

# Effect of Silica Gel Addition on Cotton Dyeing with Sirius Dyestuff

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(Received September 30, 1989)

## ABSTRACT

The dyeing properties of cotton with direct dyes are influenced by the addition of auxiliary reagents such as neutral salts. The effects of silica gel addition on cotton dyeing with Sirius dyestuffs were investigated. L a b values as color difference of dyed cloths were measured on a color difference meter.

Up to 10% o. w. f. addition of silica gel into the dyebath, L values were decreased, and above 15% addition L values were increased. In an occasion of coexistence of silica gel particles and neutral salt, the effects of silica gel addition were not observed so clearly. The addition of silica gel particles in the dyeing with Sirius Black L having greater minus charge was more effective than that in the dyeing with Sirius Red 4B.

The adsorption of these dyestuff on silica gel particles filtered from dyebath was not observed, and neither was the weight increase of dyed cloth.

## INTRODUCTION

One of the dyeing method of silk, wool and polyamide fiber such as nylon is the acidic dyebath method, the other the natural dyebath method. Owing to frequent occurrence of unevenness in color, especially in silk dyeing the natural dyebath method which gives good evenness in color has been well introduced. But the color fastness of silk dyed by the natural dyebath method is not so good. The natural dyebath method of silk having higher fastness must be developed.

On the other hand, the problem of antipollution management of residual solution from dyeing processes is very significant in a dye works. One approach to this problem is that by the rise of dye uptake the dye concentration in the residual solution is decreased.

Some papers reported the influences of various auxiliary reagents such as neutral salts in dyeing<sup>1)-5)</sup>. Neutral salts assist the approach of dye molecules to the surface of fibers.

In this report, the effects of silica gel addition on the dyeing of cotton by Sirius dyestuffs have been examined. There are few reports about the influences of silica gel in dyeing process<sup>6)</sup>.

## EXPERIMENTALS

### *Materials*

The cotton cloths (NISSHINBO KK white poplin NO.4000) were used after the destarch treatment by a conventional method. The size of cotton cloth used in the following experiments was 10×10 cm and its average weight was 1.245g.

As the dyestuffs for cotton, Sirius Red 4B [FBy] (C.I. Direct Red 81) and Sirius Black L [FBy] (C.I. Direct Black 51) were used without further purifications.  $\lambda_{\max}$  (pyridine-water) of Sirius Red 4B is 525 nm, and that of Sirius Black L is 590.0 nm<sup>7)</sup>.

Sodium sulfate was used as an auxiliary neutral salt.

Silica gel particles (Wakogel C-300) for column chromatography were used without further purifications. Ion-exchanged distilled water was used in the following experiments.

### *Method*

#### (1) Dyeing of cotton cloth

The concentrations of dyestuff were 0.5%, 2.0%, and 5.0% o.w.f.. For each concentration the amounts of Silica gel addition were changed as o.w.f. 0%, 5%, 10%, 15% and 20%. 5% o.w.f. sodium sulfate was added into 0.5% o.w.f. dye concentration dyebaths and as references a series involving no sodium sulfates in dyebaths was prepared. Similarly, 15% sodium sulfate was added into 2.0% dyebaths, 25% sodium sulfate was added into 5.0% dyebaths, and the references were prepared respectively.

The flasks involving cotton cloth, dyestuff, silica gel particles and sodium sulfate were dipped into a shaking water bath (TOKYO RIKAI KIKAI KK NTS-211) at room temperature and the temperature was raised to  $90^{\circ} \pm 2^{\circ}\text{C}$  for 30 minutes and at this temperature dyeing was continued for 30 minutes. After cooling to room temperature, the cotton cloths were taken out of flasks and washed in water carefully.

#### (2) Measurement of color difference

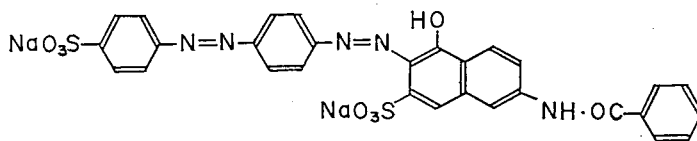
The color differences of cotton cloths dyed with Sirius Red 4B and Sirius Black L were measured on a NIPPON DENSHOKU ND-101DP color difference meter. The diameter of measurement area was 30 mm. L a b expression and X Y Z expression were both measured. The measurement of color difference was done for four points and the mean value was determined. One point in

figure indicates the average of at least four samples.

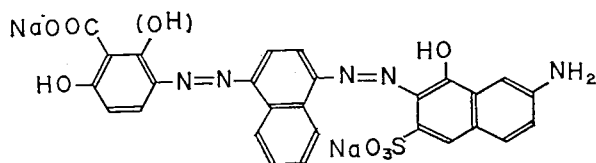
## RESULTS AND DISCUSSION

In almost all cases of dyeing of cellulose fiber by direct dyestuff, some neutral salts are added into dyebath. The amount of dye adsorption is generally increased by addition of neutral salts, but the degree of increase shows great difference with reference to the structure of dye<sup>1),2)</sup>. Many studies have been reported the effects of auxiliary reagents in dyeing of cellulose fiber<sup>1-5)</sup>. But there are few reports about the role of an auxiliary reagent in the dyeing of hydrophobic fibers such as PET by functional dyestuff having greater molecular size rather than general dyes.

In this series of experiments silica gel particles were chosen as an auxiliary reagent having no chemical interaction in the dyeing process. Upon this, as the first examination, the influences of silica gel addition to the cotton dyeing with direct dyes were studied. Figure 1 shows the structure of direct dyes used.



Sirius Red 4B



Sirius Black L

Figure 1 Structure of Sirius dyes used.

For the purpose of comparison of the influence of silica gel and it of neutral salt, two series of experiments were prepared.

In all experiments silica gel particles were filtrated from residual dye solution, and washed by water. This washed and dried silica gel particles were not dyed by dyestuff.

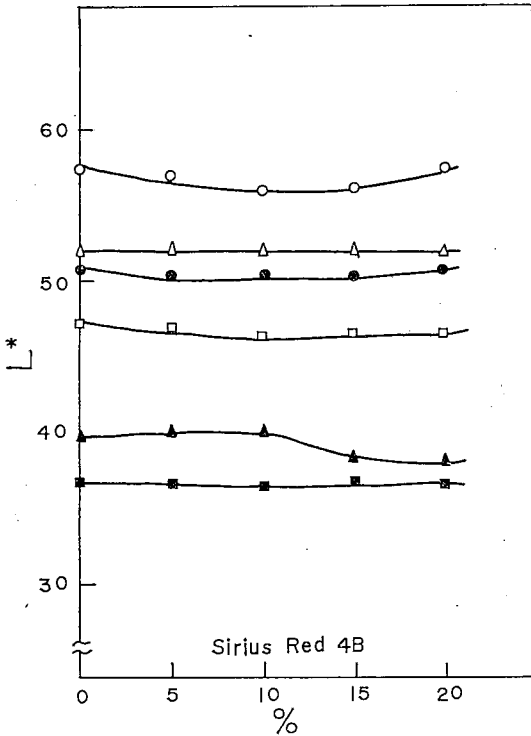


Figure 2 Relations between L and additional amounts of silica gel in the dyeing of cotton with Sirius Red 4B: (○) 0.5 % dye, (●) 0.5% dye and 5.0% sodium sulfate, (△) 2.0% dye, (▲) 2.0 % dye and 15.0% sodium sulfate, (□) 5.0% dye, (■) 5.0% dye and 25.0% sodium sulfate.

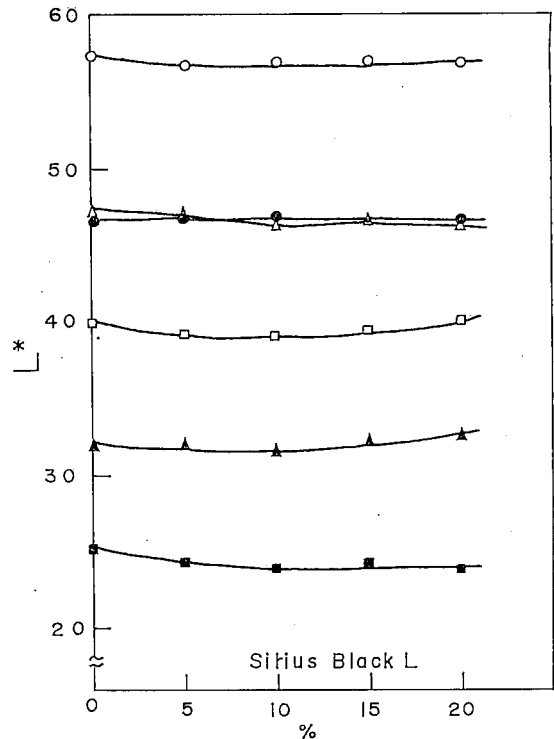


Figure 3 Relations between L and additional amounts of silica gel in the dyeing of cotton with Sirius Black L: (○) 0.5% dye, (●) 0.5% dye and 5.0% sodium sulfate, (△) 2.0% dye, (▲) 2.0% dye and 15.0% sodium sulfate, (□) 5.0% dye, (■) 5.0% dye and 25.0% sodium sulfate.

Figure 2 shows the L values of cotton cloths dyed with Sirius Red 4B, and Figure 3 shows that with Sirius Black L. White dots, white triangles and white squares indicate the influences of silica gel only. Black dots, black triangles and black squares indicate the influences of coexistence of silica gel and sodium sulfate. L values were decreased with the increase of silica gel and again increased. As shown in Figure 2, in Sirius Red 4B the influences of added silica gel particles is remarkable at 0.5% o.w.f. dyestuff concentration. On the other hand, as shown in Figure 3 in Sirius Black L it is obvious at 5.0% o.w.f. concentration.

One of difficulties of dye molecule approach to fiber surface is the electrostatic repulsion between minus charge of dye and it of fiber. Sirius Red 4B has two sulfonate groups and one hydroxyl group, on the other side Sirius Black L has one sulfonate group, one carboxylate group, one amino group and three hydroxyl groups. As can be seen from these figures, the degree of decrease of Sirius Black L in case of neutral salt addition into dyebath was greater than that of Sirius

Red 4B. L value indicates the reflection ratio, so the decrease of L values reflects the increase of adsorption of dyestuff on cotton cloth. Because of the greater minus charges on Sirius Black L than Sirius Red 4B, the effects of neutral salt addition is seemed to be greater at Sirius Black L. But the degree of L decrease in case of the addition of only silica gel particles was not so extensive.

Silica gel exhibits the nature of minus charged colloid<sup>8)</sup>. Up to 10% o. w.f. addition of silica gel the electric double layer of it is seemed to assist the approach of dye molecule toward surface of fiber, but the degree of assistance is lower than that of neutral salt.

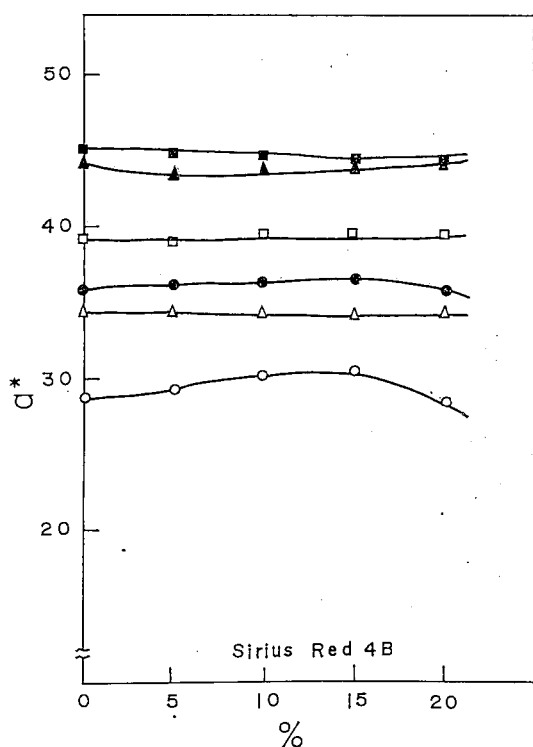


Figure 4 Relations between  $a^*$  and additional amounts of silica gel in the dyeing of cotton with Sirius Red 4B: (○) 0.5% dye, (●) 0.5% dye and 5.0% sodium sulfate, (△) 2.0% dye, (▲) 2.0% dye and 15.0% sodium sulfate, (□) 5.0% dye, (■) 5.0% dye and 25.0% sodium sulfate.

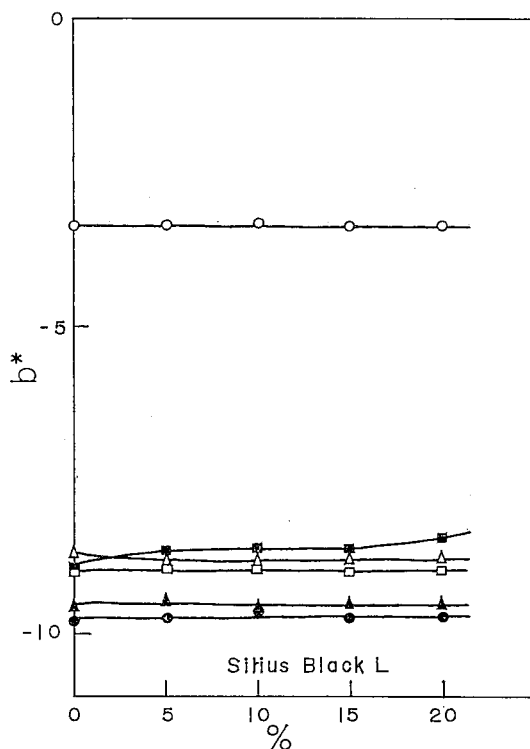


Figure 5 Relations between  $b^*$  and additional amounts of silica gel in the dyeing of cotton with Sirius Black L: (○) 0.5% dye, (●) 0.5% dye and 5.0% sodium sulfate, (△) 2.0% dye, (▲) 2.0% dye and 15.0% sodium sulfate, (□) 5.0% dye, (■) 5.0% dye and 25.0% sodium sulfate.

Figure 4 shows the change of  $a^*$  value in Sirius Red 4B, and Figure 5 shows that of  $b^*$  value in Sirius Black L. Both  $b^*$  values in Sirius Red 4B and  $a^*$  values in Sirius Black L were scarcely changed by the addition of silica gel particles and sodium sulfate. At dilute coccentration of Sirius Red 4B,  $a^*$  values were increased with the

increase of silica gel addition up to 15% o.w.f.. In occasion of coexistence of sodium sulfate, the degree of  $a^*$  increase was smaller than that of occasion involving silica gel only.

As shown in Figure 5, the effect of neutral salt addition was not observed at higher concentration of dyestuff, and neither was that of silica gel addition.

Extracted solution from silica gel particles was neutral pH and the addition of this solution into dyebath did not exhibit the change of color difference.

In conclusion, the effect of silica gel particles addition in the cotton dyeing was observed up to 10% o.w.f., but it was not so effective in comparison with that of neutral salt addition.

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