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〈Research Paper〉

# Dyeing Characteristics of Casein Protein Fiber with Acid Dyes and Reactive Dyes

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**Abstract**— The present paper focuses on the application of commercial acid dyes and wool reactive dyes to casein protein fabric. Nine acid dyes and six wool reactive dyes were compared their dyeing properties as well as color fastness. The exhaustion yields were higher than 80 % which dramatically increased at pH 3. Excellent wash fastness was obtained with metal-complex acid and wool reactive dyes. Both light and rubbing fastness were overall good, but perspiration fastness was comparatively poor.

**Keywords:** *casein protein fiber, acid dyes, reactive dyes, buffer solution, dye exhaustion*

## 1. Introduction

In recent, some sorts of natural fibers are now attracting interest in the textile market. For instance, those based on bamboo, soybean and casein fibers have biodegradable, non allergic, microbiocidal and anti-ageing properties[1]. Another type of polyester, poly(lactic acid) (PLA), has also attracted attention as a polymer for textiles because, in addition to offering similar technical properties to PET, it is produced from renewable resources and can be biodegradable[2,3,4,5].

Casein fiber is one of the man-made protein fibers of animal origin (milk protein). The casein is separated from the whey by dissolving in caustic soda, thereafter spun in a precipitating bath with sulfuric acid, sodium sulfate[6]. Therefore, casein fibers have a certain similarity to sheep's wool. Lanital was the first commercial protein fiber obtained from milk casein, as patented by Antonio Feretti in 1935[7]. As the important ingredients of milk protein fiber are casein proteins, which can nourish and lubricate the skin. The milk protein contains the natural humectant factor, which can capture moisture and will maintain the skin's moisture[7]. Some properties of casein protein fiber are compared with those of other natural fibers in Table 1.

The content of amino acids in milk fiber is around 30% which was tested by SGS-CSTC Standards Technical Services Co. Ltd Shanghai Branch. As shown in Table 2, milk fiber contains 18 amino acids similar to those found in wool and silk, but the proportions are different. The main components are Glutamic acid, Proline, Leucine and Aspartic acid. The nature of side-chain of Glutamic acid and Aspartic acid can be featured as an acidic group compared with those of other amino acids, as illustrated in Fig. 1.

For the dyeing of new materials, as mentioned above, there have been some research results on poly(lactic acid) fiber[10,11] and soybean fiber[1]. However in the case of coloration of casein fiber, very limited research results have been reported dyeing properties of casein fiber.

Due to the similarity in chemical compositions between casein fiber and wool, dyeing of casein fiber with acid dyes and some reactive dyes can be utilized.

In this paper, the optimum conditions for pretreatment have been studied, then dyeing performance of a range of acid dyes and wool reactive dyes containing different reactive groups have been assessed in terms of exhaustion yield depending on the pH of the dyebath.

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Table 1. Property comparisons between casein fiber and other natural fibers[7)]

| Property | Casein fiber | Cotton | Silk | Wool |
|---|---|---|---|---|
| Fineness (dtex) | 1.52 | 1.2-2.0 | 1.0-2.8 | 6-9 |
| Tensile strength (dry, CN/dtex) | 2.8 | 1.9-3.1 | 3.8-4.0 | 2.6-3.5 |
| Tensile strength(wet, CN/dtex) | 2.4 | 3.2 | 2.1-2.8 | 0.8 |
| Breaking elongation rate(dry, %) | 25-35 | 7-10 | 11-16 | 14-25 |
| Breaking elongation rate(wet, %) | 28.8 | 13 | 27-33 | 50 |
| Moisture regain (%) | 5-8 | 7-8 | 8-9 | 15-17 |
| Specific weight (g/cm3) | 1.22 | 1.50-1.54 | 1.46-1.52 | 1.34-1.38 |

Table 2. Amino acid composition of casein and other protein fibers[7,8,9)]

| Amino acid | Casein (%) | Wool (%) | Silk (%) |
|---|---|---|---|
| Glutamic acid | 20.3 | 11.9 | 1.3 |
| Proline | 9.3 | 6.6 | 0.4 |
| Leucine | 9.1 | 7.7 | 0.6 |
| Lysine | 8.4 | 2.9 | 0.4 |
| Aspartic acid | 7.5 | 6.5 | 1.6 |
| Valine | 6.3 | 5.6 | 2.4 |
| Tyrosine | 5.8 | 3.8 | 5.1 |
| Serine | 5.2 | 10.4 | 11.9 |
| Alanine | 3.3 | 5.4 | 28.9 |
| Glycine | - | 8.4 | 43.9 |
| Others | 24.8 | 30.8 | 3.5 |

$\mathrm{HOOCCH_2CH_2\underset{\underset{NH_2}{|}}{CH}COOH}$ $\quad$ $\mathrm{HOOCCH_2\underset{\underset{NH_2}{|}}{CH}COOH}$

(1) (2)

Fig. 1. Chemical structure of Glutamic acid (1) and Aspartic acid (2).

The reactive groups studied in this investigation included -bromoacrylamide (Lanasol dyes) and difluorochloropyrimidine (Drimalan F dyes)[12,13)].

# 2. Experimental

## 2.1 Materials

100% Casein plain-fabric was woven by Cho Yang Wool Textile Co. The casein fabric was scoured and bleached prior to dyeing. Scouring and bleaching was carried out in one stage using with 3g/l of scouring agent (Sunmorl CS-1 supplied from Nicca Korea), 3g/l of stabilizer (Neorate PLC-8800D supplied from Nicca Korea) and 8g/l of $H_2O_2$ (30% wt, Aldrich) at 95 C for 60 minutes.

## 2.2 SEM analysis

The surface change of the casein fabric by scouring and bleaching treatments was investigated by a scanning electron microscope (Jeol JSM 5800-LV).

## 2.3 Commercial acid and wool reactive dyes

To evaluate the dyeability of casein fiber, 9 acid dyes containing levelling, milling and metal-complex types, and 6 wool reactive dyes were used in this study. The commercial dyes provided by corresponding manufacturers, as listed in Table 3 and Table 4.

## 2.4 Dyeing procedure

The dyeings were performed under an exhaust dyeing method in a laboratory dyeing machine (KS-W24 Inter Cooler IR Dyeing machine, Korea Scientific Co.). The casein fabric specimens were dyed at a liquor-to-goods ratio of 20:1. Dye bath pH was adjusted to pH 3 using sodium acetate/acetic acid buffer system. In case of acid dyes a levelling agent (1% o.w.f., Newbon MG, Nicca Korea)was added into the dye bath. The dyeing commenced at 40℃ which was then raised to 95℃ at a gradient of 1.5℃/min. After holding at this temperature for 40 minutes, and then cooled to 70℃ at 2℃/min. In the case of wool reactive dyeings, the fabric was removed from the dyebath and rinsed with a soaping agent (2 g/l, Lipotol RF-101, Nicca Korea).

## 2.5 Effect of dyeing temperature on exhaustion

To determine the optimum dyeing temperature, the temperature was varied as 90℃, 95℃ and 100℃ using levelling type acid dyes, Telon Yellow RNL micro, Telon Red FRL micro and Telon Blue BRL micro. The corresponding K/S values at $\lambda_{max}$ were evaluated.

## 2.6 Effect of pH on exhaustion

The pH in the dyebath was adjusted 3, 4, 5 and 7 using acetic acid. Dyeing at each pH conditions was performed at 95℃ for 40 min, and the corresponding *K/S* values at $\lambda_{max}$ were evaluated.

## 2.7 Build-up properties

Dyeing concentrations of 1, 2, 3 and 4% o.w.f. were employed and the corresponding *K/S* values were measured. The dyeing conditions were described above.

## 2.8 Determination of dye exhaustion yield

The dye uptake was obtained using a UV-visible spectrophotometer (Shimadzu UV-2100, Japan), measuring the absorbance at the wavelength of maximum absorption of the dye. The % percentage dye bath exhaustion (%*E* )was calculated using equation(1):

$$\%E=(A_0-A_1)/A_0 \times 100 \qquad (1)$$

where, $A_0$ and $A_1$ are the absorbance at $\lambda_{max}$ of the dye originally in the dyebath and of the residual dye after dyeing, respectively.

## 2.9 Colorimetric measurements

The colorimetric properties of the dyeing were measured by a Datacolor SF 600 plus spectrophotometer (Data color International) using $D_{65}$ illuminant and 10° observer, UV excluded and specular component included. The fabrics were measured at three locations and the results averaged.

## 2.10 Color fastness

The wash fastness of each dyeing was performed using the test method ISO 105 C06 B1S to evaluate color change and staining of adjacent multifibers. The light fastness test was conducted according to ISO 105 B02 method assessing fading with gray scale by exposure at 63℃ for 20 hours. The color fastness to rubbing was evaluated using ISO 105-X12 method for 10 times forwards and backwards. The perspiration fastness test of acid and alkaline was conducted according to ISO 105-E04 method. All the fastness was evaluated at 2% o.w.f. dyeings.

# 3. Results and Discussion

## 3.1 Scouring and oxidative bleaching

The scouring and bleaching conditions were optimized by varying the amount of desizing agent, hydrogen peroxide, stabilizer and scouring agent. Due to the weakness of casein fiber in alkaline solution[14)], it was not suitable to use an alkali so that the fiber to be intact.

Although the use of alkali combined with a desizing agent, such as $Na_2CO_3$, improved the efficiency of removal of impurities, the observed yellowing effect was extremely unacceptable. Stabilizer is necessary to retard the decomposition of hydrogen peroxide to control the speed of bleaching process. In this study, a stabilizer based on poly hydroxy acrylic acid was used[15)].

Fig. 2 shows microscopic photographs of scoured and bleached fabrics in comparison with those of untreated.

## 3.2 Effect of dyeing temperature on exhaustion

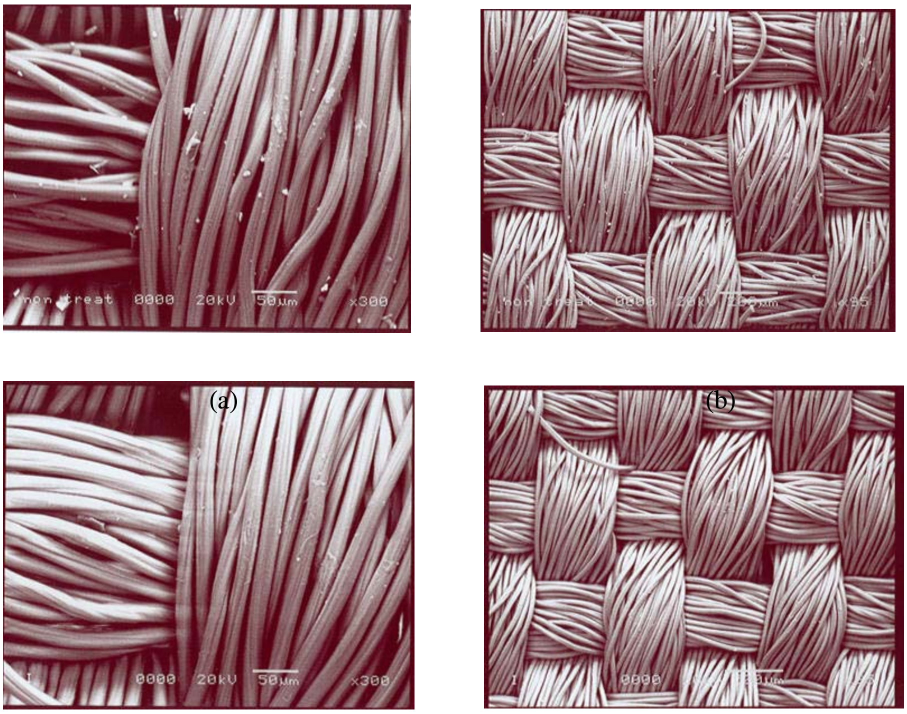
The level of exhaustion depending on dyeing temperature in the range of 90℃-100℃ indicated its very low variation. The *K/S* value at 90℃ was slightly higher than 95℃ for dye **2**, whereas dye **1** and dye **3** showed an identical performance between 90℃ and 95℃ dyeings. In this study, the dyeing at

Table 3. Acid dyes used in this study

| Dye | Type | Commercial name | C. I. generic name | Manufacturer |
|---|---|---|---|---|
| 1 | Levelling | Telon Yellow RNL micro | C.I. Acid Yellow 230 | DyStar |
| 2 | | Telon Red FRL micro | C.I. Acid Red 337 | |
| 3 | | Telon Blue BRL micro | C.I. Acid Blue 324 | |
| 4 | Milling | Dorasyn Yellow C3GL | C.I. Acid Yellow 236 | M. Dohmen |
| 5 | | Dorasyn Red C2B | C.I. Acid Red 138 | |
| 6 | | Dorasyn Blue C4RNL | C.I. Acid Blue 277:1 | |
| 7 | Metal complex | Lanaset Yellow 2R | Unknown | Ciba |
| 8 | | Lanaset Red 2B | Unknown | |
| 9 | | Lanaset Blue 2R | C.I. Acid Blue 225 | |

Table 4. Wool reactive dyes used in this study

| Dye | Commercial name | C. I. generic name | Manufacturer |
|---|---|---|---|
| 10 | Lanasol Yellow 4G | C.I. Reactive Yellow 39 | Ciba |
| 11 | Lanasol Red 6G | C.I. Reactive Red 84 | |
| 12 | Lanasol Blue 3G | C.I. Reactive Blue 69 | |
| 13 | Drimalan G/Yellow F-3RL | C.I. Reactive Yellow 125 | Clariant |
| 14 | Drimalan Br. Red F-B | C.I. Reactive Red 147 | |
| 15 | Drimalan Blue F-GRL | C.I. Reactive Blue 209 | |



(a) (b)

(c) (d)

Fig. 2. SEM photographs of casein fabric; (a) before scouring and bleaching (×300) (b) before scouring and bleaching (×95) (c) after scouring and bleaching (×300), and (d) after scouring and bleaching (×95).

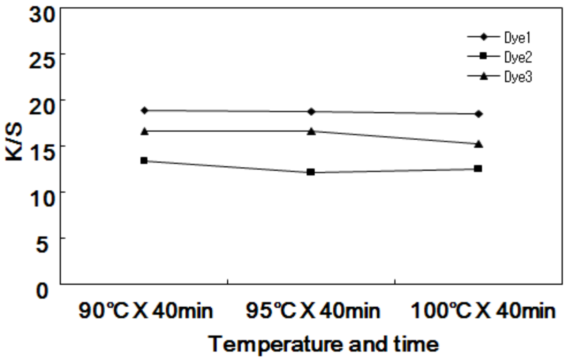


Fig. 3. Color yield of levelling type acid dyes 1-3 at different dyeing temperature.

95℃ was selected as an optimum condition by considering the dyeing temperature of general acid dyes. The results of color yield exerted by temperature dependence are given in Fig. 3.

## 3.3 Effect of pH on dye exhaustion

As illustrated in Table 2, casein fiber consists of various proteins, thus the adsorption of ions presented in the dye by casein fiber is directly associated with the presence of both $NH_3^+$ and $COO^-$ groups of the casein protein[16]. Since the proportions of amino acids included in casein fiber are more similar to wool compared to those in silk, it can be assumed that the behavior of dye adsorption onto casein fiber to be coincided with wool. The effect of pH on the dyeing is decisive, since it not only controls the rate of dye uptake into the fiber, but also the subsequent properties of the dyed polymer system. Dye uptake is one such factor and here this is related to variation in pH of the dye bath. The titration of a hypothetical, water-soluble proteins, including aspartic acid, lysine and arginine, suggested the isoelectric point of the proteins exists at a pH between 4 and 5 where the number of negative groups will equal the number of positive groups[16].

To investigate the influence of pH on dye exhaustion, acetic acid was used to adjust the starting pH as 3, 4, 5 and 7. As shown in Figs. 4-8, most of dyes used exerted much lower exhaustion at pH 4 compared to pH 3 with exceptions of dye **1** and dye **4**. In particular, wool reactive dyes **10-15** showed dramatic decrease in K/S values at pH 4, as also observed with some acid dyes, such as dyes **2, 3, 5** and **6**.

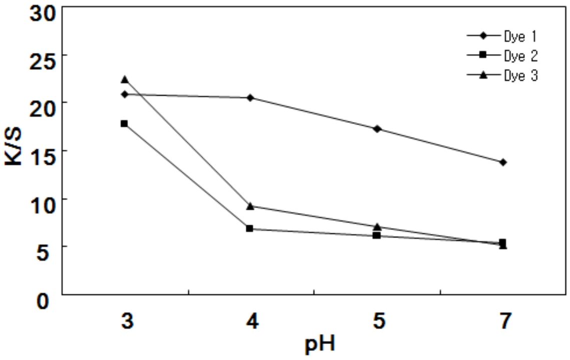


Fig. 4. Color yield of levelling type acid dyes 1-3 to dye casein fabric at different pH.

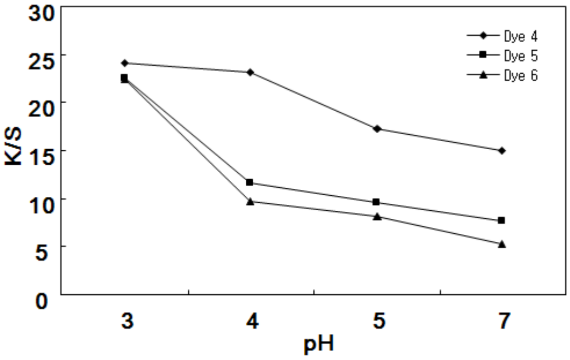


Fig. 5. Color yield of milling type acid dyes 4-6 to dye casein fabric at different pH

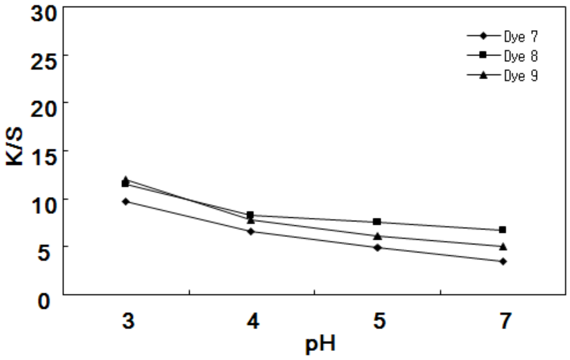


Fig. 6. Color yield of metal-complex acid dyes 7-9 to

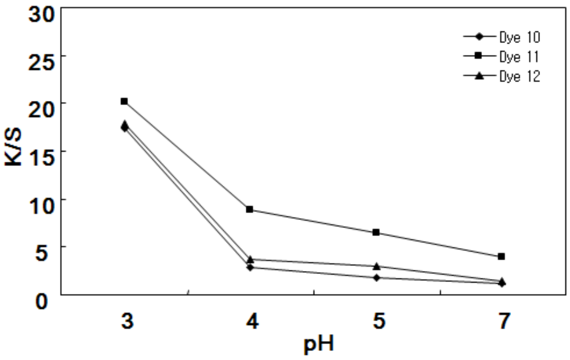


Fig. 7. Color yield of wool reactive dyes 10-12 to dye casein fabric at different pH.

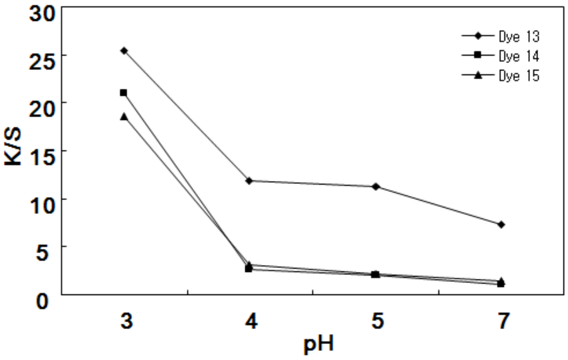


Fig. 8. Color yield of wool reactive dyes 13–15 to dye casein fabric at different pH.

The extent of decrease was comparatively smaller with metal-complex acid dyes than those with other types. The name acid dyes is derived from the fact that water soluble dyes can be more readily applied to wool and silk from aqueous dyebaths containing acids than from neutral dyebaths[17]. It can be assumed that the dyeing of wool reactive dyes under acidic conditions was mainly contributed by the ionic interactions between negative charge of dye molecule and positive charge developed in casein fiber rather than the formation of a covalent bond.

Therefore it was concluded that the low pH less than isoelectric point most enhanced dye exhaustion which behaved like acid dyes to dye protein fibers.

## 3.4 Build-up properties

To achieve deep shades it is required to use the dyes can be increased in dyeing depths at high concentrations. Proper selection requires the build-up properties of the individual dyes to be known. The color yields depending on the dye concentrations were studied are illustrated in Figs. 9-13.

Judging from these results, the highest build-up properties were found with metal-complex dyes **7-9**, as the *K/S* values continued to gradually increase up to 4% o.w.f. dyeings. Lanasol wool reactive dyes **10-12** also exhibited good build-up properties compared to other dyes. However, levelling and milling type acid dyes **1-6** seemed to have less efficient performance in terms of build-up properties than those of dyes **7-12**. This was particularly noticeable with dyes **4** which at 3% o.w.f. was completely saturated, as also shown with dyes **1, 3, 5, 6**.

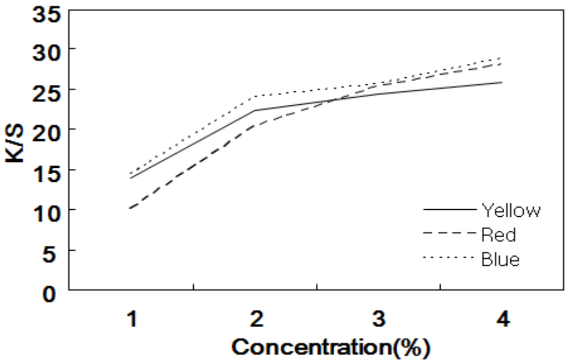


Fig. 9. Build-up properties of levelling type acid dyes at different concentrations.

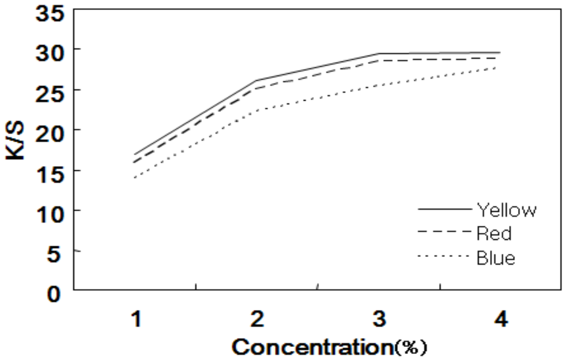


Fig. 10. Build-up properties of milling type acid dyes at different concentrations

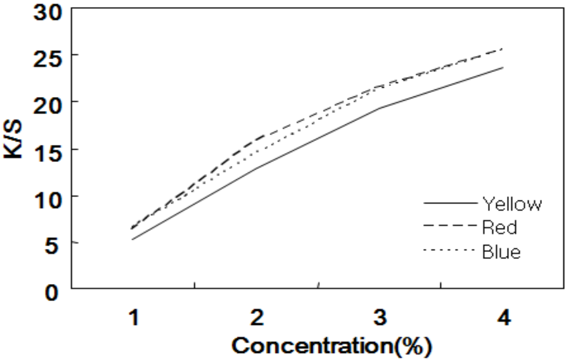


Fig. 11. Build-up properties of metal-complex type

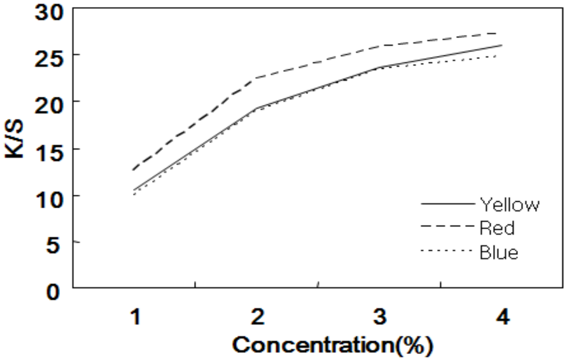


Fig. 12. Build-up properties of wool reactive dyes (Lanasol) at different concentrations.

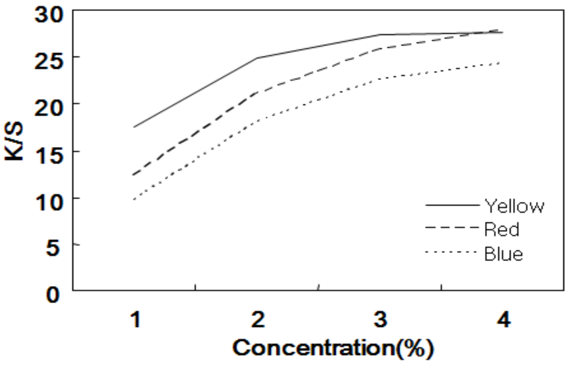


Fig. 13. Build-up properties of wool reactive dyes (Drimalan) at different concentrations.

Table 5. Exhaustion yields (%E ) for the dyes 1-15 (2% o.w.f.) used to dye casein fabric

| Dye | *%E* |
|---|---|
| **1** | 95.2 |
| **2** | 89.9 |
| **3** | 91.0 |
| **4** | 91.1 |
| **5** | 82.8 |
| **6** | 90.7 |
| **7** | 84.2 |
| **8** | 79.1 |
| **9** | 95.9 |
| **10** | 93.1 |
| **11** | 95.7 |
| **12** | 93.7 |
| **13** | 96.8 |
| **14** | 99.6 |
| **15** | 98.6 |

## 3.5 Exhaustion yields

Exhaustion yields (*%E*, 2% o.w.f.) for acid dyes **1-9** and reactive dyes **10-15** were assessed using an exhaustion dyeing method, and the results are shown in Table 5. Within same type of acid dyes, the dye uptake varied depending on the dye structures which are largely responsible for the substantivity, for instance dye **5** appeared to have 8% lower exhaustion yield compared to dye **4**. Larger difference in *%E* was found in comparison of dye **8** and dye **9** these are metal-complex dyes, where 79.1% exhaustion by dye **8** was much lower than 95.9% of dye **9**.

Percentage of exhaustion for wool reactive dyes exhibited greater than those for acid dyes with exception of dye **1** and dye **9**. Among these groups, slightly higher exhaustion yields were provided by dyes containing a difluorochloropyrimidine group.

## 3.6 Color fastness

The wash fastness of each sample was assessed by the staining of adjacent multifibers, particularly on cotton and nylon. As expected, the poorest overall wash fastness in the series was given by levelling type acid dye **1-3**, with ratings of 2 for staining of nylon and 3 for cotton in averaged. In contrast, wool reactive dyes **10-15** provided minimal staining of both nylon and cotton indicating their best selection for

Table 6. Wash fastness for dyes 1-15 on casein fiber

| Dye | Color change | Staining on multifiber | | |
|---|---|---|---|---|
| | | Cotton | Nylon | Polyester |
| 1 | 4-5 | 4 | 2-3 | 4-5 |
| 2 | 4-5 | 2-3 | 1-2 | 4-5 |
| 3 | 4-5 | 3 | 2 | 4-5 |
| 4 | 4-5 | 4 | 4 | 4-5 |
| 5 | 4-5 | 4 | 4 | 4-5 |
| 6 | 4-5 | 3 | 2 | 4-5 |
| 7 | 4-5 | 4-5 | 4 | 4-5 |
| 8 | 4-5 | 4 | 3-4 | 4-5 |
| 9 | 4-5 | 4-5 | 4 | 5 |
| 10 | 4-5 | 3-4 | 4-5 | 5 |
| 11 | 4-5 | 4-5 | 4-5 | 5 |
| 12 | 4-5 | 4-5 | 4-5 | 5 |
| 13 | 4-5 | 4-5 | 4-5 | 5 |
| 14 | 4-5 | 4-5 | 4-5 | 5 |
| 15 | 4-5 | 4-5 | 4-5 | 5 |

Table 7. Light, rubbing and perspiration fastness for dyes 1-15 on casein fiber

| Dye | Light | Rubbing | | Perspiration (nylon) | |
|---|---|---|---|---|---|
| | | Dry | Wet | Acid | Alkali |
| 1 | 4-5 | 4-5 | 4-5 | 2 | 1-2 |
| 2 | 4-5 | 4-5 | 4 | 1 | 1 |
| 3 | 4-5 | 4-5 | 4 | 1 | 1 |
| 4 | 4-5 | 4-5 | 4-5 | 4 | 3 |
| 5 | 4 | 4 | 3 | 3 | 2-3 |
| 6 | 4-5 | 4-5 | 4 | 1-2 | 1 |
| 7 | 4-5 | 4-5 | 4-5 | 4 | 4 |
| 8 | 4-5 | 4-5 | 4-5 | 4 | 3-4 |
| 9 | 4 | 4-5 | 4-5 | 3 | 2-3 |
| 10 | 4-5 | 4-5 | 4-5 | 2-3 | 2 |
| 11 | 4-5 | 4-5 | 4-5 | 2-3 | 2 |
| 12 | 4 | 4-5 | 4-5 | 2 | 1-2 |
| 13 | 4-5 | 4-5 | 4-5 | 3 | 2-3 |
| 14 | 4 | 4-5 | 4 | 3-4 | 3 |
| 15 | 4 | 4-5 | 4-5 | 4-5 | 4 |

high wash fastness. For milling type and metal-complex acid dyes **4-9**, their ratings were satisfied except dye **6** which gave significant staining of nylon. In overall, the data shown in Table 6 are in agreement with general results of wash fastness by acid dyes and wool reactive dyes.

In case of light fastness evaluations, all the dyes revealed to have excellent photo-stability with rating of over 4. The rubbing fastness also exhibited satisfactory levels better than rating 4 with an exception of dye **5** for wet rubbing fastness. Apparently, metal-complex acid dyes **7-9** and wool reactive dyes **10-15** seem to be the most suitable combinations to give good rubbing fastness.

However, for perspiration fastness most dyes deteriorated both acid and alkali conditions. In particular, levelling type dyes **1-3** gave significant staining of nylon rated less than 2. The dyeings with wool reactive dyes except dye **14** and **15** also showed poor results with ratings lower than 3. It was thus revealed that metal-complex dyes **7-9** were superior to the remainder of the series in terms of perspiration fastness.

## 4. Conclusions

Prior to the dyeing, scouring and bleaching treatments of casein fiber were required. The use of alkali, such as $Na_2CO_3$, improved the efficiency of removal of impurities, however the resulting yellowing effect was extremely unacceptable. Therefore oxidative bleaching excluding an alkali was the best compromised condition.

Both acid dyes and wool reactive dyes used exhibited good exhaustion yields in the range of 79-99%.

In particular, the effect of pH in the dyebath was significant between pH 4 and pH 3, where dramatic decrease in *K/S* values was observed at pH 4 in comparison with those at pH 3.

The data obtained clearly indicate that the dyeing of reactive dyes under low pH conditions was mainly attributable to the ionic interactions between negative charge of dye molecule and positive charge developed in casein fiber rather than the formation of a covalent bond.

In terms of build-up properties, metal-complex dyes exhibited best performance and followed by wool reactive dyes containing -bromoacrylamide group.

As expected, the poorest overall wash fastness in the series was given by levelling type acid dyes rated less than 3.

Wool reactive dyes gave minimal staining of both nylon and cotton. All dyes provided good light and rubbing fastness, but comparatively poor perspiration fastness was observed.

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