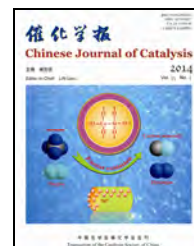




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Review

Wet air oxidation for the decolorization of dye wastewater: An overview of the last two decades

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ABSTRACT

Wet air oxidation (WAO), a liquid phase reaction between organic materials in water and oxygen, is one of the most economical and technologically viable advanced oxidation processes for wastewater treatment, particularly toxic and high organic content wastewater. WAO is the liquid phase oxidation of organics or oxidizable inorganic components at elevated temperatures (125–320 °C) and pressures (0.5–20 MPa) using gaseous oxygen (or air) as oxidant. In the past two decades, the WAO process was widely studied and applied in the treatment of dye wastewater. Compared to conventional WAO, catalytic WAO processes have higher efficiency and use moderate reaction conditions. The catalysts included homogenous and heterogeneous types. The key points that need to be solved are recycling of homogenous catalysts and better stability of heterogeneous catalysts. In the present review, the technological processes are first introduced, then some research history and hotspots of WAO research are presented, and finally, its application in the treatment of dye wastewater in the past two decades is summarized to reveal the impressive changes in modes, trends, and conditions used. The application includes model pollutant studies and wastewater tests.

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1. Introduction

Dye wastewater from the printing and dyeing industry is a major source of environmental contamination. Dyestuff, slurries, dyeing aids, acids and alkalis, and fibers and inorganic compounds are present in dye wastewater [1]. Generally, dye wastewater is characterized by a strong color, high pH, high chemical oxygen demand (COD), and low biodegradability [2]. Such wastewater is difficult to treat, especially with color removal, using conventional wastewater processes [3].

In a brief search in Scopus, many hits were obtained for the term “dye oxidation” (Fig. 1). The major peak for works on the wet air oxidation (WAO) process was in the 21st century. The

present review first introduces the technology and process, and the research history and research hotspots of WAO. Its application in dye wastewater treatment is then described in detail. Major conclusions were obtained from the different procedures during the past decades, which strongly influenced the new trends and economic aspects of each time period. All these confirmed the sustainability of this process.

Recently, advanced oxidation processes (AOPs) have received attention as effective pretreatment processes for less biodegradable wastewater [4]. AOPs were first proposed by Glaze [5] in 1987. They were defined as near ambient temperature and pressure water treatment processes based on the generation of hydroxyl radicals ($\bullet\text{OH}$) to initiate the oxidative

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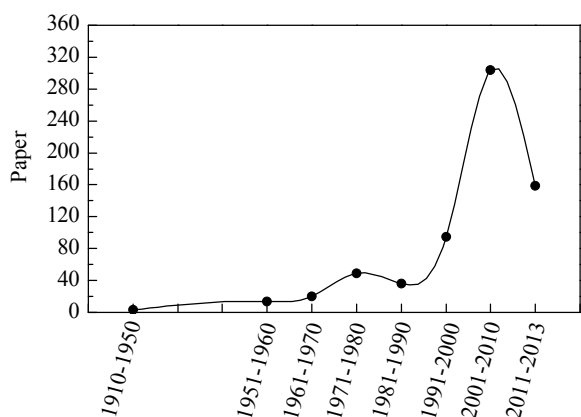


Fig. 1. Works published with the “dye oxidation” keyword (data search in Scopus).

destruction of organics [5]. $\bullet\text{OH}$ is a powerful, non-selective chemical oxidant, which reacts a million to a billion times faster than either ozone (O_3) or H_2O_2 (Table 1) [6].

AOPs can achieve the oxidative destruction of refractory compounds that conventional oxidation cannot. In addition, they have the potential to completely oxidize organic contaminants to CO_2 , H_2O , and mineral salts. AOPs can also oxidize inorganic contaminants such as cyanides, sulfides, and nitrites [6]. Their advantages for the treatment of dye wastewater include fast reaction, complete treatment, and being pollution-free and of wide application.

WAO, which was established and developed by Zimmermann [7–10], is one of the most economical and technologically viable AOPs for wastewater treatment, particularly toxic and high organic content wastewater. The application range of each AOP is different and depends on the flow rate and organic content of the effluent [11]. Unlike other AOPs, WAO is suitable for high organic loads at high flow rates. Especially, WAO has great potential for treating effluents containing a high content of organic matter (about 10–100 g/L of COD) and/or toxic con-

Table 1

Relative oxidation power of some oxidizing species with chlorine as the reference species [6].

Oxidation species	Oxidation power
Hydroxyl radical	2.05
Atomic oxygen	1.78
Ozone	1.52
Hydrogen peroxide	1.31
Permanganate	1.24
Chlorine	1.00

taminants, for which a direct biological treatment is not feasible [12]. WAO is not only eco-friendly, but also economical compared to other AOPs that use harmful and expensive oxidants like O_3 and H_2O_2 .

2. Technology and process

WAO is the liquid phase oxidation of organics or oxidizable inorganic components at elevated temperatures (125–320 °C) and pressures (0.5–20 MPa) using a gaseous source of oxygen (O_2 or air) as oxidant. The solubility of O_2 in aqueous solutions is greatly enhanced at elevated temperatures and pressures, which provide the strong driving force for oxidation. WAO has been demonstrated to oxidize organic compounds to CO_2 and other end products (carbon is oxidized to CO_2 , nitrogen is converted to NH_3 and nitrate ions (NO_3^-), and halogen and sulfur are converted to inorganic forms). WAO is useful for the treatment of hazardous, toxic, and non-biodegradable waste streams. The process becomes self-sustaining when the feed COD is about 20000 mg/L [13].

Figure 2 shows a typical WAO treatment system. The wastewater is brought into the system using a high pressure pump. Air (or O_2) is added to the reactor using a compressor. Preheating may be necessary to raise the temperature of the wastewater. The feed temperature is adjusted to let the exothermic heat of reaction raise the mixture temperature to the

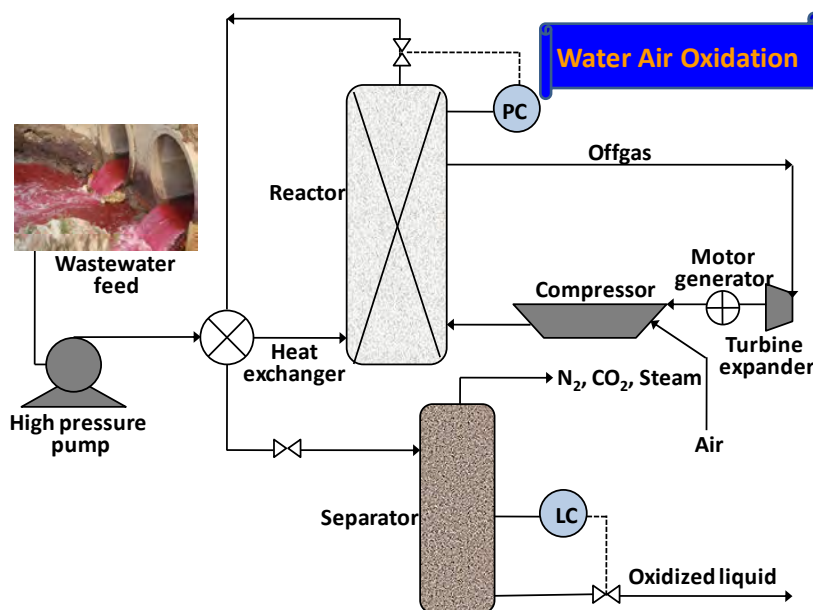


Fig. 2. Basic wet air oxidation plant flow sheet.

operating temperature. The treated effluent can be used to preheat fresh wastewater [14]. Liquid and non-condensable gases are separated in a separator. The off-gases can be expanded in a turbine to recover energy. The gasses are then treated using carbon absorption beds or afterburners to reduce the concentration of organic matters. The oxidized liquid phase can be directly disposed of or, most often, subjected to biological treatment.

WAO requires much less fuel than any other thermal oxidation process such as incineration. This is because the only energy required for WAO is that used to make up the difference in enthalpy between the incoming and outgoing streams. The capital cost of a WAO system is higher, and the reactor itself can account for a significant fraction (50%) of the total equipment cost. The capital investment can be reduced if one selects a WAO system based on O₂ instead of air. Prasad et al. [15] studied the technological and economic aspects of both air- and O₂-based systems and concluded that O₂-based WAO systems gave higher profitability. The operating costs are almost entirely for power consumption to compress air and to pump liquids [16]. Furthermore, WAO becomes self-sustaining with no additional fuel requirement when the COD is above 20000 mg/L [17], that is, energy recovery (thermal or mechanical) becomes possible when the feed COD is sufficiently high [18]. Costs can further be reduced by reducing the severity of the oxidation conditions by the use of suitable catalysts.

3. Research history – first attempts

The first patent on wet air oxidation was 102 years ago. In 1911, Strehlenert applied for a patent for the treatment of sulfite liquor (from pulp production) by oxidation with compressed air at 180 °C [13]. In 1927, Hanglin and Stauf [19] purified a solution containing metallic salts and organic impurities using wet air oxidation. The temperature was above 130 °C, and the pressure was above 0.2 MPa. The WAO technology was firmly established and developed by Zimmermann [7–10]. The first known WAO plant was set up in 1958 in Norway for the treatment of sulfite liquors, but this was later closed down due to uneconomical operation.

A truly successful application of the WAO technique as a commercial process for wastewater treatment came in the early 1960s with its application for the recovery of pulping chemicals from waste liquors and the complete oxidation of sewage sludge. The Zimpro Corporation built several WAO plants for the oxidation of sewage sludge. On the basis of the work with sewage sludge, new applications for WAO were discovered, such as the conditioning of sludge for easy dewatering and improving settling characteristics. Commercial applications for hazardous wastes were started in the early 1970s. Up to 1995 more than 200 full scale WAO plants were in operation for the treatment of a wide variety of effluent streams [13]. The major application of WAO is still for the treatment of sewage sludge, with more than 50% of the WAO plants built being used for this purpose [20].

4. Research hotspots

Although WAO can quickly and completely treat high concentration and highly toxic pollutants, its operational conditions and equipment requirements are severe. Capital and operating costs are high compared to other treatments, such as biological treatments. Therefore, the wide application of WAO has restrictions.

During the past two decades, the application of catalysts for the catalytic WAO (CWAO) process has been a research hotspot. CWAO not only reduces the severity of the reaction conditions but also more easily decomposes even refractory pollutants, thereby reducing capital and operational cost [13,21–31]. Regardless of the wastewater type, the operating cost of CWAO is about half that of non-catalytic WAO due to milder operating conditions and shorter residence time [32]. According to the type of catalyst, CWAO can be divided into homogeneous CWAO processes and heterogeneous CWAO processes.

Homogeneous copper salts are the most active catalysts in the homogeneous CWAO process [13,33–35]. As there is no additional third phase, mass transfer and reaction in a homogeneous CWAO process are therefore similar to the non-catalytic system. Reactor operation is also simpler compared to the heterogeneous CWAO process [25]. The main disadvantage of the homogeneous CWAO process is that an additional separation step is required to remove or recover the metal ions (homogeneous catalyst) from the treated effluent due to their toxicity, which increases operational cost [12]. Several heterogeneous CWAO processes have been developed in the last two decades, including the Ciba-Geigy process, LO-PROX (low pressure wet oxidation) process, and WPO (wet peroxide oxidation) process [28].

In recent years, heterogeneous CWAO has received much attention because a separation step is not necessary. Many solid catalysts including noble metals, metal oxides, and mixed oxides have been widely studied for the CWAO process [12]. However, in addition to being cost-effective, the solid catalyst must also show satisfactory stability and durability under the severe operating conditions and acidic environments in WAO systems. Catalyst deactivation can occur due to leaching, sintering, poisoning of active sites or fouling of the catalyst surface by deposition of organic or inorganic compounds [25]. To overcome these problems, several solid catalysts based on supported precious metals were studied [36–43]. Supported precious metal catalysts not only are less prone to deactivation by the leaching of the active phase, but they also have higher overall activity for the oxidation of various pollutants. Several commercial heterogeneous CWAO processes have been developed, such as the NS-LC (Nippon Shokubai liquid chromatography) process, Osaka Gas process, and Kurita Process [28].

5. Application in dye wastewater

Synthetic dyes are widely used in many industries such as the textile, cosmetic, printing, pharmacy, and food processing industries [44]. These dyes, which exist in large volumes of wastewater generated by the factories, threaten human health by their toxicity and potential carcinogenicity [45]. The removal of these dyes in the wastewater has attracted much interest

and is an important practical problem [46]. There were many studies for dye removal with WAO in the past two decades. According to the treated agents, these studies can be divided into those on pure compounds (important model pollutants) and wastewater containing toxic and hazardous compounds. From their findings, a better understanding of the reactions taking place during WAO (reaction mechanism and kinetics) was obtained, which is important as it leads to the reliable design of oxidation reactors and also to cost reduction by optimization of the operating conditions.

5.1. Model pollutants

Studies on the WAO of model dyes are listed in Table 2. Lin et al. [47] studied the WAO of a prepared direct dye solution using a CoAlPO₄₋₅ catalyst. The results showed that CoAlPO₄₋₅ effectively improved the rate of color removal, and the activation energy of the color removal reaction was decreased from 110 to 75 kJ/mol as the catalyst loading was increased from 0 to 3.0 g/L. In their subsequent work [48], the performance of CoAlPO₄₋₅ and CeO₂ as catalysts in the WAO process was stud-

ied. By adsorption and oxidation, CoAlPO₄₋₅ effectively decreased the American Dye Manufacturers Institute color and COD values in the dye solution. At a reaction temperature of 135 °C and a pressure of 1.0 MPa, color and COD removal after 2 h reached 95% and 90%, respectively.

Zhu et al. [49] used four types of powder catalysts whose main active components were Cu, Ce, Cd, and Co-Bi in the CWAO treatment of a H-acid solution. The comparison of the efficiency of the different catalysts showed that the Ce3Cu1 (3:1) catalyst was the best. At the conditions of 200 °C, 3 MPa of O₂ partial pressure, pH = 12, and 30 min of reaction time, the COD removal rate was over 90%.

To get further insight into the dominant reaction pathway of the catalytic and non-catalytic WAO processes, Arslan-Alaton et al. [50] added isopropyl alcohol and bromide as •OH scavengers into the reaction solution. The catalytic processes were appreciably less sensitive to the presence of •OH scavengers than non-catalyzed WAO, implying that CWAO did not mainly use a free radical type reaction mechanism. The results supported the free radical chain mechanism only for non-catalyzed WAO [50].

Table 2

Summary of WAO studies with model dyes.

Year	Dye	Reactor	Catalyst	Conditions	Effect	Ref.
2001	Direct dye	Autoclave	CoAlPO ₄₋₅	135 or 145 °C, 1 MPa O ₂	Activation energy of color removal 75 kJ/mol	[47]
2002	H-acid	Autoclave	Ce3Cu1	135 or 145 °C, 1 MPa O ₂	Removal of 90% COD in 30 min	[49]
2002	Acid dye Orange II	Stainless steel unions	Non-catalytic; H ₄ SiW ₁₂ O ₄₀ and Na ₂ HPW ₁₂ O ₄₀	160–290 °C, 0.6–3.0 MPa O ₂	Arrhenius parameters E _a = 84 kJ/mol and A = 5.6 × 10 ⁷ /min	[50]
2003	Reactive dye	Autoclave	CoAlPO ₄₋₅ & CeO ₂	135 °C, 1.0 MPa O ₂	Color and COD removal were 95% and 90%, respectively, after 2 h	[48]
2004	Phenol	Trickle bed reactor	Active Pt and Ru	393 and 473 K, 5 and 8 MPa O ₂	58–107 kJ/mol of activation energy	[51]
2005	Azo dyes	Stainless-steel high-pressure reactor	Pt/MWNT	150 °C, 6.9 MPa O ₂	Almost 100% removal of dyes in 2 h	[52]
2007	Methyl orange	Glass reactor	Fe ₂ O ₃ -CeO ₂ -TiO ₂ /γ-Al ₂ O ₃	25 °C, atm. pressure air	Removal of 98.09% color and 96.08% TOC in 2.5 h	[53]
2007	Cationic red X-GRL	Autoclave	Non-catalytic	60–180 °C, 0–1.2 MPa O ₂	Removal of 92% dye at 180 °C in 1 h	[54]
2009	Safranin-T	Three-neck glass flask	Zn _{1.5} PmO ₁₂ O ₄₀ nanotube	25 °C, atm. pressure air	Removal of 98% color and 95% COD within 40 min	[55]
2011	Basic Yellow 11	Stainless-steel high-pressure reactor	Ni- and Fe-doped mixed oxides	120 °C, 5 MPa O ₂	High TOC and toxicity removal efficiency	[56]
2011	Cl Basic Yellow 11	Autoclave	Pt nanotube	120–180 °C, 5 MPa O ₂	Effective to destroy the dye structure	[57]
2011	Basic Yellow 11, Crystal violet, Naphtol Blue Black	Autoclave	Ni catalysts supported on hydrotalcites	120 °C, 5 MPa O ₂	Removal of 95% dye	[58]
2012	Crystal violet	Autoclave	Hi/HT	220 °C, 5 MPa O ₂	11.15 kJ/mol of activation energy	[59]
2012	Cationic red GTL	Glass reactor	Mo-Zn-Al-O catalyst	25 °C, atm. pressure air	Removal of 80.1% color and 50.9% TOC	[60]
2012	Crystal violet	Trickle-bed reactor	Ni/MgAlO	120–180 °C, 2.5–6 MPa O ₂	Removal of 64% TOC in 350 h	[61]
2012	Crystal violet	Autoclave	Ni/MgAlO	140–200 °C, 5 MPa O ₂	Removal of 98% dye	[62]
2013	Azo dyes	Autoclave	CuO/γ-Al ₂ O ₃	80 °C, atm. pressure O ₂	Removal of 99% color and 70% of TOC in 2 h	[63]
2013	Chromotrope 2R	Trickle-bed reactor	Ni/MgAlO	100–180 °C, 2.5 MPa air	Conversion of 82% TOC at 150 °C	[64]
2013	Basic Yellow 11	Autoclave	Ni/MgAlO	100–200 °C, 3–6 MPa O ₂	Improvement of 49.1% for the CWAO process	[65]

Cybulski et al. [51] developed active Pt and Ru catalysts for the CWAO of phenol solution. A kinetic analysis of phenol conversion and COD changes was given based on a lumped reaction network. Most of the activation energy obtained ranged from 58 to 107 kJ/mol. The reaction orders for phenol ranged from 0.94 to 2.0 and those for O₂ from 0.22 to 0.85. They later use supported Pt catalysts to treat wastewater from a fine chemicals plant and also obtained good results [66].

Garcia and co-workers [52] have done much work on the CWAO of model dyes. They first used multi-walled carbon nanotubes (MWNT) to prepare supported Pt catalysts and applied them in the CWAO of azo dye solutions [52,57,67]. The CWAO of a textile wastewater was also performed using the same catalyst. Complete color removal and 51.2% TOC abatement were obtained. Later, they used Ni catalysts supported on hydrotalcites [58] and mixed oxides [56,59,61,62,64,65], and especially for the Ni catalyst supported on Mg-Al mixed oxides (Ni/MgAlO), much work was done to apply it in different reactors [61,64] and with different dyes [64,65].

In order to develop a catalyst with high activity and stability for the CWAO process at room temperature and atmospheric pressure, Liu et al. [53] prepared Fe₂O₃-CeO₂-TiO₂/γ-Al₂O₃ by sequential impregnation. The degradation of an azo dye (methyl orange) in the CWAO process with this catalyst at room temperature and atmospheric pressure was investigated, and it was shown that the catalyst had excellent catalytic activity in treating synthetic wastewater containing 500 mg/L methyl orange. The 98.09% of the color and 96.08% of TOC were removed in 2.5 h. In their recent work [60], a new Mo-Zn-Al-O catalyst was developed for the CWAO of cationic red GTL at room temperature and atmospheric pressure. The optimal conditions of the pH value, initial concentration of dye, and catalyst dosage were 4.0, 85 mg/L, and 2.72 g/L, respectively, for the maximum decolorization of 80.1% and TOC removal of 50.9%. Zhang et al. [55] have also developed a catalyst with high activity and stability for the CWAO of pollutant dyes at room condition. Using the new polyoxometalate Zn_{1.5}PMo₁₂O₄₀ with a nanotube structure as the catalyst, the CWAO of Safranin-T (10 mg/L), a hazardous textile dye, using air at room

temperature and atmospheric pressure, the removal of 98% color and 95% COD within 40 min was achieved. Hua et al. [63] also prepared a catalyst (CuO/γ-Al₂O₃) that was used under atmospheric pressure. At 80 °C, almost 99% of color and 70% of TOC of azo dye solution were removed in 2 h.

5.2. Wastewater studies

Studies on the WAO of wastewater are summarized in Table 3. Lin and Ho [68] treated desizing wastewater, a typical high strength industrial wastewater, with WAO. The desizing wastewater sample was obtained from a large textile dyeing and finishing plant. They found that over 60% COD removal efficiency was easily obtained in 1 h. Also, it was observed that WAO can increase the biodegradability of the treated desizing wastewater, thus rendering it suitable for subsequent activated sludge treatment. In their subsequent work [69], catalysts (CuSO₄ and Cu(NO₃)₂) were introduced into the WAO process. The COD removal percentage was increased from 60% to 80% by 1 h treatment. Kinetic studies indicated that the generalized kinetic model yielded a better fit to the kinetic data than the two stage, first order model [70].

Lei et al. [74] studied the treatment of dyeing and printing wastewater from the textile industry by WAO. They found that an alumina-supported catalyst had an advantage over other metal salt catalysts in that it can be easily separated from the treated wastewater by filtration and recycled. The reaction was carried out in a 2 L high pressure reactor. In another work, by a homogeneous catalyst screening experiment, they found that of all catalysts tested, copper salts were the most effective. Anions in the salt solutions were active in the catalytic process. Nitrate ions were more effective than sulfate ions. A mixture of salts containing different metals performed better than a single salt [71]. They introduced a modified first order kinetics model to study the WAO of printing and dye wastewater [72]. The model simulations were in good agreement with experimental data. The results indicated that the ratio of degraded organic matter was independent of temperature and can be improved by using a catalyst. In their subsequent work, their focus changed from

Table 3
Summary of studies on dye wastewater WAO.

Year	Wastewater	Reactor	Catalyst	Conditions	Effect	Ref.
1996	Desizing wastewater	High pressure Parr reactor	CuSO ₄ and Cu(NO ₃) ₂	150–250 °C, 7 MPa air	80% COD removal in 1 h [69]	
1997	Desizing wastewater	High pressure Parr reactor	CuSO ₄ and Cu(NO ₃) ₂	175–260 °C, 4–8 MPa air	70% COD removal in 1 h [70]	
2000	Textile wastewater	Autoclave	CuSO ₄ , FeSO ₄ , Fe ₂ (SO ₄) ₃ , MnSO ₄ , Cu(NO ₃) ₂ , Fe(NO ₃) ₃ , Mn(NO ₃) ₂	240 °C, 1.5 MPa O ₂	61%–77% COD removal in 30 min [71]	
2001	Textile wastewater	Autoclave	CuO and Cu(NO ₃) ₂	150–300 °C, 2.65 MPa O ₂	1st order rate constant: 0.0492–0.0559/min [72]	
2003	Textile wastewater	Autoclave batch reactor	Non-catalytic	423–563 K, 1.69 MPa O ₂	activation energy: 37.5 kJ/(mol·K) [73]	
2005	Textile wastewater	Stainless-steel high pressure reactor	Pt/MWNT	150 °C, 6.9 MPa O ₂	51.2% TOC abatement [52]	
2006	Chemical plant wastewater	Fixed-bed reactor	CBC-supported Pt	393–473 K, 5.0 MPa O ₂	High COD removal rates [66]	
2011	Ink-plant wastewater	Autoclave	Pt nanotube	120–180 °C, 5 MPa O ₂	Effective to remove TOC and toxicity [57]	
2011	Textile wastewater	Autoclave	Ni catalysts (supp. Hydrotalcites)	120 °C, 5 MPa O ₂	Effective to remove TOC [58]	

the use of industrial wastewater to simulated wastewater to study the degradation mechanism in the WAO of dyes [54].

Chen and coworkers [73] designed a WAO system to carry out a COD kinetic study of the treatment of printing and dye wastewater from the textile industry. A simple first order kinetic model described the removal of WAO-removable COD well. The activation energy was 37.5 kJ/mol.

6. Conclusions

Studies on the WOA of dye wastewater began from the middle 1990s. The early studies focused on the treatment of wastewater. In the last decade, research focus changed to model dye studies. Most WOA studies are on CWOA, especially heterogeneous catalytic WOA. The use of a solid catalyst not only improved treatment efficiency but also reduced the severity of the reaction conditions, e.g., a Mo-Zn-Al-O catalyst can be active under room conditions. Many present studies are fundamental research focusing on the development of new catalysts for the WAO process. Process technology and modeling techniques should also be developed to optimize the process and solve problems in the practical application of WAO for the treatment of dye wastewater.

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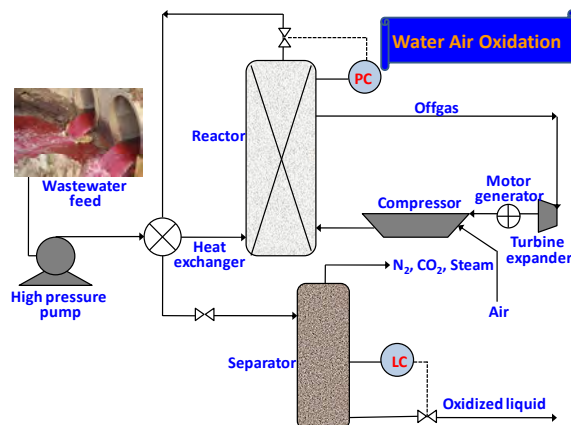
Graphical Abstract

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Wet air oxidation for the decolorization of dye wastewater: An overview of the last two decades

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Wet air oxidation (WAO) is one of the most economically and technologically viable advanced oxidation processes for dye wastewater. The wide studies on WAO of dye wastewater began from 1995. Recent studies focused on real wastewaters.



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