

Jan. 11, 1966

L. C. GALATIOTO

3,228,745

PROCESS OF SHRINKING NYLON FABRICS WITH MIXTURES OF SPECIFIC
CHEMICAL SHRINKING AGENTS

Filed Jan. 10, 1961

8 Sheets-Sheet 1

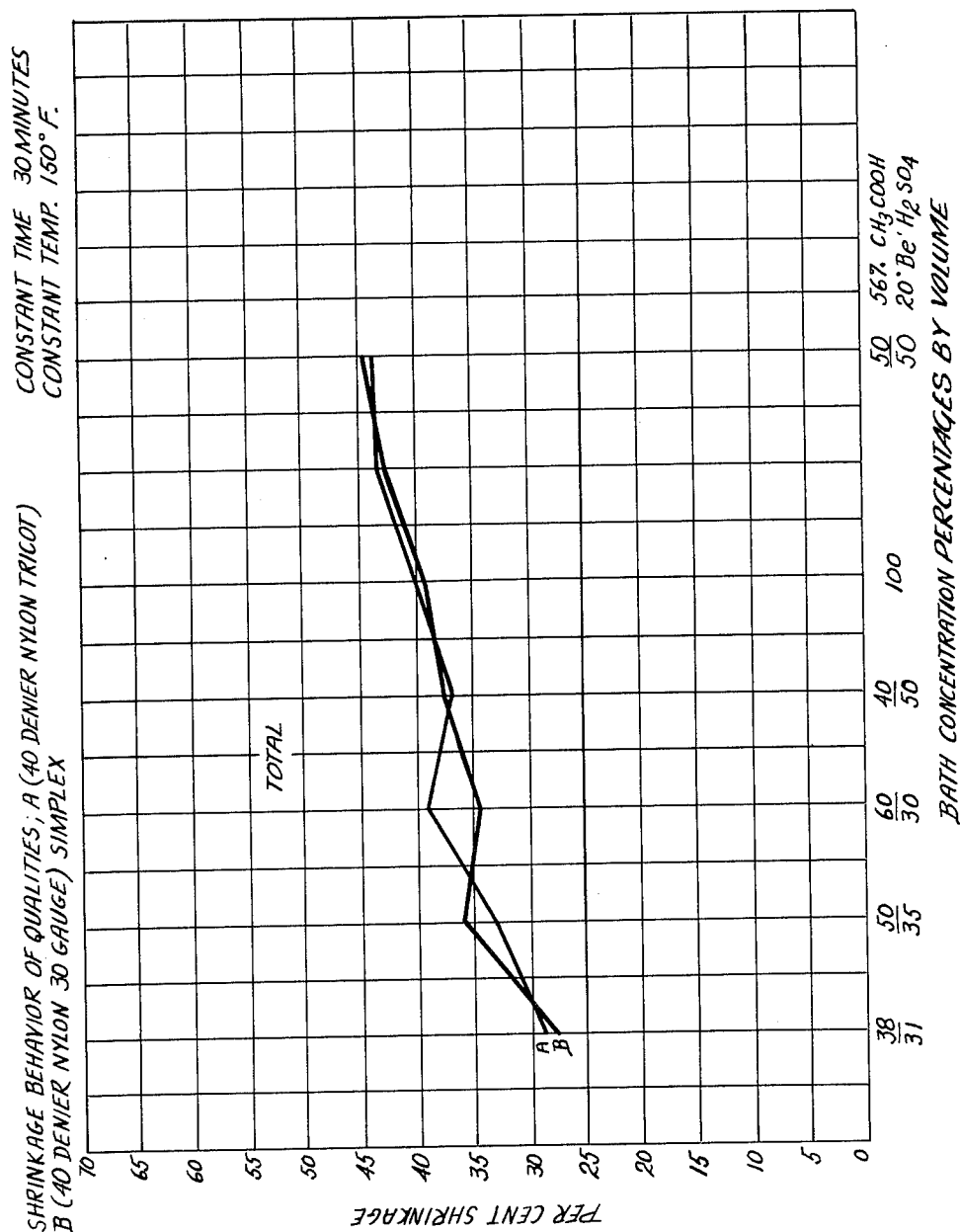


Fig. 1

INVENTOR.

Louis C. Galatioto

BY

Harold R. Savage.

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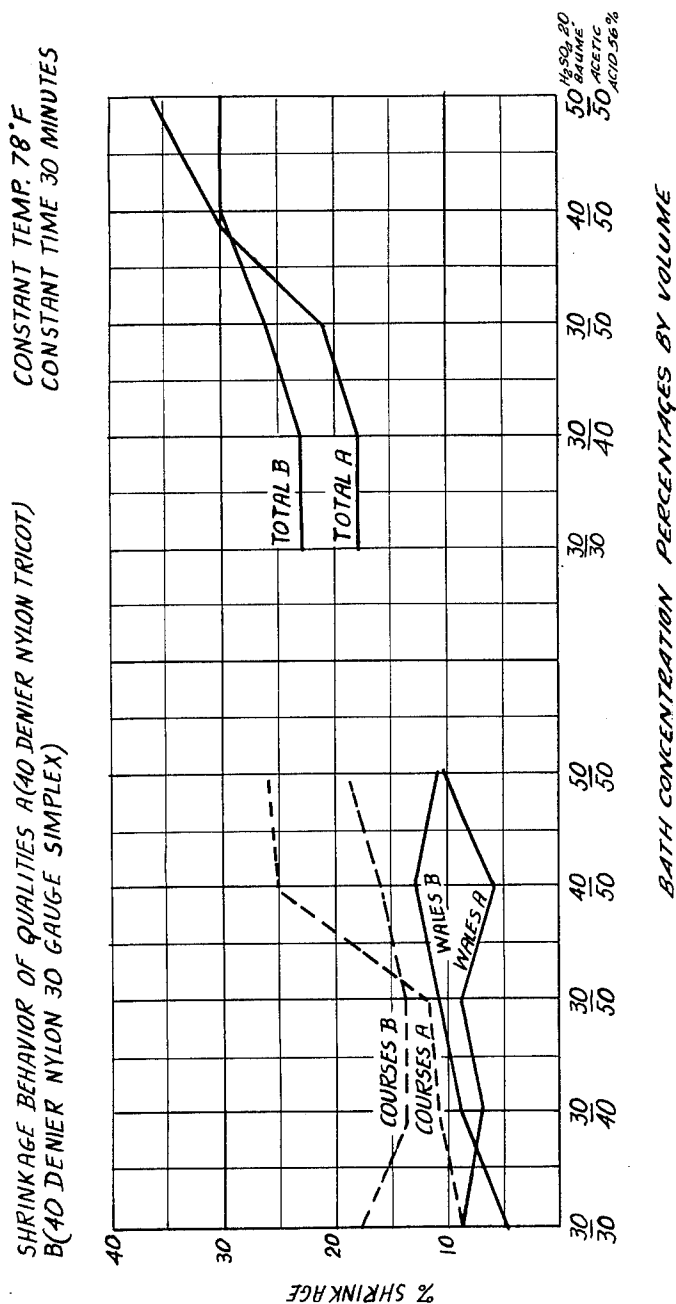
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8 Sheets-Sheet 2



INVENTOR.

Louis C. Galatioto

BY

Harold R. Savage

ATTY

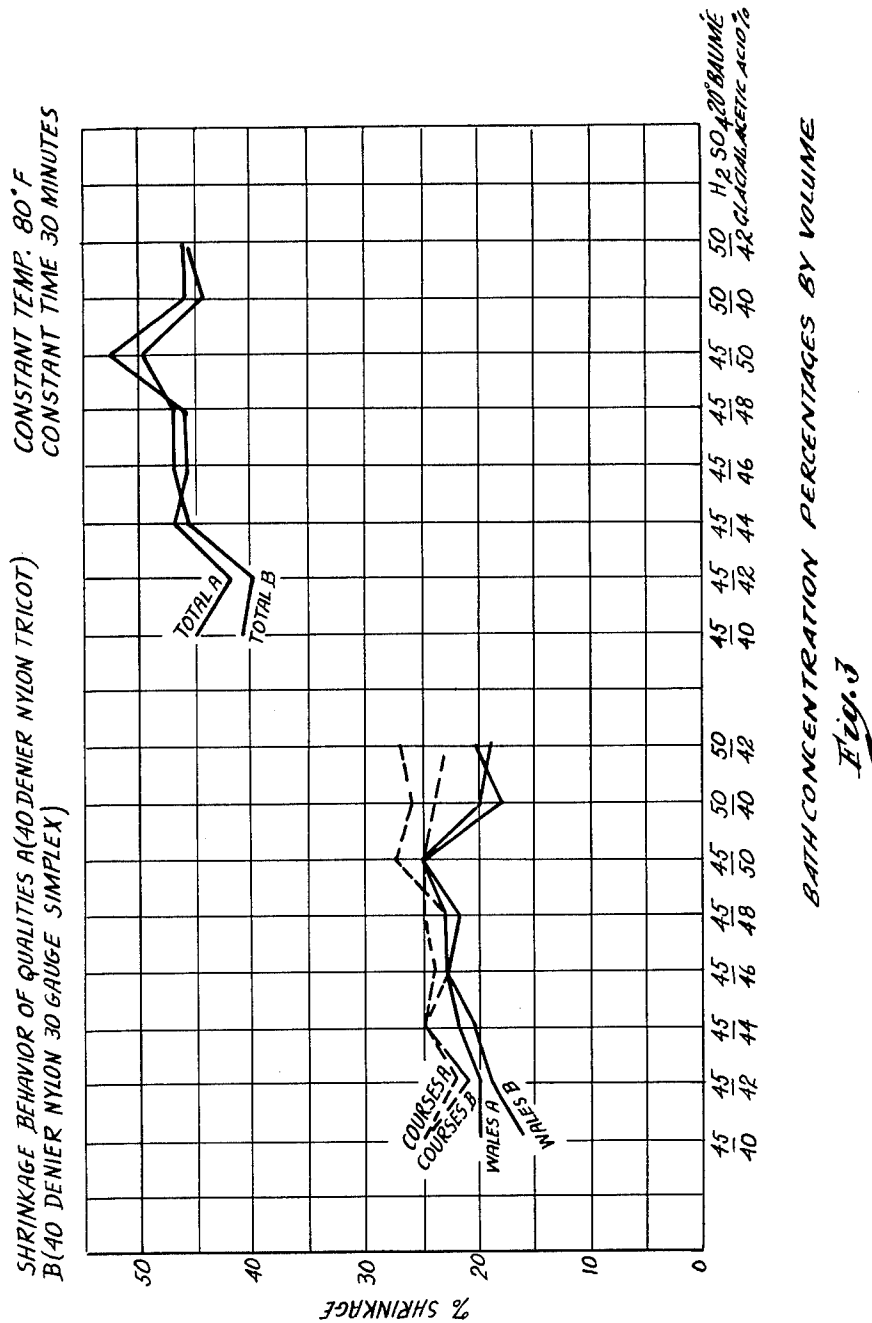
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8 Sheets-Sheet 3



INVENTOR.

Louis C. Galatioto

BY

Francis R. Savage

ATTY

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L. C. GALATIOTO

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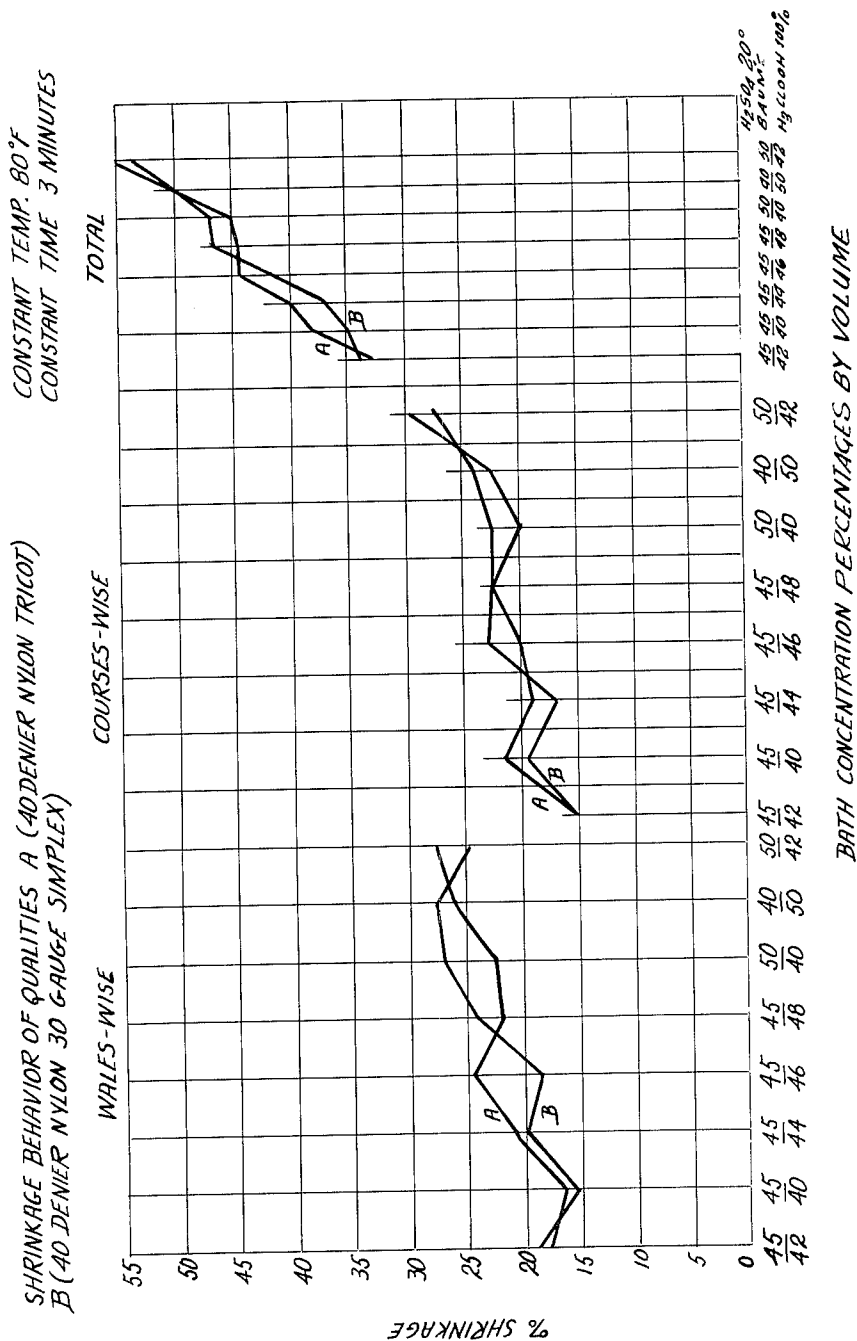


Fig. 4

INVENTOR.

Louis C. Galatioto

BY

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Hazel R. Savage

ATTY

Jan. 11, 1966

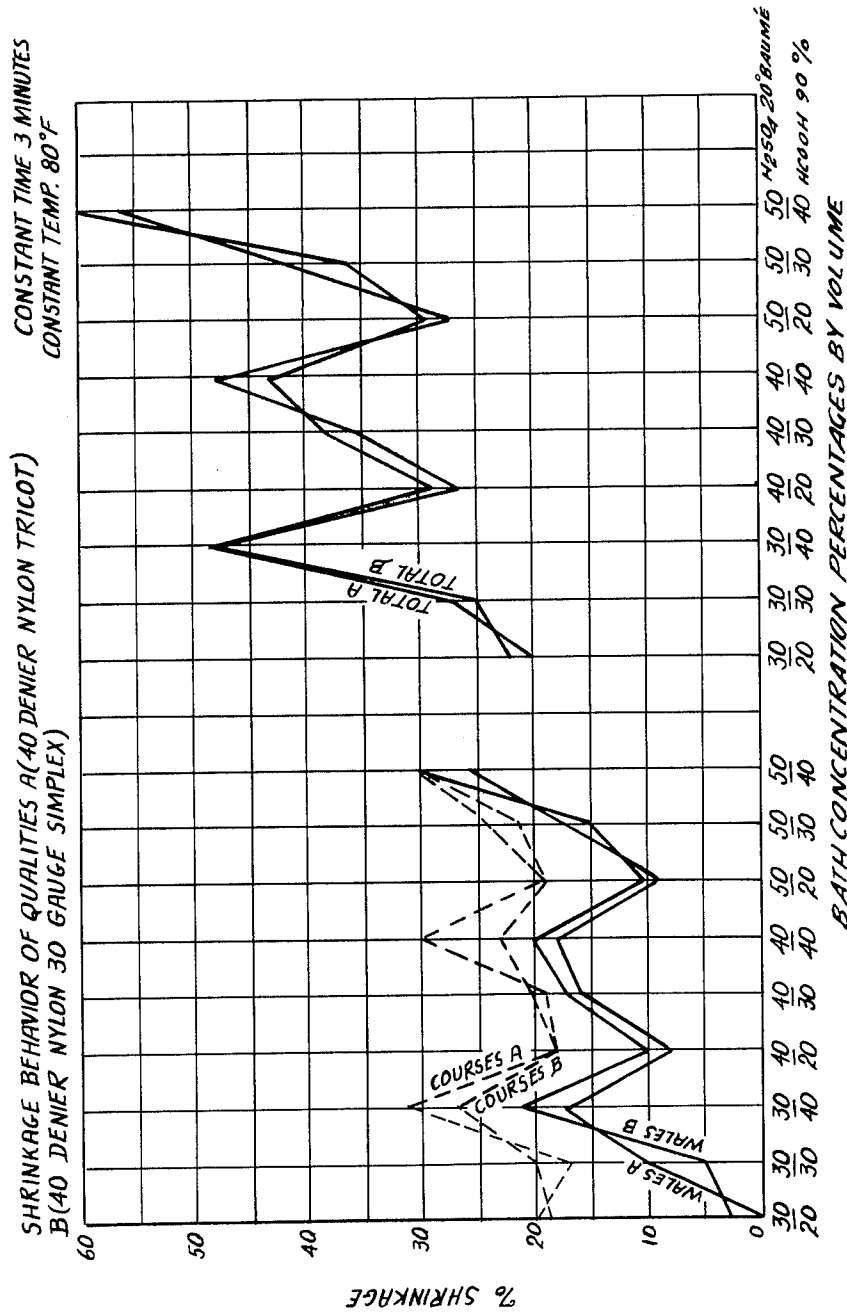
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8 Sheets-Sheet 5



INVENTOR.

Louis C. Galatioto

BY

Harold A. Savage.

ATTY

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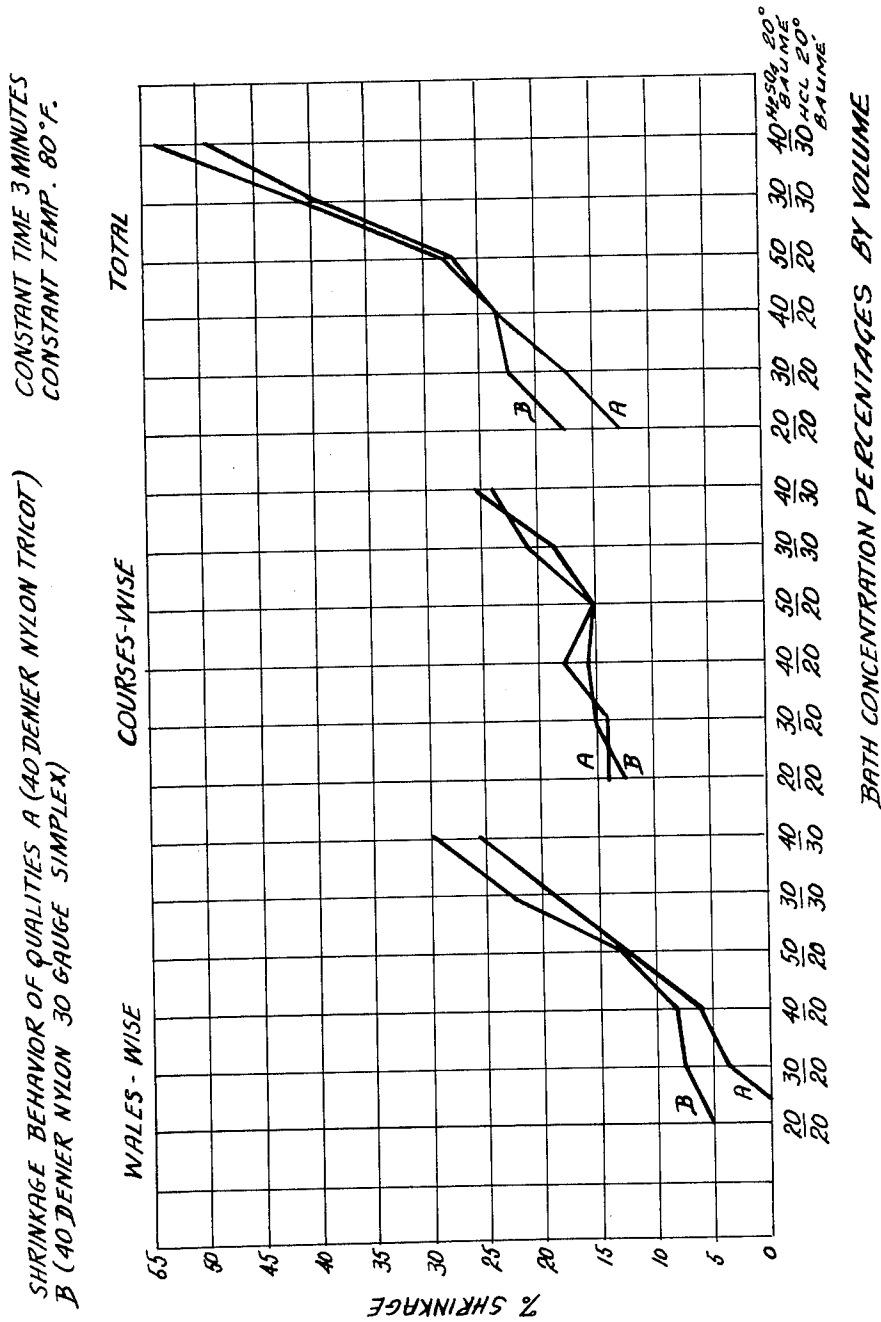


Fig. 6

INVENTOR.
Louis C. Galatioto

INVENTOR.
Louis C. Galatioto

INVENTOR.
Louis C. Galatioto

INVENTOR.
Louis C. Galatioto

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L. C. GALATIOTO

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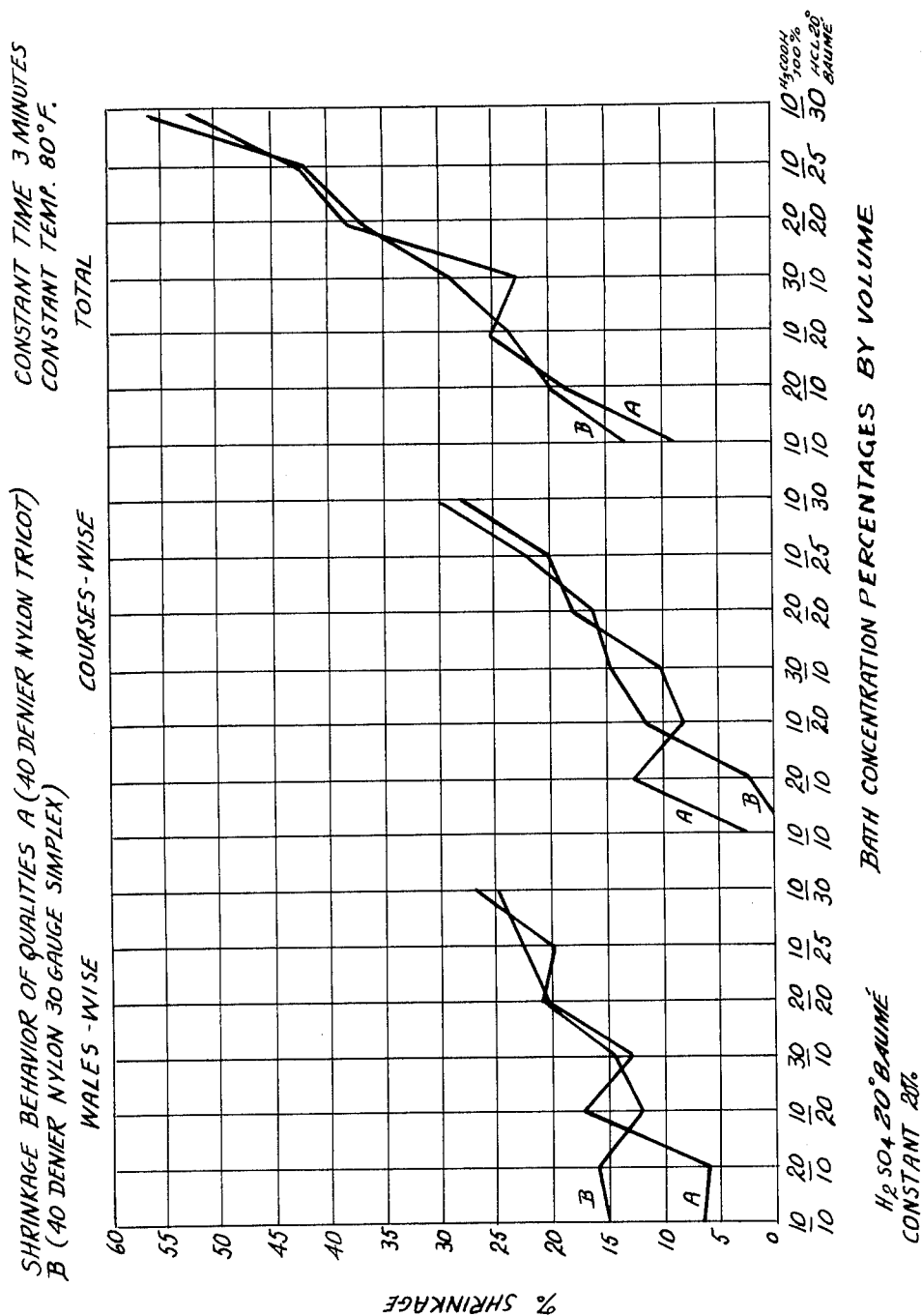


Fig. 7

INVENTOR.

Louis C. Galatioto

BY

BY *Harold R. Savage.*

ATTY

Jan. 11, 1966

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SHRINKAGE BEHAVIOR OF QUALITIES A (40 DENIER NYLON TRICOT) CONSTANT-TIME 3 MIN. TEMP. 80°F
B (40 DENIER NYLON 30 GAUGE) SIMPLEX. CONSTANT H₂SO₄ 30% CONSTANT H₃CCOOH-20%
20° BAUME 100%

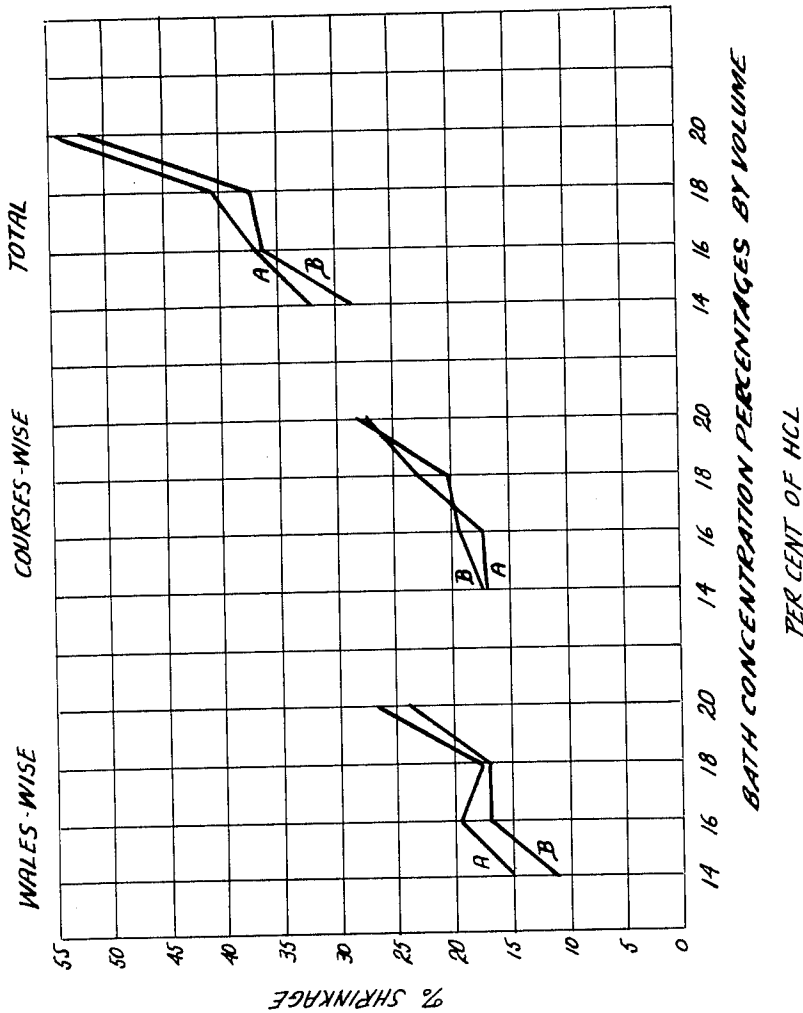


Fig. 8

INVENTOR.
Louis C. Galatioto
BY
Harold R. Savage
ATTY

1

3,228,745

PROCESS OF SHRINKING NYLON FABRICS WITH MIXTURES OF SPECIFIC CHEMICAL SHRINKING AGENTS

Louis C. Galatioto, Roseto, Pa., assignor, by mesne assignments, to Lehigh Valley Industries Inc., New York, N.Y., a corporation of Delaware

Filed Jan. 10, 1961, Ser. No. 81,874

1 Claim. (Cl. 8—130.1)

The present invention relates to a method of treating certain synthetic textile materials, such as filaments, fibers, yarns and threads, more especially in knitted form or woven condition, characteristically to shrink the same, and to the resulting products.

It is a fundamental, characteristic property of all synthetic filaments, fibers, yarns or threads, and hence of fabricated textile or other products made from them—that their surfaces are relatively physically and chemically resistant, continuous, smooth and hard—and hence non-felting inter se, and of low frictional propensities, if any, with respect to themselves and to other surfaces and materials, tending to give them a slick feel and a loose, free hand.

These properties are well known and recognized as being very valuable features of such synthetic textile materials, for many purposes. However, it is sometimes desirable that they should have supplementary properties and characteristics which would render them applicable or adaptable to further uses, functions and conditions, which, in their usual state of manufacture and use, they are quite incapable of serving.

Thus, if an open or looser weave or structure is desired, this can be provided in the original spinning, twisting, weaving or knitting operations or even in the finished goods, by tensioning.

But if a very firm, compact weave or structure is to be desired, this may not be so readily provided in the originally spun yarns or in the twisted threads, nor in the woven or knitted goods made from them. And, insofar as it can be and is effected, the original spun structure of the yarn and twisted structure of the threads and woven or knitted weave or structure of the goods may, each and all, be subject to appreciable degrees of reduction (and consequent warping) in the course of the usual winding, handling and storing of the goods themselves and before or during or after use, for the ultimate purposes which they are intended to serve.

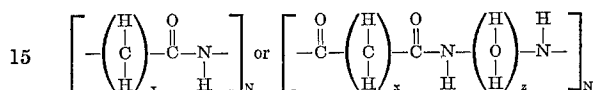
Accordingly, it is an object of the present invention to provide a method of treating synthetic filaments, fibers, yarns, threads, and woven, knitted or otherwise fabricated textile materials or goods made from them, which are characteristically relatively physically and chemically resistant, partially water-repellent, and of continuous, hard, smooth surfaces, whereby these inherent tendencies to lose their firm, compact structure of spinning, twisting, weaving, knitting or other type of fabrication tending to integrate them, may be checked; or, more particularly, whereby the firm, compact structure of the spun yarns, twisted threads, woven or knitted or otherwise fabricated textile materials or goods, may be positively enhanced and preserved throughout the normal courses of their manufacture, handling, storage and the intermediate or ultimate uses to which they are to be put.

Briefly, it is an object of the invention to effect the positive, uniform, controlled shrinkage of such materials in length and/or cross-section of filaments, fibers, yarns, or threads, in length and/or width of woven goods and wale-wise and/or course-wise of knitted goods, to the degree desired.

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Other objects will appear from the following disclosures and descriptions of the invention and from the claim.

By the present invention, it has been discovered that the more stable, chemically and physically resistant, synthetic filaments, fibers, yarns and threads, and knitted, woven or otherwise fabricated textile goods made from them, such as nylon which is a generic term for any long chain synthetic polymeric amide, having recurring amide groups as an integral part of the polymer amide chain, and which is capable of being formed into a filament in which the structural elements are oriented in the direction of the axis:



cellulose tri-acetate; and the like, may be given a felting characteristic or function, and consequently a shrinking action, whereby the filament, yarn, fiber or thread, or fabricated textiles made from or containing it, may be felted and/or shrunk, to substantially any desired degree or effect, uniformly throughout their dimensions or surfaces, or selectively in this respect, and without deleterious action thereon but improvements in other respects, such as feel, hand, strength, susceptibility to dyes and dyeing, and the like.

It is well known that such synthetic, chemically resistant filaments, fibers, yarns, threads, and the various products made from them, are sensitive to some solvents, which may be active enough, under sufficient conditions of concentration, temperature and time, to dissolve them. In the course of such action, the solvent penetrates first the superficial surfaces of the product and then progressively into an ultimately throughout its structure, reducing it to a dispersed condition or to a liquid solution.

It is also well known that such synthetic, chemically and physically resistant filaments, fibers, threads, and the various textile products made from them, are sensitive to other reagent liquids, which penetrate the superficial surfaces, under sufficient conditions of concentration, temperature and time. They are not dissolved by such reagents but tend to gelatinize. In the course of such action, the superficial surfaces treated swell and subsequently the body structure of the filament, fiber, yarn or thread, or of the goods treated, as a whole. But the integrity and general shape and solid characteristics, generally are maintained.

It is found by the present invention that by treating chemically and physically resistant synthetic filaments, fibers, yarns, threads, and woven, knitted or otherwise formed textile goods, with an aqueous solution containing a solvent of the same in a concentration sufficient partially to dissolve the surface and/or with an aqueous solution containing a gelatinizing agent, in sufficient concentration partially to effect gelatinization of the surface, at a temperature not above the boiling point of the solution and for a time to control the action of the same, the thus treated surfaces acquire the characteristic properties of felting and can be given substantially any degree of shrinkage that may be desired and that it can be controlled and regulated, not only by the concentrations and conditions of the treating solution but definitively by the removal of the treating solution from the treated surface and subsequent neutralization and stabilization.

It is believed that the shrinking action thus produced is a composite of the dispersing or dissolving action of the solvent upon the treated surface and of the opposite of restraining action or contracting tendency of the gelatinizing agent upon the thus partially dissolved, relaxed or dispersed

superficial surface or surfaces. These opposed forces result in a fine wrinkling or folding of the superficial surface areas which, upon removal of the solution, are capable of manifesting a typical felting action between such surfaces, per se, and also with other similar surfaces, with a consequent shrinking action upon the filaments, fibers, yarns, threads, individually, and also between them and others in the woven, knitted or otherwise fabricated textile goods which they may constitute or of which they may form a part.

In accordance with the present invention, it may be said that the action of the solvent reagents upon the surfaces of the goods, when applied, per se, is more rapid and more drastic than the action of the gelatinizing agents, per se. Moreover, at high concentrations, at high temperatures and for prolonged periods of treatment, the solvents will not only wet, attack and soften, but ultimately disperse or dissolve the surface or even the entire body structure of the filament. The gelatinizing agents will also attack and soften the surfaces and structure of the filament. On the other hand the gelatinizing agents will not disperse nor dissolve the surface but will cause it to swell and gelatinize and at the same time leave it in its original shape and general conformation—though swollen in its dimensions and hence susceptible to distortion and to folding or wrinkling, superficially.

Both reagents, when applied in the higher ranges of concentration or at higher temperatures or for prolonged periods of time, will soften the surfaces of the chemically and physically resistant synthetic filaments and textile goods made from them, and, as a result will render them susceptible to appreciable shrinkage. It would therefore be possible, within the present invention, to shrink such filaments or the goods made from them, by treating with concentrated solvent reagents or concentrated gelatinizing agents, severally or successively, at low temperatures or even at high temperatures, if suitably confined so as to avoid fuming into the atmosphere and checked in respect of their actions upon the goods, as by prompt removal—such as vaporization, cooling, neutralization or dilution with an inert solvent, such as water or the like. But such actions and reactions are apt to be too rapid, the reagents subject to vaporization and escape or develop in too short a time for effective or convenient control. It is therefore generally desirable that the reagents be reduced in concentration, with an inert solvent, such as water.

Thus, a practical limitation applies to the concentration of the solvent reagent and of the gelatinizing reagent, or mixture of them. As a general rule, the active reagent or total of active agents used will never be above 90% by volume of the treating solution. Such concentrations, being very high, would make the resulting reagent solutions very active, very expensive and very difficult to handle in operation, as above pointed out. As a matter of practice, therefore, lower concentrations will ordinarily be preferred and used. Accordingly, an upper limit of 75%, more or less, is both safer and less expensive and in fact an upper limit, from the standpoint of efficiency, economy and convenience, will usually be adopted in commercial operations and practice. On the other hand the treating solution should contain at least about 25%, by volume, of the solvent reagent or gelatinizing reagent, or both, in order to effect a shrinkage of 16% or greater (in one direction or the other, or the sum of the two), and hence the inert solvent or diluent component of the solution, such as water, in association therewith, will not exceed about 75%, more or less.

Some latitude, either way, in respect of these limitations, in terms of concentration of the reagents is therefore permissible and will be necessary and/or desirable. Moreover, their selection will be governed or modified by the temperature and time of treatment, as above pointed out, with added precautions accordingly to suit the exigencies of any specific case or condition of operation. These will include, to some extent, the appurtenant type and char-

acter of the apparatus available, and of course the material to be treated and the effects and results desired to be accomplished therewith. But, in general, the usual equipment, means and apparatus for the handling of textiles, in whatever stage of fabrication the material to be treated may be, will ordinarily be sufficient and satisfactory for carrying out the present invention therein in order to effect the desired shrinking action and results, if made of a material sufficiently resistant to the acids, such as a good grade of stainless steel, wood, or plastics.

In general, of course, efficient means and apparatus for rapid and uniform handling, wetting out, treating, treatments, concentrations, temperature and time controls, and removal of reagent solutions, as by neutralization, washing or the like, squeeze rolls, etc., will all be beneficial, in their respective actions and effects on the functions, operations and results to be produced.

The solvent reagents, as above referred to, for the purposes of the present invention are: formic acid (90%) at 80° F. and above, phenolic compounds such as carbolic acid, cresols, xylenols and chlorinated phenols, calcium chloride in methanol (saturated solution at 80° F.), hot solutions of calcium chloride in glacial acetic acid, ethylene chlorhydrin and ethylene glycol, hot solutions of zinc chloride in methanol and benzyl alcohol at the boil. All dissolve nylon.

Mineral acids, such as sulphuric and hydrochloric, in low concentrations, cause loss in strength of nylon yarn and in higher concentrations completely dissolve nylon.

The gelatinizing reagents, as above referred to, for the purposes of this invention are: acetic acid, adipic acid, aniline, benzene sulphonc acid, benzoic acid, boric acid, chloral hydrate, chloro-acetic acid, formic acid diluted, glycol, lactic acid, lithium bromide, metacresol, parahydroxybenzoic acid, phenol (e.g. 2% in H₂O), phosphoric acid and zinc chloride. All cause nylon to swell or gelatinize.

It will be noticed that formic acid appears in both categories, that is to say, above 80° F. and in the more concentrated solutions containing formic acid it acts as a solvent, or dispersing reagent, while at lower temperatures and in solutions of lower concentrations its activities are those of gelatinizing or swelling reagents.

The mineral or inorganic acids, sulphuric and hydrochloric, while active solvent reagents upon nylon are so much more so, even in low concentrations and at low temperatures, that they may more especially be regarded as catalysts of the solvent action of the organic solvent reagents and also as imparting increased acidity or lower pH values to the solutions to which they are added, even when in small amounts or relative proportions.

For example, all of the reagent solutions which are found to be sufficiently active in the shrinking of the synthetic filaments, etc., and products made from them, are highly acidic and manifest a pH value of 1, or below 1, and usually and preferentially a pH value below 1 and even of 0 and below 0. The pH values of such solutions, accordingly, are negative, such as -1, -2, etc. This means, as a practical matter, that these solutions, upon dilution with water, have their pH values gradually raised thereby from their negative values to less negative values, then to 0 and then to a positive pH value above 0 and between 0 and 1, in concentrations of the order of those solutions used in this invention. Such pH values, therefore, of the orders of 1 to 0 and from 0 to -1, -2, etc., are to be considered as representing the range of acidity of the reagent solutions which are satisfactory for use in the present process of shrinking synthetic filaments and the textile and like products made from them.

Example 1

A preliminary example of the invention was carried out with a solution of 25% (of 90%) formic acid and 75% water and applied to swatches of 40 denier nylon tricot knitted fabric at 100° F., 120° F., and 150° F. for

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30 minutes, rinsed, neutralized with sodium bicarbonate and dried, with the following results:

	100°	120°	150°
Shrinkage wales-wise.....percent..	7	13	10
Shrinkage course-wise.....do.....	8	7.5	11

Example 2

Swatches of the same material were treated with a 100% solution of commercial acetic acid (56%) with the following results:

	Shrinkage, percent
Wale-wise	20
Course-wise	20

While the shrinkage in Example 1, with formic acid, was barely sufficient to be regarded as commercially satisfactory, especially at the lower temperatures, it was promising and that obtained in Example 2 was highly satisfactory and sufficient for many purposes.

Example 3

On account of the high cost and consequent expense of using formic acid in higher concentrations and the operation difficulties involved in using high temperatures, tending to render the atmosphere irritating to the operators if not properly confined, it was then tried to replace a part of the formic acid or acetic acid with sulphuric acid. To this end an experiment was made with a solution containing 30% of formic acid (90%) and 30% of sulphuric acid (20° Baumé). Four swatches of the same material were used. One was treated with plain water at 140° F. for 20 minutes; as a control. The second was treated for 20 minutes at 90° F. The third was treated for 20 minutes at 120° F. The fourth was treated for 20 minutes at 140° F. They were then rinsed, neutralized and dried, as before.

All swatches were observed for shrinkage and appearance. Except for the control, or first sample, they all showed a marked increase in swelling as compared to the results obtained with 25% formic acid (90%) alone, as in Example 1.

The shrinkages obtained were the following:

Control	Wales-wise 1%, course-wise 5%.
Second	Wales-wise 13%, course-wise 13%.
Third	Wales-wise 17.5%, course-wise 20%.
Fourth	Wales-wise 23%, course-wise 22%.

These results were satisfactory for many purposes, but still the expense of using formic acid was a factor to be resolved.

Example 4

For this reason, acetic acid was similarly treated with sulphuric acid, using a commercial solution containing 56% acetic acid, and preparing a treating solution of 50% of such acid and 50% of sulphuric acid (20° Baumé).

Swatches of the same material were treated with this solution for 30 minutes at 150° F. Then the former procedure of rinsing, neutralizing, drying and observing was carried out.

The results obtained showed the following shrinkage:

Wales-wise 22%, course-wise 22%

This proved that acetic acid could be substituted for formic acid and that the shrinking effects of both were enhanced by the presence of sulphuric acid. The shrunken fabric, especially where the shrinkage was 20% or more, had a beautiful hand and a superior appearance.

Example 5

The above examples were carried out in small containers and upon small swatches of the goods. It was therefore proposed to treat 5 yards of material (simplex

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knitted 40 denier nylon) in a laboratory tub of larger volume capacity.

For this purpose, 30 liters of the solution used in Example 4 were prepared. The sample of cloth was loaded in the tub and it was treated for 30 minutes at 160° F. The fabric was then rinsed and neutralized with sodium bi-carbonate solution. It was dried in a commercial type of frame and sueded. Then it was dyed a shade of dark ivory and finished, suitable for use in gloves.

A shrinkage of 35%, both wales-wise and course-wise, was obtained. A beautiful, heavy cloth resulted.

Various experiments were continued with various qualities of cloth, including mixtures of nylon with Arnel, Arnel and viscose, cotton and nylon, Arnel and silk, Arnel and Bemberg, etc. While performing these experiments, which in the majority of cases gave satisfactory results, it was noted that the commercial acetic acid (56%) was quite often contaminated by metals, producing a reddish coloration in the treating solution which consequently left the treated cloth not in a condition to be dyed white or pastel shades without a further bleaching. It was then decided to substitute it with pure, glacial acetic acid. Of course all previous formulas had to be adjusted with the new concentration of acid accordingly.

Hundreds of experiments were performed with various concentrations of the acids and at various temperatures and it was found from them that the best formula for the majority of fabrics containing only nylon fibers, was 50% sulphuric acid (20° Baumé), 31% glacial acetic acid and 19% water. Time of treatment was ½ hour and temperature of solution was 160° F. However, experiments performed in the plant, with open containers proved that the acetic acid at 160° F. produced so strong fumes, that it was almost impossible for the operators to work under these conditions. Therefore a new series of experiments was made to develop a formula for a treating solution which would give the same results at room temperatures.

The concentrations of the acid treating solutions used were increased and many variations of them tried, in order to determine the best possible combinations.

The results obtained by these experiments are represented in the graphs of the accompanying drawings, in which:

FIG. 1 is a chart, plotting the shrinkages obtained by the procedure of Example 4, above, upon (A) knitted 40 denier nylon tricot and (B) 40 denier nylon 30 gauge simplex, in totals of percentages (that is shrinkage wales-wise plus shrinkage course-wise) with a constant time period of treatment, of 30 minutes, and at a constant temperature of 150° F., against varying percentages of sulphuric acid (20° Baumé) and acetic acid (56%) in the treating solutions used.

FIG. 2 is a chart, plotting the shrinkages obtained from (A) knitted 40 denier nylon tricot and (B) 40 denier nylon 30 gauge simplex in percentages (wales-wise, course-wise and wales-wise plus course-wise) with a constant time period of treatment of 30 minutes and a constant temperature of 78° F., against varying percentages of sulphuric acid (20° Baumé) and acetic acid (56%) in the treating solutions used.

FIG. 3 is a chart plotting the shrinkages obtained (upon the same materials as in FIG. 2) with a constant time period of treatment of 30 minutes and at a constant temperature of 80° F. (wales-wise, course-wise and wales-wise plus course-wise) against various concentrations of sulphuric acid (20° Baumé) and of acetic acid, as glacial acetic acid (100%) in the treating solutions used.

FIG. 4 is a chart plotting the shrinkages obtained using the same materials with a constant time period of treatment of 3 minutes and a constant temperature of 80° F. (wales-wise, course-wise and wales-wise plus course-wise) with mixtures of various concentrations of sulphuric acid (20° Baumé) and of acetic acid, as glacial acetic acid (100%) in the treating solutions used.

From these graphs of the results obtained, it was concluded that the best combinations for the shrinking of 40 denier nylon treated in these examples may be taken as 48% sulphuric acid (20° Baumé) and 42% glacial acetic acid (100%) and 10% of water.

Further experimentation was made to reduce the time of treatment, so as to make the process as practical as possible for commercial production and operation. It was found, as can be derived from the accompanying charts, that a treating solution of the above formula can also be used in a continuous machine, where the cloth is immersed in the liquor for 3 minutes, or for even less, namely 1½ minutes, or less, as well as in the vat or tub for 30 minutes, at lower acid concentrations.

Thus, through experimentation, a swelling and shrinking process for nylon and other fibers has been attained, which is commercially possible and commercially satisfactory, as demonstrated from the various experiments and pilot plant operations which have been performed as described above. It was started with the use of formic acid alone at high temperatures for 30 minutes. Then a combination with a mineral acid gave the desired results and also lowered the cost. The possibility of working it out at room temperature made it commercially feasible and the reduction of time to about a minute made it possible as a continuous process and consequently a very economical one.

This process is useful for many purposes:

First, by swelling synthetic fibers in a knitted or woven fabric, the texture of such treated fabric will become tighter and, therefore, improve the quality and the weight per square yard, substantially.

Second, it makes the yarn dull, giving the fabric a better appearance.

Third, in some cases, undesirable stretch has been completely eliminated.

Fourth, it has been shown that cloth, treated as described above, has its substantivity toward dyestuffs increased by 25% and more.

Fifth, it has been found possible, by increasing the concentrations of the acid components of the above developed formula, to control the degrees of stiffness of various fabrics, such as with a 70 denier nylon simplex knitted fabric, in which a leather-like appearance was obtained.

Pursuant to the results obtained by combining acetic acid, either commercial (56%) or glacial (100%) with sulphuric acid (20° Baumé) in the treating solution, parallel experiments were conducted with treating solutions containing other combinations, additions or substitutions of their components. The results thus obtained are given in the accompanying drawings, in which:

FIG. 5 is a chart plotting the shrinking results obtained, at a constant temperature of 80° F. in a constant time period of 3 minutes, with solutions containing from 20% to 40% of formic acid (90%) plus from 30% to 50% of sulphuric acid (20° Baumé), upon (A) 40 denier nylon tricot and (B) 40 denier nylon 30 gauge simplex.

FIG. 6 is a chart plotting the shrinking results obtained, upon the same goods as in FIG. 5 at a constant temperature of 80° F. and in a time period of 3 minutes, with a treating solution containing from 20% to 50% sulphuric acid (20° Baumé) and 20% to 30% of hydrochloric acid (20° Baumé) only.

FIG. 7 is a chart, plotting the shrinkage results obtained upon the same goods as in FIG. 5 at a constant temperature of 80° F. and in a constant time period of 3 minutes, with a treating solution containing in each instance 20% sulphuric acid (20° Baumé) plus from 10% to 30% glacial acetic acid (100%) and from 10% to 30% of hydrochloric acid (20° Baumé).

FIG. 8 is a chart, plotting the shrinkage upon the same goods as in FIG. 5, obtained at a constant temperature of 80° F., in a constant time period of 3 minutes, with a treating solution containing a constant concentration of 20% glacial acetic acid (100%) and constant concentra-

tion of 30% sulphuric acid (20° Baumé) plus additions of hydrochloric acid (32%) of varying amounts from 14% to 20%.

From these experimentations and from the results obtained by carrying them out, it has been demonstrated that by the addition of sulphuric acid or hydrochloric acid to organic acid concentrations, the treating solution markedly increases its shrinking action and the degree of shrinkage obtained in the resulting product.

It is also known that the inorganic or mineral acids, sulphuric and hydrochloric, while reactive separately as solvent reagents upon the synthetic filaments and fabrics, etc., made from them, may be combined and used, without any of the organic acid solvent reagents or acidic gelatinizing reagents and give effective shrinking action and results.

It is further shown that a solution containing a given concentration of sulphuric acid (20° Baumé) may be increased in its shrinking power and effects by additions of both glacial acetic acid (100%) and hydrochloric acid (32%) in varying amounts.

It is also shown that treating solutions containing a constant concentration (20%) of glacial acetic acid (100%) and of 30% sulphuric acid (20° Baumé) may be increased in their shrinking functions and effects by the successive additions of 14% to 20% of hydrochloric acid (32%) thereto.

It is also observed that, while shrinkage of the materials treated may be effected with the acid solvent solution organic or inorganic, alone, or by the gelatinizing or swelling reagent solution, alone, the use of mixtures of the solvent reagent and of the gelatinizing or swelling reagent, in various combinations and proportions, imparts an addition to the shrinkage effects and improved qualities of hand, texture and finish to the product obtained.

While the inorganic acids, sulphuric and hydrochloric, are active as solvent reagents toward the synthetic filaments and goods made of them, as above pointed out and demonstrated, they present the additional function of activating the solvent reagents and/or gelatinizing reagents in the treating solutions when added thereto. This is thought to be in part attributable to the fact of their being much stronger acids than the organic acids and characterized by presenting and providing greatly lowered pH values, accordingly, e.g., to below 1, to 0, and even to below 0, as -1, -2, etc. This has been mentioned above, but is demonstrated by the following table of potentiometrically determined and recorded values of pH of some of the treating solutions, containing various combinations of organic acid solvent reagents, organic acid gelatinizing reagents, per se and in admixture, and of sulphuric acid and hydrochloric acid, per se and in admixture, in various concentrations with the others:

Formic Acid, 90%	Acetic Acid, 56%	Sulphuric Acid (20° Baume)	Water	pH
Percent	Percent	Percent	Percent	
25			75	0.9
30			70	.75
40			60	.5
25		25	50	-.25
35		35	30	-.6
40		50	10	-.85
20	20		60	1.0
30	30		40	.6
	40	40	20	-.2
	40	50	10	-.3
	50	50	0	-.4
	100		0	1.1
	30	60	10	-.3

Sulphuric acid of a concentration of 35.4% H_2SO_4 gives a pH value of -1.9.

Acetic Acid, Glacial, 100%	Sulphuric Acid, 20° Baumé	Water	Temperature, ° F.	pH Value
Percent	Percent	Percent		
40	44	16	80	-.6
42	44	14	80	-.6
44	44	12	80	-.65
44	46	10	80	-.7
44	48	8	80	-.7
44	50	6	80	-.75
46	50	4	80	-.8
48	50	2	80	-.85
50	50	0	80	-.90

Without going into the theory of ionization, potentials or pH values, generally, it may be said that for the purposes of the present invention the negative pH values as thus indicated constitute a reverse acid capacity of the system in which they are thus found and determined. To test this aspect of the case, applicant prepared 150 cc. of a solution of 40% formic acid (90%), 50% sulphuric acid (20° Baumé) and 10% of water. This solution, at 80° F., had a pH value of -.3. Upon successive dilutions of this solution with 5 cc. of distilled water and making corresponding determinations of the pH value of each resulting solution, the pH values were, respectively, -.3, -.3, -.3; -.2, -.2, -.2; -.1, -.1, -.1, -.1; 0, 0, +0.1.

In general, therefore, it may be followed as an underlying principle of the invention to control the solvent reagent and/or the gelatinizing reagent in the treating solution used, in terms of its concentration its temperature and the time period of its effective action upon the goods and consequent function of creating and developing felting characteristics and actions upon the surfaces of the filaments or goods under treatment and also to activate or catalyze such characteristics, functions, actions and results, by lowering the pH value of the solution by means of strong inorganic acids as sulphuric and hydrochloric acids. These acids may be said to be effective themselves, to activate or catalyze their solvent actions upon the goods in these respects, under suitably sharply regulated conditions.

It may be pointed out that in the above described examples of the invention the proportions of the several components of the treating solutions are given in the percentages of each, by volume (as one would naturally measure them out in the laboratory, with a graduate) and not by weight. The water component, therefore, having a density or specific gravity of 1, will be present in a somewhat lesser proportion or percentage, if considered in terms of proportions or percentages by weight, than when as thus given by volume—for most of the other components to be added thereto have a density or specific gravity which is greater than 1. For example, if a solution were prepared from 75 cc. of water (75% by volume) and 25 cc. of hydrochloric acid (25% by volume) having a specific gravity of 1.2, the resulting solution would have a volume of 100 cc. and contain 30 (25×1.2) grams of the hydrochloric acid component to 75 grams of water and hence weigh about 105 grams.

The figures above given, therefore, are subject to this slight correction, if they are to be considered in terms of relative proportions or percentages, by weight.

While the method of the invention has been described as being carried out through the several steps and stages

of procedure continuously, so as to effect the ultimate degree of the felting and shrinking functions of the solutions upon the goods or materials treated, it is to be understood that various modifications may be made in such operations without departing from the invention, as described and claimed. Thus, the goods or materials may be treated with the solution and then neutralized, and/or dried, and/or washed, without permitting felting or shrinking, as by intermediate or concurrent simple retention or positively tensioning the same to arrest such felting or shrinking action. Subsequently, such stage product may be relaxed and manipulated to permit or develop the felting and shrinking to take place and thus impart their effects and results to the treated goods or materials, of the kind and degree desired.

The shrinking solutions can be used, and we are using them, over and over again; occasionally it is desirable to clarify and/or to filter such solutions in order to eliminate small particles and light color which could have accumulated during the various treatments.

I claim:

The method of effecting the characteristics of inherent felting upon and the consequent shrinking of uniformly chemically and physically resistant, smooth surfaced, synthetic fibers of nylon, in knitted and woven fabricated textile goods made of them, which comprises the steps of subjecting said textile goods to an aqueous solution of the group consisting of (A) a mixture of 46% to 50% glacial acetic acid of 100% and 45% to 50% sulphuric acid of 20° Baumé; (B) a mixture of 40% formic acid of 90% and 30% to 50% sulphuric acid of 20° Baumé; (C) a mixture of 30% hydrochloric acid of 20° Baumé and 30% to 40% sulphuric acid of 20° Baumé; (D) a mixture of 10% glacial acetic acid of 100% and 25% to 30% hydrochloric acid of 20° Baumé; and (E) a mixture of 20% glacial acetic acid of 100%, 30% sulphuric acid of 20° Baumé and 18% to 20% hydrochloric acid of 20° Baumé, said percentages being by volume, at room temperature and for a period of time of approximately 3 minutes to regulate, develop and determine the degree of felting and shrinking to a total added shrinkage, in length and width, of over 40%, and arresting the action of the solution thereon by removing the active agent from the goods.

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NORMAN G. TORCHIN, *Primary Examiner.*

WILLIAM B. KNIGHT, MORRIS O. WOLK, *Examiners.*