Removal of Oils and Waxes from Fabric with Supercritical Fluid Extraction

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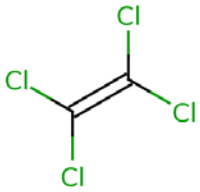
Introduction

Perchloroethylene (as seen in the figure below) has been the standard dry cleaning solvent for over seven decades. Dry cleaning with perchloroethylene not only leaves a distinctive chemical odor on clothes, but can damage colored clothing, buttons, and beads. Perchloroethylene is also highly carcinogenic substance. Perchloroethylene remains in clothes; ergo consistent clothing exposure to perchloroethylene substantially raises levels of the carcinogen.



SFT Inc. has developed a method utilizing the lab-scale SFT-110 SFE to remove stains from clothing utilizing supercritical carbon dioxide. Supercritical CO_2 is extremely successful at extracting oils from plants, seeds, and other types of matrices and we have applied this technology to clothing.

The SFT-110 SFE utilizes pressurized carbon dioxide, allowing extraction to take place in room temperature environments, so a clean, energy efficient product is generated. Carbon dioxide is also advantageous, because it does not introduce any residual organic chemicals, meaning that all extracts are safe to wear. Carbon dioxide extraction using the SFT-110 SFE can separate stains from fabric in a clean, energy efficient fashion. Recent reports have even indicated that supercritical fabric cleaning far surpasses typical stain removal, but can also recover water and fire damaged fabric.

Equipment

- Analytical Balance
- SFT-110 SFE Unit
- SFT 100 cc Sample Vessel

Materials

- 100% Cotton T-shirt
- Paraffin Wax
- Mineral Oil
- 3 SFT Collection Vials
- 1 SFT Vent Tube

Figure 1. SFT-110 SFE Unit

Experimental Procedure

A simple SFE extraction method using the SFT-110 SFE was employed to extract mineral oil and paraffin wax from cotton clothes.

Experiment #1 Paraffin Extraction

Weigh 15 grams of the paraffin wax on an analytical balance. Melt and place on a 100% cotton t-shirt. After a drying time of 24 hours, roll the t-shirt tightly. Place the rolled paraffin covered t-shirt into the SFT 100 cc vessel (10kpsi, 200°C operation).

Seal the vessel and set into a SFT-110 SFE unit according to manual instructions. Affix a SFT-vent tube into a clean, tared SFT-collection vial to the extract collection tube. One fraction of oil will be collected via multiple (20) soak and dynamic flow steps. Extract the sample according to the following parameters:

Experiment #2 Mineral Oil Extraction

Weigh 12 grams (approximately 15 mL) of the mineral oil on an analytical balance. Pour on a 100% cotton t-shirt. After a drying time of 72 hours (or until the shirt is completely dry), roll the t-shirt tightly. Place the rolled oil covered t-shirt into the SFT 100 cc vessel (10kpsi, 200°C operation).

Seal the vessel and set into a SFT-110 SFE unit according to manual instructions. Affix a SFT-vent tube into a clean, tared SFT-collection vial to the extract collection tube. One fraction of oil will be collected via multiple (20) soak and dynamic flow steps. Extract the sample according to the following parameters:

Experiment #3 Paraffin and Mineral Oil Extraction

Weigh 7.5 grams of the paraffin wax on an analytical balance. Melt and place on a 100% cotton t-shirt. After a drying time of 24 hours, add 6 grams (7.5 mL) of the mineral oil to the t-shirt. Allow for additional drying time until the shirt is completely dry.

Roll the t-shirt tightly. Place the rolled paraffin and oil covered t-shirt into the SFT 100 cc vessel (10kpsi, 200°C operation).

Seal the vessel and set into a SFT-110 SFE unit according to manual instructions. Affix a SFT-vent tube into a clean, tared SFT-collection vial to the extract collection tube. One fraction of oil will be collected via multiple (20) soak and dynamic flow steps. Extract the sample according to the following parameters:

Extraction Parameters

One fraction of oil will be collected via multiple soak and dynamic flow cycles according to the guidelines below. These following parameters will work for all 3 experiments.

Essential Oil Extract

- Pressure: 6800 psi
- Oven Temperature: 65°C
- Restrictor Temperature: 80ºC
- CO_2 Flow Rate: 10 mL/min
- 20 static and dynamic steps for 10 minutes apiece

Results

All three of the fabric samples were bright white when removed from the 100 cc sample vessel (see *Figures 2-4)*. The

paraffin wax and mineral oil extract will yield products that look exactly like

Figure 2. Wax Stained Fabric before and after Supercritical Extraction

Figure 3. Oil Stained Fabric before and after Supercritical Extraction

the starting materials. The paraffin wax extract (see *Figures 2 & 5*) will be a white thick solid product. The mineral oil extract (see *Figures 3 & 5*) will be a clear liquid product. Naturally, the mineral oil and paraffin wax extract will be the combination of the 2 (*Figures 4 &5)*.

Figure 4. Oil and Wax Stained Fabric before and after Supercritical Extraction

For the sample containing strictly paraffin wax extract, there was a recovery rate of 100% or 15.0001 grams (see *Figure 6*). For the sample containing strictly mineral oil wax extract, there was a recovery rate of 104.63% or 15.6944 grams. For the sample containing both paraffin wax and mineral oil extracts, there was a recovery rate of 105.21% or 15.7822 grams.

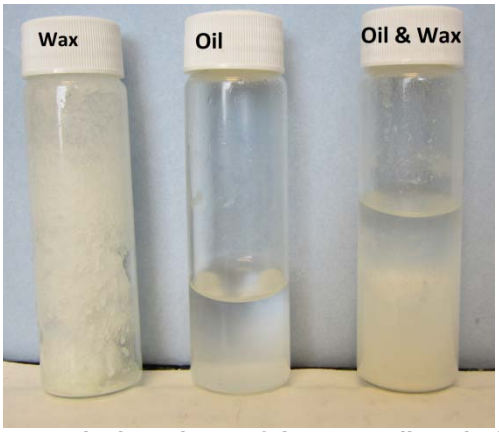


Figure 5. Final Products of the Wax, Oil, and Oil & Wax Extracts

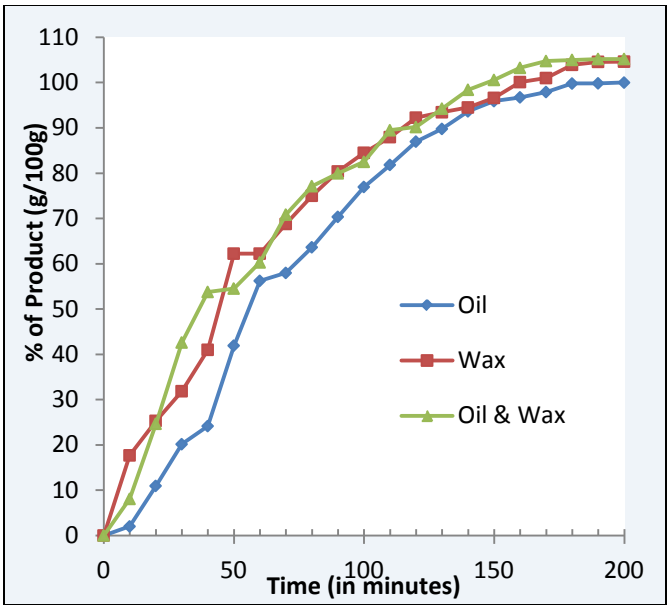


Figure 3. Supercritical Carbon Dioxide Extraction

For the mineral oil extraction and paraffin wax/mineral oil extraction, there was a recovery rate greater than 100%. This indicates that the SFT-110 can extract mineral oil and paraffin wax left over from the manufacturing process at 6820 psi.

CONCLUSIONS

The SFT-110 can remove any paraffin wax and mineral oil deposits at 6820 psi, including residual manufacturing oils. It is a green and safe alternative to traditional dry cleaning methods, such as perchloroethylene. Supercritical carbon dioxide will remove stains effectively and will leave no harsh chemical smells on clothing.

REFERENCES

Beck, Keith and More, Lynn. "Extraction of Cotton Impurities: Supercritical C02 vs. SoxhIet/TCE." Textile Chemist and Colorist. 29.8 (1997): 66-70. Print.