

Water free dyeing of polypropylene fabric under supercritical carbon dioxide and comparison with its aqueous analogue

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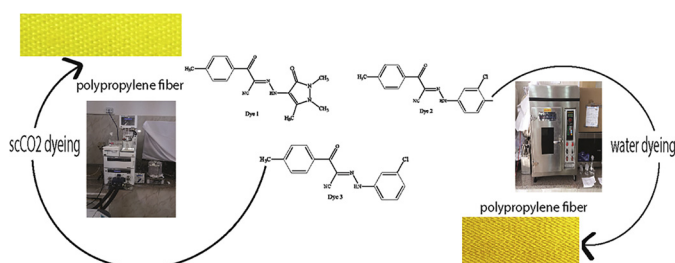
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GRAPHICAL ABSTRACT



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ABSTRACT

A facile one step method was developed for dyeing polypropylene fabrics with hydrazono propanenitrile disperse dyes under both aqueous and supercritical carbon dioxide mediums. The dyeing was carried out without any modification of the fabric and the results were compared. The dyeing parameters, mainly, dyeing concentration, temperature, time, and pressure in both water and scCO_2 mediums were studied. Also, the dye uptake expressed as colour strength (K/S) was measured for both dyeing mediums and it was observed that colour strength of the dyed polypropylene fibers in scCO_2 was significantly better than in water. The fastness properties (washing, rubbing, light and sublimation) of dyed samples in scCO_2 and water exhibited very good results. The ATR-FTIR of dyed samples in water and scCO_2 were found to be in good agreement with the structure of the dyestuff under application.

1. Introduction

Polypropylene (PP) is a remarkable member of the stereo regular polymer. It has several feature properties that have become of essential commercial importance in the manufacture of home furnishing and industrial applications. The characteristic features are good abrasion resistance, high stain resistance, good antistatic character, good chemical resistance, low cost, high toughness, and resilience [1].

Despite the unique characteristics, the dyeing of polypropylene fabrics in water is almost impossible. This goes back to its high

crystallinity and non-polar aliphatic structure. Besides, the absence of sites where dye molecules can be chemically attracted to the fibers [2]. Therefore, many attempts have been made to overcome this problem [3–5]. Structural modification of the fabrics before dyeing to yield water-bath dyeable PP fibers by chemical reaction and graft co-polymerization [3,4]. Another method for enhancing dyeability to polypropylene fiber by imparting low temperature plasma technique [5]. Also, incorporation of hyper-branched macromolecules into the polymer prior to fiber spinning was reported [1]. Sahinbakh et al reported the dyeing of PP using microwave energy [6] while John et al

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used nanoclay modified with quaternary ammonium salt to make nanocomposite polypropylene (nanoPP). This novel polypropylene is dyeable with both acid and disperse dyes [7,8].

Although modifications of polypropylene fabrics have made it dyeable, the physical and chemical properties of the fiber were changed. Synthesis of hydrophobic dyes is the most successful methods for polypropylene dyeing [2,9].

β -Oxoalkanenitriles are highly multifunctional reagents which undergo a wide range of condensation and cyclization reactions. Azolylketonitriles compounds especially those bearing antipyrine moieties play an important role in modern organic synthesis, because they constitute a particularly useful class of heterocyclic compounds and they can also be used as intermediates in the dyestuff industry. Our group succeeded in synthesizing a set of oxoalkanenitriles, mainly hydrazonepropane nitriles disperse dyes [10] and applied these dyes in dyeing polyester and nylon fabrics [11,12].

Supercritical carbon dioxide is the perfect replacement to overcome the problems of dyeing. Dyeing in supercritical carbon dioxide is based on replacing water with carbon dioxide [13]. The distinctive properties of carbon dioxide are low critical parameters, an inexhaustible resource, non-toxic, no disposal problems [14,15]. There has been a lack of research of the hydrophobic dyes that have high affinity for supercritical carbon dioxide dyeing on polypropylene fiber. Of those that have ability to dye PP, suffered from weak light and wash fastness [14,16]. On the other hand, many dyes used in $scCO_2$ were difficult to apply under conventional aqueous system [17]. In continuation of our previous work, we investigated the affinity of hydrazonepropane nitrile dyes towards polypropylene fabric under aqueous and supercritical medium. The proposed methodology, in addition to ease of dyeing offers a solution of most of the disadvantages reported in previous research for dyeing PP.

2. Experimental sections

2.1. Materials and dyes

A set of 3 disperse dyes were used all over the study. The structure of dyes is illustrated in (Scheme 1) and it was prepared and used in pure form according to our procedure which previously reported [10].

A 100% polypropylene fabric supplied by Shikisen-sha Company (Osaka, Japan) was used as the dyeing substrate. The crystallinity of polypropylene fabrics is given according to the Eq. (1):

$$\text{Crystallinity (\%)} = \Delta H / \Delta H_m * 100 \quad (1)$$

ΔH = melting enthalpy: 105.8 J/g,

ΔH_m = ideal crystal melting enthalpy: 209.1 J/g [18]

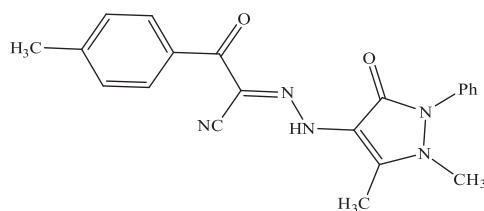
Crystallinity: 50.6%

2.2. Theoretical solubility of the dyestuff

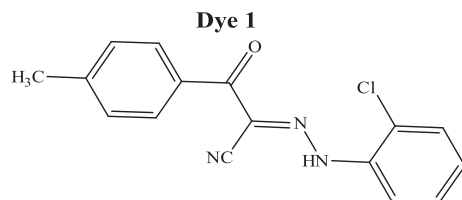
The theoretical solubility parameters (SP) of the disperse dyes under study have been calculated according to Robert F. Fedors's method [19] and was showed in Table 1. The table also represented the solubility of both Polypropylene fiber and $scCO_2$. It can be concluded that the large CO_2 -ligand affinity of the dyes as depicted in (Scheme 1) may be attributed to the presence of functional groups having lone pair of electrons or polar groups including acidic hydrogens.

2.3. Water dyeing apparatus

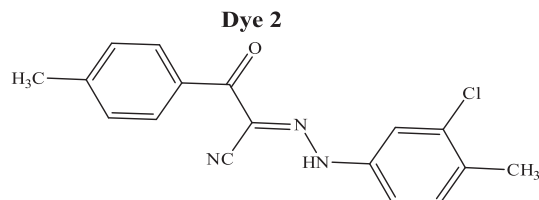
Dyeing in water was carried out using Infra colour dyeing machine. It consisted of beakers that are mounted in a rotating beaker carrying wheel. Heating came through infrared radiation, cooling through air, automation through microprocessor programmer DC4 F/R. The maximum temperature was up to 140 °C, a maximum rate of heating up to



(*E*)-*N'*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)-2-oxo-2-(*p*-tolyl)acetohydrazone nitrile



(*E*)-*N'*-(2-chlorophenyl)-2-oxo-2-(*p*-tolyl)acetohydrazone nitrile



(*E*)-*N'*-(3-chloro-4-methylphenyl)-2-oxo-2-(*p*-tolyl)acetohydrazone nitrile

Scheme 1. Structures of dyes.

Table 1

The theoretical solubility parameters (SP) of the disperse dyes [1–3], polypropylene and $scCO_2$.

| Substrate | SP/ unit |
|-----------|-----------------------------------|
| PP | 8.31 (Cal/cm ³) (1/2) |
| $scCO_2$ | 4.27 (Cal/cm ³) (1/2) |
| D1 | 12.9 (Cal/cm ³) (1/2) |
| D2 | 13.2 (Cal/cm ³) (1/2) |
| D3 | 13.0 (Cal/cm ³) (1/2) |

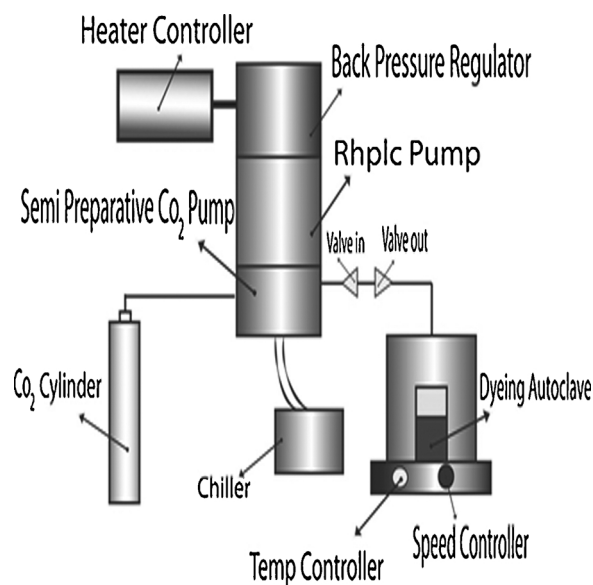


Fig. 1. Diagram of the whole $scCO_2$ apparatus.

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