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Study of dichlorotriazine reactive dye hydrolysis in siloxane reverse micro-emulsion

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A B S T R A C T

The dyeing ability of the dichlorotriazine dye on cotton fiber in siloxane media was investigated using a water/oil emulsion system. Compared with traditional water base dyeing, a deeper color depth of fabric and 90% fixation rate of dye could be achieved without using salts in siloxane reverse micro-emulsion system. The hydrolysis mechanism of dichlorotriazine dye, which deeply affects the dyeing performance, was analyzed using high performance liquid chromatography and mass spectrometric methods. In siloxane reverse micro-emulsion dyeing system, the lower hydrolysis of dye is probably due to the grasping water ability of surfactant, the reaction between fiber and dye, and the dye aggregation under a high concentration of dye. Furthermore, the hydrolysis model shows that the hydrolysis rate constants in traditional water base was about three to four times faster than the one in siloxane reverse micro-emulsion system. The dye hydrolysis mechanism in siloxane reverse micro-emulsion system in this study could successfully guide the siloxane reverse micro-emulsion dyeing technology.

1. Introduction

Conventional dyeing for textile is generally performed in water base. In such a process, waste aqueous effluent contains hydrolyzed dyes, inorganic salts, alkalis, and other chemicals such as surfactants etc. (Rattanaphani et al., 2007; Khatri et al., 2015; Akbari et al., 2006). Effluents from dyeing cellulose fiber with reactive dye are particularly highly polluted and have a high oxygen demand (Verma et al., 2012; Ali et al., 2014). In order to reduce or even eliminate this problem, new concepts (Banchero, 2013; Tehrani-Bagha and Holmberg, 2013; Haddar et al., 2014; Fu et al., 2013) that avoid use of water are being investigated and evaluated.

Reverse micro-emulsion dyeing is one of representative dyeing techniques (Xie et al., 2011; Sun et al., 2007) to substitute the traditional water base dyeing, which uses an organic solvent as the continuous phase (Wang et al., 2016; Padasala et al., 2016). A major advantage of reverse micro-emulsion dyeing is just need a little water (Petcu et al., 2016), which might influence the hydrolysis of dye and the fixation rate and color fastness (Sawada et al., 2002). Since the hydrolyzed dye has no additional affinity to the fibers, hydrolysis of dye reduces dyeing efficiency and leads to waste of dye and environmental pollution (Cai et al., 2015; Zotou et al., 2002). It is of great importance studying the hydrolytic property of dye in reverse micro-emulsion dyeing system. Compared with traditional dyeing, not only a much higher fixation rate and color fastness can be achieved in reverse micro-emulsion system, but also the dyeing waste water and ecological problems can be decreased. For reverse micro-emulsion dyeing, most of researches were carried out by using hydrocarbon solvents as continuous phase media (Sawada and Ueda, 2003), such as hexane, cyclohexane and *n*-heptane etc, which are not only unfriendly to the environment but also harmful to human.

In our previous work (Fu et al., 2015; Liu et al., 2012; Li et al., 2011; Miao et al., 2013), reverse micro-emulsion dyeing for cellulose fiber with water soluble dye was successfully prepared by taking D5 (Decamethyl cyclopentasiloxane) as a continuous phase. The chemical structure of D5 is shown in Fig. 1. D5 is a clear, odorless, colorless, and non-oily cyclic siloxane fluid, which is widely used in consumer and industrial applications. Researches demonstrated that D5 is safe both to human health and environment (Burns-Naas et al., 1998; Reddy et al., 2007; Jovanovic et al., 2008). In recent years, D5 is applied widely in dry cleaning as a new media (Gao et al., 2007). It was indicated in the previous

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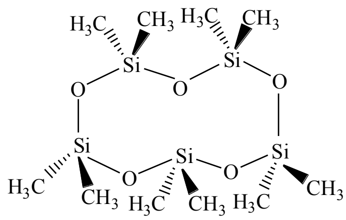

Fig. 1. Chemical structure of D5.

studies that substituting D5 for traditional alkanes was feasible for cellulose fiber dyeing in a reverse micro-emulsion system. The D5 reverse micro-emulsion dyeing technology can achieve a higher dye uptake and a higher fixation than that in the traditional water base dyeing. However, information about why the reactive dye has a higher fixation rate in the D5 reverse micro-emulsion dyeing is limited. In particular, the hydrolysis behavior of reactive dye in the D5 emulsion system is not systematically studied.

In this work, the D5 reverse micro-emulsion system was successfully prepared with nonionic surfactants and co-surfactants, together with a small amount of dichlorotriazine dye aqueous solution. The dyeing performance and the fixation rate of dichlorotriazine dye were analyzed in D5 reverse micro-emulsion dyeing system and compared with conventional water base dyeing. The hydrolysis kinetic of dichlorotriazine dye under different conditions was studied using a HPLC (high performance liquid chromatography) analysis in the D5 reverse micro-emulsion system and conventional water base dyeing, respectively. Impacting factors on the hydrolysis of dichlorotriazine reactive dye from surfactant, co-surfactant, dye concentration, cellulose fiber addition as well as the dyeing temperature were systematically studied.

2. Experimental

2.1. Materials

Fabric: A 100% cotton woven fabric (127.2 g/m^2, yarn count: 40 S × 40 S, yarn density: 146 × 287) was used for the dyeing performance which was collected from Shaoxing Furun Dyeing and Finishing Co., Ltd. D5 (purity>96%) was purchased from GE Toshiba Silicone Ltd. Dichlorotriazine dye, ammonium dihydrogen phosphate and acetonitrile (HPLC grade) were purchased from Sigma-Aldrich. The molecular structure of dichlorotriazine dye is shown in Fig. 2. Alkyl alcohol polyoxyethylene ether (AEO-3) was received

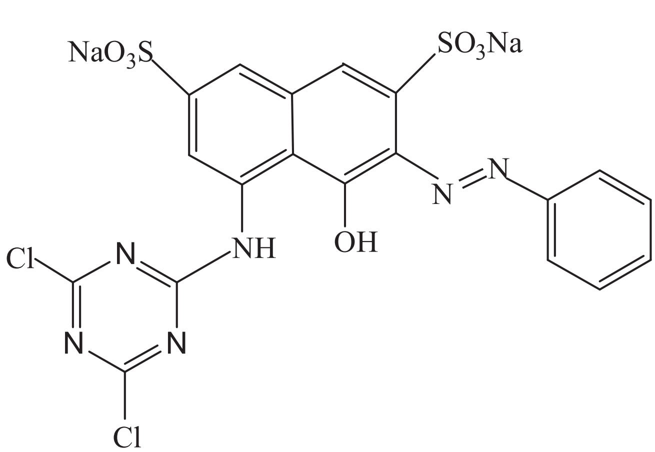

Fig. 2. Structure of dichlorotriazine dye.

from Tianjing Haoyuan Chemical Co., Ltd. Sodium carbonate (A.R.), sodium bicarbonate (A.R.), acetic acid (A.R.), sodium acetate (A.R.), sodium sulfate(A.R.) and *n*-octanol were obtained from Hangzhou Gaojing Chemical Reagent Co., Ltd.

2.2. Preparation of D5 reverse micro-emulsion

AEO-3 (1.25 g) and *n*-octanol (1.25 g) were added in a reactor and mixed evenly (Pei et al., 2017). Then 20 mL D5 was added in the system. After stirring for 10 min, 30 mL D5 was added and stirred for another 10 min. At last, amount of dye solution (2.5 mL) was added to the above mixture and stirred for another 15 min. The system would be transparent, indicating that the D5 reverse micro-emulsion had been prepared.

2.3. Dyeing method

Dyeing was carried out on DYE-24 dyeing machine (ShangHai Chain-Lih Automation Equipment Co., Ltd). Two different dyeing methods are as follows:

D5 reverse micro-emulsion: 5 g cotton fabric was dyed with 2% (o.w.f) dichlorotriazine dye, 7.5% (o.w.f) sodium carbonate and 100% water (o.w.f) at a liquor ratio of 20:1 in D5 media. Dyeing was started for 15 min at room temperature then it was raised to 80 °C at 2 °C/min. Fixation process was carried out at 80 °C for 30 min. After dyeing, fabric sample was washed using tap water, cooked in soap solution under 95 °C for 10 min, and washed thoroughly using warm (50 °C) water and cold water.

Traditional water base: 5 g cotton fabric was dyed with dichlorotriazine dye at a liquor ratio of 20:1 and the concentration of dye was 2% (o.w.f), sodium sulfate and sodium carbonate were 40 g/L and 15 g/L, respectively. Half of sodium sulfate was added into the dye bath after dyeing 10 min at 50 °C and rest in next 10 min. After 30 min, the temperature was raised to 80 °C at 2 °C/min and sodium carbonate was added into dyeing bath, then fixation was carried out for 30 min. After dyeing, fabric sample was soaped and rinsed.

2.4. Dyeing evaluations

The color depth of dyed fabric in percentage was calculated first by determining the reflectance R of the dyed fabric at the wavelength of minimum reflectance (maximum absorbance) on a Datacolor SF600X spectrophotometer. The color yield (*K/S*) value was then calculated by using the Kubelka-Munk equation (Eq. (1)).

$$K/S = (1 - \mathrm{R})/2R \tag{1}$$

Dye fixation is the dye bonding with fiber to the percentage of dye which is added in dyeing solution. The dye fixation rate was evaluated using Eq. (2).

$$E = \left(1 - \frac{C_1V_1 + C_2V_2}{C_0V_0}\right) \times 100\% \tag{2}$$

where: E refers to the dye fixation rate. C_0, C_1, C_2 refer to the concentration of initial dye, the concentration of dyeing residual solution and the concentration of soaping residual solution, respectively. V_0, V_1, V_2 refer to the volume of initial dye solution, the volume of dyeing residual solution and the volume of soaping residual solution, respectively.

2.5. Preparation of the buffer solution

The buffer solutions were prepared to perform the dye