



## Water reuse in the textile industry with integrated treatments: membranes and advanced oxidation processes

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### ABSTRACT

Nowadays, traditional methods for dye removal from industrial effluents have been integrated with chemical oxidation processes, mainly advanced oxidation processes (AOPs) which are able to degrade complex organic substances. In this paper, the integration of membrane process and hydrodynamic cavitation for the treatment of a dyeing wastewater is presented. The degradation efficiency of two dyes (methyl orange and methylene blue) obtained in previous experimental activity and here resumed are used as an input to simulate an integrated treatment cycle in which dyeing wastewater is pre-treated, filtrated through a membrane operation, subjected to an AOP with hydrodynamic cavitation in a hybrid configuration. The integration among physical treatment (membrane) and chemical/physical treatment (hydrodynamic cavitation coupled with Fenton treatment) allows to obtain a depurated stream (permeate) that can be reused as process water and a concentrated stream (retentate) that after the treatment can be safely discharged into superficial body.

*Keywords:* Water reuse; Dyes; Textile industry; Membrane; Advanced oxidation processes; Hydrodynamic cavitation

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### 1. Introduction

Nowadays, dyeing wastewater pollution is considered a serious environmental problem. Dyeing processes in the textile industry and fabric manufacturing require the use of various chemical dyes and other additives [1]. When these substances are discharged into wastewater without proper treatment, they can have a significant negative impact on the environment [2]. This particular type of industrial wastewater contains high concentrations of dyes, salts, heavy metals, and other chemicals that can cause severe pollution [3]. Dyes can discolour the water, making it unsightly, and can also inhibit sunlight penetration. Additionally, many dyes and chemicals used in the dyeing process are toxic to

aquatic life. They can disrupt the natural balance of ecosystems and harm fish, and invertebrates [4]. Additionally, some dyes contain heavy metals like chromium, lead, or cadmium, which can be highly toxic and bioaccumulate [5]. Chemicals and dyes present in the wastewater can also seep into the soil, potentially reaching groundwater and causing long-term contamination, thereby introducing toxins into the human food chain. Exposure to certain dyes and chemicals has been associated with adverse health effects such as skin irritation, respiratory problems, and even cancer [6].

To mitigate the environmental impact of dyeing wastewater, industries typically implement wastewater treatment processes, such as biological treatment, adsorption on activated carbon, membrane filtration, ion exchange,

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coagulation precipitation, and extraction, to remove or reduce the concentration of pollutants before the water is discharged [7]. As a matter of fact, these traditional schemes, combining biological/physical/chemical processes, if used alone, are often unsatisfactory because of the high treatment times, especially for biological operations; other problems are the low efficiency and the transfer of pollutants from the liquid stream to the solid or another liquid phase that is an additional secondary source of contaminants. For example, membrane processes are commonly used for the treatment of dyeing wastewater. They involve the use of selectively permeable membranes to separate pollutants from the wastewater based on their size, charge, or solubility [8]. Even if they are very effective for wastewater depuration, these processes generate a concentrated waste stream, known as concentrate or brine, containing the rejected contaminants. Depending on the characteristics of the concentrate, additional treatment steps may be required. Appropriate management and disposal of the concentrate are thus necessary for its safe discharge or proper disposal, as well as to prevent environmental impacts [9]. On the contrary, the integration of traditional treatments such as membranes with advanced oxidation processes (AOPs) has the main advantage of reducing the volume of liquid to be treated and thus the oxidant dosage, with degrading up to complete mineralization pollutants, and obtaining fresh and safe water, free from any dangerous contaminant [10].

Advanced oxidation processes are highly effective methods for treating dyeing wastewater. AOPs involve the generation of powerful oxidizing agents, hydroxyl radicals  $\cdot\text{OH}$ , to break down and remove organic pollutants [11]. Each AOP has its own mechanism and operating conditions. It's important to study and select the most suitable AOP based on the characteristics of the dyeing wastewaters and the desired treatment objectives. Moreover, prior to applying AOPs, it's advisable to perform preliminary treatments such as pH adjustment, filtration, or sedimentation to remove solid particles, oil, and other substances that can interfere with the AOPs [12]. This step ensures optimal performance and prolongs the life of the AOP equipment. Based on the characteristics of the dyeing wastewater, the most appropriate AOP method is to be chosen. The most common are: ozone-based processes [13] that use ozone ( $\text{O}_3$ ) alone or in combination with hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) or ultraviolet (UV) light to enhance treatment efficiency [14]; hydrogen peroxide-based processes [15], which use hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) as the oxidant that can be activated by UV light or other catalysts to produce highly reactive hydroxyl radicals ( $\cdot\text{OH}$ ), breaking down the pollutants. In the photocatalytic processes [16], titanium dioxide ( $\text{TiO}_2$ ) is commonly used; when irradiated with UV light,  $\text{TiO}_2$  generates electron–hole pairs that react with water and oxygen to produce hydroxyl radicals, which degrade the pollutants. Finally, hydrodynamic cavitation (HC) is an emerging AOP technology that can be used for the treatment of dyeing wastewater [17]. It involves the generation and subsequent collapse of cavitation bubbles in a high-velocity flow, resulting in the release of intense energy that can degrade and remove pollutants. Hydrodynamic cavitation occurs when a liquid is subjected to rapid changes in pressure, resulting in the formation and collapse of cavitation

bubbles [18]. The bubbles are generated in a low-pressure region, for example, in a Venturi tube's throat. During bubble collapse, high temperatures, pressures, and localized shock waves are generated, resulting in the generation of highly reactive radical species, which can cause the degradation of organic compounds present in the wastewater. Moreover, HC can remarkably enhance the oxidative degradation of organics by promoting the mass transfer and  $\cdot\text{OH}$  formation in combination with other AOPs, such as hydrogen peroxide, Fenton oxidation, and ozonation [19].

The use of HC in environmental engineering technologies allows processes to be greatly effective during water and effluent treatment [20]. This technology can be considered as a non-waste technology and environmentally friendly due to the possibility of degrading low biodegradable and hazardous organic compounds, resistant to conventional disposal methods, such as pesticides, dyes, or complex organic compounds, which in the cavitating liquid environment become susceptible to biodegradation. This is particularly important when water is intended for reuse since these compounds are almost "transparent" to the depuration treatments carried out in conventional wastewater treatment plants, so if they are present in the effluent, apart from very low removal percentage due to sludge sedimentation, they will be almost unaffected, thus entering the water cycle [21,22]; it is expected that developed process of wastewater treatment with aid of cavitation will considerably reduce the presence of these micropollutants in purified water. Moreover, the use of HC appears of major importance since it can diminish the use of expensive chemical reagents for advanced treatment processes, whose residual streams need to be further treated and managed.

In this paper, a combined process that integrates membrane and hydrodynamic cavitation is proposed, for the treatment of dyeing wastewater aiming not only at wastewater depuration but also at water reuse. Process analysis is carried out on simulated typical wastewater coming from a tannery, and HC is used alone and in combination with hydrogen peroxide (hybrid configuration) to reduce the oxidant dose. The degradation efficiency results obtained in previous experimental activity are used as an input to simulate an integrated treatment cycle in which wastewater is pre-treated, filtered through a membrane operation, subjected to an AOP with HC in a hybrid configuration, and then can be reused; while the concentrated stream is purified and discharged. Material balance is presented.

## 2. Materials and methods

HC experiments were carried out using the apparatus reported in Fig. 1. The cavitation device in the HC reactor is a Venturi. The details of the reactor configuration and Venturi were previously reported in our previous study [23]. All experiments were performed on a constant effluent volume of 1 L. The temperature of the solution ( $T = 20^\circ\text{C}$ ) was kept constant by circulating cooling water through the jacket. The total treatment time for all experiments was 60 min, and samples were taken at regular time intervals for analysis. In order to improve HC efficiency, the effect of advanced oxidative reagents such as,  $\text{H}_2\text{O}_2$  in combination with HC was also investigated at the optimized inlet pressure.

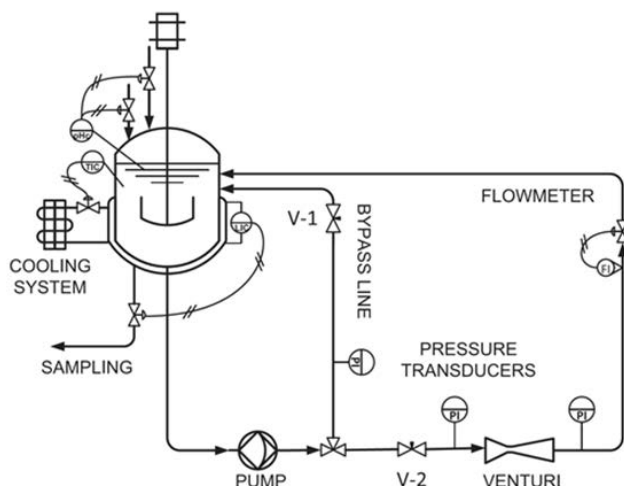


Fig. 1. Lab-scale experimental apparatus.

Experiments were carried out at an inlet pressure in the range 0.2–0.5 MPa to investigate the combined effect of HC/hydrogen peroxide on colour reduction of textile wastewater. The concentration of  $H_2O_2$  was varied between 0.1–0.9 M. Methylene blue (MB;  $C_{16}H_{18}N_3ClS$ ; molecular weight: 319.85 g/mol), methyl orange (MO;  $C_{14}H_{14}N_3NaO_3S$ ; molecular weight: 327.33 g/mol) and distilled water were used to prepare the dye solutions. Dye concentrations varied between 5 and 100 ppm, according to the adopted experimental procedures. Sodium hydroxide (Fluka Chemika, Switzerland, >97%) and sulfuric acid (Carlo Erba, Milano, Italy, 98%) were used to adjust the pH of the solution.

The reduction of colour was measured in order to study the efficiency of the hybrid process. The collected samples were analyzed using a UV spectrophotometer (Cary 1E UV/Visible Spectrophotometer Varian, Agilent Technologies, Santa Clara, California, USA) in order to observe a change in the absorbance of dyes with time at a specific wavelength ( $\lambda$ ).

### 3. Results and discussion

#### 3.1. Experimental results with HC alone (dyes: MO and MB)

The experimental apparatus previously described has been used to perform experimental cavitation tests on several dyes [23,24], in stand-alone configuration and in hybrid treatments.

Here are reported in an aggregated form the results obtained for two kinds of contaminants, (methyl orange, MO; methylene blue, MB) as for the effect of inlet pressure (inlet pressure range: 0.2–0.5 MPa) with using HC in a stand-alone configuration.

The results are expressed in terms of a removal efficiency, defined as:

$$\eta = \text{removal efficiency} = \frac{[c]_0 - [c]_t}{[c]_0} \times 100 \quad (1)$$

where  $[c]_t$  and  $[c]_0$  are the concentrations of contaminant in ppm at a treatment time of 60 min ( $t$ ) and at the initial time.

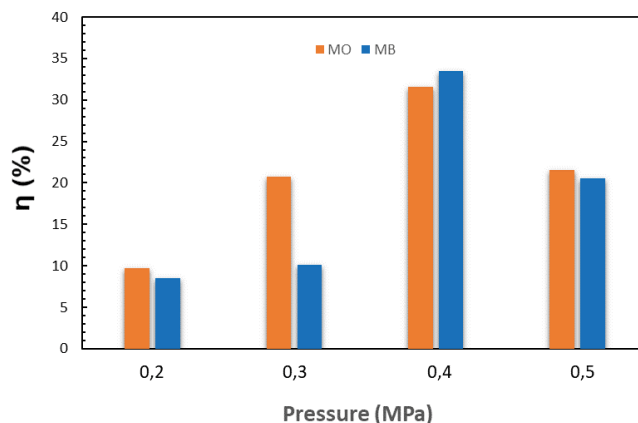


Fig. 2. Effect of the inlet pressure on removal efficiency of methyl orange and methylene blue.

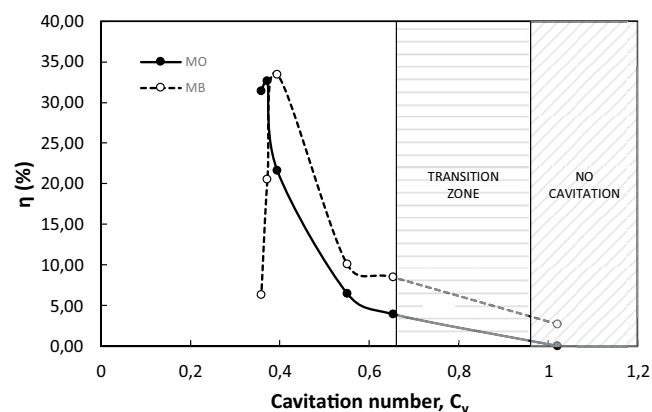


Fig. 3. Effect of the cavitation number on the removal efficiency of methyl orange and methylene blue.

Fig. 2 shows the positive effect of the inlet pressure for all the tested contaminants, since the increase in pressure leads to an increase in the removal efficiency, up to a maximum value of pressure in the range 0.3–0.4 MPa, above which the efficiency starts to decrease. This trend is similar to that already found in the literature for other compounds, and the increase of HC efficiency on removal efficiency can be attributed to the enhanced OH radical production as a result of the intensification of the cavitation activity at the higher pressures. However, above certain values of pressure, this positive outcome disappears, and the efficiencies start to decline.

In the graph in Fig. 3 the degradation efficiency of the tested compounds is reported as a function of the cavitation number.

$$C_v = \text{cavitation number} = \frac{P_2 - P_v}{0.5\rho v_0^2} \quad (2)$$

where  $P_2$  and  $P_v$  are the pressure downstream of the Venturi device and the vapor pressure of the liquid, respectively;  $v_0$  is the fluid velocity at the convergent. The cavitation number is a dimensionless parameter, extensively used in the

literature to characterize this kind of device; as the inlet pressure increases, the flow rate increases and the cavitation number decreases because of the higher kinetic energy of the liquid flow. As  $C_v$  becomes less than one, the fluid velocity grows, resulting in increased turbulence and, therefore, cavitation occurs with the formation and subsequent implosion of the cavitation bubbles.

In the plot, three regions can be individuated, separated by a transition zone: on the right part of the plot, a region where no cavitation occurs is present, since  $C_v$  exceeds the unity for an inlet pressure below 0.2 MPa, and the degradation efficiencies are very low (below 3%) for all the tested compounds; in the central zone of the plot, the cavitation zone is individuated;  $C_v$  falls below the unity for an increase in the pressure upstream of the Venturi, due to the greater kinetic energy of the circulating liquid. Therefore, to be sure to work in cavitating conditions, experiments are conducted for an inlet pressure well above 0.2 MPa and  $C_v < 0.8$ . In this zone, with increasing pressure, thus with decreasing  $C_v$ , the degradation efficiencies grow for all the tested compounds, up to a maximum, then start to decrease. This phenomenon, called choked cavitation, characterizes the region on the left of plot, it was observed for  $C_v < 0.4$  and is located on the decreasing branch of the curves, and it is characterized by many cavities that merge to form vapor bubbles without collapsing to produce radicals useful for the oxidation reaction. In the choked cavitation zone, degradation efficiencies decrease significantly. This confirms that there is a range of pressures, and consequently a range of  $C_v$ , in which cavitation experiments have to be carried out in for the specific device, independently from the compound to be degraded. For the experimental apparatus here presented, the range is 0.3–0.6 MPa.

However, by the reading of Figs. 2 and 3, it appears that for all the tested molecules, the removal efficiencies with using only cavitation after 60 min of treatment time are in the range 30%–35% at the best pressure values (MO, MB).

### 3.2. Experimental results with HC in hybrid configuration (dye MO)

For the same contaminant MO, experimental results carried out in hybrid configurations with adding hydrogen peroxide in the same experimental apparatus have shown that the degradation efficiencies grow up significantly. As for example, Fig. 4 shows the degradation efficiency of MO in the cavitation device with different concentration of  $H_2O_2$  added in solution [25].

As can be seen from the analysis of Fig. 4, the value of MO/ $H_2O_2$  molar ratio equal to 1:25 with a treatment time of 60 min appears to be as the optimal dosage. The worsening of performance at higher hydrogen peroxide concentrations is due to the recombination of OH radicals, a possible negative outcome of the oxidant which may react with the hydroxyl radicals thus decreasing the radicals present in solution, destined to degrade methyl orange.

Moreover, the efficiency of the hydrodynamic cavitation process in the presence of the metals (iron and nickel) and hydrogen peroxide (0.005%v/v of hydrogen peroxide, iron (20 mg/L) and nickel (10 mg/L) = 0.4 MPa, was tested, showing that combining HC,  $H_2O_2$  and metals, the

degradation yields were near to 90%, meaning that the presence of iron induced an advanced Fenton process which improved the efficiency of the entire process of cavitation.

### 3.3. Process analysis (real wastewater)

A process analysis is discussed below, considering a real wastewater, whose average characteristics are reported in Table 1. It is a real wastewater taken from a textile industry, which operates in Abruzzo region.

Two different cases were investigated: one involving hydrodynamic cavitation (HC) process alone, the second adding a mixture of  $H_2O_2$ +Ni+Fe before HC occurs. The aim of both plants is the production of a high-quality water reusable for industrial purposes, in particular in textile industry.

Fig. 5a and b shows the process flow diagram proposed for both cases.

The two schemes share the following steps. The stream n. 1, which is the dyeing wastewater effluent produced by the textile industry and characterized in Table 1, undergoes a screening operation, which is able to reduce the biochemical oxygen demand (BOD) and the total suspended solids (TSS) values. The most efficient fine screen devices are the rotary drums, which are often used to replace primary sedimentation. Typical data about removal percentage spam within 25%–50% for BOD and 25%–45% for TSS [26]. Considering a mean value, the BOD of stream n. 2

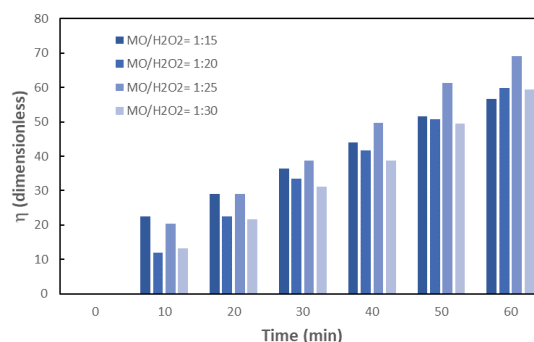


Fig. 4. Effect of the hydrodynamic cavitation with  $H_2O_2$  on the removal efficiency of methyl orange.

Table 1  
Characteristics of wastewater

Parameter	Concentration
COD, mg/L	140
BOD, mg/L	10
TSS, mg/L	1,500
TOC, mg/L	64.5
Cl <sup>-</sup> , mg/L	140
NO <sub>3</sub> <sup>-</sup> , mg/L	6.15
SO <sub>4</sub> <sup>2-</sup> , mg/L	172.6
Total surfactant, mg/L	3.848
PO <sub>4</sub> <sup>3-</sup> , mg/L	0.253
Colour, abs	0.78

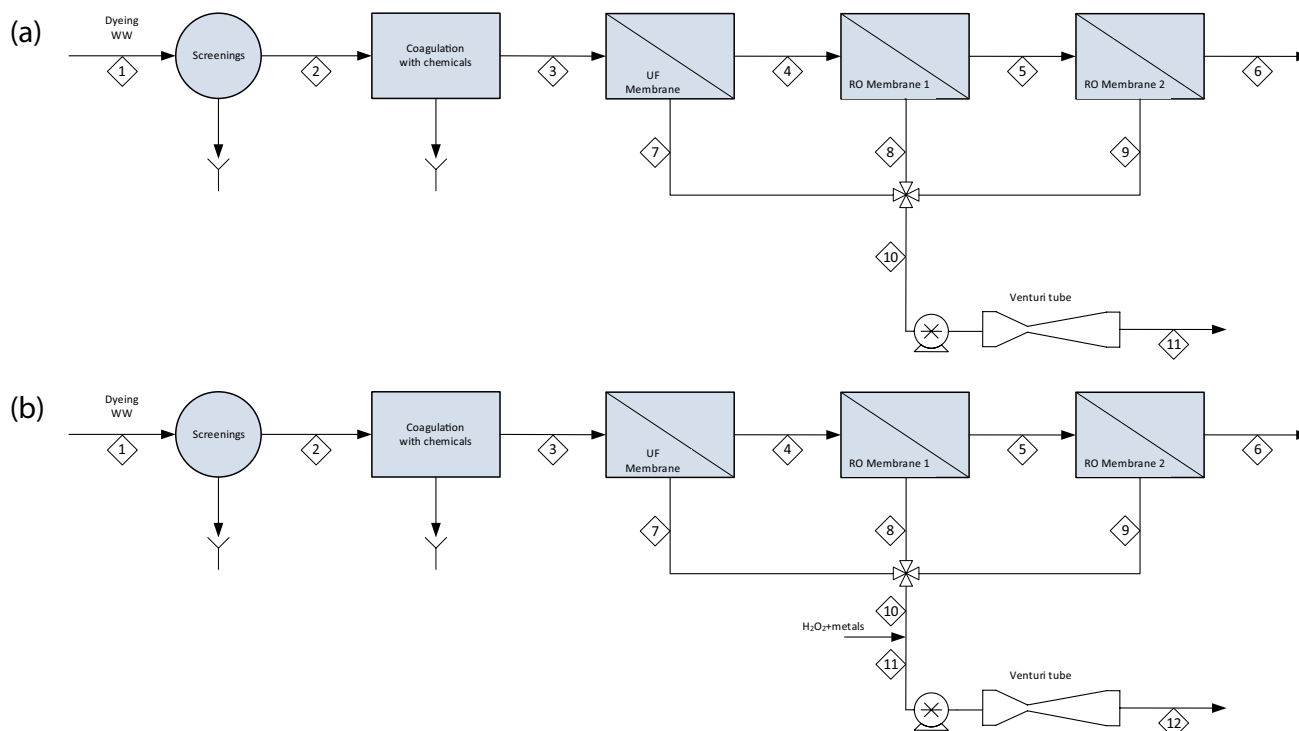


Fig. 5. (a) Process analysis with hydrodynamic cavitation alone and (b) process analysis with hydrodynamic cavitation preceded by adding a solution of H<sub>2</sub>O<sub>2</sub>+metals.

decreases from 10 mg/L (Table 1) until 6.3 mg/L, while TSS falls from 1,500 mg/L (Table 1) to 975 mg/L.

Subsequently, the effluent is subjected to a coagulation process with chemicals. Different substances can be added to the wastewater: ferric chloride, ferric chloride with lime and ferric sulphate with lime. Anyway, if they are correctly dosed, if the mixing is efficient and the process is properly controlled, TSS may be reduced from 80% to 90%, while BOD from 50% to 80% [26]. Again, considering the mean values, the BOD and TSS of stream n. 3 reduce respectively to 2.2 and 146 mg/L.

These preliminary steps are followed by tangential membrane sections, which consists of three different stages: the first is an ultrafiltration process (UF), while the next two are both reverse osmosis (RO) modules. During the UF step the incoming effluent named as n. 3, is divided into a permeate and retentate streams. Here it is crucial the correct choice of the module, in terms of material and MWCO. The dyes inside wastewater usually show a rejection of about 97%, if the membrane material is made of polysulfone with a MWCO greater than 700 Da [27]. Simultaneously, this configuration permits to reach an interesting reduction of both chemical oxygen demand (COD; 75%–85%) and total organic carbon (TOC) (50%–60%). Finally, UF realizes the almost total rejection of residual TSS [28] and surfactant [29,30] amounts. Consequently, the stream n. 4, that is the permeate stream of UF membrane, shows these characteristics: COD 28 mg/L, TOC 29 mg/L, TSS 0 mg/L, total surfactant 0 mg/L.

RO is the most selective tangential membrane process. A typical performance summary is showed in Table 2.

Table 2  
Typical performance summary for reverse osmosis process

Parameter	Average reduction, %	References
COD	94	[31]
BOD	30–60	[32]
TOC	90–98	[26]
Cl <sup>-</sup>	98.9	[33]
NO <sub>3</sub> <sup>-</sup>	84–86	[26]
SO <sub>4</sub> <sup>2-</sup>	99.8	[31]
PO <sub>4</sub> <sup>3-</sup>	99	[32]
Colour	90–96	[26]

With the average reduction showed above the n. 5 permeate stream is a good quality water, except for Cl<sup>-</sup> concentration as its value is still too high (1.54 mg/L). This is the reason why the second RO membrane module is necessary, by which not only the Cl<sup>-</sup> concentration decreases to very low value (0.0169 mg/L), but also the other parameters improve further, to obtain a high-quality effluent water that can be reused for industrial purposes or even discharged in superficial water. Its characteristics are reported in Table 3.

The three retentate streams, n. 7, 8 and 9, are mixed all together and they undergo a different treatment, as shown in Fig. 5a and b.

Referring to Fig. 5a, the stream n. 10 is treated in a hydrodynamic cavitation reactor, with proper value of pressure and time as defined in the experimental section (inlet pressure  $p = 0.4$  MPa, treatment time  $t = 60$  min).

Table 3  
Characteristics of permeate stream n. 6

Parameter	Concentration
COD, mg/L	0.10
BOD, mg/L	0.66
TSS, mg/L	0.00
TOC, mg/L	0.10
Cl <sup>-</sup> , mg/L	0.0169
NO <sub>3</sub> <sup>-</sup> , mg/L	0.14
SO <sub>4</sub> <sup>2-</sup> , mg/L	0.000690
Total surfactant, mg/L	0.0
PO <sub>4</sub> <sup>3-</sup> , mg/L	0.0000253
Colour, abs	0.0

Table 4  
Characteristics of permeate stream n. 11, after HC alone

Parameter	Concentration
COD, mg/L	119
BOD, mg/L	1.53
TSS, mg/L	146
TOC, mg/L	8.37
Cl <sup>-</sup> , mg/L	140
NO <sub>3</sub> <sup>-</sup> , mg/L	6.01
SO <sub>4</sub> <sup>2-</sup> , mg/L	172.6
Total surfactant, mg/L	4.0
PO <sub>4</sub> <sup>3-</sup> , mg/L	0.253
Colour, abs	0.53

The HC alone is able to reduce TOC amount to 87% [34] and the COD almost by 15% [35]. Furthermore, as showed in Fig. 2, this process is effective on colour reduction, reaching a percentage decrease between 30%–35%, in case of pressure range between 0.3–0.4 MPa. Even ions in solution may be strongly lowered, as showed by Khajeh et al. [36]: they can chemically interact with OH<sup>-</sup> radicals, giving rise to the following percentage reductions: Cl<sup>-</sup> 69.5%, NO<sub>3</sub><sup>-</sup> 94.3%, SO<sub>4</sub><sup>2-</sup> 98.6%, PO<sub>4</sub><sup>3-</sup> 90.6%.

Table 4 reports the characteristics of stream n. 11. There are some critical values in terms of TSS, Cl<sup>-</sup>, and colour, which make this stream not suitable to be reused, even for textile industrial applications. A further process is necessary, for example an adsorption on active carbon to improve the effluent and to make him suitable for water reuse.

Better performances may be obtained if hydrodynamic cavitation process is performed in presence of both hydrogen peroxide and metals. Authors, using 0.005% v/v of H<sub>2</sub>O<sub>2</sub> together with 20 mg/L of iron and 10 mg/L of nickel, with an inlet pressure of 0.4 MPa, reached a colour degradation yield of about 90%. This satisfactory result was due not only to the presence of hydrogen peroxide which favours the formation of more OH<sup>-</sup> free radicals, that are available to degrade organic substances. In addition, the presence of metals promotes the reaction with oxygen, to produce further OH<sup>-</sup> radicals, which react with the dye, allowing to

Table 5  
Characteristics of permeate stream n. 12, after adding H<sub>2</sub>O<sub>2</sub>+metals before HC

Parameter	Concentration
COD, mg/L	7.0
BOD, mg/L	1.53
TSS, mg/L	146
TOC, mg/L	8.35
Cl <sup>-</sup> , mg/L	43
NO <sub>3</sub> <sup>-</sup> , mg/L	0.33
SO <sub>4</sub> <sup>2-</sup> , mg/L	2.4
Total surfactant, mg/L	4.0
PO <sub>4</sub> <sup>3-</sup> , mg/L	0.024
Colour, abs	0.078

achieve such a degradation yield. Further benefits were also found by Agarkoti et al. [35], who added H<sub>2</sub>O<sub>2</sub> and metals inside the wastewater before HC occurs, achieving a COD reduction yield until 95% with a proper Fe/H<sub>2</sub>O<sub>2</sub> ratio. Table 5 shows the characteristics of stream n.12 of Fig. 5b.

Looking at Tables 4 and 5 it is possible to notice that stream n. 12 of Fig. 5b is highly improved if compared with stream n. 11 of Fig. 5a, in terms of colour, TOC and COD reduction. Anyway, there is still a critical value, which is the concentration of chlorine ion (43 mg/L Table 5). Another step of HC, optimized to remove Cl<sup>-</sup>, will be able to make also this stream suitable for textile industrial reuse, reaching the ambitious target of zero-waste discharge process.

#### 4. Conclusions

This paper reports the results of a process analysis about the integration of membrane process and hydrodynamic cavitation for the treatment of an industrial wastewater containing dyes. Based on the degradation efficiency of methyl orange and methylene blue in an experimental plant with a Venturi orifice, an integrated treatment cycle is simulated, in which dyeing wastewater is pre-treated, filtered through a membrane operation, subjected to an AOP with HC in a hybrid configuration. The material balance on different stages of the treatment train shows that the integration among membranes and hydrodynamic cavitation in a hybrid configuration (cavitation with H<sub>2</sub>O<sub>2</sub> and Fenton treatment) allows to obtain clean water that can be reused as process water and a concentrated stream that after a tertiary treatment can be safely discharged.

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