Supercritical CO_2 Technology in Resource-effective Production of Functional and Smart Textiles *

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Abstract

The purpose of this work was to investigate the potential application of $scCO_2$ dyeing technology to produce functional and smart textiles. Selected dyes and functional agents were applied to polyester fabric using the $scCO_2$ impregnation technique. The effects of the processing variables on the functional and colour performances were explored. The results showed that $scCO_2$ is a viable technique to produce functional polyester fabric in a resource-efficient and eco-friendly way. Dyed polyester fabric with additional functionalities such as antimicrobial, antioxidant, UV protection, and UV sensing properties were realised. The fabrics developed have demonstrated desirable colour and functional properties without affecting each other confirming compatibility. Moreover, the functional fabrics exhibited the required durability and fastness properties sufficient for various applications. This research project contributes towards widening the application of the supercritical CO_2 dyeing technique and paves a way for sustainable production of functional and smart textiles in a resource-efficient and eco-friendly way.

Keywords: Supercritical CO₂; Functional and Smart textiles; Antimicrobial; Antioxidant; UV protective textile; Textile UV-sensor; Curcumin natural dye; Chitosan biopolymers; Photochromic textiles

1 Introduction

Functional clothing is a domain of textiles that are designed to deliver specific functions to the users in addition to their usual wearing purposes. Functional textiles encompass areas like

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protective textiles, medical textiles, industrial textiles, sports textiles, automotive textiles, and packaging textiles. In recent times, functional and smart textiles have emerged as a growth field. The market for functional clothing is predicted to reach 244.6 billion USD by 2025 and the global smart textile market size is expected to reach 5.5 billion USD by the same year [1]. Among the textile fibres used for smart and functional textile applications, polyester and viscose (rayon) fibres are the basic and most used ones [2]. The worldwide production volume of textile fibres has increased from 23.94 million metric tons in the year 1975 to 98.5 million metric tons in 2017. Among these, about 53.7 million metric tons (54.5%) were polyester (PET) fibres and the volume is estimated to increase in the coming years [3]. These statistics clearly show that smart and functional textiles are one of the most important fields in the textile industry and polyester is the most important textile fibre in the production of functional and smart textiles.

These functional textiles are mainly manufactured by conventional dyeing and/or finishing process or incorporating various functional finishing agents into the textile structure during the fibre production process. However, the conventional wet textile processes; particularly the dyeing and finishing processes consume a huge amount of water, chemicals, and energy. It is estimated that 100–150 liters of water are required to produce 1 kg of finished textile, on average [4]. This, in turn, results in a huge amount of wastewater discharge which causes large scale water pollution, one of the global environmental problems. The wastewater discharge of the textile wet processing industry lies between 40 and 300 m^3 per ton of finished textiles [5]. This wastewater discharge is contaminated with dyes, finishing agents, salt, alkalis and acids, auxiliary chemicals, and other solvents. A large amount of unfixed dyes and finishing chemicals leaves the process in which the exact amounts depends on the type of dyes or functional agents and the process used. In conventional polyester dyeing, it is estimated that approximately 5-10% of the dye is lost and ends up in the effluent [6, 7]. According to the World Bank estimate, 17 to 20% of industrial water pollution comes from textile dyeing and finishing operations [8]. This is a huge problem posing a serious threat to the flora and fauna and due to this the textile industry is mostly criticized for its role in environmental pollution.

To solve these issues, the textile industries have focused on the use of alternative eco-friendly techniques and agents to minimize waste generation and reduce toxic chemicals. In this regard, more emphasis has been given to the development of cleaner, cost-effective, and value-added textile products to solve the issues related to health and the environment. Not only the product but also the methods of production of the future itself must bring technological innovation and sustainability for possible advancements while keeping the balance of nature. Thus, the textile industries have focused on the use of green technologies as an alternative to conventional textile wet processing to promote sustainable production and reduce waste generation. Some of the important green technologies and renewable natural materials such as biopolymers, enzymes, and natural dyes used for sustainable textile production have been reviewed by [9]. As discussed in the review, some of the green technologies adopted recently include, supercritical CO_2 , microwave, plasma, ultrasound, and electrochemical reduction of dyes.

The use of environmentally friendly supercritical CO_2 as a dyeing medium has shown promising results owing to many advantages over conventional systems [10]. Supercritical CO_2 dyeing has several advantages such as it does not require any water, surfactants, or dispersing agents, and does not involve drying at the end of the procedure. It is non-toxic, non-flammable, inexpensive, chemically inert under many conditions, and easily manageable critical conditions. It was shown that the scCO₂ dyeing process is not only environmentally superior to aqueous dyeing but also economically [11]. Due to these attributes, it has been given considerable attention and the potentials of $scCO_2$ in textile dyeing have been investigated extensively for the past three decades as described in these two review papers [12, 13]. To date, $scCO_2$ dyeing of polyester fibres with disperse dyes is successful on an industrial scale [14]. In addition to dyeing, $scCO_2$ technology has also a promising potential to overcome the environmental and technical issues in many other commercial textile applications for example in the textile functionalization or finishing industry. However, the use of the $scCO_2$ processing technique to produce functional textiles is very limited so far. Therefore, further development and adaptation of this technology to the textile finishing industry is crucial and timely.

Thus, in this study, the possible use of the $scCO_2$ dyeing technique for the development of functional and smart textiles in an eco-friendly way as an alternative to the conventional dyeing and finishing methods has been explored. Several functionalities of interest have been considered based on literature data and broad screenings and those which gave good results were selected and investigated in this study. Generally, many kinds of functional finishing agents are used to impart various functionalities to textiles. However, most of the agents are based on synthetic compounds in which many of them are not environmentally safe with a harmful effect on humans and animals. Considering this, this study also gives due attention to the possibilities of using sustainable and green agents based on natural products such as bioactive functional agents.

2 Experimental

2.1 Materials

Two kinds of polyester fabrics, pique-knitted (123 g/m^2) and plain-woven (147 g/m^2) supplied by DyeCoo (Weesp, Netherlands) and FOV Fabrics (Sweden), respectively were used for all experiments. The fabrics were washed at 60 °C for 30 minutes with a standard detergent using a domestic laundry washing machine before use to remove spin oil and other contaminants. High performance disperse dyes (TERASIL®SC) tailored for scCO₂ dyeing received from DyeCoo (Weesp, Netherlands) were used. Several chitosan and its derivatives having different molecular weight and viscosity were acquired from Sigma-Aldrich AB, Sweden. Two kinds of curcumin natural dyes, having the same molecular weight 368.38 g/mol but with different purity were used. Two commercial photochromic dyes; Sea Green from Spirooxazine (SO) and Ruby Red from Naphthopyran (NP) dye classes (Vivimed Labs, UK) were used for the development of photochromic polyester fabric. Reagents and products such as Agar (select agar), Nutrient broth, Hexamethylene diisocyanate (HDI), Isopropanol, dodecylamine, ABTS, and others were used. The CO₂ used was purchased from AGA Industrial gases (Lidingo, Sweden) with 99.5% purity.

2.2 Method

The study is a fully experimental, and technology-driven approach based on the $scCO_2$ dyeing technique. For all $scCO_2$ dyeing and functionalization experiments, the polyester fabric was used, and the fabrics were washed using standard detergent before use. Different functional finishing agents were selected based on literature data and screening experiments and the selected agents were applied to polyester fabric under different dyeing conditions.

In this study, two strategies were followed to functionalize polyester fabric using the $scCO_2$

dyeing and impregnation route. In the first strategy, a combined process was followed in which the dye and functional agent (i.e. disperse dye and selected chitosan biopolymers) were applied in a single bath to dye and functionalize polyester simultaneously. In the second strategy, selected dyestuffs that offered additional functionalities to the textile were chosen and directly applied to polyester fabric in a $scCO_2$ bath. In the second strategy, the curcumin dye powder and photochromic colorants were directly applied into the $scCO_2$ dyeing vessel along with the polyester fabric without fabric modification and any auxiliary agent.

In the first strategy, selected chitosan biopolymers and disperse dye were used in a single bath to develop dyed and antimicrobial finished fabric simultaneously. In this strategy, two approaches were followed. In the first approach, a washed pristine polyester fabric was used without modification and in the second approach the polyester fabric was modified before $scCO_2$ treatment to acquire amino functionality on the fabric surface. In this approach, the modified fabric was treated in the presence of crosslinking agent to link chitosan molecules with the amine groups of modified polyester. Surface modification of polyester fabric was carried out according to the procedure previously used by Knittel *et al.* [15].

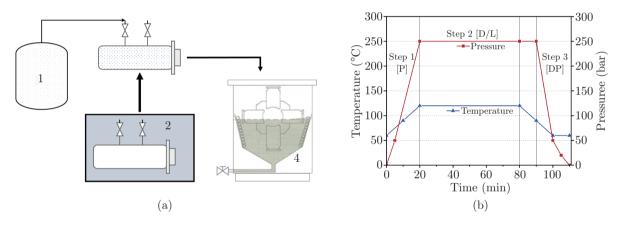


Fig. 1: (a) Schematic of $scCO_2$ lab dyeing procedure (1) CO_2 tank, (2) Freezer, (3) Dyeing vessel, (4) High-temperature oil bath (b) Dyeing step showing the temperature and pressure profile of the dyeing vessel. [P] Pressurization, [D/L] – Dyeing and Levelling, and [DP] – Depressurization

Fig. 1(a) shows the dyeing procedure and Fig. 1(b) illustrate the dyeing steps for running an experiment at 120 °C, 25 MPa for 60 min of dyeing time as an example. In all the experiments, the oil bath is pre-heated at least to about 60 °C before the introduction of the vessels. After the vessels are introduced into the oil bath, the temperature rose at a rate of 3°C per minute until the required dyeing temperature reaches. Initially, when the vessels are introduced into the oil bath is around 60 °C and the pressure is around 50 bars. During the pressurization step [P], the temperature rose to 120 °C causing the pressure to increase to 250 bars. Once the required temperature is reached, the dyeing and levelling step [D/L] continues for the desired time (60 min in this case). After the desired dyeing time is reached, the depressurization step [DP] continues by reducing the temperature until about 60 °C at a rate of 2-3 °C per minute and dyeing is completed.

Based on preliminary experiments, the condition of 120 °C, 25 MPs for 1 h was determined optimum and used for the dyeing/impregnation experiments throughout this study. For the simultaneous dyeing antimicrobial functionalization experiment, a fixed amount of dye (0.4 wt.%), chitosan (3 wt.%), a crosslinking agent (5 wt.%) was used for comparison purposes. For the

coloration and bio-activation experiment of polyester fabric using curcumin natural dye, an orthogonal array design was used to study the effects of the dyeing variables on the colour properties. To study the functional properties of curcumin-treated samples, higher purity curcumin was used and four different concentrations (0.25, 0.5, 0.75, and 1 wt.%) were selected to study the effect of dye concentration on the functional property. For the photochromic dyeing experiment, a small amount of photochromic dye powder (0.027 wt.% ≈ 2.56 mg) was used.

2.3 Characterizations

Surface characterizations fabric samples were performed using FTIR, Zeta potential, Water contact angle, and Scanning electron microscopy (SEM) measurements. The colour properties were evaluated in terms of colour strength (K/S) values using a spectrophotometer. The functional properties such as antibacterial, antioxidant, UV protection, and photochromic performances were assessed using standard testing methods as explained in the thesis in detail [16].

3 Summary of Results

3.1 Simultaneous Disperse Dyeing and Antimicrobial Functionalization of Polyester Fabric with Chitosan Biopolymers

This experiment aimed to investigate the suitability of the $scCO_2$ technique for dyeing and impregnation of polyester fabric with disperse dye and chitosan biopolymers to produce colored and functional polyester in a single step [17]. Among the chitosan types investigated during the broad screening experiment, two chitosan biopolymers exhibited better bacteria inhibition and weight gains, namely, chitosan (10 cps) with very low molecular weight (CLW) and chitosan lactate (CL) were selected. According to the results, it was observed that chitosan was hardly impregnated into the polyester fabric as displayed by lower weight gains and a considerable amount of chitosan powder remained inside the vessels after the impregnation experiments. As the weight gains were less reproducible, those samples that have similar impregnation yields were chosen for further characterization and comparisons. Even though weight gains were small, the amount impregnated was enough to evaluate the behavior of chitosan incorporated into the polyester fabric using the $scCO_2$ impregnation method. Among the two chitosan types, CL was impregnated in large amounts possibly due to the higher number of carboxylic and amino reactive groups available in CL that could interact with the anchoring agent and improved the amount of chitosan add-on.

Surface characterizations of the samples were carried out to assess the impregnation efficiency through FTIR, SEM, Zeta potential, and water contact angle measurements. All the results of the surface characterization showed the presence of chitosan molecules on the fabric surface confirming the potentialities of the $scCO_2$ impregnation process for the development of colored and antibacterial polyester fabric in a single step.

According to the results of antibacterial activity, all the chitosan treated samples exhibited higher antibacterial reduction compared with the dyed only sample. The antibacterial property of chitosan is believed to originate from its polycationic nature of chitosan. The cationic group (positively charged amino groups) of chitosan can interact with negatively charged residues of macromolecules at the cell surface of bacteria, which causes extensive cell surface modifications and alters cell permeability and subsequently inhibit the growth of bacteria [18]. Among the treated samples, samples treated with CL exhibited the highest antimicrobial activity (93% reduction) compared with the other samples. This could be due to the highest positive charge density as established from the ζ potential results. A marginal improvement in the durability could be possible after fabric surface modification and crosslinking mechanism but the antimicrobial activity was not improved as expected. Though promising results were obtained, the antimicrobial activity should be improved further to be applied as a true antimicrobial fabric especially in medical and hygienic conditions.

Finally, the colour strength (K/S value) of samples were assessed to evaluate the compatibility between the dye and chitosan biopolymers. For this, the colour strength of samples dyed with and without chitosan were compared. Based on the results there was no significant difference between the K/S values between the samples dyed separately and in combination with chitosan confirming compatibility. A slightly higher colour strength properties were observed when the dyeing was performed in the presence of chitosan compared with the dyed only samples. This could be attributed to the positive effect of chitosan molecules towards improving the hydrophilicity and increased localized amorphous domains of the fibre which promotes the sorption and diffusion of the dyes into the fibre. Furthermore, all the samples exhibited excellent colour fastness to washing and rubbing with a rating between 4/5 to 5 for both fade and stain fastness on an adjacent fabric of SDC Multi-fibre DW (wool, acrylic, polyester, polyamide 66, cotton, and acetate). In the first strategy, selected chitosan biopolymers and disperse dye were used in a single bath to develop dyed and antimicrobial finished fabric simultaneously. Within this strategy, two approaches were followed. In the first approach, a washed pristine polyester fabric was used without modification and in the second approach the polyester fabric was modified before $scCO_2$ treatment to acquire amino functionality on the fabric surface. in this approach, the modified fabric was then used in the presence of crosslinking agent to link chitosan molecules with the amine groups of modified polyester. Surface modification of polyester fabric surface was carried out according to the procedure previously used by Knittel et al. [15].

3.2 Coloration and Bio-activation of Polyester Fabric with Curcumin in $scCO_2$

This study aimed at developing colored and functionalized polyester fabric with curcumin natural dye using the scCO₂ dyeing technique. The effects of processing conditions on the colour and functional properties were investigated in detail [19, 20]. According to the results, the polyester fabric could be successfully dyed with curcumin natural dye in scCO₂ dyeing medium without fabric pre-or post-treatment avoiding mordant chemicals which otherwise are required in the conventional dyeing process of natural dyes. Results of the colour strength showed that the average K/S values increased with the system pressure, temperature, and dye concentration and slightly decreased with the dyeing time. Among the processing variables, the dyeing behavior of polyester fabric with curcumin in scCO₂ is strongly affected by system pressure followed by temperature. Based on the results, above 20 MPa, 100 °C, and at least 1 h dyeing time is enough to get acceptable quality. The highest K/S and levelness was obtained at 25 MPa, 120 °C, during 1 h dyeing time which could be regarded as the optimum condition. Moreover, an excellent colour fastness towards washing and rubbing was achieved using the optimum condition.

40

The effects of dye concentration on the functional properties were also investigated [20]. According to the results, all curcumin dyed samples exhibited good UV protection, antioxidant, and antibacterial properties. All curcumin dyed samples provided UV protection factor (UPF) values above 35%, which means a very strong UV protection level according to Australian/New Zealand standard (AS/NZS4399:2017). However, no clear trend between the UPF value dye concentration was observed. All curcumin dyed samples also exhibited UVA and UVB transmittance values of less than 5%, which meets the standard requirement. Results also showed that all the samples treated with curcumin showed a significantly higher antioxidant activity compared with the sample treated in $scCO_2$ without curcumin (non-dyed). The non-dyed polyester exhibited the lowest antioxidant activity (17%) revealing its poor radical scavenging activity compared with curcumin-treated polyester samples. An increase in the antioxidant activity was observed with an increase in the dye concentration attaining an activity of more than 60% using only 1 wt.% curcumin concentration. According to this trend, a further increase in dye concentration could lead to a more efficient antioxidant activity. From the results of antibacterial activity, untreated polyester samples had a poor antibacterial activity with an inhibition rate of around 24% against the E. coli bacteria strain. On the other hand, curcumin-treated polyester fabrics demonstrated higher activity compared with untreated ones. Moreover, the bacteria inhibition increases when the dye concentration increase attaining a bacteria reduction percentage of above 60% using a small amount of curcumin (1 wt.%). However, the level of antibacterial activity achieved in this study seems not enough to be applied as a true antimicrobial fabric. Still, compared with conventional dyeing, the current process highly resource-efficient with the possibility of improving the activity by increasing the dye concentration.

3.3 Functionalization of Polyester Fabric with Photosensitive Dyes in scCO₂ for UV Sensing Smart Textile Application

In this study, two commercial photochromic dyes (Ruby red from Naphthopyran and Sea green from Spirooxazine dye classes) were applied to the polyester fabric to fabricate photochromic fabrics for UV sensing smart textile application. The photochromic behavior of these dyes in solution and applied on polyester fabric has also been investigated [21]. The behavior of photochromic dyes applied to polyester fabric from $scCO_2$ solvent has been compared with those applied using the traditional dyeing and printing methods [22]. According to the results, both photochromic dyes were successfully incorporated into the polyester fabric using the $scCO_2$ dyeing technique resulting good photochromic behavior. Both dyes exhibited reversible colour changing behavior when exposed to UV light and revert to their original colour fast enough when the UV light is removed both in solution as well as applied on polyester fabric. When incorporated into the polymeric matrix, photochromic molecules showed a similar trend of colour strength but contrasting reaction kinetics was observed compared with their behaviors in solution. On the fabric, the sea-green from the spirooxazine family had a slightly higher colour yield, faster coloration, and de-coloration, and poor wash fastness properties compared with the ruby red from the naphthopyran dye class. However, the colour yield and rate kinetics were significantly reduced after washing and abrasion of sea-green dyed samples while the effect was almost negligible for ruby dyed samples. One interesting observation was that the half-lives of fading of both dyes applied from $scCO_2$ solvent were found to be relatively low compared to conventional exhaust and solvent dyeing techniques and are comparable with samples produced with the digital inkjet printing method. Overall, the results showed that supercritical CO_2 dyeing is a viable technique to incorporate photochromic spirooxazine and naphthopyran based dyes into the polyester fabrics which can be used for UV sensing smart textile applications. Photochromic textile samples are shown in Fig. 2 when activated by UVA light torch.

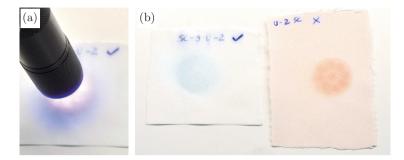


Fig. 2: (a) Activation of the photochromic textiles using a UVA light torch and (b) Locally activated spirooxazine sea green (left) and naphthopyran ruby red (right) samples after washing

4 Conclusion

This study explores the production and characterization of functional and smart textiles using a resource-efficient and eco-friendly $scCO_2$ processing technology. It aimed at investigation of the feasibility of $scCO_2$ processing technology towards sustainable and economic production of functional and smart textiles. The results obtained in this study are encouraging and gives an insight for a wider application of $scCO_2$ technology in the textile processing industry in the future. It will also open more opportunities in innovative green processing of materials useful for the development of new functional products. Based on the obtained results and observation made during the study, the following conclusions have been drawn.

- The results obtained in this study showed the feasibility and significant potential of the scCO₂ impregnation and dyeing technique to produce various functional and smart textile products.
- In this work, suitable functional finishing agents have been identified and successfully applied to polyester fabric using the scCO₂ processing technique.
- An approach towards combining the dyeing and functionalization processes through a single bath (i.e., the dye and functional agent in a single formulation) as well as employing multi-functional colorants was proven effective. This is very important from economic production and environmental safety viewpoints for the designing of sustainable production processes.
- Novel single-step dyeing and functionalization of polyester using green agents such as curcumin and chitosan biopolymers in scCO₂ medium have achieved promising results. Using scCO₂, it was possible to avoid the substrate pre/post-treatment and use of the toxic metallic mordants which otherwise are required procedures in the case of conventional natural dyeing. This will be highly useful in the designing of an innovative and eco-friendly process to develop new functionalized materials in the future.

- The study showed that scCO₂ is a viable technique for dyeing polyester fabric with photochromic spirooxazine and naphthopyran based dyes to produce photochromic smart textiles in a resource-efficient and eco-friendly way achieving faster color ability and fading kinetics compared to exhaust dyed and screen-printing methods and comparable performance with inkjet printing.
- The experimental studies also showed that scCO₂ processing parameters greatly influence the colour and functional performances and an optimum processing condition has been determined. Among the processing variables, pressure and temperature were identified as the two important factors that affect colour and functional performances.

Overall, in this study, an attempt has been made to address important challenges related to the conventional textile dyeing and functionalization processes and new knowledge has been gained to widen the application of $scCO_2$. The obtained results showed that $scCO_2$ processing is a feasible technique to produce functional and smart textiles and could be an attractive alternative to conventional aqueous dyeing and impregnation methods with significant environmental and economic advantages. Thus, adopting this technique is highly important in improving the economics and environmental safety of textile dyeing and finishing processes eliminating wastewater discharge, reducing water, chemical, and energy consumptions, reducing drying and the associated air emissions.

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