



## Green reducing agents for indigo dyeing on cotton fabrics

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### ABSTRACT

A reducing agent is used in indigo dyeing to convert an indigo pigment to a water-soluble leuco form prior to its application on cellulosic fibers. Sodium dithionite ( $\text{Na}_2\text{S}_2\text{O}_4$ ), the most widely used reducing agent for industrial indigo dyeing, is considered environmentally unfavorable because sulfite and sulfate generated in the dyebath can cause various problems when discharged into wastewater. In this work, monosaccharides (glucose, fructose and galactose) and reducing disaccharides (lactose and maltose) were investigated as green reducing agents in order to be a substitute for  $\text{Na}_2\text{S}_2\text{O}_4$  in the technical indigo dyeing. The redox potential measurement coupled with photometric studies revealed that reducing sugars could be applied in the alkaline reduction of indigo at an elevated temperature. Within 10 min of adding reducing sugars, the redox potentials of the indigo dyebaths were lower than  $-700$  mV and remained nearly constant for an hour. The dyebaths with use of sodium hydroxide led to greater color strength compared to experiments with use of calcium hydroxide. Replacing  $\text{Na}_2\text{S}_2\text{O}_4$  with reducing sugars in the technical scale denim production was found to be possible in the preparation of the stock vat. This can lead to substantial reduction in  $\text{Na}_2\text{S}_2\text{O}_4$  consumption.

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## 1. Introduction

Natural indigo is a well-known vat dye that can be obtained from leaves and stems of indigo-bearing plants (*Indigofera tinctoria*) mainly cultivated in Africa, India and Southeast Asia (Teanglum et al., 2012). Due to the variation of dye content, the dyeing process of natural indigo requires a high level of expertise and experience to achieve the desired color and shade (Tinnaluck, 2005). The first commercial synthetic indigo was launched by BASF in 1897. Hence, uniform and consistent dyeing by indigo was achieved industrially (Glowacki et al., 2012; Schmidt, 1997).

The chemistry of indigo dyeing involves the oxidation-reduction reaction as shown in Fig. 1. Water-insoluble blue indigo is reduced (vatted) to a yellow leuco indigo by reducing agents in alkaline conditions (pH 11–14) (Komboonchoo et al., 2009). The leuco indigo is substantive toward cellulosic fibers. Air-drying subsequently oxidizes the leuco dye back to the water-insoluble indigo, resulting in a blue shade on the fiber. Indigo is entrapped inside the fiber and

thus is not washed out (Aspland, 1992). The ionization form of leuco molecules depends on pH. The mono-ionic form of leuco indigo is predominant in solution at pH 11.5 and exhibits high affinity to cellulosic fibers, thus indigo dyeing is usually performed in the range of such pH values. The di-anion form of leuco indigo is present at higher pH e.g. 13 which exhibits much lower affinity to cellulose, resulting in lighter dyeings (Etters, 1998; Kumbasar et al., 2006).

Various chemicals and techniques have been employed for the indigo reduction process. Sodium dithionite (hydrosulfite:  $\text{Na}_2\text{S}_2\text{O}_4$ ) is the most common reducing agent used industrially because of its effective reducing power and its availability in an easy-to-handle solid form. Indigo is reduced rapidly by  $\text{Na}_2\text{S}_2\text{O}_4$  even at room temperature hence dye reduction can be achieved in a very short time which is important to maintain stable dyebath conditions in continuous dyeing. However,  $\text{Na}_2\text{S}_2\text{O}_4$  can easily be oxidized by atmospheric oxygen and it is not stable in an alkaline solution particularly at elevated temperatures (Blackburn et al., 2009). By-products generated from the reaction of  $\text{Na}_2\text{S}_2\text{O}_4$  especially sulfite and sulfate ions can cause an adverse effect in the environment due to their toxicity and corrosive nature (Meksi et al., 2012).

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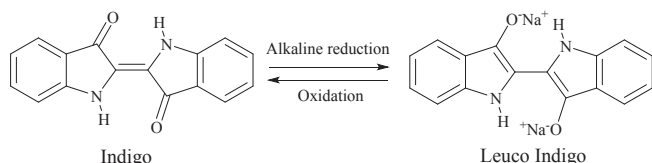


Fig. 1. Oxidation-reduction reaction of indigo.

Possible environmentally favorable alternatives for indigo reduction have been proposed such as electrochemical techniques, hydroxyacetone, bacterial reduction, fruit extracts, and glucose. Various electrochemical reduction techniques for the reduction of indigo have been employed such as the indirect electrochemical reduction method using a mediator (Chavan and Chakraborty, 2001; Kulandainathan et al., 2007a), direct electrochemical reduction via the radical process (Roessler et al., 2001) or on graphite electrodes (Roessler and Crettenand, 2004) and electrocatalytic hydrogenation (Bechtold et al., 2008; Roessler et al., 2002). The advantages of electrochemistry for indigo reduction are to minimize the utilization of chemicals and to monitor the process by controlling the redox potential. However, a large consumption of electrical energy and the requirement of electrodes with high surface area need to be considered (Teli et al., 2001).

Since the redox potential of indigo was reported to be more negative than  $-700$  mV (vs. Ag/AgCl, 3M KCl) (Bechtold et al., 1998),  $\alpha$ -hydroxyketones such as hydroxyacetone that has the redox potential of  $-810$  mV (vs. Ag/AgCl, 3M KCl) was used as a biodegradable reducing agent (Chakraborty and Chavan, 2004). However, the reduction rate for indigo at pH 11.5–12.0 was unsatisfactory and an unpleasant smell was also produced from the reaction (Kulandainathan et al., 2007b; Vuorema et al., 2008).

For bacteria induced reduction, Nicholson and John (2005) discovered that *Clostridium isatidis* can decrease the particle size of indigo and produce adequate redox potential to be successfully used in the indigo reduction process. Aino et al. (2010) found that strains belonging to genera *Amphibacillus* and *Oceanobacillus* have major roles in supporting the reduced state of indigo dye during fermentation. Nevertheless, the mechanism of bacterial indigo reduction to date is still unknown (Vuorema, 2008).

Different kinds and parts of fruits i.e. date palm, apple, banana (Hossain et al., 2017), ripe banana (Lasopha et al., 2015), banana peel (Shin et al., 2013) and bokbunja (*Rubus coreanus* Miq.) sludge (Shin et al., 2014) were applied as reducing agents in the indigo dyeing process. The reduction system using these natural products and by-products is claimed to be greener and more eco-sustainable. However, the inconsistency of the material composition can cause problems in reduction efficiency (Shin et al., 2013).

Glucose was successfully employed as a reducing sugar in an indigo alkaline reduction (Shin et al., 2016). An aldehyde group of glucose can be oxidized to carboxylic acid as shown in Fig. 2, while indigo is reduced to leuco indigo dyes (Božić and Kokol, 2008). The combination of glucose with NaOH solution at high temperature

provides stable dyebath conditions and the redox potential in the dyebath is sufficiently negative to maintain indigo in its reduced form (Saikhao et al., 2017). Glucose is eco-friendly, non-toxic, biodegradable and inexpensive (Blackburn and Harvey, 2004).

An attempt to use reducing sugars in the reduction of sulfur dye was reported by Blackburn and Harvey (2004). Hexose and pentose monosaccharides and reducing disaccharides gave the optimum redox potential at about  $-650$  mV to achieve maximum color strength. These reducing sugars could be used as an alternative to standard commercial sulfide-based reducing systems in sulfur dyeing.

However, there have been no studies performed on the possibility to use various reducing monosaccharides and disaccharides as a reducing agent in the indigo reduction system in terms of reduction power together with the absorbance of the dyebath. Hence, reducing monosaccharides and disaccharides were investigated in this current research as green reducing agents to replace Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> in the indigo dyeing process on cotton. Synthetic indigo was used in this study to avoid the inconsistency of the dye content that would happen from using natural indigo. Moreover, relatively high dye aggregation and poor penetration of natural indigo could lead to dye wastes in the effluent, one of the problematic pollutant groups (Kawahito and Yasukawa, 2009). The focus is on the evaluation of redox potentials of sugar-alkali systems used in indigo reduction and dyeing. The reduction potential measurement (vs. Ag/AgCl, 3M KCl) coupled with photometric study of reducing sugars was designed to record redox potential and absorbance at 405 nm simultaneously as a function of reduction time. All these reducing sugars were able to reduce indigo because the respective redox potential in the dyebath was more negative than  $-700$  mV. Based on the experimental data, the direct introduction of reducing sugars instead of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> in the technical scale will be difficult due to high alkalinity and elevated temperatures usually being required in the application of reducing sugars. A strategy is presented here to replace a substantial part of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> in the denim production by use of reducing sugars in the stock vat process.

## 2. Experimental

### 2.1. Chemicals and materials

Indigo (C.I. Vat Blue 1; Class IK) was used as received without further purification (DyStar Textilfarben GmbH, Frankfurt a.M. Germany). For the redox experiments of indigo, reducing agents and alkali (NaOH and Ca(OH)<sub>2</sub>) used were of technical grade quality. D-glucose (Carl Roth GmbH), D-fructose (Riedel de Haen), D-galactose (Carl Roth GmbH), lactose (Carl Roth GmbH), maltose (SERVA) and sodium dithionite (AR grade) were used as reducing agents. The redox buffers were prepared by dissolving 0.528 g K<sub>4</sub>[Fe(CN)<sub>6</sub>]·3H<sub>2</sub>O (Sigma-Aldrich), 0.411 g K<sub>3</sub>[Fe(CN)<sub>6</sub>] (Riedel de Haen), 0.180 g KH<sub>2</sub>PO<sub>4</sub> (Merck) and 0.390 g Na<sub>2</sub>HPO<sub>4</sub>·2H<sub>2</sub>O (Merck) in 100 mL deionized water. Argon gas was employed for de-aeration in the redox measurement. Plain woven cotton fabrics

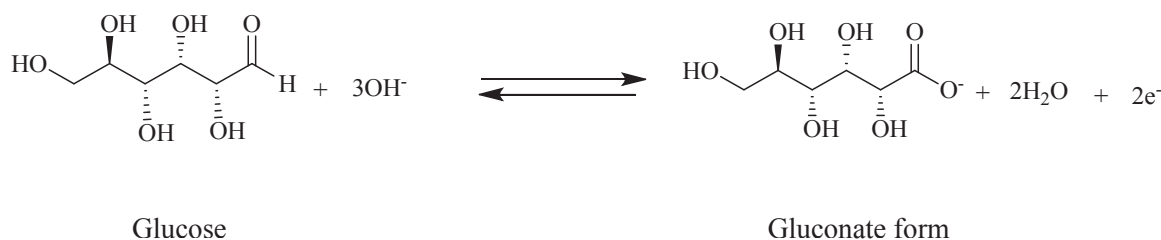


Fig. 2. Oxidation of glucose in alkaline solution.

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