetable yarns and fabrics with wool-like effects, such for example as cotton goods which have been treated so as to chemically structurally change the fibres to produce permanent finish wool effects.

The production of various permanent finishes in cotton goods is known, such for example as permanent organdie effects, wool-like effects and linen-like effects which have been produced by processes hereinafter referred to.

The object of the present invention is to improve the character and quality of permanent finish goods and also the process of producing same. According to the preferred manner of practicing the present invention, the hydrolyzation of the fibres by the action of a concentrated mineral acid which is used in producing these permanent finishes, is stabilized so that the acid may be allowed to act on the goods for a greater length of time so as to effect a more gradual and greater penetration of the goods and hence a more thorough and uniform conversion of the fibres, and with much less risk of producing detrimental effects, damage or destruction of the goods.

This application is a division of my copending application Serial No. 631,374, filed April 11, 1923, and in this present application I aim to cover the permanent finish wool-like effect goods of the character resulting from the process herein described and claimed broadly in my said copending application.

The invention consists in the wool-like effect fabrics and yarns hereinafter described. These are produced according to the novel process or processes hereinafter described, by way of example, in accordance with certain ways of carrying out the same. The invention will be particularly pointed out in the appended claims.

In order that the scope of the use and application of the invention to goods for the production of permanent finishes may be more readily understood, I will first refer to some of the prior processes for producing permanent finishes and to which this invention is applicable and upon which it constitutes an improvement.

In United States Patent No. 1,141,872, granted to Georges Heberlein, there was described the process of treating cotton fabric to produce permanent wool-like effects by subjecting the fabric to two treatments, one a mercerizing treatment such as with caustic soda, and the other a treatment with concentrated sulfuric acid of less than 51° Bé. It was described in that patent specification that if cotton fabric which has been mercerized and also preferably bleached, be subjected to the action of sulfuric acid of from 49° to somewhat under 51° Bé. (the best action being obtained between 49½° and 50½° Bé.), the mercerizing lustre disappears and the fabric appears fuller, softer and more wool-like and is generally improved, so as to take on the character of fine thin wool muslin.

It was also pointed out in United States Patent No. 1,439,514, to Georges Heberlein, that this wool-like effect could also be obtained if in said process there was substituted for the sulfuric acid, other reagents, including other strong mineral acids, such for example as hydrochloric acid of the specific gravity of 1.19 at low temperature, or nitric acid of 43° to 46° Bé., or phosphoric acid of 55° to 57° Bé.

It was also pointed out in United States Patent No. 1,144,655, granted to Georges Heberlein, that when the cotton fabric was subjected to the mercerizing or caustic soda treatment and the sulfuric acid treatment, utilizing the acid at a higher concentration than that used for the wool effect, i. e., over 50½° Bé., for example, about 54° Bé., then the said transparent permanent effect or permanent organdie would result.

It was further pointed out in the United States patent to Eduard Heberlein, No. 1,265,082, that the permanent transparent effect could be obtained if the cotton fabric was treated for a sufficient period with the caustic alkali cooled below 0° C., and of such concentration as would mercerize the cotton at the ordinary temperature. That is to say, of at least 15° Bé. specific gravity; and that if in conjunction with the treatment of said cold caustic alkali the fabric was also treated with the last mentioned sulfuric acid treatment, a more enhanced transparency

would result. Furthermore, it was pointed out in United States Patent No. 1,439,519, to Eduard Heberlein, that when the treatments set forth in said Patent 1,265,082 (i. e., a treatment with the caustic soda at a temperature below 0° C. or said treatment in conjunction with a treatment with the sulfuric acid of over 50½° Baumé, and preferably about 54° Baumé) were carried out upon yarns or webs constituted of threads whose fineness does not exceed English yarn No. 80, permanent linen-like effects would be produced. It was also pointed out in said Patent 1,439,519 that more enhanced linen-like effects would be produced if in conjunction with the acid treatment and the cold caustic soda treatment the goods were also subjected to the caustic soda or mercerizing treatment at normal temperature. Among other Letters Patent disclosing processes producing permanent finishes and permanent finish products and upon which the present invention is an improvement, I might mention the following, i. e., 1,288,884, 1,258,225, 1,285,738, 1,392,264 and 1,392,265.

In the practicing or execution of these processes it has been found that due to the detrimental effects that these concentrated acids will quickly produce on the goods if not very accurately controlled, it was very important to accurately measure and limit the action of the acid in order to safeguard the fabric. It was therefore found to be necessary to interrupt or arrest the reaction suddenly after an interval of time that had to be strictly adhered to, inasmuch as an excess of the action always resulted in an undesirable hydrolyzation or decomposition of the cellulose. This decomposition even makes itself evident during the swelling reactions. The consequences of any excess of this hydrolytic decomposition of the cellulose made themselves evident, on the one hand, in the form of a diminution of the toughness or strength of the fibre, and on the other hand, in the form of an excessive hardening of the fibres.

Now I have discovered that the reaction of the concentrated mineral acids on these materials, which is quite aggressive, may to some degree be regulated and stabilized by the use of suitable heterocyclic compounds miscible with the particular mineral acid used, as for example, sulfuric acid, and thereby it is possible to somewhat increase the strength of the acid used in the sense of decreasing the proportion of water thereto and so decreasing the water acid ratio and the hydrolyzing action that water exerts in the presence of acid. Furthermore, I have discovered that the preferred heterocyclic compounds are heterocyclic bases such as pyridine, etc. I have discovered that in a surprising manner, for example, salts of pyridine, quinolin, their homologues and derivatives, manifest the property, when added to mineral acids, of stabilizing the swelling action of the fibres due to the action of the acid, i. e., of holding back the hydrolytic decomposition of the cellulose so that the swelling may be completely realized without injurious hydrolyzation. These deterring agents, I have found, exert a specific action in partially and temporarily neutralizing a portion of the acid without at the same time permanently inhibiting its action as would a strong alkali. I have found that certain of the heterocyclic compounds such as salts of the heterocyclic bases and their derivatives, particularly pyridine and quinolin, their homologues and derivatives, produce a toning down of, or a sort of retarding effect upon the reaction during the treatment of the goods with the acid, in the sense that by their presence they retard the rate of reaction and have thereby the great commercial advantage of allowing much more time in which to adjust and regulate the precise degree of action required to attain the most desirable appearance effects on the surface of the fibre undergoing treatment, while still enabling the core or center of the fibre or yarn to be thoroughly treated to attain more desirable qualities in the fabric in addition to the more desirable appearance effects.

In this manner it is possible to prolong the duration of the action to fifty times the normal period without injury to the goods being processed or ennobled; and it is also possible to safely use more concentrated acids for a given finish. If, for example, a mixture consisting of one part of pyridine and four parts of sulfuric acid of 66° Baumé is allowed to act upon previously bleached mercerized muslin fabric, the latter after about four minutes comes to have a transparent appearance, in connection with which a remarkable softness of effect is to be noted. Again, if for example, we operate in such a way that a suitable mercerized cotton fabric is subjected for 5 minutes to the action of a mixture of one part of pyridine and 9 parts of sulfuric acid of 56.5° Baumé, a pronounced soft wool effect is obtained having a sort of discernible "bloom" or overlaid opalescence which slightly lights up the dull tone of the permanent finish wool-like effects. The improvement is therefore greater smoothness and softness to the touch and a slight lighting up of the dull tone wool effect. The proportion of acid and pyridine in the mixture may vary within wide limits, according to the desired effect and the goods to be worked up.

According to the present invention it is also feasible to thoroughly improve very compact fibrous structures, such for example

as coarse yarns to produce the transparent and the wool-like effects, which heretofore has not been possible as regards transparent effects and has not been possible with some of the acids as regards wool-like effects, since in the treatment of such coarse yarns either a merely superficial change took place, or in case the action of the acid was allowed to continue for a sufficient duration to cause greater penetration of the yarn, then partial or even complete parchmentizing or destruction of the yarn resulted.

I am aware of the fact that it has been proposed (see German Patent No. 139,669 to A. Wohl, and Schwalbe, Chemie de Cellulose, 1911, p. 320) to use a heterocyclic base in producing cellulose acetate by means of acetylchloride. But that proposed use of a heterocyclic base in the presence of an organic acid chloride for the purpose of converting chopped or pulverized paper directly into cellulose acetate, is quite a different matter from that of the present invention relating to the ennobling or finishing of fibrous goods (yarns and webs) to produce permanent effects thereon.

According to the present invention, in all cases in which concentrated mineral acids have heretofore been employed for producing permanent effects in fibrous materials, such for example as in the production of said transparent organdie, wool-like effects, linen effects, etc., the acid may advantageously be used in the presence of a suitable heterocyclic compound, such for example, as the salts of the heterocyclic bases miscible with the acid, or, in other words, the concentrated acids of the strengths heretofore used may be replaced by a mixture of said acid and a heterocyclic base, in which case the acid at even greater concentration may be allowed to act for a much longer period of time to effect a more complete and desirable finish, without the heretofore accompanying danger. As is understood in the art, it is desirable in all instances to wash the goods after the acid treatments and also after the caustic treatments when used.

For the purpose of this invention, suitable heterocyclic compounds or heterocyclic bases are such heterocyclic compounds or bases as have pyridin characteristics for the purpose, and as are miscible in the respective mineral acids with which they may be used for producing the particular permanent finish. Among these compounds or bases are included for example various heterocyclic compounds or bases containing a pyridin nucleus. For example, in the case of the use of sulfuric acid, such heterocyclic compounds or bases may be used as have pyridin characteristics for the purpose, and as are miscible in the sufuric acid, and in the case of nitric acid such heterocyclic compounds or bases would be used as have pyridin characteristics for the purpose, and as are miscible in nitric acid.

Also for the purpose of this invention suitable mineral acids are those that are capable of dissolving cellulose fibres so as to produce a permanent effect without detrimental effect, such for example, as the acids mentioned in the above set forth United States patents.

Such features of my invention herein described but not herein claimed are covered by my copending application Serial No. 631,374, filed April 11, 1923, for permanent finish vegetable material and process of producing same.

While I have described my invention with particularly, by way of example, with respect to certain preferred specific deterring or stabilizing reagents, it will be understood by those skilled in the art, after understanding my invention, that other reagents may be substituted without departing from the spirit or scope of the invention, and I aim in the appended claims to cover the use of all such reagents as come within the scope of the invention.

What I claim as new and desire to secure by Letters Patent, is:

1. Vegetable fibrous material (yarns and goods made therefrom) the fibres of which have been structurally altered by chemical treatment, which treatment comprises a treatment with a suitable concentrated mineral acid in the presence of a suitable heterocyclic compound having pyridin characteristics for the purpose, to produce a permanent finish wool-like effect.

2. Vegetable fibrous material (yarns and goods made therefrom) the fibres of which have been structurally altered by chemical treatment, which treatment comprises a treatment with sulfuric acid in the presence of pyridine to produce a permanent finish wool-like effect.

3. Vegetable fibrous material (yarns and goods made therefrom) having a permanent finish wool-like effect produced by chemical treatment of the material, which treatment comprises a treatment with a suitable concentrated mineral acid containing a suitable heterocyclic compound containing a pyridin nucleus, and a treatment with caustic alkali.

4. Vegetable fibrous material (yarns and goods made therefrom) in which the fibres have undergone a permanent structural change by chemical treatment, which treatment comprises a treatment with a suitable concentrated mineral acid containing a suitable heterocyclic compound having pyridin characteristics for the purpose, and a treatment with caustic alkali, to produce a wool-like effect with a bright tone.

5. Vegetable fibrous material (yarns and goods made therefrom) having a permanent wool-like effect produced by chemical treat-

ment of the material, which treatment comprises a treatment with a suitable concentrated mineral acid containing pyridine, and a treatment with caustic soda.

6. Vegetable fibrous material (yarns and goods made therefrom) the fibres of which have been structurally altered by chemical treatment, which treatment comprises a treatment with sulfuric acid of a concentration substantially over 51° Bé. having mixed therewith a heterocyclic compound having pyridin characteristics for the purpose and which stabilizes the reaction of said acid on the material.

In testimony whereof I have signed my name to this specification.

ALBERT BODMER.