

POTENTIAL OF RED MUD IN FENTON PROCESSES FOR THE DEGRADATION OF ORGANIC POLLUTANTS IN WATER - A MINI REVIEW

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Abstract: Fenton processes, in which hydrogen peroxide in the presence of divalent iron ions generates hydroxyl radicals ($\cdot\text{OH}$), are widely used for the degradation of organic pollutants (phenols, antibiotics, dyes). In this review, red mud is analysed as a cheap source of iron ions in Fenton processes. Raw red mud can be used without additional modifications, but to increase the catalytic efficiency, its modification is required, which includes chemical reduction, carbothermal treatment or doping with metals. Particular attention is given to photo-Fenton and electro-Fenton processes, where red mud doped Co, Sn or Ce, or in combination with reduced graphene oxide and biochar, allow the generation of not only hydroxyl radicals ($\cdot\text{OH}$) but also singlet oxygen ($^1\text{O}_2$) and superoxide radicals ($\cdot\text{O}_2^-$), achieving $\geq 99\%$ pollutant removal. At the same time, the synthesised catalysts showed high stability and reusability. Based on a comparative analysis of more than 30 studies, it is concluded that red mud represents a cheap source of iron ions for heterogeneous Fenton processes, with significant potential for industrial application.

Keywords: Fenton process, hydroxyl radicals, organic pollutants, red mud.

1. INTRODUCTION

Red mud, or bauxite residue in some literature, is industrial waste that is produced during the processing of bauxite into aluminium oxide by the Bayer process [1]. However, around 180 million tons of red mud are accumulated annually in the world, which represents a serious environmental risk [2]. The main problem of its disposal lies in the fact that it has a very high pH due to the presence of alkali, and the presence of heavy metals (Cr, Ni, V), which can penetrate to the surrounding soil and groundwater [3]. On the other hand, red mud contains significant amounts of useful compounds, such as iron oxides (Fe_2O_3 , 30–60% in dry weight), aluminum (Al_2O_3 , 10–20%), silicon

(SiO_2 , 5–15%) and titanium (TiO_2 , 2–5%), which can be used for numerous purposes [4].

To prevent the pollution of the ecosystem, and at the same time valorise the red mud, a circular approach is needed in which the red mud is not disposed of, but turned into valuable materials [5,6]. The possibilities of using red mud are shown in Figure 1a. One way is its use as a catalyst carrier or active agent in advanced oxidation processes (AOP), e.g. in Fenton processes, which is based on the generation of hydroxyl radicals ($\cdot\text{OH}$) from hydrogen peroxide and in the presence of iron ions to decompose organic pollutants into harmless end products such as carbon dioxide and water [7]. Figure 1b shows the reaction path for a typical Fenton process.

The classical homogeneous Fenton system (ions $\text{Fe}^{2+}/\text{H}_2\text{O}_2$) is often used due to its high affinity for generating hydroxyl radicals $\cdot\text{OH}$ which then serve to degrade contaminants, but the main problem is that iron remains in the solution after treatment, which leads to secondary pollution [8]. In contrast, heterogeneous Fenton processes, in which iron is in a solid aggregate state, allow the catalyst to be easily separated and reused after the process is completed. Also, with their application, it is possible to adjust the pH (optimally 3–5), temperature (20–60 °C) and peroxide concentration, which affects the degree of radical formation [5].

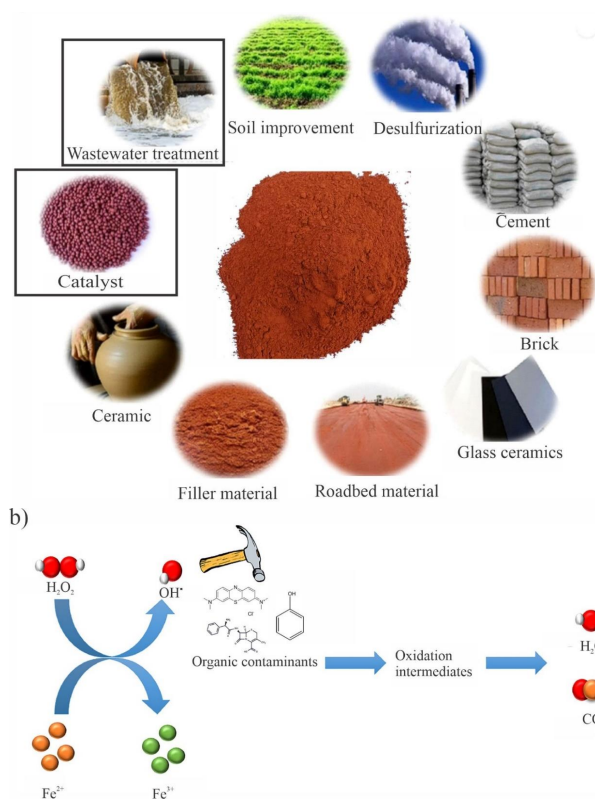
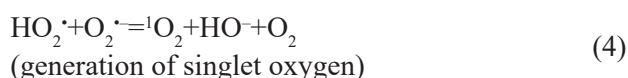
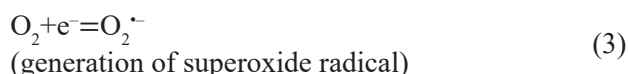
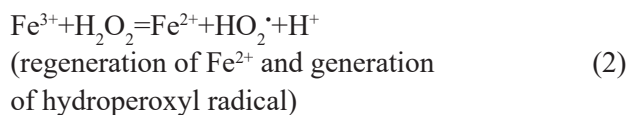
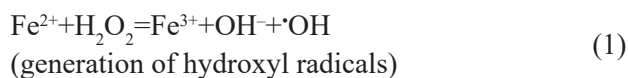


Figure 1. Red mud applications (a) and Fenton reaction pathway (b). Figure (a) adapted and modified from [9]; Figure (b) adapted and modified from [10].

Red mud has a favorable physicochemical composition (due to the presence of iron) for the generation of hydroxyl radicals and other reactive species such as superoxide radical ($\text{O}_2^{\cdot-}$), hydroperoxyl radical (HO_2^\cdot) and singlet oxygen ($^1\text{O}_2$). It is also possible to modify it (reduction, acid washing, calcination, doping with other metals, etc.) to improve its porosity, pore volume and specific surface area, thereby increasing its efficiency [5].

The main reactions related to the generation of reactive oxygen species (ROS) are follows [11]:



The application of red mud in Fenton processes opens perspectives for the degradation of organic pollutants in water (dyes, pharmaceuticals, pesticides), while reducing overall operating costs and environmental impact. The continuation of this paper presents a brief review of the application of raw and modified red mud in heterogeneous Fenton systems and the key factors that determine their application in water treatment.

2. RED MUD IN FENTON PROCESSES

2.1. Application of unmodified red mud

Red mud can be used in Fenton processes without previous modifications, thanks to the high content of iron in its composition.

In the work of Albqmi et al., red mud was used as a catalyst in the Fenton process for the treatment of wastewater from an olive processing plant, which is extremely toxic due to the presence of phenolic compounds [12]. Experiments were conducted at different concentrations of red mud: 0.05, 0.10, 0.5, 1.0, 2.0, 4.0, 5.0, 20 and 30 g/L. Increasing the concentration of red mud increased in the degradation of phenolic compounds, until stable values were reached at red mud concentrations higher than 5 g/L. At 0.5 g/L of red mud, 58.1% of the total organic carbon (TOC) was removed, while at 5 g/L of red mud, 74.4% of the TOC was removed. The optimal operating conditions were pH = 3, magnetic stirring speed = 460 rpm, ambient temperature and H_2O_2 at a concentration of 10 w/v relative to the wastewater. A similar study was carried out by Domingues et al. [13]. It was found that the adsorption of the effluent on the red mud was negligible and that the degradation of phenolic acids from an olive processing plant was

due to oxidation by hydroxyl radicals. At a catalyst concentration of 1 g/L and 100 mg/L hydrogen peroxide, 100% degradation of phenolic acids and 25% mineralisation occurred after 60 minutes of reaction.

Raw red mud can be used for the hydrothermal degradation of antibiotics (e.g., norfloxacin - NOR) from pharmaceutical wastewater using H_2O_2 to produce formic acid [14]. When 10 mL of a 40 mg/L solution was used, the optimal reaction conditions were 0.02 g of red mud activated at 100 °C, 0.2 mL of added H_2O_2 , a reaction time of 0.5 h, and a temperature of 90 °C, resulting in a NOR degradation of 69.26% and a formic acid yield of 58.36% (selectivity: 87.56%). In a similar study, acidified red mud was used for the degradation of amoxicillin and the production of formic acid [15]. Based on various analyses, it was determined that the degradation of amoxicillin is most influenced by the interactions between Fe_3O_4 on the surface of the red mud and H_2O_2 in the solution, while formic acid is a product of the decarboxylation of amoxicillin. The optimal conditions, under which the highest yield of formic acid was achieved, are: reaction temperature of 90 °C, reaction time of 30 minutes, H_2O_2 concentration of 20 ml/L, addition of acidified red mud of 0.8 g/L, pH = 7 and stirring speed of 500 rpm.

2.2. Application of modified red mud

The modification of red mud is carried out to improve its surface structure and chemical properties. The most common techniques are reduction, acid activation, metal doping or combination with carbon materials. Some of the modification techniques are shown in Figure 2.

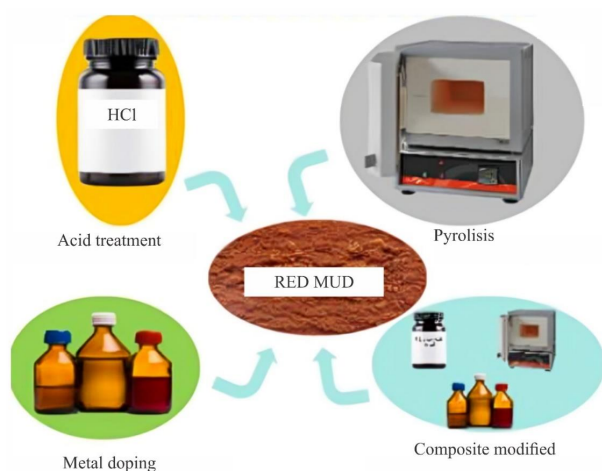


Figure 2. Red mud modification techniques.
Adapted and modified from [16].

2.2.1. Common physicochemical modification techniques of red mud

Modification of red mud by reduction is a process in which oxidised forms of metals in the mud composition (such as Fe^{3+}) are reduced to more chemically active forms (e.g. Fe^{2+}), thereby improving its catalytic activity in Fenton processes.

In the work of Chen et al., red mud was modified by reduction with oxalic acid and l-ascorbic acid in an acidic solution [17]. The results showed that red mud treated with 2 g of oxalic acid dehydrate and 2 g of l-ascorbic acid and red mud treated with 3 g of oxalic acid dehydrate and 3 g of l-ascorbic acid showed removal efficiencies of over 99.1% of phenol with a concentration of 200 mg/L within 5 minutes. The excellent catalytic performance of the modified red mud was attributed to the presence of Fe_3O_4 , Fe_2O_3 , Mn_2O_3 , Fe_2SiO_4 and $FeTiO_3$. The rate constants for the two previously tested catalysts, based on a pseudo-first-order kinetic model, were 1,000 and 1,073 min^{-1} , respectively. In a similar study by Chen et al., red mud was first treated with HCl to extract iron. The dissolved iron was then reduced with ascorbic acid, and finally the filtrate was precipitated to obtain the final catalytic form [18]. The results showed that the catalyst had the highest phenol degradation efficiency (99.3%) after just 5 min, due to the production of iron polymetallic oxides on the catalyst and the formation of mesoscopic particles and microcellular structures. The optimal conditions were: catalyst dosage of 1 g/L, H_2O_2 concentration of 5 mM, 3–6 initial pH, and 100 mg/L initial phenol concentration. The degradation data fit a pseudo-first-order kinetic model, and the reaction rate constant (k) was 0.865 min^{-1} . The possible degradation pathway for phenol is: phenol \rightarrow catechol \rightarrow benzoquinone \rightarrow muconic acid \rightarrow low molecular weight organic acids \rightarrow CO_2 and H_2O .

A heterogeneous catalyst can be synthesized by direct reduction of iron oxide from red mud to zero-valent iron at 800 °C using H_2 as the reducing agent [19]. Using 2.86 mM H_2O_2 and 1 g/L of catalyst, 95% of acid red G (ARG), whose initial concentration is 100 mg/L, was removed after just 10 minutes. The analyses showed that $\cdot OH$ was the main reactive species responsible for the degradation of pollutants, and that the catalyst/ H_2O_2 system could also be used for the degradation of antibiotics (sulfa-

methoxazole, ibuprofen, and primidone). A catalyst containing zero-valent iron was also synthesized in a study by Sun et al. [20]. In comparison to the previous study, here the carbon black red sludge obtained by pyrolysis of waste tires was subjected to carbothermal reduction. The characterization results indicate that the iron transformation, i.e. $\text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{FeO} \rightarrow \text{Fe}^0$, is achieved by varying the carbothermal reduction temperature. The catalyst reduced at 900 °C showed much higher efficiency in the degradation of methylene blue (MB) than the catalyst reduced at 850 °C and lower temperatures. This indicates that zero-valent iron nanoparticles obtained at higher temperatures are more efficient than iron oxides in heterogeneous Fenton processes. Free radicals ($\cdot\text{OH}$ and O_2^-) and singlet oxygen ($^1\text{O}_2$) contribute most to the degradation of MB in the Fenton process.

In addition to iron reduction as a modification method to obtain a more efficient catalyst, it is possible to include some other methods, such as acid washing, calcination, neutralization and precipitation.

Thus, a red mud composite, which was first modified with reduced graphene oxide (rGO) and then acid-washed and calcined, can be used for the degradation of rhodamine B (RhB) [21]. The modification with rGO led to the inhibition of charge

carrier recombination and the acceleration of $\text{Fe}^{3+}/\text{Fe}^{2+}$ cycling during the process. The acid treatment and calcination reduced the impurities in the catalyst, which allowed $\alpha\text{-Fe}_2\text{O}_3$ to be in greater contact with H_2O_2 . Due to this treatment, 99.8% degradation of RhB was achieved within 20 min, and the catalyst itself showed high stability over a wide pH range. The reaction pathway of rhodamine B degradation is shown in Figure 3a.

In a study by Chen et al., a catalyst was synthesized from red mud by a method that included acid treatment, neutralization, and precipitation [24]. $\alpha\text{-Fe}_2\text{O}_3$ and $\beta\text{-Fe}_2\text{O}_3$ played a key role in the activation of peroxide (H_2O_2), with a phenol removal efficiency of 96.8% under optimal conditions (0.02 M H_2O_2 , 50 mL of phenol at a concentration of 100 mg/L, 0.05 g of catalyst, and a reaction time of 120 min). The phenol removal efficiency (96.8%) was not consistent with the chemical oxygen demand removal efficiency (88.2%), indicating that carboxylic intermediates were formed during the degradation process. The degradation pathway of phenol is shown in Figure 4a [25].

In the study by Liu et al., a composite of red mud and Prussian blue was synthesized by the co-precipitation method in an acidic solution, which

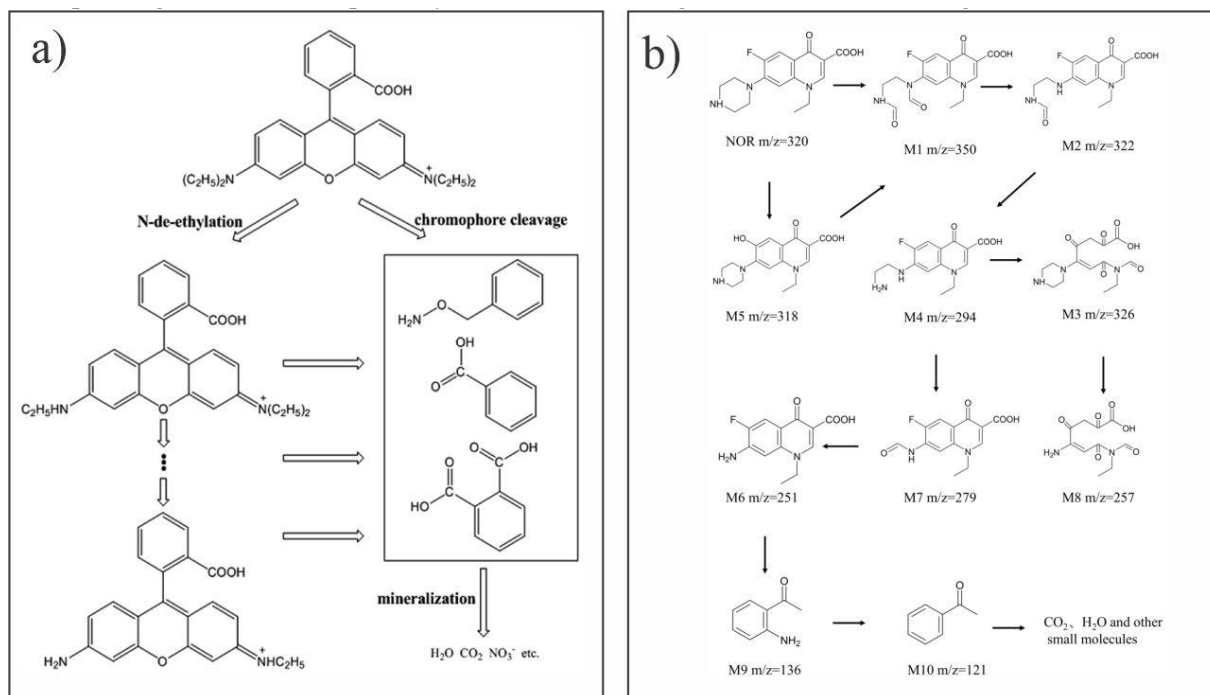


Figure 3. Reaction path of degradation: (a) rhodamine B and (b) norfloxacin. Figure (a) adapted and modified from [22]; figure (b) adapted and modified from [23].

was then used for H_2O_2 activation and norfloxacin degradation [26]. It was found that 90% norfloxacin degradation could be achieved at $\text{pH} = 5$ within 60 min in a wide pH range (3–11). Also, the removal rate of norfloxacin by the composite/ H_2O_2 system was 8.58 times and 2.62 times higher than that of the red mud/ H_2O_2 system and the Prussian blue/ H_2O_2 system due to the numerous reactive sites. The reactive oxygen species (ROS) included $^1\text{O}_2$, $\cdot\text{OH}$, $\cdot\text{O}_2^-$,

with $^1\text{O}_2$ playing a dominant role. The reaction pathway of norfloxacin degradation is shown in Figure 3b. In a similar study, a catalyst based on red mud, Prussian blue and potassium cobalt cyanide was synthesized by the acid washing-reduction-precipitation method for the degradation of ciprofloxacin (CIP) [27]. The catalyst obtained by treating with 2.4 M HCl and adding 4 mM potassium cobalt cyanide showed the best dispersibility and regular shape,

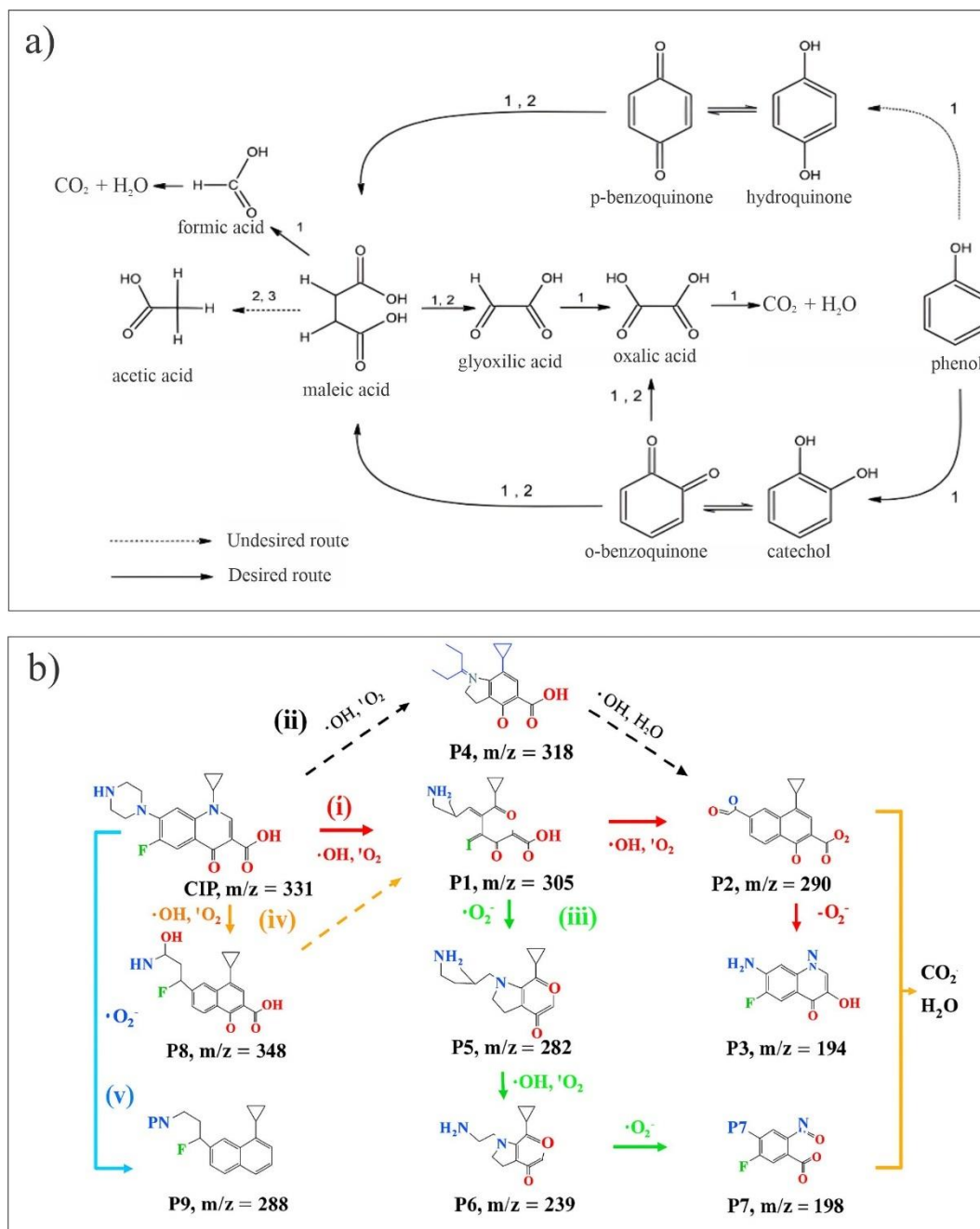


Figure 4. Reaction path of degradation: (a) phenol and (b) ciprofloxacin. Figure (a) adapted and modified from [25]; figure (b) adapted and modified from [27].

which then influenced the increase of its catalytic activity. The degradation efficiency of the composite/ H_2O_2 system reached 75.79% in 10 minutes, which was 10.54 times higher than that of the red mud/ H_2O_2 system. The catalyst was almost not affected by pH, had resistance to inorganic anions, and in addition, it could be reused. Superoxide radical ($\cdot O_2^-$) was the main ROS attacking CIP, which continuously accumulated in the process of Co(III)/Fe(III) and Co(II)/Fe(II) cycling. The degradation pathway of ciprofloxacin is shown in Figure 4b.

2.2.2. Surface modification of red mud via metal doping

Red mud can be modified by doping with metals such as Al, Co, Zn, Sn, and Ni, in order to improve its reactivity in heterogeneous Fenton advanced oxidation processes.

In the study by He et al., a catalyst based on Fe_2O_3 from red mud and Co-Al layered double hydroxide was prepared by mechanochemical synthesis, which was then used as a catalyst for a photo-Fenton system for the degradation of gatifloxacin (GAT) under visible light [28]. After a reaction time of 120 min, 94.0% of GAT (20 mg/L, 300 ml) could be removed under the optimal reaction conditions: catalyst concentration of 0.03 g/L, hydrogen peroxide concentration of 90 mmol/L, and initial pH = 6.5. Co(IV) and Fe(IV) are the active species responsible for the degradation of GAT in a weakly acidic system, while h^+ and $\cdot OH$ are the active species for the degradation of GAT in a strongly acidic system. In a similar work, the catalytic activity of $Fe_2O_3/Zn-Al$ layered double hydroxide in the photo-Fenton reaction was studied [29]. The apparent rate constant of the composite/ H_2O_2 system in the photo-Fenton reaction was about 4 times higher than that of the system containing only Zn-Al layered double hydroxide and about 10 times higher than that of the system containing only red mud (which was the main raw material). It was found that in addition to the increased surface area (45.64 m^2/g) and increased pore volume of the composite, the increased oxygen vacancy content also contributed to the improved catalytic activity of the composite.

In the work of Li et al., a catalyst was developed by doping red mud with Sn. The resulting catalyst was then used in a heterogeneous electro-Fenton

system for the removal of antibiotics [30]. The catalyst removed over 90% of nine antibiotics and two phenolic compounds from water within 90 min. The addition of Sn led to the formation of internal oxygen vacancies, which act as electron donors, significantly improving the internal electron transfer between Sn and Fe, thereby ensuring a sustainable iron cycle. An electrode with red mud particles doped with CuO prepared by the ultrasound-assisted physical impregnation method can be used in an electro-Fenton system for the degradation of ciprofloxacin (Figure 5a) [31]. The synthesized electrode had a developed mesoporous structure and high specific surface area, and doping with CuO reduced the electrode resistance and accelerated electron transfer. The high electrocatalytic activity of this system is due to the combined effects of adsorption by the particulate electrode, direct oxidation from both the anode and the particulate electrode, and indirect oxidation by active species generated in the system. Ni-doped red mud can be used in the Fenton reaction for the degradation of antibiotics in water [32]. Within 15 min, 0.02 g/L sulfamethoxazole (SMX) can be completely degraded using 0.1 g/L catalyst and 6 mM H_2O_2 . Hydroxyl radicals ($\cdot OH$) and Ni are the key contributors to the removal of SMX. This system also efficiently degraded other antibiotics such as LFX, NFX, CIP and TC, with the degradation byproducts having low or no toxicity.

2.2.3. Synthesis of red mud–biochar catalysts through co-pyrolysis

To obtain catalysts in Fenton processes, pyrolysis of red mud and lignocellulosic biomass is possible. Due to this approach, the synthesized catalyst has functional groups that enable the generation of reactive oxygen species.

In a study by Li et al., a heterogeneous Fenton catalyst with zero-valent iron was synthesized by pyrolysis (900°C) of red mud and straw (Figure 5b) [33]. At 2.86 mM H_2O_2 , 100 mg/L acid red G (ARG) and 1 g/L catalyst, 98% of acid red G was removed within 10 min. Comparing the reaction rate constants of this catalyst and that of commercial zero-valent iron, it was found to be 6.7 times higher for this catalyst. In addition to the degradation of acid red G, this catalyst also showed good degradation of antibiotics, such as sulfamethoxazole, ibuprofen and carbamaz-

epine. The degradation of contaminants was mainly due to FeO, which was used to generate $\cdot\text{OH}$, and the catalyst itself showed excellent catalytic performance after four months and after being used nine times. In another work, red mud was ultrasonically impregnated onto biochar produced by pyrolysis of rice husk waste for the removal of ciprofloxacin (CIP) from water [34]. The results showed that at pH 3, catalyst dosage of 1.0 g/L, CIP concentration of 20 mg/L, treatment time of 180 min and temperature of 50 °C, the degradation efficiency was the highest and was 75.97% for sono-Fenton (SF) and 95.65% for sono-photo-Fenton (SPF) with in situ generation of H_2O_2 during the process.

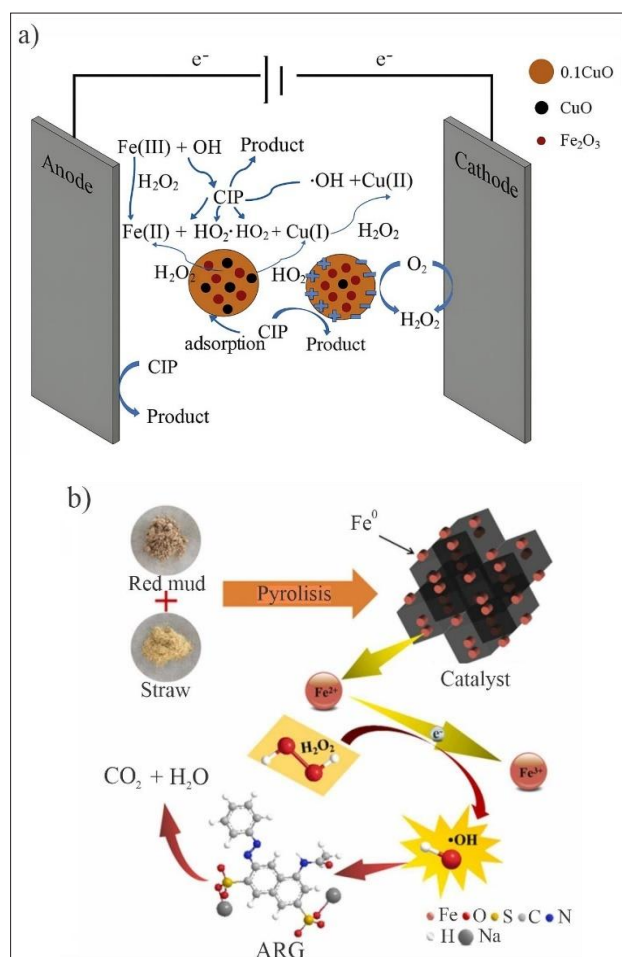


Figure 5. Contaminant removal mechanism using:
(a) CuO-doped red mud particle electrodes and
(b) red mud pyrolyzed together with biochar.
Figure (a) adapted and modified from [31];
Figure (b) adapted and modified from [33].

In the work of Lin et al., a red mud-based catalyst was synthesized by one-step co-pyrolysis of red

mud and grapefruit peel as a photo-Fenton catalyst for the activation of H_2O_2 and the degradation of acid orange 7 (AO7) [35]. The catalyst exhibited an AO7 removal efficiency of nearly 100% and a mineralization efficiency of 87%, which was maintained stable over five consecutive reuses. The synthesized catalyst provided Fe^{2+} for the activation of H_2O_2 , while light irradiation facilitated the $\text{Fe}^{2+}/\text{Fe}^{3+}$ redox cycle in the system, thereby producing more reactive oxygen species (ROS, i.e. $\cdot\text{OH}$) for the degradation of AO7. The $\cdot\text{OH}$ radical was the dominant ROS under light-free conditions, while in the light-irradiated system, $^1\text{O}_2$ was the primary ROS, followed by $\cdot\text{OH}$ and $\text{O}_2^{\cdot-}$. A composite material obtained by pyrolysis of red mud and coffee grounds can be used for the photocatalytic Fenton degradation of rhodamine B (RhB) under simulated visible light conditions [36]. During the pyrolysis process, the coffee residue is transformed into a biochar with a porous structure, and the hematite from the red mud is gradually reduced to Fe_3O_4 and Fe^0 . After 30 minutes of reaction, the catalyst showed a degradation efficiency of 94.8% under light conditions, significantly exceeding the 48.8% efficiency observed in the Fenton system that took place without light. Hydroxyl radicals ($\cdot\text{OH}$) produced by the activation of H_2O_2 by Fe^{2+} dominate the degradation of RhB. At the same time, photogenerated holes directly participate in the degradation of RhB by oxidizing OH^- ions to generate $\cdot\text{OH}$ radicals, while photogenerated electrons participate in the reaction process by promoting the $\text{Fe}^{2+}/\text{Fe}^{3+}$ cycle.

Heterogeneous photo-Fenton catalysts can be obtained by a single-step co-pyrolysis process using red mud and pomace from the distillation of alcoholic beverages [37]. The catalysts showed the best performance when the pyrolysis temperature was 900°C. For this catalyst, within 10 min, the degradation rates of tetracycline, rhodamine B, methylene blue and acid orange 7 under visible light were 91.6%, 94.5%, 77.2% and 94.2%, respectively. The primary reactive oxygen species (ROS) responsible for the degradation were $^1\text{O}_2$, followed by h^+ , $\cdot\text{OH}$ and $\text{O}_2^{\cdot-}$. In a similar work, a Fenton catalyst was developed for the degradation of sulfamethoxazole. Red mud (RM), bagasse pulp and molasses wastewater were used as raw materials, and the catalyst was synthesized through acidification and calcination steps [38]. The optimal conditions for catalyst

preparation were a mass ratio of bagasse pulp to red mud of 0.033:1, a particle size of bagasse pulp of 0.10~0.20 mm, a calcination temperature of 773 K and a calcination time of 2 h. It was found that the iron in red mud was completely transformed into $\alpha\text{-Fe}_2\text{O}_3$ after the acidification and calcination process, and the addition of bagasse pulp significantly improved the specific surface area of the prepared catalyst. By testing the catalyst for the decomposition of sulfamethoxazole, it was found that at a catalyst dosage of 2 g/L, an initial pH of 3 and a reaction time of 90 min, it showed the highest catalytic activity. The catalyst had significantly higher activity than red mud, good recyclability and stability during use. Kinetic studies showed that the degradation process can be described by a first-order model.

3. CONCLUSION

This review paper highlights the potential of red mud as a catalyst in advanced oxidation processes. Unmodified red mud, due to its iron content, is useful in heterogeneous Fenton systems, as it efficiently degrades organic pollutants such as phenols, dyes and antibiotics. Its main problem lies in its low specific surface area, low porosity and therefore insufficient active Fe^{2+} sites. Therefore, its physico-chemical modification can be performed, which can include carbothermal reduction, acid washing, calcination, neutralization and precipitation. Doping with metals (e.g. with Co, Zn, Sn, Ni, Cu) and co-pyrolysis with biomass can also be used as modification techniques, as it increases the number of functional groups that can participate in the formation of reactive oxygen species. The modified catalyst degrades organic pollutants very rapidly, often achieving >90% removal within minutes. In future research, it is necessary to improve scalable processes and better elucidate the reaction mechanism. This application of red mud shows that it is a functional material, but its application as a Fenton catalyst still cannot significantly reduce the environmental pollution associated with its large-scale disposal.

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ПОТЕНЦИЈАЛ ЦРВЕНОГ МУЉА У ФЕНТОН ПРОЦЕСИМА ЗА РАЗГРАДЊУ ОРГАНСКИХ ПОЛУТАНАТА ИЗ ВОДЕ – КРАТКИ ПРЕГЛЕД

Сажетак: Фентон процеси, у којима водоник пероксид у присуству двовалентног јона жељеза генерише хидроксилне радикале ($\cdot\text{OH}$), се нашироко користе за разградњу органских загађивача (феноли, антибиотици, боје). У овом прегледном раду је анализиран црвени муљ као јефтин извор јона жељеза у Фентон процесима. Сирови црвени муљ се може без додатних модификација користити, али за повећање каталитичке ефикасности, потребна је његова модификација, која обухвата хемијску редукујућу, карботермалну обраду или допирање металима. Посебно су значајни фото-Фентон и електро-Фентон процеси, у којима црвени муљ допиран са Co, Sn или Se, или у комбинацији са редукованим графен оксидом и биоугљем, омогућавају генерисање не само хидроксилног радикала ($\cdot\text{OH}$) већ и синглетног кисеоника ($^1\text{O}_2$) и супероксидних радикала ($\text{O}_2^{\cdot-}$), постижући $\geq 99\%$ уклањања загађивача. Уједно, синтетисани катализатори су показали високу стабилност и могућност поновне употребе. На основу компаративне анализе више од 30 студија, закључује се да црвени муљ представља јефтин извор јона жељеза за хетерогене Фентон процесе, са значајним потенцијалом за индустријску примену.

Кључне ријечи: органски полутанти, Фентон процес, хидроксилни радикали, црвени муљ.

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