The Retention and Removal Behavior of Perchloroethylene and Dichloromethane in Polyester Fabric

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Synopsis

Polyester fabric was exposed to solvent at different bath temperatures. The retention of solvent by the fabric was determined. The removal of the retained solvent from the fabric was determined as a function of time and also as a function of temperature. The retention of perchloroethylene varied from >9% at 121°C to <0.01% at room temperature. The removal of perchloroethylene from the fabric was also a function of temperature, but even at 120°C significant quantities of perchloroethylene remained after 5 min of heating with nitrogen flow across the fabric. Dichloromethane was retained (>5%) at its boiling point and had similar slow removal rates as were found for perchloroethylene. Retention and removal rate data for both solvents as a function of time and temperature are presented.

INTRODUCTION

The use of organic solvents in textile dyeing and finishing has been explored by industry and independent researchers extensively for the past several years. This research was prompted by the need for an alternative solvent to replace the traditional solvent, water. This interest in solvent processing dates back earlier than 1937 when a Celanese patent¹ referred to the use of organic solvents for continuous dyeing. In other early research, Garret^{2,3} described the use of trichloroethylene vapor to fix disperse dyes on polyester and cellulose acetates. More recently, solvent processing has been demonstrated to be economical in the removal of oils from knitted fabrics.

As an outgrowth of studies⁴ on dyeing rates from perchloroethylene solvent with polyester fibers, a new study was initiated in an attempt to define the various economic and processing conditions and materials and also the environmental significance of a solvent slashing and desizing operation for cotton/polyester fabrics. One of the obvious questions to be answered was the effect and extent of solvent retention by the polyester fibers. The answers here are important from two basic standpoints; the first is the economics of solvent recovery, and the second is an environmental question of the release of solvent from the fiber after processing has been completed.

Previous reports have indicated that retention of perchloroethylene by polyester fiber may be of concern. Byland et al.⁵ gave a brief indication of the drying conditions of polyester under conditions of superheated perchloroethylene. His reported data showed some information on vapor concentration at various processing conditions over short drying time (seconds). More recently, Brodmann⁶ gave retention data for chlorinated solvents in fabrics under conditions of drycleaning processing. These data again indicate some retention of solvent by polyester. A more in-depth study was performed to provide a better description of the total retentions and rates of loss of chlorinated solvents from polyester fabric. This information was necessary for a thorough economical and environmental evaluation of solvent slashing and desizing processes for fabrics containing polyester fibers.

EXPERIMENTAL

The fabrics used in this study were obtained from Test Fabrics, Inc. The fabrics were 100% spun Dacron T54 polyester, one was heat set, one was without heat set. Solvents used were of reagent grade. Thermal analysis experiments were performed using a du Pont thermogravimetric analyzer.

The fabrics used were exposed to solvent in the following manner in order to determine the retention of solvent as a function of temperature. Fabric pieces which had been previously taken to constant weight were immersed in a solvent bath that was maintained at the desired temperature. The solvent and fabric were stirred at regular intervals. After immersion in the solvent bath for 15 min, the fabric was removed and allowed to dry at room temperature for 10 min. This allowed surface solvent to evaporate. The fabric was then weighed to determine the percent retention of solvent within the fiber. After weighing, the fabric was allowed to expire solvent under ambient room conditions and was reweighed at various time intervals. This experiment therefore gave data of percentage retention versus time. Different bath temperatures were employed, and the procedure above was repeated in order to determine the retention of solvent under these conditions.

A second experiment was devised in order to determine the rate of removal of solvent from the fiber at various drying temperatures and under flow conditions. Small pieces of fabric were heated in the solvent for 15 min at the boiling point of the solvent. This gave a maximum value for initial retention. The fabrics were then allowed to dry as before. The dry samples were then placed in the thermal gravimetric analyzer. The sample was then heated under nitrogen gas flow and at isothermal conditions. The percent weight (solvent) loss of the sample versus time was obtained. The experiment was performed in triplicate at several temperatures.

The two solvents tested were perchloroethylene and dichloromethane.

Retention of Perchloroethylene

In any solvent process the economics will be partially dependent on the ability to recover the solvent efficiently. One possible pathway for solvent loss in finishing of polyester or other synthetic fibers is by retention in the fibers.

The retention of perchloroethylene by two specific fabrics as a function of temperature is shown in Figure 1. These data indicate the temperature-dependent nature of perchloroethylene sorption by polyester. The curves in Figure 1 show that between bath temperatures of 60° and 100°C, there is a direct relationship of retention with temperature. After 100°C these data indicate a saturation level. Obviously, if the processing temperature is maintained below 60°C, there will be little or no retention of solvent. At room temperature (22°C) there is no significant retention of perchloroethylene in the fabric. Therefore, solvent processing at this temperature would cause no solvent loss by retention.



BATH TEMPERATURE (^OC)

Fig. 1. Effect of bath temperature on the retention of perchloroethylene in polyester fabrics after air drying at ambient temperature with (O) and without (\Box) heat set.

Removal of Perchloroethylene

Further experiments were conducted to determine the removal behavior of the solvent from polyester fabric. Data showing the removal of perchloroethylene from the fabric at room temperature as a function of drying time are presented in Figure 2. These data show a gradual reduction of solvent in the fabric. The initial concentrations were those achieved by treating the fabric at different bath temperatures. The rates of loss of solvent versus time at constant temperature are the same and therefore independent of initial retention level in the fabric. Also, these data indicate the slow loss of the retained solvent under ambient drying conditions.

Perchloroethylene Removal Versus Drying Temperature

As shown by the ambient condition data, elevated temperatures will be necessary to remove the solvent from fabric after it has been retained. Polyester fabric was treated at the maximum perchloroethylene bath temperature (121°C) in order to achieve a high level of retention. This fabric was then heated isothermally using a thermogravimetric analyzer as previously described. The isothermal conditions were set at different levels to obtain the rates of removal and removal behavior at these temperatures. Table I shows the rates of removal, in percent per minute, of the perchloroethylene from polyester fabrics. The removal behavior of the perchloroethylene indicated two different curves. An initial, very rapid removal was followed by a much slower removal. This is reflected in the initial and final rate data shown in Table I. As expected, the initial rates of removal of solvent are highly temperature dependent. At 60°C the removal is slow so that one cannot distinguish between the initial and final rates.

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Fig. 2. Effect of perchloroethylene bath temperatures on the retention of perc in polyester fabrics without and with heat-set. Bath temperatures: A-121°C, B-100°C, C-80°C, D-60°C. Fabric immersion time: 15 min.

Retention of Perchloroethylene After Drying

The information shown in Figures 3 and 4 are of more significance to industrial processing than the previously discussed rates of removal. This table gives the retention of solvent after 2.5 and 5.0 min of drying at the indicated temperatures. It is shown that at the lower temperatures most of the retained solvent is still in the fabric after 5 min of drying and that a significant amount of the retained solvent is present after 2.5 min of drying at the higher temperature, 120°C. Therefore, under normal industrial processing conditions retained perchloroethylene would not be efficiently removed.

Thus, the temperature of perchloroethylene solvent in the finishing of poly-

TABLE I ates of Removal of Perchloroethylene from Polyester Fabrics at Various Drying Temperatu								
Temperature	Rate of remova Heat set		I, %/min W/O heat set					
°C	Initial	Final	Initial	Fina				
60		0.03		0.03				
80	1.0	0.03	1.1	0.03				
100	2.4	0.02	2.5	0.03				
120	4.5	0.01	4.1	0.02				



Temperature (°C)

Fig. 3. Weight percentage of perchloroethylene retained by polyester fabric after drying for 2.5 min at various temperatures: with heat set (\Box) ; without heat set (\Box) .

ester fiber or fabric must be maintained low enough to prevent the solvent from entering the fiber and being retained. Low treatment temperatures should therefore be used in sizing processes using perchloroethylene so that complete drying of the yarn or fabric could be accomplished in a reasonable time. At elevated drying temperatures the solvent would be evaporated from the surface before the fabric temperature is raised high enough to allow significant sorption.

With the data described herein, one should be able to calculate any of the retention information needed for any type of solvent finishing of polyester fiber or fabric using perchloroethylene solvent.

Dichloromethane Solvent

The rates of removal of dichloromethane from the two polyester fabrics previously described were determined at drying temperatures ranging from 40° to 120°C. These rate data are shown in Table II. These data again indicate a two-slope behavior, with the initial slope being very temperature dependent.

The retention of dichloromethane after drying intervals of 2.5 and 5.0 min are shown in Table III. These data show that there is significant retention of the solvent even at temperatures well above the solvent boiling point. The use of the low-boiling solvent therefore shows no particular advantage over perchloroethylene as far as its retention behavior is concerned.



Fig. 4. Weight percentage of perchloroethylene retained by polyester fabric after drying for 5.0 min at various temperatures: without heat set (\Box) ; with heat set (O).

SUMMARY

The sorption and retention behavior of solvents by polyester has been shown to be dependent on the solvent bath temperatures and on the drying temperatures. At high bath temperatures, there is a large quantity of solvent absorbed, greater than 9%. At low bath temperatures, for example, room temperature, there is little or no perchloroethylene absorbed. The rates of removal of the solvent from the fiber were higher at higher temperatures and lower at low temperatures. The obvious conclusion is that in order to avoid solvent retention, the solvent must be prevented from entering the fiber. This can be accomplished by using using low bath temperatures (room temperature) and by removing the surface solvent rapidly, preventing penetration of the solvent into the fiber during drying.

 TABLE II

 Rates of Removal of Dichloromethane from Polyester Fabric (Fabric Saturated at 41°C)

	Rate of removal, %/min				
Drying temperature,	Heat set		W/O heat set		
°C	Initial	Final	Initial	Final	
40	0.2	0.026	0.2	0.033	
60	1.2	0.021	0.9	0.025	
80	1.4	0.008	1.2	0.014	
100	2.1	0.001	2.5	0.004	
120	5.6	~0	4.5	~0	

Temperature, °C	Retention, wt-%					
	2.5 min		5.0 min			
	Heat set	W/O heat set	Heat set	W/O heat set		
60	2.9	3.8	2.2	3.0		
80	1.8	2.6	1.1	1.7		
100	0.8	1.3	0.3	0.7		
120	0.2	0.5	0.08	0.12		

 TABLE III

 Retention of Dichloromethane in Polyester Fabric after Drying the Saturated Fabric for 2.5 and 5.0 Minutes at Various Temperatures

Prevention of retention of solvent in the fiber removes the danger of air pollution by expiration of the solvent after the fabric has left the processing facility.

The loss of solvent would have a significant effect on the economics of any process. As can be shown in the economics evaluation of solvent processes, material costs are important. However, from the information on solvent retention presented herein, the loss of solvent through retention can be avoided to such a degree as to make the loss insignificant.

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