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# Enaminones-Assisted Synthesis of Disperse Dyes. Part 2: High Temperature Dyeing of Polyester Fabrics

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SOME new disperse dyes has been prepared and used in dyeing polyester fabrics at a temperature of 130 °C and a study of the optimum conditions for the use of the dispersing agent. The relationship between the dye concentration used in dyeing polyester fabrics with dispersed dyes and the color strength K/S were studied using three different concentrations of dye weight. Finally, polyester fabrics dyed with disperse dyes have shown that they possess very good fastness against washing, perspiration and rubbing and acceptable fastness properties of light.

## Introduction

The chemistry of enaminones has gotten a lot of consideration because of their easy accessibility through synthesis of disperse dyes [1]. The significance of azo enaminones dyes is exhibited by the high number of publications [2,3] managing somehow with that class of dyestuffs, noted for their profoundly fascinating dyeing properties, specifically their high level of brilliance contrasted and azo dyes got from anilines [4] and their magnificent shades [5]. Their derivatives have for some time been utilized as intermediates in the dyestuff chemistry. Enaminones compounds are helpful as forerunners for the union of melded heterocyclic ring frameworks, which show a significant scope of organic and pharmacological activities [6–9]. Disregarding the huge number of reports on the utility of these compounds in the dye chemistry. Right now, the dyeing performance of disperse dyes 1-6 was analyzed within the sight of a dispersing agent ranged from 0.5 to 2% at increasing dye bath concentrations from 1% to 3% to get the optimum dyeing conditions. The coloring performances of the individual colors are shown by using the K/S esteems as dye uptake of the colored examples.

## Materials and Methods

*General Procedure for the Synthesis of Disperse Dyes 1-6*

The disperse dyes were prepared according to the method that we published in our previous research [10].

*Dyeing at 130 °C (high temperature dyeing)*

A- Study the optimum concentration of dispersing agent.

The disperse dyes 1-6, a dispersion of the dyes were produced by dissolution of the appropriate amount of dyes (1% shades) in 2 ml DMF and then added dropwise with stirring to the dye bath (liquor ration 1:30) containing different concentration (0.5, 1, 1.5, 2%) of levegal MDL as anionic dispersing agent (TANATEX chemicals). The pH of the dye bath was adjusted to 5.5 with aqueous acetic acid, and the wetted-out polyester fabrics (3 gm) were added. We performed dyeing by raising the dye bath temperature to 130°C at a rate of 3°C/min and holding it at this temperature for 60 min. After they were cooled to 50°C, the dyed fibers were rinsed with cold water and reduction-cleared (1 g/L sodium hydroxide, 1 g/L

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sodium hydrosulfite, 10 min, 80°C). The samples were rinsed with hot and cold water and, finally, air-dried.

B- Dyeing at optimum concentration of dispersing agent.

The same procedure performed for disperse dyes 1-6 (1% - 3% shades), using concentration (1.5%) of levegal MDL as dispersing agent.

*Color Measurements*

The colorimetric parameters of the dyed polyester fabrics were determined on a reflectance spectrophotometer. The color yields of the dyed samples were determined by using the light reflectance technique performed on an UltraScan PRO D65 UV/VIS Spectrophotometer. The color strengths, expressed as K/S values, were determined by applying the Kubelka-Mink equation [10].

$$K/S = (1- R)^2 / 2R$$

*Color fastness to washing*

The color fastness to washing was determined according to the ISO 105-C02:1989 method [10]. The composite specimens were sewed between two pieces of bleached cotton and wool fabrics, and then immersed into an aqueous solution containing 5 g/L of nonionic detergents at a liquor ratio of 1:50. The bath was thermostatically adjusted to 60 °C for 30 min. After the desired time, samples were removed, rinsed twice with occasional hand squeezing, and then dried. Evaluation of the wash fastness was established using the grey scale for color change.

*Color fastness to rubbing*

Color fastness to rubbing was determined according to the ISO 105-X12:1987 test method. The test is designed for determining the degree of color that may transfer from the surface of the colored fabrics to another surface by rubbing. The current test can be carried out on dry and wet fabrics.

*Dry crocking test*

The test specimen was placed flat on the base of the crockmeter. A white testing cloth was mounted. The covered finger was lowered onto the test specimen and caused to slide back and forth 20 times. The white test sample was then removed for evaluation using the grey scale for staining.

*Wet crocking test*

The white test sample was thoroughly (65%) wetted with water. The procedure was run as before. The white test samples were air dried before evaluation.

*Color fastness to perspiration*

Two artificial perspiration solutions (acidic and alkaline) were prepared according to the ISO 105-E04 : 1989 test method. The acidic solution was prepared by dissolving L-histidine monohydrochloride monohydrate (0.5 g), sodium chloride (5 g), and sodium dihydrogen orthophosphate dihydrate (2.2 g) in one liter of distilled water. Then, the pH was finally adjusted to 5.5 using 0.1 N NaOH. To prepare the alkaline solution, L-histidine monohydrochloride monohydrate (0.5 g), sodium chloride (5 g), and disodium hydrogen orthophosphate dehydrate (2.5 g) were all dissolved in one liter of distilled water. The pH was adjusted to 8 using 0.1 N NaOH. The fastness test was performed as follows. The 5 cm × 4 cm colored specimen was sewn between two pieces of uncolored specimens to form a composite specimen. The composite samples were immersed for 15-30 min in both solutions with well agitation and squeezing to ensure complete wetting. The test specimens were placed between two plates of glass or plastic under a force of about 4-5 kg. The plates containing the composite specimens were then held vertically in an oven at 37 ± 2 °C for 4 h. The effect on the color of the tested specimens was expressed and defined by reference to the grey scale for color change.

*Color fastness to light*

The light fastness test was carried out in accordance with the ISO 105-B02:1988 test method, using a carbon arc lamp and continuous light for 35 h. The effect on the color of the tested samples was recorded by reference to the blue scale for color change.

## Results and Discussion

Related to our enthusiasm for creating proficient courses to polyfunctional heteroaromatics as disperse dyes for polyester textures or potentially Dye Diffusion Thermal Transfer (D2T2) printing, we report here our outcomes on the reactivity of enaminones toward some aryldiazonium chlorides. Acetophenones or substituted acetophenones reacted with dimethylformamide dimethylacetal (DMFDMA) afforded enaminones that reacted with aryldiazonium salts to obtain disperse dyes 1-6 (Figure 1) [10]. In this study these new disperse dyes were used in dyeing polyester fabrics at a high temperature of 130 °C, in the presence of the dispersing agent.

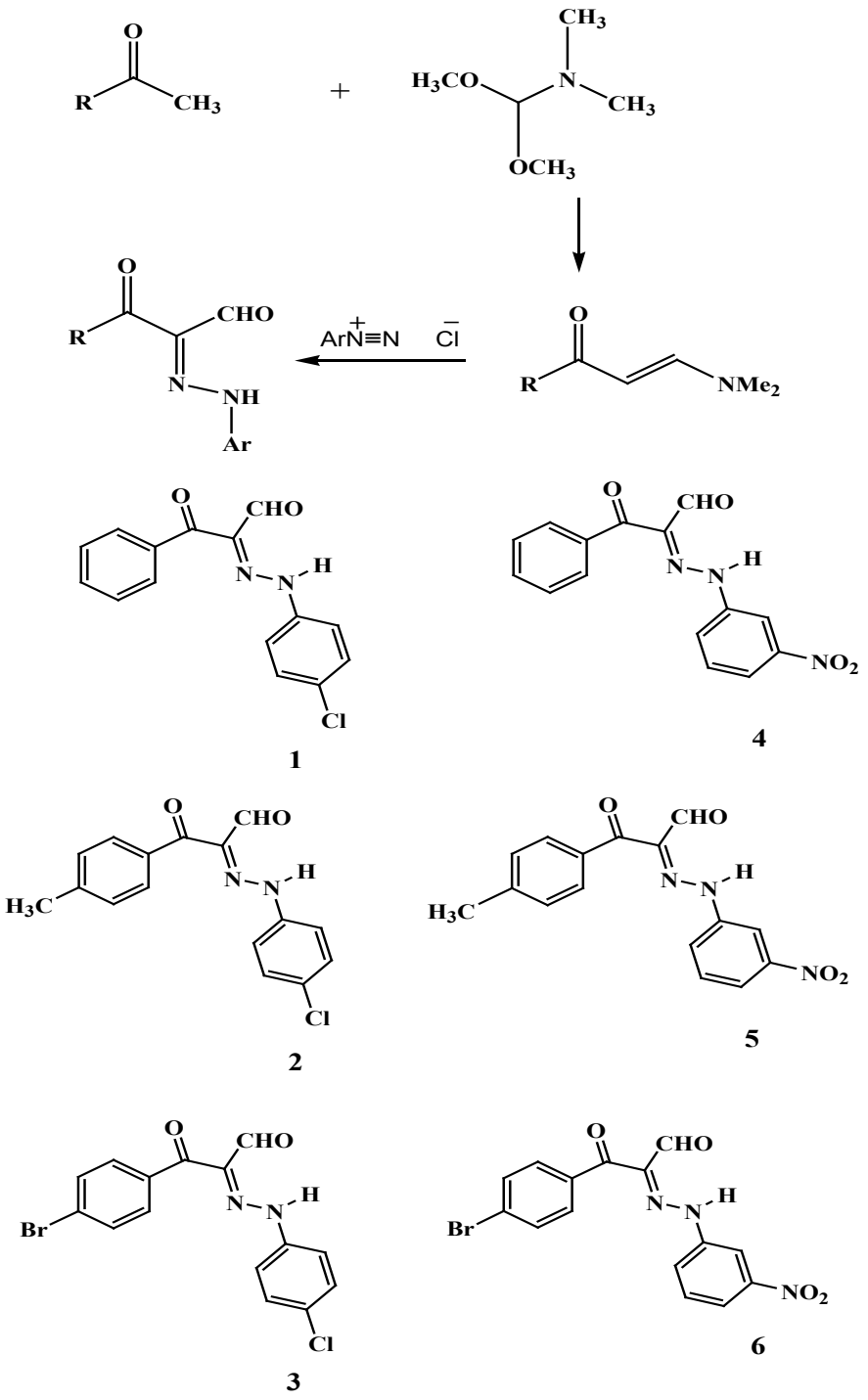


**Fig. 1. Chemical structures of the disperse dyes.**

*Effect of dispersing agent on K/S*

We dyed a polyester fabrics with the new disperse dyes 1-6 based on enaminones [11,12] at 1% shade, and we studied the use of the dispersing agent at different concentrations from 0.5 to 2% to study the optimum concentration giving the best value of color strength K/S. The results set out in Table 1 indicate that the K/S values of the polyester fabrics dyed with the disperse dyes of the dyes 1, 4 and 5 increase with the increase of the dispersing agent concentration and reach their highest value (13.18, 10.27 and 12.41) at a concentration of 1.5 %. The K/S values for the polyester fabric dyed with the disperse dyes of

the 2 and 3 dyes also increase with increasing dispersing agent concentration and reach their highest value (16.99 and 10.96) at a concentration of 1%, while, the highest K/S value for dye No. 6 (10.60) at a concentration of 0.5%. It is clear from the results described in Table 1 that when dyeing polyester fabrics with new dispersed dyes at a temperature of 130 °C the most appropriate optimum conditions for the use of dispersing agent are 1.5%.

*Relation between dye concentrations of the disperse dyes and K/S*

While trying to discover the connection between the concentration of the dye used in dyeing polyester fabrics with the new dispersed dyes and the intensity of the color resulting from that dyeing process on the dyed fabrics, the dyeing process was done by using three concentrations of each dye 1, 2 and 3%, and the color strength was measured and results are presented in Table 2 and Figure 2. Table 2 indicate that the color strength K/S values for the polyester fabrics dyed with the disperse dyes of the dyes 1- 6 increase with increasing the dye concentration and reach their highest value (17.14, 15.54, 13.76, 19.93, 16.47 and 13.95) at dye concentration 3%.

*Fastness properties* of the disperse dyes.

The fastness properties of polyester fabrics dyed with the new disperse dyes have been done at shades from 1% to 3%, and the results set in Tables 3-5 show the fastness against washing, rubbing and perspiration gave very good results. The fastness properties against light gave acceptable results, and the results of shade 1% were slightly better than both shades 2% and 3%, and also shade 2% was slightly better than shade 3%.

**TABLE 1. Dispersing agent effects on the dyeing process of disperse dyes by using 1% shade.**

| Dye No | % Dispersing agent | L* | *a** | *b** | *C** | *h** | K/S |
|---|---|---|---|---|---|---|---|
| 1 | 0.5% | 83.08 | -11.19 | 57.60 | 57.00 | 103.59 | 11.80 |
| | 1% | 80.30 | -9.12 | 57.25 | 57.97 | 99.05 | 9.94 |
| | 1.5% | 81.63 | -10.29 | 57.58 | 58.50 | 100.13 | 13.18 |
| | 2% | 80.70 | -10.17 | 57.31 | 58.21 | 100.06 | 10.88 |
| 2 | 0.5% | 81.19 | -11.61 | 56.42 | 57.60 | 101.62 | 12.34 |
| | 1% | 81.85 | -12.04 | 56.33 | 57.60 | 102.06 | 16.99 |
| | 1.5% | 82.78 | -12.12 | 56.60 | 57.88 | 102.09 | 11.63 |
| | 2% | 81.76 | -12.43 | 56.92 | 58.27 | 102.32 | 15.85 |
| 3 | 0.5% | 82.03 | -12.19 | 52.52 | 53.91 | 103.07 | 9.43 |
| | 1% | 81.68 | -12.20 | 53.78 | 55.14 | 102.78 | 10.96 |
| | 1.5% | 82.79 | -12.75 | 51.75 | 55.30 | 103.84 | 10.31 |
| | 2% | 81.66 | -11.10 | 53.72 | 55.05 | 102.59 | 10.60 |
| 4 | 0.5% | 78.40 | -5.11 | 33.44 | 33.55 | 98.60 | 7.48 |
| | 1% | 77.95 | -5,90 | 37.40 | 37.86 | 98.97 | 9.53 |
| | 1.5% | 81.64 | -7.19 | 38.56 | 39.22 | 100.56 | 10.27 |
| | 2% | 80.60 | -6.22 | 39.57 | 40.05 | 98.94 | 9.47 |
| 5 | 0.5% | 80.63 | -5.85 | 38.02 | 38.47 | 98.75 | 11.13 |
| | 1% | 82.47 | -6.94 | 35.54 | 36.21 | 101.06 | 9.03 |
| | 1.5% | 80.32 | -6.59 | 37.88 | 38.45 | 99.87 | 12.41 |
| | 2% | 79.19 | -5.82 | 34.30 | 34.79 | 99.62 | 8.57 |
| 6 | 0.5% | 81.98 | -7.54 | 38.20 | 38.93 | 101.16 | 10.60 |
| | 1% | 81.65 | -6.51 | 36.28 | 36.86 | 100.17 | 4.67 |
| | 1.5% | 81.14 | -6.54 | 39.58 | 40.12 | 99.39 | 9.22 |
| | 2% | 81.78 | -7.74 | 35.58 | 36.41 | 102.28 | 7.48 |

**TABLE 2. Effect of the dye shades used in dyeing process at 100 $^0$C and K/S of dyed fabrics.**

| Dye No | % shade | L* | *a** | *b** | *C** | *h** | K/S |
|---|---|---|---|---|---|---|---|
| 1 | 1% | 86.92 | -7.92 | 65.08 | 65.56 | 96.94 | 9.70 |
| | 2% | 84.86 | -3.70 | 79.77 | 79.85 | 92.66 | 16.04 |
| | 3% | 83.26 | -0.12 | 83.09 | 83.09 | 90.08 | 17.14 |
| 2 | 1% | 88.18 | -12.01 | 64.81 | 65.92 | 100.49 | 13.92 |
| | 2% | 86.89 | -9.44 | 70.33 | 70.96 | 97.64 | 13.70 |
| | 3% | 85.47 | -7.64 | 75.87 | 76.26 | 95.75 | 15.54 |
| 3 | 1% | 88.83 | -13.10 | 62.39 | 63.75 | 101.86 | 11.49 |
| | 2% | 89.66 | -9.77 | 73.76 | 74.41 | 97.55 | 13.51 |
| | 3% | 86.63 | -9.02 | 75.02 | 75.56 | 96.86 | 13.76 |
| 4 | 1% | 86.78 | -4.95 | 42.07 | 42.36 | 96.71 | 7.94 |
| | 2% | 82.00 | 1.29 | 53.86 | 53.87 | 88.63 | 15.60 |
| | 3% | 78.44 | 5.33 | 59.76 | 60.00 | 84.91 | 19.93 |
| 5 | 1% | 84.60 | -1.77 | 48.85 | 48.88 | 92.08 | 11.90 |
| | 2% | 82.48 | 0.92 | 54.46 | 54.46 | 89.04 | 15.18 |
| | 3% | 81.06 | 2.85 | 57.38 | 57.45 | 87.15 | 16.47 |
| 6 | 1% | 87.53 | -6.23 | 41.10 | 41.57 | 98.62 | 7.19 |
| | 2% | 85.33 | -3.24 | 48.30 | 48.41 | 93.84 | 11.19 |
| | 3% | 79.00 | -3.11 | 47.46 | 47.57 | 93.84 | 13.95 |

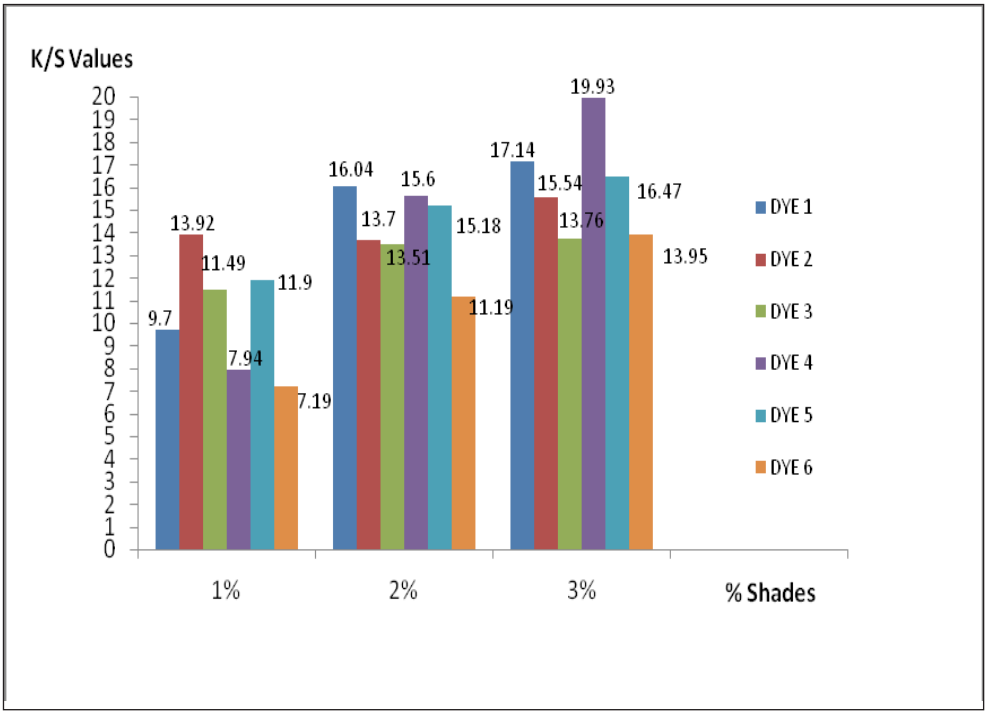


**Fig. 2. Relation between dye concentrations and shades.**

**TABLE 3. Fastness properties of disperse dyes on polyester fabrics at shade 1%.**

| Dye No | Washing fastness | | | Rubbing fastness | | Perspiration fastness | | | | | | Light fastness |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Acidic | | | Alkaline | | | |
| | SC | SW | Alt | Dry | Wet | SC | SW | Alt | SC | SW | Alt | |
| **1** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4–5 | 4 | 4–5 | 4 | 4 | 3 |
| **2** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4 | 4 | 4–5 | 4–5 | 4 | 3 |
| **3** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4–5 | 4 | 4–5 | 4–5 | 4 | 3 |
| **4** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4 | 4 | 4–5 | 4 | 4 | 4 |
| **5** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4 | 4 | 4–5 | 4 | 4 | 3 |
| **6** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4 | 4 | 4–5 | 4 | 4 | 3–4 |

**TABLE 4. Fastness properties of disperse dyes on polyester fabrics at shade 2%.**

| Dye No | Washing fastness | | | Rubbing fastness | | Perspiration fastness | | | | | | Light fastness |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Acidic | | | Alkaline | | | |
| | SC | SW | Alt | Dry | Wet | SC | SW | Alt | SC | SW | Alt | |
| **1** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4 | 4 | 4–5 | 4 | 4 | 2–3 |
| **2** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4 | 4 | 4–5 | 4 | 4 | 3 |
| **3** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4–5 | 4 | 4–5 | 4 | 4 | 2–3 |
| **4** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4–5 | 4 | 4–5 | 4–5 | 4 | 3–4 |
| **5** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4 | 4 | 4–5 | 4 | 4 | 3 |
| **6** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4–5 | 4 | 4–5 | 4–5 | 4 | 3 |

**TABLE 5. Fastness properties of disperse dyes on polyester fabrics at shade 3%.**

| Dye No | Washing fastness | | | Rubbing fastness | | Perspiration fastness | | | | | | Light fastness |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Acidic | | | Alkaline | | | |
| | SC | SW | Alt | Dry | Wet | SC | SW | Alt | SC | SW | Alt | |
| **1** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4 | 4 | 4–5 | 4 | 4 | 2–3 |
| **2** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4–5 | 4 | 4–5 | 4–5 | 4 | 2–3 |
| **3** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4–5 | 4 | 4–5 | 4 | 4 | 2–3 |
| **4** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4–5 | 4 | 4–5 | 4 | 4 | 3–4 |
| **5** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4 | 4 | 4–5 | 4 | 4 | 2–3 |
| **6** | 4–5 | 4 | 4 | 4–5 | 4 | 4–5 | 4 | 4 | 4–5 | 4–5 | 4 | 3 |

## Conclusion

We prepared some new disperse dyes and using them in dyeing polyester fabrics at a temperature of 130 °C, and the optimum conditions for using the dispersed agent is 1.5%. The K/S values of dyed polyester fabrics with the disperse dyes 1-6 increase with increasing dye concentration and reached to the top its values at the dye concentration 3%. The fastness properties of polyester fabric dyed with disperse were acceptable for light fastness and very good for fastness against washing, rubbing and perspiration.

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## تشييد بعض الصبغات المنتشرة بمساعدة الإينامينونات. جزء ٢: صباغة أقمشة البولى استر فى درجة حرارة عالية

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تم تحضير بعض من الأصباغ الجديدة المنتشرة واستخدامها في صباغة أقمشة البولي استر عند درجة حرارة ١٣٠ درجة مئوية ودراسة الظروف المثلى لاستخدام العامل المشتت. تمت دراسة العلاقة بين تركيز الصبغة المستخدمة في صباغة أقمشة البولي استر وشدة اللون باستخدام ثلاثة تركيزات مختلفة من وزن الصبغة. أخيرًا ، أظهرت أقمشة البولي استر المصبوغة بالأصباغ المنتشرة أنها تمتلك خصائص ثبات جيد جدًا للغسيل والعرق والاحتكاك و خصائص ثبات مقبولة للضوء.