



Clean fixation of dye on cotton in supercritical carbon dioxide with a heterogeneous and phase transfer catalytic reaction



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ABSTRACT

A clean fixation of disperse reactive dye on cotton was investigated by employing a heterogeneous and phase transfer catalytic reaction with a phase transfer agent in a liquid-supercritical-solid triphase system, as an environmentally friendly alternative to conventional and heavy polluting wet-chemical process. The catalytic fixation reaction was characterized by color intensity, fixation efficiency and Fourier transform infrared spectrometry (FT-IR), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM). The results show that the dye fixation through a nucleophilic addition at a solid cellulosic polymer substrate phase could be effectively facilitated as the transportation of the anion of OH^- or CO_3^{2-} by the phase transfer catalyst of FC-134 from a liquid phase to supercritical phase and then onto the solid substrate in a self-built reactor. A heterogeneous and triphase transfer catalytic reaction mechanism was also supposed for the dye fixation at the solid substrate in supercritical carbon dioxide fluid with the presence of a base.

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1. Introduction

The fixation of reactive dyes on cotton in conventional wet chemical processes usually involves hard conditions such as in a strong alkaline solution or at a temperature as high as 150 °C–160 °C for baking, etc. Moreover, a large amount of water, chemicals and energy consumptions are involved, and then various colorful and toxic effluents are discharged to environment, which usually cause serious environment concerns. Therefore, it is very important and urgent to develop some effective, useful and environmental friendly methodologies for dye fixation or uptake on cotton substrate with water free and clean media.

Fortunately, supercritical carbon dioxide fluid (SCF- CO_2) as a green, ecological and safety medium has been proved as a promising alternative to the conventional water bath for textile substrate colorations. Furthermore, a catalytic reaction especially for phase transfer catalytic reaction in supercritical carbon dioxide fluid has been paid widely attention in recent years, due to its unique properties and advantages as reaction medium, such as a liquid-like density for dissolving some reactants and gas-like viscosities and diffusivities for achieving an improved mass transfer. Those result

in a high rate of reactivity and selectivity, especially for those reactants from different phases. Moreover, it is most important that the SCF- CO_2 medium is water and organic solvents free, which exhibiting favorable features of environmentally friendly and sustainable production in industry, especially compared with the uptake and fixation reactions of various reactive dyes on a polymeric fiber substrate in conventional processes with a high consumption of water resource and a large amount of effluents discharge.

Up to date, numerous phase transfer catalytic reaction processes in SCF- CO_2 have been well developed and defined [1–3], such as the first phase transfer catalytic reaction in SCF- CO_2 for a nucleophilic displacement of benzyl chloride with potassium bromide by employing a phase transfer agent of tetraheptyl-ammonium bromide (THAB) [2], the intramolecular cycloalkylation of phenyl-acetonitrile with α , ω -dibromoalkanes in SCF- CO_2 medium or other solvents [4], the synthesis of diethyl carbonate from ethanol and SCF- CO_2 in the presence of potassium carbonate by employing a phase transfer catalyst [5], a similar displacement reaction of benzyl chloride with a nucleophile of potassium cyanide in the presence of tetraheptyl ammonium chloride (THAC) [6], as well as other nucleophilic displacements using supported phase transfer agents in SCF- CO_2 [7,8], etc. Furthermore, reports about the modification of polymeric materials and free-radical initiated polymerization by catalysis in SCF- CO_2 are also available recently [9–11], as well as some dispersion polymerization [12], or homogeneous

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catalysis [13], etc. However, most of the demonstrated phase transfer catalytic reactions are simple biphasic system involving SCF-CO₂, such as liquid–solid, supercritical–solid and/or supercritical–supercritical systems. Especially, most of them belong to homogeneous catalytic reactions. But in practice, especially for industrial applications, more phase systems and more complicated phase transfer catalysis are necessary and frequently encountered, such as the dye fixation in a liquid–supercritical–solid triphase system.

Furthermore, the medium of supercritical carbon dioxide fluid is very different from traditional water bath due to its hydrophobic properties. Many hydrophilic materials, especially for some inorganic anions (also as important accelerants for some reactions), such as OH[−], CO₃^{2−}, etc., are insoluble in the hydrophobic supercritical fluid. These result in impossibility for a lot of reactions to occur, such as the nucleophilic addition for reactive dye molecules on a cellulosic substrate, although it is very easy and common in conventional water bath. Therefore, the key point for the fixation of a reactive dye at a cellulosic substrate through covalent bond in supercritical carbon dioxide is how to transfer those anionic accelerants into the hydrophobic medium and then access to the reactants for the reactions to be occurred readily. But unfortunately, all these concerns are still under research, and have not been reported so much in literature, although they are very important for many reactions, and also a crucial basis for the applications of the green medium in textile industry as an alternative to conventional wet-chemical dyeing processes for fiber substrates [14,15].

The purpose of the present work is to investigate the fixation of disperse reactive dye molecules at cotton substrate by employing a heterogeneous and phase transfer catalytic reaction in a triphase system of liquid–supercritical–solid with the presence of perfluoroalkylsulfonyl quaternary ammonium iodides (FC-134), and to get some basic information of a clean fixation for dyes on cotton in a liquid–supercritical–solid triphase system. Moreover, the phase transfer catalytic reaction for dye fixation was characterized and confirmed by color intensity, fixation efficiency and X-ray photoelectron spectroscopy (XPS), Fourier transform infrared spectrometry (FT-IR), scanning electron microscopy (SEM) analysis.

2. Experimental

2.1. Materials and chemicals

A cellulosic substrate, which a fully scoured cotton fabric (a woven, with a fabric weight of 140.0 g m^{−2}) was kindly supplied by Jiangsu Shazhou Printing and Dyeing Co., Ltd. (China) in this study. A 2.0 g of the fabric sample with a dimension of about 10.0 cm × 14.3 cm (height × length) was used in the dye uptake process, and then applied to a phase catalytic reaction for dye fixation. All the substrate samples before subjected to further analysis were extracted by a Soxhlet extraction method with acetone, except an additional indication.

A pure reactive disperse red dye with one vinylsulfone as reactive group (the chemical structure depicted as in Fig. 1), was synthesized and kindly supplied by Everlight Chemical Industrial Corporation (Taiwan of China), and used without further purification. A commercial phase transfer catalyst—Perfluoroalkylsulfonyl quaternary ammonium iodides (FC-134; CAS NO: 1652-63-7;

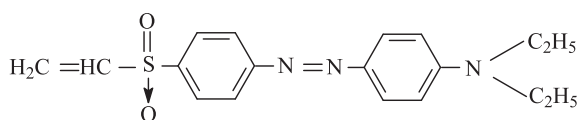


Fig. 1. The chemical structure of the applied disperse reactive dye.

≥98.0 wt.%) was purchased from Wuhan Institute of fluoride (Wuhan of China). Analytical reagent grade acetone was used as a solvent for a Soxhlet extraction of the cellulosic substrate after a phase catalytic reaction to remove the excess dye for evaluation of dye fixation. A pure carbon dioxide gas (99.6 vol.%) was used for the dye uptake process and a phase transfer catalytic fixation reaction in supercritical carbon dioxide system, respectively. Analytical reagent grade sodium carbonate used as a base was supplied by Sinopharm Chemical Reagent Co., Ltd. (Shanghai of China).

2.2. Apparatus and procedures

2.2.1. The uptake of the disperse reactive dye in supercritical carbon dioxide bath

The uptake or adsorption of the vinylsulfone reactive disperse red dye onto the cellulosic polymer samples was carried out in the same batch system as described elsewhere [15], except the dyestuff vessel was not employed in this study.

The vinylsulfone reactive disperse red dye with a ratio of 0.20% o.m.f. (on the mass of fabric substrate), was packed into a filter and hung on the top of the dyeing autoclave that filled with supercritical carbon dioxide fluid, and then dissolved (the packed solid dyes were all dissolved during the dyeing process under all tested conditions according to our previous studies) and diffused into the supercritical fluid when circulated by the syringe pump without an entrainment. The substrate sample for dye uptake in the dyeing autoclave was wrapped around an improved beam as described in detail elsewhere [15].

As the pressure and temperature of the system were attained a request condition of 20.0 MPa and 80 °C, the dye uptake process was begun by circulating the fluid and dissolved dye with a request time ratio of fluid circulation to static dyeing (R_{time}) at 0.10. After a request treatment time of 60 min, the fabric was removed and used for further catalytic fixation reaction experiment. The whole batch system was completely cleaned after every run, and other procedures were described in our previous report [15].

2.2.2. The phase transfer catalytic reaction between the dye and substrate in a triphase system

The catalytic reaction for disperse reactive dye fixation on cotton substrate was performed in a self-built reactor, as shown in Fig. 2. The cotton substrate (a woven cotton fabric (6)) previously adsorbed by the disperse reactive dye molecules (as depicted in Fig. 1) in an uptake process (20.0 MPa, 80 °C, 60 min), was wrapped around a mesh and top-sealed beam [15] which was set at the upper section of the reactor as shown in Fig. 2. An aqueous solution (2) involving a mass fraction of 10% the mixture of FC-134 with sodium carbonate, which were previously mixed with a mole ratio of 1:3, was initially charged into the lower section of the reactor. Then the system was pressurized and heated, and the supercritical carbon dioxide fluid was circulated by a circulation pump from inlet (1) and passed through a three-way pipe (3) in a reverse direction, which two outlets were immersed in the liquid phase for effective ion-exchange and mass transfer between the supercritical and the liquid phases. Then the supercritical fluid bubbled through the liquid phase for picking up the reactant of OH[−] and/or CO₃^{2−} anions by the phase transfer agent in a form of ion complex of FC-134⁺:OH[−] or/and FC-134⁺:(1/2)CO₃^{2−}, and thus penetrated into the polymeric substrate layers (6) after passing through a filter (4) and a mesh beam (7) which connected with a horn shaped cover (5), for the release of the anion reactants on the solid polymer, where the reaction occurred for the dye fixation. An entrainment of the aqueous solution was avoided by the filter (4) and the horn shaped cover (5), as well as by a controlled flow rate of the supercritical fluid. Finally, the supercritical fluid involving the re-freed phase

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