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Cotton and polyester dyeing using nanofiltered wastewater

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Abstract

Cotton and polyester dyeing using effluents treated by nanofiltration was evaluated on laboratory and pilot scale. Wastewaters were collected separately from reactive and direct dyeing (for cotton) and from disperse dyeing (for polyester). For reactive and direct dyeing, experiments were carried out with both NaCl and Na_2SO_4 .

Flux decline due to fouling was observed during nanofiltration. Additionally, retention of ions, SO_4^{2-} in particular, led to lower fluxes or a higher pressure to be applied. After nanofiltration, not all permeates were colourless and in most cases the COD was above the limit for good quality process water. Considerable differences in pH and conductivity were observed between the different fractions.

For reactive dyeing, reuse tests were carried out with fresh water for wetting and dyeing, and 50% permeate for rinsing. Large differences in colour and deviations in fastness to laundering and rubbing were observed when NaCl was used. However, when Na₂SO₄ was used, colour and fastness figures were similar to the reference, in spite of the presence of organic matter.

For direct dyeing, a 75/25 mixture of permeate and process water was used for dyeing and rinsing. In case of NaCl, fastness to laundering and wet rubbing was not satisfactory; the colour difference was, however, limited. In case of Na₂SO₄, all results were satisfactory. Since NaCl is hardly rejected by nanofiltration membranes, while Na₂SO₄ is well rejected, it was concluded that the conductivity of the permeate determines the possibility of reusing the water for direct and reactive cotton dyeing, while the remaining colour and COD have a minor influence.

For disperse dyeing, 100% permeate was used for wetting, dyeing and rinsing. This corresponds to a 'worst case scenario'. However, no differences in colour and fastness to laundering and rubbing were observed. The components causing colour in the permeate apparently do not disturb the dyeing process.

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1. Introduction

Since several years, the textile sector in Flanders, Belgium is facing a restriction on the use of groundwater, and more in particular of water from the Palaeozoic layer, which is an important source of water for many textile companies. There are various strategies to tackle the water issue in a sustainable way. Textile companies have firstly focused on mapping the

specific water use (expressed in 1 per kg textiles). On the basis of this information, several water-saving measures have been introduced, whenever it was possible from a qualitative and economic point of view. This has resulted in a decreased average water use of 58 l/kg in Flanders, whereas the European average amounts to 102 l/kg [1]. The most obvious measures leading to the best saving results have been applied in a vast majority of companies. Further savings are hard to introduce because of technical/qualitative or economic reasons.

It becomes increasingly obvious that for a number of applications, one will have to look for alternative water sources, such as purified wastewater. This strategy to 'purify and reuse

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wastewater' is also dictated by the fact that many companies are faced with the so-called disconnection policy (making individual companies responsible for their own wastewater treatment, and thus disconnecting these companies from the public sewage system) and the expected adaptation of sector-related standards for the discharge of textile wastewater.

A technology that may offer a solution to both issues (process water supply and wastewater discharge) is membrane filtration. Initially, tertiary treatment of wastewaters was emphasized, in view of discharge [2,3]. Many research groups also studied the suitability of membrane filtration, mostly nanofiltration, for producing process water of sufficient quality from wastewater [4-14]. In most cases it was concluded that the quality of the produced water was high enough for reuse in dyeing processes. This conclusion is usually based on the absence of visible colour. However, it is obvious that other parameters, such as the ionic composition, the presence of organic matter and the hardness of the water also influence the dyeing process. Thus, the quality of textile dyed with reused water is possibly different; this aspect was not investigated before. Colour and fastness to laundering were evaluated in one case [15] and were considered satisfactory. In this framework, the possibilities of nanofiltration for the treatment and reuse of coloured wastewater from direct, reactive and disperse dyeing processes were examined from the point of view of the dyeing process itself.

2. Materials and methods

2.1. Nanofiltration: type of membrane and conditions

The filtration tests were carried out at 50 $^{\circ}$ C, since this is the average temperature of dyeing wastewater. On the one hand, this is a worst case scenario, because rejections are generally lower at high temperature [16]; on the other hand, the higher temperature possibly allows to recycle energy as well [17]. However, it was impossible to dye and filter in one single day. Therefore, the wastewater had to be heated to 50 $^{\circ}$ C before the filtration could start.

The nanofiltration experiments were performed on a pilot scale installation using the DESAL Duratherm Pro NF membrane (Osmonics, Vista, California, USA) (Fig. 1). This membrane element can be used at elevated temperatures [18].

The membrane's specifications are mentioned in Table 1.

The pilot scale filtration (Fig. 2) was performed in cross flow mode with a feed flow of $1000 \, l/h$. The wastewater was pre-filtered over a candle filter of 5 μm . The applied pressure over the nanofiltration membrane varied between 10 and 15 bar, depending on the composition of the wastewater to be treated.

The filtration performance is expressed in the recovery, i.e. the ratio of the permeate flow to the sum of permeate and concentrate flows. The flow $(l/h \, m^2)$ as well as the retention of both salt and colour have been monitored.



Fig. 1. DESAL Duratherm Pro NF membrane (Osmonics, Vista, California, USA) used in experiments.

2.2. Cotton dyeing

For the dyeing of cotton fabric direct and reactive dyestuffs were used. For both dyestuffs, a dyeing recipe has been selected to dye a medium shade. These dyeing recipes have been performed with both NaCl and Na₂SO₄. The dosage of dyestuff and electrolyte is summarised in Table 2.

The dyeing was done on a pilot scale by using a discontinuous dyeing apparatus (a jet) in a liquid ratio of 1/10: 10 kg of fabric in 100 l water per bath. All baths for each type of dyeing have been collected in a mono-container.

2.3. Polyester dyeing

Spools with polyester yarns have been dyed in a light and dark shade. Apart from the difference in dosed dyestuff (0.1-1%) there is also a difference in post-treatment. A reductive cleaning has been carried out to remove non-fixated dyestuff only after the dark dyeing.

The dyeing was performed on a pilot scale by using a spool dyeing apparatus with a liquid ratio of 1/22: 2 kg yarn in 45 l water per bath. All baths per type of dyeing were collected in a mono-container.

2.4. Evaluation of reuse

During the reuse tests, the permeate was reused purely or mixed with fresh process water for the same kind of dyeing.

Table 1
Specifications: DESAL Duratherm Pro NF (2540)

Specifications. DESAL Durauterin 110 W (2.	D + 0)
Surface	4.3 m ²
pH range	2-10
Max. temperature	70 °C
NaCl removal efficiency (50 °C)	10-25%
MgSO ₄ removal efficiency (50 °C)	85-95%
Clear water	$14.5 \text{ l/h m}^2 (10 \text{ bar})$
flow (determined at 20 °C)	



Fig. 2. Membrane filtration equipment (frontal and lateral views).

To assess the reuse tests, a reference dyeing per type of dyeing was carried out to determine the difference in colour on the one hand, and to assess the difference in fastness figures on the other, i.e. fastness to laundering and rubbing.

The difference in colour is determined in conformity with the ISO 105-J03 standard, by which the reflection of the dyed textile is measured by means of a specified light source. The measuring results are converted into 3 colour co-ordinates (L,a,b) and the difference in colour (ΔE) is expressed by means of the CMS system. There is no generally set maximum, but in practice 0.8-1 is accepted as the maximum difference.

The fastness to laundering is determined in conformity with the ISO 105-C06 standard. This is a method to determine the colour fastness of textiles to domestic and commercial laundering. Depending on the application and composition of the textiles, various conditions may be chosen in respect of temperature, alkalinity, bleaching activity, etc. The change in colour of both the textile to be tested and the standard test fabric is compared to grey fastness scales and assessed on a scale from 1 to 5; 1 being very poor (important difference in colour) and 5 being very good (no difference in colour). Differences of up to 0.5 are considered as irrelevant.

The fastness to rubbing is determined in conformity with the ISO 105-X12 standard. This is a method to verify the degree of bleeding of a standard test fabric and to assess the degree of discoloration of a textile when being rubbed under preset conditions (wet, dry, warp or weft direction of fabrics). The assessment is done in the same way as for fastness to laundering, i.e. by means of grey fastness scales.

3. Results and discussion

3.1. Nanofiltration of coloured wastewater from dyeing plant

For all selected dyeing cycles, the wastewater, i.e. exhausted dye bath and all subsequent rinsing baths, was collected separately per type of dyeing. For cotton dyeing, depending on the application, one frequently uses reactive and direct dyestuffs. Polyester, on the other hand, is always dyed by using disperse dyestuff. Because these are two important raw materials for the finished products marketed by Flemish textile companies, these three kinds of dyeing processes (two for cotton and one for polyester) were examined extensively in this study.

Nanofiltration is a membrane separation process suitable to retain dissolved molecules with a size above ca. 1 nm, corresponding to a molecular weight of ca. 200. In addition, charge interactions result in rejection of ions, even those smaller than the membrane pores. These interactions are more distinct for bivalent ions than for monovalent ions. Since the selected nanofiltration membrane is negatively charged at neutral to high pH, it is to be expected that sulphate (SO_4^{2-}) is better retained than chloride (CI^{-}) . This is also shown by the characteristics of the membrane (see Table 1). Because the type of salt has a clear influence on the composition of the permeate, both reactive and direct dyeing were performed by using NaCl and Na₂SO₄. Table 2 shows that in order to arrive at a medium shade in the case of direct dyeing, the salt and dyestuff dosages are smaller than in the case of reactive dyeing.

For the dyeing of the polyester spools, two formulae were selected with disperse dyestuff. The light shade does not need reductive cleaning, in contrast to the darker shade.

Table 2
Dosage of dyestuff and electrolyte for cotton dyeing

Type of dyeing	Dyestuff (%)	NaCl (g/l)	Na ₂ SO ₄ (mg/l)
Reactive - NaCl	2.6	60	_
Reactive - Na ₂ SO ₄	2.6	_	72.9
Direct - NaCl	0.8	10	_
Direct - Na ₂ SO ₄	0.8	_	12.5

All subsequent baths per type of dyeing have been collected in a mono-container and the quality of the wastewater was determined (Table 3).

The COD (chemical oxygen demand) measurements reveal a moderate organic charge of wastewater from reactive and direct dyeing processes. Wastewater from disperse dyeing processes has a much higher COD value. This is explained by the smaller number of rinsing baths and the higher COD charge of dyestuff and dosed chemicals. The conductivity, on the other hand, is highest in the case of reactive dyeing, followed by direct dyeing. The concentration of suspended solids is low in all water samples. This is a prerequisite to carry out nanofiltration with a limited pre-filtration, i.e. a candle filter of 5 μm .

At the start of the filtration of wastewater from *reactive dyeing with NaCl*, the pressure was set at 10 bar. This resulted in a recovery of 74%, which dropped to 55% during the experiment (300 min). Therefore, the pressure was raised to 13 bar after 75 min. The flow was relatively high at the beginning, i.e. 13.3 l/h m² (cfr. fresh water flow at 20 °C: 14.5 l/h m²), but decreased to 8.1 l/h m². As indicated by the specifications of the membrane (see Table 1) the retention for NaCl was poor, and the decrease of conductivity amounted to 9%. Since conductivity is mainly caused by the dosed NaCl, it can be concluded that the retention of NaCl corresponds to the specifications as indicated by the supplier. The colour removal was 99.5%. The mixed wastewater had a deep red colour, whereas the residue colour of the permeate is pink.

The wastewater from reactive dyeing with Na_2SO_4 was filtered at a pressure of 15 bar instead of 10 bar, because it was expected that because of the better retention of SO_4^{2-} the developed osmotic pressure (counteracting the applied pressure) would be higher.

At the start, the recovery amounted to 75% but after 30 min it had already dropped to 63%. This corresponds to a decrease of the flow from 14 l/h m² to 9.3 l/h m². In the next 225 min the flow and recovery remained relatively stable, but another slight decrease to 58% recovery and a flow of 6.5 l/h m²

Table 3 Quality of the wastewater to be treated

Type of dyeing	COD (mg/l)	Suspended solids (mg/l)	pН	Conductivity (mS/cm)
Reactive - NaCl	535	0	9.86	26.2
Reactive - Na ₂ SO ₄	539	7	9.9	21.7
Direct - NaCl	345	11	9.45	6.43
Direct - Na ₂ SO ₄	403	13	8.63	5.88
Disperse - light	2128	8	4.74	0.91
Disperse – dark	1893	6	8.81	2.62

occurred. The salt retention is 56%, which is clearly less than the specifications for sulphate retention indicated by the supplier, i.e. 85-95%. The colour retention is 90%, corresponding to the visual observation. The residue colour is still distinctly red, but much more limpid than the wastewater. Comparing the residue colour in both permeates of reactive dyeing processes shows that colour retention is much lower in case Na_2SO_4 is applied as electrolyte.

Wastewater from *direct dyeing with NaCl* is filtered at a pressure of 10 bar. At first, the recovery amounted to 87% and slightly dropped to 83%. This corresponds to a decrease in the flow from 23.3 l/h m² to 16.5 l/h m². The obtained flow is higher than the measured fresh water flow at 20 °C. This may be explained by the lesser viscosity of the water at higher temperatures. The obtained permeate is almost colourless (98% colour removal), despite the fact that the wastewater had a deep blue—grey colour. The salt retention, on the other hand, is limited, i.e. 10%. This corresponds to the specifications of the membrane supplier.

Direct dyeing has also been performed with Na_2SO_4 . The salt dosage in direct dyeing is much smaller than in reactive dyeing. Therefore it is expected that the development of osmotic pressure by retaining sulphate ions will be much smaller. For this reason the filtration has been started at a pressure of 10 bar. This has resulted in a recovery of 82% and a flow of 16.3 l/h m². After 60 min the flow had already dropped to 10.5 l/h m², hence the decision to raise the pressure to 12 bar. Since the flow had dropped again after 180 min, the pressure has been further raised to 16 bar. This has resulted in an increased recovery of 79% and a flow of 12.8 l/h m². A sulphate retention of 81% corresponds to the indicated specifications, i.e. 85-95%. The colour retention is also rather good, i.e. 98%. Visually there still is a yellowish shine in the permeate but the deep blue colour of the wastewater has completely disappeared.

Disperse dyeing of polyester has been carried out in a light and a dark shade. Both wastewaters were filtered at 10 bar with similar performances in respect of recovery, flow and salt retention. The recovery and flow remained almost constant in both cases, being 75% and 12.5 l/h m², respectively. Despite the fact that no salt has been dosed, the wastewater has conductivities of viz. 0.9 mS/cm (light shade) and 2.52 mS/cm (dark shade). This is due to salts that are present in the process water, the dosed dyestuff and the hydrosulphite used for the reductive cleaning. The decrease of conductivity is 50% in both cases. The colour retention reveals clear differences, that of 86% for the light shade and only 69% for the dark shade.

3.2. Reuse of permeate on lab scale and pilot scale

The permeate of each filtration was again collected in mono-containers with the intention of reusing it for the same application. Before starting the reuse tests the quality of the respective permeate flows is determined and compared to the requirements for process water. This information is summarised in Table 4.

Table 4 Quality of permeate flows and requirements for process water

	Units	Reactive NaCl	Reactive Na ₂ SO ₄	Direct NaCl	Direct Na ₂ SO ₄	Disperse light	Disperse dark	Process water
COD	mg/l	216	91	443	22	377	438	60
Colour	_	Pink	Light red	Colourless	Yellowish	Colourless	Light brown	Colourless
pН	_	9.33	9.52	7.34	7.37	5.87	8.49	6.5-8
Conductivity	mS/cm	21.9	9.32	5.82	1.06	0.51	1.11	
Chloride	mg/l	7000	_	1750	_	_	_	
Sulphate	mg/l	_	5400	_	140	_	_	
Iron	μg/l	< 20	30	< 20	< 20	< 20	40	100
Manganese	μg/l	<1	5	3.5	<1	<1	<1	100
Hardness	$^{\circ} F$	2	2	2	<1	<1	<1	5

The COD concentration is relatively high in all permeate flows, except in direct and reactive permeate flows with Na₂SO₄. It is, however, not obvious which components are causing this COD and whether they will disturb the reuse.

Only two permeate flows are virtually colourless, i.e. those from direct dyeing with NaCl and the light disperse dyeing. The other flows are still clearly coloured. However, even a slight concentration of dyestuff leads to a visually noticeable colour in water.

The pH in permeates from both reactive dyeing processes is too alkaline, whereas the pH value of permeate from the light disperse dyeing is too low. On the one hand, an improvement is to be expected as a result of mixing fresh process water and on the other hand, a pH adjustment causes few practical problems.

In permeates from cotton dyeing, the conductivity is too high. This is most clearly in the case of reactive dyeing processes. Since the presence of salt in a dye bath is an important parameter for the fastness and degree of dyeing, it is necessary to reckon with increased residual electrolyte concentrations. In this test series, the concentrations of chloride and sulphate ions have been determined prior to performing the reuse tests. In this way, it is possible to reckon with the remaining concentration. However, on line reuse of the permeate may cause practical problems, unless an on line sensor is foreseen.

The degree of hardness and the concentrations of iron and manganese do not cause any problems for reuse.

Despite the fact that the quality of the permeates differs from the requirements for high quality process water, the reuse tests were still carried out, firstly on laboratory scale. The objective of the respective series of reuse tests on laboratory scale was to choose the best option (acceptable quality of the textile material with the highest percentage of permeate reuse) per type of dyeing. This selected procedure was afterwards tested on pilot scale. The laboratory tests had to clarify the following points:

- reuse of permeate pure or mixed with fresh process water;
- use of permeate merely for rinsing or both for dyeing and rinsing;
- need to adapt the salt dosage in case permeate is used for dyeing of cotton since the electrolyte concentration in the permeate can be substantial;
- necessity to carry out the last rinsing bath with fresh process water in the case of reactive dyeing;
- possibility to use pure permeate for both dyeing and rinsing of polyester.

These laboratory scale dyeing cycles have only been assessed on the basis of the difference in colour in comparison with the referential dyeing since this is the most critical parameter.

The biggest differences were measured in the reactive dyeing processes. The colour differences measured in the series with direct and disperse dyeing were always below 1, which is acceptable.

Based on these results the reuse strategies for pilot scale testing were selected (Table 5).

Prior to the dyeing process, the textile is wetted to facilitate an even intake of dyestuff and auxiliary products. The reactive and direct dyeing processes on pilot scale were again carried out on a cotton fabric whereas disperse dyeing processes were carried out on polyester spools. Spools function as a kind of filter since the water is always pumped from the outside to the inside through the spool and vice versa. The odds of residual dyestuff or other components are much higher in the case of spool dyeing than in the case of fabric dyeing. In unwinding dyed spools one may assess the influence on the evenness and the affinity to dyeing.

Of those textiles that have been dyed during the pilot tests, both the difference in colour and the fastness figures to laundering and rubbing have been determined.

In the case of *reactive dyeing* no permeate was used to dye, since the permeate still contains large residual electrolyte concentrations. More in particular, the permeate from reactive dyeing with NaCl contains 7 g/l chloride. This may cause the too large differences in colour (Table 6) and the deviating figures of fastness to laundering and rubbing (bleeding on cotton, fastness to wet rubbing).

The rinsing was done with a 50/50 mixture (permeate/process water) still containing a rather high amount of electrolyte.

Table 5
Pilot scale: water source used for each process step of the selected types of dyeing

Type of dyeing	Wetting (permeate/ process water)	Dyeing (permeate/ process water)	Rinsing (permeate/ process water)
Reactive - NaCl	0/100	0/100	50/50
Reactive - Na ₂ SO ₄	0/100	0/100	50/50
Direct - NaCl	0/100	75/25 ^a	75/25
Direct - Na ₂ SO ₄	0/100	75/25	75/25
Disperse - light	100/0	100/0	100/0
Disperse – dark	100/0	100/0	100/0

^a Adaptation for the residual electrolyte concentration in the permeate.

Table 6
Reuse test with permeate of reactive dyeing

	NaCl reference	NaCl reuse	Na ₂ SO ₄ reference	Na ₂ SO ₄ reuse
Colour difference (CMC 2:1)	_	1.25	_	0.81
Laundering (C2S)				
Decolouration	4-5	4-5	4-5	5
Cotton	4	3	4	4
Viscose	4-5	4-5	4-5	4-5
Rubbing (warp)				
Dry	4-5	4-5	4-5	4-5
Wet	4	3-4	4	4
Rubbing (weft)				
Dry	4-5	4-5	4-5	4-5
Wet	3-4	3-4	4	3-4

The sodium ions are dosed during the cotton dyeing in order to diminish the repulsion between negatively charged dyestuff molecules and the cotton fabric. The rinsing aims at removing hydrolysed and unfixed reactive dyestuff from the textile material. Rinsing with water still containing many sodium ions creates an extra attraction between the dyestuff and the textile material whereas the opposite effect is needed during rinsing. It is therefore recommendable to carry out the last rinsing bath with fresh process water.

The reuse of permeate in reactive dyeing with Na_2SO_4 has better results, since the residual sodium concentration is much smaller. Fastness to wet rubbing is only 0.5 units worse, which cannot be considered as significant. The difference in colour is limited and still within the accepted deviation. By comparing the results of reactive dyeing processes it is obvious that the rest colour in the permeate has a limited influence. The results of the reuse tests with Na_2SO_4 are, however, acceptable, in contrast to the ones with NaCl.

The reuse tests on a pilot scale have been carried out in case of direct dyeing processes with a mixture (75/25) of permeate and process water for both dyeing and rinsing. The results are summarised in Table 7.

Because the permeate's conductivity is 5.8 mS/cm, the NaCl dosage in the dye bath was adapted. However, the fastness to laundering (bleeding on cotton) and the fastness to wet rubbing are worse than the ones of the reference materials. The colour difference, however, is limited and is also visually satisfactory.

Reuse of the permeate for direct dyeing with Na_2SO_4 on the other hand may be done without any problem. No adaptation has been carried out in view of the limited presence of electrolyte. Also in these reuse tests, the rest colour in the permeate is of no consequence. In the dyeing processes with NaCl, the permeate is colourless, whereas the permeate of the dyeing process with Na_2SO_4 is still yellowish.

For both light and dark *disperse dyeing*, 100% permeate was used in all process steps. This option has been tested as a "worst case scenario". In practice, it is recommendable to work with a mixture of permeate and fresh process water in successive reuse cycles.

Table 7
Reuse tests with permeate of direct dyeing

	NaCl reference	NaCl ^a reuse	Na ₂ SO ₄ reference	Na ₂ SO ₄ reuse
Colour difference (CMC 2:1)	_	0.51	-	0.37
Laundering (A2S)				
Decolouration	4-5	4-5	4-5	4-5
Cotton	4	3	3-4	3-4
Wool	4-5	4-5	4-5	4-5
Rubbing (warp)				
Dry	4-5	4-5	4-5	4-5
Wet	4	3-4	4	4
Rubbing (weft)				
Dry	4	4-5	4-5	4-5
Wet	3-4	3	3-4	3-4

^a Adaptation for the residual electrolyte concentration in permeate.

In both cases of disperse dyeing, no significant difference in parameters, colour differences and figures of fastness to laundering and rubbing, was observed in relation to the reference dyeing (Table 8). However, the permeate of the dark dyeing contains a yellow-brown rest colour. The components causing this colour apparently do not disturb the dyeing process and cause no sediment onto the spool. In order to verify the evenness of the dyeing, the colour difference is determined by taking the middle of the spool as point of reference (Table 9). The differences are very small both in the reference and in the reuse dyeing cycles.

4. Conclusion

In order to arrive at a conclusion it is important to mention that the various wastewater flows have been kept apart during filtration as well as during reuse and that the permeate has each time been used in the same application.

Nanofiltration experiments and reuse tests prove that the organic charge of the effluents has little influence on the efficiency of the filtration and the possibilities to reuse the permeate as process water. Despite the higher COD values in the effluents of disperse dyeing processes the filtration was carried out without problems with an acceptable recovery and flow.

Table 8
Reuse tests with permeate of disperse dyeing

	Light reference	Light reuse	Dark reference	Dark reference
Colour difference (CMC 2:1)	-	0.25	-	0.09
Laundering (C2S)				
Decolouration	5	5	4-5	4-5
Cotton	4-5	4-5	4-5	4-5
Polyester	5	5	5	5
Rubbing				
Dry	5	5	5	5
Wet	5	4-5	5	4-5

Table 9
Evenness in reuse of permeate of disperse dyeing

	Colour difference (CMC 2:1) against middle spool (2)		
	Inside (1)	Outside (3)	
Light — reference	0.15	0.11	2 3
Light - reuse	0.11	0.36	
Dark - reference	0.17	0.23	
Dark – reuse	0.32	0.31	

The remaining COD in the permeate still amounted to about 400 mg/l, but this did not disturb the reuse tests in which the undiluted permeate was applied for the entire production process, comprising wetting, dyeing and rinsing.

The dyeing of cotton with a medium shade was performed four times to verify the influence of the type and dosage of electrolyte on the one hand and the used dyestuff, on the other.

If the dosage of NaCl or $\rm Na_2SO_4$ is compared, it seems that, as expected, the monovalent ions are hardly being retained whereas the bivalent ions are removed up to 80% in the permeate. This has, however, important consequences on the flow and recovery. The high retention of sulphate brings along a lower flow, certainly in the case of reactive dyeing in which large amounts of salt are required. This proves the necessity to assess the possibility to lower the salt dosage by selecting new reactive dyestuff.

Colour retention seems to be inversely proportional to the COD removal and salt retention. In the case of reactive as well as of direct dyeing processes, the least coloured permeates are obtained by filtering effluents with NaCl, whereas the retention of chlorides and COD are very low. The evaluation of the reuse tests proves that the presence of a limited rest colour is no hindrance. The conductivity of the permeate has a much larger impact on the possibilities of reuse. The best results have been obtained both in reactive and direct dyeing by using Na₂SO₄ since this could be retained much more efficiently by the membrane. As a consequence, the sodium concentration in the permeate is lower and does not disturb the reuse. It has to be mentioned that in the reactive and direct dyeing a mixture of permeate and fresh process water has been used to limit the influence of the higher conductivity.

The pH values of the permeate and the used dyestuff, direct or reactive, do not determine the results of the reuse tests neither the degree of hardness, the iron and manganese concentrations, which comply with the requirements for process water.

As a general conclusion of filtration and reuse tests on a pilot scale one may state that the remaining electrolyte concentration in the permeate determines the possibilities for reuse to a large extent. This is most distinct in reactive dyeing processes in which a considerable quantity of salt is dosed. Another important aspect of reuse is the measure in which the quality of the permeate remains stable in time. This is determined by the efficiency of the membrane filtration on the one hand, and the measure in which certain components will

concentrate in the permeate during the successive reuse cycles. This, however, fell outside the scope of the present tests.

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