United States Patent Office

2,993,746

Patented July 25, 1961

2,993,746

TREATMENT OF THPC FLAME-RESISTANT FABRICS WITH STRONG MINERAL ACIDS TO IMPROVE THEIR TEAR STRENGTH AND PRODUCTS PRODUCED THEREBY

Thomas D. Miles, Southboro, and Francis A. Hoffman, Millis, Mass., and Armando C. Delasanta, Woonsocket, R.I., assignors to the United States of America as represented by the Secretary of the Army

No Drawing. Filed Mar. 27, 1959, Ser. No. 802,579

14 Claims. (Cl. 8—116)

(Granted under Title 35, U.S. Code (1952), sec. 266)

The invention described herein, if patented, may be manufactured and used by or for the Government for governmental purposes, without the payment to us of any royalty thereon.

This invention relates to a method of improving the tear strength of cellulosic fabrics which have been rendered flame resistant by impregnation with tetrakis-hydroxymethyl phosphonium chloride (THPC).

It is known that treatment of cotton fabrics and other cellulosic fabrics (e.g. linen fabrics), with THPC imparts a large degree of flame resistance to the treated fabrics. However, this desirable effect is largely offset by a marked decrease in tear strength, which greatly decreases the military and other utility of the flame-resistant fabrics thus produced.

We have now discovered a simple process whereby the tear strength of THPC-treated cellulosic fabrics can be greatly improved without substantially reducing their flame resistance.

More particularly, we have now found that a substantial and surprising improvement in the tear strength of THPC treated fabrics can be effected by partial hydrolysis for a short time in a dilute mineral acid, preferably hydrochloric acid, e.g. of about 1–10% concentration. This hydrolysis is performed as an aftertreatment, and is preferably carried out at normal fabric washing temperatures, e.g., at 75–100° C., followed by substantially neutralizing the fabric.

The following examples illustrate the practice of our invention:

EXAMPLE I

Carded sateen, cotton cloth (8.5 oz. weight) was treated with THPC as follows: a Solution A, containing 467 parts THPC, 450 parts water, and 84.5 parts triethanolamine, and Solution B, containing 267.8 parts methylol melamine resin, 278.5 parts urea, and 1265 parts water were prepared. The fabric was then passed through a solution made by combining Solution A and Solution B in a 1 to 1 ratio, using 2 dips and 2 nips to achieve a 60% wet pick-up. The treated fabric was oven dried at 85° C. for approximately 10 minutes followed by a 5-minute cure at 140° C. Partial hydrolysis was carried out, by immersing the fabric in a solution of 2% hydrochloric acid and 5% urea, at 85° C. for various lengths of time, as shown in Table I below. The partially hydrolyzed fabric was then neutralized by immersion in a solution of 1% sodium carbonate, at 38° C., for 5 minutes, rinsed and dried at 90° C. The following results were obtained in determining the tear strength of the treated fabric samples:

Table I

Time of Hydrolysis (minutes)	Tear Strength (lbs.)		Fire Resistance		
	Warp	Filling	After Flame (sec.)	After Glow (sec.)	Char Length (in.)
0 untreated (no THPC)	9.6	11.7			
0 treated (THPC)	4.1	3.7	0	1.6	3.3
1 treated (THPC)	7.1	7.1	0	0	3.5
2 treated (THPC)	6.4	6.8	0	0.7	3.7
3 treated (THPC)	5.8	5.4	0	0.7	3.2
4 treated (THPC)	6.8	7.5	0	1.2	3.8
5 treated (THPC)	6.0	7.7	0	0.3	6.5

EXAMPLE II

Carded sateen (8.5 oz. wt.) was treated with the standard THPC formulation as described in Example I except that plant equipment was used. The tear strength of the THPC treated fabric was 6.6 lbs. in the warp and 5.4 lbs. in the filling. This treatment was followed by a cold water rinse and drying at 90° C. The tear strength of the partially hydrolyzed and neutralized fabric was again determined and was found to have risen to 8.9 lbs. in the warp and 8.5 lbs. in the filling, which represents a substantial improvement. The fabric retained its flame resistance, and was resistant to subsequent water leaching.

EXAMPLE III

Example I was repeated by substituting a stoichiometric equivalent proportion of phosphoric acid in lieu of hydrochloric acid. The tear strength of the thus treated fabric was improved to about the same degree as in Example I. However, the fabric was less resistant to leaching during subsequent laundering.

EXAMPLE IV

Example II was repeated with the use of a 2% hydrochloric acid solution, without urea, for the partial hydrolysis of the dried THPC-treated fabric. The results summerized in Table II, below, were obtained in determining the tear strength of fabric samples treated in accordance with this example:

Table II

Time of Hydrolysis (minutes)	Tear Strength (lbs.)	
	Warp	Filling
Untreated (no THPC)	11.6	12.5
0 treated (THPC)	5.3	5.5
2 treated (THPC)	7.3	7.3
4 treated (THPC)	7.0	7.8

Laundering tests of the treated samples showed that fire resistance of the treated samples was retained through up to 9 laundering cycles.

It will thus be seen that by the use of our method of after-treating a THPC-impregnated flame-resistant fabric with a strong mineral acid such as hydrochloric acid to effect a partial hydrolysis of the fabric, its tear strength is materially improved. Prior to our invention, it was not possible to use a THPC treated cellulosic fabric for any purpose for which tear strength is important. While our invention does not completely restore the tear strength

of the THPC treated fabric to the equivalent of that of the untreated (i.e. not flameproofed) fabric, it does improve the tear strength characteristics of the THPC-treated fabric to such an extent that it can be used as a tent fabric, tarpaulin, or other similar military or civilian textile item, for purposes where both flame strength and a degree of tear resistance are required. Thus, we have effected an acceptable and practical compromise between the high tear strength of a non-flameproofed cellulosic fabric, and the desirable flame resistance characteristics of a cellulosic fabric whose tear strength has been substantially reduced, however, by a flame-resistance-imparting THPC treatment.

We are of course aware that we are not the first to treat cellulosic fabrics, such as cotton fabrics, with hydrochloric or other mineral acids. However, such treatments of the prior art are for an entirely different purpose, namely to modify the appearance and feel of a non-flameproofed cotton fabric ("parchmentizing") and such treatments have no effect on the tear strength of a chemically untreated cotton fabric. On the other hand, we believe ourselves to be the first to have discovered the beneficial effect of a hydrochloric acid or other mineral acid after-treatment of a THPC-impregnated flame-resistant fabric, so as to restore a large part of the loss of tear strength suffered by the fabric by reason of the THPC treatment.

Without confining ourselves to any particular theory on which to base the efficiency of our invention, we postulate that the partial hydrolysis of the cellulose molecules effected by the hydrochloric acid (or other mineral acid) treatment counteracts the adverse effect on the tear strength of the cellulosic fibers exerted by the bonding of the flame-resistance-imparting impregnant.

It will of course be apparent to those skilled in the art that the operating conditions described in our foregoing specification and examples may be varied in a number of details without departing from the spirit of our invention. We thus intend to include any such changes and modifications within the scope of our invention, and to that end define our invention by the appended claims.

We claim:

1. In a method of improving the tear strength of a cellulosic fabric which has been rendered flame-resistant with tetrakis-hydroxy-methyl phosphonium chloride and a methylol melamine resin, the improvement which comprises immersing the thus treated flame-resistant fabric in a dilute aqueous solution of a strong mineral acid until the cellulose molecules of said fabric have been partially hydrolyzed and the tear strength of said fabric has been increased, then removing said fabric from said aqueous solution, substantially neutralizing said fabric, and rinsing and drying said fabric.

2. In a method of improving the tear strength of a cellulosic fabric which has been rendered flame-resistant with tetrakis-hydroxy-methyl phosphonium chloride and a methylol melamine resin, the improvement which comprises immersing the thus treated flame-resistant fabric in an aqueous solution of a strong mineral acid of about 1–10% concentration until the cellulose molecules of said fabric have been partially hydrolyzed and the tear strength of said fabric has been increased, then removing said fabric from said aqueous solution, substantially neutralizing said fabric, and rinsing and drying said fabric.

3. In a method of improving the tear strength of a cellulosic fabric which has been rendered flame-resistant with tetrakis-hydroxy-methyl phosphonium chloride and a methylol melamine resin, the improvement which comprises immersing the thus treated flame-resistant fabric in an aqueous solution of hydrochloric acid of about 1–10% concentration until the cellulose molecules of said fabric have been partially hydrolyzed and the tear strength of said fabric has been increased, then removing said fabric from said aqueous solution, substantially neutralizing said fabric, and rinsing and drying said fabric.

4. In a method of improving the tear strength of a cellulosic fabric which has been rendered flame-resistant with tetrakis-hydroxy-methyl phosphonium chloride and a methylol melamine resin, the improvement which comprises immersing the thus treater flame-resistant fabric in an aqueous solution of hydrochloric acid of about 2% concentration until the cellulose molecules of said fabric have been partially hydrolyzed and the tear strength of said fabric has been increased, then removing said fabric from said aqueous solution, substantially neutralizing said fabric, and rinsing and drying said fabric.

5. In a method of improving the tear strength of a cellulosic fabric which has been rendered flame-resistant with tetrakis-hydroxy-methyl phosphonium chloride and a methylol melamine resin, the improvement which comprises immersing the thus treated flame-resistant fabric in an aqueous solution of hydrochloric acid of about 1–10% concentration in the presence of urea until the cellulose molecules of said fabric have been partially hydrolyzed and the tear strength of said fabric has been increased, then removing said fabric from said aqueous solution, substantially neutralizing said fabric, and rinsing and drying said fabric.

6. In a method of improving the tear strength of a cellulosic fabric which has been rendered flame-resistant with tetrakis-hydroxy-methyl phosphonium chloride and a methylol melamine resin, the improvement which comprises immersing the thus treated flame-resistant fabric in an aqueous solution of about 2% hydrochloric acid and about 5% urea until the cellulose molecules of said fabric have been partially hpdrolyzed and the tear strength of said fabric has been increased, then removing said fabric from said aqueous solution, substantially neutralizing said fabric, and rinsing and drying said fabric.

7. Method according to claim 1, wherein said fabric is neutralized in a dilute aqueous solution of sodium carbonate.

8. Method according to claim 3, wherein said fabric is neutralized in a dilute aqueous solution of sodium carbonate.

9. The method according to claim 1, wherein said aqueous solution has a temperature of about 75°–100° C.

10. The method according to claim 3, wherein said aqueous solution has a temperature of about 75°–100° C.

11. The method according to claim 5, wherein said aqueous solution has a temperature of about 75°–100° C.

12. The method according to claim 11, wherein said fabric is immersed in said aqueous solution for about one to five minutes.

13. The method according to claim 1, wherein said fabric is immersed in said aqueous solution for about one to five minutes.

14. A flame-resistant cellulosic fabric of improved tear strength produced by the method of claim 1.

References Cited in the file of this patent

UNITED STATES PATENTS

2,264,229	Wallach	Nov. 25, 1941
2,648,597	Nielsen	Aug. 11, 1953
2,668,096	Reeves	Feb. 2, 1954

OTHER REFERENCES

Campbell: Abstract 95,869, April 29, 1952 (657 O.G. 1586).

Ind. and Engineering Chem., December 1943, pp. 1281–1283.