

1

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WATER-SOLUBLE YARN

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This invention relates to a process for producing water-soluble polyvinyl alcohol yarn of improved properties.

The demand for a water-soluble yarn has been increasing steadily for several years. Uses for such yarn include various plied structures wherein the water-soluble yarn is used as a strengthener for very fine yarns during weaving or other fabrication steps. They may be plied with cotton, nylon, or other synthetic polymer yarn and subsequently dissolved from the fabric.

A preferred polyvinyl alcohol material for this use is grade 52-22 "Elvanol" manufactured by E. I. du Pont de Nemours and Company, inasmuch as this polymer type exhibits complete and rapid solubility in hot and cold water. It is about 88% hydrolyzed polyvinyl acetate. Polyvinyl alcohols which have been hydrolyzed to substantially higher or lower degrees are not as suitable for the manufacture of water-soluble yarns since they are not as readily water-soluble.

The very fact that the most satisfactory material is rapidly and completely soluble makes it difficult to prepare products with filament properties necessary to withstand the required textile operations and storage under normal humidity conditions while yet retaining the needed ease of solubility.

It is of course known that water-soluble polyvinyl alcohol yarns coagulated in highly concentrated salt solution can be rendered more difficultly soluble in water by after-treatment as, for instance, with boric acid which may affect a partial cross-linking and blocking or substitution of the water-sensitive hydroxyl groups. However, this is undesirable for the purpose of this invention for, while the salts of coagulation must be removed without re-dissolving the yarn, the yarn must remain in a readily soluble form while maintaining desirably high tenacity, elongation and modulus of elasticity within the filaments.

It is, therefore, an object of this invention to provide a process for producing stabilized water-soluble yarn of improved properties for the above-mentioned textile purposes. Other objects will become apparent from the description and the appended claims.

This invention comprises spinning a solution of 82% to 94% saponified polyvinyl ester into a concentrated sulfate salt coagulating bath, removing such sulfate salt from the resulting yarn with an aqueous solution of a volatile salt containing a small concentration of boric acid, and drying the yarn at elevated temperature to remove the volatile salt as well as the water.

Preferably the saponified polyvinyl ester is grade 52-22 "Elvanol," an 88% saponified polyvinyl acetate, but any other saponified polyvinyl esters may be used, e. g., polyvinyl formate, polyvinyl propionate and polyvinyl butyrate, and the degree of saponification may be varied from about 82% to 94% depending on the polyvinyl ester and the viscosity of the polymer.

The sulfate salt coagulating bath may be the usual type, i. e., sodium sulfate, ammonium sulfate or mixtures thereof. Preferably the bath consists of about 45% am-

2

monium sulfate, although the concentration may vary from 40% to saturation.

The aqueous solution of a volatile salt may be a concentrated solution, e. g., 40% to 60%, of ammonium acetate, ammonium formate, ammonium oxalate, ammonium carbonate, ammonium bicarbonate, or other salt which is volatile below temperatures which would injure the yarn. In this aqueous, volatile salt bath about 1% of boric acid is dissolved. This boric acid concentration may be varied from 0.5% to as much as 2% without adverse effect but 1% appears to be about optimum.

Best results are obtained under certain conditions of spinning, lagging before drawing, drawing 4 to 8 times, washing and drying, as will be shown by the examples that follow.

EXAMPLE 1

A yarn of sixty filaments was spun from a 20% aqueous solution of about 88% saponified polyvinyl acetate (type 52-22 "Elvanol") by extruding the solution into a 45% ammonium sulfate bath at 70° C., leading the yarn through the bath a distance of about 13 inches to a draw-off wheel at 25 yards per minute and, after several passes around the wheel, to a bobbin rotating at the same peripheral speed as the draw-off wheel, on which bobbin the yarn is wound up. This yarn of about 1300 denier, calculated on the basis of the polymer present, was held several hours and then drawn through a 30% ammonium sulfate bath at 60° C. to a second bobbin at a draw ratio of 4.6X. From this bobbin, yarn was withdrawn and passed through 60% ammonium acetate bath at 25° C., to replace substantially all the ammonium sulfate with ammonium acetate, and the yarn was again wound on a bobbin. After centrifuging, the bobbin of yarn was subjected to overnight drying at 35° C. and, thereafter, to overnight drying at 65° C. This is sample A in the table below. Another sample of yarn was similarly prepared except that the ammonium acetate washing bath also contained 1% boric acid. This is sample B in the table below.

Table 1

Sample	Denier	Modulus	Yield Point		Tenacity, gm./denier	Elong., percent
			Tensile	Elong.		
A-----	286	37	0.3	1.5	1.9	27
B-----	301	108	1.1	1.7	2.1	24

It will be noted that the sample treated with boric acid showed a marked improvement in modulus and yield point and noticeably higher tenacity. When these two samples were subjected to solubility tests under low tension (i. e., 0.02 to 0.1 gram per denier) sample A was found to break in about thirty seconds in water at 1° C., while sample B required a water temperature of about 37° C. to bring about tensile failure in the same time. This decrease in solubility points to some chemical reaction having taken place between the polyvinyl alcohol and boric acid, but the surprising part is that this modified product is still relatively soluble and entirely satisfactory for commercial use, while acquiring much needed stability for textile processing and storing. The following data are given to bring out further advantages gained from the use of this invention.

Comparative data on tensile recovery, wherein samples were subjected to stress to elongate them 1% and 3%, as indicated, and then allowed to recover, show desirable tensile recovery properties resulting from this boric acid treatment. Sample B was compared with a sample designated C in the tabulation below and prepared as described

above for sample A and with properties very much like sample A.

G. The following results were obtained from tests on these samples:

Table IV

Sample	Denier	Modulus	Yield Point		Tenacity, gm./den.	Elong., percent	Tensile Recovery	
			Tensile	Elong.			1%	3%
F.....	168	99	0.7	0.7	3.3	10	56	41
G.....	170	180	2.2	1.7	3.4	6	105	81

Table II

Sample	Denier	Modulus	Tenacity, gm./denier	Elong., percent	Tensile Recovery	
					1%	3%
C.....	280	24	1.9	30	51	40
B.....	301	108	2.1	24	93	56

Shrinkage of the yarn is largely affected by relative humidity, being small below 50% relative humidity and much greater at 60% and higher humidities. It is much reduced by the boric acid treatment of this invention as shown below. Here sample D was prepared similarly to A and the preparation of E differed from the preparation of D only by the addition of about 1% boric acid to the ammonium acetate washing bath used in the preparation of sample D.

Table III

Sample	Shrinkage at 60% Relative Humidity, percent	
	1 day	3 days
D.....	17.5	18.0
E.....	9.4	9.5

This reduction of shrinkage gained by the use of boric acid is desirable in order that the polyvinyl alcohol component of a plied yarn shall not shrink and cause unsightly puckering, or the like, during use before it is washed out. Moreover, it minimizes tightening of the yarn on the package during storage.

EXAMPLE 2

The spinning, lagging, drawing and washing steps were carried out in much the same manner as described under Example 1. In this case, however, the coagulating bath was composed of 47% ammonium sulfate maintained at a temperature of 70° C. The draw bath was 50% ammonium sulfate at 90° C., and the draw ratio was 6.3X. Again, the ammonium sulfate was displaced by passing it through a 60% ammonium acetate bath at 25° C. for the control sample designated F, while 1% boric acid was added to the ammonium acetate displacement bath for the production of the yarn of this invention designated sample

Starting with less readily soluble polyvinyl alcohol, wet spun yarns could not be directly produced or modified to give as desirable physical properties with satisfactory solubility whereas, by the use of this invention, polyvinyl alcohol yarns of excellent textile properties may be produced. These yarns can easily be plied with yarns of cotton, rayon, nylon, polyesters, polyacrylics and of other synthetic filament-forming polymers to produce improved and useful composite yarns, the polyvinyl alcohol yarn component being easily removed by washing in luke-warm water, i. e., from 100° to 110° F.

Since many different embodiments of the invention may be made without departing from the spirit and scope thereof, it is to be understood that the invention is not limited by the specific illustrations except to the extent defined in the following claims.

What is claimed is:

1. A process for stabilizing freshly spun yarn formed by spinning a solution of 82% to 94% saponified polyvinyl ester through a spinneret into a concentrated sulfate salt coagulating bath which comprises removing the sulfate salt from the yarn with a concentrated aqueous solution of a volatile salt containing from 0.5% to 2.0% of boric acid and drying the yarn at elevated temperature to remove the water and volatile salt.

2. A process as defined in claim 1 in which the saponified polyvinyl ester is saponified polyvinyl acetate.

3. A process as defined in claim 1 in which the saponified polyvinyl ester is 88% saponified polyvinyl acetate.

4. A process as defined in claim 1 in which the volatile salt is an ammonium salt and the concentration of the volatile salt solution is from 40% to 60%.

5. A process as defined in claim 1 in which the concentration of boric acid in the volatile salt solution is about 1%.

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