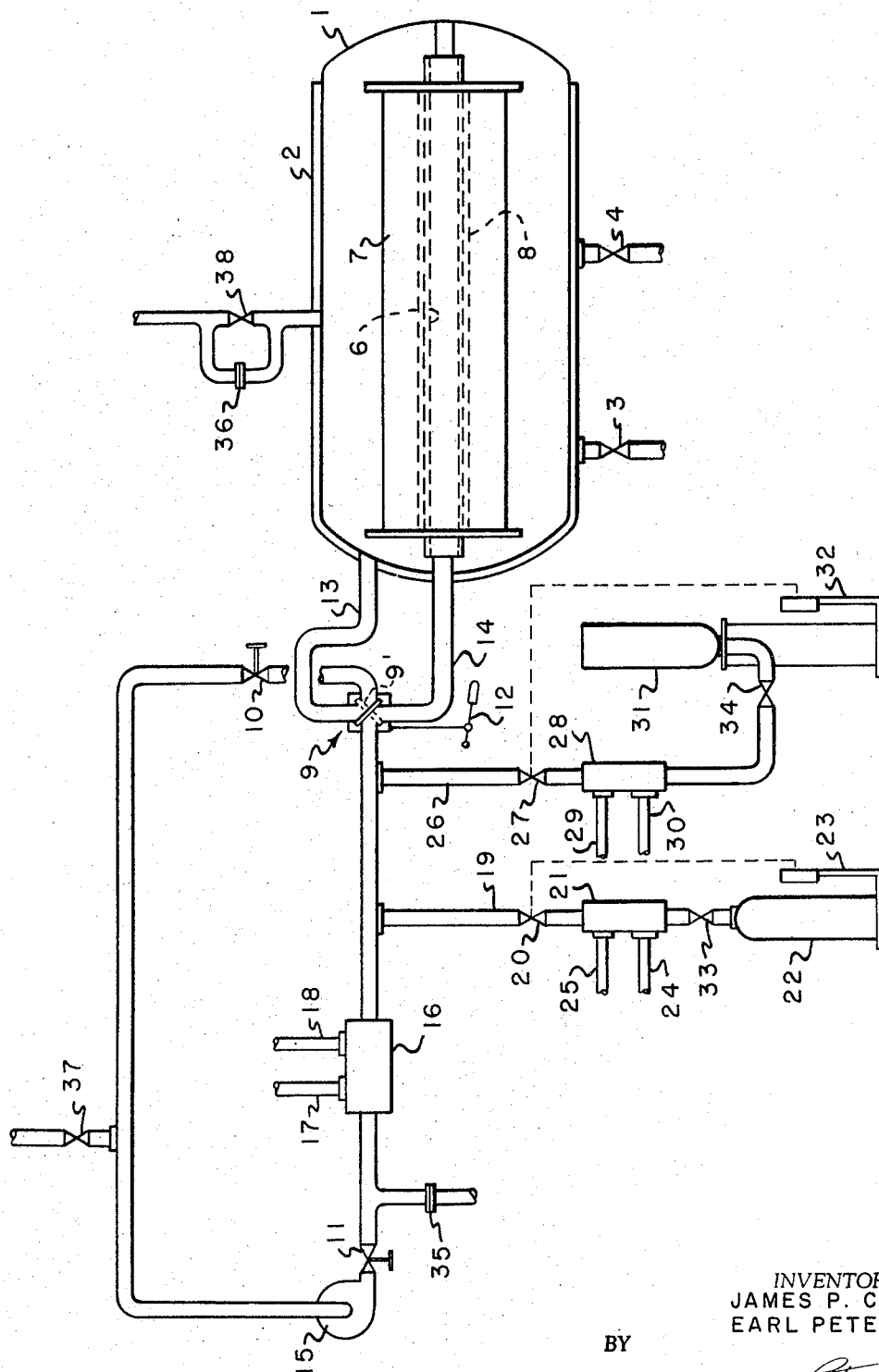


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WOOL FIBERS SENSITIZED FOR SETTING BY APPLYING THERETO ONE
MEMBER OF A REDUCING AGENT-PRODUCING COREACTANT
PAIR, MECHANICALLY FINISHING AND APPLYING
THE SECOND MEMBER OF SAID PAIR
Filed May 6, 1963



INVENTORS
JAMES P. CAIN
EARL PETERS

BY

William Peters
ATTORNEY

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WOOL FIBERS SENSITIZED FOR SETTING BY APPLYING THERETO ONE MEMBER OF A REDUCING AGENT-PRODUCING COREACTANT PAIR, MECHANICALLY FINISHING AND APPLYING THE SECOND MEMBER OF SAID PAIR

Earl Peters and James Palmer Cain, Spartanburg, S.C., assignors to Deering Milliken Research Corporation, Spartanburg, S.C., a corporation of Delaware

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This invention relates to novel processes for modifying the characteristics of keratin fibers and, more particularly, to novel processes for presensitizing fabrics containing keratin fibers for subsequent durable setting, the presensitization being accomplished in such a manner that desirable finishes on the fabrics may be provided. This invention relates particularly to such presensitizing processes whereby the final setting of the fabrics, such as in the form of garments produced therefrom, may be accomplished under normal moisture conditions, i.e., without the addition of moisture beyond the moisture regain level of the fibers.

Processes for the presensitization of fabrics containing keratin fibers, particularly wool fabrics, have recently been developed whereby the final setting of the fabric, e.g., in the form of garments produced therefrom, may be accomplished without the addition of large amounts of water as required by the prior art. Such processes are disclosed in U.S. patent applications, Ser. No. 111,447 and, more particularly, 167,420. These processes have become known to the art as "dry-crease" presensitizing processes, in recognition of the fact that configurations, e.g., creases, pleats and the like, of a durable nature can be accomplished by simple pressing, such as on a Hoffman press, without the previously required water spraying. These processes overcome the necessity for spray equipment and the multiplicity of undesirable manipulations of wet fabric inherent in "wet" presensitizing processes which require addition of water to the fabric prior to pressing.

The above novel "dry crease" presensitizing processes have been widely acclaimed and generally accepted, but in the early stages of this new concept in presensitization some problems have arisen. For example, wool fabrics are presensitized by the process of U.S. patent application, Ser. No. 167,420, by impregnation with an aqueous solution of a reducing agent and a low molecular weight polyhydroxy compound, such as ethylene glycol. The dried fabric does not have a particularly pleasing finish so that it has been necessary to subject the fabrics so treated to mild, conventional finishing operations.

Typical finishing operations include full decatizing, wherein the fabric, in rolled form and in contact with a particular leader cloth, is exposed to steam in a pressurized autoclave for several minutes after which vacuum conditions are imposed on the fabric. An alternative operation, called semi-decatizing, is similar to full decatizing, except that steam at atmospheric pressure is forced through the rolled fabric. These finishing techniques, however, are somewhat similar to the conditions necessary to set the fabric, e.g., in garment form, to a desired configuration. For example, the full decatizing operation substantially destroys the presensitization in the fabric. Consequently, the somewhat milder semi-decatizing operation has been used. Even this mild finishing operation, however, can cause the loss of a substantial degree of the presensitization if care is not taken.

A further difficulty arises from the fact that from the

outset of the finishing operations, the fabric is in a reduced state and, as a result, is more susceptible to stresses which induce in the fabric a higher degree of residual relaxation shrinkage. Normal fabrics have some residual relaxation shrinkage, but this difficulty is overcome by wetting the fabric thoroughly prior to cutting into garment form and drying in a relaxed state. This technique is not practicable for presensitized fabrics since the presensitizing chemicals are substantially removed by the wetting operation. Consequently, it has been necessary to exercise a high degree of control over these novel presensitizing processes to avoid building into the fabrics a high residual relaxation shrinkage.

While an excellent presensitized fabric can be provided by the above processes, the high degree of control required during the finishing operation to avoid both destruction of presensitization and increased residual relaxation shrinkage adds materially to the processing cost of such fabrics.

These difficulties have been overcome in accordance with this invention by forming a reducing agent in situ on the fabric after conducting the desired finishing operations. This procedure is most efficaciously accomplished by contacting the fabric with a reducing agent precursor, conducting the desired finishing operations, and then exposing the fabric to a gaseous reducing agent activator. In this manner, all wet procedures are conducted prior to application of the desired finish and this finish is substantially retained since the gaseous activator does not disturb the finish.

Furthermore, the keratin fibers of the fabric are not in reduced form until after all finishing operations have been completed, so that no extraordinary care must be taken to avoid inducing high residual relaxation shrinkage properties into the fabric.

Also, since the fabric is not presensitized until after exposure to the gaseous activator chemical, full finishing operations can be practiced to impart a high degree of finish to the fabric with no effect whatsoever on the degree of presensitization.

By "reducing agent precursor" as utilized herein is meant a chemical compound which forms a reducing agent for keratin fibers upon reaction with another chemical compound. It is generally preferred that the precursor compound have a pH of about 7 or greater as a 1% solution in water. Particularly suitable compounds include lower alkanolamines, such as monoethanolamine, diethanolamine, triethanolamine, N-methyl ethanolamine, N-ethyl ethanolamine, N,N-dimethyl ethanolamine, N,N-diethyl ethanolamine, N,N-diisopropyl ethanolamine, N-aminoethyl ethanolamine, N-methyl diethanolamine, n-propanolamine, isopropanolamine, triisopropanolamine, n-butanolamine, dimethyl-butanolamine, dimethylhexanolamine, polyglycolamines of the general formula $\text{HO}(\text{C}_2\text{H}_4\text{O})_x\text{RNH}_2$ wherein x is a positive integer and R is alkyl, e.g., the compound where $x=2$ and $\text{R}=\text{C}_3\text{H}_7$, and the like. These compounds readily form reducing agent compounds upon exposure to SO_2 gas and other activators.

While the above alkanolamines constitute the preferred embodiment of the reducing agent precursor compounds, additional compounds include other amines, for example those characterized by the formula $\text{R}(\text{NH}_2)_x$ wherein x is a positive integer of from 1 to about 4 and R is alkyl (e.g., ethylamine, hexylamine and the like); aryl (e.g., aniline, toluidines, benzidine, and the like); $\text{R}'\text{CONH}-$ wherein $x=1$, and R' is alkyl or aryl, (e.g., hydrazides, such as acetoxy hydrazide $\text{H}_3\text{C}-\text{CONH}-\text{NH}_2$, butyrylhydrazide, benzoylhydrazide, and the like); hydrazines of the formula $\text{R}'\text{NHNH}_2$ wherein R' is selected from hydrogen, alkyl, aryl, and the like; e.g., hydrazine, methyl-

hydrazine, phenylhydrazine and the like; piperazine compounds, such as piperazine, homopiperazine, N-methyl piperazine, N-hydroxyethyl piperazine, N-aminoethyl, N-phenyl piperazine and the like.

Additional suitable basic precursor compounds include alkalis, such as the alkali-earth metal and alkali-metal compounds, including the hydroxides, carbonates, borates, phosphates, e.g., sodium hydroxide, potassium hydroxide, lithium hydroxide, strontium hydroxide, barium hydroxide, sodium carbonate, sodium bicarbonate, potassium carbonate, potassium bicarbonate, sodium borate, potassium borate, sodium perborate, disodium monohydrogen phosphate and the like.

Additional reducing agent precursor chemicals include aldehydes, particularly formaldehyde and glyoxal, although other aldehydes are suitable, e.g., saturated aliphatic aldehydes containing up to about 18 carbon atoms, such as acetaldehyde, propionaldehyde, butyraldehyde, valeraldehyde, caproaldehyde, enanthaldehyde, nonaldehyde, palmitic aldehyde and the like; unsaturated aliphatic aldehydes, such as acrolein, crotonaldehyde, tiglic aldehyde, citral, propionaldehyde and the like; alicyclic monofunctional aldehydes, such as formylcyclohexane and the like; aliphatic dialdehydes, such as glyoxal, pyruvaldehyde, malonaldehyde, succinaldehyde, glutaraldehyde, adipaldehyde and the like; aromatic aldehydes, such as benzaldehyde, tolualdehyde, α -tolualdehyde, cinnamaldehyde, salicylaldehyde, anisaldehyde, phenylacetaldehyde, α -naphthaldehyde, anthraldehyde, pyrocatechualdehyde, veratraldehyde and the like; heterocyclic aldehydes, such as α -formylthiophene, α -formylfuran, and the like; dialdehyde starch, and other aldehyde carbohydrates and aldehydic cellulosic materials.

It has also been discovered that ammonia per se can be utilized as a reducing agent precursor, e.g., in combination with SO_2 or N_2O_3 activator gases. Ammonia may be provided as a gas, preferably anhydrous, or as ammonium hydroxide, methylammonium hydroxide, ethylammonium hydroxide and similar compounds. When applied as a gas, it may be applied before, after or during the desired finishing operation.

The procedure of applying the ammonia to the fabric after finishing, and before or after application of the activator gas is highly preferred for its simplicity and for the excellent results which are obtained without a wet finishing operation, which involves the added expense of padding and drying. In addition, ammonia gas may be utilized in combination with other reducing agent precursors. For example, a wool fabric can be impregnated with an alkanolamine, finished, and then exposed to both ammonia and SO_2 gases. This technique provides particularly excellent presensitization in that better creases, or other configurations, of improved durability can be accomplished in this matter.

On the other hand, ammonia may be utilized as a reducing agent activator per se, particularly in combination with nitrites to form ammonium nitrites, ammonium complexes and the like.

As noted above, the reducing agent precursor, except for ammonia gas, is preferably applied to the fabric prior to finishing in that these compounds are most conveniently applied to the fabric in liquid media which would substantially destroy the finish on the fabric. Most of the precursors are soluble in water and can be applied to the fabric as aqueous solutions, although dispersions, emulsions and other systems are suitable. Uniform impregnation of the fabric is readily accomplished by conventional techniques, such as padding, spraying and the like. It should be appreciated, however, that the precursor chemical may be applied in gaseous form before or after finishing if the practitioner prefers to volatilize the normally liquid precursor systems.

By "reducing agent activator" as utilized herein is meant a chemical compounds, preferably in a gaseous state,

which can react with one of the above reducing agent precursors to form a different chemical compound which is a reducing agent for keratin fibers, i.e., is capable of rupturing the disulfide bonds of the keratin fiber molecular structure. It is not known with certitude if a reducing agent per se is formed in situ on the keratin fibers being treated, although the formation thereof is highly probable.

Most of the preferred activator gases are reducing agents for keratin fibers and it is possible that the pretreatment of the fibers with one of the precursor chemicals may merely sensitize the fibers for more efficient reaction of the keratin fibers with the reducing agent gas, whereby presensitization of the fibers for subsequent durable setting is effected.

Regardless of the mechanism of the presensitization, however, the reducing agent precursor chemicals and the reducing agent activator gases can react in the absence of keratin fibers to form different reducing agent compounds. Furthermore, presensitization is effected by the process of this invention where treatment with a reducing agent per se is essentially ineffective for such purpose. For example, excellent presensitization of keratin fibers for subsequent durable setting in the absence of large amounts of water is effected by the process of this invention wherein the fabric is first impregnated with monoisopropanolamine, subjected to the desired finishing operation and then exposed to SO_2 , possibly to produce monoisopropanolamine sulfite in situ on the fabric. On the other hand, if monoisopropanolamine sulfite per se is utilized, no such presensitization is effected and a durable crease could only be obtained by pressing the fabric while it is wet with large quantities of water, e.g., on the order of 40% by weight or more. Similarly, treatment with SO_2 gas alone is generally ineffective in producing a fabric presensitized for subsequent durable setting, with or without large amounts of water.

Since sulfites are generally excellent reducing agents for keratin fibers, SO_2 is a highly preferred reducing agent activator gas. Other suitable activator gases include, however, hydrogen sulfide; mercaptans, such as methyl mercaptan (B.P. 6°C .), ethyl mercaptan (B.P. 37°C .) and the like; mercaptan alcohols, such as 2-mercaptoethanol (B.P. $50-52^\circ \text{C}$. at 10 mm. Hg) and the like; nitrogen oxides, such as N_2O_3 and the like; phosphorus-containing gases, such as phosphine and the like; nitrosating agents, such as NOCl , NOBr and the like.

The amount of reducing agent precursor and activator gas can be readily determined by one skilled in the art depending on the fabric being treated and the extent of presensitization desired.

The preferred precursor chemicals are fairly strong bases. Keratin fibers tend to degrade considerably during prolonged storage under basic conditions. It is preferred, therefore, that a sufficient amount of the reducing agent activator gas, which generally is acidic, be utilized for substantially complete reaction with the precursor chemical or until substantially neutral fabric is produced. Obviously, the fabric may be shipped under slightly acidic or basic conditions, or even under highly acidic or basic conditions, but the optimum degree of physical properties in combination with the presensitized characteristic is obtained when the fabric is shipped essentially neutral.

In this regard, the fabric treated in accordance with this invention has a higher degree of creasability after prolonged storage than fabrics treated by previous techniques. In fact, the performance of the fabric after storage is generally superior to the performance immediately after gassing. Consequently, conventional storage time has become an asset rather than a liability in the present presensitizing technique.

Fabric containing the precursor chemical may be exposed to the gaseous activator in conventional equipment. For example, steam boxes, decating apparatus, beam and package dye machines, drying ovens and the like may be utilized.

Slightly improved results, though at increased cost, are obtained when a swelling agent or "low molecular weight polyhydroxy compound" is applied to the fabric in combination with the reducing agent precursor.

By the term "low molecular weight polyhydroxy compound" is meant a compound containing more than one hydroxy group and having a molecular weight no greater than about 4000. Of these compounds, the most readily available and desirable compound, from the standpoint of ease of application, comprises ethylene glycol. A particularly preferred group of glycols includes the polyfunctional glycols having terminal hydroxyl groups separated by 2 to 10 methylene groups, including, of course, the preferred ethylene glycol as well as trimethylene glycol, tetramethylene glycol, pentamethylene glycol, hexamethylene glycol, heptamethylene glycol, octamethylene glycol, nonamethylene glycol, and decamethylene glycol, or such glycols as 1,2-propylene glycol, dipropylene glycol, 1,3-butylene glycol, diethylene glycol, polyethylene glycol or the like.

Polyfunctional compounds containing more than 2 hydroxyl groups include the polyfunctional alcohol glycerols such as glycerine, quaternyl glycerin, diethylglycerol and mesicerin, as well as trimethylol ethane, trimethylol butane, tris(hydroxymethyl)aminomethane and others. Glycol ethers, such as the water-soluble or dispersible polyethylene glycols or polypropylene glycols having molecular weights no greater than about 4000 also provide satisfactory results when utilized in accordance with this invention.

Urea constitutes the most readily available and desirable swelling agent, although any other material which will swell wool fibers in an aqueous medium is suitable. For example, guanidine compounds such as the hydrochloride; formamide, N,N-dimethylformamide, acetamide, thiourea, phenol, lithium salts, such as the chloride, bromide and iodide and the like are similarly useful.

The swelling agent or low molecular weight polyhydroxy compound may be utilized in any desired amount depending on requirements for particular fabrics. For example, as little as about 0.5 to about 1% of the additives, based on the weight of the fabric, provides some improvement, although, in general, larger amounts, e.g., from about 3 to about 10% by weight provide noticeable improvement. Larger amounts of up to about 50% or higher may be utilized, of course, if the particular end use justifies the increased chemical cost in the use of these additives.

Since the application of the swelling agent and/or the polyhydroxy compound is generally a wet process, it is preferably conducted prior to finishing.

By "finishing" as utilized herein is meant a mechanical operation whereby at least one surface of the fabric is treated to impart thereto a more lustrous appearance and/or pleasing hand. In this context, finishing is a well-known and conventional operation.

Although any desired finishing technique may be utilized, e.g., rotary pressing and the like, steaming techniques such as semi-decating and full decating are preferred. These techniques apply little or no tension to the fabric, thereby minimizing residual relaxation shrinkage. Full decating involves wrapping the fabric around a perforated drum located in a pressure autoclave. Steam is forced through the drum and the fabric under a constant pressure, generally up to about 30 pounds per square inch gauge for several minutes, e.g., about 1-5 minutes, after which vacuum conditions across the fabric are maintained for another several minutes, e.g., 2-20 minutes.

In a semi-decating operation, steam at atmospheric pressure is forced through a perforated drum and fabric rolled thereon until it emerges from the outer layer. After this steam "breakthrough," the steam is continued to be passed through the fabric for a few minutes, e.g., up to 2 or more minutes. The extent of decating may be modified

to provide the desired degree of luster of the finishes obtained.

Treatment of fabrics in accordance with this invention may be conducted under atmospheric conditions if desired. It is generally preferred, however, to conduct the treatment under conditions of elevated temperatures and pressures. Improved results are obtained when the fabric containing a precursor chemical is heated slightly before, during or after treatment with the activator gas.

Although the reason for the improvement is not fully understood, it is believed that the probable reaction which occurs between the precursor chemical, activator gas and, possibly, the keratin fibers proceeds more efficiently at the elevated temperature.

The level of temperature is preferably below the volatilization temperature of the precursor chemical and below the decomposition temperature of the expected reaction product. For most of the preferred combinations of precursor and activator gas, this temperature will be below about 212° F., preferably below 180° F.

By evacuating the chamber containing the fabric to be treated, most efficient impregnation of the fabric with the activator gas may be accomplished. For example, vacuum conditions can be produced in the chamber. When the activator gas is introduced into the chamber, the vacuum can be broken and the gas will be forced more efficiently into the interstices of the fabric as a result of the pressure increase.

Since the keratin fibers of the fabrics being treated in accordance with the process of this invention are not in a reduced state until after gassing, the fabric, after padding with the precursor chemical, may be dried at levels normally encountered in mill operations, for example, about 200° F. and usually not in excess of 250° F. When this drying procedure is conducted with the fabric under substantially relaxed conditions, the resulting fabric is characterized by even lower area shrinkage than is untreated fabric. In this condition, the fabric may be shipped to a garment manufacturer who can cut the fabric as received without conducting the sponging or shrinking operations which he would be forced to conduct with conventional fabrics. These operations are particularly undesirable to the garment manufacturer since he must not only wet out the fabrics received but must permit the fabric to dry under relaxed conditions to insure that the fabric shrinks in its uncut configuration rather than upon subsequent wetting in the form of garments. This sponging or shrinking procedure, although a great nuisance to garment manufacturers, heretofore has been as essential step in the manufacture of garments from conventionally finished fabrics.

Regardless of drying procedure, however, fabric treated as disclosed herein has low residual relaxation shrinkage since it is not handled in a wet, reduced state. The garment manufacturer may cut the fabric of this invention into the desired configuration necessary for the production of a garment, sew the garment, press the garment without any additional step of treatment required and obtain a garment characterized by durable creases or pleats, as the case may be. In addition, the flat areas of the garment manufactured from the fabric of this invention are also characterized by a tendency to maintain a desirably flattened configuration, even under hot, wet conditions.

It may be readily seen, therefore, that the process of this invention not only provides the wearer of the garment with a most desirable garment but also completely eliminates many of the undesirable techniques and additional procedures required heretofore on the part of garment manufacturers in the production of such garments.

While the process of this invention is particularly adapted to fabrics composed essentially of keratin fibers, particularly those composed entirely of wool fibers, it is also applicable to fabrics wherein synthetic, natural, or other keratin fibers are blended with the wool component. Other keratin fibers include mohair, alpaca,

cashmere, vicuna, guanaco, camels hair, Llama and the like. Preferred synthetic fibers for blending with wool fibers include polyamides, such as polyhexamethylene adipamide; polyesters, such as polyethylene terephthalate; and acrylic fibers, such as acrylonitrile homopolymers or copolymers containing at least about 85% combined acrylonitrile, such as acrylonitrile/methyl acrylate (85/15), and cellulose, such as cellulose acetate and viscose rayon. Of the natural fibers which may be blended with the keratin fibers, cotton is preferred.

Furthermore, the fibers need not be in fabric form during treatment. For example, the process may be conducted on top, tow, roving, sliver, yarn and the like.

The process of this invention may be performed on woven, non-woven, or knitted fabrics of any type, dyed or undyed, provided, of course, that the dyes are stable to the precursor and activator chemicals. In this regard the dyes are far less sensitive to the precursor chemicals than they are to the reducing agents utilized in previous processes.

Another advantage of the process of this invention is that the fabric may be treated over a wide range of pH. For example, a conventional finishing treatment applied to wool fabrics involves the application to the fabric of concentrated sulfuric acid in order to carbonize cellulosic impurities present in the fabric. After this treatment, from 2 to 3% by weight of sulfuric acid is often left on the fabric. One of the advantages of this invention is that fabrics so treated may be subjected to the process of this invention without being neutralized, although, obviously, the fabric may be neutralized if, for some reason, the operator prefers. As noted above, the preferred reducing agent precursors are basic. Wool fabrics, for example, degrade if maintained under basic conditions for extensive periods of time. Consequently, it is preferred that the gas reaction be conducted to an extent sufficient to provide an essentially neutral fabric shipment.

A typical apparatus for conducting the processes of this invention is shown schematically in the drawing. The apparatus includes reaction chamber 1 partially surrounded by jacket 2 for heating or cooling the chamber and its contents. Steam or other heating or cooling fluid can be admitted through inlet valve 3 and withdrawn through outlet valve 4. Mounted within the reaction chamber 1 in circulating relationship with conduit 14 is a perforated spindle 6. Full width lengths of fabric 7 supported on a perforated beam 8 is mounted over the perforated spindle 6. A four-way reverse valve 9 is provided within the recirculation system for changing the direction of flow of the gas or gases utilized in the process, valves 10 and 11 being provided for flow rate control. Hand lever 12 is provided for manual operation of the valve, although automatic control can be readily provided.

The recirculating system connected to four-way valve 9 includes recirculating conduits 13 and 14, motor-driven air pump 15 (motor not shown), heat exchanger 16 connected to steam or water line 17 and return line 18, gas supply conduit 19 connected through an automatic valve 20 and heat exchanger 21 to pressurized gas container 22 mounted on scale 23 electrically connected to valve 20 for automatic control. Heat exchanger 21 is provided with steam or water line 24 and return line 25.

A similar system for an additional gas is connected into the recirculating system through conduit 26, automatic valve 27, heat exchanger 28 having steam or water line 29 and return line 30 to pressurized gas container 31 mounted on scale 32 electrically connected to valve 27 for automatic control.

Additional valves 33 and 34 are provided for control of the flow of the gas or liquids fed into heat exchangers 21 and 28, respectively.

Blow-out disk 35 is provided in the recirculating system and a similar disk 36 is provided in the vent for

reaction chamber 1. An air intake valve is provided in the recirculating system at 37 and a vent valve is provided in the reaction chamber at 38.

In operation of this apparatus, the fabric beam is mounted within reaction chamber 1 onto perforated spindle 6 in circulating relationship with conduit 14 of the recirculating system. The fabric can be preheated if desired by admitting steam to jacket 2.

Ammonia gas from container 22 is fed into heat exchanger 21 and through automatic valve 20 into conduits 19 and 14 until a predetermined amount is registered on scale 23 at which time the coordinated automatic valve 20 closes. This gas is then passed through conduit 14 into perforated spindle 6, perforated beam 8 and fabric 7 and back into the recirculating system conduit 13 where it passes through heat exchanger 16 for maintenance of the desired gas temperature. This direction of flow is called inside-out flow.

After a predetermined period of time, the direction of flow is reversed by switching valve 9 to the position shown by the dotted lines at 9'. In this position, the flow of the gas is reversed through the valve 9 so that the gas enters the area surrounding the fabric beam where it is pulled through fabric 7, perforated beam 8 and perforated spindle 9 back into conduit 14 of the recirculating system. This direction of flow is called outside-in flow.

This procedure is continuously repeated to provide any desired cycle of inside-out, outside-in flow, which cycle is normally continued throughout all treatments.

After a predetermined period of exposure to the ammonia gas, the machine is vented by closing valve 10, opening inlet valve 37 and venting valve 38 of the reaction chamber 1, while continuing the cycling operation. After a few minutes, valve 10 is opened and valves 37 and 38 are closed.

Liquid sulfur dioxide from container 31 is then passed into heat exchanger 28 which is heated by steam. The liquid immediately vaporizes and is then admitted through valve 27 which automatically closes upon signal from scale 32 that a predetermined amount of liquid has been utilized.

After recirculation for the desired time, the machine is vented to the atmosphere as before, after which additional ammonia is fed into the system for recirculation.

The cycling operation is then discontinued and the reaction chamber 1 is again vented to the atmosphere, air being forced through the fabric in inside-out flow. After this flushing operation, the machine is opened and the fabric beam removed.

This apparatus can be used for any embodiment of this invention, e.g., wherein the fabric has been pretreated with a reducing agent precursor chemical and wherein one or more gases are utilized.

In the following examples, various properties of fabrics treated in accordance with this invention are measured. Tongue tear strength is measured according to the A.S.T.M. Test for Woven Fabrics (Designation 39), Tongue Method; flex abrasion according to A.S.T.M. Test for Abrasion of Textile Fabrics (Designation 1175), Flexing and Abrasion Method; and tensile strength according to A.S.T.M. Test for Woven Fabrics (Designation 39), Grab Method. The pH values are obtained by the Approved Method for the Determination of the pH Value of a Water Extract of Wool adopted by the International Wool Textile Organization Technical Committee in May 1960.

Reducing power (expressed as percent SO_2) is determined by extracting a 50 gram sample of presensitized wool fabric in a Soxhlet extractor with methanol for four hours under nitrogen atmosphere. After extracting and cooling, the extract is diluted to 250 ml. in volumetric equipment. A ten-ml. aliquot of the extract is placed in a 125 ml. Erlenmeyer flask along with 50 ml. water, 10 drops of 6 N H_2SO_4 and 1 ml. starch indicator solution.

The resulting system is titrated with 0.1 N KI_3 solution to the first blue end point that persists for 10 seconds.

Dry crease performance data are obtained from presensitized fabric samples having dimensions of $4\frac{1}{2}$ inches in the filling direction by 6 inches in the warp direction. These samples are folded in half with the fold parallel to the warp yarns. The samples are then placed on a Hoffman press, the cover is closed and locked and the samples are pressed with 30 seconds top steam and 30 seconds baking, followed by 10 seconds vacuuming.

The creased samples are then opened and placed in a standing water bath which contains a wetting agent and is heated to 170° F. After 30 minutes the samples are removed, folded along the original crease line and allowed to air dry. After drying, the creases remaining in the samples are rated subjectively by at least three observers, the crease ratings running from 1 (no appreciable crease) to 5 (very sharp crease).

In the 120° F. Hanging Test, $4\frac{1}{2}$ by 12 inch samples are similarly pressed. In this test, however, the samples are opened and suspended from a clip frame and lowered into a similar bath at 120° F. After 30 minutes, the samples are raised from the bath and allowed to dry suspended on the frame.

Wet creases are measured by either of the above techniques by wetting the presensitized fabric samples with water to 40% wet pick-up prior to Hoffman pressing.

EXAMPLE I

An all wool fabric, Deering Milliken Style 8012 worsted fabric, is impregnated with an aqueous solution containing 5.5% monoethanolamine and 0.1% Sulfonic N-95 non-ionic ethylene oxide condensate type wetting agent. After drying, the fabric is steamed on a Hoffman press to impart a desirable finish thereto. The finished fabric is then rolled onto a perforated spindle and SO_2 gas at 3 liters/minute is forced through the spindle and fabric for 3 minutes.

After storage for 24 hours, the fabric is creased without the addition of water and tested as set forth above to provide a crease rating of 3.0.

This procedure is repeated except that different reducing agent precursor solutions are utilized, as set forth in Table I.

Table I

Treatment prior to finishing:	Crease rating (dry)
Control	1.0
No pretreatment (SO_2 gas only)	1.0
5.5% monoethanolamine	3.0
9.6% monoethanolamine carbonate (pH 8.0)	2.5
6.2% monoisopropanolamine	3.0
11% monoisopropanolamine carbonate (pH 8.0)	2.5
7.7% tris(hydroxymethyl)aminomethane	3.5
13% tris(hydroxymethyl)aminomethane carbonate (pH 8.0)	3.0
20% ethylene glycol	1.3
5.5% monoethanolamine+20% ethylene glycol	3.5
9.6% monoethanolamine carbonate+20% ethylene glycol	3.0
6.2% monoisopropanolamine+20% ethylene glycol	3.5
11% monoisopropanolamine carbonate+20% ethylene glycol	3.0
7.7% tris(hydroxymethyl)aminomethane+20% ethylene glycol	4.0
13% tris(hydroxymethyl)aminomethane+20% ethylene glycol	3.5

EXAMPLE II

A sample of worsted fabric Style 8012 is padded to 80% pick-up with an aqueous solution of 6.0% mono-

isopropanolamine and 0.1% Syn-Fac 905, a non-ionic ethylene oxide condensate type wetting agent. The temperature of the pad-bath is 75° F. The impregnated fabric is then dried in a tensionless dryer at 215° F. to a moisture level of 10%. After drying, the fabric is semi-decated, the cycle consisting of steam breakthrough plus $1\frac{1}{4}$ minutes of steam at 100 p.s.i. gauge, followed by three minutes vacuum pumping at 500 mm. mercury.

After semi-decating, the fabric is rolled onto a perforated spindle for a full-decater autoclave. The spindle is placed in the autoclave, the unit is sealed, and the fabric is heated to 140° F. Sulfur dioxide gas is then introduced into the system in stoichiometric proportions to that amount of monoisopropanolamine previously padded onto the fabric. After introduction of the sulfur dioxide into the closed system, the fabric is allowed to stand in the sulfur dioxide vapors for an additional 10 minutes, after which air is forced through the fabric, the autoclave being ventilated to permit removal of any remaining and/or unreacted sulfur dioxide.

After creasing and testing as shown above, a crease rating of 2.4 is obtained.

EXAMPLE III

The procedure of Example II is repeated except that a Gaston County (Theis) Drying Machine is utilized for the gassing step. In this machine, SO_2 gas is recirculated back and forth through the fabric until all the amine has, theoretically, reacted. Fabric treated in this manner has a crease rating of 2.5.

EXAMPLE IV

The procedure of Example II is repeated except that the semidecating cycle is 5 seconds steaming after breakthrough, followed by 2 minutes vacuum pumping. In addition, the fabric is placed in a Burlington Dyeing Machine and pre-heated to 80° F. prior to gassing. Crease ratings of 2.0 are obtained in this manner. This value increases to 2.5 after 1 week's storage.

EXAMPLE V

The procedure of Example II is repeated except that 5% ethylene glycol is added to the pad bath. Crease ratings after 24 hours are 2.3; after 1 week, 2.5.

EXAMPLE VI

Samples of the worsted fabric of Example I are padded to a 70% wet pick-up of two pad solutions:

(1) 6.1% monoisopropanolamine +0.1% Syn-Fac 905 wetting agent;

(2) 6.1% monoisopropanolamine +20% ethylene glycol and 0.1% Syn-Fac 905.

After drying at 200° F. and semi-decating at a cycle of 5 seconds steam followed by 2 minutes vacuum pumping, the fabric (weight of 70 lbs.) are rolled onto the beam of a Burlington Dyeing Machine. The unit is then sealed and SO_2 is forced through the beam at 15 liters per minute, corrected to atmospheric pressure, for a time period computed at 0.28 minutes per pound of fabric. This amount of SO_2 gives the stoichiometric amount necessary to combine with the amine. The gassing operation is continued for 20 minutes, after which the fabric is permitted to stand for an additional 20 minutes in the SO_2 -laden atmosphere within the machine. Compressed air is then introduced into the beam inlet to expel excess SO_2 from the fabric prior to removal of the beam from the machine.

Samples of fabric, after decating and after SO_2 treatment, are analyzed for reducing power (percent SO_2 extractible) free amine content, pH, crease performance and physical properties. These fabrics are compared with an untreated control.

TABLE II.—AFTER AMINE PAD—BEFORE SO₂ TREATMENT

Solution	Instron test		Tongue tear (grams)	Flex abrasion resistance (cycles to failure)
	Grab tensile			
	Strength (lbs.)	Elongation percent		
Control-----	77.5	60.5	1,926	-----
1-----	70.4	50.6	1,841	741
2-----	73.2	53.0	1,698	925

TABLE III.—AFTER SO₂ TREATMENT AND 4 DAYS AGEING

Solution	Relaxation shrinkage (percent)		Instron test			Flex abrasion resistance (cycles to failure)
	Warp	Fill- ing	Grab tensile		Tongue tear (gms.)	
			Strength (lbs.)	Elonga- tion (percent)		
Control			77.5	60.5	1,926	
1	1.43	3.03	72.7	51.6	1,851	803
2	1.70	3.17	80.4	58.7	1,878	938

TABLE IV.—REDUCING POWER AND DRY CREASE RATINGS

Solution	Percent SO ₂	Dry crease rating after 4 days
Control.....	0	1.0
1.....	0.24	3.1
2.....	0.23	2.5

EXAMPLE VII

Samples of the fabric of Example I are padded to 100% pick-up with each of the following solutions:

- 6.1% monoisopropanolamine +20% ethylene glycol +0.1% Syn-Fac 905
- 6.1% monoisopropanolamine +0.1% Syn-Fac 905
- 9.15% monoisopropanolamine +20% ethylene glycol +0.1% Syn-Fac 905
- 9.15% monoisopropanolamine +0.1% Syn-Fac 905
- 12.2% monoisopropanolamine +20% ethylene glycol +0.1% Syn-Fac 905
- 12.2% monoisopropanolamine +0.1% Syn-Fac 905

After drying in a mechanical convection oven at 200° F., a lustrous finish is imparted to the fabrics by placing them on a Hoffman press between two pieces of cotton leader cloth, press-steaming for 15 seconds and baking for 60 seconds under vacuum conditions. Some samples are removed for pH measurements, amine content determination and physical tests.

The remaining samples are exposed to SO₂ gas until a definite excess is detected by moist indicator paper outside the fabric roll. Creases durability tests, extractions for reducing power and physical tests are performed on these samples.

TABLE V.—REDUCING POWER AND DRY CREASE RATINGS AFTER 1 DAY

Solution	Percent SO ₂	Dry crease rating at 170° F.
A.....	0.79	4.9
B.....	0.46	4.4
C.....	0.42	4.0
D.....	0.65	4.5
E.....	1.27	5.0
F.....	0.47	4.8
Control.....	0.0	1.0

TABLE VI.—pH OF FABRIC

Solution	After amine pad, before SO ₂ treatment	After SO ₂ treatment
Control.....	5.4	5.3
A.....	8.1	7.5
B.....	9.2	7.7
C.....	9.5	7.4
D.....	9.5	5.4
E.....	9.6	8.9
F.....	9.6	

¹ No amine.

TABLE VII.—PHYSICAL TEST

Solution	Instron test, grab tensile		Tongue tear (gms.)	
	Strength (lbs.)	Elongation percent		
5	After amine treatment and finish- ing—before SO ₂ treatment:			
	Control.....	71.1	44.0	1,719
	A.....	77.6	62.3	1,678
	B.....	71.7	55.3	1,633
	C.....	74.6	64.1	1,524
10	D.....	75.1	60.6	1,615
	E.....	75.9	61.3	1,551
	F.....	71.0	55.6	1,488
	After SO ₂ treatment:			
	A.....	81.6	73.0	1,696
	B.....	85.0	65.3	1,814
	C.....	81.5	68.6	1,583
15	D.....	83.9	70.6	1,864
	E.....	83.3	71.1	1,696
	F.....	83.9	74.3	1,746

EXAMPLE VIII

In this example, the various fabric samples are pre-treated with solution B of Example VII, dried and finished on a Hoffman press as in Example VII. The fabric is then rolled tightly on the spindle of a package dyeing machine, the machine is locked, and SO₂ is admitted to the spindle inlet under various conditions of pressure. By admitting the SO₂ to an evacuated autoclave, the fabric is more readily impregnated with the gas. In addition, the pressure decreases as the SO₂ reacts, thereby providing the practitioner with a method of controlling the amount of SO₂ utilized through measurement of pressure drop.

In Table VIII, there are shown various treatments involving the evacuated package dye machine and crease ratings obtained. The initial MIPA designate monoisopropanolamine.

TABLE VIII

Treat- ment num- ber	Description of treatment	Crease rating
1.....	Treat with 6.0% MIPA; semi-decate on Hoffman press; break vacuum of 71 cm. with SO ₂ (5 times).	3.7
2.....	Treat with 6.0% MIPA; semi-decate in dye machine; SO ₂ under 10 p.s.i. pressure.	2.3
3.....	Treat with 6.0% MIPA; semi-decate in dye machine; break vacuum of 70 cm. with SO ₂ (1 time).	3.1
4.....	Treat with 6.0% MIPA; semi-decate in dye machine; break vacuum of 30 cm. with SO ₂ (1 time).	3.7
5.....	Treat with 6.0% MIPA; semi-decate in dye machine; break vacuum of 20 cm. with SO ₂ (1 time).	3.9
6.....	Treat with 6.0% MIPA; semi-decate in dye machine; break vacuum of 10 cm. with SO ₂ (1 time).	3.0
7.....	Treat with 6.0% MIPA; semi-decate in dye machine; break vacuum of 71 cm. with SO ₂ ; allow pressure to increase; break vacuum of 47 cm. to 0 with NH ₃ .	5.0
8.....	Treat with 6.0% MIPA; semi-decate on Hoffman press; break vacuum of 71 cm. with SO ₂ ; allow pressure to increase; break vacuum of 46 cm. to 0 with NH ₃ .	4.6
9.....	Treat with 6.0% MIPA; do not semi-decate; blow SO ₂ through fabric at 2 liters/min. for 5 minutes; pump for 10 minutes at 40 cm. vacuum for flushing.	4.0
10.....	Treat with 6.0% MIPA and 1.0% Synsoft LS; do not semi-decate; blow SO ₂ through at 2 liters/min. for 5 minutes; age for 5 minutes; blow with air for 30 minutes.	3.5
11.....	Treat with no chemicals and do not semi-decate; blow SO ₂ through fabric at 2 liters/min. for 5 minutes; no ageing; blow NH ₃ and air through until fabric pH is 7.	1.8
12.....	Treat with no chemicals and do not semi-decate; blow in SO ₂ at 2 liters/min. for 3 minutes; close and let age 10 minutes; air for 5 minutes; blow NH ₃ through fabric until pH is 7.	3.4
13.....	Treat with no chemicals and do not semi-decate; blow in NH ₃ for 3 minutes; close and let age for 5 minutes; blow with air for 3 minutes; blow in SO ₂ until fabric pH is 7.	4.2

EXAMPLE IX

The fabric of Example I is treated as in Example VIII under varying conditions of pressure of the package dye machine and with the fabric pre-heated to various temperatures. The conditions are set forth in Table IX, the pH of the fabric after such treatments, the reducing power, various physical properties and crease performances, both with and without added moisture, being shown in Table X. The wet crease ratings are obtained by wetting

the fabric to 40% moisture prior to pressing on the Hoffman press.

TABLE IX

Treat- ment	Description
G.....	Fabric treated with 6% monoisopropanolamine, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 81° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 100° F. Pressure at start 54 mm.
H.....	Fabric treated with 6% monoisopropanolamine, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 152° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 182° F. Pressure at start 190 mm.
I.....	Fabric treated with 6% monoisopropanolamine, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 202° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 214° F. Pressure at start 70 mm.
J.....	Fabric treated with 6% monoisopropanolamine, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 80° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 99° F. Pressure at start 60 mm.
K.....	Fabric treated with 6% monoisopropanolamine, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 137° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 175° F. Pressure at start 62 mm.
L.....	Fabric treated with 6% monoisopropanolamine, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 122° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 156° F. Pressure at start 58 mm.
M.....	Fabric treated with 6% monoisopropanolamine, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 139° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 183° F. Pressure at start 60 mm.
N.....	Fabric treated with 6% monoisopropanolamine, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 142° F. inside the fabric and SO ₂ is added. After SO ₂ is added, the temperature rises to 160° F. Pressure at start 60 mm.
O.....	Fabric treated with 6% monoisopropanolamine, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 137° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 157° F. Pressure at start 365 mm.
P.....	Fabric treated with 6% monoisopropanolamine, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 138° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 167° F. Pressure at start 365 mm.
Q.....	Fabric treated with 6% monoisopropanolamine and 5% ethylene glycol, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 146° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 168° F. Pressure at start 365 mm.
R.....	Fabric treated with 6% monoisopropanolamine and 5% ethylene glycol, padded to 100% wet pick-up, dried at 200° F. Temperature is brought to 150° F. inside the fabric and SO ₂ is added. After SO ₂ is added, temperature rises to 174° F. Pressure at start 365 mm.

EXAMPLE X

The fabric of Example I is semi-decated, rolled onto the perforated spindle of a package dye machine and sealed as in Example VIII. The temperature of the fabric is 77° F. when anhydrous ammonia gas is admitted to the machine. After the temperature rises to 105° F., air is forced through the machine for 10 minutes to flush out the ammonia gas, after which the temperature is 66° F. With the machine under a pressure of 50 mm. Hg, SO₂ gas is admitted to the machine and continued until the temperatures reaches 96° F. The machine is then flushed with air as before, the spindle is removed, and the fabric pH and crease performance are measured. The pH is 4.9, dry crease is 2.6, and wet crease is 4.7.

EXAMPLE XI

The procedure of Example X is repeated except that SO₂ gas is added initially, followed by anhydrous ammonia gas. The temperature of the fabric is 77° F. prior to addition of SO₂ and 92° F. when SO₂ admission is discontinued. The temperature of the fabric is 77° F. and pressure is 57 mm. prior to the addition of ammonia. When a temperature of 115° F. is reached, the reaction is discontinued and the spindle, after flushing, is removed from the machine. The fabric pH is 5.9, dry crease is 3.2 and wet crease is 5.0.

EXAMPLE XII

The fabric of Example I is padded with various pad baths to 80% pick-up finished and exposed to an activator gas as set forth in Example I. Table XI set forth the properties of fabrics so treated.

TABLE XI

Percent composition of pad-bath	pH of pad- bath	Gas treat- ment	pH before gas treat.	pH after gas treat.	Dry crease after 1 hour	Dry crease after 24 hours	Percent SO ₂
H ₂ O.....	3.0	SO ₂	-----	4.8	1.8	1.9	0.728
6.0 MIPA.....	3.0	SO ₂	5.4	4.6	2.2	1.9	0.317
8.2 hexylamine.....	3.0	SO ₂	5.0	4.4	2.3	2.3	1.96
4.9 NH ₄ OH (28%).....	7.0	SO ₂	4.8	4.4	2.1	2.1	0.912
6.0 MIPA.....	7.0	SO ₂	5.9	4.7	1.8	2.3	1.338
8.2 hexylamine.....	7.0	SO ₂	5.5	4.6	2.1	1.8	1.221
4.9 NH ₄ OH (28%).....	10.0	SO ₂	4.9	4.4	2.2	2.0	0.803
6.0 MIPA.....	10.0	SO ₂	8.8	8.3	2.2	2.6	0.912
8.2 hexylamine.....	10.0	SO ₂	9.4	6.0	2.7	2.5	2.423
Untreated control.....		None	4.3	-----	1.4	1.8	-----
5.3 strontium hydroxide 8H ₂ O.....	12.3	SO ₂	9.2	6.0	2.3	2.8	.21
1.6 sodium hydroxide.....	12.1	SO ₂	9.0	5.8	3.6	4.2	1.96
3.3 formalin (37%).....	4.3	SO ₂	7.5	3.0	2.4	2.1	.27
7.8 glyoxal (30%).....	4.5	SO ₂	6.9	3.2	1.8	2.1	.43
4.9 NH ₄ OH (28%).....	10.9	SO ₂	7.2	3.1	2.1	2.5	.30
8.2 hexylamine.....	11.2	SO ₂	9.6	6.0	4.4	4.5	1.13
8.8 aniline sulfate.....	4.5	SO ₂	4.4	4.2	1.7	1.8	.38
1.6 NaOH.....	11.9	H ₂ S	10.4	10.1	2.4	3.3	.29
6.0 MIPA.....	11.0	H ₂ S	9.0	8.9	2.6	3.4	.29
Water.....	7.5	H ₂ S	7.4	5.4	1.3	1.8	.09
6.0 MIPA.....	11.0	SO ₂	9.0	6.7	3.6	3.9	.87

TABLE X.-PARTIAL GAS PROCESS

Treat- ment	Instron						Creases	
	Grab tensile							
	pH	Percent SO ₂	Tongue tear (gm.)	Strength (lb.)	Elong- ation (percent)	Dry.	Wet	
Control.....			1,926	65.9	47.0	1.1	2.3	
G.....	7.2	1.68	1,874	77.5	60.5	3.6	5.0	
H.....	5.2	2.23	1,838	70.2	45.8	3.5	5.0	
I.....	4.6	1.68	1,757	66.1	55.0	3.1	5.0	
J.....	7.1	1.16	1,957	70.1	45.4	2.5	4.9	
K.....	7.0	2.24	1,844	75.4	50.7	2.9	4.8	
L.....	7.1	1.13	1,913	78.0	54.3	3.8	5.0	
M.....	7.5	1.10	1,868	78.3	52.9	3.6	4.9	
N.....	7.5	1.78	1,825	70.2	53.2	3.9	5.0	
O.....	7.9	1.67	1,803	64.5	48.1	3.8	4.9	
P.....	5.6	2.75	1,752	69.6	54.2	3.7	4.6	
Q.....	6.1	2.85	1,719	66.1	55.0	3.6	5.0	
R.....	5.3	2.50	1,688	67.7	62.8	3.2	4.6	

EXAMPLE XIII

The fabric of Example I is padded to 82.0% wet pick-up with a solution containing 6.0% monoisopropanolamine, 5.0% ethylene glycol and 0.1% Syn-Fac 905. After padding, this fabric is dried at 200-225° F. to about 10% moisture regain, semi-decated at 1.25 minutes steam followed by 3 minutes vacuum pumping. After semi-decating, the fabric is cut into strips 11½ inches wide and 40 yards long. Each fabric sample is individually mechanically wound onto the spindle of a gas treating machine, and an attempt is made to manually hold the fabric tension close to the same from one roll to the next. Actual gas treatment is accomplished by various procedures as set forth below.

(1) Pre-treated fabric (1869 gms.) is placed in the

machine and heated to 150° F. Sulfur dioxide gas is introduced in an amount (51 gms.) sufficient to titrate the amine in the fabric plus sufficient excess to occupy the void spaces in the fabric and in the machine. This gaseous material is circulated for 15 minutes and then ammonia gas is introduced in amount (7 gms.) sufficient to titrate the excess sulfur dioxide in the system. Ammonia is circulated for 5 minutes, the experimental treatment is stopped, and the fabric is removed from the machine.

(2) Pre-treated fabric (1869 gms.) is placed in the machine and heated to 83° F. Sulfur dioxide gas and ammonia are introduced in the same sequence and amounts as in (1). A very slight amount of "snow" (believed to be ammonium sulfite and/or bisulfite) is noted along the fringes of the fabric.

(3) Pre-treated fabric (1823 gms.) is placed in the machine and heated to 135° F. Sulfur dioxide gas is introduced into the system in an amount (41.4 gms.) sufficient to titrate the amine in the fabric plus an amount sufficient to occupy the void spaces in the fabric. The gases are circulated for 10 minutes and an amount (6.3 gms.) of ammonia gas is introduced sufficient to titrate the excess sulfur dioxide in the system. After an additional circulation time of 10 minutes, the top is removed and the fabric blown with air for 5 minutes.

(4) Pre-treated fabric (1908 gms.) is placed in the machine and sulfur dioxide gas is introduced into the system in an amount (43.0 gms.) sufficient to titrate the amine in the fabric and occupy the void spaces in the fabric. After circulating for 10 minutes, ammonia gas is introduced in an amount (6.6 gms.) sufficient to titrate the excess sulfur dioxide. The gases are circulated for 5 minutes and the system and fabric blown with air for 5 minutes.

No heat is applied during this experiment. The heat of compression of the air plus the heat of reaction in the acid-base titrations involved raises the temperature of the fabric to 102° F. during the course of the experiment.

(5) Pre-treated fabric (1886 gms.) is placed in the machine and sulfur dioxide gas is introduced into the system in an amount (42.5 gms.) sufficient to titrate the amine in the fabric and occupy the void spaces in the fabric. This gas is circulated for 5 minutes after which ammonia gas is introduced in an amount (6.5 gms.) sufficient to titrate the excess sulfur dioxide. At this point, the fabric temperature is about 100° F. The fabric is then heated to 140° F. by means of the electric heating system connected to the machine. After this temperature is reached, the fabric is blown with air for 5 minutes.

(6) Pre-treated fabric (1791 gms.) is placed in the machine and heated to 134° F. Sulfur dioxide gas is introduced into the system in an amount (40.5 gms.) sufficient to titrate the amine in the fabric and occupy the void spaces in the fabric. After circulation of this gas for 5 minutes, ammonia gas is introduced in an amount (6.2 gms.) sufficient to titrate the excess sulfur dioxide. After an additional 5 minutes circulation, the system is opened and the fabric blown with air for 5 minutes.

(7) Pre-treated fabric (1920 gms.) is placed in the machine and heated to 140° F. Sulfur dioxide gas is introduced in an amount (38.4 gms.) sufficient to titrate the amine in the fabric and occupy the void spaces in the fabric. This gas is circulated for 5 minutes, after which ammonia gas is introduced in an amount (6.4 gms.) sufficient to titrate the excess sulfur dioxide. After circulating an additional 5 minutes, the system is opened and the fabric blown with air for 5 minutes.

(8) Pre-treated fabric (1874 gms.) is placed in the machine and heated to 145° F. Sulfur dioxide gas is introduced in an amount (37.0 gms.) sufficient to titrate the amine in the fabric. The gases are circulated for 5 minutes and ammonia gas is introduced in an amount (14.0 gms.) sufficient to titrate all the sulfur dioxide previously admitted to the system. The gases are circulated for an addi-

tional 5 minutes and the system is opened and the fabric blown with air for 5 minutes.

(9) Pre-treated fabric (1982 gms.) is placed in the machine and heated to 145° F. Sulfur dioxide gas is introduced in an amount (39.5 gms.) sufficient to titrate the amine in the fabric. After circulating this gas for 5 minutes, ammonia gas is introduced in an amount (10.5 gms.) corresponding to half of the amount necessary to titrate the sulfur dioxide previously admitted to the system. These gases are circulated for 5 minutes and the system opened and blown with air for 5 minutes.

(10) Pre-treated fabric (1883 gms.) is placed in the machine and sulfur dioxide gas is introduced in an amount (38.0 gms.) sufficient to titrate the amine in the fabric. After circulating this gas for 5 minutes, ammonia gas is introduced in an amount (3.3 gms.) only sufficient to occupy the void spaces in the fabric. These gases are circulated for 5 minutes, and then the system is opened and blown with air for 5 minutes.

(11) Pre-treated fabric (2021 gms.) is placed in the machine and heated to 136° F. Sulfur dioxide gas is admitted to the system in an amount (40.0 gms.) sufficient to react with the amine in the fabric. This gas is circulated for 5 minutes, after which ammonia gas is introduced in an amount (3.6 gms.) sufficient to occupy the void spaces in the fabric. After 5 minutes additional circulation, the system is opened and the fabric blown with air for 5 minutes.

Data are shown in Table XII.

TABLE XII

Experiment No.	Dry crease rating, 170° F. test		pH of fabric after treatment
	1 day	5 days	
1.....	2.7	3.3	6.6
2.....	2.4	3.0	6.8
3.....	3.9	3.1	6.5
4.....	1.9	2.3	6.8
5.....	1.9	2.9	6.7
6.....	2.1	2.3	6.4
7.....	2.8	2.4	6.2
8.....	3.0	3.3	6.5
9.....	2.6	2.5	6.5
10.....	2.3	2.8	6.5
11.....	1.9	2.4	6.9

EXAMPLE XIV

The fabric of Example I is treated with 4.50% mono-isopropanolamine, 0.10% Syn-Fac 905 and 7.20% urea, based on the weight of fabric.

The fabric is dried at 200–225° F. and semi-decated on a Hoffman press at 15 seconds steam followed by 60 seconds vacuum while contained between two pieces of decater fabric.

This fabric, weighing 1482 grams, is then rolled onto the beam of a laboratory gas treating machine, placed in the machine and heated to 140° F. Ammonia gas (2.6 grams) is added to the system and circulated for six minutes. The excess ammonia is vented to the outside. Sulfur dioxide gas (51.4 grams) is then added to the system and, after complete addition, is circulated for six minutes. The excess sulfur dioxide gas is vented to the outside.

Additional ammonia (1.3 grams) is then added to the system and circulated for six minutes. A final venting and exhausting is conducted for 10 minutes. The fabric is removed from the machine and tested.

The initial dry crease rating is 3.5. This rating after aging at room conditions for one (1) week, increased to 4.3.

That which is claimed is:

1. A novel process for presensitizing keratin fibers for subsequent durable setting comprising

(1) contacting keratin fibers with a reducing agent precursor;

(2) subjecting said fibers to any desired mechanical finishing operation; and

(3) then exposing said fibers to a gaseous reducing

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agent activator chemical which can react with the precursor to form a different chemical compound which is a reducing agent for the keratin fibers whereby said fibers are presensitized for subsequent durable setting.

2. The process of claim 1 wherein the reducing agent activator is in a gaseous state.

3. The process of claim 2 wherein the keratin fibers are contacted by the reducing agent precursor prior to the reducing agent activator.

4. The process of claim 3 wherein the reducing agent precursor has a pH greater than 7 as a 1% solution in water.

5. The process of claim 4 wherein the reducing agent activator comprises a reducing agent.

6. The process of claim 5 wherein the reducing agent activator comprises SO_2 .

7. The process of claim 2 wherein the precursor is in a gaseous state.

8. A novel process for presensitizing fabrics containing keratin fibers for subsequent durable setting while providing a desirable finish on said fabric comprising

(1) impregnating said fabric with a reducing agent precursor;

(2) drying said fabric;

(3) imparting a desirable finish to said fabric by subjecting the fabric to heat and pressure; and

(4) then exposing said fabric to a gaseous reducing agent activator chemical which can react with the precursor to form a different chemical compound which is a reducing agent for the keratin fibers whereby a desirable finish on the fabric is retained and whereby said fabric becomes presensitized for subsequent durable setting.

9. The process of claim 8 wherein the fabric is dried under relaxed conditions after impregnation.

10. The process of claim 9 wherein the fabric is impregnated with an aqueous solution of the reducing agent precursor.

11. The process of claim 10 wherein the reducing agent precursor has a pH greater than about 7 as a 1% solution in water.

12. The process of claim 11 wherein the reducing agent activator comprises SO_2 .

13. The process of claim 12 wherein the reducing agent precursor comprises an amine.

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14. The process of claim 13 wherein the amine comprises an alkanolamine.

15. The process of claim 9 wherein the desirable finish is imparted to the fabric by a steaming operation.

16. The process of claim 9 wherein the reducing agent activator is present in an amount at least substantially equivalent to the reducing agent precursor.

17. The process of claim 10 wherein ammonia gas is applied to the fabric after finishing.

18. The process of claim 17 wherein sulfur dioxide gas is applied to the fabric after finishing.

19. The process of claim 18 wherein at least a portion of the ammonia gas is applied to the fabric after treatment with sulfur dioxide gas.

20. The process of claim 17 wherein the reducing agent precursor solution contains an alkanolamine.

21. The process of claim 20 wherein the precursor comprises ammonia gas and the activator comprises SO_2 .

22. The process of claim 21 wherein the reducing agent precursor comprises ammonia.

23. The process of claim 22 wherein the reducing agent activator comprises sulfur dioxide.

24. The process of claim 22 wherein at least a portion of the ammonia is applied to the fabric after treatment thereof with sulfur dioxide.

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NORMAN G. TORCHIN, *Primary Examiner.*

J. CANNON, *Assistant Examiner.*

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

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Earl Peters et al.

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 3, line 3, after "aminoethyl" insert -- piperazine --; line 75, "compounds" should read -- compound --. Column 4, line 38, "highely" should read -- highly --. Column 7, line 25, "sulfric" should read -- sulfuric --; line 37, after "fabric" insert -- for --. Column 9, line 19, "inch" should read -- inches --. Column 11, line 50, "Creases" should read -- Crease --. Column 14, Table XI, the last 7 numbers in the last column under Percent SO₂ should read -- .30 --, --1.13 --, -- .38 --, -- .29 --, -- .29 --, -- .07 --, and -- .83 --. Column 16, line 16, "mania" should read -- monia --.

Signed and sealed this 28th day of April 1970.

(SEAL)

Attest:

Edward M. Fletcher, Jr.

Attesting Officer

WILLIAM E. SCHUYLER, JR.

Commissioner of Patents