Recovery and Re-use of Reactive Dyes from Textile Effluents for Colouration of Jute Substrate – An Environmental-Friendly Approach

Dr. Bipin J. Agrawal
Associate Professor
Department of Textile Chemistry, Faculty of Technology & Engineering,
The Maharaja Sayajirao University of Baroda, Vadodara -390 001 (Gujarat), India
e-mail ID: bjagarwal@yahoo.com

***Abstract*—In today's world, the availability of fresh water is one of the many environmental challenges on the universal horizon, which has arisen due to water scarcity, increasing population and water pollution. Therefore, it is important that water is used efficiently. As the cost of water increases, legislation becomes more stringent and enforcement stricter; the recycling of used water becomes a very viable option.** The textiles chemical wet processing industry is also one of the biggest consumers of water, with conventional de-sizing, scouring, bleaching, dyeing, and finishing processes utilizing large amounts of fresh water. This water is ultimately disposed off as waste water containing dyestuffs and chemicals. **It is necessary that the industries must be encouraged to implement efficient water management practices and also to investment in various water recycling methods. Various physico-chemical and biological techniques are utilized for the treatment of textile effluent before discharging it into land and water bodies. Apart from these physico-chemical and biological processes, several membrane technologies have been successfully employed to recover dyes, chemicals and water from the textiles effluents. In the present work, a membrane technology has been utilized for treatment of effluent liquor of reactive dye. The permeate obtained by the effluent treatment has been successfully utilized for its re-application on the jute substrate. The results have been compared with the conventionally reactive colour dyed jute samples on the basis of their colour strengths, in terms of K/S values, and the fastness characteristics.**

Keywords*— *Wastewater recycling, reactive dyes, jute fabric, dyeing, membrane filtration

I. INTRODUCTION

Water is an essential natural resource for sustaining life and environment, which is always thought to be available in abundance and free gift of nature [1]. The amount of water on earth has remained the same and is regarded as an unlimited resource since time immemorial; however, freshwater is become a scarce commodity that is becoming ever increasingly hard to find. Many countries around the world are experiencing some form of water scarcity and more are going to experience it in the future. The surface water sources are limited and availability of water from them vary from year to year depending upon monsoon conditions. The underground water resources are also getting depleted with the increasing amount of water drawn from them every year without adequate replenishments [2, 3].

The textile industry consumes large amount of water in its varied processing operations. In the mechanical processes of spinning and weaving, water consumed is very small as compared to textile wet processing operations, where water is used extensively. Almost all dyes, specialty chemicals, and finishing chemicals are applied to textile substrates from water baths. In addition, most fabric preparation steps, including desizing, scouring, bleaching, and mercerizing use aqueous systems [4]. According to USEPA a unit producing 20,000 lb/day of fabric consumes 36000 liters of water [5].

Textile industries not only consume large quantity of water but also dispose large volumes of effluent to the environment. Textile waste occurs in a variety of forms throughout production process [6]. There is need to adopt economical practices for the use of water in textile industries. It has been estimated that 3.5 % of the total cost of running the industry is required for water utilization in textile industry. The cost of water is rising steeply and it is, therefore, essential that the textile industries should take efficient measures to conserve water [5, 7].

The wastewater from dyeing processes contains a lot of components in various concentrations, for example, dyestuff, alkali, acid, salt, and auxiliaries. In a first basic step, a separation of the wastewater stream according to the degree of chemical load should be performed. The technical and economic feasibility of treating textile waste water can be demonstrated by using physical, chemical and biological processes. Efficient effluent treatment technologies can be implemented not only to reduce the pollution load but also to recycle and reuse the treated water for numerous processes within the industry itself. These techniques are to be applied within a new wastewater treatment system, which are aimed to significantly reduce fresh water consumption and pollutant discharge in chemical wet processing of textiles. A treatment of wastewater with low pollution for reuse can be achieved by the combination of various approaches, such as adjustment of pH and temperature; sedimentation, precipitation; precipitation/flocculation ($Fe^{2+/3+}$, Al^{3+}, polyelectrolyte); filtration (e.g., sand filter); adsorption (e.g., activated carbon); oxidative processes (based on ozone treatment, UV treatment, hydrogen peroxide, Fenton's reagent for the destruction of the chromophore); reductive destruction of coloured dye baths (performed by the addition of reducing chemicals such as $Na_2S_2O_4$ and Fe^{2+} salts); membrane technologies; evaporation; incineration; anaerobic degradation; etc.

In many cases, combinations of the techniques are applied to obtain an optimized process fitting on the individual

situation of the textile dye-house, such as nanofiltraton–oxidation processes; nanofiltration–evaporation–oxidation; evaporation–oxidation, etc. Another full-scale process combines catalytic oxidation including biodegradation, adsorption, precipitation/flocculation, and reverse osmosis.

In the present paper, various technical aspects of membrane filtration technology have been discussed, along with utilization of one membrane filtration technique for the recovery of reactive dyes from textile effluents and their dyeing on jute substrate.

II. TECHNICAL ASPECTS OF MEMBRANE TECHNOLOGY

The increasing cost of water and its wasteful consumption have now induced a treatment process which is integrated in in-plant water circuits rather than as a subsequent treatment. From this standpoint, membrane filtration offers potential applications. Membrane filtration technique is a practical and cost effective solution to the problems for handling textile wastewater pollution [8-10]. The main objectives of the membrane filtration technology are

- to purify incoming water so as to obtain clear water (permeate) through membrane filtration and help recycle used water as many times as is practically feasible so that generation of effluent and treatment costs are reduced. The technique thus helps to conserve water.
- to reduce the volume of wastewater for effective treatment at effluent treatment plant (ETP).

Process houses in Gujarat, Rajasthan and also Tamil Nadu are affected by strictly enforced pollution norms. Existing ETP are not able to achieve these norms. By utilization of membrane filtration technology, one can overcome this obstacle by multiple recycling of their effluent speedily without loading ETP.

Membranes are of different pore size and it is necessary to select membranes of appropriate pore size for specific purpose so that effluent dye liquor (EDL) from different dyes, wash liquors and wastewater could be purified and permeate could be recycled a number of times.

There are three broad categories of membrane filtration. They are –

- Micro- and Ultra-filtration (M.F. and U.F),
- Nano-filtration (N.F) and
- Reverse Osmosis (R.O)

Processes using membranes provide very interesting possibilities of separating hydrolyzed dyestuffs and dyeing auxiliaries, thus simultaneously reducing colouration and the BOD/COD of the wastewater. It is difficult to recommend a particular membrane process (MF, UF, NF and RO) for textile effluent treatment, and the choice is guided by the desired quality of the permeation results.

The dispersions and solutes separated by these membranes are mentioned in Table I and Figure 1, while pore size of membranes vis-à-vis approx. molecular weight cut off (MWCO) point is given in Table II.

TABLE 1. SOLUTES SEPARATED BY MEMBRANE FILTRATION

Type of filtration	Dispersions & Solutes blocked	Solutes allowed to pass through
Micro-/ Ultrafiltration	Pigments, resins, latex, emulsifiers, enzymes, oils, polymers, binders, thickeners	Dyes, salts, detergents
Nanofiltration	Polyvalent salts, dyes, detergents	Monovalent salts
Reverse osmosis	Salts, sugars, ions	None; only dissolved gases

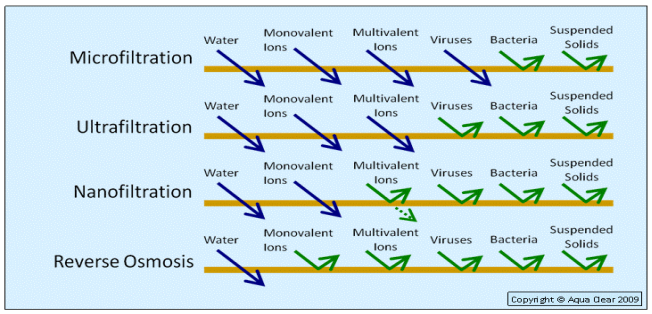


Fig. 1. Schematic representation of solute separation by different membranes

TABLE II. SALIENT FEATURES OF MEMBRANE FILTRATION

Type of filtration	Pore size	MWCO point (approx.)	Operating filtration pressure (kg/cm^2)
Ultrafiltration	5–100	1000	10
Nanofiltration	1–5	200–1000	15 – 30
Reverse osmosis	< 1.0	< 200	30 – 60

Membrane filtration is not like conventional filtration where under pressure insoluble matter remains on the filter and liquid passes through [11, 12]. If such high levels of pressure are applied to membranes, it will tear apart. Membrane module is so designed that the liquid, free from particulate matter, passes through a rolled up module of membranes and separators, when higher molecular weight compounds slip out as a separate stream of concentrates while lower molecular weight compounds pass through the membrane and are recovered as permeates as shown in Figure 2.

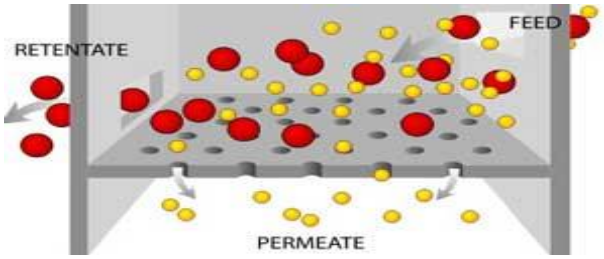


Fig. 2. Working principle of a typical membrane

There are many ways in which membrane modules can be made e.g. tubular, plate and frame, spiral and hollow fibre type; but the most common ones are spirally wound modules. For water purification and recycling processes the following aspects have to be considered:

- high permeate flow;
- selectivity;
- stability and life-time of membrane and equipment;
- cleaning of membrane;
- tendency for membrane fouling;
- cost.

III. Utilization Of Membrane Technology For Recovery Of Reactive Dyes From Textile Effluents

In case of reactive dyes, wastewater problem mainly arise from three different sources [13]:

- dyestuff: coloured effluents, AOX, heavy metal content (Cu, Ni);
- dispersing agents in dyestuff formulation: COD, poor biodegradability;
- auxiliaries, chemicals added: salt content (NaCl, Na2SO4), pH value (caustic soda, soda ash, silicates), N-content (urea).

For reactive dyes, the content of heavy metals (e.g., Cu, Ni from phthalocyanine dyes) and AOX values from covalently bound halogen are quite high. Selection of processes with a high fixation of dyestuff yields a considerable decrease in Cu/Ni concentrations and AOX values. For the fixation of reactive dyes on cellulosic substrates, certain amounts of alkalies are added to the dyebath. As the total amount of alkali used is low compared to the consumption of alkali during mercerization, scouring, and bleaching, high pH due to the alkali from reactive dyeing is of minor relevance. Two main problems have to be mentioned in connection with reactive dyeing [14]:

- High load of soluble salt (NaCl, Na2SO4): For acceptable exhaustion of dyes, considerable concentrations of salt (up to 50 g/l) are required in exhaust dyeing processes. The release of the used dyebath transports a rather high load of salt into the wastewater stream. When a liquor ratio of 1:10 is applied, 10 litres of dyebath are used for dyeing of 1 kg of goods; thus at a salt concentration of 50 g/l an amount of 0.5 kg salt is released for dyeing of 1 kg of goods.
- Coloured wastewater: The problem of relatively high dyestuff concentrations in wastewater particularly arises when dyestuff exhaustion and fixation proceed only to a limited degree, typically only 70–80%, so that between 30 and 20% of the dye is released with the spent dyebath and the washing baths that follow. Such a situation is observed particularly with reactive dyeing processes where a covalent reaction of the dye with the fiber takes place. However, some of the reactive groups may get hydrolyzed during dyeing and thus some dye remains unfixed in the dyebath. Depending on the general method of dyeing, two different qualities of coloured wastewater can be identified from exhaust and continuous dyeing processes as discussed below –

Exhaust dyeing method: For reactive dyes possessing limited degree of fixation, the unfixed reactive dye goes into the effluent liquor leading to intensively coloured wastewater. As the reactive group of the unfixed dyestuff is hydrolyzed into an inactive form, its reuse is not possible [8]. For exhaust dyeing with 5% colour depth on the substrate, using a liquor ratio of 1:10, the degree of dyestuff fixation achieved with most of the reactive dyes is in the range of 70 – 80% corresponding to 3.5 – 4 g/l of dye is fixed on the fabric and 1.5–1 g/l of hydrolyzed dye is released with the dyebath as represented in Figure 3

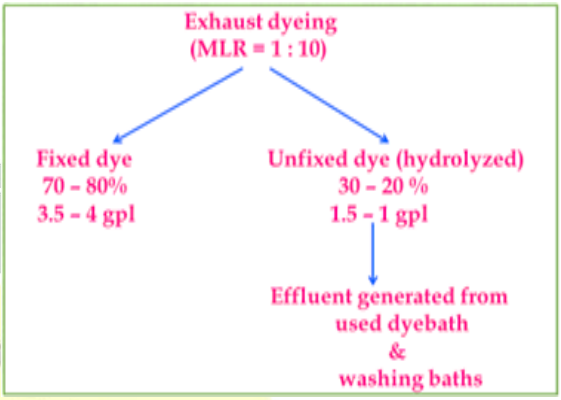


Fig. 3. Sources of coloured waste from exhaust dyeing of reactive dyes

For exhaust dyeing process, a reduction of the liquor ratio leads to significant improvements. When the dyestuff fixation is known for a certain liquor ratio, the lowering of the amount of unfixed dye released into the wasted water can be estimated as a function of the liquor ratio. When a color depth of 5% (50 g dyestuff per 1 kg of goods) is used as basis for a calculation and a dyestuff fixation of 80% is observed at a liquor ratio of 1:10 (10 litres of dyebath for 1 kg of goods), then a mass of 40 g dyestuff is fixed on the textile while 10 g remain in the dyebath as hydrolyzed dye.

On the other hand, a reduction of liquor ratio from 1:10 to 1:5 decreases the loss of dyestuff to approximately 11%, and a degree of fixation of 89% is expected. These results clearly indicate the importance of a low liquor ratio to optimize the degree of dyestuff fixation.

Continuous dyeing process: Continuous dyeing of reactive dyes by pad-dry-cure and pad-dry-steam processes is another source of creating highly coloured dyebaths, where the last filling in the trough of the padding mangle, required to complete the process at well-defined conditions, has to be withdrawn at the end of the padding process. The concentration of reactive dyes, commonly employed in such processes, is 50 g/l. For the application of a commercial reactive dye, having a degree of fixation of 70-80%, at 5% colour depth on the substrate, a concentration of 1.5-1 g/l hydrolyzed dye is expected in the wastewater, when 10 litres of washing water is utilized for washing 1 kg of fabric. The emission of coloured wastewater here can be divided into two different sources (Figure 4), namely

- the fillings of the trough of the padding mangle, which constitute high concentration of reactive dyes, up to

approximately 50 g/l, as well as high concentration of alkali;

- spent dyebaths and washing baths, which contain low concentration of dyestuff, approximately 1 g/l, as well as low concentration of alkali.

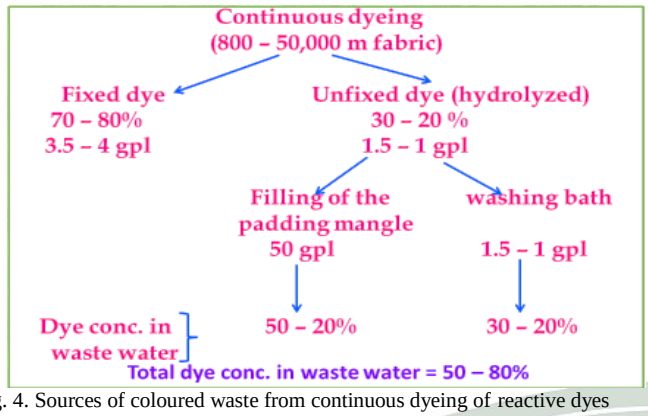


Fig. 4. Sources of coloured waste from continuous dyeing of reactive dyes

A large number of techniques have been described in the literature, for example, dyestuff adsorption, oxidative and reductive treatments, electrochemical oxidation or reduction methods, electrochemical treatment with flocculation, membrane separation processes, and biological methods [15]. Each of these techniques offers special advantages, but they can also be understood as a source of coupled problems, for example, consumption of chemicals, increased COD, AOX, increased chemical load in the wastewater, and formation of sludge that has to be disposed. The utilization of various membrane separation processes for reactive dye effluent treatment has been discussed below.

A. Different membrane filtration techniques [16]

(1) Reverse osmosis (RO): Reverse osmosis membranes have a retention rate of 90% or more for most types of ionic compounds and produce a high quality of permeate (Figure 5). Decolourization and the elimination of chemical auxiliaries in dye house wastewater can be carried out in a single step. Reverse osmosis permits the removal of all mineral salts, hydrolyzed reactive dyes and chemical auxiliaries. The problem involved is that the higher the concentration of salt, the more important the osmotic pressure becomes and therefore the greater energy is required during process.

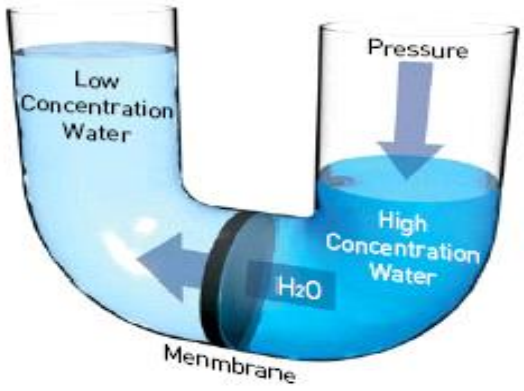


Fig. 5. Reverse osmosis membrane filter

(2) Nanofiltration (NF): Nanofiltration membranes (Figure 6) retains organic compounds of low molecular weight, divalent ions or large monovalent ions, concerning dye house effluents, the concentration of mineral salts does not exceed 20 g/l and the concentration of dyestuff 1.5 g/l. The effluents are reconstituted with generally only one dye and the volume studied is low. The treatment of dyeing wastewater by nanofiltration thus represents one of the rate applications possible for the treatment of solutions with highly concentrated and complex solutions.

Fig. 6. Nanomembrane filtration plant

(3) Ultrafiltration (UF): Ultrafiltration (Figure 7) enables the elimination of macromolecules and particles but the elimination of polluting substances, such as colour is never complete (between 31% and 76%). Even in the best of cases, the quality of the treated wastewater does not permit its reuse for feeding sensitive processes, such as the dyeing of textile. It has been emphasized that 40% of the water treated by ultrafiltration can be recycled to feed processes termed "minor" in the textile industry (rinsing, washing) in which salinity is not a problem. Ultrafiltration can only be used as a pretreatment for reverse osmosis or in combination with a biological reactor.

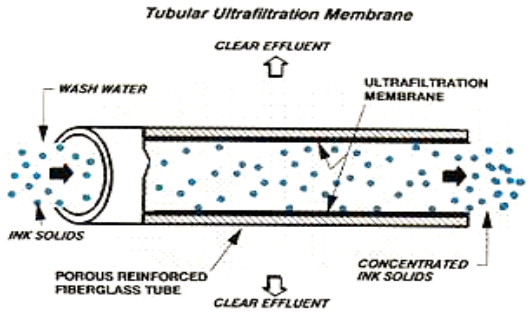


Fig. 7. Ultrafiltration membrane

(4) Microfiltration (MF): Microfiltration is a membrane technical filtration process which removes contaminants from a fluid (liquid and gas) by passage through a microporous membrane (Figure 8). A typical microfiltration membrane pore size ranges from 0.1 to 10 micrometres (µm). Microfiltration is fundamentally different from reverse osmosis and nanofiltration because those systems use pressure as a means of forcing water to go from low pressure to high pressure. Microfiltration can use a pressurized system but it does not need to include pressure.

Fig. 8. Micro-membrane filter

Microfiltration is suitable for treating dye baths containing reactive dyes as well as subsequent rinsing baths. The auxiliaries remain in the retentate. Microfiltration can also be used as a pretreatment for nanofiltration or reverse osmosis.

IV. MATERIALS & EXPERIMENTAL METHODS

A. Materials

(1) Fabric: Plain-weave jute fabric (weight: 0.277 g/in2, 10 ends/inch and 13 picks/inch) was used for the study. The fabric was scoured with 5 g/l non-ionic detergent (Lissapol N) and 2 g/l caustic soda at boil for 90 min. The scoured fabric was then bleached with 3% hydrogen peroxide (30 volumes) using sodium silicate (5 g/l) as stabilizer. The sodium silicate also helps to maintain the pH value at 9.5. The bleaching process carried out at 80o C for 1 hour, after which the fabric was subsequently washed thoroughly till it became neutral and then dried.

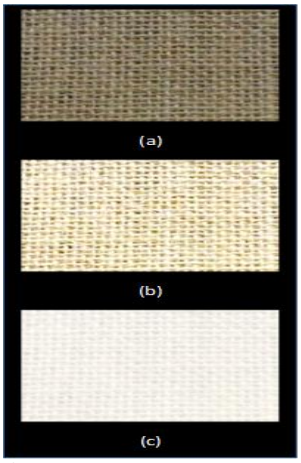
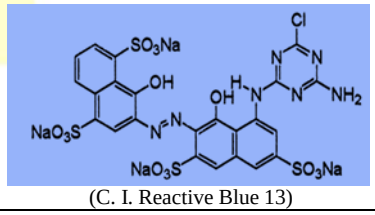
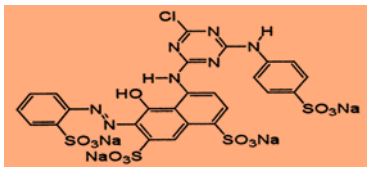
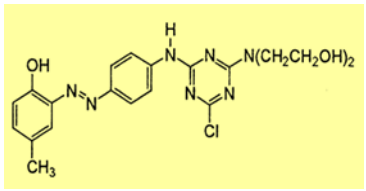


Fig. 9. Jute fabrics: (a) Grey, (b) Scoured & (c) Bleached

(2) Dyes and Chemicals: Three commercial monochlorotriazine (hot brand) reactive dyes were used for the present work without any purification (Table III). Various chemicals used were of Laboratory reagent grade.

TABLE III. VARIOUS SPECIFICATIONS OF DYES USED

Reactive dye	Chemical Structure (Colour Index Number)
D1 Reactive Brilliant Blue K3RL	 (C. I. Reactive Blue 13)
D2 Reactive Brilliant Red K2G	 (C. I. Reactive Red 15)
D3 Procion Yellow G	 (C. I. Reactive Yellow 5)

B. Experimental Procedures

(1) Dyeing procedure: Dyeing of jute substrate was performed by conventional exhaust dyeing method on Laboratory constant temperature water bath. The dyebath was prepared as follows –

Reactive dye	3% (*owf*)
Glauber's salt	20 gpl
Soda ash	5 gpl
M:L ratio	1:20
Temperature	60° C
Time	60 min

After dyeing, all samples were washed, soaped at 50o C with 2 g/l non-ionic detergent and 2 g/l soda ash, and then dried at ambient temperature.

(2) Effluent treatment: All the left over dyeing effluents were collected and were nanofiltered. The nanofiltration technique was performed at CHT laboratories, Mumbai in an arrangement of equipment as shown in the Figure 10. The permeates of each separation treatment were collected separately and were used for dyeing 2% shades by above mentioned dyeing procedure.

Fig. 10. Nanofiltration set up (ERD: Effluent after reactive colour dyeing and P4: permeate obtained after dyeing of jute was completed with 3rd permeate)

(3) Testing and Analysis

- Evaluation of Colour Strength: The dyeing performance of various dyed samples was assessed on Data Spectraflash SF 600 Spectrophotometer by measuring the relative colour strength (*K/S* value) spectrophotometrically. The colour measurement is based on the ratio between total light absorbed (K) and scattered (S) by the substrate as defined by the Kubelka-Munk equation given in below equation:

$$K/S = (1-R)^2/2R$$

where, K = absorption coefficient,
S = scattering coefficient,
R = reflectance at a given wavelength.

The *K/S* value is commonly used as a basis for evaluating dye build-up or change in colour strength. Comparison of colour strength can be made based either on the *K/S* values at maximum absorption wavelength (λ_{max}) or on the sum of *K/S* values across the visible spectrum when no specific peaks are identifiable. Any particular colour may be represented by a graph of the *K/S* or reflectance values (across the visible spectrum) plotted against corresponding wavelength [17].

- Assessment of Fastness Properties [18]**:** Wash fastness was evaluated according to ISO Standard Test No.3 on Launder-o-meter; and light fastness on fade-o-meter using carbon-arc continuous illumination (BS 1006: 1987).

By using grey scale, the change in shade was evaluated and given grades. For fastness to washing, the grades were given from 1 to 5, where 1 stands for poor and 5 for excellent. The samples for light fastness were grades from 1 to 7, where 1 indicates poor fastness and 7 represents excellent fastness towards light.

V. Results And Discussion

The permeate obtained after membrane separation treatment was dyed with individual reactive dye and the colour strength values of the dyed samples were evaluated spectrophometrically. The colour strength values were compared with conventionally dyed samples as shown in Table IV.

TABLE IV: COLOUR STRENGTH VALUES FOR MULTIPLE RECYCLING OF PERMEATE OF VARIOUS INDIVIDUAL REACTIVE DYES

Sample	Colour strength (*K/S*) values for primary shades		
	D1	***D2***	***D3***
Fresh water dyeing	18.413	39.76	18.66
After 1st permeate	16.87	38.45	18.24
After 2nd permeate	15.24	36.78	17.76
After 3rd permeate	14.11	34.56	15.23

From the table, it can be seen that the colour strength values for the samples dyed using permeate after membrane filtration are quite comparable with the standard samples dyed using fresh water. The values are only slightly lower but the shades obtained were quite uniform. Addition of very little amount of fresh dye solution will give similar shades in case of each individual reactive dye.

TABLE V: FASTNESS GRADES FOR MULTIPLE RECYCLING OF PERMEATE OF VARIOUS INDIVIDUAL REACTIVE DYES

Sample	Fastness grades for					
	D1		D2		D3	
	W	L	W	L	W	L
Fresh water dyeing	4-5	6-7	5	6-7	4-5	6
After 1st permeate	5	6-7	5	6-7	4-5	6
After 2nd permeate	4-5	6	4-5	6	4-5	5-6
After 3rd permeate	4-5	6	4-5	5	4	4-5

The wash and light fastness characteristics of the reactive dyes used for the study were quite adequate (Table V). However, slight impairment in the light fastness grades has been observed for C.I. Reactive Yellow 5 dye, predominantly after dyeing with 3rd permeate.

Conclusions

Large amount of dyestuff and chemical remain after completion of the dyeing process. These ingredients, if not reused, will be disposed off as waste materials, thus adding up to the cost of dyeing process as well as enhancement in the effluent treatment cost. Membrane filtration technology has definite utility in reducing the cost of dyeing as well as the subsequent pollution load. It is essential to select a membrane of appropriate pore size for membrane filtration for specific

purpose. The permeate obtained after membrane filtration gave uniform dyeing and the colour yield was almost similar to those of conventionally dyed samples. Fastness characteristics were also adequate. Thus, membrane filtration technique can be adopted as a suitable tool for water conservation and reduction of the dyeing process cost. However, it may be sometimes beneficial to use membrane technique in combination with various other physico-chemical effluent treatment processes to get better results.

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