단일상 천연쪽의 특성 및 응용

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Application and Characterization of Single Phase Polygoum Tinctoria

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Introduction

Indigo is one of the oldest dyes used throughout the history of mankind. There are many indigo dye precursor-containing plants all over the world, but the most common ones are indigofera tinctoria, isatis tinctoria and *polygonum tinctorium*. Indigofera has more than 800 different types of its own kinds and it spreads from India to tropical regions of Southeast Asia. *Polygonum tictorium* is most widely grown in the Far East region like China, Japan, and Korea [1].

Traditional dyeing method used in Korea is to make the indigo paste. The leaves of indigo plant are soaked in water for couple of days to extract indican. The leaves are then eliminated and the indican containing solution is mixed with lime water and stirred vigorously to oxidize the dye which becomes insoluble in water and settles at the bottom of the vessel. The water is then poured out to finally obtain the indigo paste. The carbonyl group in the insoluble dye is reduced in alkaline state to make the leuco dye. The fiber is dyed in this solution and the dye is oxidized to indifotin to give the blue color[2,3].

Generally, natural dyes have moderate colors and are environmentally friendly, while harmful mordants must be used for fixing them and obtaining a full color range on fabrics and it is very hard to improve K/S of the dyeing and mordanting[4]

In this study, physicochemical characterization of *polygoum tinctoria* dye substance was investigated, and *polygoum tinctoria* dyeing was carried out various fabrics according to the various dyeing conditions - fermentation temperature, dyeing temperature, dyeing time, and kinds and amounts of alkali. And then the values of K/S according to dyeing conditions were investigated by colorimeter. Also we investigated to dyefastness against the light, washing, perspiration and friction by KS (Korea Industrial Standard).

Experimental

Materials

Indigo and fabrics (silk, cotten, yam) were purchased from Ban-Suk (Jeonnam, Korea). Synthetic indigo, acetonitrile, cholroform and DMSO (Aldrich), as a reagent grade were used without further purification.

Preparation and Characterization of single phase polygonum tinctroium

The single phase *polygonum tinctroium* dye was prepared by dorr agitator and lyophilization and physicochemical characterization of polygoum tinctoria dye substance was investigated by UV/VIS spectrophotometer, TLC, DSC, FT-IR spectrophotometer, ¹H-NMR spectrophotometer, HPLC, MALDI-TOF mass, CHNS/O. Also cytotoxicity study of *polygonum tinctroium* dye was carried out MTT-assay.

Dyeing and Evaluation of dyeability

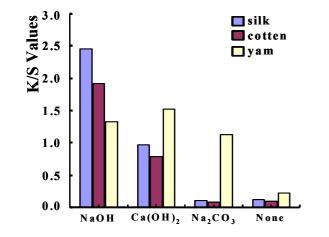
The dyeing was carried out at various dyeing conditions- kinds of alkali, amounts of alkali, dyeing temperature, and dyeing time. Reflectance of dyed fabrics was measured at every 10 nm between 400 to 700 nm by Chroma meter (CR-340, MINOLTA). The K/S values were calculated using Kubelka-Munk equation. $K/S=(1-R)^2/2R$

Where in, R: Surface reflectance at wavelength of maximum absorption, K: Absorption coefficient, and S: Difussion coefficient, respectively.

Results and discussion

We confirmed that ingredients of *polygonum tinctorium* were indigo (70.50wt%) as blue pigment and indirubin (29.50wt%) as red pigment by TLC and HPLC. Also, we confirmed quantitative results of indigo and synthetic indigo by various spectroscopic methods such as UV/VIS spectrophotometer, TLC, DSC, FT-IR spectrophotometer, ¹H-NMR spectrophotometer, and MALDI-TOF mass.

From the results of K/S values of according to the kinds of alkali, the highest K/S values was obtained in sodium hydroxide in all fabrics, and the sodium carbonate did not influence to K/S values except yam (Fig. 1). From the results of K/S values of according to the amounts of sodium hydroxide, the K/S values increased until the sodium hydroxide concentration reached 3 g/L in all fabrics (Fig. 2).



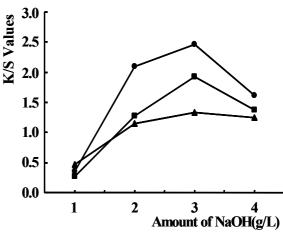


Fig. 1. K/S values of silk, cotten, and yam with *polygoum tinctoria* dyes as a function of various alkali.

Fig. 1. K/S values of silk(\bullet), cotten(\blacksquare), and yam(\blacktriangle) with *polygoum tinctoria* dyes as a function of various sodium hydroxide concentration.

From the results of K/S values of according to the amounts of dextrose, the K/S values increased until the dextrose concentration reached 3 g/L in all fabrics. From the results of K/S values of according to the vatting temperatures, the highest K/S values were obtained at 95 $^{\circ}$ C in all fabric. From the results of K/S values of according to the dyeing temperatures, the highest K/S values was obtained at 30 $^{\circ}$ C and the K/S values decreased noticeably at 50 $^{\circ}$ C in all fabric (Fig. 3). This shows that the dye is quite unstable at high temperatures. From the results of K/S values of according to the dyeing to increase the dyeing times, while for yam, K/S values was increased for the first 10 minutes, and then decreased to increase the dyeing times (Fig. 4).

Table 1, 2 show the colorfastness to light, washing, perspiration and friction by KS(Korea Industrial Standard). The colorfastness to light, washing, perspiration and friction showed grade of $4 \sim 5$ in all fabrics.

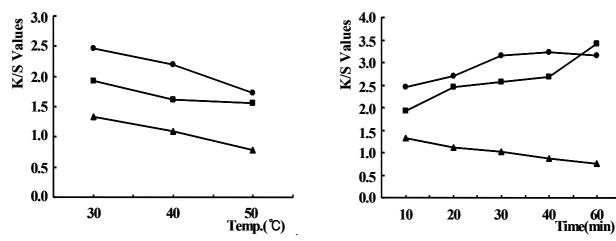


Fig. 3. K/S values of silk(\bullet), cotten(\blacksquare), and yam(\blacktriangle) with *polygoum tinctoria* dyes as a function of various dyeing temperature

Fig. 4. K/S values of silk(\bullet), cotten(\blacksquare), and yam(\blacktriangle) with *polygoum tinctoria* dyes as a function of various dyeing times.

 Table 1. Color fastness to light, washing, perspiration and friction of fabrics with polygoum tinctoria dyes*.

Fastness	washing	light	perspiration		friction
fabrics			acid	alkali	
silk	4~5	4~5	5	5	4~5
cotton	4~5	4~5	5	5	4~5
yam	4~5	4~5	5	5	4~5

*dyeing conditions: *polygoum tinctoria* dyes: 40Kg, dextrose: 30g, sodium hydroxide: 30g, vatting temperature: 95° C, dyeing temperature: 30° C, dyeing time: 10min

Table 2. Color fastness to light, washing, perspiration and friction of fabrics with mixing dyes composed *polygoum tinctoria* dyes and synthetic indigo dyes*.

Fastness	washing	light	perspiration		friction
fabrics			acid	alkali	
silk	4~5	4~5	5	5	4~5
cotton	4~5	4~5	5	5	4~5
yam	4~5	4~5	5	5	4~5

*dyeing conditions: mixing dyes (*polygoum tinctoria* dyes: synthetic indigo dyes=7:3): 40Kg, dextrose: 30g, sodium hydroxide: 30g, vatting temperature: 95° C, dyeing temperature: 30° C, dyeing time: 10min

Conclusions

From result that analyze *polygoum tinctoria* dyes and synthetic indigo dyes to Solve problem of dyeing that use *polygoum tinctoria* dyes and for more efficient dyeing, we got conclusion as follow.

1. *polygoum tinctoria* was composed indigo (70.50wt%) and indirubin (29.50wt%). And we introduced to quantitative results of natural indigo by various spectroscopic methods.

2. The highest K/S values was obtained when concentration of dextrose were 3 g/L, concentration of sodium hydroxide were 3 g/L, vatting temperature and dyeing temperature were 95 °C and 30 °C, respectively, and dyeing time was 10min.

3. The colorfastness to light, washing, perspiration and friction of all fabrics showed grade of $4 \sim 5$ when dyeing was carried out optimum conditions

References

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