SUSTAINABLE DYEING OF DENIM USING INDIGO DYE RECOVERED WITH POLYVINYLIDENE DIFLUORIDE ULTRAFILTRATION MEMBRANES

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Abstract

Indigo is one of the most consumed dyes in the textile sector, as it is widely used for the dyeing of denim clothes. About 15% of indigo used in the dyeing process is discharged to the wastewater treatment plants or sometimes into rivers, in countries where regulations are not strictly applied.

In this work, real effluents that contained indigo dye were treated by means of 4 different ultrafiltration membranes. The feasibility to recover the concentrated dye with lab and semi-industrial pilots was also investigated. The studied membranes achieved up to 99% colour removal and 80% chemical oxygen demand (COD) decrease. Finally, the concentrates containing 20 $g \cdot L^{-1}$ of indigo dye were reused in new dyeing processes. Colour differences (DE_{cmc}) and rubbing and washing fastnesses were evaluated. Fabrics dyed with the recovered indigo concentrates exhibited similar characteristics than the ones obtained with the commercial dye.

Keywords: dye recovery; indigo; reuse; membrane technology; ultrafiltration.

1. Introduction

The textile industry consumes a large amount of water in their processes, especially in the dyeing and subsequent washing steps (Fersi et al., 2005). The wastewater generated is reported to be in the range of 21–377 m^3 per ton of textile products (Asghar et al., 2014). In addition, the wastewater generated from the textile industry is characterized by high colouration, biological oxygen demand (BOD), chemical oxygen demand (COD) and salinity (Riera-Torres et al., 2010).

Physico-chemical and biological processes are currently applied to treat textile wastewater (Blanco et al., 2013). In general, conventional biological treatment provides good COD removal, but low efficiencies in discoloration due to the chemical stability and resistance to microbiological attack of the dyes (Bes-Piá et al., 2005; Robinson et al., 2001). Chemical coagulation is a very common treatment but this method generates a sludge which requires an additional treatment to be destroyed (Sala and Gutiérrez-Bouzán, 2012). These methods are able to meet legislative requirements but they do not enable water reuse in textile processes (Barredo-Damas et al., 2010).Recently, there are an increasing number of studies based on industrial waste reuse (Huber et al., 2014; Sathishkumar et al., 2012) and on water reuse (Othman et al., 2014). In particular, advanced oxidation treatments have been applied to achieve the reuse textile wastewater (Li and He, 2013; Sala and Gutiérrez-Bouzán, 2014). Membrane technology is also an attractive alternative to treat and reuse textile wastewater (Tahri et al., 2012; Zheng et al., 2013), because it is able to remove many kind of dyes.

In general, studies published on membrane technology applied to textile effluents are focused on the reuse of permeate (Arnal et al., 2008; He et al., 2013; Sahinkaya et al., 2008). Studies on concentrated dyes reuse are very scarce. Although some types of dyes cannot be reused, the sulphur dyes and vat dyes, especially indigo, are suitable to be recovered with membrane filtration because they are insoluble in water and can be easily separated from the effluent. This is especially interesting as the annual consumption of sulphur and vat dyes is about 120,000 t (Roessler, 2004).

Indigo ($C_{16}H_{10}N_2O_2$) is one of the oldest known dyes and currently it still occupies an important place in textile dyeing. Its importance is especially due to the popularity of blue jeans, which are dyed with indigo (Meksi et al., 2012; Vuorema et al., 2008). About 15% of the indigo used is lost during the dyeing process (Vedrenne et al., 2012), but to our knowledge, only two studies have been published on indigo dye reuse after membrane technology treatment. Crespi (1989) reported the recovery of the residual indigo from washing wastewater by ultrafiltration membranes in the Belgian company UCO. Porter et al. (1990) studied the recovery of indigo dye with a vinyl-sulfone membrane using a multistage system, where the indigo concentration became progressively higher.

The aim of this work is to apply ultrafiltration membranes to effluents containing indigo in order to achieve the dye reuse. Nowadays, polyvinylidene difluoride (PVDF) is the most used material due to its thermal stability, high hydrophobicity, and resistance to corrosion from many chemicals (Zhang et al., 2014). As far as we know, this material has not still been applied to indigo wastewater treatment. Consequently, in this work different PVDF hollow fibre ultrafiltration membranes were tested to treat effluents containing indigo dye. The dye was firstly concentrated up to 20 $g \cdot L^{-1}$ and subsequently the concentrate was reused in a new dyeing process. Fabrics dyed with the recovered indigo were evaluated with respect to the reference ones (dyed with commercial indigo) by means of spectrophotometric colour difference, dry rubbing and washing fastness tests.

2. Experimental

2.1 Reagents

Indigo dye (95%) and sodium dithionite (85%) were supplied by ACROS. Sodium hydroxide (98.5%) was obtained from Panreac. Sodium hypochlorite solution (6-14%

chlorine active) was acquired from Sigma-Aldrich and 1-methyl-2-pyrrolidine (99.5%) from Scharlau.

2.2 Wastewater

Three industrial effluents supplied by the denim yarn factory “Tejidos Royo” (Alcudia de Crespins, Valencia, Spain) were selected to be treated. They were collected from the first washing tank, after the dyeing process, and correspond to different type of fibres and production periods.

The effluents used for the lab tests were preserved in a thermostated room at 20ºC. Before the membrane treatment, samples were pre-filtered (pore diameter 500 µm) in order to remove the higher size particles and fibres. The concentration of indigo was immediately determined before and after the ultrafiltration.

2.3 Ultrafiltration module

In this work, four hollow fiber membranes were selected to carry out the indigo dye recovery tests: ZeeWeed-1 (GE Power & Water, Canada), UOF-1b (Motimo Membrane Technology, China), UOF-4 (Motimo Membrane Technology, China) and FP-T0008 (Motimo Membrane Technology, China), referred herein after as ZW-1, U-1b, U-4 and FP-T, respectively. In Table 1 are described the main characteristics of these membranes.

Table 1- Membranes characteristics

Membrane	Configuration	Pore size (µm)	Membrane Area (m^2)
U-1b	External	0.04	0.5
ZW-1	Submerged	0.04	0.05
U-4	External	0.03	40
FP-T	Submerged	0.1	1

Three pilot plants were built to position the different membrane modules, according to their geometry and specific requirements.

Pilot 1 (Figure 1) was equipped with U-1b membrane. It was fed by a 100 L tank. Peristaltic pumps were used for feed, permeate, and concentrate effluents. Pilot 2 operated in cycles of 15 minutes of filtration and 30 seconds of backwashing with permeate.

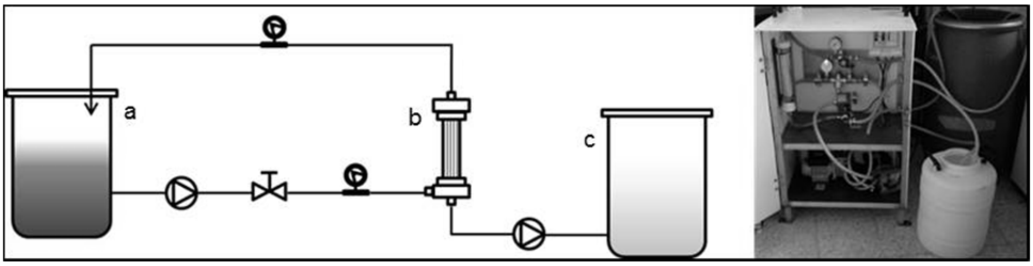

Figure 1- Pilot 1 with U-1b membrane. a) Feed tank, b) U-1b Membrane, c) Permeate tank

Pilot 2 (Figure 2) was designed to be equipped with ZW-1 module. The membrane reactor was a 20 L cylindrical vessel. It was fed from a 20 L tank by a centrifugal pump. A peristaltic pump was used for the permeate effluent. The membrane module had an air inlet with the purpose to decrease the fouling. This pilot also operated in cycles of 15 minutes of filtration and 30 seconds of backwashing with permeate.

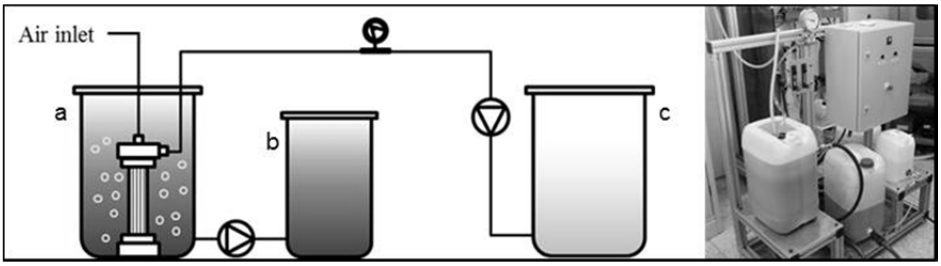

Figure 2- Pilot 2 with ZW-1 membrane. a) ZW-1 Membrane, b) Feed tank, c) Permeate tank

Pilot 3 (Figure 3) was a semi-industrial system designed to place two membrane modules. The first one was the U-4 membrane able to concentrating up to 3 g·L^{-1}. The volume of feeding tank was 1000L. The concentrate obtained was then applied as a feed to FP-T module which volume was 100 L. In this way, the indigo was concentrated until 20 g·L^{-1}. U-4 membrane operated in cycles of 30 minutes of filtration and 30 seconds of backwashing with permeate and FP-T membrane worked in cycles of 15 minutes of filtration and 30 second of backwashing.

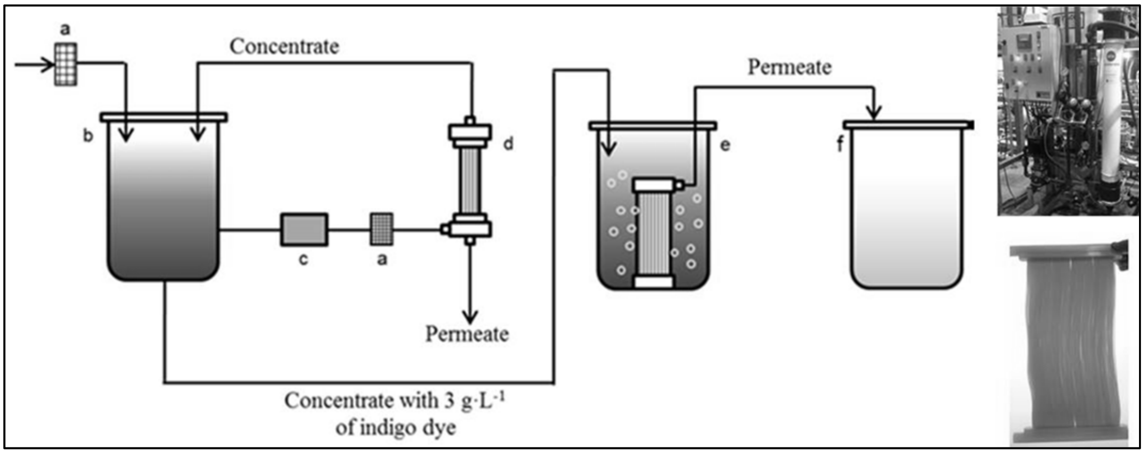

Figure 3- Semi-industrial system. a) Meshes, b) Feed tank, c) Control system, d) U-4 membrane, e) FP-T membrane, f) Permeate tank.

Finally, after each filtration process, membranes were cleaned with a sodium dithionite alkaline solution (pH 11), followed by rinsing with a sodium hypochlorite solution (5 $mg\cdot L^{-1}$).

2.4 Analytical methods and measurements

Permeate flux was determined by measuring the permeate volume collected in a certain period of time and using the following equation:

$J\ (L/m^2\cdot h) = V/A\cdot\Delta t$

Where J is the volumetric flux, A is the effective area of the membrane and V is the collected volume in a time interval Δt.

Indigo removal was calculated from concentrations of feed and permeates using the following equation:

$R_{indigo} = ((C_f\text{-}C_p)/C_f)\cdot 100$

where C_f and C_p are the concentrations of indigo in feed and permeate respectively.

Indigo was reduced with a solution which contains sodium dithionite, sodium hydroxide and 1-methyl-2-pyrrolidine and determined by UV-Vis spectrophotometry (Shimadzu

UV-2401) in the maximum of the visible spectrum (407 nm). The absorbance and dye concentration were linear in the range between 0.10 and 10 $mg \cdot L^{-1}$.

COD was determined according to the methods recommended by American Public Health Association (Standard Methods for the Examination of Water and Wastewater, 2012). The COD reduction was calculated using the following equation:

$$R_{COD} = ((COD_f - COD_p)/COD_f) \cdot 100$$

where COD_f and COD_p are the COD values in feed and permeate respectively.

Finally, the conductivity and pH were determined using a Conductimeter GLP 31 (CRISON) and a pHmeter GLP 21 (CRISON) respectively (Standard Methods for the Examination of Water and Wastewater, 2012).

2.5 Dyeing tests and dyed fabric evaluation

Dyeing experiments were carried out, with synthetic indigo dye and 100% recovered dye, in a foulard designed especially for laboratory tests by Tejidos Royo. Cotton fabrics were passed through a bath which contained the dyeing bath with 3 $g \cdot L^{-1}$ of reduced indigo and then exposed to the air.

The quality of fabrics dyed with indigo recovered was studied from colour differences (DE_{cmc}(l:c)) and fastness to rubbing and washing respect to fabrics dyed with commercial pre-reduced indigo dye.

Colour differences were measured using a Macbeth Colour Eye 7000A spectrophotometer according to the Standard UNE-EN ISO 105-J03 (AENOR, 2009). The equation for DE_{cmc}(l:c) describes an ellipsoidal volume with axes in the direction of lightness (L), chroma (C), and hue (H) centered about a standard. Colour difference is composed of three components:

- Lightness component (DL_{cmc}) that is weighted by the lightness tolerance (DL^*/lS_L). If DL_{cmc} is positive, the reused dyeing is lighter than the standard. If DL_{cmc} is negative, the reused dyeing is darker than the standard.

- Chroma component (DC_{cmc}) that is weighted by the chroma tolerance (DC^*_{ab}/cS_c). If DC_{cmc} is positive, the reused dyeing is more chromatic than the standard. If DL_{cmc} is negative the reused dyeing is less chromatic than the standard.
- Hue component (DH_{cmc}) that is weighted by the hue tolerance (DH^*_{ab}/S_H). It describes the difference between the hue angle of the standard and the hue angle of the reused dyeing in a polar coordinate.

Colour difference is calculated from the following equation:

$$DE_{cmc}(l:c)= [(DL^*/lS_L)^2+(DC^*_{ab}/cS_c)^2+(DH^*_{ab}/S_H)^2]^{1/2}$$

The lengths of the semi axes of the ellipsoid are calculated from the values L^*_r, $C^*_{ab,R}$ and $h_{ab,R,}$ that correspond to the reference as follows:

$$S_L=0.040975\ L^*_R/(1+0.01765\ L^*_R) \text{ if } L^*_R \geq 16 \text{ or } S_L= 0.511 \text{ if } L^*_R <16$$

$$S_C=[0.0638\ C^*_{ab,R}/(1+0.0131\ C^*_{ab,R})] + 0.638$$

$$S_H=(FT+1-F)S_c$$

where

$$F=((C^*_{ab,R})^4/((C^*_{ab,R})^4+1900))^{1/2}$$

$$T=0.36+|0.4\cos(35+h_{ab,R})| \text{ if } h_{ab,R} \geq 345^o \text{ or } h_{ab,R} \leq 164^o$$

$$\text{or } T=0.56+ |0.2\cos(168+ h_{ab,R})| \text{ if } 164^o < h_{ab,R}< 345^o$$

Colour fastnesstorubbing was determined according to the Standard UNE-EN ISO 105-X12 (AENOR, 2003). This test determines the amount of colour transferred from the surface of dyed fabric to other surface by rubbing. Colour fastness to washing was determined following the Standard UNE-EN ISO 105-C10 (AENOR, 2008). Rubbing and washing fastnesses are evaluated on a scale of 1-5, where value 1 indicates very low fastness and 5 corresponds to high fastness.

3. Results and discussion

3.1 Wastewater characterization

Before the membrane filtration study, the effluents were characterized. The characteristics of each effluent (Table 2) depend mainly on the type of fibre and on dyeing conditions.

Table 2- Effluents characterization

Parameter	Effluent 1	Effluent 2	Effluent 3
Dyed Fibre	Cotton	Lyocell	Cotton
pH	8.6	9.5	10.4
COD ($mg\cdot L^{-1}$)	320	800	572
Conductivity ($\mu S\cdot cm^{-1}$)	6751	7266	4230
Indigo ($mg\cdot L^{-1}$)	58	118	82

As can be observed, the effluents generated by denim industry are mainly characterized by its high pH and conductivity. The alkaline pH is attributed to the dyeing process, as it requires pH between 11.5 and 12. The high conductivity is due to the presence of sulphates, which are generated from the oxidation of sodium dithionite. This reagent is used for the indigo reduction during the dyeing process.

The two lab pilots cannot run simultaneously as they share the same air inlet. For this reason, a different effluent was treated in each pilot. Effluent 1 was treated in U-1b membrane (pilot 1) and effluent 2 in ZW-1 membrane (pilot 2).

The effluent 3 was treated in situ in Tejidos Royo. The semi-industrial system, equipped with two ultrafiltration membranes, operated in the mill during 2 weeks.

3.2 Ultrafiltration experiments

3.2.1 Pilot 1 with U-1b membrane

The efficiency of the membrane process in the recovery of indigo dye from textile effluents was determined by means of permeate characterization. Permeate samples were taken and analysed daily.

It can be seen in Figure 4 that 96% dye removal was achieved. This result confirmed the almost full retention of the dye in the concentrate, which indicated the high efficiency of the ultrafiltration membranes in the recovery of indigo dye. In addition, the COD removal was about 40%, increasing at the end of the experiment. A layer of dye was formed on the membrane surface, which acted as a barrier and increased the process efficiency. The increase of colour and COD removal along the treatment has also been reported by different authors (Alventosa-deLara et al., 2014; Aouni et al., 2011; He et al., 2008; Kim et al., 2005) .

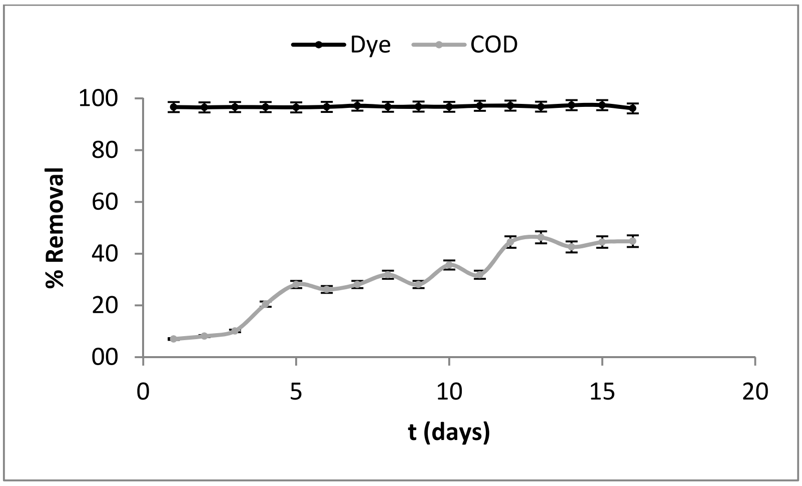

Figure 4- Dye and COD removal with U-1b membrane

The pH and conductivity values were 8.4 and 6720 $\mu S \cdot cm^{-1}$ respectively, which evidenced the low efficiency of the ultrafiltration membrane in ions removal. Ions such as sodium or sulphate pass through ultrafiltration membranes, therefore when the permeate has to be reused, nanofiltration or reverse osmosis membranes must be selected to remove inorganic ions.

After 5 days of operation, this pilot exhibited a remarkable membrane flux decrease up to 57% (Figure 5), which was mainly attributed to the indigo layer formed on the membrane surface. The classic washing was not able to remove this layer from the membrane surface and to restore the initial membrane flux.

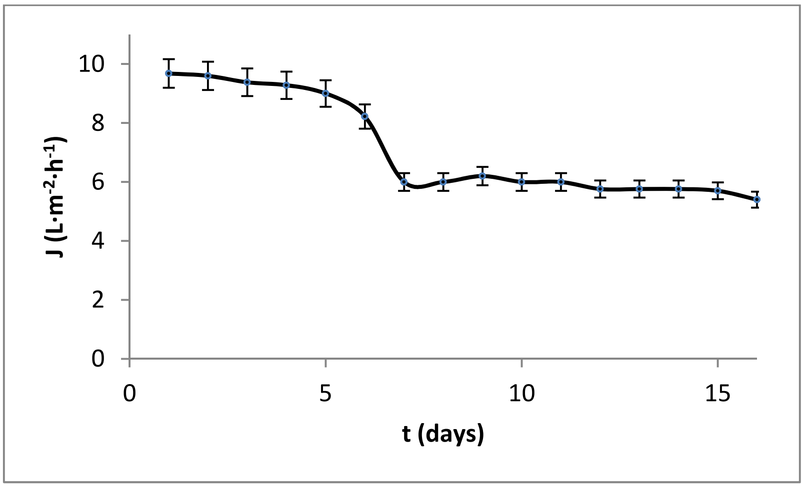

Figure 5- Flux values of U-1b membrane

Taking into account that higher size particles of effluent 1 were separated before ultrafiltration treatment, it was concluded that the fouling observed in U-1b membrane was due to its physical configuration as it is constituted by a packed column. This structure complicates the washing process and results in the production of a cake of indigo particles (Figure 6).

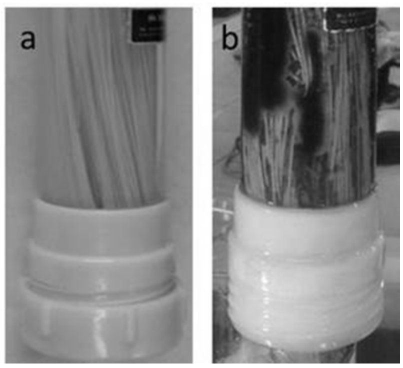

Figure 6- U-1b membrane: a) Before the study, b) After the study

Thus, this type of membrane was only considered able to obtain concentrates up to 3 $g{\cdot}L^{-1}$ indigo dye. This concentration is high enough to be directly reused in new dyeing processes. However, the automated dyeing process used in Tejidos Royo requires the dosage of an initial dye bath of 20 $g{\cdot}L^{-1}$. For this reason, the dye reuse study was not carried out with the effluent treated with the U-1b membrane.

3.2.2 Pilot 2 with ZW-1 membrane

As in the previous section, permeate samples were taken and analysed daily. Along the filtration process, 99% dye removal was obtained and no coloration was observed in the permeate (Figure 7). In addition, at the end of the study, 80% COD removal was also achieved. This phenomenon was also observed with U-1b membrane and it was discussed in section 3.2.1.

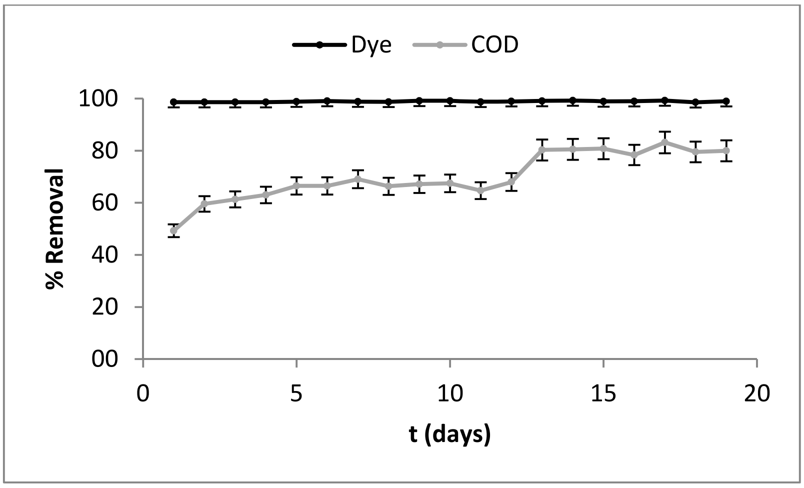

Figure 7- Dye and COD removal with ZW-1 membrane

The pH and conductivity values were 8.7 and 5027 $\mu S \cdot cm^{-1}$ respectively.

Regarding the membrane behaviour, the fouling was controlled by measuring the permeate flux during the treatment. The permeate flux was stable throughout the study, that indicated low fouling on the membrane. The importance of the fouling in a membrane process should be highlighted. In fact, membrane fouling has become in a major obstacle to the further application of UF technology in water treatment (Qu et al., 2014), because it causes a reduction in the permeability and therefore the maintenance cost of the membrane is increased and its life time is reduced (Ellouze et al., 2012). The washing step was able to clean and recover the initial membrane flux value, which indicated that the cake formed on the membrane surface was fully removed, although at the end of the study, the membrane surface was just dyed with a blue coloration.

The low fouling observed in this pilot shows the feasibility of applying this type of membranes to recover indigo dye.

The ZW-1 membrane enabled to obtain a concentrate containing 20 $g \cdot L^{-1}$ indigo dye. This concentration is adequate to be used in an automated dyeing process. Thus, the indigo dye reuse study was carried out with this concentrated effluent (section 3.3).

3.2.3 Semi-industrial system

In the lab tests, both membranes provided almost full retention of indigo. For this reason, in the semi-industrial study both membrane configurations were combined to evaluate their behaviour at semi-industrial scale. The design of the semi-industrial pilot was mainly based on practical considerations such as operational factors, maintenance, cleaning and membrane cost. The first membrane (U-4) was able to concentrate up to 3 $g \cdot L^{-1}$ and it was combined with a second membrane module (FP-T) in order to achieve the 20 $g \cdot L^{-1}$ as final concentration. The submerged configuration of the second membrane enabled to reach the required dye concentration for the automated dyeing process employed in Tejidos Royo.

Due to the high volume of effluent required, experiments of membrane characterization were not carried out, to avoid interference in the production process of the company. Permeate was characterized and at the end of the process. It was obtained 98% of dye removal and 67% of COD decrease. As in the case of the first lab pilot; the layer of dye formed on the membranes surface increased the indigo dye rejection. The concentrated was kept for the reuse tests (section 3.3).

With the semi-industrial pilot, dye removal results (98%) were into the range of lab pilots (96-99%). COD removal (67%) was also found to be into the range of the lab pilots (40-80%).

3.3 Indigo dye reuse

Indigo dye reuse tests were performed from solutions containing 20 $g \cdot L^{-1}$ of indigo dye, which correspond to concentrates of pilots 2 (lab scale) and 3 (semi-industrial). The concentrate of pilot 1 was not included in this study as it did not meet the requirements for the automatic dosage of the mill.

Dyeings obtained from the two concentrates were evaluated with respect to a reference dyeing performed with commercial indigo. Their chromatic coordinates and colour differences ($DE_{CMC\ (2:1)}$) are shown in Table 3.

Table 3- Colour differences values

Pilot plant	$DE_{CMC\ (2:1)}$	DL	DC	DH
ZW-1 membrane	0.84	-0.76	-0.26	0.22
Semi-industrial	1.19	-1.19	-0.31	-0.35

From these values it can be concluded that the main differences between colour of the reference fabric and the ones dyed with reused indigo ($DE_{CMC\ (2:1)}$) were mainly due to the influence of DL. Thus reused dyeings had similar chroma (C) and hue (H), but they exhibited more differences on the intensity values (L). In addition, the negative value of DL means that dyeings with indigo recovered were darker than the reference. This fact was attributed to dispersant that can be retained with the indigo dye. It is necessary either determine the amount of dispersant in the concentrate and adjust it or decrease the dye concentration of the bath to obtain the same intensity. Both cases provide an important advantage from the economical point of view. This study is been currently carried out.

The indigo recovered with pilot ZW-1 membrane provided $DE_{CMC\ (2:1)}$ lower than 1, which is considered the acceptance limit of most industrial dyeing processes. However, in the denim industry the acceptance limit can be higher than 20% (1.2), as generally fabrics are submitted to subsequent bleaching or sandwashing processes. For this reason, $DE_{CMC\ (2:1)}$ corresponding to the semi-industrial test, is also considered acceptable.

Fastness properties are also an important factor to be considered in textiles. In this case, colour fastnesses to washing and rubbing were evaluated (Table 4).

Table 4- Colour fastnesses to washing and rubbing values

Pilot plant	Washing	Rubbing
Reference	4	2
ZW-1 membrane	5	2
Semi-industrial	5	2

Both, dyeings with reused and dyeings with commercial dye exhibited the same rubbing fastness. Fabrics dyed with recovered indigo showed higher values of washing fastness than the fabric dyed with commercial indigo. This difference could probably be attributed to the higher amount of dispersing agent in the recovered dyeing bath (part of the dispersant may probably be retained with the dye). This point is under research and should be confirmed by further investigations.

3.4 Permeate reuse

Although the aim of this work was the indigo dye reuse, it is important to highlight the quality of the permeate obtained after the membrane treatment (Table 5).

Table 5- Permeate characterization

Pilot Plant	Dye concentration ($mg \cdot L^{-1}$)	pH	Conductivity ($\mu S \cdot cm^{-1}$)	COD (mg $O_2 \cdot L^{-1}$)
U-1b membrane	1.8	8.6	6750	230
ZW-1 membrane	1.3	8.7	5028	239

Both membranes provided permeates with similar characteristics: very low dye concentration (<2 mg·L^{-1}) and low organic matter content (<250 mg·L^{-1}). According to the mill experience (Tejidos Royo), these values enable to reuse the effluent in a new indigo dyeing process directly or partially diluted.

Permeate reuse is an important challenge. It is estimated that the annual consumption of fresh water in the textile industry at the European level is 600 million m^3 (Vajnhandl and Valh, 2014). Regarding the denim industry, Chico et al. (2013) reported that about 3000 m^3 of water is needed per trouser from fibre production stage to fabric production. Despite the clear benefits of water reuse, its implementation is still not a common practice in the textile sector.

4. Conclusions

Indigo dye from wastewater can be successfully removed by means of PVDF ultrafiltration membranes.

Among the studied membranes, the external hollow fibre module (U-1b membrane) was able to treat wastewater containing indigo dye and it enabled to concentrate the dye up to 3 g·L^{-1}. Higher concentration was discarded due to the fouling on the membrane surface. This concentration is acceptable when the effluent is directly reused. The U-1b membrane allowed obtain a permeate free of dye (96% dye removal) and a 40% COD reduction.

According to the results, the submersible hollow fibre module (ZW-1 membrane) enabled to obtain a concentrate with 20 g·L^{-1} indigo dye, which is the required concentration for automated dyeing processes. In addition, the permeate characterization showed 99% dye retention and 80% COD reduction.

The study in semi-industrial system showed that the combination of external and submerged PVDF membranes was able to treat wastewater that contained indigo dye. The COD and dye removal obtained was 67 and 98% respectively.

Finally, the study of indigo dye reuse carried out with the concentrate obtained in both submersible hollow fibre module and semi-industrial system, showed the feasibility of the membrane technology to recover indigo dye. The dyeing made with recovered dye exhibited similar characteristics than dyeing with commercial dye.

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