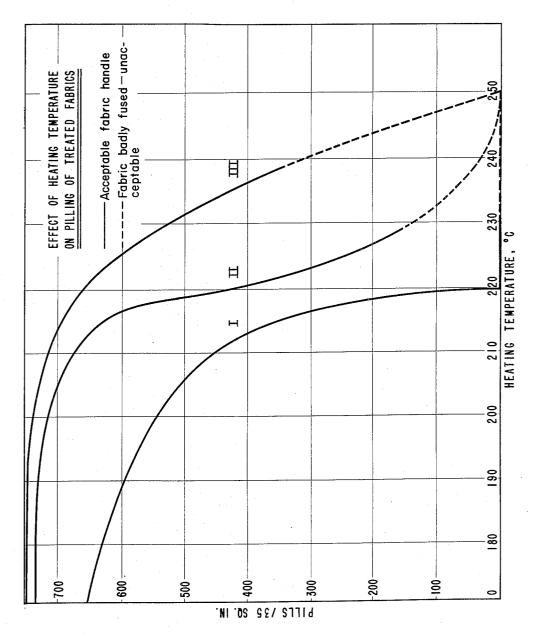
FINISHING FABRICS

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FINISHING FABRICS

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This invention relates to a process of finishing syn- 15 thetic fabrics and more particularly to a process of reducing the pilling tendency of fabrics containing polyester fibers. It has been observed for some time that fabrics made from certain synthetic fibers have a tenare known in the trade as "pills." It has also been observed that this undesirable property is found to a greater extent on fabrics woven from synthetic fibers which have high strength and abrasion resistance. One explanation of this tendency is that the fibers of these polymers do 25 not wear away as readily as those of the lower strength fibers either before or after the formation of "pills." This explanation is supported by the fact that pill formation is almost directly proportional to the abrasion resistance of a given fabric.

It is an object of this invention to provide a process for finishing fabrics containing polyester fibers which avoids formation of "pills" or reduces the formation of "pills" to an acceptable level. Another object is to provide a process for reducing or eliminating pill formation 35 in fabrics containing a major proportion of ethylene terephthalate polymer staple fibers without significant impairment of the fabric strength, abrasion resistance or other desirable properties of these fabrics. Other objects of the invention will be apparent as the description 40 proceeds.

These and other objects are accomplished by treating woven or knitted fabrics containing linear condensation polyester fibers in major proportion with a liquid impregnating medium containing at least 5% by weight 45 of certain polyether glycol compounds. These compounds are polyether glycols, their esters, or ethers and mixtures thereof having the formula

$$A-(O-R_1-O-R_2-O-R_3)_Y-B$$

in which R₁, R₂, and R₃ are the same or different lower alkylene units such as methylene, ethylene, propylene and so forth up to about 5 carbon atoms. In the formula, A stands for hydrogen, alkyl, aryl, or an acyl group; and B is OH, alkyl ether, aryl ether or an alkyl or aryl ester 55radical. In the formula, Y has a value to produce a molecular weight of the alkylene oxide component of at least about 300.

The liquid is applied to the fabric by impregnation, padding or spraying. After the fabric is treated with the 60 impregnating liquid, excess liquid is usually removed by squeeze rolls, extraction or other conventional means. and the fabric dried. Then the surface only is heated (i.e., either one or both surfaces) rapidly to a temperature of at least 200° C., but below the fusion temperature of the surface fibers. The heat treatment should be such that the surface fibers are heated, and the source of heat, or the fabric, removed before the temperature of the entire fabric is raised to the temperature of its surface. The back of the fabric may be cooled if necessary 70 while the surface is being heated.

The time-temperature cycle of heating is thereby con-

trolled to avoid degradation of the fibers of the main body of the fabric beneath the surface. One convenient way of determining whether the fabric itself has retained its desirable properties or whether the severity of the finishing treatment has appreciably changed the aesthetic and other physical properties of the body of the fabric is to measure the weight loss of the treated and untreated fabric after subjection to a standard abrasion test defined hereinafter. In order to preserve the good aesthetic and other properties of the polyester fabrics, it has been found that independent of the fabric composition or construction, the fabric must not undergo an absolute percent weight loss in the adopted abrasion test of more than 5 percentage units greater than the absolute percent weight loss of the untreated control fabric when subjected to the same abrasion test; i.e., the differential weight loss between treated and untreated fabric must not be more than

The abrasion resistance of the fabrics is measured in dency to form small fuzz balls on the surface which 20 terms of fabric weight loss in the abrasion test described on page 3 of the A.A.T.C.C. Bulletin entitled, "The A.A.T.C.C. Accelerotor," July 1953, using a 4½" square sample and a 434" propeller blade driven at 1800 r.p.m. with a 400 E. grit closed coat "Durite" abrasive paper (Behr-Manning Co.) as the abrasive liner for a five-minute testing period.

The efficiency of the process in reducing pilling is determined by number of pills formed per 35 in.2 of surface fabric as determined in accordance with ASTM D-1375-55T, "Test for Pilling Propensity of Textile Fabrics-Part C-Brush and Sponge Method."

As mentioned above, the value of Y in the formula

$$-(O-R_1-O-R_2-O-R_3)_{Y}-$$

is such as to yield a molecular weight of at least about 300. There is no known maximum limitation for the molecular weight although the lower range, particularly that of from 400 to 600, is preferred since compounds in this range are more rapidly soluble as well as being more available and economical. The critical nature of the minimum molecular weight of the polyether glycol is shown in Table I below, wherein one particular polyester fabric is treated under optimum conditions in accordance with this invention while varying only the average molecular weight of the alkylene oxide unit in a series of polyethylene glycols.

TABLE I

	Average mol. wt. of glycol: Pills/35	in.2
0	62	535
	150	410
	200	350
	400	0
	~~~	0
ŏ	1000	0
	4000	0
	20000	0
	20000	0

The polyether glycol may be etherified or esterified without affecting the pill reducing tendency of the treatment so long as the average molecular weight of the alkylene oxide unit is maintained above about 300. The glycol treatment may involve the use of a single glycol or derivative thereof, or more practically, it will involve the use of mixtures of glycols which are commercially available and, therefore, much less expensive for large-scale use. Specific examples of the polyether glycols and their derivatives which may be used in accordance with this invention include polyethylene glycol 400, polyethylene glycol 600, polymethylene glycol 400, polypropylene glycol 600, polyethylene glycol 400 monolaurate, poly-

ethylene glycol 400 monostearate, polypropylene glycol 400, polytetramethylene glycol 600, polyethylene glycol 600 monolaurate, polyethylene glycol 1000 monolaurate, polyethylene glycol 1500 mono aurate, polyethylene glycol 400 monooleate, polyethylene glycol 600 monostearate, polyethylene glycol 600 monopalmitate, monomethoxy polyethylene glycol 550, dibutyl ether of polyethylene glycol 400, trimethylnonyl ether of polyethylene glycol 400, methylphenyl ether of polyethylene glycol 400, p-n-butylphenyl ether of polyethylene glycol 400, 10 p-isobutylphenyl ether of polyethylene glycol 400, pdodecylphenyl ether of polyethylene glycol 400, p-isooctylphenyl ether of polyethylene glycol 400, oleyl ether of polyethylene glycol 400, 1-hexyl-2-naphthyl ether of polyethylene glycol 600, 1-octyl-2-naphthyl ether of poly- 15 ethylene glycol 600, 1-hexyl (2,3,4,5-tetrahydroxy, 6-stearate) ether of polyethylene glycol 400, and the like. The numbers after the name indicate the approximate average molecular weight, for example, polyethylene glycol 400 is a mixture of polyethylene glycols, the weight 20 average molecular weight of the mixture being about 400.

The liquid treating medium which is applied to the polyester fabric may be composed wholly of the polyether glycol or more practically, the glycol or derivative thereof will be dissolved in a miscible solvent such as water, alcohol, acetone and the like. The use of solutions of the glycol is more economical when carried out on a commercial scale and involves the use of less glycol and, therefore, less loss or recovery of glycol is involved. If the liquid medium contains any diluent for the glycol, the medium must be applied to the fabric with a minimum concentration of 5% of the glycol based on the total weight of the liquid medium in order to achieve a satisfactory reduction of the pilling count in the resulting fabrics. In general, for the lowest levels of pilling, the 35 preferred operating concentration for the padding bath is an aqueous medium containing 20%-40% by weight of polyether glycol.

Pillable fabrics may be treated by the process of this invention (i.e., glycol treatment followed by heat treatment), either with or without benefit of an additional modifying agent. Typical modifying agents which may be employed in the liquid medium, together with glycols, include ester exchange catalysts, such as the metallic salts or oxides of calcium, magnesium, lead, tin and zinc (e.g., zinc nitrate, zinc acetate, magnesium oxide, and the like).

In addition to the critical nature of the liquid-treating medium, it has also been found that the heat-treating step is likewise critical for achieving the objects of this The use of too high a temperature of heat 50 treatment will cause fusion of the surface fibers which in turn destroys the aesthetic properties of the fabric, as well as causing too great a drop in abrasion resistance and other strength properties of the fabric.

The maximum temperature will be governed by the 55 melting point or degradation of the particular fibers used, which, of course, will change somewhat with the composition of any copolyesters making up the fabric, as well as the nature of other fiber components when used in a blend with polyester fibers.

The minimum temperature necessary to achieve a satisfactory reduction in pilling propensity in this process has been found to be about 200° C. when polyethylene terephthalate is the polyester fiber in the fabric. Furthermore, the time/temperature relationship of the heat 65 treatment must be carefully controlled so that the fabric properties do not suffer impairment. Too long a treatment, even at optimum temperatures, will tend to degrade the fibers within the body of the fabric. Normally, the heat treating step will be carried out on the impregnated 70 fabric so that the surface of the fabric will be raised rapidly to a temperature of 200° C.-230° C., preferably 218° C.-222° C., and kept at that temperature for several seconds, although it will be obvious to those practicing the invention that no absolute time can be set for optimum 75

pill reduction without also specifying a particular temperature for the particular fabric being treated.

A satisfactory criterion for determining the best time/temperature relationship is to measure the abrasion resistance of a sample of fabric, both untreated and treated by the process of this invention. Since the absolute weight loss during abrasion testing is dependent on fabric construction and composition, the best criterion for the effect of the instant process on the fabric is that the absolute percent weight loss of the treated fabric by the standard abrasion test specified should not be more than 5 percentage units greater than the absolute percent weight loss of the untreated control fabric subjected

to the same abrasion test.

A guide for selection of optimum temperatures for use in this invention will be found in the drawing in which the single figure is a graph in which the number of pills per 35 in.² section is plotted against temperature of the surface of the fabric treated. The curves show the pills formed at various temperatures when a 100% polyethylene terephthalate fabric is treated (I) with 20%-50% polyethylene glycol (av. M.W. 600) in aqueous solution, (II) with 5% to 10% of the same glycol in aqueous solution and (III) with no treating agent. These three curves involved the use of the gabardine fabric described in Example VI. The dotted lines represent portions on the curves where the fabric fused or had deteriorated to an extent that its feel or handle was considered unsatisfactory commercially. The solid lines represent conditions which result in little or no impairment in the handle or feel of the fabric. The curves represent data obtained from one particular type of fabric. Curves for other types have the same general shape but do not necessarily coincide with those shown.

The number of pills per 35 sq. in., as measured in the standard ASTM test, considered acceptable depends upon the ultimate use of the fabric and its construction. Naturally zero pills is desirable, but up to 50 or even 100 is sometimes acceptable, and, considering the wide variety of fabric constructions and compositions available on the market, these levels represent a considerable improve-

ment over the prior art.

The particular manner by which the heat is applied to the surface of the impregnated fabric is also a critical part of the heat-treating step. It has been found that merely heating the fabric in an ordinary air oven is not a satisfactory method of achieving the objects of this invention, chiefly because it does not raise the temperature of the surface fibers to 200° C. rapidly enough. Using this method by the time the surface fibers reach the critical temperature of this invention, the fibers through the body of the fabric have become degraded and the abrasion resistance and strength of the whole fabric is severely impaired.

Any means for heating the surface of the fabric only may be used and a large variety of suitable apparatus is available in the trade for rapidly raising the temperature of the surface fibers to 200° C., such as, for example, a preheated ordinary hand iron, a standard series of National hot rolls, and an Andrews-Goodrich oven or a Morrison oven, both of which contain rolls within an oven which is preheated to the desired temperature. All of this apparatus involves direct contact between a heated surface and the surface of the fabric, which is the preferred manner of carrying out the heating step in this invention. Normally, with such contact heating equipment, the fabric will be kept taut so that the surface of the fabric conforms accurately to the contour of the heated surface, although this is not a critical limitation in the process.

It has been noted that the increased pilling resistance resulting from the instant process is developed selectively only on the surface of the fabric in contact with the heated surface; i.e., the side of the fabric in contact with the hot roll, when a hot roll is employed as the heating means. Any other apparatus or method for rapidly achieving the minimum temperature selectively at the surface of the fabric may be used such as radiant heat, and the like. In such instances when the heating means does not contact the surface of the fabric, the increased pilling resistance will be developed selectively on the side, or sides, of the fabric facing the source of heat.

The woven and knitted fabrics which may be advantageously treated in accordance with this invention include those composed of 100% linear condensation poly- 10 ester fibers, as well as those constructed of blends of other fibers or yarns containing some polyester fiber, the preferred one being polyethylene terephthalate. Other fabrics which may be treated to reduce the pill count may contain other polyester fibers such as those made from 15 ethylene glycol and bibenzoic acid, ethylene glycol and hexahydroterephthalic acid, trimethylene glycol and terephthalic acid, or tetramethylene glycol and terephthalic acid, as well as fabrics containing copolyester fibers such as those made from tetramethylene glycol, hexa- 20 methylene glycol and terephthalic acid, or those from dimethyl terephthalate, dimethyl sebacate and ethylene glycol, or similar copolyesters. The fabric may also be blends of polyester fibers with wool, cotton and other natural fibers and/or rayon, nylon, polyacrylonitrile, 25 cellulose acetate, and other synthetic fibers. It is obvious one would not employ blend fibers which will not withstand the heat treating temperatures of this invention.

Preferably, the two-step treating process of the invention is applied to fabrics before they are dyed, although 30 in some instances, the glycol or derivative may be applied in the dye bath (e.g., when dyeing in accordance with the procedure described by Gibson in U.S. 2,663,612), or the two-step treatment may be applied after dyeing and after or during conventional finishing if the 35 dyes and finishes used are stable to the final heat-treating step.

The following examples illustrate specific embodiments of this invention without intending to limit the invention. It is to be understood that the treating agent in the examples may be replaced by a like amount of any compound falling within the formula given above.

#### Example 1

A 3 x 2 twill, sheen gabardine woven of 100% polyethylene terephthalate staple yarn was padded with an aqueous solution containing 40% by weight of polyethylene glycol 600 (i.e., average molecular weight=600). The fabric was then heat treated on a National hot roll machine under tension at 220° C. and an exposure time 50 of about 25 seconds, each face of the fabric receiving about 12.5 seconds exposure. The fabric was then scoured and dried. The fabric, before treatment, showed 435 pills/35 in.² as measured by the ASTM pilling test, while the fabric, after treatment, showed 72 pills/35 in.². 55 The untreated control fabric after dyeing and conventional finishing showed 315 pills/35 in.², whereas the treated fabric after the same final dyeing and finishing steps showed 10 pills/35 in.².

Another sample of the same fabric was padded with an aqueous solution containing 40% by weight of polyethylene glycol 600, and 1% by weight zinc nitrate. The fabric was dried in a hot-air pin tenter for 1.5 minutes at 121° C., and was then heat treated on a National hot roll machine at 220° C., as described above. The fabric 65 after treatment showed 2.5 pills/35 in.², and a weight loss of 2.65% in the standard abrasion test compared with a weight loss of 2.61% for the untreated fabric, showing the properties of the fabric had not been degraded by the finishing treatment.

#### Example II

A 3 x 2 twill, sheen gabardine fabric composed of 65% polyethylene terephthalate staple and 35% by weight rayon staple was padded and dried in the same manner 75

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as the fabric of Example I using the same polyethylene glycol and catalyst. It was heat cured on a National hot roll machine for 25 seconds at 220° C. then scoured and dried. The untreated fabric showed 325 pills/35 in.² and a weight loss in the abrasion test of 6.5%, whereas, the treated fabric showed only 7 pills/35 in.² and a weight loss of 7.8%.

#### Example III

A 2 x 1 twill gabardine composed of 55% polyethylene terephthalate staple fiber and 45% rayon staple was treated in the same manner as the fabric of Example II. The untreated fabric developed 710 pills/35 in.² and the treated fabric only 20 pills/35 in.². When subjected to heat alone at 220° C. the fabric developed 180 pills/35 in.² section.

#### Example IV

A 3 x 2 twill sheen gabardine composed of 55% polyethylene terephthalate staple fiber and 45% wool was treated as in Example II. The untreated fabric showed 684 pills/35 in.² and the treated fabric only 35 pills/35 in.².

#### Example V

Two samples of batiste shirting fabric were woven from 65% polyethylene terephthalate staple yarn and 35% cotton. The fabric was padded with two aqueous solutions. The first sample was padded as in Example II with an aqueous solution of 40% by weight polyethylene glycol 600 and 1% by weight zinc nitrate catalyst. The second sample was padded the same way with the same glycol solution but in the absence of the catalyst. The two fabrics were heated on the National hot roll machine for 25 seconds at 220° C, then scoured and dried. The untreated fabric showed 175 pills/35 in.², and a weight loss in the abrasion test of 3.9%, the fabric sample padded with glycol alone showed 20 pills/35 in.², and the sample padded with glycol and zinc nitrate catalyst showed 0 pill/35 in.² and a weight loss of 6.8%.

#### Example VI

A 2 x 2 twill gabardine fabric composed of 100% polyethylene terephthalate staple fiber was padded with an aqueous solution containing 20% by weight of alkyl phenyl ether of polyethylene glycol 350 ("Tergitol" NPX commercially available from Union Carbide and Carbon Corporation, New York, New York). The treated fabric was then heated at 220° C. using a preheated hand iron. It was then scoured and dried. The untreated fabric showed 450 pills/35 in.² and the treated fabric 15 pills/35 in.².

#### Example VII

An oxford shirting fabric composed of 65% polyethylene terephthalate staple fiber and 35% cotton was padded with an aqueous solution containing 5% by weight of polyethylene glycol 600. The treated fabric was then heated on a National hot roll machine for 25 seconds, the temperature of the roll surface being 220° C. The fabric was then scoured and dried. The untreated fabric showed 630 pills/35 in.² and the fabric treated with glycol 150 pills/35 in.² in the standard pilling test. A sample of the same original shirting fabric subjected to the same heat treatment on hot rolls at 220° C. but without glycol pretreatment showed 380 pills/35 in.².

#### Example VIII

The 2 x 2 twill gabardine fabric, described in Example VI, was padded with an aqueous solution containing 40% 70 by weight polyethylene glycol 600 and 1% by weight zinc nitrate. Samples of the fabric were heat treated in a hot-air oven for various times. Table II below illustrates the pilling data and abrasion resistance of these fabrics, acceptable pill protection being afforded only at the expense of degraded fabrics.

Time of Heating In Hot Air Oven (220° C.)	Pills/35 in.²	A.A.T.C.C. Abrasion Resistance (Percent Weight Loss)
0 min	450 200 50 0	5. 8 10. 2 35. 0 100

Example IX

Three samples of an oxford fabric having a pill count of 100/35 in.² and having the same composition as the fabric of Example VII were padded with an aqueous solution containing 20% by weight of polyethylene glycol 600. The surface of each treated fabric was then ironed using a hand iron preheated to a different temperature. The sample, the surface of which was heated to 200° C., showed 25 pills/35 in.², the sample heated to 210° C showed 15 pills/35 in.², and the sample heated to 220° C showed 0 pill/35 in.².

The chief advantage of this invention is based on the fact that the polyester fabrics which normally have a high tendency to form pills can be given a simple and commercially cheap two-step finishing treatment which greatly lowers or eliminates the pilling and fuzzing tendency without significant impairment of other fabric properties such as strength, abrasion resistance, liveliness, crease recovery, wrinkle resistance, and the like.

The finishing process also improves the static resistance of the fabric, as well as improving the resistance of the fabric to soiling by liquid oils. It also permits the fabric to be dyed much faster than a control fabric, and the dyeings on these specially finished polyester fabrics are deeper and they exhaust better.

The finishing treatment is most advantageously applied to wearing apparel fabrics having almost any type of construction, because it is in these fabrics that the necessity for retaining the aesthetic properties and the undesirability of pill formation is most commonly apparent. However, the treatment may also be applied to industrial and other types of fabrics where there is a need for pill reduction, such as papermakers felts, filter fabrics, roll covers and the like.

It will be apparent that many widely different embodiments of this invention may be made without departing from the spirit and scope thereof, and therefore it is not intended to be limited except as indicated in the appended claims.

### I claim:

1. The process of finishing fabrics containing substantial amounts of linear condensation polyester fibers and which have a tendency to form surface pills in use which comprises reducing their pilling tendency without impairment of the properties of the fabrics by impregnating the said fabrics with a liquid medium comprising at least 5% by weight of a material selected from the group con-

sisting of polyether glycols, their esters, their ethers, and mixtures thereof, the alkylene oxide component of which has a molecular weight of at least 300; and thereafter heating at least one surface of the impregnated fabric to a temperature of from about 18° C. to the fusion temperature of the surface fibers while maintaining the temperature of the body of the fabric sufficiently below the temperature of the fabric surface so as to avoid degradation of the body portion of the fabric.

2. The process of claim 1 in which the weight loss differential between treated and untreated fabric is not

more than about 5%.

 The process of claim 1 in which the surface fibers contain linear condensation polyester fibers and the polytether glycol has the formula

$$A(O-R_1-O-R_2-O-R_3)_{Y}-B$$

in which R₁, R₂, and R₃ are the same or different alkylene units having up to 5 carbon atoms; A is selected from 20 the group consisting of hydrogen, alkyl, aryl and acyl groups; and B is selected from the group consisting of OH, alkyl ether, aryl ether, alkyl ester and aryl ester radicals, and Y has a value to produce a molecular weight of the alkylene oxide component of at least 300.

4. The process of claim 1 in which the surface fibers

are 100% polyethylene terephthalate.

5. The process of claim 1 in which the polyether glycol component has a molecular weight of from 400 to 600.

The process of claim 1 in which the polyether glycol
 is polyethylene glycol monolaurate, the alkylene oxide component of which has an average molecular weight of about 400.

7. The process of claim 1 in which the surface of the fabric is heated momentarily to a temperature of from

35 218° C. to 230° C.

8. The process of claim 6 in which the temperature is from 218° C. to 222° C.

9. The process of claim 1 in which the concentration of the polyether glycol component in the liquid is from 10 20-40%.

10. The process of claim 8 in which the polyether glycol is dissolved in water.

11. The process of claim 1 in which the polyether glycol is a polyethylene glycol having an average molecular weight of about 600.

12. The process of claim 1 in which the fabric is composed of a blend of linear condensation polyester fibers

and natural fibers.

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# UNITED STATES PATENT OFFICE CERTIFICATE OF CORRECTION

Patent No. 2,920,980

January 12, 1960

David D. Mooberry

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 53, for "through" read -- throughout --; column 8, line 5, for "180 C." read -- 2180 C. --.

Signed and sealed this 28th day of June 1960.

(SEAL)
Attest:
KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON Commissioner of Patents