



## Role of major constituents of coconut fibres on absorption of ionic dyes

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### ABSTRACT

Effort was made to understand the role of major constituents of coconut fibres i.e., lignin, hemicelluloses and cellulose, on its absorption of different ionic dyes. Composition and functional groups of raw coconut fibre were changed by progressive removal of lignin, and hemicelluloses. Differently treated fibres were analyzed through FTIR, TGA, composition analysis and were dyed with anionic (acid, reactive) and cationic (basic) dyes separately. Absorption performance was assessed by surface and core colour uptake. Progressive removal of lignin resulted in reduction of uptake of anionic (acid and reactive) dyes, while increase in uptake of cationic (basic) dye. Treatment causing removal of hemicelluloses improved uptake of basic dye due to structural swelling. Increase in  $\alpha$ -cellulose (29.9%), favours uptake of reactive dye to a higher extent. Loss of lignin occurred in case of both delignification and hemicelluloses removal treatments to the extent of 6.3% and 11.9% respectively, consequently uptake of acid dyes reduced. It may be concluded that treatment causing higher removal of hemicelluloses are beneficial and loss of lignin is disadvantageous for absorption of dyes.

### 1. Introduction

Bio-based materials on selective treatments are unanimously accepted as superior absorber for various dyes for treatment of wastewater (Laszlo, 1994; Namasivayam and Sureshkumar, 2006; Kaushik et al., 2008; Parab et al., 2009; Zhang et al., 2013). Absorption has been found to be superior to other techniques (membrane-separation, electrochemical, flocculation-coagulation, reverse osmosis, ozone oxidation, and biological treatment) for wastewater treatment for their simplicity of design, user friendliness, immunity to toxic substance and cost effectiveness (Calabro et al., 1991; Niranjana and Karthikeyan, 1992; Hu, 1994; Churchley, 1994; Lin and Peng, 1994; Stephenson and Sheldon, 1996; Slokar and Marechal, 1997; Vandevivere et al., 1998; Sanghi and Bhattacharya, 2000; Robinson et al., 2001; Dinçer et al., 2007; Joshi and Purwar, 2004; Ugurlu et al., 2007; Kaushik et al., 2008). It is principally a surface phenomenon, which utilizes surface forces viz., physical forces; involves Van der Waals forces and/or hydrogen bonding, and chemical adsorption is due to electrostatic forces and covalent bonding. Adsorption and subsequent absorption of colour particles from the solution to the natural material involves (i) transport of dye particles from the bulk bath solution to the surface of adsorbent; (ii) penetration/migration of adsorbed dye molecules into the pores of adsorbents; (iii) interactions of dye molecules with interior surface bounding of the pores of the adsorbents. However the transport or migration of the adsorbed dye from bath solution to surface and surface

to the core of the materials is the deciding factor in the treatment of dye effluent, which depends on the natural/induced affinity of the dye for natural substrate. Further, effectiveness of different decolourisation techniques for removal of dyes also depends on the dye class (due to the chromophore and auxochromes). Practically, the important textile dyes can be classified into acid, basic, direct, reactive, sulphur, vat, metal complex, and disperse. Generally, highly water soluble dyes, i.e. reactive, acid, basic, sulphur and other dyes are more difficult to remove by conventional techniques than poorly soluble dyes, i.e., vat and disperse dyes (Kaushik et al., 2008).

The natural materials consist of the three major chemical constituent's viz., lignin, hemicelluloses and cellulose. Constituent of cellulose (pulp and paper, cotton), containing more than 90% cellulose, shows much similar absorption behavior in regards of different class of dyes. However, the effect of other constituents like lignin, hemicelluloses, present along with cellulose may also have influence on absorption and/or reaction and retention of different dyes. Direct and reactive dyes (anionic in nature) have inherent affinity for cellulose, even though an electrolyte is used to neutralize the negative potential developed by the cellulose on wet, for improving the migration and adsorption of dyes (Giles, 1971; Chauhan, 2003; Shenai, 2003). Drzewińska, 2008 reported that, application of cationic direct dyes on paper pulp without addition of any dyeing auxiliaries illustrates better attraction compared to its anionic form. This may be due to the modified ionic nature of the dye, enhancing inherent affinity to the

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cellulosic materials, which fits itself in between the planar macromolecular chains of cellulose. However, anionic nature acid dyes are not substantive for the cellulosic materials due to their nonlinear (non planar) structure of the dyes. In cotton, treatment with 18% (w/w) NaOH improves the fine structure, strength and dyeability for dyes (Shenai, 2003). In case of multi constituent cellulosic materials viz., flax, ramie, sisal, pineapple, jute, banana fibre; (containing 3–12% lignin; 10–24% hemicelluloses and 60–80% cellulose) (Satyanarayana et al., 1982; Pandey, 2007; Parab et al., 2009; Rahman and Khan, 2007) they can be dyed with almost all classes of ionic dye (Giles, 1971; Chauhan, 2003; Wang and Ramaswamy, 2005; Perdih and Perdih, 2011) due to their lignocellulosic constituents, which reacts with both, anionic (direct, reactive, acid) and cationic (basic) dyes. Such studies of effect of individual constituents on reactivity or dyeing performance are scanty.

In the present work, an attempt was made to study systematically the influence of major compositions (lignin, hemicelluloses, cellulose) and/or combined or interactive influence of each components of coconut fibre effecting absorption of three differently charged dyes by varying the composition of the fibre. Coconut fibre was chosen; as it has a unique composition having the three constituents in major proportion (contains cellulose (40–45%), lignin (30–40%), and hemicelluloses 24%) (Bledzki et al., 1996; Esmeraldo et al., 2010; Rahman and Khan, 2007). Study on coconut fibre for removal of methylene blue, malachite green blue and congo red dye were already reported (Wong et al., 2013; Etim et al., 2016; Rani et al., 2017). Since, no systematic study on colouration of coconut fibre was carried out till date (van Dam, 2002). Hence, the result of present work will also guide us in finding an appropriate dye and colouration technique for obtaining higher dye uptake and better yield of the colour for coconut fibre. Authors observed in the preliminary study that, conventionally coloured coconut fibre shows poor penetration of the dye (direct, reactive) resulting in surface dyeing leaving core of the fibre undyed and low washing and rubbing fastness properties. It was also thought worthwhile to understand the effects of individual constituent(s) in bringing about the changes in functional groups of coconut fibre.

Coconut fibres having progressively lowered lignin (treatment in acidic condition) and hemicelluloses (treatment in alkaline condition) were prepared and colouration was carried out using acid, basic and reactive class of dyes separately. Changes in composition (analyzed by composition analysis and TGA) effecting the dyeing performance was evaluated by the change in functional groups of the fibre through FTIR. Aesthetic value in terms of surface colour strength (K/S value) of dyed fibres was evaluated. Depth of penetration of dye molecules towards the core of the fibre were assessed through optical microscopy of fibre cross section. Consequently, retention of the dyes was analyzed by fastness property of the coloured substrate using standard methods.

## 2. Materials and methods

### 2.1. Materials

Coconut fibres were collected from Kerala, India. Laboratory reagent grades chemicals, viz., sodium chlorite, sodium hydroxide, sodium chloride, sodium carbonate, glacial acetic acid, polyoxyethylene based non ionic surfactant were procured from E Merc, India. Acid green 25 dye (C.I.61570) was purchased from Atul Industries Ltd. India. C.I. Basic Violet 1 (C.I. 42525) and reactive dye HE8B (C.I. Reactive red 152) were purchased from Hauntsman, India. The structure of the dyes is given in Fig. 1.

### 2.2. Methods

#### 2.2.1. Cleaning of coconut fibre

Raw coconut fibres were boiled in water containing non ionic surfactant (2%) for 1 h to remove surface adhered impurities and some

waxy materials. The cleaned/defatted fibres were then washed before dried in air. The major compositions of defatted coconut fibres are 38.9% cellulose, 32.6% lignin and 24.2% hemicelluloses.

#### 2.2.2. Partial and progressive removal of lignin and hemicelluloses from coconut fibre

Progressive removal of lignin partially from cleaned coconut fibres were accomplished by treating the cleaned fibre with 0.7% NaClO<sub>2</sub> solution at 100 ± 2 °C for 1 h, 3 h, 5 h at pH 4.0. The pH buffer of the bath was maintained by adding 1% (v/v) CH<sub>3</sub>COOH and 1% (v/v) CH<sub>3</sub>COONa. The treated coconut fibers were then subjected to antichlor with 2% (v/v) sodium metabisulphite solution at 50 °C for 30 min, washed and dried in air. Coconut fibres with progressively lower hemicelluloses were obtained by treating the cleaned fibre with 6%, 12%, 18% and 24% (w/w) NaOH solutions separately at different temperature and time. The alkali treated fibers were washed with water, subjected to neutralization with 5% (v/v) CH<sub>3</sub>COOH solution for 30 min, washed again with water and dried in air. For all the treatments, material to liquor ratio was maintained at 1:15 (Ganguly and Chanda, 1993).

#### 2.2.3. Colouration of coconut fibre with acid and basic dyes in hot acidic condition

The dye bath was prepared adding dye (3% acid or basic dye) and acetic acid in water at ambient temperature. The cleaned and treated coconut fibres of varying composition were entered into the dye bath. Then the temperature of the dye bath enhanced to 100 °C within 30 min, and dyeing was carried out at boil, maintaining material to liquor ratio at 1:30. After 60 min, the dye bath was cooled down to 40 °C and the fibers were removed and washed in hot and cold water successively and dried in air (Giles, 1971).

#### 2.2.4. Colouration of coconut fibre with reactive dye in hot alkaline condition

Coconut fibres (both, the cleaned and treated) were added in a bath containing 3% reactive dye, 1% acetic acid (pH 6) at ambient temperature (35 ± 2 °C). Aqueous solution of 3% sodium chloride was added and temperature was slowly raised to 50 °C in 15 min. Another 3% sodium chloride solution was added and temperature is further raised to 85 °C and dyeing was continued for 15 min maintaining the temperature. At 85 °C, After 15 min, 1% sodium carbonate aqueous solution was added in two steps at an interval of 15 min and dyeing was continued further for 45 min, maintaining material to liquor ratio at 1:30. The dye bath was cool down to 40 °C and fibres were removed and washed in water. The dyed fibres were treated with 2% nonionic detergent at 60 °C for 30 min to remove unfixd, hydrolyzed dyes present on the surface of fibre. It was then further washed in hot and cold water successively and dried in air (Chauhan, 2003).

#### 2.2.5. Estimation of major constituents

Analysis of the major constituents (lignin, hemicelluloses, and α-cellulose) of coconut fibre samples was determined using the standard method as described by (van Dam et al., 2004). The results were obtained taking an average of five repeat tests for each case.

#### 2.2.6. Thermal gravimetric analysis (TGA)

To reduce the error in determination of cellulose and hemicelluloses content in conventional process, TG analysis were also done as suggested by Carrier et al. (2011). TGA was carried out using DuPont 1090 thermal analyzer equipped with a 951 TGA module. Finely cut samples weighing 10 ± 2 mg were used for each sample and the experiment was done in a nitrogen atmosphere at a flow rate of 60 cm<sup>3</sup>/min and heating rate of 10 °C/min from 30° to 500 °C.

#### 2.2.7. Analysis of functional group through FTIR

Finely chopped coconut fibre samples were examined in FTIR

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