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The effect of tris(2-carboxyethyl)phosphine on the dyeing of wool fabrics with natural dyes

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ABSTRACT

The influence of the introduction of tris(2-carboxyethyl)phosphine on the dyeing of wool with natural dyes was studied and the kinetics and thermodynamics of the dyeing process were also investigated. None of the previous techniques employed report the possibility of modification of wool fibers with tris(2-carboxyethyl)phosphine for dyeing. The modified wool fabrics represented important advances in dye adsorption and decrease in dye desorption at a low dyeing temperature (80 °C) that are not available in the other systems without metallic mordants. Our results demonstrated that the adsorption of natural dyes on wool fabrics was greatly improved through the selective reduction of disulphide bonds with tris(2-carboxyethyl)phosphine. In addition, experiment data was analyzed using pseudo-second order kinetics and Langmuir type isotherms. The results indicated that the diffusion coefficient of dyes was increased while the dyeing entropy and enthalpy were decreased with the introduction of tris(2-carboxyethyl)phosphine.

1. Introduction

Wool, which exhibits the characteristics of feeling soft, heat retention and comfortable to wear, is amongst the earliest natural fibers for textiles and very important for human being to live in the eco-friendly world. Due to this, eco-friendly processing of wool (pre-treatment, dyeing and after-finishing) is critical to the added-value of the resulting materials. Natural dyes are believed to be suitable for the coloration of wool fabrics, because of their generally low toxicity, reduced pollution and biodegradable nature [1–4]. Recently, most researchers have focused on the studies of plant and microorganism-pigments, such as chlorophyll [5], laccaic acid [6], tea pigment [7], prodigiosin [8], rumexmadaio [9], curcumin and brazilin [10], gardenia [11]. Among these, the gardenia has attracted most attentions due to their simple extraction, strong acceleration and good compatibility with other ions, and is believed to be the most promising natural dye for dyeing of wool fibers (the structure is shown in Fig. 1) [12].

Unfortunately, the broad development of natural dyes in dyeing is thwarted by the high molecular mass, which disfavors their penetration into the inner wool fiber, resulting in the decrease adsorption of dyes on the fibers and increase desorption of dyes from the fibers. Nowadays, dyeing mordants have been used to improve dye adsorption on wool fiber and its dyeing fastness. Ferrous sulfate, aluminum sulfate and potassium sodium tartrate have been introduced in the dyeing of wool with chlorophyll and carminum, resulting in the significant improvement of coloration and fastness [5]. Burkinshaw et al., studied the effect of the presence of tannic acid and $FeSO_4$ on the dyeing of wool with C. I. mordant black 8. The results showed that the dye/(tannic acid + $FeSO_4$) systems yielded superior dyeing fastness compared with other dyeing systems [13]. However, due to the lack of an ideal accelerating effect, the resulting dyed fibers based on these mordants have coloration and dyeing fastness that fall far below the expected theoretical values of the resulting fibers. In addition, most mordants used in the dyeing of wool contain metal ions, which affect the coloration of the dyed wool fibers, and are harmful to the environment. Thus, there is a critical need for designing and establishing eco-friendly mordants, which are effective on the dyeing of wool fibers.

Tris(2-carboxyethyl)phosphine (TCEP) is a thiol-reducing agent, which has strong selective reduction for disulfide bonding in proteins [14]. With the introduction of TCEP, the disulfide bonding can be opened, and meanwhile TCEP is oxidized to TCEP oxide. The toxicities of TCEP and TCEP oxide were evaluated using rat hepatocyte and human neuron as models [15,16]. It was proved that both TCEP and TCEP oxide are low in toxicity and environmentally friendly. Thus, TCEP was used in this

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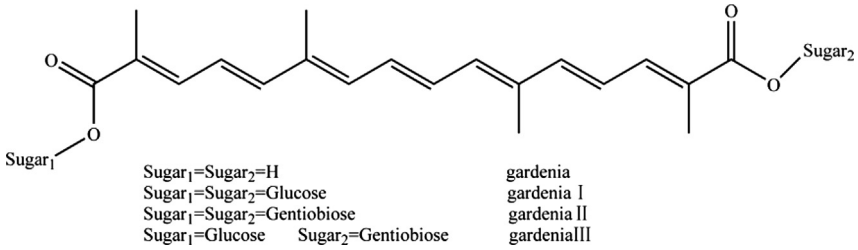

Fig. 1. The chemical structure of mainly components of gardenia.

research. In our work, the effect of the introduction of TCEP on the dyeing of wool fabrics with gardenia yellow at low temperature was studied and the accelerating mechanism of TCEP was also explored. Furthermore, the kinetics and thermodynamics of dyeing process were investigated.

2. Experiment section

2.1. Materials

Wool fabrics with diameter of 19.88 μm were purchased from Zhejiang Xinao Textiles Inc., TCEP were purchased from Tianjin Liankuan Fine Chemicals Co., Ltd, Tianjin, China and Gardenia yellow was obtained from Qianjiang Green Sea Treasury Biotechnology Co., Ltd, Guangdong, China. The Gardenia yellow was extracted with alcohol (content and temperature ~50% and 50 °C, respectively) three times before use. All chemicals used in our work were of laboratory grade.

2.2. Dyeing of wool

Dyeing of wool fabrics was carried out in a dyeing machine (HWX-23, Zhenhe Chemical Instruments Co., Ltd, Zhejiang, China) equipped with timer and temperature controller. A pH meter (FE20-FiveEasy, Mettler-Toledo International Inc.) was used to measure the pH values of the dyeing solutions. The dyeing was carried out using different concentrations (o.w.f%) of gardenia yellow and 0.3% of TCEP, at a material to liquor ratio of 1:1000 and maintaining at a pH value of 4 and temperatures at 80 or 90 °C, respectively.

2.3. Fastness and color testing

The color fastness of the samples to washing and rubbing (wet and dry) was determined using the ISO 105: CO6 2010 and ISO 105: X12 2001test methods, respectively. K/S value of dyed samples was calculated from the reflectance at the wavelength of 450 nm using a spectrophotometer (X-Rite, Gretag Macbeth Colour-Eye 7000A) with UV component included and specular component excluded [17].

2.4. Dyeing kinetics

The dyeing kinetics were studied through the adjusting the dyeing time of wool (5 min, 10 min, 20 min, 30 min, 40 min, 50 min, 60 min, 70 min, 80 min, 100 min, 150 min and 200 min). The liquor ratio was 1:1000. The amount of dyes remaining in the solutions was detected by measuring the absorbance of the solutions using a UV–Vis spectrophotometer (721, Jinghua Scientific Instruments Co., Ltd, Shanghai, China). The absorbance of the solutions was measured and Eq. (1) was used to calculate the amount of dye adsorbed at equilibrium (q_t, mg/g):

$$q_t = 4\% \times \left(1 - \frac{A_t}{A_0}\right) \times 100\% \times 10^3 \quad (1)$$

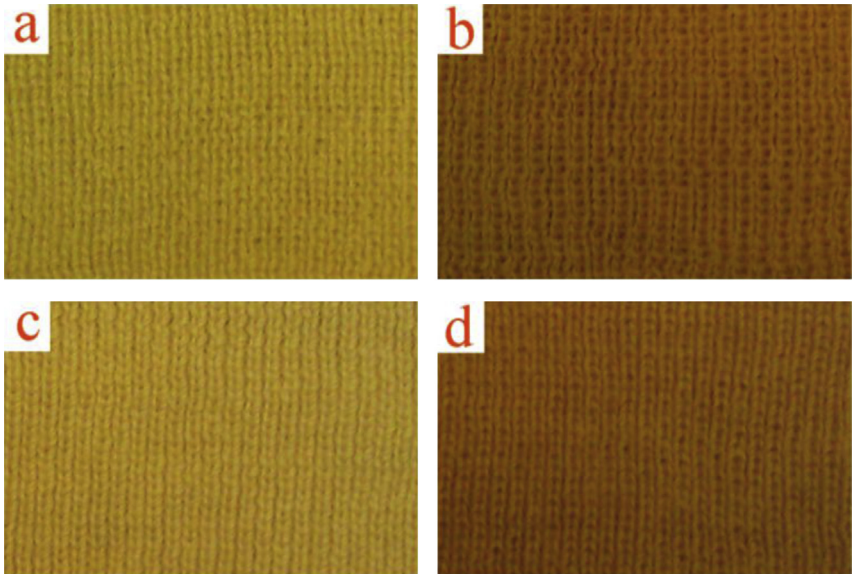

Fig. 2. Various colors of knitted wool fabrics dyed with gardenia yellow. (a) 80 °C without TCEP, (b) 80 °C with TCEP, (c) 90 °C without TCEP, (d) 90 °C with TCEP.

Table 1
The fastness properties of knitted wool dyed with gardenia yellow.

Dyeing method		Wash fastness			Rub fastness	
		c.c.	c.s.	c.w.	Dry	Wet
80 °C	Without TCEP	3	3–4	3–4	3–4	3
	With TCEP	4–5	4	3–4	4	3–4
90 °C	Without TCEP	3–4	4	4	4	3–4
	With TCEP	4	4–5	4	4–5	4

c.c. = colour change; c.s. = colour staining of cotton; c.w. = colour staining of wool.

whereas q_t is the amount of dyes on the fiber at different times (t). A_0 and A_t are the absorbance of the dyeing solutions without and with wool fibers at different times, respectively.

2.5. Dyeing thermodynamics

The studies of the dyeing thermodynamics were realized by the adjusting the amount of the gardenia yellow used. Herein, the dyeing time of wool fabrics was 100 min. After the dyeing process, the wool fibers were cleaned and the cleaning solutions were added into the dyeing solutions together, and the absorbance of total solutions was measured. Eq. (2) was used to calculate the amount of dyes in the residual solutions ($c_{\varepsilon,\ \mathrm{mg/mL}}$):

$$c_\varepsilon = x\% \times \frac{A_\varepsilon}{A_0} \times 100\% \tag{2}$$

where c_ε is the amount of dyes in the residual solutions at dyeing equilibrium; x is the amount of the gardenia yellow used; A_0 and A_ε are the absorbance of original and dyeing solutions at dyeing equilibrium, respectively. The amounts of dyes on wool fiber are calculated by Eq. (1).

3. Results and discussion

3.1. The coloration and fastness of dyed wool fibers

Fig. 2 shows the various colors and colorimetric parameters of wool fabrics dyed with gardenia yellow. As shown in Fig. 2A–D, with the use of TCEP, the dyes wool fabrics (Fig. 2B and D) exhibited deeper color than those without TCEP (Fig. 2A and C). The apparent depth was also expressed through the K/S value. With the introduction of TCEP, the K/S value (80 °C ~ 22.55 and 90 °C ~ 20.15) was higher than that without TCEP (80 and 90 °C ~ 9.23 and 12.14 respectively). It is worthy noticing that the K/S value at a temperature of 80 °C was higher than that at 90 °C, indicating that the dyeing wool fabrics obtained at the temperature of 80 °C exhibited better dyeing depth. It could be inferred that the increase of dyeing temperature had a negative effect on the selective reduction of TCEP.

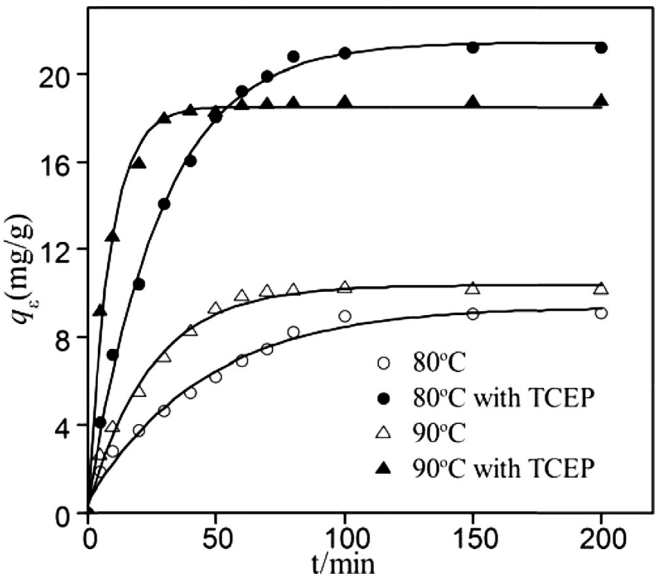

Fig. 4. The adsorption kinetics curves of gardenia yellow.

In addition, the dyed wool fabrics were subjected to tests for fastness towards wash and rub (dry and wet) properties and the results are shown in Table 1. It was observed from the color fastness data that the dyed wool samples with TCEP have shown an improvement in wash fastness ratings (most values are >4) and rub fastness ratings (3–4 for 80 °C and 4 for 90 °C), as compared to wash fastness ratings (3–4 for 80 °C and 4 for 90 °C) and rub fastness ratings (3 for 80 °C and 3–4 for 90 °C) of dyeing wool fabrics without TCEP.

Fig. 3 shows the accelerating mechanism of TCEP for dyeing process. As we know the surface of wool fibers is covered with lamellar scales, which contains lots of –S–S– bonds. With the introduction of TCEP, –S–S– bonds could be broken and –S–H bonds were constructed, resulting in the formation of a loosened structure of wool fibers. So, the adsorption amounts and rate of dyes on wool fiber were improved in the presence of TCEP. Many other agents could also be employed to modify wool fibers, such as dithiothreitol (DTT) [18,19], tri-butyl phosphine (TBP) [20] and tributylphosphine (DTNB) [21]. Comparatively, TCEP exhibits stronger selective-reduction, resulting in the further development of modifications of wool fibers [22].

3.2. Adsorption kinetics of dyeing process

Fig. 4 shows the adsorption kinetics curves of gardenia yellow on the wool fiber at different conditions. The adsorption rate of

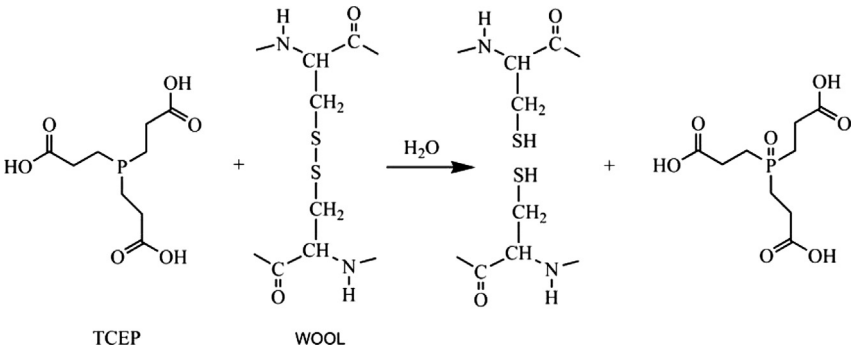

Fig. 3. The effect of TCEP on the structure of wool fiber.

Table 2
The diffusion index of gardenia yellow on wool fiber.

Samples		Diffusion index ($cm^2\ min^{-1}$)
80 °C	Without TCEP	1.83×10^{-9}
	With TCEP	2.65×10^{-9}
90 °C	Without TCEP	3.06×10^{-9}
	With TCEP	6.84×10^{-9}

dyes on the wool fiber was slow at temperature of 80 and 90 °C, and the amounts of dyes on wool fiber were only 9.11 mg/g (80 °C) and 10.16 mg/g (90 °C) at dyeing time of 200 min. With the introduction of TCEP, both the adsorption rate and adsorption amounts of dyes on wool fiber were increased. It was observed that the adsorption of dyes on wool fiber was almost finished before 80 min. The resulting adsorption amounts of dyes could be increased to 21.20 mg/g (80 °C) and 18.75 mg/g (90 °C), and afterwards the equilibrium was reached. The results demonstrated that TCEP played a critical role in the improvement of the adsorption amounts and rate of uptake of the dyes on wool fiber.

The diffusion index is important parameter that indicates the penetration and diffusion capability of dyes on wool fiber. Herein, the diffusion index can be calculated according to Hill formulation [23]:

$$\frac{q_t}{q_\varepsilon} = 4\sqrt{\frac{Dt}{\pi r^2}} \tag{3}$$

where q_t (mg/g) and q_ε (mg/g) is the dye exhaustion at different times (t) and at the equilibrium, respectively; r (cm) is the radius of fibers, and D (cm^2/min) is the diffusion index.

Table 2 shows the diffusion index results. As shown in the table, the diffusion index of dyes on wool fiber was 1.83×10^{-9} cm^2 min^{-1} at 80 °C. With the introduction of TCEP, it was increased by 42% and reached 2.65×10^{-9} cm^2 min^{-1}. At a dyeing temperature of 90 °C, the diffusion index had a value of 3.06×10^{-9} cm^2 min^{-1} but increased to 6.84×10^{-9} cm^2 min^{-1} after the introduction of TCEP. The results indicated that TCEP had an important effect on the diffusion index of natural dyes on wool fiber.

3.3. The kinetics model of dyeing process

In order to investigate the adsorption mechanism of gardenia yellow on wool fiber, two kinetic models were used, including the pseudo-first order model (4) and pseudo-second order model (5):

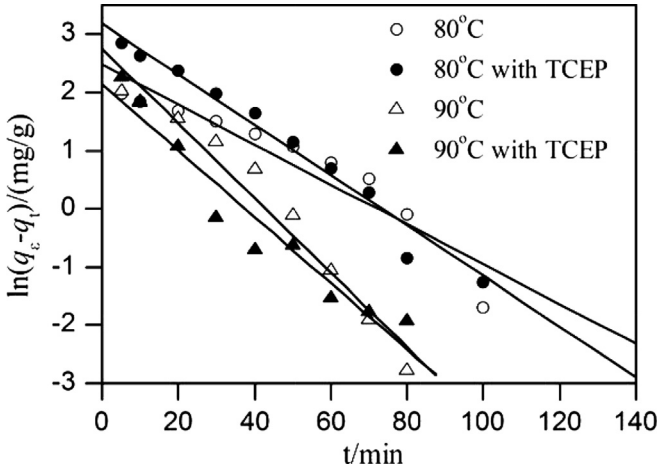

Fig. 5. The pseudo-first order model of gardenia yellow on wool fiber.

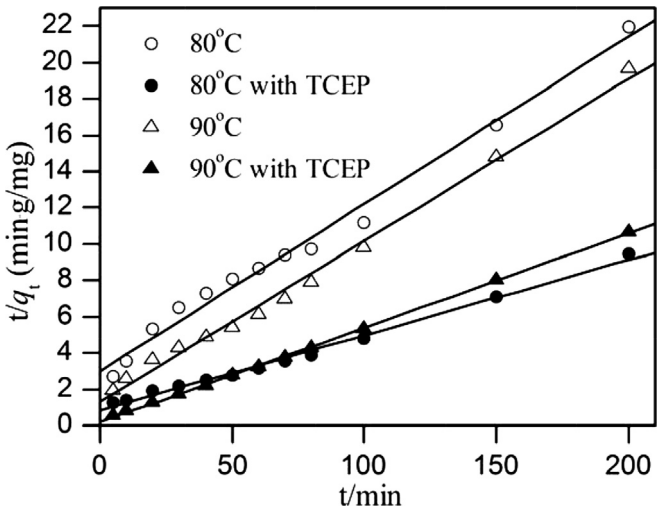

Fig. 6. The pseudo-second order model of gardenia yellow on wool fiber.

$$\ln(q_\varepsilon - q_t) = \ln q_\varepsilon - k_1 t \tag{4}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_\varepsilon^2} + \frac{1}{q_\varepsilon} t \tag{5}$$

where q_ε is the adsorption amount of dyes on wool fiber when the equilibrium was reached (mg/g); q_t is the adsorption amount of dyes on wool fiber at different times (mg/g); k_1 and k_2 are the rate constant of first and second order adsorption of dyes on wool fiber, respectively.

Firstly, plotting the ln (q_ε−q_t) against t, a line about the pseudo-first order model can be obtained as shown in Fig. 5 and k_1 can be calculated according to the slope of the line. In addition, another line about the pseudo-second order model can be obtained through the plotting the t/q_t against t (Fig. 6). The fitness index (R^2), which can be obtained through the fitness of the experimental data, is determined to the anastomosis degree of data and models. As shown in Fig. 5, the anastomosis degree of the former of the experimental data and the fitness line of pseudo-first order model was higher than that of the latter of the experimental data and the fitness line of pseudo-first order model. After the addition of TCEP, the total deviation degree of became smaller. On the contrary, the anastomosis degree of the latter of the experimental data and the fitness line of pseudo-second order model was better and it was almost identical after the addition of TCEP (Fig. 6). The results indicated that the adsorption process of dyes on wool fiber could be changed by using the TCEP.

Table 3 shows the adsorption parameters of dyes on wool fiber of different models. As shown in Table, the fitness index of the

Table 3
The adsorption parameters of gardenia yellow on wool fiber of different kinetics models.

Samples		Pseudo-first-order model			Pseudo-second-order model		
		k_1 (min^{-1})	q_e (mg/g)	R^2	k_2 (g/mg min)	q_e (mg/g)	R^2
80 °C	Without TCEP	3.42×10^{-2}	11.84	0.9454	2.84×10^{-3}	10.84	0.9878
	With TCEP	4.33×10^{-2}	24.06	0.9864	2.09×10^{-3}	24.08	0.9931
90 °C	Without TCEP	6.42×10^{-2}	15.66	0.9628	6.19×10^{-3}	11.23	0.9932
	With TCEP	5.70×10^{-2}	8.47	0.9371	15.6×10^{-3}	19.18	0.9995

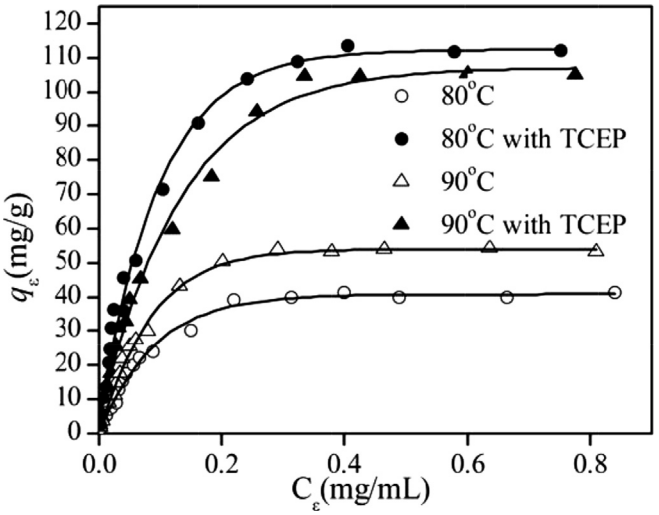

Fig. 7. The adsorption isotherms curves of gardenia yellow on wool fiber.

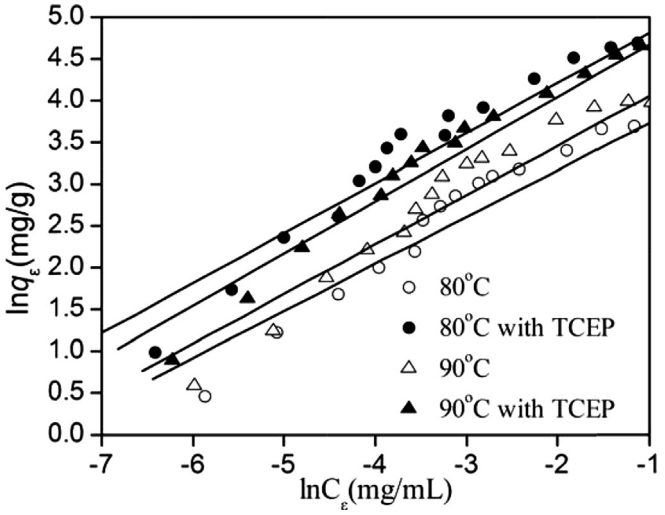

Fig. 9. The Freundlich model of gardenia yellow on wool fiber.

pseudo-first order model was very low (80 and 90 °C ~ 0.9454 and 0.9864). It is worthy to note that the fitness index was increased when the experimental data were calculated using the pseudo-second order model, R^2 at 80 and 90 °C were 0.9878 and 0.9931 respectively. Especially, the fitness index was 0.9932 ~ 80 °C and 0.9995 ~ 90 °C after the introduction of TCEP. The results demonstrated that the adsorption process of gardenia yellow on wool fiber under the accelerating effect of TCEP could be fitted the pseudo-second model very well.

As far as we are aware, the adsorption of dyes on wool fiber is mainly due to the localized adsorption between the charges of dyes and fibers. The results, which the fitness index (R^2) between experimental data and theoretical models was less than 0.98, showed that TCEP molecules play an important role in the promotion of electron transfer.

3.4. Adsorption isotherms of dyeing process

In order to further explore the adsorption isotherms of gardenia yellow on wool fiber, two parameter equations including Langmuir and Freundlich equation were used to analyze the experiment data as shown in Fig. 7.

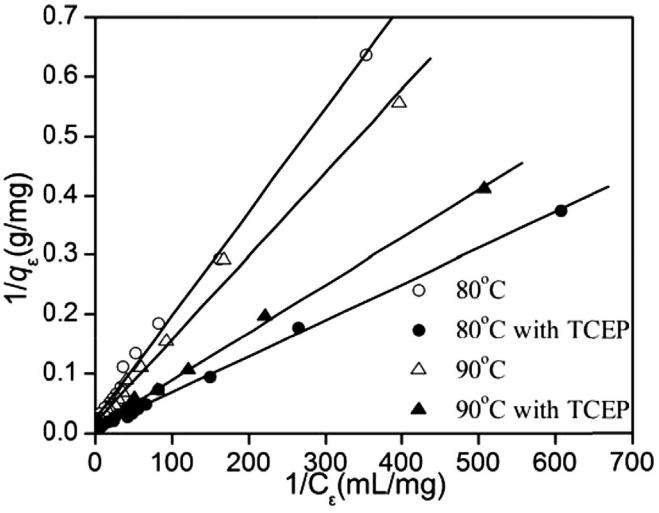

Fig. 8. The Langmuir model of gardenia yellow on wool fiber.

The Langmuir Eq. (6) is an important model for the analyzing of the adsorption process of dyes:

$$\frac{1}{q_\varepsilon} = \frac{1}{Q_L} + \frac{1}{K_L Q_L C_\varepsilon} \tag{6}$$

where q_ε (mg/g) and C_ε (mg/mL) are the adsorption amount of dyes on wool fiber and the concentrations of dyes in final solutions at equilibrium, respectively; Q_L (mg/g) is the amount of the dye on per unit weight of wool fiber to form a monolayer coverage on the surface bound at equilibrium; K_L is the constant related to the localized adsorption.

The line could be obtained through the plotting $1/q_\varepsilon$ against $1/C_\varepsilon$ (Fig. 8), and Q_L and K could be calculated from the intercepts and slopes of the line. The index (R^2) was used to evaluate the fitness of experiment data and thermodynamic models.

In addition, the Freundlich Eq. (7) is another important model about the adsorption process of dyes.

$$\ln q_\varepsilon = \ln Q_f + \frac{1}{n} \ln C_\varepsilon \tag{7}$$

where Q_f (mg/g) and $1/n$ are the adsorption capacity and adsorption intensity of fibers, respectively, and Q_f and n can be calculated from the intercepts and slopes of the line of ln q_ε versus ln C_ε as shown in Fig. 9.

Table 4 shows the parameters of gardenia yellow on wool fiber of thermodynamics models. As shown in table, for Langmuir model, the adsorption capacity (Q_L) at 80 and 90 °C were 41.84 mg/g and 55.31 mg/g respectively. With the introduction of TCEP, the adsorption capacity increased to 118.91 mg/g (80 °C) and 104.93 mg/g (90 °C). For Freundlich model, the adsorption capacity

Table 4
The adsorption parameters of gardenia yellow on wool fiber of two thermodynamics models.

Samples		Langmuir model			Freundlich model		
		Q_L (mg/g)	K_L(mL/g)	R^2	Q_f (mg/g)	n	R^2
80 °C	Without TCEP	41.84	13.74	0.9953	73.63	1.77	0.9118
	With TCEP	118.91	13.85	0.9970	224.01	1.67	0.9278
90 °C	Without TCEP	55.31	12.79	0.9923	104.34	1.69	0.9102
	With TCEP	104.93	11.91	0.9990	199.14	1.60	0.9506

Table 5
The thermodynamics parameters of gardenia yellow on wool fiber.

Samples		$-\Delta G^\circ$ (kJ)	ΔH° (kJ/mol)	ΔS° (J/mol K)
Without TCEP	80 °C	7.69	−8.09	−1.08
	90 °C	7.69		
With TCEP	80 °C	7.71	−16.13	−23.81
	90 °C	7.48		

of dyes was elevated more obviously. The value of Q_f was changed from 73.63 mg/g (80 °C) and 104.34 mg/g (90 °C) to 224.01 mg/g (80 °C) and 199.14 mg/g (90 °C). It can be concluded that the addition of TCEP could enhance the adsorption capacity of the dye on the fiber of both models very effectively.

In addition, the fitness index (R^2) of different samples calculated from the Langmuir model exceeded 0.99. However, they were decreased when the model was changed to Freundlich model. Thus, the adsorption process of gardenia yellow on wool fiber showed better agreement with the Langmuir model.

The thermodynamic parameters, including free energy change (ΔG°), enthalpy change (ΔH°) and entropy change (ΔS°), were evaluated using the Eqs. (8)–(10):

$$\Delta G^\circ = -RT\ln(K_L) \tag{8}$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{9}$$

$$\ln(K_L) = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \tag{10}$$

where R is the universal gas constant (8.314 J mol^{-1} K^{-1}) and T is the absolute temperature in K. Enthalpy change ΔH^o and entropy change ΔS^o are determined from the slope and intercept of the plot $1/T$ versus ln K_L [24].

Table 5 shows the thermodynamics parameters of gardenia yellow on wool fiber. Herein, the negative values of ΔG° indicated that the adsorption process of dyes on the fiber was spontaneous. With the introduction of TCEP, the negative values of ΔG° became bigger, indicating that the driving force of the adsorption process was increased. The entropy change (ΔS°) represents the entropy difference of the systems when the dyes are adsorbed from solutions to fibers. The entropy of the system with TCEP showed smaller negative values, which represented the better orientation of dye molecules on wool fiber. The enthalpy change (ΔH°) values were observed to be also negative, which indicated the adsorption process was exothermic. With the addition of TCEP, the enthalpy of the system became smaller than that without TCEP, indicating that the degree of the combination between dyes and fibers was higher. Our results in Table 5 demonstrated that the adsorption process of gardenia yellow on wool fiber with TCEP was similar to that of acid dyes on wool fiber.

4. Conclusions

In this work, TCEP was introduced into the adsorption process of natural dyes on wool fiber. Through the strong selective reduction for the disulfide bonding of wool fibers, TCEP could improve the adsorption amount and rate of dyes on the fiber at low temperatures (80 °C). The results from the kinetic studies indicated that the localized adsorption between natural dyes and wool fibers was strengthened with the introduction of TCEP. The experimental data showed better agreement with a pseudo-second model. Moreover, the thermodynamics results demonstrated that the adsorption capacity of dyes on wool fiber was increased with the addition of TCEP. The adsorption process of gardenia yellow on wool with the aid of TCEP was fitted the Langmuir model very well.

Acknowledgments

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