



Applications of Supercritical Carbon dioxide in Textile Finishing: A Review

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SUPERCritical carbon dioxide (scCO₂) impregnation is growing in popularity due to its unique characteristics such as high diffusivity, low surface tension, and simplicity of solvent removal at the end of the process. Furthermore, scCO₂ is the most environmentally friendly solvent, with several advantages over traditional aqueous and solvent-based procedures. ScCO₂ impregnation has a wide range of uses, mostly for incorporating various active principles into a polymeric matrix, such as medicines, functional finishing agents, dyes, and other agents. This study reviews some previous investigations on the use of scCO₂ as an impregnation medium in the development of various functional materials, to stimulate additional research into the use of scCO₂ in textile finishing.

Keywords: Textile finishing, supercritical carbon dioxide (scCO₂).

Introduction

The textile sector is one of the largest users of water. Every day, the textile industry consumes a huge amount of water for the processes of textile material. processes involved: (a) pretreatment, (b) dyeing process, and (c) after treatment (Finishing).

On average, 100-145 liters of water are required to produce 1 kilogram of textile material. Water is utilized as a solvent in various pretreatment and finishing procedures, as well as coloring processes, such as washing, scouring, bleaching, dyeing, and imparting specific finishing effects into textile materials. Despite attempts to minimize water input, such as changing traditional equipment, recycling water, and reusing wastewater- water use in the textile sector remains high. As a result, the textile industry is increasingly focusing on alternative green technologies and eco-friendly chemical agents to mitigate these issues. [1]

The need for functional and smart textiles has increased in recent years as people's lifestyles

have changed. In addition, the manufacturing of functional and smart fabrics is steadily expanding. On the other hand, traditional dyeing and finishing procedures used to make functional textiles have drawbacks such as the use of a lot of fresh water, energy, and chemicals, as well as the accompanying wastewater pollution, which is damaging to humans, animals, and the environment.

Furthermore, it has become an economic concern for the textile sector as a result of the rigorous environmental regulations on effluent release and, as a result, the requirement for wastewater treatment [2]. As a result, specialists are attempting to design new technology to solve this problem. Experts searched for a chemical that could function as both a liquid and a gas at a certain temperature and pressure. Finally, a new method called Supercritical Fluid Dyeing Technology has been presented to dye textile materials without the need for water. CO₂ is utilized for this because it can exist in both a liquid and a gas state at a certain pressure and temperature [3].

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Supercritical carbon dioxide (scCO₂) technology has taken attention in the textile industry. This approach provides several benefits over conventional aqueous procedures. Notably, carbon dioxide is harmless, nonflammable, and recyclable; dyes may be reused, and no additional chemicals are required. Furthermore, energy may be conserved because a substrate-drying process is eliminated, and the dyeing time is reduced as compared to previous procedures.

Supercritical carbon dioxide technology

A “supercritical fluid” (SCF) is a substance that exists above its critical temperature (T_c) and critical pressure (P_c). The critical point is the greatest temperature and pressure at which the material may exist in equilibrium as a vapor and liquid. The phenomena are simply described using the phase diagram for pure carbon dioxide. (Figure 1). This diagram depicts the states of carbon dioxide as a gas, liquid, solid, or SCF. Temperatures and pressures when two phases coexist in equilibrium are shown by the curves.; (at the triple point, the three phases coexist)[4].

When carbon dioxide is heated to over 310°K and pressured to over 74 bar, it becomes supercritical, a state of matter that resembles an expanded liquid or a strongly compressed gas. In other words, above the critical point, carbon dioxide has both liquid and gas properties. In this way, supercritical CO₂ has liquid-like densities, which is beneficial for dissolving hydrophobic dyes, as well as gas-like low viscosities and diffusion characteristics, which can result in faster dyeing times than water. In comparison to water dyeing, the extraction of carbon dioxide dyeing method includes just changing the temperature and pressure conditions; drying is not necessary

because CO₂ is released in the gaseous state after the process.[1].

The most widely utilized supercritical fluid is carbon dioxide.:

- Low cost and easy availability in high purity.
- Chemically inert, non-toxic, non-flammable, and bacteria-free.
- Low critical point: 31°C and 74 bar.
- Allows the fluid to be employed in low-temperature conditions, preserving the integrity of thermo-sensitive components.
- CO₂ is recycled from industrial waste.
- The products are oxidation-resistant.
- There are no solvents in the products or residues.

Supercritical carbon dioxide apparatus

The supercritical carbon dioxide device, shown in Figure 2, is commonly used for dyeing fabrics. The supercritical carbon dioxide apparatus was loaded with fibers. as indicated in Figure 2, The procedure was carried out in the dyeing kettle (7). A purifier (2) filtered the carbon dioxide stored in the carbon dioxide tank (1), which was then cooled into liquid by a pre-cooler (3). When the pressure boosted by a high-pressure pump (4) and the temperature raised by a heat exchanger (5) in the treatment system reached a certain level, the procedure began. The flowmeter (9) determined the rate of pressure release. The fibers were removed after the appropriate treatment period. Finally, carbon dioxide would be converted to gas in a separator (8) and chilled to liquid in a refrigerator (10)[5].

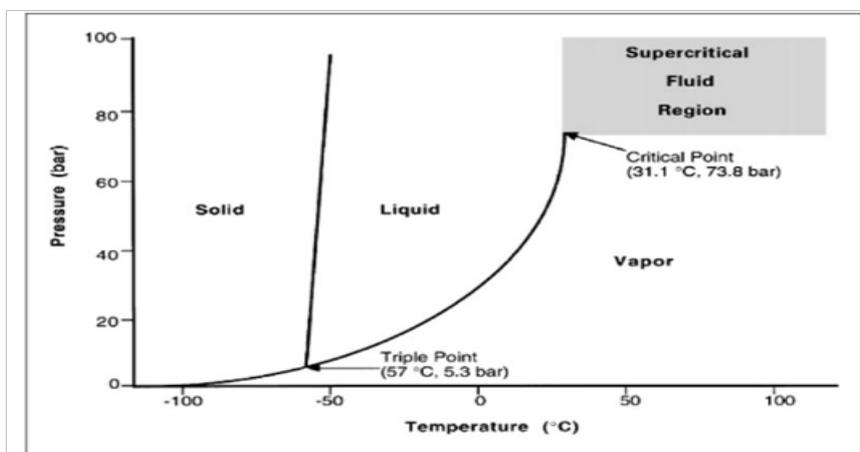


Fig.1. Phase diagram for pure carbon dioxide[4].

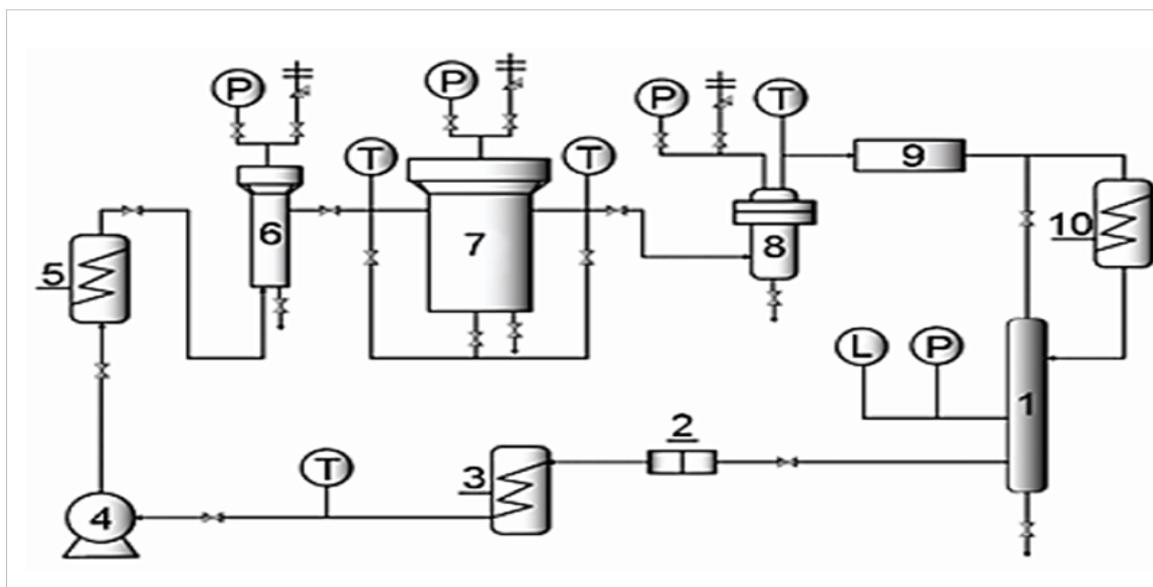


Fig. 2. The flow diagram of the supercritical carbon dioxide system [5].

(1) carbon dioxide tank, (2) purifier, (3) pre-cooler, (4) high-pressure pump, (5) heat exchanger, (6) dye kettle, (7) dyeing kettle, (8) separator, (9) flowmeter, (10) refrigerator.

Impregnation of materials in $scCO_2$

Impregnation of polymeric materials using the SCF approach is becoming a popular method for developing new functional products. The use of SCF impregnation is motivated by the environmental and economic advantages it has over conventional impregnation processes. Traditional impregnation procedures include disadvantages such as the use of dangerous solvents, significant energy consumption, and extremely slow diffusion rates. As a result, it necessitates a long contact time, and the process's low efficiency necessitates the use of a significant number of chemicals and solvents [6]. Because of its unique characteristics and environmental friendliness, $scCO_2$ aided impregnation is a viable solution to these issues. Besides the environmental benefits, this also results from the ability to make use of the $scCO_2$ process's strong diffusivity, solubility, low surface tension, and facile solvent removal, ensuring high product purity free of residual solvent.

The ability to obtain a product that has no trace of solvent is very significant in the development of functional products[7]. This also lowers the expense of removing the leftover solvent after the impregnation process. Another benefit of CO_2 that makes it suited for polymer impregnation is its swelling and plasticizing impact on many polymeric materials, which is important for solute loading. Because of these benefits, the $scCO_2$ aided impregnation technology has been adopted for the

impregnation of various polymeric materials with various additives such as dyes, medicines, and functional finishing agents.

The main impregnation process in $scCO_2$ consists of three major steps: (1) solute dissolution, (2) sorption of the mixture ($scCO_2$ and solute) in the polymer matrix, and (3) system depressurization. Figure 3 depicts a schematic illustration of the impregnation process. In most cases, the dissolution of the solute and the sorption of the solute mixture occur simultaneously. Total or partial dissolution may occur during dissolution, depending on the additive's properties and/or the processing circumstances. The sorption of CO_2 and active compounds is determined by the degree of polymer swelling and plasticization, which is also determined by the chemical nature of the polymer and the system's impregnation conditions (mostly temperature and pressure)[8].

The final depressurization step eliminates CO_2 and is also one way utilized to incorporate solute molecules into SCF-specific polymer matrices. Due to a quick decrease in the solvating power of $scCO_2$, rapid depressurization may improve the quantity of solute incorporated into the polymer. on the other hand, Rapid depressurization may induce polymer foaming, cracking, or even degradation. [9] As a result, the rate of depressurization should be carefully chosen based on the polymer and additive applied.

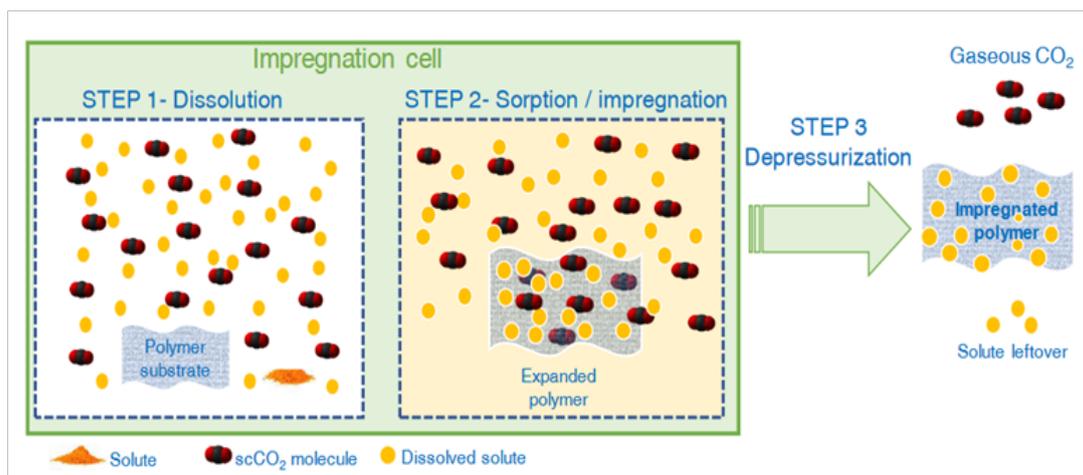


Fig. 3. Schematic of the three steps of the impregnation process of a polymer substrate

Functionalization of materials in scCO₂

The use of scCO₂ impregnation methods to impart diverse functionalities to textiles has also been reported. scCO₂ has been used to incorporate a variety of polymeric materials with various functional compounds, including poly (methyl methacrylate), polyethylene, polyvinyl chloride, polycaprolactone, polyamide, polycarbonate, polytetrafluoroethylene, linear low-density polyethylene, polyester, and so on. These polymeric materials have been impregnated with a variety of functional agents, ranging from metal carbonyl complexes to organic dyes. [10] The scCO₂ impregnation technology has been used to treat textile materials with various functional agents. Functional dyes (natural and synthetic origin), silicon and fluoropolymer, natural extracts, and organometallic-based agents are the most common active chemical species used to functionalize textiles utilizing the scCO₂ impregnation approach. [11]

Functional dyes

One technique for functionalizing textiles in scCO₂ has been to use various dyes with added functional properties. In this procedure, functional dyes are either synthesized by modifying them to incorporate functional groups via molecular design, or dyes with the desired functional characteristic are employed directly. Most dispersed dyes are modified to incorporate functional groups dependent on the required functionality, and some of them are shown in this section. [11].

Abou Elmaaty *et al.* produced novel hydrazonepropanenitrile dyes and used scCO₂ to apply the new species to polyester fabric for possible antibacterial applications. Using the scCO₂ dyeing technique, efficient dyeing, *J. Text. Color. Polym. Sci.* **Vol. 19**, No. 2 (2022)

excellent antibacterial, and fastness characteristics were produced [12]. The same research team also created a series of disperse azo dyes with possible antimicrobial activity, which they applied to Nylon 6 through the scCO₂ impregnation approach. Higher antibacterial properties and better colorfastness properties were obtained. When compared to traditional exhaust dyed samples [13].

Abou Elmaaty *et al.* also developed a new method for depositing selenium nanoparticles (SeNPs) onto polypropylene (PP) textiles using an IR-dyeing machine under hydrothermal conditions in a one-step procedure. The hue of PP textiles ranged from light to dark orange, depending on the treatment period and sodium hydrogen selenite concentration. The results show that including SeNPs into PP fabric improves UV protection significantly. Furthermore, the treated materials had excellent washing, rubbing, and lightfastness. In addition, the colored PP fabric demonstrated outstanding antimicrobial effect against *Staphylococcus aureus*, *Bacillus cereus*, and *Escherichia coli*, as well as very strong antibacterial activity against *Pseudomonas aeruginosa* [14].

Many natural dyes have been studied as functional dyes, including curcumin, berberine, and henna. It has been reported that curcumin's natural dye was impregnated into polyester (PET) films and poly(hydroxybutyrate) (PHB) granules in scCO₂. In this study, The impregnation technique was successfully established with varying quantities of curcumin add-on depending on the dyeing conditions, and no substantial adverse influence on the material properties was detected [15]. In another study, Curcumin has been used in scCO₂ to dye and functionalize polyester. Dyed samples

demonstrated excellent color strength and fastness, as well as increased antibacterial, antioxidant, and UV protection. Thus, using functional dyes that are compatible with the $scCO_2$ process is a viable method for producing colorful and functional material in a single step [16].

Antimicrobial agents

Many researchers have investigated the SCF impregnation of organometallic compounds into polymer matrices. Silver in various forms has been widely used to develop different functionalities in fibers. In $scCO_2$, silver nanoparticles were employed to modify wool fabric, the results showed that the modified fabric demonstrated good catalytic, antistatic, and antibacterial properties [17]. Two different silver precursor materials, AgNPs, were applied to cotton fabric using pressured CO_2 , and the subsequent reduction of the impregnated precursor with hydrogen gas was studied. Cotton fabrics treated with these silver precursors showed excellent antibacterial activity [18].

To produce antimicrobial fabric, a CO_2 -philic silicon-containing quaternary ammonium salt (QAS) was synthesized and applied to cotton in $scCO_2$. The treated fabric had strong antibacterial activity and was resistant to washing and UV exposure. They also produced silicone-based 2,2,6,6-tetramethyl-4-piperidinol (TMP)-based N-chloramine and used a $scCO_2$ impregnation approach to apply it to polyethylene (PE) fiber. Using 28×10^6 Pa pressure, a homogeneous coating of TMP-based N-chloramine with a thickness of 70×10^{-9} m was achieved. PE treated with TMP-based N-chloramine has strong and long-lasting biocidal

action [19]. The same research team produced a CO_2 -philic biocidal fluorinated pyridinium silicon that was impregnated into cotton yarn using $scCO_2$. At 24×10^6 Pa and $50^\circ C$, a biocidal layer with pyridinium groups separated on the top surface can reach a thickness of 50×10^{-9} m. The material obtained has greater biocidal effectiveness [20].

Supercritical carbon dioxide application using N-halamine, according to Orhan et al., could be an alternate option for getting antibacterial function on the polyester surface. Firstly, N-(2-methyl-1-(4-methyl-2,5-dioxo-imidazolidin-4 yl) propane-2-yl) acrylamide was produced and applied to polyester in supercritical carbon dioxide medium at $120^\circ C$ and 30 MPa for various processing periods (Figure 4). The addition of N-halamine to the surface resulted in substantial antibacterial action against *E. coli*. The chlorine loadings revealed that a 6-hour exposure duration was required to achieve adequate antibacterial activity [21].

Several natural-based substances, including thymol, carvacrol, eugenol, pyrethrum extract, and others, have been utilized to impregnate various polymers to modify the material characteristics and generate new functions. Zizovic et al. conducted extensive research on the application of thymol to various textile-based substrates in $scCO_2$ to generate various functional materials. They investigated thymol solubility in $scCO_2$ and its impregnation on cotton gauze. The impregnated gauze had significant antibacterial action against *Escherichia coli*, *Staphylococcus aureus*, *Bacillus subtilis*, *Enterococcus faecalis*, and *Candida albicans* [22].

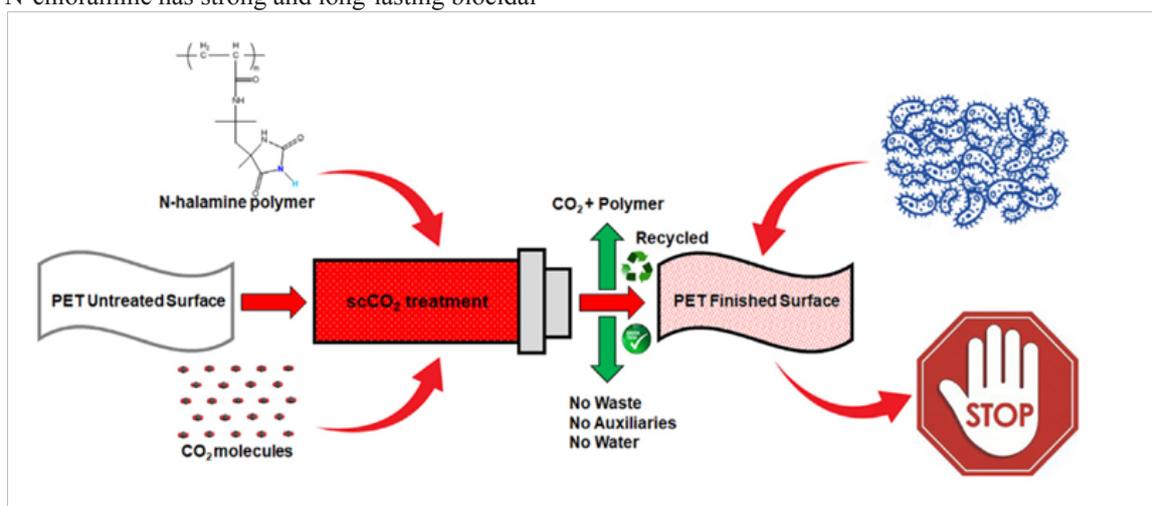


Fig. 4. Application of supercritical carbon dioxide using N-halamine on polyester fabric

Chitosan and its derivatives were utilized to impregnate polyester in a scCO₂ bath. The results showed that the low molecular weight chitosan and chitosan lactic acid salt were effectively impregnated, while chitin could not be impregnated [23]. Recently, low molecular weight chitosan and chitosan lactate were effectively integrated into the polyester fabric using the scCO₂ dyeing process, resulting in high antibacterial action. Overall, natural-based functional agents have demonstrated great potential for the production of different functional materials using the scCO₂ impregnation approach [24].

In a scCO₂ medium, Ivanovic et al. impregnated polycaprolactone (PCL) and polycaprolactone hydroxyapatite (PCL-HA) composites with thymol. Thymol was shown to be soluble in scCO₂, and the impregnation technique was successful. All of the samples prepared by scCO₂ impregnation had significant antibacterial activity against a diverse spectrum of bacteria strains [25].

Pajnik et al. impregnated sutures with the natural bioactive material thymol under mild process conditions (35°C, 10 MPa, 1–6 h) using supercritical CO₂. The proposed technique permitted thymol loadings of up to 5.9 %. All of the samples obtained had an antibacterial impact on the bacteria strains tested (*E. coli* and *S. aureus*) [26].

Water and oil repellent-based functional agents

As previously stated, silicon and amorphous fluoropolymers are known to be soluble in scCO₂. As a result, functional agents based on these compounds have been used to functionalize a wide range of fabrics, polymers, and films. For covalently bonding silicon and cellulose, several crosslinking agents were utilized. The findings show that the scCO₂ medium coats the cotton surface well with a 3D network of DMS compound and crosslinker.

Another study treated polyester fabric with low molecular weight polytetrafluoroethylene in a scCO₂ medium and consistently obtained a high degree of water repellency [27].

Recently, nylon fabric was treated in scCO₂ medium with perfluoroalkyl methacrylate/hydroxyalkyl methacrylate and a crosslinking agent (diisocyanate) to produce a durable water and oil resistant coating [28]. When compared to a coating applied with a liquid solvent, scCO₂ treatment produced a homogeneous, highly repellent, and durable coating. These researches reveal that silicon and fluoropolymer-based

materials have played an important role in the use of the scCO₂ processing technique to functionalize textiles and polymeric materials.

Using an organic fluorine solution in scCO₂, Xu et al. produced a water/oil resistant polyester fabric. An evenly distributed fluorine may be achieved with good water/oil repellency while maintaining good air permeability and increased strength [29]. In another study, Hematite nanoparticles were loaded onto cellulosic fiber under scCO₂ to produce a water-resistant composite fiber [30].

Other functional agents

Supercritical CO₂ was used as the medium for impregnating cotton fabric with 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO), 2,20-oxybis-(5,5-dimethyl-1,3,2,-dioxaphosphorinane-2,20 -disulfide) (5060), SiO₂ and their mixes for flame retardant finishing. Vertical flame tests show that increasing the finishing time, pressure, and temperature considerably improves the flame retardant effect of the samples. DOPO-treated samples had the highest flame-retarding performance under the finishing conditions of 120°C, 22 MPa, and 120 minutes, followed by DOPO/5060 (1:1) and DOPO/SiO₂ (1:1). When the temperature, time, and flame-retardant ratio are increased to 130°C, 120 minutes, and 10% (o.m.f.), the after-glow time is lowered to 0 s. TG studies reveal that the char residues rise to 85.3 % at 305°C and 22.2 % at 800°C, which is superior to the results of the untreated cotton sample. [31]

Peng et al. used silane to pre-treat silk fabric with the aid of sc-CO₂, and then microwave irradiation to deposit silver nanoparticles on the silk fabric. The number of silver nanoparticles (AgNPs) coating the silk fabric pretreated with silane using the scCO₂ procedure is substantially higher than that achieved using the conventional method. Silver-coated silk fabric prepped with silane by the scCO₂ procedure has a considerably lower UV-vis transmittance than the original silk fabric. The findings suggest that the silane pretreatment could provide good UV radiation protection for silk fabrics. The silane pretreatment by sc-CO₂ procedure enhances the adhesion strength between the silver coating and the silk fabric substantially [32].

Cotton fabric has been treated with palladium (II) hexafluoro acetylacetonate to produce conductive textiles. [33] Peng et al. coated wool

textiles in scCO₂ with silver nanoparticles, and the coated fabric had good catalytic, antistatic, and antibacterial properties [17].

Supercritical carbon dioxide was utilized by Amina et al. to finish cotton textiles with modified dimethylsiloxane polymers terminated with silanol groups. For covalently bonding between silicon and cellulose, several cross-linkers such as 3-isocyanatepropyltriethoxysilane (IPES) and tetraethyl orthosilicate (TEOS) were utilized. SEM/EDX analysis and Confocal Raman microscopy (CRM) gave qualitative and quantitative information on the distribution of silicon molecules over the fiber cross-section, respectively. The results indicate that all fibers treated with PDMS and IPES have more silicon than fibers treated with TEOS. The sc-CO₂ medium coated the cotton surface with a 3D network of DMS compound and cross-linker, resulting in the formation of the maximum DMS concentration in a layer under the surface of cotton fibers. [34, 35]

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تطبيقات ثاني أكسيد الكربون الفائق الحرج في تجهيز المنسوجات

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يزداد التشريب فوق الحرج لثاني أكسيد الكربون ($scCO_2$) بسبب خصائصه الفريدة مثل الانتشار العالي والتوتر السطحي المنخفض وبساطة إزالة المذيبات في نهاية العملية. علاوة على ذلك، يعتبر $scCO_2$ أكثر المذيبات الصديقة للبيئة، مع العديد من المزايا التي تتفوق على الإجراءات التقليدية القائمة على المياه والمذيبات. التجهيز باستخدام $scCO_2$ له مجموعة واسعة من الاستخدامات، في الغالب لدمج مختلف المبادئ النشطة في مصفوفة بوليمرية، مثل الأدوية، وعوامل التجهيز الوظيفية، والأصبغ، وعوامل أخرى. تستعرض هذه الدراسة بعض التحقيقات السابقة حول استخدام $scCO_2$ كوسيط تشريب في تطوير مواد وظيفية مختلفة، بهدف تحفيز البحث الإضافي في استخدام $scCO_2$ في تشطيب المنسوجات.

الكلمات الدالة: تجهيز المنسوجات، الحالة الحرجة لثاني أكسيد الكربون ($scCO_2$)