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Cashen et al.

[11] **3,872,693**

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[54] **LABORATORY APPARATUS FOR TREATING COTTON AND COTTON-BLEND TEXTILES WITH ORGANIC SOLVENT VAPORS**

[75] Inventors: **Norton A. Cashen,** Metairie; **Austin C. F. Mason,** New Orleans, both of La.

[73] Assignee: **The United States of America as represented by the Secretary of Agriculture,** Washington, D.C.

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[52] **U.S. Cl.** ..... **68/6,** 68/15, 68/18 C

[51] **Int. Cl.** **D06c 1/04**

[58] **Field of Search** ..... 68/5 R, 5 C, 15, 18 R, 68/18 C, 6

[56] **References Cited**

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2,359,736	10/1944	Kienle et al.	68/15 X
2,405,167	8/1946	Royer et al.	68/15 X
2,593,640	4/1952	Whittington	68/18 R X
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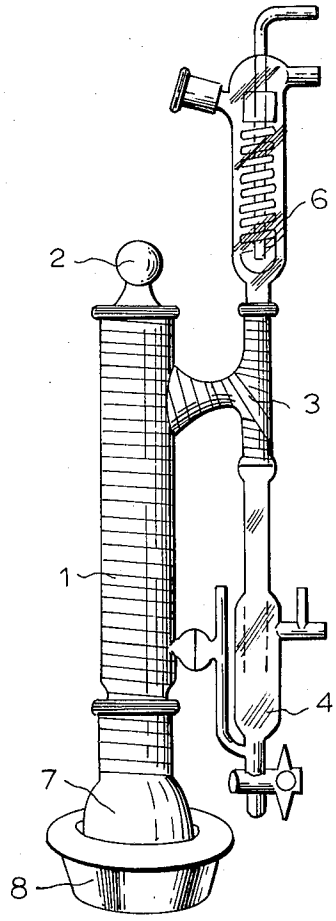
3,801,274	4/1974	Gleason	68/18 C X

Primary Examiner—Harvey C. Hornsby
Assistant Examiner—Philip R. Coe
Attorney, Agent, or Firm—M. Howard Silverstein; Max D. Hensley

[57] **ABSTRACT**

The present invention relates to a laboratory scale apparatus for treating fabrics from an organic solvent vapor, e.g. durable press finishing, and exhaustion dyeing, which apparatus comprises: a vessel, such as a distillation flask which is means for containing an organic solvent, in contact communication with a heating source, such as an electrical mantle for bringing the solvent to the boil, a chamber in the form of a column for containment of solvent vapors to exchange heat and solvent vapors into the fabric that is held extended in a tubular configuration by a stainless steel wire fabric holder; a means for exchange vapors (azeotropic) to condense and return the organic vapor condensate to distillation flask after water has been purged from the system by a solvent separator.

1 Claim, 4 Drawing Figures



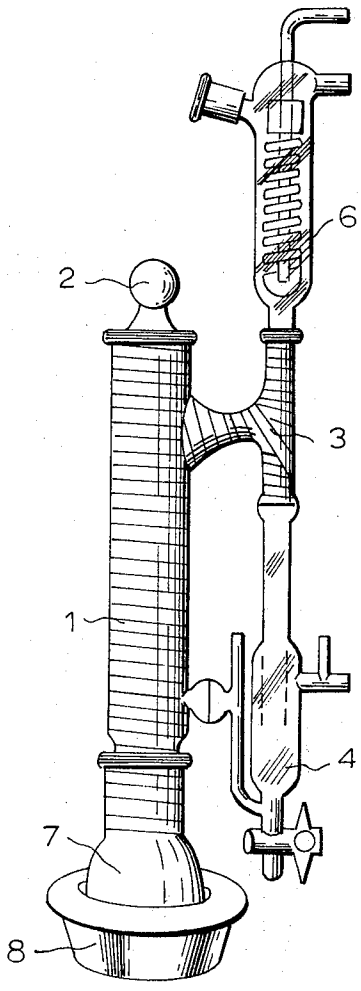


FIG.1

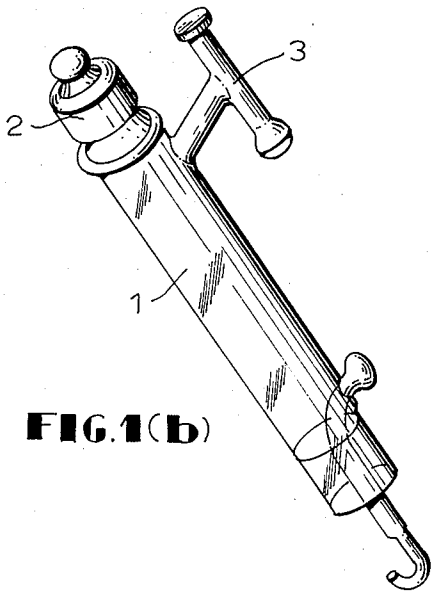


FIG.1(b)

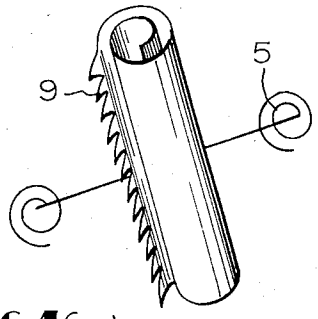


FIG.1(a)

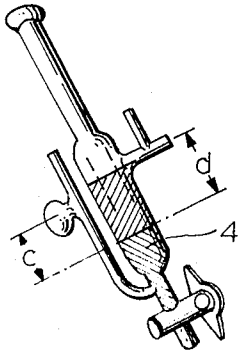


FIG.1(c)

LABORATORY APPARATUS FOR TREATING COTTON AND COTTON-BLEND TEXTILES WITH ORGANIC SOLVENT VAPORS

This invention relates to a laboratory apparatus which is useful in facilitating research with cotton and cotton-blend textiles. More specifically this invention is employed in the durable press chemical finishing by heating a cotton textile fabric that has been impregnated with an aqueous solution of the crosslinker and catalyst, or with an aqueous solution of a pre-polymer and catalyst, with a boiling organic solvent vapor, the temperature of which serves to promote the cure, and the vapor of which serves to simultaneously and azeotropically remove the water from the fabric. A desired cure temperature is achieved by an appropriate choice of solvent with a suitable boiling point, and a controlled degree of moisture may be reached by distillation rate considerations.

The main object of this invention is to provide a laboratory apparatus for facilitating durable press finishing of cotton fibers (in the form of fabric) on a research basis employing hot organic vapors as curing medium.

Another object of this invention is to provide a means of azeotropically removing water or other fraction from a reaction wherein the water or other solvent produces an azeotrope with the distillation solvent for the purpose of effecting a textile chemical finish.

A further object of this invention is to provide a means, on a laboratory scale, of azeotropically displacing a dye-solvent from the textile fiber with a dye-insoluble solvent, thus effecting an exhaustion type dyeing system with the textile fiber retained in a wet and swollen state.

A fourth object of this invention is to provide a means, on a laboratory scale, of water-drying textile fibers in a yarn or fabric form, by azeotropic distillation, to a predetermined and desired water content.

BACKGROUND AND PRIOR ART OF THIS INVENTION

D. M. Jones, S. A. Allen, A. J. Hall, and N. A. Cashen, recently published, individually, results that suggest promise for durable press chemical finishing systems that employ the vapors of boiling organic solvents as curing media. These publications were respectively in Colourage, XII, No. 24, page 31 (1970); International Dyer and Textile Printer, 146, No. 8, page 441 (1971); Textile Manufacturer, 98; No. 1162, page 39 (1971); Proceedings of Textile Solvent Technology—Update '73, A.A.T.C.C., page 79 (1973).

At this writing there has been found a paucity of literature pertinent to laboratory scale apparatus suitable for the treatment of fabrics with organic solvent vapors in particular.

The literature does reveal, however, certain equipment for treatments on a continuous basis, which, of course, are designed for pilot scale operations. These revelations were in the form of: U.S. Pat. No. 3,593,543; publication, Textile Chemist and Colorist, 4, No. 2, page 39 (1973). The instant invention although specifically designed for the needs of laboratory scale research activities could conceivably be projected to at least pilot-scale proportions wherein the basic principles utilized here could be slightly altered to accomodate the larger scale needs.

H. R. Byland, et al. in Textile Chemist and Colorist, 3, No. 10, page 33 (1971), describes a laboratory scale solvent steamer which utilizes superheated solvent vapor for the fixation of disperse dyes on 100 percent man-made fibers. The disadvantage noted in Bylands steamer is that of lack of compactness, and the apparatus facilitates only dyeing studies that are related to superheated solvent vapor disperse dyeing of synthetic fibers.

The apparatus of this invention has been utilized and found suitable in laboratory-scale finishing of cotton and blend fabrics in various organic solvent vapors to impart to the treated fabrics durable press properties.

To illustrate the apparatus which is the invention a Figure is provided and a specific illustration of how the apparatus has been employed with success is also provided.

FIG. **1** is an elevation view wherein exploded portions are also provided to illustrate pertinent paraphernalia.

FIG. **1**(*a*) is a schematic view of a fabric sample and holder.

FIG. **1**(*b*) is a perspective view of the vapor-curing chamber, showing details.

FIG. **1**(*c*) is a schematic view of the solvent separator chamber, showing details.

In the figures it is to be noted that heating mantle **8** supports and provides heat to distillation flask **7**, which in turn supports the columnar reaction chamber **1**, through which pass vapors of boiling solvent contained in distillation flask **7**. The columnar reaction chamber **1** can be about from 10 inches in height to as much as about 5 feet, for laboratory scale investigations, the size being compatible with the particular investigation. This reaction chamber **1** and lateral side arm **3** are generally wrapped with 1-inch asbestos tape or glass fabric tape to insure uniform heat and vapor dispersion.

Fabric sample **9** is generally hemmed at two opposing ends and inserted on a coiled wire hanger **5**, which for most chemical reactions should be made of stainless steel. The fabric sample and hanger are then inserted into the reaction chamber **1**. This arrangement reneders a maximum fabric surface area exposable to the solvent vapors while maintaining a smoothness for better reaction. The solvent vapors would, of course, be emanating from the direction of the distillation flask **7** as well as returning from the side arm **3**. These vapors, mixed azeotropically with the pad solvent of the fabric, should move from the distillation flask **7** upward, vertically and through the cylindrical axial configuration of the fabric sample, thence to return by the side arm **3**, wherein the vapors are cooled by the influence of the cold finger condenser **6** and returned to the distillation flask **7** through automatic solvent separator **4**. The vapors would generally contain water removed from the fabric to form an azeotropic mixture, which upon sufficient cooling, separates as imissible layers; i.e., when a chlorinated hydrocarbon solvent is boiled in the distillation flask, and the fabric pad solvent is water, the solvent of lower specific gravity, namely water, disperses to the top layer, thence is purged out of the system, and the solvent of higher specific gravity, namely chlorinated hydrocarbon solvent, disperses to the bottom, and is then returned to the distillation flask **7**, for recycling. The automatic solvent separator **4**, being a crucial component part of the apparatus, is attached to the laterial arm **3** for direct return of the condensate to the distillation flask, as is required for studies involving total reflux. For laboratory scale investigations it is ad-

visable that the entire apparatus be made of borosilicate glass and having Standard Taper ground glass fittings. The automatic solvent separator **4** of the preferred embodiment of this apparatus was one wherein glass ball and socket joints were most suitable for efficient removal of that portion of the apparatus.

Of course, there are various other essential parts which complete the picture of this apparatus, such as the ground glass stopper **2,** and the heating mantle **8,** as well as the means of energizing the mantle and cooling the cold finger condenser **6.** Also, note that distance *d* is greater than *c*.

ADVANTAGES OF USING THE APPARATUS WHICH IS THE INVENTION

1. Facilitates absolute and finite control of fabric cure temperature.

2. Facilitates instantaneous matching of fabric temperature with that of solvent vapor which is the curing medium: no uncertainties and irregularities caused by air circulation as in convection ovens.

3. Facilitates a highly efficient curing system: 12–73 percent greater than that of equivalent oven treatments and 34 percent greater than that of boiling liquid solvent treatments.

4. Facilitates very rapid removal of water in textile fabric by azeotropic distillation: efficiency of this type of system requires maximum fabric surface area (back and front) exposure to solvent vapors.

5. Facilitates very close control of fabric water content during cure.

6. Facilitates complete removal of fabric water, rendering a means of studying various exhaustion type dyeing techniques.

7. Facilitates extremely rapid curing of textile fabrics for durable press finishing: as low as 30 seconds with n-acyl acetate as curing solvent vapor.

The following examples are provided to illustrate the use of the vapor-treating apparatus.

A sample of 3.2 ounce per sq. yd. cotton printcloth was impregnated with an aqueous solution containing 15 percent dimethylolmethylcarbamate, and 2 percent aluminum chloride hexahydrate as catalyst (solids basis). The fabric was then dried in a forced draft oven at 60°C to 4–5 percent water. After this the sample was placed in the vacuum-curing chamber and exposed to vapors of the perchloroethylene containined in the round bottom distillation flask at boil (121°C) for one minute. Washing and tumble drying of the fabric followed.

Another sample, specifically 8 ounce per sq. yd. cotton twill, was impregnated with an aqueous solution containing dimethyloldihydroxyethyleneurea and a melamine, 14 percent solids each. The impregnated fabric was then dried at 60°C in a forced draft oven to approximately 28 percent residual water. Utilizing the described vapor-treated apparatus of the invention, the fabric was exposed to boiling trichloroethylene vapors (87°C) for 5-minutes, after which time approximately 11 percent water remained at completion of the fixation. The sample was then washed and dried, and 12 percent solids add-on was observed. A re-impregnation (pad) was then necessary; sample was padded with a solution containing 0.6 percent zinc nitrate hexahydrate, and 3 percent of a polyurethane. After drying, steam pressing, and oven-curing for 5 minutes at 160°C an excellent smooth-drying and durable creasing finish was obtained.

Physical Properties Obtained in Chemical Finishing of Above Fabrics, Using the Apparatus of this Invention

Cotton Fabric	Break.Str. Retained,%	W.R.A. Cond.	(W+F) Wet	Moisture Regain,%	Durable Press Rating, T.D.
3.2 oz. Printcloth	50	275	308	4.9	4.7
8.0 oz. Twill	85	285	287	6.3	4.0

One of the crucial components of the apparatus which is the invention is the automatic solvent separator. Although modifications can be made on the various components which comprise the apparatus of this invention the component referred to as the automatic solvent separator can best be described as . . . . "an automatic solvent separator which adapts to the side arms of the vapor-curing chamber at the ball and socket joints, said automatic solvent separator comprising a solvent-separator chamber which received the condensed water and other solvent to form a two-layer system of solvents therein; said solvent separator chamber extending downward to form the right arm of a Y, the stem of which Y being provided with a stopcock, the left arm of which extends upward to end in an open syphon-breaker aperture, said left arm having a socket joint which recycles the desired solvent back into the said vapor-curing chamber; said solvent separator chamber having a high, small-diameter tube extending perpendicularly to the right, to drain-off the unwanted water; said small diameter tube also having an upward extension which also serves as a syphon-breaker; the said socket joint extending from the left arm of said Y being lower than the axis of the said small diameter tube which extends to the right."

We claim:

1. A laboratory apparatus for treating fabric samples with vapors, the apparatus comprising:

a. a vapor-curing chamber comprising a cylindrical tube having top and bottom apertures, the top aperture being a standard female joint of dimensions which permit the passage of a fabric and holder, the bottom aperture being a standard male joint of dimensions compatible with those of a round bottomed flask onto which it would fit; the vapor-curing chamber having an upper and a lower side arm of about one-fourth the diameter of the said vapor-curing chamber, both arms being extended at right angles to the chamber; the said upper side arm extending a short distance thereafter to form a T, said T being tubular and open at each end, to describe an upper and a lower aperture of the crosspiece of the said T; the said upper aperture being a female standard taper joint to fit thereinto a cold-finger type condenser, the said lower aperture being a ball joint extending downward; the said lower side-arm extending a shorter distance outward than the distance of the upper side-arm and terminating outward as a socket joint, and the other end routing through the vapor chamber downward, to allow the distillate to return;

b. a ground glass standard taper stopper to close the upper aperture of the vapor-curing chamber when apparatus is in operation;

c. a cold-finger type condenser to fit onto the upper arm of the upper side arm;

d. an automatic solvent separator which adapts to the side arms of the vapor-curing chamber at the ball and socket joints, said automatic solvent separator comprising a solvent-separator chamber which received the condensed water and other solvent to form a two-layer system of solvents therein; said solvent separator chamber extending downward to form the right arm of a Y, the stem of which Y being provided with a stopcock, the left arm of which extends upward to end in an open syphon-breaker aperture, said left arm having a socket joint which recycles the desired solvent back into the said vapor-curing chamber; said solvent separator chamber having a high, small-diameter tube extending perpendicularly to the right, to drain off the unwanted water; said small diameter tube also having an upward extension which also serves as a syphon-breaker; the said socket joint extending from the left arm of said Y being lower than the axis of the said small diameter tube which extends to the right;

e. clamps to secure said socket joints;

f. a rigid, stainless steel, fabric sample holder, said holder comprising a vertical straight shaft terminating at each end in a coiled configuration, the plannar configuration of each coil being perpendicular to the said vertical straight shaft; said rigid, stainless steel, fabric sample holder being of a dimension compatible to the inner dimensions of the said vapor-curing chamber of (a) and comprising a means of supporting a fabric sample hung thereonto;

g. a round-bottomed flask with ground glass standard taper joint compatible with the bottom aperture of the said vapor-curing chamber of (a) and being of a volumetric capacity compatible with the requirements of the entire apparatus; and

h. a heating mantle, a source of energy to actuate the mantle, an ice-cold source of water for the cold-finger condenser, supporting brackets and clamps to provide the apparatus with adequate rigidity.

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