Extraction of Cotton Impurities: Supercritical CO_2 vs. Soxhlet/TCE

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ABSTRACT

Determination of residual natural waxes and processing lubricants on yarns and fabrics has traditionally been accomplished by Soxhlet extraction with an organic solvent. A variety of concerns about these solvents has driven a search for alternative methods. Hydrocarbon solvents; e.g., petroleum ether, are flammable and the manufacture of chlorofluorocarbon solvents was phased out in 1996 as a result of their effect on the ozone layer. Purchase and disposal of chlorinated solvents such as 1,1,1-trichloroethane (TCE) are costly. One possible solution to this problem is to use a nontoxic supercritical fluid, carbon dioxide, as a medium for extracting materials from textile substrates. This paper describes development of a supercritical fluid extraction (SFE) method for the quantitative determination of natural cotton wax, paraffin winding wax, and knitting oil from cotton fibers, yarns, and fabrics. Analysis of the extracts by various analytical methods, including differential scanning calorimetry (DSC), Fourier transform infrared (FTIR) spectroscopy, gas chromatography (GC), and gas chromatography/mass spectrometry (GC/MS) techniques are also presented.

KEY TERMS

Analytical Techniques
Cotton
Supercritical Fluid Extraction

Any material above its critical pressure (P_c) and temperature (T_c) is a supercritical fluid. At the critical pressure and temperature only a single unique phase which is neither liquid or gas exists.[1] T_c is the highest temperature at which a gas can be liquefied by increasing pressure and P_c is the highest pressure at which a liquid can be vaporized by increasing the temperature.[2] This phase behavior is illustrated in Fig. 1. Table I contains critical pressures and temperatures for several common materials.[3,4]

Supercritical fluids (SF) exhibit the diffusion properties of a gas and the solvating properties of a liquid. The data in Table II show that the characteristics of a SF are between those of a liquid and a gas.[1] A SF can be used to solvate materials because it has a density similar to a liquid, yet it can diffuse like a gas into matrices containing extractables. Density of a SF can be varied with temperature and pressure. Because of this, one SF can mimic the solvating properties of a range of liquid solvents.

Carbon dioxide is the most widely used SF because of its cost, low T_c and P_c, and low toxicity. It is used on a large commercial scale to decaffeinate coffee and to defat cocoa. Supercritical CO_2 has also been investigated as a fluid for drycleaning[5] and disperse dyeing of polyester.[6] Laboratory investigations utilizing supercritical CO_2 have been simplified by the development of convenient-to-use supercritical fluid extractors. These instruments typically consist of a pump to compress the CO_2, an extraction cell, a restrictor that simultaneously allows compression and passage of the fluid, and a trap to collect or condense the extract. Some extractors allow only dynamic extractions; i.e., continuous flow through the sample, while others allow both dynamic and static extractions. Both types of instruments were used in this work.

Variables that need to be determined to develop a supercritical fluid extraction method include extraction pressure, extraction temperature, flow rate, extraction time, trap temperature, sample size, use of a modifier, and whether extraction should be static, dynamic, or a combination of both. Modifiers are solvents that can be added to the sample before extraction or pressurized with the CO_2. Depending on their characteristics, modifiers can increase the solubility of the extractable materials in the SF or they can interact with the matrix surrounding the extractable to facilitate extraction.

A gravimetric determination is the simplest method for analyzing the effectiveness of an extraction. Two different gravimetric methods can be

Table I. Supercritical Conditions for Various Materials

Solvent	Critical Temperature, C	Critical Pressure, atm.
Carbon Dioxide	31.1	72.8
Propane	96.7	41.9
Isopropanol	235.2	47.0
Ammonia	132.5	111.3
Water	374.2	217.6
Ethanol	241	61

Table II. Properties of Gases, Liquids, and Supercritical Fluids

Phase/Property	Gas	Liquid	Supercritical Fluid
Density, g/cm^3	10^{-3}	1	0.1^{-1}
Diffusion Coefficient, cm^2/s	10^{-1}	$<10^{-5}$	10^{-3}-10^{-4}
Viscosity, g/cm·s	10^{-4}	10^{-2}	10^{-3}-10^{-4}

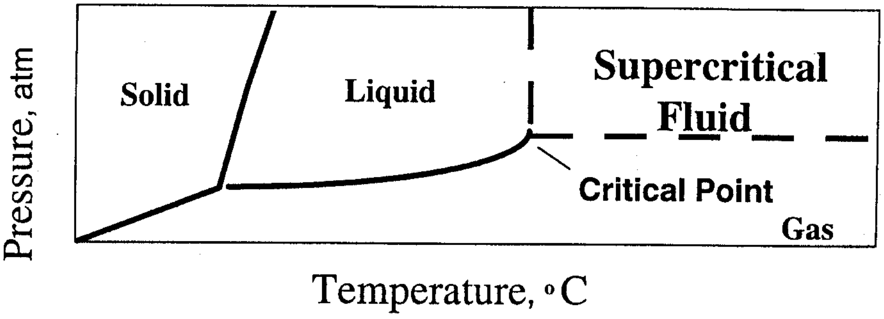

Fig. 1. Phase diagram of a supercritical fluid.[3]

used. Percent weight loss is determined from the weight of the sample before and after extraction. Percent recovery is the weight of the recovered extract relative to the sample weight. The major disadvantage to these methods is that they are not analyte specific. In spite of this limitation, % weight loss and % recovery have been the traditional means for determining the amounts of materials removed from textile substrates by Soxhlet extraction.

Use of supercritical fluid extraction (SFE) for quantitative determination of residues on textile materials continues to increase. The amount of grease on raw wool has been determined by SFE.[7] Taylor et al. have coupled SFE with FTIR for the quantitative on-line determination of a spin finish on nylon and Kevlar.[8,9] Drews et al. have determined the level of spin finish on nylon, polyester, and polypropylene by SFE.[10] Quantitation of finishing oils on spandex by SFE and FTIR has been reported by workers at Isco.[11] Extraction of cotton waxes by SFE has been published in a Dionex application note.[12] This project was undertaken to comprehensively study the supercritical extraction of nonpolar impurities from cotton fibers, yarns, and knit fabrics.

Composition of Cotton Wax

Lewin and Carroll, in an unpublished report to Cotton Inc., used GC/MS to determine the composition of solvent ($HCCl_3$:CH_3OH, 6:1) extracts from cotton.[13] They silylated the extracts to volatilize alcohols and acids. They found many components in the natural wax but concluded that the major ones were high molecular weight esters; e.g., ceryl oleate and montanyl triacontanoate (10-15%), montanol (25%), 1-triacontanol (18%), ß-sitosterol (10%), and $C_{26}H_{52}O$ (13%). Other components were palmitic acid, stearic acid, oleic acid, n-triacontane, and hentriacontane.

Experimental

Materials

Cotton fibers and yarns were obtained from Cotton Inc. Yarns donated by Parkdale Mills were knitted into fabric. The winding wax was described as a paraffin wax and the knitting oil as a mineral oil. The greige knit was scoured and bleached in a Thies sample jet using normal scouring and bleaching formulations.

Soxhlet Extractions

In the past, solvent extraction of cotton wax has been done with benzene, chloroform, carbon tetrachloride, ethanol, isopropyl alcohol, ethanol/benzene, trichloroethylene, and diethyl ether. A comprehensive study of these extractions was carried out by Goldthwait et al.[14] Those authors reported that some cotton wax components decompose as a result of long exposure to hot solvent. The current solvent recommended in AATCC Test Method 87 and the solvent used in the Soxhlet extractions reported here is 1,1,1-trichloroethane (TCE).[15]

Soxhlet extractors (Fisher) were either medium (250-mL) or large (500-mL) capacity. Extractions utilized refluxing TCE (150 or 200 mL) for six hours unless otherwise specified. The flasks and extractors were insulated with aluminum foil. Because new cellulose extraction thimbles were shown to contain extractables, glass thimbles (Fisher) were used for samples that might contain loose fibers. In some large-scale extractions, no thimble was used. The solvent was recovered on a rotary evaporator. The extract was quantitatively transferred from the flat-bottomed Soxhlet flask to a preweighed glass vial with a disposable pipette and TCE. After ambient evaporation of the solvent in a hood, the vials were transferred to a convection oven for drying (30-60 min) at 105C. A glass vial of known dry weight (glass standard) was dried with the sample vials. Weights were determined to 0.01 mg on a Mettler AT201 balance.

Cotton fibers, yarns, and fabrics were initially dried in weighing bottles for 2-72 hours depending on the sample weight. A cotton fiber or greige fabric sample of known dry weight (fiber or fabric standard) was dried with fiber, yarn, or fabric samples to monitor the drying process. Typical drying times for 5- to 10-gram samples were 24 hours. This meant that it took a minimum of two complete days to determine the sample weight before and after extraction. To accelerate the drying process, a six-hour vacuum drying procedure was developed. Cotton samples and a standard in weighing bottles were placed in a vacuum oven (Fisher Scientific Model 280) with lids off. Samples were dried (105C) at full vacuum (29 in. Hg) for three hours. The oven, while under vacuum, was turned off and allowed to come to ambient temperature over three hours. After the vacuum was released, the samples and standard were quickly capped and weighed. Weighing bottles were handled with lint-free gloves. Weight losses (%) and weight recoveries (%) were calculated in the normal manner.

Samples for SFE were dried in the same manner as those for Soxhlet extraction. To ensure ease of handling and uniform packing in the SFE sample cells, fabrics were cut in 0.5" x 2" strips.

Table III. SFE Manufacturers Instrument Specifications

Manufacturer/Specifications	**Hewlett Packard**	**Dionex**	**Isco**	**Suprex**
Model	HP 7680	SFE 723/723M	SFX 1220	PrepMaster w/AccuTrap
Pump, Type	Reciprocating	Reciprocating	Syringe	VariPump-Cont. Flow
Pressure Max., atm	360	680	510	510
Rest. Type	Variable	Fixed	Fixed Capillary	Automated Variable
Rest. Temp Max., C	5-120	250	150	100
Extract. Temp. Max., C	40-120	35-150	150	150
Flow Max., mL/min (comp. CO_2)	0.5-4.0	250, 500[a]	4	5
Extraction Mode(s)	Static, Dynamic	Dynamic	Static, Dynamic	Static, Dynamic
Sample Cell Size, mL	7	10	10	9
Samples per Extraction	1 (8 sequentially)	8	2	2[b]
Modifier Pump, Type	No	Yes	No	Yes
Trap Type	Stainless Steel or Octadecyl Silane	Collection Vial	None	S. Steel or Custom Packed
Trap Temp. Range, C	-30 to 80	Room Temp. or 2-3C	Room Temp.	-50 to 100
Collection	Glass Vial	Glass Vial	Glass Test Tube	Glass Vial
Comp. CO_2 Req.	Dip Tube	Dip Tube	Dip Tube, He Head Space	Dip Tube, He Head Space
Other Req. Gases	None	Air for Pump	None	N_2 and CO_2, industrial grade

[a]Fixed restrictor flow rate in mL/min. [b]One extraction must be in static mode while the other is in dynamic.

Instrumentation

Four different supercritical fluid extractors were used in this work. The manufacturers and some important specifications of each instrument are shown in Table III.

FTIR spectra were collected on a Nicolet 510P. Samples were deposited from solutions as films on silver chloride plates which were used to record background spectra. Differential scanning calorimetric data were obtained using a Perkin Elmer DSC 7. Gas chromatographic analyses were performed on a Hewlett Packard 5890 GC equipped with a split/splitless injector and flame ionization detector. The GC was controlled by a 5895A data station. Separations were effected on a DB5-HT column (J&W Scientific), 15 m, 0.32 mm i.d., 0.25 µm film thickness. This column was designed for high temperature (up to 400C) analyses. GC/MS data were obtained on a Hewlett Packard 5840 GC coupled to a Hewlett Packard 5985B quadrupole mass spectrometer with TRE-IV data system. The same column used for GC analysis was used to collect GC/MS data.

Silylation Procedure

Cotton extracts were silylated prior to GC or GC/MS analysis. Known samples of winding wax, knitting oil, and authentic samples of suspected components of cotton wax were silylated by placing the sample (1-2 mg weighed to 0.01 mg) in a 0.6-mL Reacti-Vial (Pierce Scientific) followed by injection of 400 µL of tetrahydrofuran (THF) and 200 µL of N,O-bis[trimethylsilyl]trifluoroacetamide (BSTFA, Pierce Scientific) containing 1% trimethylchlorosilane (TMCS). The vial was shaken and allowed to stand overnight to complete the silylation. Cotton extracts in glass vials were melted in an oven and dissolved in THF. Volumes of this THF solution were adjusted so that approximately 1 mg of extract was silylated using the above procedure.

Fluidity Measurements

Fluidity measurements were made on cotton samples which had been milled through a 20-mesh screen and stored in a desiccator containing concentrated sulfuric acid as a desiccant. Efflux times were determined at 25 ± 1C on cupriethylene diamine (Synmet Inc.) solutions prepared according to AATCC Test Method 82-1989.

Results and Discussion

Soxhlet Results

Table IV. Soxhlet Recoveries from Fibers, Yarns, and Knitted Fabrics

Source	% Recovery Fiber (n=2)	% Recovery Yarn (n=2)	% Recovery Fabric (n=2)
3.4 Micronaire Fiber	0.82 ± 0.03	0.70 ± 0.00	1.36 ±0.21
4.2 Micronaire Fiber	0.62 ± 0.02	0.64 ± 0.02	1.17 ±0.23
5.0 Micronaire Fiber	0.74 ± 0.14	0.59 ± 0.13	1.21 ±0.16
Greige	0.95 ± 0.02		
Scoured	0.80 ± 0.05		
Bleached	0.48 ± 0.06		

Soxhlet extraction was used to determine the level of extractables on cotton fibers, yarns spun from those fibers, and fabrics knitted from those yarns. Only weight recoveries were determined. Results are shown in Table IV. As expected, the percent recovery increased during the knitting process because of the addition of knitting oil. According to the percent recovery of the greige, scoured, and bleached fabric, which was different from the fabric knitted from the cotton of known fineness, the preparation processes left more than the desirable 0.3% extractables on the fabric. Results of SF extraction of these samples will be discussed later.

SFE Results

To determine the ease of removal of the paraffin winding wax and the knitting oil, known amounts of these components were applied to Soxhlet-extracted bleached cotton samples as 1% (w/w) solutions in dichloromethane. These individual components were quantitatively removed from the cotton fabrics at 353 atm, 75C, in 25 minutes of dynamic extraction. Under these conditions, only 50% (relative to the Soxhlet results) of the cotton wax was extracted from unprocessed fibers.

To expedite the method development, a set of designed experiments was run on duplicate greige knit samples at pressures of 200, 325, 450, 525, 600, and 670 atm at 40, 65, and 90C for 60 min. These samples were dried before and after extraction using the vacuum drying procedure. Analysis of variance (ANOVA) showed temperature to be the most significant variable, followed by the temperature-pressure interaction. Maximum recoveries were obtained at 65C/325 atm, 65C/450 atm, and 40C/325 atm. However, the maximum recovery (0.68%) was still only about 85% of that obtained by Soxhlet (0.80%). SFE values were consistently lower than Soxhlet recoveries, indicating incomplete removal by carbon dioxide.

Table V. SFE Recoveries - Greige Knit: Pure CO_2, Static, and Dynamic Conditions

Extraction Conditions	% Recovery
60-min dynamic	0.90
10-min static, 60-min dynamic	0.60
20-min static, 60-min dynamic	0.65
10-min static, 10-min dynamic	0.74

To determine the effect of static and dynamic conditions, four different extractions were run at 350 atm, 90C at a flow rate of 2 mL/min CO_2. Because this was exploratory work to determine optimum temperature and pressure conditions for extraction of cotton waxes, the samples were not pre- or post-dried. The extractions were carried out at 90C because DSC data had shown a melting range of 38-68C for the winding wax and 37-86C for the cotton wax. Table V shows the results of these experiments. Even though a single 60-min SFE gave a recovery comparable to Soxhlet extraction, the results indicated no advantage to adding a static step to a dynamic extraction.

To determine if a modifier would enhance extraction efficiency, greige knit samples were spiked with a volume (2.5 mL) of modifier equal to the weight of the sample (2.5 g). Both TCE and ethanol improved the recoveries. Without the modifier the recovery was 0.74%, while with the TCE the recovery was 0.89% and with ethanol it was 0.86%. Since one of the goals of the project was to eliminate the use of halogenated solvents, ethanol was chosen as the modifier for further study.

The effect of the volume of ethanol on extraction efficiency was determined by measuring recoveries from samples of greige fabric that had been spiked with different volumes of modifier. Results of these extractions are shown in Table VI. Recovery of extractables was maximized at 2.5 mL/2.5 g of sample. Immersion of the sample in ethanol gave the same level of recovery. By evaporation of the residual ethanol in which the sample had been dipped, it was shown that no extractables were lost by this procedure.

Table VI. Effect of Ethanol Volume on CO_2 Extraction Recoveries

Ethanol, mL	% Recovery
0.25	0.64
0.75	0.73
1.25	0.79
1.75	0.81
2.5	0.89
3.33	0.90
6.67	0.90
Sample dipped in ethanol	0.87

Once it had been determined that ethanol-modified CO_2 yielded recover-

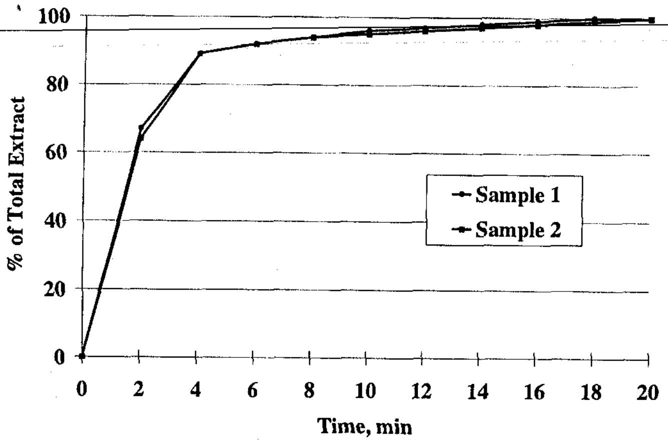

Fig. 2. Ethanol-modified SFE: greige knit, recovery during dynamic extraction.

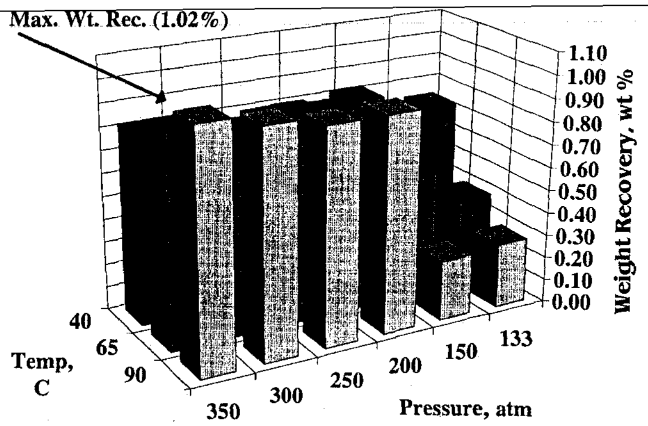

Fig. 3. Ethanol-modified SFE: greige knit, weight recoveries for various pressures and temperatures.

ies comparable to Soxhlet extractions, the time of the supercritical extraction was optimized. Fig. 2 shows results from duplicate supercritical fluid extractions in which recovery vials were changed every two minutes. After five minutes of dynamic extraction, 90% of the materials extracted during the run have been collected. The extraction was continued to 20 minutes to ensure complete removal of the extractables.

To determine minimum pressure and temperature requirements for an ethanol-modified SFE of cotton, an experimental design was completed at 2 mL/min flow and 10 minute static/10 minute dynamic extraction at three temperatures and six pressures. Samples were not pre-dried. Recovery data are shown in Fig. 3. In a statistical design such as this, it is common practice to randomize the variables; i.e., temperature and pressure. Because of the time required to cool the oven, these extractions were completed by starting at the lowest temperature and extracting at randomized pressures before increasing the temperature. One pressure setting at each temperature was evaluated the following day. ANOVA showed that the extraction day did not significantly affect the results. This indicates that lack of temperature randomization did not significantly affect extraction recoveries. ANOVA showed that recoveries were significantly affected by temperature, pressure, and the pressure/temperature interaction. One set of conditions, 350 atm and 90C, was chosen as a reference point for further comparisons. Dunnett's test was applied to determine which temperature/pressure combinations differed significantly (95% confidence interval) from the reference condition. Temperature/pressure conditions which were not significantly different from 90C/350 atm were 90C/300 atm, 90C/250 atm, 90C/200 atm, and 65C/350 atm. From Fig. 3 it is apparent that the highest recoveries were obtained at 90C between 200 and 350 atm. Those recoveries were all greater than 0.91% compared to 0.95% for Soxhlet recoveries from the same greige knit under the identical weighing conditions.

Table VII shows Soxhlet and SFE recovery data for seven different fabric samples. The correlation coefficient (r^2) for this set of samples was 0.93 indicating very good correlation between Soxhlet and SFE recoveries. The correlation coefficient for Soxhlet recovery vs. SFE recovery for a set of five fibers was only 0.82 and when recoveries for three yarns were combined with the fiber and fabric data, the correlation coefficient dropped to 0.66. Fig. 4 shows a plot of all 16 of these data points. It is apparent from this plot that SFE did not recover as much material as Soxhlet extraction when extractables exceeded 1.0%. This may indicate the need to extend the extraction times.

Instrumental Analysis of Extracts

Analysis of the extracts by FTIR showed the expected absorption bands for the cotton wax and the paraffin wax. The knitting oil was not apparent in FTIR spectra of extracts. Only small differences were shown between spectra in the following comparisons:

- Soxhlet vs. ethanol-modified SF extracts of cotton samples
- Ethanol-modified SF extract vs. TCE-modified SF extracts of greige knits
- Ethanol-modified SF extracts of greige and scoured cotton knits
- Ethanol-modified SF dynamic vs. ethanol-modified SF static extracts of cotton knits

FTIR spectra did show some differences between the extract from one instrument and the extracts from two other instruments. In one instrument, some of the extract seemed to condense in an exit vent. This may mean that some of the more volatile components are lost through aerosoling with this instrument.

Previous GC/MS analysis of cotton wax indicated the presence of several alcohols, acids, esters, hydrocarbons, and steroids.[13] The chromatograms of winding wax showed the expected envelope of well-resolved peaks between 8 and 14 minutes. The knitting oil gave a very distinct broad unresolved mineral oil band from 5-10 minutes. The cotton wax contained 70 peaks between 4 and 17 minutes. The largest seven peaks were selected for identification by GC and GC/MS. Of the seven major peaks in the gas chromatogram, only two (1-triacontanol and triacontanoic acid) were identifiable by comparison with retention times of available authentic samples. However, no peaks eluted at the retention times cor-

Table VII. SFE and Soxhlet Recoveries: Cotton Fabrics

Knit Sample	Soxhlet Avg. % Recovery (n=2)	SFE Avg. % Recovery (n=2)
Greige	0.95 ± 0.02	0.88 ± 0.06
Scoured	0.80 ± 0.05	0.75 ± 0.06
Bleached	0.48 ± 0.04	0.41 ± 0.00
Test Greige	1.56 ± 0.08	1.45 ± 0.11
4.2 micronaire	1.17 ± 0.23	1.03 ± 0.05
3.4 micronaire	1.36 ± 0.21	1.04 ± 0.08
5.0 micronaire	1.21 ± 0.16	0.95 ± 0.06

Table VIII. Components in Cotton Wax

Analyte	Retention Time (min)	Silylated Mol. Wt. (m/e)
Palmitic Acid	6.79	328
Stearic Acid	7.96	356
Montanol	12.44	482
Alcohol-X	13.16	482
Triacontanol	13.25	510

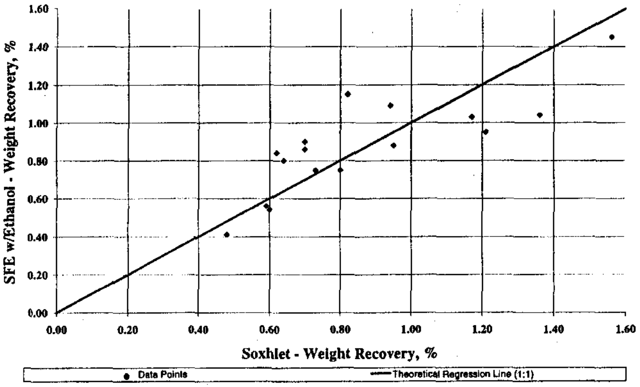

Fig. 4. SFE vs. Soxhlet recoveries for fibers, yarns, and knit fabrics.

Table IX. Fluidities Before and After SFE and Soxhlet Extraction

Fiber Sample	Before Extraction Fluidity, Rhes (n=8)	Soxhlet Extraction Fluidity, Rhes (n=2)	SFE-w/EtOH Fluidity, Rhes (n=2)
Low Strength	0.569	0.639	0.654
High Strength	0.529	0.729	0.670

responding to hexacosanoic acid, β-sitosterol, or triacontane. A significant peak (12.64 min) appeared in several extract GC chromatograms but did not appear in the GC/MS chromatograms of those same extracts. Gas chromatography proved to be a better tool than FTIR for detecting the presence of knitting oil and winding wax in the cotton extracts. With the exception of differences in intensity and the 12.64-min peak, the chromatograms from SF and Soxhlet extracts were quite similar. Some minor differences were noted in the extracts from two extractors. Analysis of the difference showed that it was probably due to the use of a higher boiling trap-rinsing solvent (toluene). Evaporation of the toluene required higher temperatures and may have resulted in the loss of some volatile knitting oil components.

Mass spectral analysis of the silylated extracts allowed identification of five components (Table VIII) in cotton wax. Alcohol-X was not identified but its mass spectrum indicates a primary alcohol of formula $C_{28}H_{58}O$. In contrast to previous work[13] no β-sitosterol was found in either Soxhlet or SF extracts. There were minor differences between the composition of Soxhlet and SF extracts; however, the two extraction techniques appear to be equally efficient in removing the cotton fiber wax constituents. This strongly suggests that there are no significant qualitative differences between extracts from solvent and carbon dioxide or ethanol-modified carbon dioxide extractions. Additional analyses of these extracts may be reported at a later date, but copies of representative FTIR spectra and gas chromatograms are available from the authors upon request.

Fluidity Results

Table IX shows fluidity data for two different fiber samples before extraction, after Soxhlet extraction, and after supercritical extraction. The differences between Soxhlet-extracted and greige samples was statistically significant. The same is true of the SFE and greige samples, but there was not significant difference between Soxhlet and SFE samples. Even though there was a significant difference, indicating some decrease in the molecular weight of the cellulose, the difference is not practically significant because the value is still well below 5 rhes.

Conclusions

Unmodified supercritical carbon dioxide quantitatively extracts paraffin winding wax and mineral oil-based knitting oil from cotton yarns and fabrics, but does not completely extract the cotton wax. Addition of a modifier; e.g., TCE or ethanol, increases the supercritical extraction efficiency to that of solvent-based Soxhlet extraction. This work suggests that the modifier be used in quantities sufficient to saturate the sample. Satisfactory conditions for ethanol-modified supercritical extraction of cotton wax were 65C/350 atm, 90C/200 atm, 90C/250 atm, 90C/300 atm, and 90C/350 atm.

Analysis of fluidity data indicates that a slight decrease in molecular weight is caused by both SFE and Soxhlet extraction. The damage caused by extraction is slight compared to that which the fibers experience in preparation.

Dynamic and a combination of static and dynamic extraction conditions seem to give comparable recoveries. Instrumental analysis of Soxhlet and SF extracts indicates that these two techniques give quite comparable qualitative results; i.e., both extractions remove the same materials.

A 20-min SFE may not be adequate to completely remove all of the wax from samples with high cotton wax content.

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