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An industrial scale multiple supercritical carbon dioxide apparatus and its eco-friendly dyeing production

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ABSTRACT

An industrial scale multiple apparatus was designed and built successfully for the dyeing of bobbins in supercritical CO_2 fluid. The system configuration of the industrial scale multiple supercritical CO_2 apparatus and its dyeing procedure for bobbins were described for the first time. Furthermore, production experiments were conducted to determine the feasibility of bobbins dyeing and the stability of the industrial scale supercritical CO_2 apparatus. The dyeing results showed that a satisfactory dyeing effect for the dyed bobbins with commercially acceptable levelness and reproducibility was obtained in supercritical CO_2 dyeing process by employing the industrial scale multiple apparatus. The dyed products also presented excellent color fastness to washing, rubbing and light, which rated at 5, 4–5 and 5–6, respectively. In comparison with the aqueous dyeing process, dyeing of bobbins in supercritical CO_2 fluid with the industrial scale multiple apparatus displays advantages of more environmentally friendly, water and effluents free, as well as energy conservation. Moreover, it is also a novel approach for using industrial emissions of CO_2 instead of water in the textile dyeing process.

1. Introduction

Solvents, and dissolution in general, have been integral to many industries for hundreds of years [1]. At present, most manufacturing and processing industries – automotive, electronics, pulp and paper, chemical, mining and food – depend on the extensive use of solvents. Water is a universal solvent for polar molecules and the most common solvent used by living things and industrial production processes, of which textile industry is one of the biggest consumers of water resources. Traditional aqueous dyeing procedure requires a large amount of water, averagely 100–150 L of water per 1 kg of fiber [2]. Simultaneously, a large amount of dyes and other chemicals were utilized in the dyeing process so that the discharged wastewater contains various kinds of salts, surfactants and unused dyes [3]. With these additives, the wastewater is high toxicity and poor biodegradability, which cannot be treated with conventional biological treatment methods [4]. Given the extremely huge production volume of dyed textile materials (over 50 billion meters in 2015) in China, aqueous dyeing procedure not only leaves large water footprint on the planet but also suffers from the increasing costs of wastewater treatment [5]. Thus, it is highly desirable to develop an anhydrous dyeing procedure to conserve the scarce water resources, reduce the negative environmental impacts and improve the economic sustainability [6].

Considerable efforts have been made to find clean solvents to replace water in textile dyeing in recent years. CO_2 collecting from industrial combustion and fermentation processes, ammonia synthesis and mineral springs, is an attractive solvent alternative for a wide variety of chemical and industrial processes because of its low cost, wide availability, and environmentally friendly and chemically benign nature [7]. Dyeing of textile materials using CO_2 in supercritical state (critical temperature: 31.1 °C, critical pressure: 7.4 MPa) was first introduced into textile field by Pro. E. Schollmeyer in 1988. The first dyeing experiments of PET (polyethylene terephthalate) in supercritical CO_2 were made within an MSc thesis from Ruhr-University of Bochum in close cooperation with G. M. Schneider in 1989 [8]. Since then supercritical CO_2 dyeing for polyester has been the focus of numerous investigations all over the world due to the high diffusivity and solubility, zero surface tension, low energy process and almost no risk of heat deformation for supercritical CO_2 [9]. A. Schmidt examined polyester damage in supercritical carbon dioxide between 100 °C and 160 °C for 1 and 4 h at a pressure of 30 MPa, and disclosed that polyester was the most stable fiber and was not affected in supercritical CO_2 up to 160 °C [10]. J. Dai investigated the polyester fabric dyeing with Disperse Blue 79 in

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supercritical CO_2, and found that the dye uptake increased gradually with the raising of the temperature, reaching equilibrium after 60 min [11]. A. Ferri measured the equilibrium uptake of three disperse azoic dyes in a commercial PET yarn in presence of supercritical CO_2, and observed that the yarn saturation had been achieved for each dye at tested temperature so revealing that dye uptake was more dependent on temperature than pressure [12]. M. V.D. Kraan discussed the influence of temperature and density of the supercritical CO_2 on the dyeing process of polyester in the ranges of 85–125 °C and 400–550 kg/m^3, and showed that increasing the fluid density led to an increasing saturation concentration and a decreasing distribution coefficient. Their results also indicated that the dye adsorption on the polyester followed Nernst adsorption, and the dyeing was exothermic [13]. S. Okubayashi reported dyeing of polyester fabric with C.I. Solvent Blue 35, C.I. Disperse Red 60 and C.I. Disperse Yellow 54 in supercritical CO_2 by employing a mini-plant possessing internal circulator, and declared that the fluid flow was changed by adding a stainless net between the specimen and the cylindrical holder, providing less staining and showing the uniform dyeing effect [14]. T. Abou Elmaaty explored the application of new hydrazonopropanenitrile dyes with potential antibacterial activity in the dyeing of polyester fabrics with supercritical CO_2, and revealed that the optimum dyeing temperature and pressure were 120 °C and 15 MPa, the optimum dye concentration ranges from 2 to 6% o.w.f., and all dyed samples exhibited excellent antibacterial efficiency against G +ve and G −ve bacteria [15]. In addition, in order to explore loose fiber dyeing, our group investigated dyeing performance of polyester fibers with Disperse Red 153 at temperatures of 80–140 °C, pressures of 17–29 MPa and time of 20–80 min, and demonstrated that good dyeing effect, levelness, reproducibility and washing colorfastness of polyester fibers were achieved on the dyeing frame with a favorably increased temperature, pressure and time [16].

Coloration of other synthetic fibers in supercritical CO_2, for instance polypropylene, polyamide 6 and 66, polylactide, poly-m-phenylene isophthalamide and acrylic fibers, has also been conducted [17–20]. Liao et al. analyzed the dyeing performance of polypropylene fibers in supercritical CO_2, and proved the dyeing feasibility of polypropylene using CO_2 as a transport medium [21]. Elmaaty et al. constructed a supercritical carbon dioxide assembly for dyeing Nylon 6 fabric with a series of disperse azo dyes, and found that the color strength of the dyed fabric increased by increasing dye concentration from 2% to 6% o.w.f., and elimination of auxiliary chemicals such as salt, carrier or dispersing agent has no diverse effect on dyeing [22]. Wen et al. investigated the dyeing of polylactide fibers with Disperse Blue 79 in supercritical CO_2, and indicated that a major advantage of dyeing polylactide in supercritical CO_2 over dyeing polylactide in an aqueous medium was its much better retention of the fiber strength [23]. Kim et al. conducted the dry dyeing of NOMEX yarn in a temperature range of 90–150 °C and at pressures of 10–30 MPa with E-types of Disperse Red 60, Blue 56, and Yellow 54 and S-types of Disperse Red 360, Blue 79, and Yellow 114, and obtained the highest color strength (K/S) of the dyed aramid spun yarn at 150 °C and 30 MPa, and demonstrated the adsorption isotherm follows the Langmuir type [24].

Researches on dyeing of natural fibers in supercritical CO_2 have also been reported [25–27]. Sawada et al. investigated the dyeing of natural fibers from a reverse-micellar system in supercritical CO_2 using ammonium carboxylate perfluoropolyether as surfactant, and revealed that wool and silk could be dyed in deep shades with a conventional acid dye even in the absence of an auxiliary, and C.I. Reactive Yellow was adsorbed satisfactorily on cotton fiber but effective fixation was not achieved [28]. Schmidt et al. described the dyeing of natural fibers in supercritical CO_2 with C.I. Disperse Yellow 23 modified with 2-bromoacrylic acid and 1,3,5-trichloro-2,4,6-triazine, and found that using these reactive disperse dyes, the color strengths on wool and silk were higher than on cotton [29]. M.V.F Cid et al. introduced a dyeing method where cotton had been effectively dyed in supercritical CO_2. Their results showed that an outstanding dye fixation of 99% on cotton dyed in supercritical CO_2 was achieved using monofluorotriazine reactive dyes and by adding small quantities of acids [30]. Long et al. developed a dyeing procedure of cotton fabric with a vinylsulfone reactive disperse dye in supercritical CO_2, and demonstrated that significant improvements of dye fixation efficiency and color strength on wet and dry cotton fabrics were achieved by employing the catalyst of TEDA under various conditions [31]. The changes of the physico-chemical structure of different fibers in supercritical CO_2 were also discussed, and found that the supermolecular structure of PET was changed significantly with supercritical pressure and then with supercritical temperature, accompanying with a variation of the crystallinity [32]. Moreover, the effect of depressurization rate on dyeing performance of wool fibers was also studied in the supercritical CO_2 dyeing process, and disclosed that wool fibers were easy to be damaged with depressurization rate increasing [33].

Accompanied with the high temperature and high pressure dyeing process for different fibers, the typical construction of small and medium-sized equipment or industrial apparatus of supercritical CO_2 dyeing is different from the ordinary chemical plant. Therefore, dyeing apparatus plays a crucial role in the development of supercritical CO_2 dyeing technology. The breakthrough and innovation for industrial scale apparatus of supercritical CO_2 dyeing has been the research highlights in the last decades.

In 1989, the first laboratory-scale static supercritical CO_2 dyeing apparatus was constructed in Deutsche Textilforschungszentrum Nord-West e.V. (DTNW) in Germany with a 400 mL autoclave and a stirrable dyeing beam, achieving the anhydrous dyeing of PET with disperse dyes [8]. The semi-technical scale supercritical dyeing apparatus equipped with a stirrable device was then designed by Jasper Gmb H& Co. Velen and DTNW in 1991, containing an autoclave of 67 L for 4 bobbins [34]. Nevertheless, the leveling properties of the dyed yarns and fabrics were poor because the dye liquor circulation cannot occur with the stirrable device. Hence, the supercritical dyeing apparatus with a dyeing liquid circulation system was first constructed under the cooperation of Uhde High Pressure Technologies and DTNW [8]. This apparatus equipped with a gas recovery and recycling system, and had a performance of separation for spinning oils and excess dyes in the dyeing process. However, only one-way circulation could be achieved in the apparatus, thus showing the deficiency of dyeing deviation and uneven color.

In order to solve the above technical problems in the first generation dyeing apparatus, a new supercritical CO_2 dyeing apparatus was built by Uhde High Pressure Technologies at a maximum pressure 30 MPa and temperature 150 °C in 1999 [8]. The main parameters of this new apparatus was controlled automatically, and 3–7 kg polyester filaments can be dyed each time. In addition, a pilot-scale supercritical CO_2 dyeing apparatus with a capacity of 1 PET yarn package was designed by College of Textiles in NC State University in 1996 [35]. In this machine, CO_2 liquid flow was changed by adding a control unit, completing the forward and reverse loop of the liquid flow. Therefore, excellent dyeing effect was obtained, which was similar with the dyeing effect of the traditional aqueous dyeing process. Simultaneously, Applied Sepration Inc. declared that a supercritical fluid machine with an autoclave of 40 L was developed successfully, containing 4 PET yarn packages with the maximum temperature 120 °C and time 40 min [36]. Moreover, another supercritical fluid apparatus funded from European Union was manufactured in 1997 by