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Title: Treatments for color removal from wastewater: state of the art

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Abstract: This work focuses on different types of chemical/physical, chemical, electrochemical and biological processes applied in the field of color removal from industrial wastewater. Both examples of treatments already widely applied to the real scale and still in the experimental phase are reported. This paper also presents the reaction mechanisms that occur during the different discoloration processes and the advantages and disadvantages that may exist in adopting one type of treatment in spite of another. Common chemical/physical treatments such as coagulation/flocculation, adsorption and membrane filtration as well as chemical-type processes are discussed, both those that exploit the traditional oxidizing chemical agents such as Ozone, H<sub>2</sub>O<sub>2</sub> and reactive based on chlorine and those based on the principle of advanced chemical oxidation. In particular, both Hydroxyl radical based AOPs (more precisely Ozone-based, H<sub>2</sub>O<sub>2</sub> based, Fenton processes, wet oxidation and photocatalysis with TiO<sub>2</sub>/UV) and Sulfate radical based advanced chemical oxidation processes are reported. The most commonly used Electrochemical processes for the removal of color are also presented (Electro-photocatalysis with TiO<sub>2</sub>/UV, Electro Fenton, Anodic Oxidation, Electrocoagulation) as well as biological treatments (microbial biomass reactors - RBM and fungal treatments). There is also a report with some examples specifically chosen for each type of treatment.

## Title page: **Treatments for color removal from wastewater: state of the art**

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Pavia, September 4<sup>th</sup>, 2018

Dear Editor-in-Chief,

we would like to submit the manuscript titled “TREATMENTS FOR COLOR REMOVAL FROM WASTEWATER: STATE OF THE ART” for possible publication in *Journal of Environmental Management*.

This work focuses on different types of chemical/physical, chemical, electrochemical and biological processes applied in the field of color removal from industrial wastewater. Many examples of treatments already widely applied to the real scale and still in the experimental phase are reported. At the end of every chapter there is also a report with many examples specifically chosen for each type of treatment.

We find that, while some advanced chemical oxidation methods as well as chemical/physical methods are certainly already commonly used a lot of work is needed before real-scale applications of electrochemical processes or biological processes can be developed.

This paper is original and unpublished; it is not being considered for publication by any other journal.

Yours Sincerely,

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- Wastewater colour removal is a process that must be assessed on a case-by-case basis
- Chemical/physical methods are certainly still common used
- Advanced chemical oxidation processes are the most promising ones today
- Electrochemical and biological processes must still be adequately studied

## Treatments for color removal from wastewater: state of the art

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This work focuses on different types of chemical/physical, chemical, electrochemical and biological processes applied in the field of color removal from industrial wastewater. Both examples of treatments already widely applied to the real scale and still in the experimental phase are reported. This paper also presents the reaction mechanisms that occur during the different discoloration processes and the advantages and disadvantages that may exist in adopting one type of treatment in spite of another. Common chemical/physical treatments such as coagulation/flocculation, adsorption and membrane filtration as well as chemical-type processes are discussed, both those that exploit the traditional oxidizing chemical agents such as Ozone, H<sub>2</sub>O<sub>2</sub> and reactive based on chlorine and those based on the principle of advanced chemical oxidation. In particular, both Hydroxyl radical based AOPs (more precisely Ozone-based, H<sub>2</sub>O<sub>2</sub> based, Fenton processes, wet oxidation and photocatalysis with TiO<sub>2</sub>/UV) and Sulfate radical based advanced chemical oxidation processes are reported. The most commonly used Electrochemical processes for the removal of color are also presented (Electrophotocatalysis with TiO<sub>2</sub>/UV, Electro Fenton, Anodic Oxidation, Electrocoagulation) as well as biological treatments (microbial biomass reactors - RBM and fungal treatments). There is also a report with some examples specifically chosen for each type of treatment.

**Keywords:** color removal, dye degradation, advanced oxidation process, textile wastewater, electrochemical processes

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## 1. Introduction

The problem of color in wastewater is among those, together with the odor problem, more easily perceived by other people (Ghalebizade and Ayati, 2016; Gulnaz et al., 2012). Among the industrial waters are those coming from activities that use dyes in their production cycles. So obviously we are talking about textile companies, food companies, distilleries, cosmetics industries, chemical and pharmaceutical companies, paper mills, petroleum industries and printing houses (Gupta et al., 2011; Mashkooor et al., 2018; Mehrali et al., 2010; Satyawali and Balakrishnan, 2008). Some particular categories of aqueous waste such as landfill leachate or aqueous food waste are not exempt from staining problems (Foo and Hameed, 2010; Satyawali and Balakrishnan, 2008). The release of these colored wastewater into the environment is also a source of problems for the aquatic ecosystem. One of the main concerns is the reduction of the penetration of light radiation into water which can cause an alteration of the photosynthetic activity and thus alter the natural balance of flora and fauna. (Mashkooor et al., 2018).

Dye molecules are made up of a chromogen or an aromatic structure capable of absorbing visible light. This explains why they can be appreciated with the naked eye. Twelve classes of chromogenic groups are known and the most famous is certainly that of nitrogen dyes. Fall into this category for example: Orange G, Acid Orange 7, New coccine, Acid Black 1, Tartrazine, Acid yellow 17 e Congo red. They all obviously have at least one group-azo (double bond  $N = N$ ) (Tanaka et al., 2000).

Dyes, in particular the nitrogenous ones, possess complex aromatic structures. Precisely for this reason their degradation does not occur effectively with conventional biological treatment methods. Therefore, different treatments are required for discoloration of the initially colored wastewater (Ghalebizade and Ayati, 2016; Hao et al., 2000; Kim and Ihm, 2011; Muruganandham and Swaminathan, 2006a; Tanaka et al., 2000). It must be said that there is a clear distinction between discoloration and degradation. In fact, when we talk about

discoloration, we simply refer to the loss of color from the wastewater that is the object of the analysis. However, the water so bleached may still be rich in organic substance (Hao et al., 2000). Following studies analyzed below, it has been observed that there is no single method applicable to all the different types of colored water because the high variability of the nature of the coloring substances must also be taken into consideration (Tanaka et al., 2000). To date, a considerable number of chemical and chemical/physical treatments have been developed (Butani and Mane, 2017; Golob et al., 2005; Mane and Babu, 2011; Pokhrel and Viraraghavan, 2004; Sadri Moghaddam et al., 2010; Satyawali and Balakrishnan, 2008; Vijayaraghavan et al., 2017; Xiaoxu et al., 2017; Zonoozi et al., 2008). Coagulation/flocculation treatments shows some problems. Significantly colored wastewater can in fact lead to the use of an excessive amount of coagulant, to the formation of a high amount of chemical sludge, as well as in some cases fail to effectively remove the chromophoric substances (Ghalebizade and Ayati, 2016; Gulnaz et al., 2012; Rajkumar and Kim, 2006). Activated charcoal also plays a decolorizing function (Crini, 2006; Malik, 2004; Purkait et al., 2007; Robinson et al., 2001) but the convenience in its applicability is not always optimal and depends on the intensity of the initial coloration and on the nature of the chromophores. It should be noted that regenerating activated carbon is generally inefficient as well as very expensive (H.-Y. Shu and Chang, 2005; Tanaka et al., 2000). Over the years, for the resolution of the color problem, studies have also focused on the use of chemical treatments, both traditional and advanced, that is the AOPs (Franz et al., 2015; Ghalebizade and Ayati, 2016; Turolla et al., 2012).

The Advanced Oxidation Processes (AOPs) were proposed for the first time in the 80s for the treatment of drinking water but their study extended, in the following years, to the application for other types of water including obviously those of colored discharge (Deng and Zhao, 2015).

Today, as a result of increasingly stringent regulations especially with regard to the removal of refractory organic substances such as dyes, pesticides, solvents, AOPs are among the most

studied methods. They are based on the use of radical transition species, such as  $\bullet\text{OH}$  or the  $\text{SO}_4^{\bullet-}$ , with high reactivity and no selectivity (Deng and Zhao, 2015; Franz et al., 2015; Tanaka et al., 2000; Turolla et al., 2012; Zaharia and Dartu, 2010).

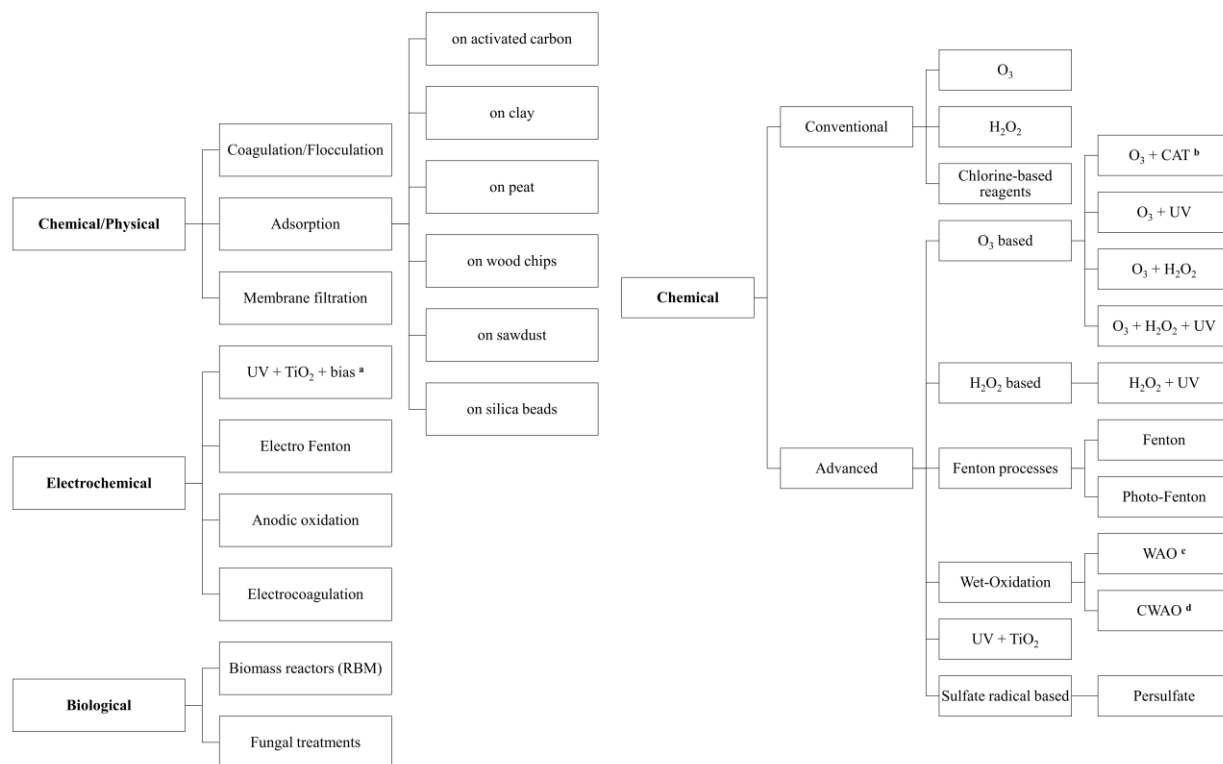
They have a very high redox potential ( $\bullet\text{OH}$ : 2.80V;  $\text{SO}_4^{\bullet-}$ : 2.60V) when compared to other oxidizing agents such as  $\text{O}_3$  (2.08 V) or  $\text{Cl}_2$  (1.36 V) (Deng and Zhao, 2015; Sharma et al., 2011). This allows them to quickly and completely degrade the pollutants present in the waters, including obviously chromophores (Patel et al., 2013).

The high versatility of the AOPs is also given by the significant number of existing methods with which to produce radical transition species (Sharma et al., 2011). This makes it possible to choose the chemical process best suited to the specific needs of the treatment examined after an in-depth study has been carried out (Andreozzi, 1999; M. Collivignarelli et al., 2017). Moreover, the integration between chemical oxidation processes and conventional (Mohajerani et al., 2009) or innovative (Bertanza et al., 2010) biological treatments can be used to take advantages of the treatments and to minimize the drawback of each methods. Electrochemical processes, which allow to obtain good discolouration yields without high use of oxidizing agents, can be a valid alternative even if they are still under study (Chu et al., 2010; Duan, 2018; El-Ashtoukhy and Amin, 2010; Gökkuş et al., 2018; Moreira et al., 2017; Song et al., 2007; Zhou and He, 2007). Finally, even biological processes can be characterized as a useful tool for the elimination of chromophores, both for more traditional biological microbial biomass reactors (Ajaz et al., 2018; Kapdan and Alparslan, 2005; Maiti et al., 2017; Robinson et al., 2001; Saratale et al., 2009; van der Zee and Villaverde, 2005) and for most innovative reactors that use particular types of fungal cultures. This last method is still the subject of analysis (Blázquez et al., 2018; Dwivedi and Tomar, 2018; Yesilada et al., 2018)

In the (Fig: 1) the most used, or under study, treatments to remove the chromophoric agents that will be treated in this article are reported. They are classified in various sub-categories.



**Figure 1:** Suitable treatments for color removal. (2-column fitting image).



<sup>a</sup>: bias= polarization of the titanium dioxide network

<sup>b</sup>: CAT= catalyst

<sup>c</sup>: WAO= Wet Air Oxidation

<sup>d</sup>: CWAO= Catalytic Wet Air Oxidation

## 2. Chemical / Physical processes

### 2.1. Coagulation / Flocculation

Coagulation / flocculation is one of the most widespread chemical-physical processes for the removal of coloring substances in wastewater (Tab:1) (Asadollahfardi et al., 2018; Mane and Babu, 2011; Pokhrel and Viraraghavan, 2004; Sadri Moghaddam et al., 2010; Satyawali and Balakrishnan, 2008; Zonoozi et al., 2008). The main advantage of traditional chemical-physical processes is that, when the dye can be removed, it comes completely eliminated from the wastewater by physical means and not simply partially decomposed as can sometimes happen with chemical methods (Mane and Babu, 2011).

The process can be divided into three phases: coagulation, flocculation and sedimentation. The first consists of a chemical process that aims to destabilize the colloidal suspensions by neutralizing their charge. The second, or flocculation, is a physical process and allows the smaller particles to be grouped (Butani and Mane, 2017; Mane and Babu, 2011; Xiaoxu et al., 2017) so that they can be easily separated from the water during the third and final phase, ie the sedimentation (Golob et al., 2005).

For example (Tab: 1), cases of color removal efficiencies of up to 99%, starting from synthetic waters containing separately Brilliant Green e Congo Red, are reported (Mane and Babu, 2011). The (Zonoozi et al., 2008) study instead came to remove the dye Acid Red 398 up to 80%.

## **2.2. Adsorption**

### **2.2.1. Adsorption on Activated Carbon**

This is another chemical/physical method commonly used for the removal of dyes (Mohammad Razi et al., 2017; Xiaoxu et al., 2017). It is very effective for adsorbing cationic, mordant and acid dyes and, to a lesser extent, dispersed, direct, and reactive dyes. The performances depend mainly on the type of active carbon used and the characteristics of the treated wastewater (Malik, 2004; Pokhrel and Viraraghavan, 2004; Robinson et al., 2001; Satyawali and Balakrishnan, 2008). For example, starting from a synthetic water colored with Congo Red, (Purkait et al., 2007) showed that it was possible to remove up to 90% of the color using an activated carbon concentration of 1 g/L.

To reduce the costs related to the regeneration of the CA there are actually two different approaches. The first consists in using low cost adsorbents that can therefore be used in a "disposable" manner. This however shifts the problem of the presence of dye molecules from the liquid phase to a solid phase. The second approach consists in the study of alternative

regeneration processes that reduce existing operating costs (Mohammad Razi et al., 2017; Purkait et al., 2007; Robinson et al., 2001).

### **2.2.2. Adsorption on other materials**

The use of these substrates such as clay, peat, coconut shells, zeolite, silica beads, sawdust, wood chips for the removal of color is essentially beneficial for two reasons: great ease with which these materials can be found and the low cost associated with them (Crini, 2006; Mashkoo et al., 2018; Mohammad Razi et al., 2017; Pokhrel and Viraraghavan, 2004; Robinson et al., 2001; Satyawali and Balakrishnan, 2008; Sia et al., 2017).

For example sawdust, like many agricultural and industrial by-products, can be considered as low-cost adsorbents as they are present in large quantities and can be used without the need for special treatments (Mashkoo et al., 2018). In the study conducted by (Mashkoo et al., 2018), the sawdust produced from a plant native to tropical countries (*Tectona grandis*) was used as an adsorbent for the removal of Crystal violet. Removal rates greater than 94% were obtained after 180 minutes of treatment at pH 7.5.

Among the disadvantages, for instance in case wood chips are used as alternative adsorbent, (Robinson et al., 2001) mention the long contact time necessary to obtain satisfactory removal rates. Advantages, in addition to the significantly low cost, are related to the possible subsequent combustion of the chips used as adsorbent substance.

### **2.3. Membrane filtration**

Membrane filtration is an alternative physical treatment that can be used to remove color from wastewater (Tab: 1) (Ahmad et al., 2017; Erkanlı et al., 2017; Pokhrel and Viraraghavan, 2004; Robinson et al., 2001; Satyawali and Balakrishnan, 2008).

It has some advantages such as: appreciable resistance to temperature, good resistance to adverse chemical environment and excellent performance of color removal. Also worthy of note are disadvantages, among which the one linked to the high cost of disposal of the remaining concentrated residue following the separation, the high management costs and the inability to deal with high flow rates (Crini, 2006; Robinson et al., 2001). For example (Erkanlı et al., 2017) have studied the effectiveness of ultrafiltration (UF) on real wastewater of textile company obtaining percentages of color removal up to 90%. (Pokhrel and Viraraghavan, 2004) have instead obtained color removal efficiencies, from real wastewater, between 98% and 100% respectively using ultrafiltration membranes (UF) and nanofiltration (NF).

**Table 1:** Applications of chemical/physical processes for color removal

CHEMICAL / PHYSICAL PROCESSES								
Process	Ind. origin	Type <sup>a</sup>	Dye	Operating conditions			Color removal [%] (other removal [%])	References
				pH	t <sub>r</sub> <sup>b</sup> [min]	Other <sup>c,d</sup>		
Coag/Flocc	Textile/dyes	S	Congo red	6	30	C <sub>0</sub> =500 mg/L	80 - 96	(Vijayaraghavan et al., 2017)
			Direct blue	4	30	C <sub>0</sub> =400 mg/L	79 - 90	
						C <sub>flocculant</sub> =1-6 mL/100mL		
	Textile	S	Reactive brilliant blue XBR	8 - 9	65	PAC= 70mg/L	75 - 76	(Xiaoxu et al., 2017)
	Textile/dyes	S	Acid red 119	1 - 6	65	C <sub>0</sub> =100-200 mg/L	82 - 89	(Butani and Mane, 2017)
			Methyl violet					
			Acid blue 92					
	Textile	R	Various	11 - 12	52 - 172	C <sub>coagulant</sub> = 20-40mg/L	98	(Golob et al., 2005)
						C <sub>flocculant</sub> =2.5-5 mg/L		
	Textile/dyes	S	Brilliant green	6 - 8	77	C <sub>coagulant</sub> = 300-400mg/L	99	(Mane and Babu, 2011)
			Congo red					
	Textile/dyes	S	Acid red 398	4 - 5	57	C <sub>0</sub> = 100 mg/L	60- 80	(Zonoozi et al., 2008)
						C <sub>coagulant</sub> =100-120mg/L		
Adsorption	Textile	S	Methyl violet	-	30	adsorbent= perlite	- <sup>e</sup>	(Crini, 2006)
			Basic blue 9					
	Textile	R	Various	3	150	adsorbent= Mahogany sawdust	65 - 70	(Malik, 2004)
	Textile/dyes	S	Congo red	6.5 - 7	-	C <sub>0</sub> =50-100 mg/L	80 - 90	(Purkait et al., 2007)
						C <sub>AC</sub> = 1g/L		
	Textile/dyes	R	Various	10	120	C <sub>AC</sub> = 2.5 g/L	88	(Popuri et al., 2016)
Filtration	Textile	R	Various	7.5 - 9.5	-	UF (2 bar)	90	(Erkanlı et al., 2017)

					(TOC=85%)		
Textile	S	Direct-15	7	15	UF; C <sub>0</sub> =5-20mg/L	40 - 80	(Ahmad et al., 2017)
Food	R	Various	11	30	Nanofiltration(NF)	81	(Jang et al., 2018)
						(COD=80)	
Paper mill	R	Various	-	-	Ultrafiltration(UF)	93 - 98	(Pokhrel and Viraraghavan, 2004)
			-	-	Nanofiltration(NF)	99 - 100	

<sup>a</sup>: R= real wastewater; S= synthetic wastewater

Synthetic wastewater (S) was created in the laboratory by simulating the hypothetical origin expressed in the apposite column

<sup>b</sup>: t<sub>r</sub>= reaction time

<sup>c</sup>: C<sub>0</sub>= initial concentration

<sup>d</sup>: C<sub>AC</sub>= active carbon concentration

<sup>e</sup>: high adsorption

### 3. Chemical oxidation processes

#### 3.1. Conventional chemical oxidation processes

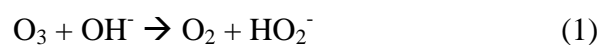
##### 3.1.1. Ozone

Ozone (O<sub>3</sub>) is a powerful oxidant with a fairly high redox potential of 2.08 V. It produces a direct and selective oxidation and reacts preferably with the ionized and dissociated forms of organic compounds (Deng and Zhao, 2015).

Ozone is an unstable gas that has a very limited life (Gerrity et al., 2017). There are several plant alternatives that guarantee a quick and optimal contact between the water to be treated and the ozone (Droste and Gehr, 2018).

In addition, methods are being studied to make ozone production more efficient and less costly. This, for example, acting on the voltage during the production process (Yuan et al., 2017).

In the process the high reactivity of the O<sub>3</sub> in water according to the reaction (1) is exploited (Gerrity et al., 2017).



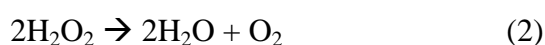
Numerous studies have been carried out on this subject (Tab: 2). With regard to acid dyes, for example, it has been shown that pH, contact time and dye concentration influence the removal of color.

(Ledakowicz et al., 2001) have shown how Acid Blue 40 can be removed up to 98 % with an optimal pH of 7.5. For the Metil Green dye, however, the maximum discoloration (86%) occurs for acidic pH equal to 3 (Kusvuran et al., 2011).

Generally longer contact times favor a more effective decolouring action while, increasing the dye concentration, the contact times necessary for a complete degradation action increase significantly. Conditions of basic or acidic pH allow complete discoloration of the water in less time than necessary in a neutral pH condition (Gerrity et al., 2017; Muthukumar et al., 2004; Perkins et al., 1996). The major disadvantage of using Ozone, however, is certainly the possible formation of toxic by-products (OBPs) starting from the biodegradable substances contained in the wastewater. We can mention the BrO<sub>3</sub> and the nitrosamines (eg. N-nitrosodimethylamine - NDMA) which are known to be possible carcinogens for humans (Bourgin et al., 2018; Droste and Gehr, 2018). The quantity of O<sub>3</sub> to be used must therefore be dosed appropriately (Selcuk, 2005).

### **3.1.2. Hydrogen peroxide**

In order to discolor the wastewater it is also possible to use another oxidizing chemical agent, that is Hydrogen Peroxide. At room temperature, it tends to dissociate according to the reaction expressed in step (2) in water and oxygen. However, peroxide has a high redox potential (1.78 V) which allows it to act as an oxidant against organic molecules, including dyes (Sharma et al., 2011; Slokar and Majcen Le Marechal, 1998).



Sometimes H<sub>2</sub>O<sub>2</sub> alone is not sufficient to discolor particularly colored wastewater in fact over the years different possible combinations to increase its effectiveness have been studied (Hao et

al., 2000). Some concern the introduction of biological enzymes such as peroxidases for the catalyzing of the dissociation reaction reported in point (2). By increasing the temperature, the concentration of peroxidase and maintaining a basic pH of around 9.5, it has been shown that the degree of efficiency of the treatment increases (Slokar and Majcen Le Marechal, 1998). Instead (Ledakowicz et al., 2001) experienced a removal of up to 98% of the Acid Blue 40 dye maintaining a solution pH of 7.5.

(Khatri et al., 2018) studied the catalysis of the reaction by adding zero-valent aluminum (ZVAI). In this case, following the hydrogen peroxide dosage, OH• radicals with high redox potential can be formed (Eq. 3) which increase the speed of discoloration.



### 3.1.3. Chlorine compounds

The discoloration of wastewater is also possible by adding Chlorine compounds that are capable of attacking the amine group present in dye molecules and accelerating the destruction of the nitrogenous bridge (Hao et al., 2000; Slokar and Majcen Le Marechal, 1998; Wu et al., 2016). Precisely for this reason dyes, that have amino groups within their molecular structure (eg dyes derived from some types of sulfonic acids), are among the most easily attacked by chlorine (Omura, 1994; Wu et al., 2016). Chlorine (Cl) is available in both liquid and gaseous or solid state.

When the chlorine is added to the water, the reaction described in point (Eq. 4) is obtained (Droste and Gehr, 2018). However, the hypochlorous acid (HClO) is a weak acid and therefore tends to partially dissociate and form the Hypochlorite ion namely ClO<sup>-</sup> (Eq. 5). The dissociation of HClO is mainly dependent on temperature and pH. (Droste and Gehr, 2018; Hao et al., 2000).



Over the years, the satisfactory use of this methodology to decolorate water from acid and direct dyes has been widely reported. On the other hand, it is less efficient in the removal of reactive dyes and formed by metal complexes where very long contact times are required and it is possible to have residual colouring. Dispersed dyes, on the other hand, are not degraded by chlorine and in particular are totally refractory to the NaClO. It has also been found that the rate of effectiveness in the removal of color increases with increasing chlorine added and decreasing in pH (Slokar and Majcen Le Marechal, 1998).

However, chlorine compounds (except ClO<sub>2</sub>), reacting with the organic substance in the water, can lead to the formation of toxic by-products such as N-nitrosodimethylamine (NDMA) and Trihalomethanes (THMs) such as chloroform and bromoform (Droste and Gehr, 2018; Zhao et al., 2017). Some of these show mutagenic properties and are known to be carcinogenic to humans (Sakarinen, 2016).

Precisely because of potential health and environmental problems, for some years now legislation has imposed a stringent limit on the use of Chlorine in industrial processes and also in the eventual treatment phases of wastewater (Slokar and Majcen Le Marechal, 1998).

In terms of results, (D. Kaur et al., 2018) achieved a 43% color removal from real wastewater. (Baddouh et al., 2018) obtained 100% removal of Rhodamine B after only 90 minutes thanks to the coupling with current in an electrochemical process. Other examples are shown in (Tab. 2).

**Table 2:** Applications of conventional chemical oxidation processes for color removal

CONVENTIONAL CHEMICAL PROCESSES								
Process	Ind. origin	Type <sup>a</sup>	Dye	Operating conditions			Color removal [%] (other removal [%])	References
				pH	t <sub>r</sub> <sup>b</sup> [min]	Other <sup>c</sup>		
O <sub>3</sub>	Dyes	R	Various	-	60	-	> 60	(Bessegato et al., 2018)
	Baker's yeast	R	Various	3 - 7.5 - 9 - 11	-	T= 25-45°C	96 - 98 (COD=56)	(Balcioglu and Gonder, 2018)
	Paper	R	Various	-	-	-	81 (COD=81)	(Tabinda et al., 2017)
	Textile/dyes	S	Methylene blue	3.3 - 5.7	30	-	> 99	(Ogata et al., 2018)
			Acid orange 7	3.5 - 6.3	70	-	> 99	



Coffee	R	Various	-	-	-	85	(Takashina et al., 2018)	
Textile	S	Various	9	-	abs=0.248	90	(Azbar et al., 2004)	
						(COD=92)		
Textile/dyes	S	Direct yellow 4	5 - 6	3.2 - 11	-	95	(H. Shu and Chang, 2005a) <sup>d</sup>	
		Acid black 1						
		Acid orange 10						
		Acid red 14						
		Acid red 18						
		Acid yellow 17						
Food	R	Various	11	30	O <sub>3</sub> = 50mg/L	34	(Jang et al., 2018)	
						(COD=27)		
Textile/printing	S	Red RB	10	30	COD=54-169mg/l	95 - 99	(Sarayu et al., 2007) <sup>d</sup>	
		Remazol red						
		Remazol blue						
		Remazol black						
		Remazol violet						
		Remazol orange						
		Golden yellow						
		Remazol yellow						
Petrol	S	Ortho-Toluidine	11	120	O <sub>3</sub> = 0.5L/min	(COD=57)	(Shokri et al., 2016)	
Textile/dyes	S	Reactive red 198	6 - 10	40	T= 25°C; O <sub>3</sub> = 0.25g/h	> 80	(Karami et al., 2016)	
Textile	S	Acid red 151	7 - 13	30	-	98 - 100	(Erol and Özbelge, 2008) <sup>d</sup>	
		Remazol brilliant blue R						
Dyes	S	Malachite green	3	10	-	86	(Kusvuran et al., 2011) <sup>d</sup>	
						(TOC=10)		
Textile/dyes	S	Congo red	8.5	6	C <sub>0</sub> = 300mg/L	> 99	(Khadhraoui et al., 2009)	
					O <sub>3</sub> = 2.7g/h			
Textile	S	Acid blue 40	7.5	60	O <sub>3</sub> = 100mg/L	98	(Ledakowicz et al., 2001)	
Textile	S	Reactive red 195	11	2	-	85 - 100	(Perkins et al., 1996)	
		Reactive blue 221						
		Reactive yellow 145						
H <sub>2</sub> O <sub>2</sub>	Textile/dyes	S	Methyl orange	-	360	C <sub>0</sub> = 25mg/L; H <sub>2</sub> O <sub>2</sub> alone	0	(Beheshtkhoo et al., 2018)
						H <sub>2</sub> O <sub>2</sub> + iron nanoparticles	> 90	
Textile	S	Acid Blue 40	7.5	-	H <sub>2</sub> O <sub>2</sub> = 2mL/L	98	(Ledakowicz et al., 2001)	
Cl <sub>2</sub>	Textile/dyes	S	Reactive red 2	7	10	Cl <sub>2</sub> = 5mg/L; chlorine alone	40	(Wu et al., 2016)
						Cl <sub>2</sub> = 5mg/L; chlorine + UV	80	
Textile	R	Various	4 - 6	10	Ca(ClO) <sub>2</sub> = 1mg/L	95	(Massoudinejad et al., 2015)	
						(COD=65)		

<sup>a</sup>: R= real wastewater; S= synthetic wastewater

Synthetic wastewater (S) was created in the laboratory by simulating the hypothetical origin expressed in the apposite column

<sup>b</sup>: tr= reaction time

<sup>c</sup>: abs= absorbance

<sup>d</sup>: some information on this case study is also reported in (Sharma et al., 2011).

## **3.2. Advanced chemical oxidation processes**

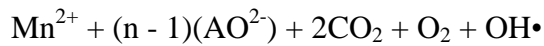
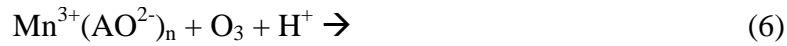
### **3.2.1. Ozone-based**

They are processes that exploit the combined action of O<sub>3</sub> and the hydroxyl radicals that are developed (Munter, 2001; Sharma et al., 2011). Unlike the O<sub>3</sub>, •OH radicals tend to attack not only the ionic and dissociated forms of the organic substance but also the neutral ones (Deng and Zhao, 2015) being endowed with a higher redox potential.

#### **3.2.1.1. O<sub>3</sub> / CAT**

Using homogeneous or heterogeneous catalysts is one of the possible opportunities for accelerating ozonation reactions. Studies that have considered the use of metal oxides and ions, such as Fe<sup>2+</sup>, Al<sub>2</sub><sup>3+</sup>-Me, MnO<sub>2</sub>, TiO<sub>2</sub>-Me, Fe<sup>2+</sup>, Fe<sup>3+</sup>, Mn<sup>2+</sup> and nZVI (zero valent iron nanoparticles), have been numerous and detailed to date (Tab: 3). It must be said, however, that in many cases the complete mechanism that leads to the catalysis of reactions still remains not completely clear (Boczkaj and Fernandes, 2017; Erol and Özbelge, 2008; Malik et al., 2018; Munter, 2001; Ogata et al., 2018; Pachhade et al., 2009; Raman and Kanmani, 2016; Selcuk, 2005; Zhuang et al., 2018).

Taking the case of the Manganese ion (Mn<sup>2+</sup>) as an example, we could see how, combined with Oxalic Acid and joined to the Ozone to form a system Mn<sup>2+</sup> / (COOH)<sub>2</sub> / O<sub>3</sub>, the oxidation of the organic substance proceeds more quickly. This presumably following the formation of OH• radicals originating from the reaction between the Mn<sup>3+</sup> complex, formed as a result of pH > 4 conditions, and ozone (Eq: 6) (Andreozzi, 1999; Asghar et al., 2015; Hilles et al., 2018; Wu et al., 2008).



Also  $\text{Fe}^{3+}$  can be used to catalyze the oxidation reaction of the chromophores. It has been shown that, by combining it with  $\text{O}_3$  for a pH of around 4, it actually improves the discolouration efficiency considerably, obtaining percentages of removal of reactive dyes up to 100% (Wu and Ng, 2008).

Studies in most cases are still ongoing (Malik et al., 2018; Ogata et al., 2018; Wen et al., 2018). Take into account that the effectiveness of the action of each catalyst must be evaluated on every single wastewater in a case-by-case perspective. For example, (Malik et al., 2018) obtained, by alternatively using  $\text{Fe}^{2+}$  and zero valent iron nanoparticles (nZVI), percentages of color removal up to 80 %. Examples of  $\text{O}_3/\text{CAT}$  systems are also reported, such as  $\text{Fe}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Al}_2\text{O}_3$ , where however the application of catalysts did not lead to a significant improvement in coloration of treated water compared to those subjected to traditional treatments with  $\text{O}_3$  (Erol and Özbelge, 2008; Selcuk, 2005). In some situations the introduction of an incorrect catalyst can even lead to an increase in operating costs. The efficiency of removing the color of the wastewater does not improve and it is also necessary to cover the costs of buying the catalyst and disposal of the chemical sludge produced (Selcuk, 2005).

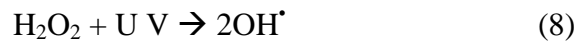
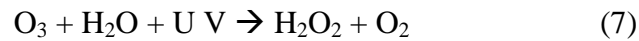
### 3.2.1.2. $\text{O}_3$ / UV

Among the most widely used treatments for discoloration of wastewater, the process that involves the combined use of Ozone and UV rays certainly stands out (Tab: 3). The aim is always to rapidly stimulate the production of hydroxyl radicals and to have a non-selective degradation of the organic substance present in the wastewater. Over the years, a considerable number of experiments, which have demonstrated the effectiveness of the treatment to remove

even highly concentrated dyes in wastewater (including textiles), have been completed (Azbar et al., 2004; Bessegato et al., 2018; H. Shu and Chang, 2005a; Wu, 2008; Wu and Ng, 2008).

For example, (Bessegato et al., 2018) obtained 75% color removal from a real wastewater of a dye-producing company after a reaction time of 60 minutes. Instead (Wu, 2008) has almost completely removed the Reactive Red 2 (>98%) from a synthetic wastewater with a pH of 7.

In the process O<sub>3</sub>/UV the ultraviolet radiation stimulates the production of hydrogen peroxide (Eq. 7) which in turn, again thanks to the UV radiation, will tend to split off forming radicals •OH (Eq. 8) (Deng and Zhao, 2015).



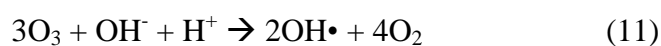
Maintaining a slightly basic pH it is possible to make sure that the •OH radicals can be generated through three different paths (Deng and Zhao, 2015):

1. ozonation at high pH (Eq. 11)
2. thanks to the presence of O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> (Eq. 13 and Eq. 14)
3. photolysis of Hydrogen peroxide (Eq. 8)

Ozone quickly absorbs ultraviolet radiation with a wavelength of 254 nm. Common mercury lamps generate more than 80% of the UV radiation at this wavelength. However, when ozone is subject to UV, before producing hydroxyl radicals, it generates H<sub>2</sub>O<sub>2</sub> which must itself be subject to photolysis. At first glance it would therefore seem more convenient to subject hydrogen peroxide directly to photolysis. However, it shows a low molecular absorption at the wavelength of 254 nm and this limits the yield of •OH. Molecular absorption of UV by Hydrogen peroxide can be increased by using lamps with emissions at lower wavelengths (Munter, 2001).

### 3.2.1.3 O<sub>3</sub> / basic pH

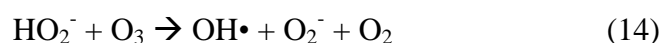
As the pH increases, the rate of decomposition of ozone in water increases. The reaction between the hydroxide ion and ozone leads to the formation of superoxide radical anions O<sub>2</sub><sup>•-</sup> and HO<sub>2</sub><sup>•</sup> (Eq. 9). O<sub>3</sub><sup>•-</sup> forms from the reaction of the ozone with the super-oxide radical anions, which decomposes immediately giving rise to •OH (Eq. 10). Overall, two hydroxyl radicals can be produced every three ozone molecules (Eq. 11) (Munter, 2001).



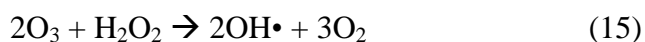
(Boczkaj and Fernandes, 2017) have shown, for example, that real wastewater from wineries is bleached over 80% with a contact time of 60 minutes and an alkaline pH of 9.

### 3.2.1.4. Peroxone

Saying Peroxone is equivalent to referring to the ozone and hydrogen peroxide mixture (O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>) (Munter, 2001). First of all it should be considered that in particular conditions ozone will tend to transform itself producing hydroxyl radicals (Eq. 12). The peroxide is able to split up forming hydroperoxide or HO<sub>2</sub><sup>-</sup> (Eq. 13). This in turn reacts with ozone to form the radicals •OH (Eq. 14) (Deng and Zhao, 2015).



The •OH can be produced following different possible routes. In fact, the combination of the different reactions shows that two molecules of O<sub>3</sub> allow to produce two hydroxyl radical molecules (Eq. 15) (Munter, 2001).



In terms of removal, for example, the results of (Wu, 2008) studies can be cited. In this case the Reactive Red dye has been removed to 99% in pH conditions of 7.

### **O<sub>3</sub> / H<sub>2</sub>O<sub>2</sub> / UV**

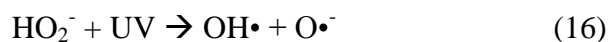
The addition of H<sub>2</sub>O<sub>2</sub> to the process O<sub>3</sub> /UV allows to accelerate the decomposition of the ozone with the consequent increase of the production rate of •OH (Bessegato et al., 2018; Boczkaj and Fernandes, 2017; Munter, 2001). This is because the amount of peroxide increases compared to what would normally be available if you simply implemented a process of type O<sub>3</sub>/UV (Eq. 7).

It has been experimentally proved that the removal yields of the coloring substances, especially the nitrogenous ones, are higher if an O<sub>3</sub>/UV/H<sub>2</sub>O<sub>2</sub> process is used compared to an O<sub>3</sub>/UV (Bessegato et al., 2018; Lucas et al., 2010; Silva et al., 2009; Wu and Ng, 2008). For example, (Bessegato et al., 2018) have shown that, in 60 minutes of reaction, a particular real wastewater is discolored at 80-85% against only 75% that would be obtained if an O<sub>3</sub>/UV treatment was adopted.

### **3.2.2. H<sub>2</sub>O<sub>2</sub>-based**

#### **3.2.2.1. H<sub>2</sub>O<sub>2</sub> / UV**

Direct photolysis of hydrogen peroxide leads to the formation of hydroxyl radicals (Eq. 8). Also HO<sub>2</sub><sup>-</sup>, which is in acid-base equilibrium with H<sub>2</sub>O<sub>2</sub> (Eq. 13), absorbs ultraviolet radiation, especially the one with a wavelength of 254 nm. Therefore, it is able to produce further hydroxyl radicals (Eq. 16) (Deng and Zhao, 2015; Munter, 2001; Muruganandham and Swaminathan, 2006b; Shireesha et al., 2017).



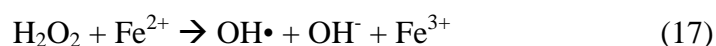
The H<sub>2</sub>O<sub>2</sub>/UV processes are successfully used to destroy dyes (Tab: 3) (Neamtu et al., 2002; Patel et al., 2013; Shu, 2006; H.-Y. Shu and Chang, 2005; Wu, 2008) but also chlorophenols and other chlorinated compounds. (Rosa et al., 2015) showed how it was possible to remove up to 99% of the color from a composite solution of dyes (Reactive Yellow 145, Reactive Orange 122, Reactive Red 239, Reactive Blue 222 and Reactive Blue 21). In another case, Reactive Red 120, Reactive Black 5 and Reactive Yellow 84 dyes have also been successfully removed (up to 99%) under pH conditions around 7 (Neamtu et al., 2002).

Good disinfectant power is also highlighted. As for the previous treatments, the operating cost depends on the type and concentration of the present dye, wastewater flow rate to be treated, removal efficiency required (Munter, 2001).

### 3.2.3. Fenton related

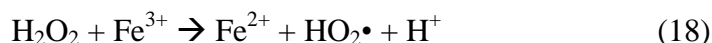
#### 3.2.3.1. Fenton

The Fenton reagent is an extremely strong oxidizing reagent that generates hydroxyl radicals thanks to the addition of ferrous ion Fe<sup>2+</sup> and hydrogen peroxide (Eq. 17) (Belalcázar-Saldarriaga et al., 2018; Cetinkaya et al., 2018; Ertugay and Acar, 2017; Forsey, 2004; Sharma et al., 2011).



$$k = 53\text{-}76 \text{ M}^{-1} \text{ s}^{-1}$$

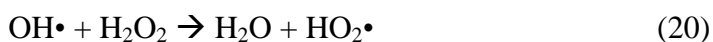
The reaction is rapidly propagated thanks to the regeneration of the ferrous ion given by the reduction of Fe<sup>3+</sup> ions by hydrogen peroxide (Eq. 18) (Belalcázar-Saldarriaga et al., 2018; Peralta-Hernández et al., 2018; Ting et al., 2009).



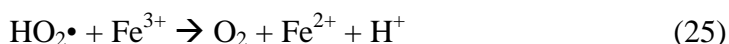
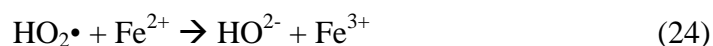
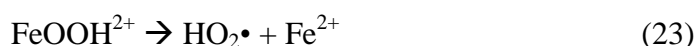
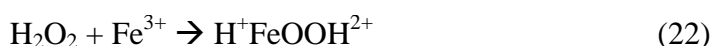
$$k = 0.01 \text{ M}^{-1} \text{ s}^{-1}$$

Once formed, the hydroxyl radicals are able to quickly attack the chromophores (Eq. 19), and the organic substance more generally, and cause its consequent decomposition through the abstraction of the hydrogen atoms and the addition to the unsaturated C-C bonds. In the

meantime other competitive reactions involving  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{H}_2\text{O}_2$ ,  $\text{OH}\cdot$  ed  $\text{HO}_2\cdot$  intervene. Therefore the hydroxyl radicals can be eliminated by reacting with  $\text{Fe}^{3+}$ ,  $\text{H}_2\text{O}_2$  (Eq. 20) or simply reacting with each other (Eq. 21) (Lucas and Peres, 2006; Sharma et al., 2011).



Within the process then other reactions, which are listed below, intervene (from Eq.22 to Eq.25) (Forsey, 2004; Muruganandham and Swaminathan, 2004).



The  $\cdot\text{OH}$  themselves can terminate the reaction by transforming the ferrous ion into ferric ion (Eq: 26) without forming further hydroxyl radicals (Forsey, 2004; Peralta-Hernández et al., 2018):



$$k = 3 * 10^8 \text{ M}^{-1} \text{ s}^{-1}$$

According to previous studies, the use of  $\text{Fe}^{2+}/\text{H}_2\text{O}_2$  as a bleach for wastewater is generally appealing for four reasons (Munter, 2001):

1. Iron is an abundant material
2. Its use does not produce toxic by-products
3. Hydrogen peroxide is easy to use
4.  $\text{H}_2\text{O}_2$  does not cause particular problems in the environment

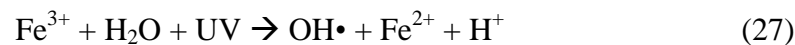


However, as can be seen from the constants  $k$  of the reactions (17) and (18) the ferrous ion is consumed much more rapidly than it is produced (Forsey, 2004). From this derives an important production of ferric hydroxide which is formed in the subsequent neutralization phase. This material must be disposed of as chemical sludge with a consequent increase in costs (Ting et al., 2009).

For example, in terms of results the study of (Ertugay and Acar, 2017) can be cited. It demonstrates the removal of color caused by Direct Blue 71 up to 80% in just 20 minutes of contact under pH conditions of 3. (Zaharia and Dartu, 2010), in the presence of pH equal to 5, have removed 90% of Orange 16 in just 5 minutes of contact time.

### 3.2.3.2. Photo-Fenton

In addition to the reactions listed above, in the Photo-Fenton process hydroxyl radicals are also formed as a result of the use of UV rays (Eq. 8 and 27) (Cetinkaya et al., 2018; Lucas and Peres, 2006; Sharma et al., 2011).



The use of UV rays is therefore an excellent ally in the degradation processes of chromophores because it stimulates the production of radicals  $\cdot\text{OH}$  (Dang et al., 2017; Lucas and Peres, 2006).

Ultraviolet rays not only produce more hydroxyl radicals but also allow the  $\text{Fe}^{3+}$  to be re-transformed into  $\text{Fe}^{2+}$  (Sharma et al., 2011). In this way the concentration of ferrous ion is increased and this allows to further speed up the reaction and reduce the consumption of  $\text{Fe}^{2+}$  (Dang et al., 2017; Peralta-Hernández et al., 2018; Sharma et al., 2011). The rate of removal of the chromophores is strictly dependent on the concentration of the dye, of the reagents  $\text{Fe}^{2+}$  and  $\text{H}_2\text{O}_2$  and it can be considered as maximum in the conditions of pH equal to 3 (Muruganandham and Swaminathan, 2004; Sharma et al., 2011).

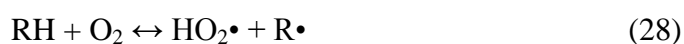
For example, (Lucas and Peres, 2006) obtained, due to this pH condition, a color removal of Reactive Black 5 up to 98%. (Muruganandham and Swaminathan, 2004) obtained the same result for the Reactive Orange 4 dye, always with a pH operating condition of 3.

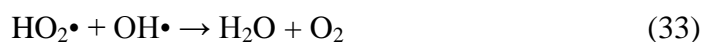
### 3.2.4. Wet Air Oxidation

#### 3.2.4.1. Conventional WAO

The wet air oxidation process (WAO) can be applied to wastewater (Luan et al., 2017) or solid matrices (Collivignarelli et al., 2018). It involves the presence of air or pure oxygen (O<sub>2</sub>), at particular temperature conditions [T= 150-350 °C], pressure [P= 60-250 bar], and wastewater that must be treated in order to eliminate chromophore substances. Generally the term "wet" is used because the water in these particular conditions of pressure and temperature remains in the liquid state optimizing the contact between substrate and oxidizing agent (Kayan et al., 2010; Zhou and He, 2007).

At first glance it seems that only the increase in redox potential of the O<sub>2</sub> due to the increase in temperature is exploited. However, in the Wet Air Oxidation process the degradation mechanism that occurs is to be considered decidedly more complex. Despite this, a considerable number of studies agree that most of the degradation occurs as a result of the formation of free radicals •OH according to the traditional start, propagation and term scheme. Numerous reactions have been proposed as significantly representative of what happens to an organic compound (RH) (from Eq. 28 to Eq. 33) (Rodríguez et al., 2009; Zhou and He, 2007).





The efficiency of the production of free radicals, and therefore the rate of color removal, depends mainly on the temperature and the amount of oxygen present (Luan et al., 2017; Zhou and He, 2007).

The results then also vary according to the type of chromophore to be eliminated. The color, caused by the presence of Acid Red 97, has been brilliantly removed up to 98% with  $T = 150\text{--}200^\circ\text{C}$  (Kayan et al., 2010) while the removal of azo dyes as Red X- GRL, with  $T = 180^\circ\text{C}$ , stops at 47% (Zhou and He, 2007). This is because azo dyes have very complex aromatic structures which make their degradation particularly difficult (Luan et al., 2017; Tanaka et al., 2000; Zhou and He, 2007).

#### **3.2.4.2. CWAO**

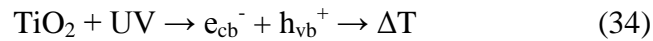
Examples of CWAO (Catalytic Wet Air Oxidation) are also reported. Catalytic complexes, such as Cu/CNFs (Carbon nanofibers) or AC (Activated Carbon), have been adopted to increase the efficiency of removing the most recalcitrant dyes (Tab: 3) (Rodríguez et al., 2009; Santos et al., 2007). For example, activated carbon was used as a catalytic complex for the removal of three different dyes ( Orange G, Brilliant Green, Methylene Blue) in three distinct solutions obtaining the removal of 80-100% of dyes (Santos et al., 2007).  $\text{LaNiO}_3$  was instead added as a catalyst in order to remove the Reactive Black 5 obtaining up to 90% removal (Palas et al., 2018).

#### **3.2.5. $\text{TiO}_2$ / UV**

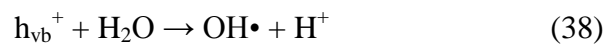
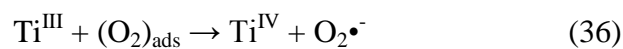
In the field of wastewater color removal, a process, that has aroused considerable interest especially in recent years and which is part of the AOPs, is the Photocatalysis with  $\text{TiO}_2$  and UV radiation in the presence of oxygen. This is due to the high photocatalytic activity and the evident stability of titanium dioxide to remove organic substances from the water (Tab: 3) (Arcanjo et al., 2018; Gupta et al., 2011; Harun et al., 2018; Islam Molla et al., 2017;

Muruganandham and Swaminathan, 2006a). The process is based on the exploitation of the photoinduced reactivity of titanium dioxide which allows the formation of highly oxidizing species such as hydroxyl radicals  $\bullet\text{OH}$ . In fact, photocatalysis can be framed as a Hydroxyl Radical-based process because the aim is nevertheless to produce hydroxyl radicals  $\bullet\text{OH}$  with high redox potential (Eskandarloo et al., 2016; Turolla et al., 2012). The process begins when photons produced by UV irradiation, carrying enough energy to bridge the  $\text{TiO}_2$  bandwidth, hit the catalytic surface. Usually it is 3.2 eV for anatase and 3.0 eV for rutile namely a wavelength of light radiation below 390 nm (Chong et al., 2010; Ghalebizade and Ayati, 2016; Miklos et al., 2018; Turolla, 2014).

At the same time, the promotion of electrons from the valence band to the conduction band is then stimulated by generating what are called lacuna-electron pairs. However, most of the electrons recombine immediately, releasing a portion of thermal energy. In fact this behavior represents the main limiting agent of the photocatalyst activity (Eq. 34) (Turolla, 2014).



In the meantime, the process continues with oxidation-reduction reactions involving electrons and electronic gaps in the phase of displacement on the surface. To enter in more detail, the titanium (IV) is reduced to titanium (III) (Eq. 35) thanks to the presence of the electrons of the conduction band. Titanium (III) in turn is able to react with oxygen (electron acceptor) to form superoxide anion radicals  $\text{O}_2^{\bullet-}$  (Eq. 36). Instead, the electronic holes  $h_{\text{vb}}$  react following two possible ways to obtain the hydroxyl radicals: oxidizing hydroxide ions (Eq. 37) or water molecules (Eq. 38) (Kistan et al., 2018; Tanaka et al., 2000; Turolla, 2014).



In some cases, the electronic gaps can participate directly in the oxidation of organic substances (Eq. 39) (Turolla, 2014).



Due to the limited life time of the species involved in the formation of hydroxyl radicals, the adsorption of the reagents on the titanium dioxide surface is the fundamental point for the reactions described above. In this way, water molecules, ions and organic compounds can easily react with each other (Turolla, 2014).

Several types of titanium dioxide ( $\text{TiO}_2$ ) photoreactive have been synthesized over the years. Basically they can all be classified into two categories: the particle and the immobilized ones. The substantial difference is in their structure and above all in their mode of use. The  $\text{TiO}_2$  in particles is in suspension phase and is usually used in reactors with an adequate and significant level of mixing. Instead the immobilized  $\text{TiO}_2$  consists of a solid substrate allocated inside a reactor (Turolla, 2014). Each system has strengths and weaknesses. Certainly the necessity of having to perform a separation treatment immediately after use is the main disadvantage linked to the use of  $\text{TiO}_2$  in the form of nanopowders. This is obviously to avoid that  $\text{TiO}_2$  nanopowders remain in the wastewater following the treatment (Franz et al., 2015; Turolla, 2014).

Especially in recent years, interest in the study of alternative methods to synthesize titanium dioxide has been increasing (Franz et al., 2016). The usefulness and practicality of adopting systems with immobilized  $\text{TiO}_2$ , which can therefore be used without having to request a post-treatment separation after use, have become increasingly evident (Turolla et al., 2012).

To give some examples we can cite the study of (Arcanjo et al., 2018) which demonstrated the possible removal of color (95%) from a real wastewater coming from a textile company in a contact time of 6 hours with pH equal to 10. Instead (Muruganandham and Swaminathan, 2006a) got 95% removal of Reactive Orange 4 after 60 minutes of contact time with pH equal to 5.

### 3.2.6. Sulfate Radical-based

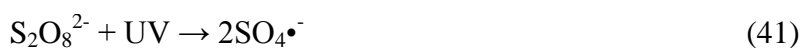
Even the  $S_2O_8^{2-}$  is a powerful oxidant. The redox potential of 2.01 V can be further increased by forming  $SO_4^{\bullet-}$  ( $E^0 = 2.6$  V). This is done by subjecting  $S_2O_8^{2-}$  to a condition that facilitates cleavage in the most reactive molecule, namely in the presence of (Brienza and Katsoyiannis, 2017; Deng and Zhao, 2015; Ishak et al., 2018; Shi et al., 2012; Wang et al., 2014; Yuan et al., 2011):

#### *Heat source*

With temperatures between 35 and 130 °C

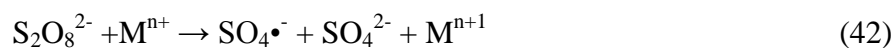


#### *Irradiation with UV rays*



#### *Transition metal*

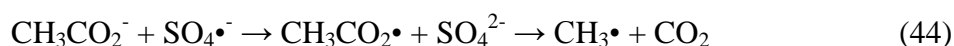
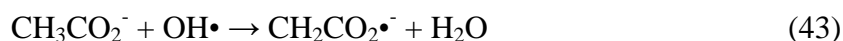
Such as  $Fe^{2+}$



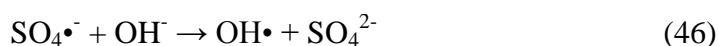
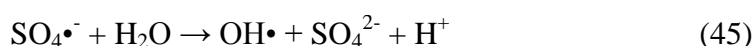
#### *Basic pH*

Although in this case the mechanisms of transformation of the Persulfate are not yet completely clear.

Exactly like hydroxyl radicals, sulfatic radicals  $SO_4^{\bullet-}$  are highly reactive species but have a decidedly short life span. It is the mechanism of action that changes.  $\bullet OH$  preferably attack double C=C bonds or untie the hydrogen atom from the C-H bond (Eq. 43). Sulphatic radicals, on the other hand, act differently. They remove electrons from organic molecules that in this way are transformed into radical organic cations (Eq. 44) (Deng and Zhao, 2015; Forsey, 2004; Ishak et al., 2018).



It should be noted that, starting from sulphonic radicals, it is possible to obtain hydroxyl radicals (Eq. 45 and Eq. 46) (Deng and Zhao, 2015).



For example, (Wang et al., 2014) obtained up to 90% removal of the Acid Orange 7 dye from synthetic waters thanks to the combined use of ultrasound and  $\text{Fe}^0$  in just 20 minutes of reaction time. Studies have also been carried out on the influence of chloride ion in the removal of color from wastewater using Sulfate Radical-based AOPs. If present in high concentrations, it can inhibit the action of discoloration (role of scavenger) and eventually lead to the development of a greater quantity of chlorinated compounds (Wang et al., 2014; Yuan et al., 2011).

Table 3: Applications of advanced chemical processes for color removal

ADVANCED CHEMICAL PROCESSES								
Process	Ind. origin	Type <sup>a</sup>	Dye	Operating conditions			Color removal [%] (other removal [%])	References
				pH	t <sub>r</sub> <sup>b</sup> [min]	Other <sup>c d e f g h i</sup>		
O <sub>3</sub> /CAT	Textile/dyes	R	Various	10	–	CAT=Perfluorooctyl alumina C <sub>CAT</sub> = 300g/L	75	(Ogata et al., 2018)
	Textile/dyes	S	Reactive red 2 Acid orange 6	–	120	CAT=MnO <sub>2</sub> C <sub>CAT</sub> = 3g/L	> 90	(Ogata et al., 2018)
	Textile/dyes	R	Various	6.5	40	CAT=Fe <sup>2+</sup> ; C <sub>CAT</sub> = 1-6g/L	50 - 70	(Malik et al., 2018)
				6.5	40	CAT=nZVI; C <sub>CAT</sub> = 1-6g/L	50 - 85	
	Waste dump	R	Various	4	180	CAT=ZnSO <sub>4</sub> C <sub>CAT</sub> = 1g/6g (COD <sub>0</sub> /Zn)	99 (COD=90)	(Amr et al., 2017)
	Textile/dyes	S	Acid red 18	–	6-25	CAT=Ca(OH) <sub>2</sub> C <sub>CAT</sub> = 2-3g/L	100 (TOC=100)	(Quan et al., 2017)
	Textile	R	Various	10	–	CAT=Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> and FeSO <sub>4</sub>	50 - 60 <sup>l</sup>	(Sharma et al., 2011)
	Textile	S	Reactive red 2	4 - 7 - 10	60	CAT=Fe <sup>3+</sup>	98 - 100	(Wu and Ng, 2008) <sup>m</sup>
	Dyes	S	Reactive red 2	4 - 7 - 10	60	CAT=Fe <sup>3+</sup>	95	(Wu, 2008) <sup>m</sup>
	Textile	S	Acid red-151 Remazol brilliant blue R	2.5 - 7 - 13	30	CAT=Al <sub>2</sub> O <sub>3</sub> and PFOA	54 - 98	(Erol and Özbelge, 2008) <sup>m</sup>
O <sub>3</sub> /UV	Textile	R	Various	7 - 8.5	2520	CAT= Ru/AC	(TOC=75)	(Wang et al., 2009) <sup>m</sup>
	Dyes	R	Various	–	60	–	> 75	(Bessegato et al., 2018)
	Coffee	R	Various	–	–	–	99	(Takashina et al., 2018)

	Textile/dyes	S	Direct yellow 4 Acid black 1 Acid orange 10 Acid red 14 Acid red 18 Acid yellow 17	–	4 - 11	–	> 95	(H. Shu and Chang, 2005a) <sup>mm</sup>
	Textile	S	Reactive red 2	4 - 7 - 10	60	–	97 - 99	(Wu and Ng, 2008) <sup>mm</sup>
	Petrol	S	Ortho-Toluidine	11	120	O <sub>3</sub> = 0.5L/min	(COD=70)	(Shokri et al., 2016)
	Dyes	S	Reactive red 2	4 - 7 - 10	60	–	98 - 100	(Wu, 2008) <sup>mm</sup>
	Textile	R	Various	9	–	abs= 0.248	93	(Azbar et al., 2004)
							(COD=94)	
O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>	Petrol	S	Ortho-Toluidine	9	120	O <sub>3</sub> = 0.5L/min H <sub>2</sub> O <sub>2</sub> = 40mM	(COD=66)	(Shokri et al., 2016)
	Textile/dyes	S	Congo red	8.5	6	C <sub>0</sub> = 300mg/L O <sub>3</sub> = 2.7g/h H <sub>2</sub> O <sub>2</sub> = 7.5mM/L	> 90	(Khadhraoui et al., 2009)
	Food	R	Various	11	30	H <sub>2</sub> O <sub>2</sub> /O <sub>3</sub> = 0.3	52	(Jang et al., 2018)
	Textile	S	Reactive red 2	4 - 7 - 10	60	–	98 - 99	(Wu and Ng, 2008) <sup>mm</sup>
	Dyes	S	Reactive red 2	4 - 7 - 10	60	–	99	(Wu, 2008) <sup>mm</sup>
	Textile/dyes	S	Rhodamine B	7	80	C <sub>0</sub> = 100mg/L H <sub>2</sub> O <sub>2</sub> = 100mg/L	38	(Mousavi et al., 2016)
	Textile/dyes	S	Reactive red 198	9	40	T= 25°C; O <sub>3</sub> = 0.25g/h H <sub>2</sub> O <sub>2</sub> = 0.01-0.04mM	> 90	(Karami et al., 2016)
O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> /UV	Dyes	R	Various	–	60	–	80 - 85	(Bessegato et al., 2018)
	Textile	S	Reactive red 2	4 - 7 - 10	60	–	94 - 97	(Wu and Ng, 2008) <sup>mm</sup>
	Petrol	S	Ortho-Toluidine	10	120	O <sub>3</sub> = 0.5L/min H <sub>2</sub> O <sub>2</sub> = 20mM	(COD=83)	(Shokri et al., 2016)
	Dyes	S	Reactive red 2	4 - 7 - 10	60	–	93 - 98	(Wu, 2008)
	Textile	S	Orange II Acid red 27	5 - 9	10	interfering: salt (0-100g/L)	75 - 98 83 - 92	(Silva et al., 2009) <sup>mm</sup>
	Textile	R	Various	3	120	abs= 0.248; O <sub>3</sub> = 2g/h H <sub>2</sub> O <sub>2</sub> = 200mg/L	96	(Azbar et al., 2004)
							(COD=99)	
	Textile	S	Direct black 22	6.5	160	–	93 - 99	(H. Shu and Chang, 2005b) <sup>mm</sup>
H <sub>2</sub> O <sub>2</sub> /UV	Textile/dyes	S	Reactive green 19	6.5	20	–	100	(Holkar et al., 2016)
	Dyes	S	Yellow 28	5 - 7 - 10	90	C <sub>0</sub> = 100-300mg/L H <sub>2</sub> O <sub>2</sub> = 25-100mg/L	98	(Beikmohammadi et al., 2016)
	Textile/dyes	S	Various	7	15	–	92 - 99	(Rosa et al., 2015)
	Textile/dyes	S	Acid black 24	3 - 5.5	–	–	99	(Chang et al., 2006) <sup>mm</sup>
	Textile	S	Reactive red 120 Reactive black 5	6.5 - 7	60	C <sub>0</sub> = 100mg/L	99	(Neamtu et al., 2002) <sup>mm</sup>





						H <sub>2</sub> O <sub>2</sub> = 0.15mmol		
	Dyes	R	Various	3	20 - 30	Fe <sup>2+</sup> = 550mg/dm <sup>3</sup>	> 90	(Szpyrkowicz et al., 2001)
						H <sub>2</sub> O <sub>2</sub> = 600mg/dm <sup>3</sup>		
	Textile	S	Reactive black 5	3	-	T=40°C	> 99	(Meriç et al., 2004)
	Textile	R	Various	5	-	-	94	(Azbar et al., 2004)
							(COD=96)	
	Textile	R	Various	3	-	T=40°C	> 40	(Pérez et al., 2002)
	Textile	S	Orange 16	5	5	T=20°C	> 90	(Zaharia and Dartu, 2010)
H <sub>2</sub> O <sub>2</sub> /Fe <sup>2+</sup> /UV	Dyes	S	Carmoisine	3.5	12	C <sub>0</sub> = 20mg/L	95	(Sohrabi et al., 2017)
						Fe <sup>2+</sup> = 0.0125mmol		
						H <sub>2</sub> O <sub>2</sub> = 0.3mmol		
	Dyes	S	Acid orange 8	3	60	Fe <sup>2+</sup> = 2*10 <sup>-5</sup>	100	(Abo-Farha, 2010)
						H <sub>2</sub> O <sub>2</sub> = 10mM		
	Dyes	S	Acid red 17	3	60	Fe <sup>2+</sup> = 1*10 <sup>-5</sup>	> 90	(Abo-Farha, 2010)
						H <sub>2</sub> O <sub>2</sub> = 5mM		
	Textile/dyes	S	Reactive yellow 14	3 - 5.5	-	-	95	(Sharma et al., 2011)
			Reactive orange 4	3	-	-	98	(Sharma et al., 2011)
			Reactive black 5	3	-	-	98	(Sharma et al., 2011)
			Reactive black B	2	-	-	98	(Sharma et al., 2011)
	Dyes	S	Reactive red 241	4 - 7 - 10	120	-	87 - 99	(Patel et al., 2013)
	Textile	R	Various	3	-	T=40°C	> 70	(Pérez et al., 2002)
WAO	Textile/dyes	S	Red X-GRL	5	180	T=180°C	47	(Zhou and He, 2007) <sup>m</sup>
							(COD<=30)	
	Textile	S	Acid red 97	-	-	T=150 - 200°C	88 - 98	(Kayan et al., 2010) <sup>m</sup>
	Textile	S	Acid orange 7	5.5	180	T= 140°C; p= 8.7bar	24	(Rodríguez et al., 2009)
			Acid orange 74	10.5	180	C <sub>0</sub> = 1g/L	0	
			Direct blue 71	6.5	180		58	
			Reactive black 5	5	180		34	
			Eriochrome blue					
			black B	4	180		97	
CWAO	Textile	S	Reactive black 5	3	120	T=50°C; p=1atm	90	(Palas et al., 2018)
						C <sub>0</sub> = 100mg/L		
						CAT= LaNiO <sub>3</sub>		
	Textile	S	Orange G	-	720	T= 160°C; p= 16bar	80 - 100	(Santos et al., 2007)
			Methylene blue			C <sub>0</sub> = 1g/L		
			Brilliant green			CAT= AC		
	Textile	S	Acid orange 7	5.5	180	T= 140°C; p= 8.7bar	100	(Rodríguez et al., 2009)
			Acid orange 74	10.5	180	C <sub>0</sub> = 1g/L	99	
			Direct blue 71	6.5	180	CAT= Cu/CNF	100	
			Reactive black 5	5	180		100	
			Eriochrome blue					
			black B	4	180		100	

TiO <sub>2</sub> /UV	Textile	R	Various	10	360	–	95	(Arcanjo et al., 2018)
	Textile	S	Reactive orange 4	5	6	–	95	(Muruganandham and Swaminathan, 2006a)
	Various	S	Orange G	2 - 6	57 - 240	–	(COD=64)	(Tanaka et al., 2000)
			Acid orange 7					
			New cocchine					
			Acid black 1					
			Tartrazine					
			Acid yellow 17					
			Congo red					
	Textile	S	Reactive violet 5	4	20	T=25°C	90	(Chung and Chen, 2009)
	Textile/dyes	S	Blue 9	7	120	–	> 99	(Dias and Azevedo, 2009)
			Red 51					
			Yellow 23					
	Textile/dyes	S	Rhodamine B	–	–	UV=365nm	50	(Zhuang et al., 2010)
	Textile/dyes	S	Methylene blue	7	60	C <sub>0</sub> = 20ppm	> 95	(Esparza et al., 2010)
	Dyes	S	Reactive yellow 14	3	–	–	91	(Sharma et al., 2011)
	Textile	S	Tropaeoline 000	10	–	–	(COD=92)	(Sharma et al., 2011)
								(Rao and Venkateswarlu, 2008) <sup>m</sup>
	Textile	S	DASDA	5	60	T= 30°C	(TOC=65-82)	
			ANSDA					
			DNSDA					
Persulfate	Waste dump	R	Various	5 - 6	–	+UV and	94 - 100	(Ishak et al., 2018)
						coagulation/flocculation	(COD=91)	
	Textile	S	Acid orange 7	5 - 8	20	+US, +Fe <sup>0</sup> , +US/Fe <sup>0</sup>	10 - 90	(Wang et al., 2014)
	Textile/dyes	S	Reactive red 198	9	30	T= 70°C; C <sub>0</sub> = 200mg/L	> 99	(Karami et al., 2016)
						persulfate= 12mM		

<sup>a</sup>: R= real wastewater; S= synthetic wastewater

Synthetic wastewater (S) was created in the laboratory by simulating the hypothetical origin expressed in the apposite column

<sup>b</sup>: t<sub>r</sub>= reaction time

<sup>c</sup>: C<sub>0</sub>= initial dye concentration

<sup>d</sup>: CAT= catalyst

<sup>e</sup>: nZVI= zero valent iron nanoparticles

<sup>f</sup>: abs= absorbance

<sup>g</sup>: AC= activated carbon

<sup>h</sup>: λ<sub>UV</sub>= UV wavelenght

<sup>i</sup>: US= ultrasound

<sup>l</sup>: high production of sludge

<sup>m</sup>: some information on this case study is also reported in (Sharma et al., 2011)

## **4. Electrochemical processes**

### **4.1. TiO<sub>2</sub> / UV / bias**

In recent years, the possibility of integrating photocatalysis with an electrochemical process has been repeatedly emphasized. Hence the name Electro photocatalysis that allows the reduction of the recombination of photogenerated electronic spaces (Tab: 4) (Cardoso et al., 2016; Eskandarloo et al., 2016; Garcia-Segura and Brillas, 2017; Turolla et al., 2012).

For example, we can cite the study of (Garcia-Segura and Brillas, 2017) that shows the removal of more than 90% of the color, caused by Methyl Orange, following 180 minutes of treatment with immobilized TiO<sub>2</sub>. The application combined with the O<sub>3</sub> is also interesting. It has been tested for example by (Cardoso et al., 2016). They have obtained the removal of more than 99% of the color of a real wastewater coming from a textile industry.

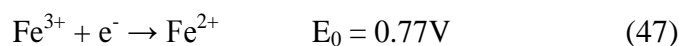
### **4.2. Electro Fenton (EF)**

Recently, due to the high efficiency found and low environmental impact, there is a growing interest (El-Desoky et al., 2010; Ganiyu et al., 2018; Ghoneim et al., 2011; Gökkuş et al., 2018; Iranifam et al., 2011; Mohajeri et al., 2010; Özcan and Özcan, 2018; Rosales et al., 2009; Zhang et al., 2006) in processes based on electrochemical degradation, direct or indirect, of organic pollutants, including chromophores, contained in wastewater (Sharma et al., 2011). Compared to the traditional Fenton process, an electrochemical unit is also applied in the Electro Fenton process. Studies, carried out in recent years on this theme, can be divided into four different categories (Ganiyu et al., 2018; Zhang et al., 2006):

1. Hydrogen peroxide is applied externally while the anode is sacrificed and used as an iron source

2. The ferrous ion and hydrogen peroxide are electro-generated by an anode and a cathode which spreads oxygen
3. The ferrous ion is applied externally while the hydrogen peroxide is generated by a cathode which spreads oxygen
4. Fenton reagents are used to produce hydroxyl radicals in the electrolyte cell while the ferrous ion is regenerated by reducing the ferric ion at the cathode level.

According to previous studies, category 4 is generally the most used and the most studied (Moreira et al., 2017; Ting et al., 2009). In addition to the already explicit reactions, which start when the Fenton reagent is prepared in the electrolytic cell, the reduction of the ferric ion can be observed at the cathode (Eq. 47). This makes it possible to develop new  $\text{Fe}^{2+}$  and thus increase the production of hydroxyl radicals, thereby improving the speed and impact on the degradation of organic substances ( $E_0$  is the standard reduction potential) (Huang et al., 2008; Moreira et al., 2017).



For example, we can cite the study of (Özcan and Özcan, 2018) which demonstrated the total removal of Naphthol Blue Black after 180 minutes of contact time. We can also mention the study of (Rosales et al., 2009) which showed the possible color removal caused by Lissamine Green B, Methyl Orange, Reactive Black 5 and Fuchsin Acid with peaks of 80%.

### 4.3. Anodic Oxidation (AO)

Anodic Oxidation is a valid alternative to previous processes. It has many advantages. In fact, it does not require significant addition of chemical oxidants, particular plant equipment and it does not produce polluting and/or toxic by-products. This makes this process particularly attractive. Over the years the use of different types of anodes (such as graphite, glassy carbon, platinum,

stainless steel and many others) has been tested (Tab: 4) (Bassyouni et al., 2017; Chu et al., 2010; Hamad et al., 2018; Matilainen and Sillanpää, 2010; Sharma et al., 2011).

In this process of electrochemical oxidation, pollutants are destroyed by a process of direct or indirect oxidation. In a process of direct anodic oxidation, pollutants are firstly adsorbed on the surface of the anode and then destroyed by the anodic electron transfer reaction. In an indirect process, strong oxidants such as hypochlorite/chlorine, ozone and hydrogen peroxide are instead electrochemically generated (El-Ashtoukhy and Amin, 2010; Moreira et al., 2017).

Unfortunately today, due to the low stability of the process and the very high operating cost, this system is not yet economically advantageous even though the removal of coloring substances, and of the organic ones in general, is decidedly effective (Chu et al., 2010; Hamad et al., 2018). In fact, for example, a 100 mg/L solution of Acid Brown 14 was successfully bleached (90%) in 60 minutes (Bassyouni et al., 2017).

#### **4.4. Electrocoagulation (EC)**

The process develops in the following way. At the anode, metal cations are generated while at the cathode, atoms of H<sub>2</sub> are also produced with the release of ions OH<sup>-</sup> and radicals •OH. The metal ions, which destabilize the electrical charges of the colloids, enter into solution and aggregate to form flakes which can then be separated from the liquid mass by sedimentation or flotation. Unfortunately, even in this case the studies are still underway and the number of experiments in the removal of color in wastewater is still small. Also the effects that there are on the coupled use of several technologies such as AO/EC (Bassyouni et al., 2017) or O<sub>3</sub> /EC (El-Ashtoukhy and Amin, 2010; Song et al., 2007; Zhou and He, 2007) are under study. The results show a good efficiency in the removal of dyes from wastewater (Adhoum and Monser, 2004; Alinsafi et al., 2005; El-Ashtoukhy and Amin, 2010; Zhou and He, 2007). (Duan, 2018) have shown removal up to 99% of the color caused by Methylene Blue in just 30 minutes of contact

time. Also the applicability of the treatment for paper mill wastewater has been studied obtaining color removals around 100% in 60 minutes and in pH condition equal to 5 (Azadi Aghdam et al., 2016).

**Table 4:** Applications of electrochemical processes for color removal

Process	Ind. origin	Type <sup>d</sup>	Dye	ELECTROCHEMICAL PROCESSES			Color removal [%] (other removal [%])	References
				Operating conditions				
				pH	t <sub>r</sub> <sup>e</sup> [min]	Other <sup>f g h</sup>		
TiO <sub>2</sub> /UV/bias	Textile/dyes	S	Methyl orange	2	150	voltage= 0.75V C <sub>0</sub> = 20mg/L	97	(Garcia-Segura and Brillas, 2017)
	Textile	R	Various	3	60	-	> 50	(Cardoso et al., 2016)
	Dyes	R	Various	-	90	+ O <sub>3</sub>	> 99	(Bessegato et al., 2018)
	Textile	S	Acid orange 7	6.5	180	+ ZnO voltage= 2.5V C <sub>0</sub> =100mg/l	> 80	(Ghalebizade and Ayati, 2016)
EF <sup>a</sup>	Textile	R	Various	3	-	-	> 91	(Gökkuş et al., 2018)
	Textile/dyes	S	Naphthol blue black	3	180	current= 60mA	(TOC>70)	(Özcan and Özcan, 2018)
	Textile/dyes	R	Various	-	90	current= 0.32A Fe <sup>2+</sup> = 0.53mM	90	(P. Kaur et al., 2018)
	Textile/dyes	R	Various	-	-	-	(COD=100)	(Roshini et al., 2017)
	Textile	S	Rhodamine B	2	180	voltage= 8V Fe <sup>2+</sup> = 15mg/L	63	(Jinisha et al., 2018)
	Textile	R	Various	2	180	voltage= 8V Fe <sup>2+</sup> = 15mg/L	(COD=48)	(Jinisha et al., 2018)
	Textile/dyes	S	Reactive Red 120	3	-	-	98	(Rostamizadeh et al., 2018)
	Paper	R	Various	-	-	-	97	(Tabinda et al., 2017)
	Waste dump	R	Various	3	75	current= 2A	53	(Zhang et al., 2006) <sup>i</sup>
	Waste dump	R	Various	3	43	-	(COD=70)	(Mohajeri et al., 2010) <sup>i</sup>
	Various	S	Lissamine green B Methyl orange Fuchsin acid Reactive black 5	2 - 5	1260	-	96	(Rosales et al., 2009) <sup>i</sup>
	Dyes	S	Sunset yellow FCF	3	120	-	(COD=94)	(Ghoneim et al., 2011) <sup>i</sup>
	Various	S	Basic yellow 28	3	40	-	60 - 80	(Iranifam et al., 2011) <sup>i</sup>

AO <sup>b</sup>	Various	S	Ponceau S	2.5	10 - 60	-	> 99	(El-Desoky et al., 2010) <sup>i</sup>
						T= 25°C; C <sub>0</sub> =		
	Textile/dyes	S	Acid orange 7	7	-	300mg/L	> 40	(Moreira et al., 2017)
	Textile/dyes	S	Acid brown 14	3	60	T= 25°C	90	(Bassyouni et al., 2017)
EC <sup>c</sup>						C <sub>0</sub> = 100 mg/L		
	Food	R	Various	5	120 - 300	I=5A	70 - 100	(Panizza and Cerisola, 2006)
	Textile	S	Acid green 50	3 - 9	21	-	> 90	(El-Ashtoukhy and Amin, 2010)
							(COD=68)	
	Textile/dyes	S	Methylene blue	8	30	voltage= 3.1V	95 - 99	(Duan, 2018)
	Paper mill	R	Various	5	60	voltage= 10V	100	(Azadi Aghdam et al., 2016)
							(COD=85)	
	Textile	S	Red X-GRL	5	120	T=25°C	50	(Zhou and He, 2007) <sup>i</sup>
							(COD>=30)	
	Textile	S	Acid green 50	7-9	21	-	> 92	(El-Ashtoukhy and Amin, 2010)
						(COD=87)		
Food	S	Various	4 - 6	25	-	95	(Adhoum and Monser, 2004)	
						(COD=76)		
			Drimarene K2LR					
Textile/dyes	S	CDG blue	7 - 10	60 - 120	COD <sub>0</sub> = 154 mgO <sub>2</sub> /l	90 - 95	(Alinsafi et al., 2005)	
					current density=	(COD=30-36)		
					10-40 mA/cm <sup>2</sup>			
Textile/dyes	R	Various	7 - 10	60 - 120	COD <sub>0</sub> = 620 mgO <sub>2</sub> /l	98 - 99	(Alinsafi et al., 2005)	
						(COD=75)		

<sup>a</sup>: EF= Electro Fenton

<sup>b</sup>: AO= Anodic Oxidation

<sup>c</sup>: EC= Electrocoagulation

<sup>d</sup>: R= real wastewater; S= synthetic wastewater

Synthetic wastewater (S) was created in the laboratory by simulating the hypothetical origin expressed in the apposite column

<sup>e</sup>: t<sub>r</sub>= reaction time

<sup>f</sup>: λ<sub>UV</sub>= UV absorbance

<sup>g</sup>: C<sub>0</sub>= initial concentration

<sup>h</sup>: COD<sub>0</sub>= initial COD

<sup>i</sup>: some information on this case study is also reported in (Sharma et al., 2011)



## 5. Biological processes

### 5.1. Microbial biomass reactors (RBM)

A further approach, aimed at removing color from wastewater, is to adopt microbial biomass reactors (RBM). These systems are currently in the research phase (Tab: 5) (Ajaz et al., 2018; M. C. Collivignarelli et al., 2017; Maiti et al., 2017; Meerbergen et al., 2018) essentially regarding aspects related to the removal of azo dyes. While in aerobic conditions the treatment does not present efficient results of color removal, the absence of oxygen instead stimulates the breaking of the azo-bonds present in the molecules thanks to the aspecific azo-reductase enzyme. In this way, colorless, but toxic and mutagenic aromatic amines are produced for the animals. They are resistant to further anaerobic treatments but are easily removed from aerobic treatments. It has been studied that sequences of anaerobic-aerobic treatment allow a satisfactory removal of the colour (Bahia et al., 2018; Chen et al., 2018; Robinson et al., 2001). Among the most promising cultures are the *Pseudomonas*, *Aeromonas jandaei*, *Bacillus firmus*, *Sphingomonas*, *Klyveromyces marxianus*, *Acinetobacter*, *Halomonas sp. e Alishewanella sp. CBL-2* (Ajaz et al., 2018; Guadie et al., 2018; Hameed and Ismail, 2018; Meerbergen et al., 2018; Pearce et al., 2003; Robinson et al., 2001; Saratale et al., 2009).

Examples of microbial complexes that allow to operate under statically anoxic conditions are also reported, as in the case of *Proteus vulgaris NCIM-202* and *Micrococcus glutamicus NCIM-2168* for removal of the Scarlet R dye (Saratale et al., 2009). Therefore the efficiency of the treatment varies according to the type of dye to be removed and the culture used. Not all cultures are suitable for removing the same types of dyes. Precisely for this reason, before a possible application to the real scale, an in-depth study should be carried out (Lalnunhlimi and Krishnaswamy, 2016; Robinson et al., 2001; Saratale et al., 2009).

The configuration of these catalysts may be different. With regard to the anaerobic processes, one example is UASB (Up-flow Anaerobic Sludge Blanket) reactors. They have already been

tested several times in relation to the removal of chromophores from wastewater produced by textile companies (Tab: 5) (Baêta et al., 2012; España-Gamboa et al., 2017; Kapdan and Alparslan, 2005; Şen and Demirer, 2003; van der Zee and Villaverde, 2005; Verma et al., 2015). For example, (Baêta et al., 2012) obtained 93% removal of Drimaren blue HFRL with yeast extract injection in UASB reactor at ambient temperature.

Most of the studies, however, focus purely on the removal of a single synthetic dye at a time unlike the real cases where there is the simultaneous presence of multiple dyes mixed together (Kapdan and Alparslan, 2005). For example, (Ajaz et al., 2018) successfully removed (83%) the ST Blue dye in 6 days while maintaining a pH of 7 and a temperature of 37 °C. (Maiti et al., 2017) have removed, in just 18 h, up to 90% of color in real wastewater from a cosmetic company.

The factors that affect the discoloration of the wastewater are obviously type and concentration of dye, contact time, presence of alternative electron acceptors, concentration of biomass and toxicity of the dye (it could in fact determine the inhibition of biomass) (Kapdan and Alparslan, 2005; van der Zee and Villaverde, 2005). The major advantage of anaerobic treatment systems, in addition to the discoloration of the initial wastewater, is the production of Biogas that can be reused to produce heat and energy, reducing energy costs (Robinson et al., 2001).

## **5.2. Fungal treatments (FT)**

It is a type of treatment that can be applied even when the dyes show strong resistance to normal microbial activity. Most experiments on the degradation of dyes have been carried out using whole cultures or simple use of extracellular raw enzymes of the ligninolytic system of fungi (Tab: 5) (Ghosh et al., 2018; Jarosz-Wilkolazka et al., 2002; Yesilada et al., 2018).

Some types of fungi are in fact able to degrade dyes using enzymes such as lignin peroxidase (LiP) and manganese peroxidase (MnP). Other enzymes used for this purpose are those that

produce H<sub>2</sub>O<sub>2</sub>, such as glucose-1-oxidase, glucose-2-oxidase, laccase and phenol oxidase. These are the same enzymes used for the degradation of lignin (Robinson et al., 2001).

Fungi that have been shown to decolorize the effluent containing the coloring substance are, for example, *Trametes versicolor*, *P. chrysosporium*, *Hirschioporus larincinus*, *Inonotus hispidus*, *Phlebia tremellosa*, *Coriolus versicolor* and *Aspergillus flavus* (Erdem and Cihangir, 2018; Ghosh et al., 2018; Robinson et al., 2001).

Thanks to the latter, for example, with a reaction time of about 2 days (50 h), it was possible to discolor up to 75% wastewater containing Acid brown 45 (Ghosh et al., 2018).

As with traditional microbial biomass reactors (RBM), even in this case, not all cultures are suitable for removing same types of dyes. Before a hypothetical application to a particular type of wastewater, a thorough characterization of wastewater and cultures to be used is therefore necessary (Bahia et al., 2018; Dwivedi and Tomar, 2018; Robinson et al., 2001).

**Table 5:** Applications of biological processes for color removal

Process	Ind. origin	Type <sup>c</sup>	Dye	BIOLOGICAL PROCESSES			Color removal [%] (other removal [%])	References
				Operating conditions				
				pH	t <sub>r</sub> <sup>d</sup> [h]	Other <sup>e f g h</sup>		
RBM <sup>a</sup>	Textile/dyes	S	Sumifex tourqi blue	7	144	T= 37°C	83	(Ajaz et al., 2018)
	Cosmetics	R	Various	–	18	–	40 - 90	(Maiti et al., 2017)
	Textile/dyes	S	Reactive orange 16 Reactive green 19	7 - 10	48 - 72	T= 30°C	80 - 100	(Meerbergen et al., 2018)
	Sugar refinery	R	Various	5.5	624	FBB + UASB <i>Trametes versicolor</i>	16 - 40	(España-Gamboa et al., 2017)
	Textile	R	Various	–	8 - 30.8	UASB+coagulant Ccoagulant= 800mg/L UASB + yeast extract (500 mg/L)	> 99	(Verma et al., 2015)
	Textile/dyes	S	Drimaren blue HFRL	–	ambient		93 (COD=35)	(Baëta et al., 2012)
	Textile/dyes	S	Direct blue 151 Direct red 31	9.5	120	C <sub>0</sub> = 200mg/L	95 - 97	(Lalnunhlimi and Krishnaswamy, 2016)
	Textile	R	Various	9 - 10	24	anaerobic + 2g/L glucose	59	(Şen and Demirer, 2003)
	Textile	R	Various	7	48	anaerobic	> 85 (COD=90)	(Kapdan and Alparslan, 2005)

	Textile/dyes	S	Scarlet Red	6.5	3	T= 37°C microbial consortium-GR	(COD<100) (TOC>90)	(Saratale et al., 2009)
	Textile/dyes	S	Amaranth Orange II Tartrazine	–	8	anaerobic; Bacteroides fragilis  T=35 °C; Shewanella	80 50 20	(Pearce et al., 2003)
	Textile/dyes	S	Remazol black B	8	–	putrefaciens	95	(Pearce et al., 2003)
		R	Various				80	
	Textile/dyes	R	Various	–	–	anaerobic biological AC Aerobic	50 65 - 90	(Pokhrel and Viraraghavan, 2004)
	Textile/dyes	S	Reactive Red 141 Reactive Blue 220 Reactive Orange 12 Reactive Orange 13 Reactive Red 218	– – – – –	4.5 1 23 50 32	anaerobic; C <sub>0</sub> =100mg/L anaerobic; C <sub>0</sub> =100mg/L anaerobic; C <sub>0</sub> =100mg/L anaerobic; C <sub>0</sub> =100mg/L anaerobic; C <sub>0</sub> =100mg/L	85 - 90 90 - 95 90 - 95 85 - 90 90 - 95	(Şen and Demirel, 2003)
FT <sup>b</sup>	Textile/dyes	S	Reactive black 5 Orange II Indigo carmine	8	24	laccase Si1A + 0.1 mM methyl syringate (MeS) T=35°C	89 - 98	(Blázquez et al., 2018)
	Textile/dyes	S	Various	–	> 480	Saccharomyces cerevisiae UASB + aerobic	23 COD=85	(Bahia et al., 2018)
	Textile	S	Reactive blue 19 Acid black 194 Reactive orange 12 Reactive red 120 Reactive blue 21	4	2	Trametes versicolor UASB + aerobic	99 83 76 56 85	(Erdem and Cihangir, 2018)
	Textile/dyes	S	Crystal Violet Methyl Violet	–	168	Coriolopsis C <sub>0</sub> = 100 mg/L	94 - 97	(Yesilada et al., 2018)

<sup>a</sup>: RBM= Microbial Biomass Reactors

<sup>b</sup>: FT= Fungal Treatments

<sup>c</sup>: R= real wastewater; S= synthetic wastewater

Synthetic wastewater (S) was created in the laboratory by simulating the hypothetical origin expressed in the apposite column

<sup>d</sup>: t<sub>r</sub>= reaction time

<sup>e</sup>: FBB= Fluidized Bed Bioreactor

<sup>f</sup>: UASB= Upflow Anaerobic Sludge Blanket

<sup>g</sup>: C<sub>0</sub>= initial concentration

<sup>h</sup>: AC= Active Carbon

## **6. Conclusions**

In this work different types of treatment that can be adopted to remove the color from industrial wastewater have been exposed. Chemical/physical, chemical, electrochemical and finally biological processes were presented. It has been found that there is no type of ideal method but the most efficient treatment to remove a certain dye should be looked for in a case by case view. While some advanced chemical oxidation methods, as well as chemical/physical methods, are certainly already commonly used (show a better margin of applicability) a lot of work is needed before real-scale applications of electrochemical processes such as electrophotocatalysis, anodic oxidation or biological processes, such as fungal treatments, can be developed.

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None

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Figure

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