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METHOD OF NITRATING CELLULOSE LACE-BACKING FABRICS

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4 Claims. (Cl. 8—120)

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This invention relates to nitrated cellulose fabrics and methods for making such fabrics. More particularly, the fabrics of this invention are especially adapted for use in the manufacture of lace and the like articles.

Heretofore, backing or ground fabrics which are to be used in the manufacture of lace, have been prepared by nitrating cotton or other cellulose fabrics in nitric acid baths. The fabric resulting from such a procedure has lace stitched thereon and the composite material is passed through a bath of caustic alkali. The nitrated backing fabric disintegrates in the caustic alkali, leaving the lace structure which is unimpaired by the caustic alkali, to be removed from the bath.

The nitrated fabrics originally used were highly inflammable due to their high nitrogen content. In order to produce fabrics having a substantially lower degree of inflammability, the nitric acid baths were modified by the addition of phosphoric acid. The nitration of the fabrics was also adjusted to materially reduce the nitrogen content of the nitrated fabric. The fabrics so produced have nitrogen contents of approximately 0.4% to 1.0% by weight.

The nitrated fabrics of low nitrogen content as described, present several difficulties. One disadvantage relates to the time intervals required for the nitrating procedure. It has been necessary to leave cotton fabrics submerged in phosphoric acid modified nitrating baths for periods extending from one half hour to as much as thirty-six hours in order to achieve the required degree of nitration. Accordingly it is an object of this invention to produce a nitrated cellulose fabric by subjecting a cellulose fabric to the action of a phosphoric acid modified nitrating bath wherein the time interval necessary to produce a predetermined degree of nitration is reduced to a fraction of that required in procedures already known in the art.

A further difficulty attendant to the use of the nitrated fabrics of low nitrogen content is their relatively low tensile strengths. Inasmuch as the nitrated fabrics are used in the manufacture of lace and the fabric is subjected to the various strains incident to its passage through the lace making machines, an adequate and uniform tensile strength is necessary. The nitrated fabrics having low nitrogen content (1.0% or less) are characterized by a relatively low and non-uniform tensile strength. Therefore, it is a further object of this invention to produce an improved nitrated cellulose fabric which shall

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have a uniform and relatively high tensile strength.

The nitrated fabrics of low nitrogen content have a tendency to deteriorate in a relatively short time during storage. It has been observed that such fabrics will disintegrate within a period of two to three months. Accordingly, a further object of this invention is to produce an improved nitrated cellulose fabric which will withstand deterioration particularly in the presence of agents promoting conditions of mildew, for an almost indefinite period of time.

Another object of this invention is the production of an improved nitrated cellulose fabric by means of a nitric acid-phosphoric acid bath wherein the final product shall have a nitrogen content of approximately 2.0% to 6.0% by weight. Such a nitrated fabric shall be further characterized by a low degree of inflammability and yet is readily disintegrated in alkali.

Still another object of this invention is to nitrate a cellulose by the use of a nitric acid-phosphoric acid bath of predetermined concentration whereby the cellulose fabric may be passed continuously through the bath and yet result in a final product having the desired nitrogen content.

Other objects of my invention will in part be obvious and in part hereinafter pointed out.

It has been found that a cellulose fabric such as cheese cloth or the like may be nitrated to a nitrogen content of more than 1.0% and less than 3.0% yet produce a nitrated fabric having a relatively high tensile strength as compared to fabrics nitrated to a nitrogen content of less than 1.0%. It has also been found that fabrics having the higher nitrogen content still retain the property of disintegration in caustic alkali as well as other alkaline baths. Furthermore the period of nitration may be considerably reduced as compared to that required in processes known in the art.

Accordingly there is provided, in accordance with the present invention, a nitrating bath which comprises a mixture of nitric acid and phosphoric acid wherein the nitric acid may constitute from about 40% to about 60% by volume. The phosphoric acid content of the bath may be from about 60% to about 40% by volume. Small amounts of sulfuric acid may be optionally added to the bath. The cellulose fabric is passed through the bath in a continuous manner, is removed from the bath, exposed to the air for an appropriate time interval and is finally washed with water and weak alkali solutions to neutralize excess acid.

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By way of illustration and without limiting myself to this particular form of my invention, the following illustrates bath compositions and appropriate procedure which will give the desired results:

1.

Parts by volume

Nitric acid—concentration about 97%----- 60
Phosphoric acid—concentration about 75%-- 40

Cotton fabric was continuously passed through the bath at a temperature of 100°–110° F. The fabric had a contact period within the bath of approximately 0.5 minute. The nitrated fabric coming out of the bath was exposed to the air for approximately 4.5 minutes and was then passed through successive washing solutions of water and weak alkali. The nitrated fabric thus produced had a nitrogen content of approximately 6.0%.

Parts by volume

Nitric acid—concentration about 97%-----40
Phosphoric acid—concentration about 75%----60

Cotton fabric was passed through the bath in the manner described in Example 1. Using the same time intervals and temperatures, the final product had a nitrogen content of approximately 2.0%.

In both of the above examples sulfuric acid may be optionally added to the baths.

The temperatures indicated in the above examples may be varied with a corresponding variation in the time intervals of nitration and exposure in air, providing the final product has a nitrogen content of between 2.0% and 6.0% by weight.

A cotton fabric nitrated in accordance with the procedures outlined above and having a nitrogen content of 4.01% was tested for its tensile strength and showed a value of 23 pounds per inch.

Similar cotton fabrics which had been nitrated to nitrogen contents of 1.0% or less were also tested and showed tensile strengths of approximately 50% to 75% less than the tensile strengths of nitrated fabrics having nitrogen contents above 2.0%.

The nitrated cellulose fabrics of this invention are also characterized by a high degree of resistance to mildew. Upon exposure of the fabric to conditions of mildew it was noted that there was no decrease in tensile strength as compared to the tensile strength of the unexposed fabric.

The nitrated fabrics of this invention are stable during storage periods of almost indefinite length.

In the case of nitrated cellulose fabrics of low nitrogen content (1% or less) it has been observed that they fail to stand up over a period of time and disintegrate while in storage within two (2) or three (3) months.

It was further determined that the nitrated cellulose fabrics of this invention are non-toxic and do not irritate the human skin upon contact therewith.

It will thus be seen that there is provided a product and a method for manufacturing the same in which the several objects of this invention are achieved and which is well adapted to meet the conditions of practical use.

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As various possible embodiments might be made of the above invention, and as various changes might be made in the embodiment above set forth, it is to be understood that all matter herein set forth is to be interpreted as illustrative and not in a limiting sense.

Having thus described my invention, I claim as new and desire to secure by Letters Patent:

1. The process of producing lace backing fabric comprising treating natural cellulose, open mesh fabric with mixed acid comprising from 60 to 40 parts by volume 97% nitric acid and from 40 to 60 parts by volume 75% phosphoric acid for about 0.5 minute and exposing the treated fabric to air for about 4.5 minutes.

2. The process of producing lace backing fabric comprising passing natural cellulose, open mesh fabric through a bath comprising a mixture of 60 parts by volume of 97% nitric acid and 40 parts by volume of 75% phosphoric acid for about 0.5 minute, exposing the treated fabric to air for about 4.5 minutes and washing the treated fabric with water and dilute alkali.

3. In the process of forming lace backing fabric, the steps of passing natural cellulose, open mesh fabric through a bath comprising from 60 to 40 parts by volume of 97% nitric acid and from 40 to 60 parts by volume of 75% phosphoric acid, retaining the fabric in the bath for about 0.5 minute, removing the fabric from the bath and permitting the nitrating action to continue for about 4.5 minutes and removing the unreacted acids from the fabric.

4. In the process of nitrating natural cellulose open mesh, lace backing fabric, the steps of continuously passing the fabric through a bath comprising 40 to 60 parts by volume of 97% nitric acid and 60 to 40 parts by volume of 75% phosphoric acid for a time interval of approximately 0.5 minute, withdrawing the nitrated fabric from the bath and exposing it to air for a time interval of approximately 4.5 minutes and neutralizing the excess acid in said fabric by washing with water and dilute alkali, the said fabric having a nitrogen content of from about 2.0% to about 6.0% by weight.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,884,802	Miles et al.	Oct. 25, 1932
2,092,749	Bradshaw	Sept. 14, 1937
2,302,107	Datlow	Nov. 17, 1942

OTHER REFERENCES

Farmer, "Mechanism of the Formation of Nitric Esters," Jour. Soc. Chem. Ind., Feb. 27, 1931, pages 76T, 77T.

Desmaroux et al., C. R. Acad. Sci., 1941, 212, pages 396–398, Abst. C:A. 1944, 38 16372; Jour. Text. Inst., Aug. 1944, A 363, 364.