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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 2391-2024

Replacing GB/T 2391-2014

Reactive dyes - Determination of degree of fixation

反应染料 固色率的测定

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1 Scope, references and terms

1 The document lays down the method for determining the degree of fixation of reactive dyes. It applies to the determination of the degree of fixation in printing and of the degree of fixation in exhaust dyeing.

2 The documents cited are GB/T 2374-2017 (dyes, general conditions for the determination of colouring), GB/T 2387-2013 (reactive dyes, determination of hue and strength) and GB/T 6687 (terminology of dyes).

3 The terms defined in GB/T 6687 apply.

4 Principle

4.1 In the printing method the test dye is printed directly on cotton cloth. A printed sample that has not been steam fixed and a printed sample that has been steam fixed are both washed thoroughly, the absorbance of each washing liquor is measured, and the degree of fixation of the dye on the fibre in printing is calculated from the two values.

4.2 In the exhaust dyeing method the test dye is dyed on cotton yarn. The exhaustion of the dye on the cotton fibre is calculated from the absorbances of the residual dye liquor and of the standard dye liquor. The unfixed hydrolysed dye is then washed off the dyed yarn and the degree of fixation on the cotton fibre is calculated from the absorbances of the standard soaping liquor and of the residual soaping liquor.

5 Reagents, materials and apparatus

5 The reagents used have to meet clause 3 of GB/T 2374-2017. They are sodium chloride, anhydrous sodium sulfate, anhydrous sodium carbonate, sodium phosphate and detergent

MA, also called detergent LS or sodium 2-methoxy-5-oleoylaminobenzenesulfonate, of industrial grade with a content of at least 95 % by mass.

6 The equipment used has to meet clause 4 of GB/T 2374-2017. It comprises a spectrophotometer, a laboratory dyeing machine, a laboratory steamer or steam box, a laboratory printing machine and a Soxhlet extractor of 150 mL.

7 Test methods

7.1.1 The printing concentration is fixed at 2 % by mass and printing follows 6.3 of GB/T 2387-2013. As soon as printing is finished, two pieces of equal area are cut from the same printed motif, normally 10 cm x 10 cm carrying two or three motif lines. One piece, sample A, goes to washing at once without drying or steaming; the other, sample B, is dried and steamed as required by 6.3 of GB/T 2387-2013 and then washed.

7.1.2 Samples A and B are each placed in a 150 mL low-form beaker holding 60 mL of water and soaked at room temperature for 15 min so that the fibre and the thickener swell fully; the sample is stirred well with a glass rod and, after 1 min, transferred into a Soxhlet extractor already charged with 100 mL of water and heated under reflux until the refluxing liquor is colourless. The sample is taken out and rinsed with a little water, and each coloured liquor is collected in a 500 mL volumetric flask, cooled to room temperature and diluted to the mark with water. If the washing liquor is too deeply coloured it is diluted further; the further dilution factor is recorded separately for the steamed and the unsteamed sample.

7.1.3 The absorbance of each washing liquor is measured with the spectrophotometer at the wavelength of maximum absorption. The degree of fixation in printing, as a mass fraction, is computed from the absorbance and the further dilution factor of the washing liquor of the steamed sample and from the absorbance and the further dilution factor of the washing liquor of the unsteamed sample.

7.2.1 The general dyeing conditions are a dyeing depth of 1 % on weight of fibre, 10 g of yarn as the fibre, a liquor ratio of 1 to 10, exhaustion temperature, fixation temperature and dyeing time as given in Table 1, and auxiliaries and their amounts as given in Table 2. Table 1 gives, for each dye type, the exhaustion temperature in degrees Celsius, the fixation temperature in degrees Celsius and the dyeing time in minutes: type X, 20, 40 and 90; type K, 40, 90 and 120; type KN, 60, 60 and 90; type M, 70, 70 and 90. A note adds that any other exhaustion temperature, fixation temperature or dyeing time is to be stated in the test report.

7.2.1 Table 2 gives the auxiliaries in grams per litre, for the dye bath and standard dye liquor an electrolyte and an alkali, and for the standard soaping liquor an alkali and detergent MA. Type X: sodium chloride 50, anhydrous sodium carbonate 10, anhydrous sodium carbonate 10, detergent MA 1. Type KN: anhydrous sodium sulfate 60, sodium phosphate 6, sodium

phosphate 6, detergent MA 1. Type K: sodium chloride 60, anhydrous sodium carbonate 15, anhydrous sodium carbonate 15, detergent MA 1. Type M: anhydrous sodium sulfate 60, anhydrous sodium carbonate 20, anhydrous sodium carbonate 20, detergent MA 1. A note adds that any other electrolyte, alkali or amount is to be stated in the test report.

7.2.2 Table 3 shows how the baths are made up, taking a type M reactive dye as the example, over four vessels: the dye bath, the standard dyeing stock liquor, the standard soaping stock liquor and the soaping liquor. Dye sample solution at 2 g/L, in millilitres: 50, 50, 50 and none. Anhydrous sodium sulfate, in grams: 6, 6, none and none. Sodium carbonate solution at 100 g/L, in millilitres: 20, 20, 20 and none. Detergent MA solution at 10 g/L, in millilitres: none, none, 25 and 25. Water, in millilitres: 30, 30, 5 and 225.

7.2.3 Before dyeing, the yarn is put into a sodium hydroxide solution of 10 g/L, heated to the boil and kept soaking for 1 h, then rubbed thoroughly and rinsed under running water until the rinse water is no longer cloudy, the alkali being removed as completely as possible and checked with pH paper if necessary. A note explains that this step removes the size on the yarn, since too much residual size affects the final degree of fixation, above all with dyes of shorter wavelength such as yellow dyes.

7.2.4 The dye bath, the standard dyeing stock liquor and the standard soaping stock liquor are made up as in Table 3, the electrolyte being added before dyeing. The three liquors go into the dyeing machine together and are brought to the exhaustion temperature of Table 1. The treated white yarn, wrung to a mass of about 20 g corresponding to a water content of about 100 %, is put into the dye bath and dyed at the exhaustion temperature for 30 min; the alkali needed is then added to each of the three liquors, the temperature is raised to the fixation temperature of Table 1, and at the end of the dyeing time of Table 1 the dyed yarn is taken out, wrung and washed several times with about 300 mL of water, the alkali being removed as far as possible and checked with pH paper if necessary, and finally wrung again to about 20 g. The liquid squeezed from the yarn and the washing liquid are recovered into the residual dye liquor.

7.2.5 The wrung dyed yarn is soaped for 15 min in the soaping liquor made up as in Table 3, at a soaping temperature of 93 °C to 95 °C and a liquor ratio of 1 to 25, then taken out and wrung to about 20 g. It is washed three times with about 200 mL of distilled water in total, being wrung to about 20 g after each wash, and the liquid squeezed out each time is recovered into the residual soaping liquor.

7.2.6 Each of the four test solutions is cooled to room temperature and made up to 500 mL in a volumetric flask. From the standard dye liquor and from the standard soaping liquor 5 mL to 20 mL is pipetted, as the case requires, into a 100 mL volumetric flask and diluted to the mark with water; from the residual dye liquor and from the residual soaping liquor 20 mL to 100 mL is pipetted in the same way. Each dilution factor is recorded.

7.2.7 and 7.2.8 The absorbances of the standard dye liquor, the residual dye liquor, the

standard soaping liquor and the residual soaping liquor are measured with the spectrophotometer at the wavelength of maximum absorption of the standard dye liquor, with water as the blank. The exhaustion is computed from the absorbance and dilution factor of the residual dye liquor and those of the standard dye liquor, and the degree of fixation in dyeing is computed from the exhaustion together with the absorbance and dilution factor of the residual soaping liquor and those of the standard soaping liquor.

8 Test report

The test report carries the name of the dye tested, the number of the document, the test method, the name and model of the apparatus used, the test results, any special circumstance arising during the test, any departure from the method and the date of the test.