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REVIEW

ENVIRONMENTAL PROGRESS
& SUSTAINABLE ENERGY

Removal of adsorbable organic halides from wastewater: A review of advanced chemical and electrochemical processes

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Abstract

To overcome the current limitations of conventional technologies for removing adsorbable organic halides (AOX) from wastewater, advanced chemical and electrochemical processes are attracting interest. This work aims to systematically review the latest findings for the removal of AOX compounds from wastewater, discussing the current main gaps and proposing possible tips for future research. Fenton and photo-Fenton, photocatalysis coupled with biological systems, and reductive processes showed encouraging results with up to 98%, 95%, and 75% of AOX mineralization, respectively. The analysis also highlighted that the studies were mainly carried out on real wastewater, but, except for Fenton and photo-Fenton, lab scale conditions are often far from the realistic ones, representing a huge gap. In all studies, a very low amount of volume has been used with significant concerns about the reliability of results in case of full-scale applications. For these reasons, the promising outcomes of chemical and electrochemical technologies for AOX removal should be fully validated before a possible application on real industrial and municipal wastewater treatment plants. In case of reductive processes, the reduction of approximately half of the AOX present in the wastewater needs days, which is not compatible with large-scale application. Moreover, more studies are also needed to understand the actual toxicity of the treated effluent to estimate the potential detrimental effect of by-products generated during the treatment. These results can be useful both for the scientific community and technical stakeholders to stimulate the scientific debate and increase awareness of these new approaches for AOX removal.

KEYWORDS

AOX, electrochemical treatments, Fenton, paper mill wastewater, photocatalysis

1 | INTRODUCTION

The global ecosystem is facing several challenges mainly due to the increasing impact of pollutants released by human activities.[1–5]

Abbreviations: AOX, adsorbable organic halides; AOPs, advanced oxidation processes; DDT, dichlorodiphenyltrichloroethane; nZVI, nano-zero valent iron; PRISMA, preferred reporting items for systematic reviews and meta-analysis; UV, ultraviolet.

Among these pollutants, adsorbable organic halides (AOX) indicate the total amount of organically bonded halogens and include different compounds, both simple ones (e.g., chloroform) and complex molecules (e.g., dioxins and furans), many of which can determine toxicological effects on health and the environment.[6,7] These compounds can be of natural origin, mainly produced by living organisms or natural processes such as geothermal activities in general, such as volcanoes, and other natural events such as fires.[7] However, in the last two centuries, industrialization determined a significant production and release of AOX.[8] Among the different industries, pulp and paper industries seem to be the major source of AOX, mainly during the phases of pulping and bleaching.[7] In fact, it has been estimated that, in this last phase in which chlorine-based oxidants are used, about 8% of the chlorine is generally converted into AOX.[9]

One of the main issues related to AOX is that some of these compounds tend to bioaccumulate in the food chain thanks to their high stability.[8] In fact, AOX has been detected in both animal and human tissues.[8,10,11] AOX compounds have also shown harmful effects on human health and the environment, such as pentachlorophenol (chronic exposure led to liver and kidney damage, loss in weight in human and metabolic disruption and deformities in fish), hexachlorobenzene (carcinogenic in animal tests), and chlorinated dibenzodioxins and dibenzofurans (endocrine and reproductive disruptors for human and fish).[12–14] For instance, Dichlorodiphenyltrichloroethane (DDT) is one of the most emblematic and famous compounds. The exposure to this substance was linked to several health issues, such as cancer, reproductive and developmental disruptions, and neurological problems.[15] For this reason, starting from the 1970s, DDT began to be banned in many countries, but it is still detected in humans and the environment due to its very high persistence.[16] For instance, according to Piscia et al.,[17] from 2011 to 2020, the concentration of all congeners of DDT in the crustacean zooplankton community of Lake Maggiore (Italy) was up to 157 ng g^{-1} dry weight. In the last five years of the same study, the DDT deposit decreased (up to 57 ng m^2 vs. 129 ng m^2) but remained high considering this substance has been banned by high-income countries for many years.[17] In developing countries, DDT is currently used to tackle malaria in indoor environments.[18] In Africa, more than a third of the samples analyzed exceeded the WHO drinking water guidelines or cancer permissible limits in terms of concentrations of DDT and congeners, implying a threat to human health.[19] In Bangladesh, Subah and Ryu[20] highlighted the impact of DDT on the health of women with several disruptions, such as the escalation of the risk of developing breast cancer and disorders of the menstrual cycle.

AOX compounds are considered recalcitrant pollutants, being not easily degraded by conventional biological processes. The current main conventional approach is to remove them from wastewater through adsorption by activated carbon or ion exchange resins.[15,21] As per definition, these compounds can be effectively removed via adsorption with yields up to 99% depending on the compound and operating conditions.[22,23] However, adsorption technologies also have three main disadvantages: (i) the non-selectivity, especially for activated carbons, which determines the need for preliminary treatments to avoid a competitive effect with other pollutants present in the matrix to be treated, (ii) the absence of reaction of mineralization only shifts the "issue" of AOX from aqueous to solid phase, and (iii) the high cost due to the use of virgin and the need to dispose/regenerate the exhausted material.[24,25] Moreover, the regeneration of spent materials for restoring the adsorptive capacity has an environmental impact due to the use of thermic and/or chemical treatments and, during thermal regeneration, toxic by-products (such as chlorodibezodioxins) could be released into the atmosphere.[26,27]

To overcome some of these disadvantages, in recent years new adsorbents from alternative materials such as agricultural solid waste, biomass, and industrial by-products have been studied.[15] Also, the combination of catalysts was tested to stimulate the oxidation of adsorbed AOX and reduce the need to regenerate the adsorbents.[28] However, some challenges still remain, such as the need for a large amount of adsorbent when the concentration of AOX is high and the difficulties in subsequently destroying AOX contaminants.[25] For this reason, in recent years the interest has moved to advanced oxidation and reductive technologies that can generally grant a fast removal of AOX (also of high molecular weight compounds) and the effective degradation of unsaturated bonds in aromatic compounds.[25,29] These technologies have already been proven to be effective in removing several different species of refractory organic pollutants with fast degradation kinetics.[30–33] In fact, in the advanced oxidation processes (AOPs), the degradation is due to the in-situ production of highly chemical oxidants (mainly hydroxyl radicals $OH^\bullet$) while the reductive processes are based on the production of hydrated electrons e_{aq}^- that have a standard reduction potential of about −2.9 V and represent one of the most promising reducing radicals.[34–38] Both radicals are characterized by a high reactivity and non-selectivity.[35,39] Despite the results of these treatments seeming promising, the knowledge about the effectiveness of these technologies for AOX removal remains heterogeneous, with more studies on Fenton but few data on photocatalytic and reductive processes.

Based on our literature research, from 2018 only 4 review papers have been published on this topic. Esmaeeli et al.[9] and Gupta and Kapoor[40] discussed the recent advances in technologies for treating pulp and paper mill wastewater but without focusing on treatments that specifically remove AOX. Yankovych et al.[7] and Xu et al.[25] reported of the feasible approaches for removing AOX from industrial streams but did not focus specifically on recent applications of advanced technologies. Therefore, the question: "What are the latest discoveries on advanced chemical and electrochemical processes for the removal of AOX compounds from polluted wastewater?" remains open. Based on the literature search, three groups of process have been identified in the recent works: (i) Fenton and photo-Fenton, (ii) photocatalysis coupled with biological systems, and (iii) reduction technologies. This work aims to review and discuss the main advantages and drawbacks of each of such technological solutions, highlighting the current gaps of the research and providing tips for future studies. The outcomes of this study can be useful for the scientific community and technical stakeholders to take consciousness of the new advance of the research on this topic and stimulate future studies in order to solve the current gaps.

2 | METHODOLOGICAL APPROACH

This work aims to highlight the latest advances in advanced chemical and electrochemical processes for the degradation of AOX in wastewater, discussing main results and proposing possible tips for future research.

The literature has been selected and screened according to the Preferred Reporting Items for Systematic reviews and Meta-Analysis (PRISMA) guidelines.[41] The keywords ("AOX" OR "adsorbent organic halogen" OR "organohalogens removal") AND ("wastewater" OR "aqueous waste") have been used to search in the "abstract, title and keywords" fields. To exclude not peer-reviewed documents, the screening was made using Scopus® database. Then, two consecutive screenings of the literature have been carried out. Original articles, books, editorials and conference proceedings written in English were considered. To focus only on new findings of the scientific community, review articles were not taken into account. In order to discuss only very recent findings, only documents published after 2017 were taken into consideration. After this first screening 76 documents were selected while 388 records were excluded (Figure 1).

However, this group of records also included documents that were not focused on the removal of AOX with advanced chemical and electrochemical processes. For this reason, a second screening has been carried out investigating titles and abstracts to avoid documents (i) focused only on the production of AOX instead of removal, (ii) only on toxicity and potential health implications, or (iii) referring only to biological processes of conventional chemical technologies. Records for which the manuscript could not be retrieved ($n = 9$) were excluded. After all screenings, 14 documents remained and were considered for the critical review. These works have been classified based on the nature of the process: (i) Fenton and photo-Fenton ($n = 8$), (ii) photocatalysis coupled with biological systems ($n = 3$), and (iii) reduction technologies ($n = 3$).

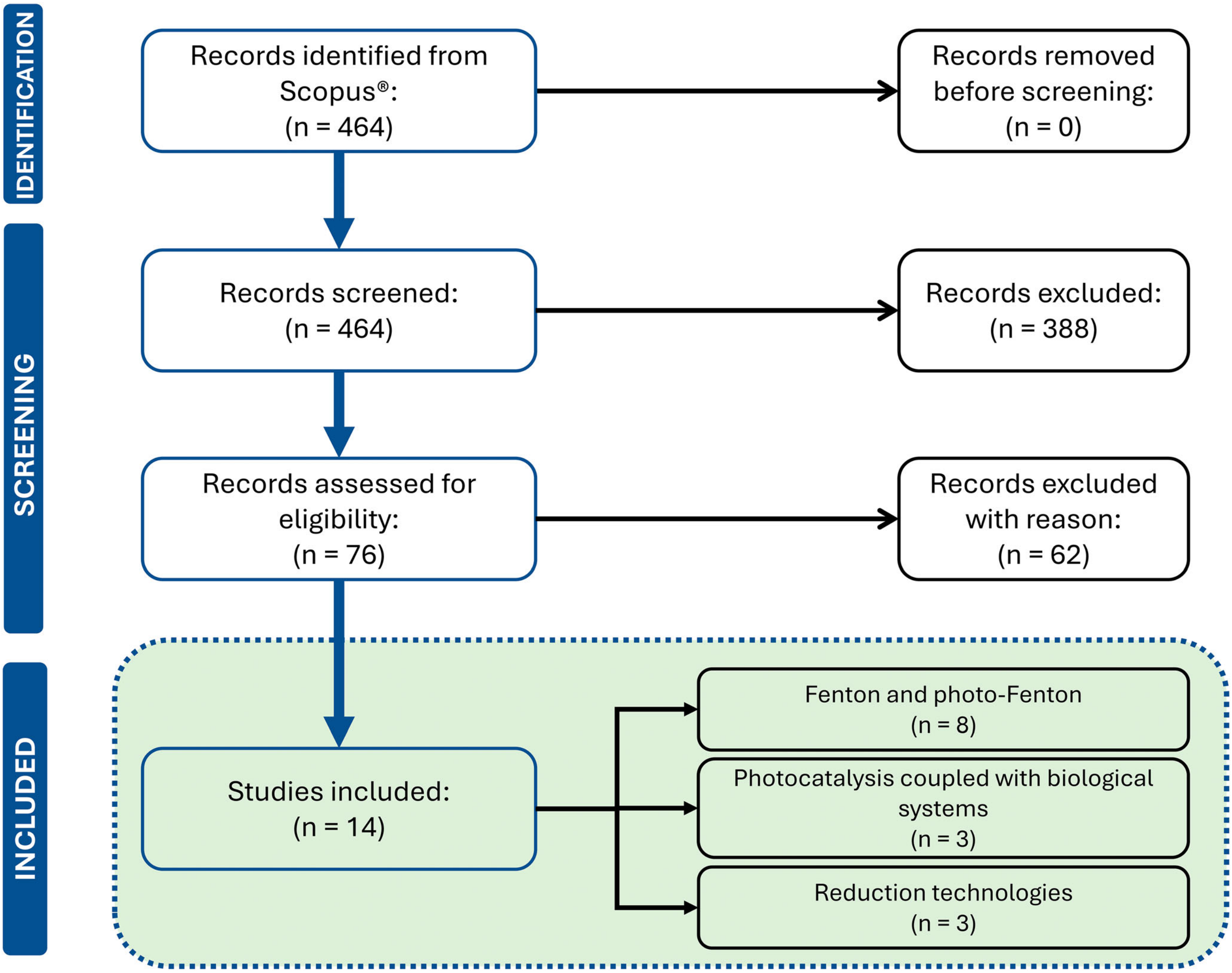


FIGURE 1 Results of the systematic literature search and studies included in the analysis. n = number of documents.

The number of the cited references of this work is higher because the documents related to aspects such as, for instance, (i) the characteristics and properties of AOX, (ii) the main sources of AOX contamination, (iii) the effect of these compounds on human health and the environment were found using more specific keywords on Scopus® and Google Scholar® and/or referring to gray literature.

3 | LATEST FINDINGS ON ADVANCED CHEMICAL AND ELECTROCHEMICAL PROCESSES

3.1 | Overview of the selected studies and initial conditions

The latest findings on advanced chemical and electrochemical technology for AOX degradation from wastewater can be summarized in three categories of technologies: (i) Fenton and photo-Fenton, (ii) photocatalysis coupled with biological systems, both approaches based on the oxidation of the AOX compounds, and (iii) reductive processes, such as bio-electrochemical treatments.

It should be noted that only 14 works corresponded to the literature search criteria (Table 1), highlighting that there is still a long way to go for greater scientific interest in the development of technologies aimed at combating the presence of AOX in wastewater.

All of the selected studies except SEL1 and SEL12 (refers to Table 1 to associate a reference to the code of the selection) used real wastewater instead of synthetic one. This represents a positive aspect because help to better simulate the real conditions with an initial concentration of AOX, presence of other pollutants and chemical–physical parameters (e.g., pH) comparable with those that would occur in the case of full-scale applications.

In terms of origin, real wastewater tested mainly came from the pulp and paper bleaching process, except for SEL13 and SEL14. In fact, paper mill wastewater is one of the major sources of AOX. During the bleaching procedure, chlorine can react with lignin fibers, producing unwanted AOX compounds. However, in recent years, the use of elemental chlorine-free or total chlorine-free reagents has been stimulated.[56] ClO_2 replaced the use of Cl_2 and NaClO, but even if the ClO_2 delignification process is highly selective, free chlorine is still generated and, reacting with lignin and hexenuronic acid, determines the production of AOX.[56,57] In SEL13, activated sludge from a wastewater treatment plant treating chemical dyestuff was tested for AOX degradation, while in SEL14, technologies were involved in the treatment of pharmaceutical wastewater. In this last case, AOX still represents an issue due to the limited number of studies in the literature and the use of organic halogens in pharmaceuticals production even today.[6]

One of the most evident limitations of the current literature on the topic concerns the absence of studies on the effectiveness of advanced chemical and electrochemical treatments on matrices such as municipal wastewater,[58] leachates,[59] and hospital wastewaters[60] that can represent a source of spread of AOX in the environment if not properly treated.

TABLE 1 Type and origin of the wastewater and initial concentration of AOX.

Selected works	References	Type of wastewater	Origin of wastewater	AOX initial concentration [mg L^{-1}]
SEL1	42	S	-	n.a.
SEL2	43	R	Pulp bleaching	123.2 ± 28.2
SEL3	44	R	Pulp bleaching	124 ± 12.0
SEL4	45	R	Pulp bleaching	47.0 ± 3.78
SEL5	46	R	Pulp bleaching	123.2 ± 28.2
SEL6	47	R	Pulp and paper bleaching	54.5 ± 4.1
SEL7	48	R	Pulp and paper bleaching	n.a.
SEL8	49	R	Pulp bleaching	45–50
SEL9	50	R	Bleaching	43
SEL10	51	R	Bleaching	12
SEL11	52	R	Bleaching	40–45
SEL12	53	S	-	n.a.
SEL13	54	R	Activated sludge of chemical dyestuff WWTP	2279[a]
SEL14	55	R	Pharmaceutical wastewater	501.2

Abbreviations: n.a., not available; R, real; S, synthetic.

[a] Expressed as mg kg^{-1} dry weight.

3.2 | Fenton and photo-Fenton processes

Fenton process involves more than 20 chemical reactions and leads to the formation of hydroxyl radicals ($OH^{\bullet}$), which are powerful oxidants.[61–64] In acidic conditions, H_2O_2 reacts with Fe^{2+} producing unselective and highly reactive radicals that allow the oxidation of AOX.[61,65] For instance, in SEL1 the removal of AOX in wastewater polluted with a halogenated anionic textile dye has been evaluated, highlighting that, after 20 h with 120 mol of H_2O_2 and 4 mol of $FeSO_4$ at a pH of 2–2.5, more than 98% of AOX were effectively degraded (Table 2).

In the case of pulp and paper mill, the high temperature of the released wastewater represents an advantage for AOX degradation. In fact, the consumption of H_2O_2 in the Fenton reaction improves with the increase of temperature, determining an intensified production of OH, and therefore stimulating the reaction of mineralization.[66] However, the main disadvantages of Fenton processes are the high costs due to (i) the addition of chemicals, and (ii) the production of chemical sludge that needs to be disposed of.[67] To overcome these disadvantages, Photo-Fenton has been proposed and applied. In this case, the use of ultraviolet (UV) rays improves the performance, limits the consumption of Fe^{2+}, and therefore reduces the production of chemical sludge.[68]

For instance, in SEL2, the AOX removal from a bleaching industrial wastewater was studied in order to optimize the process parameters. According to the same study, photo-Fenton was more effective than conventional Fenton: 90% versus 80% of AOX removed, respectively, with a lower amount of chemicals consumed. Similar results were shown in SEL3, in which the central composite experimental design tool was used to optimize the operational conditions and, in the best scenarios, 95% of AOX was removed with photo-Fenton versus the 85% removed by Fenton without UV. Also for this reason, in SEL2, photo-Fenton was evaluated as more economical with respect to the conventional Fenton reaction, with an estimated cost of 0.45 € $g^{-1}_{\mathrm{AOXremoved}}$ versus 0.78 € $g^{-1}_{\mathrm{AOXremoved}}$, respectively.

The feasibility of reducing the costs of the Fenton reaction using residual iron dust from the metallurgic industry as a catalyst for the treatment of pulp bleaching wastewater was also a matter of study.

TABLE 2 Results of Fenton and photo-Fenton processes for AOX removal.

		Treatment conditions							
Selected work	Process	Volume [L]	Fe^{2+} [mM]	H_2O_2 [mM]	UV [W m^{-2}]	T [°C]	pH [–]	Reaction time [min]	Removal [%]
SEL1	FE	0.25	4[a]	120[a]	n.p.	25	2–2.5	1200	>98
SEL2	FE	0.5	12.5	169	n.p.	60	2.2	10	80
SEL2	PhFE	0.5	1	178	142	60	2.2	10	90
SEL3	FE	0.5	8.5	178	n.p.	60	2	10	85
SEL3	PhFE	0.5	2	178	142	60	2	10	95
SEL4	FE	0.5	1[b,c]	200	n.p.	60	2–2.2	10	55–60
SEL4	FE	0.5	1[c,d]	200	n.p.	60	2–2.2	10	75–80
SEL5	FE	0.5	12.5[e]	169	n.p.	60	2.2	10	80–85
SEL5	PhFE	0.5	1[e]	178	142	60	2.2	10	60–65
SEL6	FE	0.5	6[f]	139	n.p.	60	2.8	10	70–75
SEL6	PhFE	0.5	1[f]	82	142	60	2.8	10	65–70
SEL6	FE	0.5	6[e]	139	n.p.	60	2.8	10	60–65
SEL6	PhFE	0.5	1[e]	82	142	60	2.8	10	55–60
SEL6	FE	0.5	6[g]	139	n.p.	60	2.8	10	75–80
SEL6	PhFE	0.5	1[g]	82	142	60	2.8	10	65–70
SEL7	FE	0.25	2[h,i]	5[j]	n.p.	60	2.7	30	80–85
SEL8	FE	0.5	6	139	n.p.	60	2.5	10	74–81

Note: To relate each selected work (SEL) with the proper reference, refer to Table 1.

Abbreviations: FE, Fenton; n.p., not provided; PhFE, Photo-Fenton.

[a] Expressed as mol per mol of initial dye.

[b] Residual iron dust as homogeneous catalyst.

[c] Expressed as grams per 200 mL.

[d] Residual iron dust as heterogeneous catalyst.

[e] The value expresses the sum of the concentration of two co-catalysts (i.e., Fe^{2+} and Cu^{2+}) with Fe^{2+} and Cu^{2+} in a 50:50 molar ratio.

[f] The value expresses the sum of the concentration of two co-catalysts (i.e., Fe^{2+} and Co^{2+}) with Fe^{2+} and Co^{2+} in a 50:50 molar ratio.

[g] The value expresses the sum of the concentration of two co-catalysts (i.e., Fe^{2+} and Mn^{2+}) with Fe^{2+} and Mn^{2+} in a 50:50 molar ratio.

[h] Zero valent iron.

[i] Expressed as g L^{-1}.

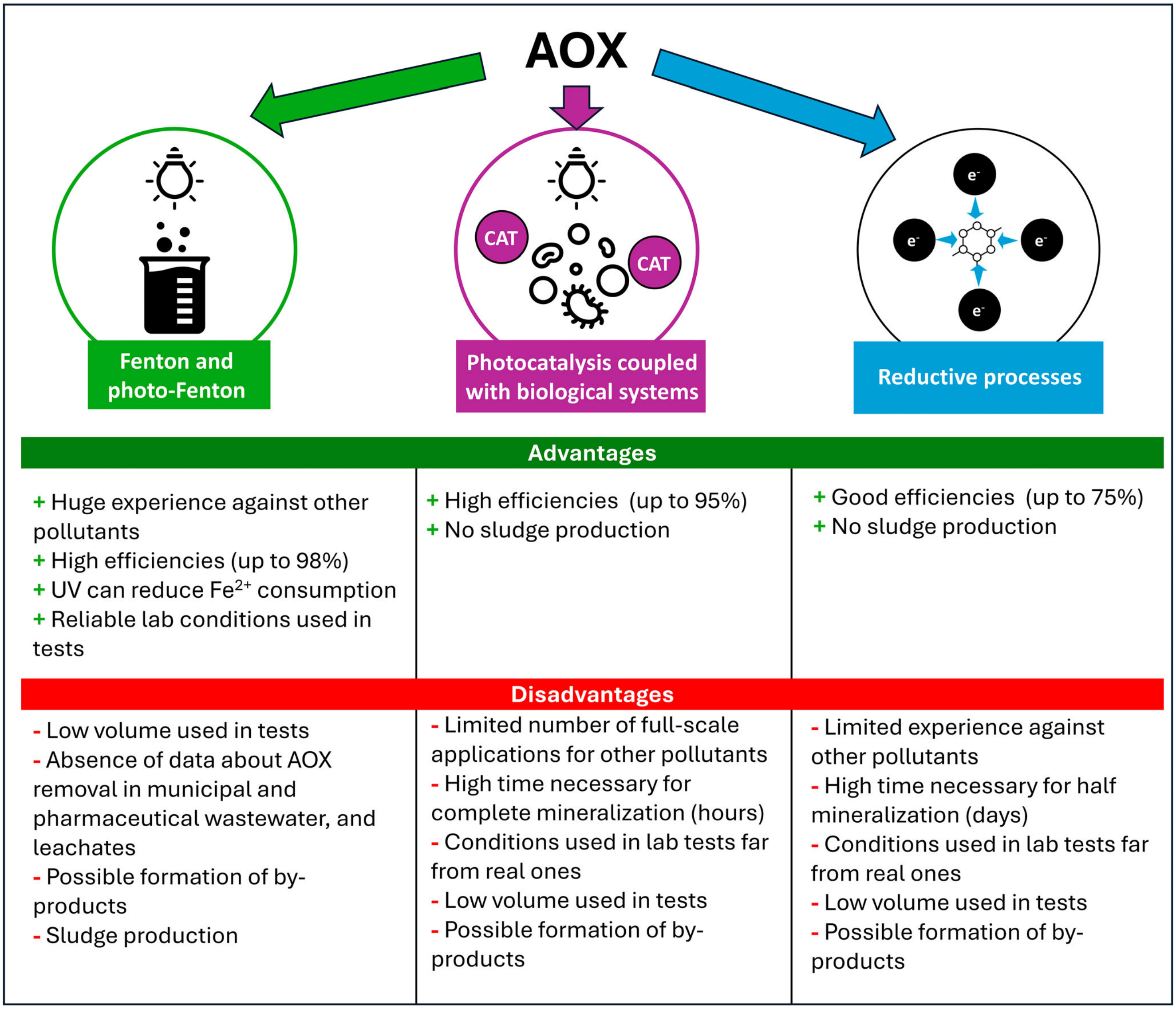
[j] Expressed as mL L^{-1}.

SEL4 proved that Fenton allowed for the removal of more than 60% of AOX and that the operating costs of the heterogeneous Fenton with alternative material were up to 10% lower than those of the conventional Fenton reaction.

The effectiveness of using different oxidation state of iron ions and combination of iron and copper as co-catalyst has been also evaluated. In SEL5, Fe^{2+} proved to be a more cost-effective catalyst than Fe^{3+} in removing AOX from industrial wastewater but the addition of Cu^{2+} proved to be not effective in improving the performance of Fenton process against AOX and synergistic effects between metals were excluded. In a similar study (SEL6), Cu, Mn, and Co were tested as co-catalysts in photo-Fenton in order to stimulate a synergistic effect and reduce the need of chemicals. These metals gave similar performance than using Fe^{2+}-only in case of Fenton, but the lower reactivity to UV rays hindered the performance of photo-Fenton reactions.[47]

A synergistic action between the Fenton process and biological treatments has also been highlighted in SEL7, proving that these two processes, when sequentially coupled, can help to reduce the concentration of AOX by up to 94%. Moreover, in recent studies, the use of nano-zero valent iron (nZVI) is gaining interest due to the high rate of hydroxyl radicals' formation, thanks to the increased surface area of the particles.[69] SEL8 showed that the optimal combination of nZVI and H_2O_2 for the degradation of AOX in the pulp and paper industry bleaching wastewater seems to be 2:5. In this condition, more than 85% of AOX can be degraded in 1 h of reaction time, while the use of ultrasound allowed for the achievement of 55% of AOX degradation in only 30 min.[48]

One of the main advantages of this technology is the significant experience in terms of full-scale applications for the removal of recalcitrant and emerging micropollutants[70,71] (Figure 2). In general, studies on Fenton and photo-Fenton processes proved that high efficiencies



Fenton and photo-Fenton	Photocatalysis coupled with biological systems	Reductive processes
Advantages		
+ Huge experience against other pollutants + High efficiencies (up to 98%) + UV can reduce Fe^{2+} consumption + Reliable lab conditions used in tests	+ High efficiencies (up to 95%) + No sludge production	+ Good efficiencies (up to 75%) + No sludge production
Disadvantages		
- Low volume used in tests - Absence of data about AOX removal in municipal and pharmaceutical wastewater, and leachates - Possible formation of by-products - Sludge production	- Limited number of full-scale applications for other pollutants - High time necessary for complete mineralization (hours) - Conditions used in lab tests far from real ones - Low volume used in tests - Possible formation of by-products	- Limited experience against other pollutants - High time necessary for half mineralization (days) - Conditions used in lab tests far from real ones - Low volume used in tests - Possible formation of by-products

FIGURE 2 Main advantages and disadvantages of latest findings for each group of technologies (indicated as "+" and "−", respectively).

can be reached against AOX compounds. As proved in SEL2, SEL3, SEL5, SEL6, the use of UV rays can reduce the iron consumption by up to over 90% without affecting the performance of the Fenton reaction towards AOX. In SEL5 and SEL6, the use of alternative (co)-catalyst proved to be promising, but further studies are necessary before possible applications on a larger scale, given the limited amount of data and quite contrasting results about the presence of a synergistic effect between two or more metals. Except for SEL1, all studies have been made in conditions of temperature (around 60°C) and pH (2–2.5) very similar to real conditions for paper mill wastewater. This represents a positive aspect highlighting the high reliability of the results. The next steps should be the scale-up of the tests. To date, SEL1 and SEL7 used only 0.25 L of wastewater while the other selected studies used 0.5 L, a very limited volume with respect to the real magnitude of the flowrate of industrial wastewater.

3.3 | Photocatalysis coupled with biological systems

Generally, photocatalysis is based on the absorption of UV light by metal oxides such as TiO_2, Fe_2O_3, ZrO_2, $NiFe_2O_4$.[72,73] The catalyst is irradiated in order to excite and move the electrons from the valence band to the conduction band. This phenomenon leaves electron-holes which can directly oxidize the organic substances or take part in the production of $OH^\bullet$.[74] This technology has been applied for the removal of AOX from bleaching wastewater coupled with a biological approach. Generally, wastewater that contains AOX can be hardly biologically treated due to the high toxicity.[51,75] At the same time, AOPs can represent a valid solution despite not being economically feasible for a complete AOX degradation.[76] Therefore, the approach followed in SEL9, SEL10, and SEL11 was to integrate the chemical and biological technologies for completely degrading AOX, overcoming the main disadvantage of the advanced chemical processes.

In SEL 9, photocatalysis has been coupled with biological treatment to treat bleaching wastewater with elemental chlorine free. In this system, a mixed activated sludge biofilm developed on sugarcane bagasse cellulose/TiO_2, which acts as a carrier. The photocatalyst was activated via the UV emitted by a xenon lamp, and the coupled activity with biomass allowed for the degradation of almost 95% of AOX in the wastewater[50] (Table 3).

In a similar study (SEL10), a porous sponge carrier with TiO_2 was used to act as a carrier for biomass for bleaching wastewater degradation. The catalyst was activated by a UV-led lamp in order to promote coupled chemical and biological reactions for AOX degradation. According to this study, the integrated system allowed to obtain better performance (more than 80%) with respect to photocatalysis and biodegradation alone (almost 65% and 30%, respectively).[51] In fact, microorganisms influence AOX degradation, but this phenomenon was very limited. However, coupling with the biodegradation help to enhance the performance of photocatalytic process. Also in SEL11 almost 95% of AOX were degraded after 7 h of treatment with a bio-photocatalytic system with respect to the 72% and 41.4% of mineralization obtained in the same reaction time with photocatalysis and biodegradation alone, respectively.

SEL9, SEL10, and SEL11 demonstrated that high efficiencies (>80%) can be reached against AOX compounds by combining photocatalysis and biological treatments. As proved in SEL11, the use of visible light for activating the catalyst could be a promising alternative, being the UV the major source of electrical consumption in photocatalytic reactors.[77] However, due to the limited amount of data, further studies are necessary before a possible scale-up of the process.

The recent interest in this wastewater treatment approach and the consequent limited number of studies represent the main barrier currently existing for a potential application on an industrial scale[78] (Figure 2). Moreover, current studies are focused on conditions far

TABLE 3 Results of photocatalysis coupled with biological systems for AOX removal.

	Operating conditions							
Selected work	Volume [L]	Catalyst	Microbial community	Lamp	T [° C]	pH [−]	Reaction time [h]	Removal [%]
SEL9	0.45	TiO_2 coated bagasse cellulose carrier; filling rate: 5%	Mainly *Sphingobacterium*, *Luminiclostridium*, and *Paenibacillus*	$p = 350$ W; $\lambda = 380$–700 nm	22	7	7	95
SEL10	0.4	TiO_2 coated sponge carrier; filling rate: 7.5%	Mainly *Proteobacteria*, *Chloroflexi*, *Bacteroidota* and *Actinobacteria*	$p = 65$ W; $\lambda = 420$–700 nm	22	7	6	80
SEL11	0.3	N-TiO_2 coated bagasse cellulose carrier; filling rate: 5%	Mainly *Phanerochaete, Paenibacilus and Ruminiclostridium*	Visible light (no lamp has been used)	n.a.	7	7	95

Note: To relate each selected work (SEL) with the proper reference, refer to Table 1.
Abbreviations: λ, emission length; n.a., not available; P, power.

from the real one of bleaching wastewater in terms of temperature, especially in the case of bleaching wastewater from paper mills which are characterized by high temperature.[79] Moreover, this technology generally requires almost 6–7 h before reaching an almost complete mineralization of AOX. In this sense, the use of electrical bias that reduces the electron hole recombination, enhancing the rate of $OH^•$ production, could be tested to investigate the feasibility of this technology (called photoelectrocatalysis[80,81]) and the performance against AOX compounds. The next steps should be the scale-up of the tests, considering that SEL9, SEL10, and SEL11 were carried out on 0.3–0.45 L wastewater. This represents a very limited volume, and results obtained in these lab-scale tests should be confirmed on larger volumes before possible full-scale application.

3.4 | Reductive processes

Among the other advanced technologies for AOX removal, the use of chemical reduction processes is attracting more and more interest. In fact, the removal of AOX with advanced oxidation could sometimes be negatively affected due to the saline environment in industrial wastewater. Reductive processes showed to effectively remove refractory compounds like AOX (Table 4). In this sense, for instance, UV/sulfite was tested for the degradation of 2,5-dichloronitrobenzene and its intermediates such as AOX (SEL12). The increase of sulfite concentration from 2.6 to 104.3 mM enhanced the apparent degradation rate of the target molecule and its intermediates by one order of magnitude, from 0.0059 to 0.0628 min^{-1}. An alkaline pH stimulates the reaction; in fact, raising the pH from 3 to 12 can enhance the rate of the reaction by one order of magnitude.[53]

The combination of metallic particles is a promising alternative solution for AO removal. In SEL13 Pd/Fe bimetallic particles were tested for AOX removal in an industrial wastewater treatment plant treating colored wastewater. The process exploits the reductive properties of these materials. Generally, the first metal has a low standard redox potential (i.e., Fe) and acts as an electron donor for the reduction of AOX while the second metal (i.e., Pd) has a high standard redox potential and acts as promoter of reaction.[82] In SEL13, these particles (Pd/Fe: 10 g L^{-1}) were used directly in the activated sludge reactor and up to 50.7% of removal was obtained after 15 days of reaction with initial slightly acidic pH (6.0).[54]

The reduction of AOX compounds has also been tested in electrochemical reactors coupled with biological systems to stimulate the reaction. For instance, in SEL14, the removal of AOX from a real pharmaceutical wastewater was assessed, highlighting that the reduction of the cathode potentials (from −300 to −1000 mV) improves the effectiveness of the process from 60% to 70% and enhances the removal rate of AOX from 0.9 to 1.2 mg_{AOX} h^{-1}. In these systems, AOX can be reduced directly on the electrode surface, while at the same time, H_2 produced during water-electrolysis and the degradation of organic compounds acts as an electron donor.[55]

The reduction processes demonstrated promising results in the mineralization of AOX even if the technologies are not yet mature for potential full-scale application[83] (Figure 2). Recent studies are few (SEL12, SEL13, SEL14) and all performed at laboratory scale on small volumes (from 0.05 L in SEL13 to 0.4 L in SEL14). Although real wastewater was used in SEL13 and SEL14, the outcomes are not robust enough to predict in detail what the results will be if applied in an industrial context. One of the main disadvantages concerns the long reaction time (days) necessary to have a degradation of at least half of the AOX present in the tested matrices. This aspect will have to be addressed in future studies as it could lead to a significant reactor volume in case of full-scale application with consequent problems of high construction and management costs.

3.5 | Current gaps and tips for future studies

Based on the results of the literature, the authors suggest carrying out tests on alternative co-catalysts in Fenton and photo-Fenton, since

TABLE 4 Results of the application of advanced reduction technologies for AOX removal.

		Operating conditions					
Selected work	Process	Volume [L]	Temperature [°C]	pH [−]	Reaction time [d]	Other	Removal [%]
SEL12	UV/sulfite	0.4	25	n.a.	n.a.	Low pressure mercury lamp	n.a.
SEL13	Biological treatment with suspended Pd/Fe particles	0.05	30	6	10	Pd loading over Fe = 0.05 weight % Pd/Fe particle dosage: 15 g L^{-1} Biomass consisted in activated sludge	50–55
SEL14	Bioelectrochemical	0.1	n.a.	7.5	1	Potential: from −300 mV to −1000 mV Addition of 40 mL of anaerobic sludge Addition of 1 mM of methyl viologen	63.8–75.2

Note: To relate each selected work (SEL) with the proper reference, refer to Table 1.

preliminary studies showed promising results although still in the laboratory phase. In this sense, it would be necessary to increase the number of studies that verify the applicability of this process on: (i) larger volumes, and (ii) wastewater other than that from paper mills. In future research, for photocatalysis combined with biological treatment systems, it would be interesting to evaluate the feasibility and effectiveness of a process upgrade with the use of bias (photoelectrocatalysis) in order to optimize the oxidative process and try to reduce the contact time necessary to achieve an almost complete mineralization of the AOX (to date still several hours). Investigating also the effectiveness on AOX of alternative semiconductors (as already tested for phenolic compounds[72]) could represent an interesting option to establish the effect on the degradation kinetics. The authors also prompt the studies on reductive processes that showed high effectiveness on AOX degradation but to date the high reaction time required and the lack of experience on the topic (to date there are no full-scale applications) represent the two main gaps that will need to be addressed in future research. Moreover, it would be useful to combine the chemical characterization of the matrices before and after the application of the different technologies with the use of ecotoxicity tests to exclude the formation of toxic by-products.

Finally, an aspect to consider concerns the possible formation of potentially toxic by-products. Advanced oxidation and reduction reactions determine the transformation of molecules, and the presence of other polluting molecules, in addition to AOX, could be the cause of the formation of unwanted reaction by-products which, if toxic, can have a deleterious effect on health and the environment.[84,85] For this reason, it could be useful to combine the chemical characterization of the matrices to be treated before and after the application of the different technologies with the use of ecotoxicity tests, as already proposed for PFAS and other emerging micropollutants,[86–88] in order to fully characterize the matrix.

In terms of process maturity among those analyzed, to date only Fenton and photo-Fenton have been sufficiently studied and tested to be applied at full scale. These processes are already used at industrial scale to treat waters particularly loaded with recalcitrant pollutants such as landfill leachates, waters produced by the petrochemical industry, and paper mill waters.[44,89–91] In terms of costs, studies suggest a strong impact related to the treatment of the sludge produced and the use of reagents (even up to 30% for H_2O_2 alone).[44] In the case of photoelectrocatalysis coupled with biological degradation and reductive processes, a process maturity has not yet been reached that can determine a large-scale application and, according to the best of the authors' knowledge, no industrial scale plants for AOX removal exist.

4 | CONCLUSIONS

In this work, a systematic review has been carried out to highlight and critically discuss the latest advances in advanced chemical and electrochemical processes for the degradation of AOX in wastewater. As with any other systematic review, it should be noted that the choice of keywords may have represented a limitation that could have led to a bias in the selected documents, limiting the number compared to those actually available.

Based on the selected documents, the current studies focus on three main technologies: (i) Fenton and photo-Fenton, (ii) photocatalysis coupled with biological systems, and (iii) reductive processes. To date, no literature is available about real case studies, but only lab-scale tests have been performed. The results of the analysis of the selected documents highlighted that:

- Fenton and photo-Fenton represent the processes that are at the most advanced stage of study with applications already on an industrial scale for the removal of other recalcitrant contaminants. In this sense, they guaranteed an oxidation of up to 98% of the AOX. Their main disadvantages linked to the consumption of chemical reagent (Fe^{2+}) and the production of sludge (present in the Fenton process) can be reduced by up to 90% by the use of UV rays.
- The photocatalysis process combined with biological treatment systems has shown promising results (up to 95% oxidation of AOX) but the number of studies is still limited, and those available were carried out on a laboratory scale.
- Studies of reduction processes are still in their infancy with a situation similar to that already found for the photocatalysis approach integrated with biological systems: (i) limited number of studies (ii) very small volumes tested. The results showed that it generally takes days for the reduction of approximately half of the AOX present in the treated matrix, but results are promising (up to 75%) depending on the applied technologies.

The main findings of this work can be useful for the scientific community and technical stakeholders to provide essential knowledge useful for overcoming the current gaps in AOX removal with advanced chemical and electrochemical technologies.

AUTHOR CONTRIBUTIONS

Marco Carnevale Miino: conceptualization, methodology, validation, investigation, writing—original draft preparation; **Maria Cristina Collivignarelli**: writing—review and editing, supervision; **Stefano Bellazzi**: writing—review and editing, visualization; **Alessandro Abbà:** writing—review and editing; **Marco Baldi:** writing—review and editing; **Alberto Pietro Damiano Baltrocchi**: writing—review and editing, visualization; **Elena Cristina Rada**: validation, writing—review and editing; **Vincenzo Torretta**: conceptualization, validation, supervision.

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The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

DATA AVAILABILITY STATEMENT

Data sharing not applicable to this article as no datasets were generated or analysed during the current study.

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