



Investigation on the Natural Indigo Dye for Cotton Fabric by using Supercritical Carbon Dioxide

Ryoi Takamatsu

Supervisors: Dr. Veronica Malm

Abstract

The textile industry is grappling with growing concerns about its environmental impact and the urgent need for sustainable technologies. Whereas conventional dyeing processes require water and chemicals, supercritical carbon dioxide scCO₂ technology is considered a resource efficient alternative. This technology uses carbon dioxide as a solvent instead of water, thereby eliminating liquid waste from the process. However, its industrial applications are mostly limited to hydrophobic synthetic fabrics dyed with hydrophobic synthetic dyes due to scCO₂'s nonpolar properties. Efforts are underway to extend the application of scCO₂ to dyeing cellulose fabrics, aiming to address environmental concerns and water scarcity issues. This thesis explores dyeing cotton with natural indigo using scCO₂, with the objective of developing dyeing methods with a lesser environmental impact. While there have been reports on scCO₂ dyeing of cotton using vat dyes, to which indigo belongs, no research is available on the application of scCO₂ dyeing using natural indigo on cotton. Natural indigo, being hydrophobic and nonpolar, is potentially compatible with scCO₂ dyeing. This dye is sustainable, biodegradable, and poses no harm to humans. The challenges of this study include the solubility and coloration of natural indigo, the influence of its impurities, and the swelling effect of hydrophilic polyethylene glycol in hydrophobic scCO₂ dyeing.

The first experiment investigated the color strength (K/S) of indigo on cotton fabric using scCO₂. Mercerized cotton fabric was dyed using 1% o.w.f. (on the weight of fabric) natural indigo under 250 bar and 80°C for 60 minutes, with or without added polyethylene glycol as a swelling agent. The samples were then washed with detergent and rinsed in an ultrasonic bath at 70°C for 20 minutes. Results suggested that indigo does increase the K/S values, albeit minimally. The K/S value increased with the use of the swelling agent, which impacted the hydrogen bonding in cotton, allowing for greater dye penetration.

Based on these findings, the second dyeing experiment focused on the effects of varied process conditions. These included time (60, 120, 180 minutes), temperature (80, 100, 120°C), indigo concentration (1, 3, 5% o.w.f.), and swelling agent amount (1, 3, 5ml) on mercerized cotton fabric dyed under scCO₂. Again, the samples were washed and rinsed in an ultrasonic bath. The maximum K/S value was achieved with a dyeing time of 120

minutes, in a temperature of 120°C, a dye concentration of 5% o.w.f., and a swelling agent amount of 3ml.

FTIR analysis showed minimal characteristic cotton spectra, with dyed samples displaying slight increases in peaks indicating the presence of hydroxyl groups, ketone groups, and double-bonded carbons from indigo. However, these increases were too slight to conclusively establish the presence of indigo on the cotton fabric. Color fastness to washing was not excellent, but rubbing fastness was good.

This study demonstrates the potential for dyeing cotton with natural indigo using scCO₂ and the role of swelling agents in increasing color strength. Adjusting temperature, time, dye concentration, and the amount of swelling agent can contribute to further enhancements in color strength. However, improving color strength or fastness to washing remains a challenge for the future, requiring innovative chemical strategies to bolster dye strength and maintain colorfastness. Exploring more suitable swelling agents or co-solvents to improve indigo solubility in scCO₂ is a future avenue worth exploring.

Keywords: Dyeing, supercritical carbon dioxide, indigo, cotton, eco-friendly, textile

Academic year 2022-2023

