Study of the Disperse Dyeing Properties of Low-Temperature Dyeable Polyesteramide Fibre

Raziskava lastnosti nizkotemperaturnega barvanja poliesteramidnih vlaken z disperznimi barvili

Abstract
Polyethylene terephthalate (PET) is the most important synthetic fibre, and is widely used in the textile industry. However, the disperse dyeing of PET fibre must be carried out at a high temperature and under high pressure. This leads to high energy consumption and damage to the wool in a PET/wool blend. Some co-polymesters and novel polyesters that can be dyed under normal pressure have proven to be good alternatives to PET. In this work, the low-temperature dyeable polyesteramide fibre was used, and its disperse dyeing properties were studied in terms of adsorption isotherms, the temperature-dependence of uptake, uptake rate, migration ability, building-up property, and colour fastness of disperse dyes. The adsorption isotherms of disperse dyes on polyesteramide fibre followed the Nernst adsorption model. Disperse dyes exhibited high exhaustion at 100 °C, indicating the good dyeability of polyesteramide fibre under atmospheric pressure. The uptake of dyes by polyesteramide fibre was considerably faster than that of PET fibre, while the majority of dye uptake occurred in the temperature range of 70 to 90 °C. Azo disperse dyes exhibited higher adsorption saturation and better a building-up property than anthraquinone dyes. Disperse dyes had a good migration ability on polyesteramide fibre, and the colour fastness of the dyed polyesteramide fabrics was also satisfactory.

Keywords: dyeing under normal pressure, partition, adsorption, dyeing rates, energy saving

Povzetek
Polietilentereftalat (PET) je najpomembnejše sintetično vlakno, ki ga v tekstilni industriji uporabljajo za različne namene. Barvanje vlaken iz PET z disperzijskimi barvili poteka le pri visoki temperaturi in visokem tlaku, kar predstavlja visoko porabo energije ter povzroči poškodbe volne v mešanicah PET/volna. Nekatera kopoliesterska vlakna in vlakna iz novih tipov poliesterov, ki so obarvilja pri normalnem tlaku, so se izkazala kot dobra alternativa vlaknom PET. V članku je predstavljeno nizkotemperaturno barvanje poliesteramidnih vlaken, raziskovane so bile adsorpcijske izoterme, temperatura odvisnost navzemanja, hitrost navzemanja, sposobnost migracije in barvna obstojnost disperzijskih barvil. Adsorpcijske izoterme disperznih barvil na poliesteramidna vlakna sledijo Nernstonovemu adsorpcijskemu modelu. Disperzna barvila so pokazala visoko izčrpanje pri 100 °C, kar je razkrilo dobro obarvanje poliesteramidnih vlaken pri atmosferskem tlaku. Vlakna iz poliesteramida so veliko hitreje navzemala barvila kot vlakna iz PET, večina barvila se je adsorbirala v temperaturnem območju od 70 do 90 °C. Azo disperzijska barvila so dala višjo nasičenost in adsorpcijo kot antrakinonska barvila. Disperzna barvila so pokazala dobro sposobnost migracije v poliesteramidna vlakna. Tudi barvna obstojnost poliesteramidnih pletiv je bila zadovoljiva.

Ključne besede: barvanje pri normalnem tlaku, porazdelitev, adsorpcija, hitrost barvanja, prihranek energije
1 Introduction

Textile processing uses an enormous amount of electricity, fuel, water and chemicals, and produces a significant amount of contaminated effluent [1–4]. Reducing energy and water consumption, as well as hazardous industrial effluents, represents the biggest sustainability challenge for the textile industry. In order to reduce energy consumption, low-temperature scouring and dyeing, and emerging techniques have become part of the industry’s strategy, and attracted a great deal of attention [1, 2, 5].

Polyethylene terephthalate (PET) is the most important synthetic fibre, and is predominant on the man-made fibre market [5]. PET fibre has been widely used in clothing, home textiles and other industries due to its numerous outstanding properties, such as high strength, good thermal and chemical stability, and excellent wrinkle resistance, as well as its relatively low price [6]. However, it also has some shortcomings, such as low moisture regain, poor antistatic properties and poor dyeability due to its high structural regularity and crystallinity, lack of reactive dyeing sites and polar groups, and high hydrophobicity [6]. The biggest disadvantage of the wet processing of PET fibre is that it must be dyed under a high pressure at 125–130 °C in a weakly acidic condition, or at about 110 °C in the presence of carriers. High-temperature dyeing not only consumes a great deal of energy, but also requires expensive dyeing equipment and causes potential risks. When used, most carriers are associated with toxicological issues [7]. In addition, the high-temperature dyeing of a PET/wool blend leads to the damage of the wool. The low-temperature dyeing of PET is a very difficult task if conventional dyes and chemicals are used. One successful approach to address this issue is to modify PET fibre for enhanced dyeability. The introduction of third and fourth monomers during the stage of polyester synthesis is a basic strategy. The copolymerisation of PET with dimethyl 5-sulfoisophthalate sodium salt as the third monomer confers cationic dyeability to PET fibre [8]. The addition of the fourth monomer, such as polyethylene glycol, 1,3-propanediol, 2-methyl-1,3-propanediol, or 2,2-dimethyl-1,3-propanediol, further improves the dyeability of PET fibre at the boiling point under normal pressure [8]. Today, cationic dyeable polyester (CDP) is deemed an industrial success. On the other hand, the application of new polyester polymers, such as polytrimethylene terephthalate (PPT), polybutylene terephthalate (PBT) and polylactic acid (PLA), is also an effective method for obtaining low-temperature dyeable polyester fibres [1, 5, 9].

In recent years, a new type of polyesteramide copolymer has been developed as an alternative to PET [10, 11]. Polyesteramide copolymer can be synthesised through the polycondensation reaction of ethylene glycol terephthalate and aliphatic amide [10, 11], and has both ester and amide blocks on its backbone. Polyesteramide fibre can be manufactured by using the melting spinning of polyesteramide chips. Previous researchers have found that while polyesteramide fibre has a lower tensile strength and whiteness than PET fibre [10–12], it possesses better dyeability, softness and anti-pilling properties [13, 14]. Very importantly, the incorporation of amide groups into an ester main chain results in a decrease in structural regularity and an increase in the amorphous region of the fibre. The glass transition and melting temperatures of polyesteramide fibre are 68–72 °C and 235 °C, respectively, which are lower than those of PET fibre [13]. These factors make polyesteramide fibre dyeable using disperse dyes at the boiling point under normal pressure.

The development of polyesteramide fibre provides the possibility of manufacturing pure polyester textiles as well as natural fibre (e.g. wool, silk and cotton) blends using a low-temperature dyeing technique, with the added advantage of energy savings. Although preliminary studies demonstrated the disperse dyeability of polyesteramide fibre [13, 14], more detailed dyeing properties need to be studied with the aim of better understanding the mechanism of disperse dyeing, and providing assistance in the selection of dyes and the determination of dyeing conditions. In this work, the adsorption isotherms of disperse dyes on polyesteramide fibre were studied in order to understand the mechanisms of dyeing, and the temperature-dependence of uptake, uptake rate, migration ability, build-up property and colour fastness of disperse dyes on polyesteramide fibre were assessed.

2 Materials and methods

2.1 Materials

Polyesteramide knitted fabric (178 g/m²) was supplied by Sinopec Yizheng Chemical Fibre Co. Ltd.,
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China. Fine denier PET knitted fabric (182 g/m²) was obtained from Suzhou TA&A Ultra Clean Technology Co., Ltd., China. In order to remove the finish oils added to the fibres during the spinning process, the fabrics were treated in a scouring bath containing 4 g/L sodium carbonate and 2 g/L Leveler O at 50 °C for 60 minutes. After scouring, the fabrics were thoroughly rinsed in distilled water and allowed to dry in the open air.

Disperse dyes were selected based on their chemical structures, energy levels, colours and applicability. The characteristics of the dyes are summarised in Table 1. Longsperse and Terasil dyes were provided by Zhejiang Longsheng Group Co. Ltd., China, and Huntsman International LLC (Shanghai Division), respectively, while other dyes were obtained from Zhejiang Runtu Co. Ltd., China. Sodium carbonate, citric acid, disodium hydrogen phosphate, sodium hydrosulphite and acetone were of analytical grade. Dispersant NNO was an industrial product from Anyang Double Circle Auxiliary Co. Ltd., China. Leveler O (polyoxyethylene alkyl ether) was provided by Jiangsu Hai’an Petrochemical Plant, China.

<table>
<thead>
<tr>
<th>Trade name</th>
<th>C.I. disperse</th>
<th>Energy level</th>
<th>Structure class</th>
</tr>
</thead>
<tbody>
<tr>
<td>Disperse Yellow Brown S-2RFL</td>
<td>Orange 30</td>
<td>High</td>
<td>Azobenzene</td>
</tr>
<tr>
<td>Disperse Scarlet S-BWFL</td>
<td>Red 74</td>
<td>High</td>
<td>Azobenzene</td>
</tr>
<tr>
<td>Longsperse Blue SE-2R</td>
<td>Blue 183</td>
<td>Medium</td>
<td>Azobenzene</td>
</tr>
<tr>
<td>Disperse Navy H-GL</td>
<td>Blue 79</td>
<td>High</td>
<td>Azobenzene</td>
</tr>
<tr>
<td>Disperse Brown 3R</td>
<td>Brown 1</td>
<td>High</td>
<td>Azobenzene</td>
</tr>
<tr>
<td>Disperse Red 3B</td>
<td>Red 60</td>
<td>Low</td>
<td>Anthraquinone</td>
</tr>
<tr>
<td>Terasil Pink 4BN</td>
<td>Red 11</td>
<td>Low</td>
<td>Anthraquinone</td>
</tr>
<tr>
<td>Disperse Violet HRFL</td>
<td>Violet 31</td>
<td>High</td>
<td>Anthraquinone</td>
</tr>
<tr>
<td>Disperse Blue 2BLN</td>
<td>Blue 56</td>
<td>Low</td>
<td>Anthraquinone</td>
</tr>
</tbody>
</table>

2.2 Dyeing methods

All dyeing processes were carried out in a laboratory using an infrared dyeing machine (FAD-7-18P; Yabo Textile Machinery Co. Ltd., Wuxi, China). The weight of the fabrics used for dyeing was 1 g, and the liquor ratio was 50:1. The dye solutions consisted of dyes, buffer and Dispersant NNO (1 g/L). The pH of the dyeing bath was adjusted to 5 by adding a McIlvaine buffer (citric acid and disodium hydrogen phosphate). At the end of dyeing process, polyesteramide fabrics were rinsed with distilled water and then dried in the open air.

Adsorption isotherm. Polyesteramide fabrics were dyed in solutions containing 0.5-12% owf (on the weight of fabric) dyes. The temperature was raised at the rate of 1.5 °C/minute from 30 to 80 °C prior to the addition of fabric samples, and then raised to 100 °C at the rate of 3 °C/minute. The dyeing process was continued at 100 °C for 150 minutes until the dye adsorption reached the equilibrium state.

Dyeing temperature. In order to study the temperature effect of dye uptake, the dyeing process was carried out according to the procedure illustrated in Figure 1. The dyeing solutions contained 2% owf dyes, 1 g/L Dispersant NNO and a buffer. The time for the dipping of each fabric in the dye solution was 120 minutes.

Figure 1: Dyeing profile for polyesteramide fabric at various temperatures

Dyeing rate. The dyeing rates of polyesteramide fibre were characterised by the uptake rates of Disperse Orange 30 for polyesteramide fibre in the conditions of 2% owf dyes, 1 g/L Dispersant NNO and pH 5. Two heating rates (2 °C/minute and 1 °C/minute) and two final holding temperatures (120 °C and 100 °C) were used. For the purpose of comparison, the uptake rate
of Disperse Orange 30 for PET fibre was also determined.

**Building-up properties.** The building-up properties of various dyes on polyesteramide fabrics were measured in a dye concentration range from 0.5% to 8% owf. The dyeing process was carried out at 100 °C for 80 minutes according to the profile illustrated in Figure 1.

### 2.3 Measurements

**Exhaustion and adsorption of disperse dyes.** The absorbance of dye solutions was measured using a Shimadzu UV-1800 UV–Vis spectrophotometer (Shimadzu Co. Ltd., Japan). The exhaustion of disperse dyes was assessed using the colourimetric method. Due to the poor water solubility of disperse dyes, a mixture of aqueous dye solution and acetone at a ratio of 30:70 (v/v) was prepared before the colourimetric analysis. The exhaustion percentage ($E$) of disperse dyes was calculated using equation 1:

$$E = \frac{A_0 - A_1}{A_0} \times 100 \ (%) \quad (1),$$

where $A_0$ and $A_1$ are the absorbance at the maximum absorption wavelength of the dye solution before and after exhaustion, respectively.

The quantity of disperse dyes ($C_f$) on polyesteramide fibre was calculated using the exhaustion percentage of dyes, the initial dye concentration and the weight of the fabric using equation 2:

$$C_f = \frac{W_d \times E}{W_f} \times 1000 \ (mg/g) \quad (2),$$

where $W_d$ is the initial dye weight (g) determined by fabric weight (1 g) and dye dosage (% owf) and $W_f$ is the fabric weight (1 g).

The quantity of disperse dyes ($C_s$) in the solution after dyeing was calculated using equation 3:

$$C_s = \frac{W_d \times (1 - E)}{V} \ (g/L) \quad (3),$$

where $V$ is the volume of the dye bath (L).

**Migration properties.** Two undyed fabrics (A and B) with the same weight were prepared. Sample B was dyed with 2% owf dyes at 100 and 120 °C. Afterwards, dyed sample B and undyed sample A, at a liquor ratio of 50:1, were immersed in a blank dyeing bath that consisted of 1 g/L Dispersant NNO and a buffer, without the addition of dyes. In order to carry out the dye migration test, the temperature was raised at a rate of 2 °C /minute from 30 to 100 and 120 °C. At this temperature, the dye migration was continued for 60 minutes, after which the temperature was lowered to 80 °C. The migration rate was calculated using equation 4:

$$\text{Migration} = \left( \frac{K}{S}_{A} \right) \times 100 \ (%) \quad (4),$$

where $(K/S)_A$ and $(K/S)_B$ are the colour depth values of samples A and B, respectively after the migration test.

**Colour characteristics.** The colour depth ($K/S$ value) of each dyed sample was measured using a HunterLab UltraScan PRO reflectance spectrophotometer at the maximum absorption wavelength. A D65 illumination and 10° standard observer were used. Each sample was folded twice to give it a thickness of four layers.

### 3 Results and discussion

#### 3.1 Adsorption isotherm of disperse dyes on polyesteramide fibre

The adsorption isotherms of disperse dyes can be described by the relationship between the adsorption quantity of dyes on polyesteramide fibre ($C_f$) and the concentration of dyes in a solution ($C_s$) at equilibrium. Figure 2 shows the adsorption isotherms of two azo dyes and two anthraquinone dyes. There was a clear linear relationship between $C_f$ and $C_s$, suggesting that the adsorption of disperse
dyes on polyesteramide fibre follows the Nernst model, and hydrogen bonding and the van der Waals forces between dyes and fibres contribute to Nernst adsorption. This observation is inconsistent with the mechanism of the distribution of disperse dyes on PET and the aqueous phase [15]. Moreover, it is evident from Figures 2a and 2b that azo dyes (Disperse Orange 30 and Red 74) did not exhibit a saturation adsorption in the range of the used dye concentration, while the adsorption saturation of the two anthraquinone dyes (Disperse Red 11 and Violet 31) approached the range of 50–60 mg/g. It is apparent that azo dyes had much higher adsorption saturation than anthraquinone dyes.

Figure 2: Adsorption isotherms of (a) azo and (b) anthraquinone disperse dyes on polyesteramide fibre at 100 °C

3.2 Dyeing temperature of polyesteramide fibre

It is a well-known fact that dyeing temperature plays a key role in the disperse dyeing of PET fibre because it affects the exhaustion, uptake rate, migration and diffusion of dyes, the colour depth and colour fastness of dyeing, and the efficiency of the dyeing process. High-temperature dyeing is typically used to achieve the high uptake of disperse dyes and the high colour depth of PET fibre because of the higher kinetic energy of dye molecules and the greater segmental mobility of the less-ordered regions within the fibre [16]. Polyesteramide fibre has lower glass transition and melting temperatures than PET fibre [13], and thus a lower dyeing temperature is to be expected. Figure 3 shows the effect of dyeing temperature on the uptake of disperse dyes by polyesteramide fibre. The exhaustion of the five disperse dyes increased with the raising of the temperature, and almost reached the maximum rate at 100 °C, irrespective of their energy level or structure class. A further increase in dyeing temperature did not improve the exhaustion of dyes, with the exception of Disperse Red 74. This finding shows that the dyeing of polyesteramide fibre can be carried out under normal pressure. The good dyeing property of polyesteramide fibre is attributable to the fact that the incorporation of amide backbones into the polyester chain decreases the structural regularity of the fibre, as well as the glass transition temperature of the fibre [13]. This study implies that the low-temperature dyability of polyesteramide fibre, as a novel synthetic fibre, has tremendous advantages in terms of energy consumption and dyeing efficiency.

Figure 3: Uptake of disperse dyes by polyesteramide fibre at various temperatures

3.3 Dyeing rate of polyesteramide fibre

It is also a well-known fact that there are four fundamental steps involved in the uptake of dyes by textile fibres: the diffusion of dyes in the external water phase toward the diffusional boundary layer.
Figure 4 shows the uptake rates of Disperse Orange 30 for polyesteramide and PET fibres at a holding temperature of 120 °C and a heat rate of 2 °C/minute. The rapid uptake of Orange 30 by polyesteramide fibre clearly occurred in the range of 70 to 90 °C, while the rapid dyeing temperature range for PET fibre was between 80 and 120 °C. Moreover, the uptake rates of Orange 30 for polyesteramide and PET fibres were 5.9 and 3.1%/minute, respectively in the temperature range for the rapid uptake of Orange 30. The dyeing rate of polyesteramide fibre was almost twice as high as that of PET fibre.

Figure 4b shows the uptake rate of Disperse Orange 30 for polyesteramide fibre at a holding temperature of 100 °C and a heat rate of 2 °C/minute. It is evident from Figure 4b that polyesteramide fibre displayed a rapid dyeing temperature range, dyeing rate and final uptake rate of dyes similar to those in Figure 4a. Figures 4a and 4b show that polyesteramide fibre exhibits a faster dyeing rate than PET fibre, which can be explained by the lower glass transition temperature of polyesteramide fibre.

Although polyesteramide fibre exhibits good low-temperature dyeability, its high dyeing rate would lead to poor dyeing levelness. Certain special measures should therefore be taken to improve the dyeing levelness of polyesteramide fibre. To that end, the most effective method is to decrease the uptake rate of disperse dyes in the faster dye-uptake temperature range or the critical temperature range (where 80% of adsorption takes place) by reducing the rate at which the temperature is raised [18]. Figure 4c shows the uptake rate of Disperse Orange 30 for polyesteramide fibre at a holding temperature of 100 °C and a heat rate of 1 °C/minute. When comparing Figures 4b and 4c, it is evident that using a heat rate of 1 °C/minute resulted in a decrease in the uptake rate of Disperse Orange 30 to 3.1%/minute from 6.0%/minute at a heat rate of 2 °C/minute. It can be suggested from this study that the dyeing of polyesteramide fibre should be carried out at a low heating rate to provide a dyeing levelness effect.

3.4 Migration ability of disperse dyes on polyesteramide fibre

The dyeing levelness of textile materials is dependent on the migration ability of dyes [18], which in turn are affected by many factors, such as fibre and dye structures, dyeing temperature and time, and
the dyeing auxiliaries used in the dye solution. The good migration ability of dyes is beneficial for dyeing levelness. Figure 5 shows the migration ability of three different disperse dyes on polyesteramide fibre at 100 and 120 °C. All three of the disperse dyes exhibited a high migration rate at 120 °C. Disperse Orange 30 and Red 74 also exhibited a high migration rate at 100 °C. However, Red 60 displayed a much lower migration rate at 100 °C than at 120 °C. Nevertheless, the migration rate of Red 60 reached 66%. Overall, the good migration ability of disperse dyes on polyesteramide fibre can be exploited to improve the dyeing levelness of polyesteramide textiles and to correct uneven dyeing.

![Figure 5: Migration ability of disperse dyes on polyesteramide fibre](image)

3.5 Building-up property of disperse dyes on polyesteramide fibre

The building-up property of dyes is of great importance for practical application. Disperse dyes with a good building-up property can impart dark shades to polyesteramide fibre. The building-up property of disperse dyes primarily depends on the dye and fibre structures, the affinity of dyes to fibres and dyeing temperature. The building-up properties of azo and anthraquinone dyes on polyesteramide fibre are illustrated in Figure 6. In the case of azo dyes, the colour depth of the polyesteramide fabrics dyed with Disperse Red 74 and Blue 183 continually increased as the dye concentration in all of the dye concentration ranges was increased, while the same trend was identified for the fabrics dyed with Disperse Orange 30 and Brown 1, where dye concentrations in the range of 1% to 6% owf were used. In the case of anthraquinone dyes, the dyed fabrics displayed continually increasing colour depth when the concentrations of Disperse Red 11 and Violet 31 were less than 4% and 6% owf, respectively. Furthermore, the fabrics dyed with azo dyes exhibited higher colour depth than anthraquinone dyes. On the whole, azo dyes demonstrated a better building-up property than anthraquinone dyes. This finding is supported by the fact that azo dyes have a much higher adsorption saturation than anthraquinone dyes, as mentioned in subchapter 3.1.

![Figure 6: Building-up properties of (a) azo and (b) anthraquinone disperse dyes on polyesteramide fibre at 100 °C](image)

3.6 Colour fastness of dyed polyesteramide fabrics

In order to assess the colour fastness of dyed polyesteramide fabrics, the dyeing process was carried out with widely used trichromatic disperse dyes at a concentration of 3% owf. Reduction cleaning was then carried out to remove loose colours. The wash, sublimation, and light fastness ratings of the dyed polyesteramide fabrics are presented in Table 2. The

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Table 2: Colour fastness of dyed polyesteramide knitted fabrics

<table>
<thead>
<tr>
<th>Dyes</th>
<th>Wash fastness</th>
<th>Sublimation fastness</th>
<th>Light fastness</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Colour change</td>
<td>Staining</td>
<td>Colour</td>
</tr>
<tr>
<td></td>
<td>Polyester</td>
<td>Cotton</td>
<td>change</td>
</tr>
<tr>
<td>Orange 30</td>
<td>4-5</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Red 74</td>
<td>4</td>
<td>4</td>
<td>3-4</td>
</tr>
<tr>
<td>Blue 79</td>
<td>4</td>
<td>4</td>
<td>4-5</td>
</tr>
</tbody>
</table>

fabrics dyed with Disperse Orange 30 and Blue 79 exhibited high wash, sublimation and light fastness levels. Good wash fastness was also identified for the fabric dyed with Disperse Red 74, while its sublimation and light fastness ratings were fair, at 3–4 and 3, respectively. According to GB 18401–2010: National General Safety Technical Code for Textile Products (Chinese National Standards for Textiles) [19], wash colour fastness ratings greater than or equal to 3–4 for baby/children products, and 3 for products that come into direct and indirect contact with skin are defined as “acceptable”. Thus, the fabrics dyed with three disperse dyes were found to meet GB 18401–2010 in terms of acceptable colour fastness to washing. On the whole, the dyed polyesteramide fabrics exhibited good fastness properties that meet the requirements of consumers.

4 Conclusion

This study presented the dyeing properties of low-temperature dyeable polyesteramide fibre. The high exhaustion of disperse dyes at 100 °C revealed the good dyeability of polyesteramide fibre under atmospheric pressure. The Nernst adsorption isotherms of disperse dyes indicated a dyeing mechanism of polyesteramide fibre similar to that of PET fibre. The higher adsorption saturation and better building-up property of azo disperse dyes compared with anthraquinone dyes made the former a good selection for dark shades. The rapid uptake of dyes by fibres in the temperature range of 70 to 90 °C suggested that raising the temperature slowly was important for the improved dyeing levelness of polyesteramide textiles. The good migration of dyes on polyesteramide fabric can also be used to improve dyeing levelness. The good colour fastness properties of the dyed polyesteramide fabrics met the requirements of consumers. In summary, polyesteramide fibre can be used as an alternative to PET fibre to develop textile products, with regard to dyeing properties.

Acknowledgement

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References


19. GB 18401-2010 National general safety technical code for textile products.