


# Effect of dissolved oxygen on the efficiency of electro-Fenton process on Fe<sub>2</sub>O<sub>3</sub>/graphite perforated tubular electrode

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## Abstract

Electrodes based on carbon materials modified with iron oxides or metallic iron have attracted much attention in the field of heterogeneous electro-Fenton process for the removal of various organic pollutants. In this study, perforated tubular graphite electrode modified with Fe<sub>2</sub>O<sub>3</sub> (Fe<sub>2</sub>O<sub>3</sub>/GT) by electrochemical deposition was used as a cathode material. The obtained electrode was characterized by electron microscopy, energy-dispersive spectroscopy and Raman spectroscopy. The oxidation of rhodamine B in the electro-Fenton process by bubbling air through the perforated tubular graphite cathode at different air pressures was investigated. The complete decolorization of the rhodamine B solution was achieved in 20 min of electrolysis using Fe<sub>2</sub>O<sub>3</sub>/GT as a cathode at a current density of 29.85 mA/cm<sup>2</sup> and a pressure of 0.1 MPa. The use of higher pressure leads to complications in the equipment design of the electro-Fenton process. Carrying out the electro-Fenton process at a pressure of 0.6 MPa leads to a decrease in the energy consumption by 0.07 kW·h/mg. A possible mechanism for the oxidation of rhodamine B by bubbling air through the perforated tubular graphite cathode modified with Fe<sub>2</sub>O<sub>3</sub> was proposed.

## Accompanying information

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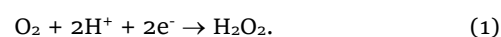
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## 1. Introduction

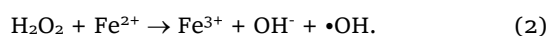
Advanced oxidation processes are currently widely used for the removal of various organic pollutants such as phenols [1,2], pesticides and insecticides [3,4], antibiotics and antiviral drugs [5–8], dyes [9–11], etc. Advanced oxidation processes are based on the generation of active oxygen species involved in the oxidation of organic pollutants. Among the active oxygen species, the hydroxyl radical (•OH) is the strongest oxidizer with a potential of 2.8 V [12, 13]. The most studied processes for •OH generation are the Fenton method and its modifications [14–16]. One of the modifications of the Fenton process is electro-Fenton, which uses the oxygen reduction reaction at the cathode to generate hydrogen peroxide and then the hydroxyl radical [17–20]. The Electro-Fenton process has attracted much attention

from researchers at present, both in terms of improving the efficiency of the process and in terms of obtaining various highly efficient oxygen reduction electrodes with simultaneous generation of •OH radicals [21–23].

Electro-Fenton, along with anodic oxidation, is one of the widely used advanced oxidation processes for the removal of organic compounds [24–27]. The process is based on the electrochemical generation of the Fenton reagent. In the implementation of the electro-Fenton process, hydrogen peroxide is formed by the reduction of oxygen [28, 29]:



Electrochemically generated H<sub>2</sub>O<sub>2</sub> reacts with an externally added catalyst (Fe<sup>2+</sup>) to form •OH homogeneously via the Fenton reaction:



Continuous formation of  $\bullet\text{OH}$  is then ensured by electroregeneration of  $\text{Fe}^{2+}$  from the reduction of  $\text{Fe}^{3+}$  formed in the Fenton reaction



$\bullet\text{OH}$  further reacts with organic pollutants, resulting in their oxidation to biodegradable species, which can be further removed by biological post-treatment [30]. The most optimal pH value is around 3 for the electro-Fenton process to occur. At higher pH values, the catalytic  $\text{Fe}^{2+}$  ions may precipitate to form iron-containing sludge. Based on this, it was proposed to use reactions of heterogeneous generation of  $\bullet\text{OH}$  radicals on the surface of solid iron-containing catalysts applied to the cathode surface as an alternative [31–34]. In particular, iron oxides  $\alpha\text{-Fe}_2\text{O}_3$ ,  $\gamma\text{-Fe}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ , etc. are widely used for this purpose on various carriers [35–38]. In this case,  $\text{Fe}_2\text{O}_3$  acts as a heterogeneous catalyst for the decomposition of hydrogen peroxide to form  $\bullet\text{OH}$  [39, 40].

In this aspect, in recent years, studies have been carried out to the selection of cathode materials for the electro-Fenton process [41–43]. Carbon based materials are widely used as cathode materials for the generation of hydrogen peroxide by oxygen reduction [44–48]. Electrodes based on carbon and graphite fibers [49–51], carbon felt [52, 53], carbon nano-tubes [13], carbon aerogel [54], etc. were studied as cathode materials in the electro-Fenton process [55, 56]. Modification of cathode materials with iron compounds or zero-valent iron allows the generation of hydroxyl radicals with simultaneous reduction of oxygen to hydrogen peroxide [57–60]. For example, the authors of the work [61] suggested using the carbon felt loaded with zero-valent iron and iron oxide as a cathode material in the electro-Fenton process for the removal of bisphenol A. Etched graphite felt was used as a matrix material for the deposition of  $\text{Fe}_2\text{O}_3$  nanoparticles doped with Cu and then – as a cathode material in the electro-Fenton process for the removal of sulfamethoxazole [62]. A carbon felt cathode coated with iron oxides was prepared by electrodeposition of  $\text{Fe}^{3+}$ . The fabricated cathodes were used in the oxidation of malachite green dye at pH 3.0 using heterogeneous electro-Fenton and photo-electro-Fenton in a stirred reactor [63–66].

Although the fabricated electrodes have a large surface area, there is still the problem with low hydrogen peroxide yield. Hydrogen peroxide generation by oxygen reduction is a key reaction to increase the efficiency of the electro-Fenton process [67, 68]. Oxygen supply is one of the factors that increase the productivity of the electro-Fenton process. For example, oxygen bubbling in the electrochemical reactor leads to an increase the concentration of dissolved oxygen and the mass transfer rate, which ultimately leads to an increase in the efficiency of the process [55, 69]. To do this, different types of reactors were developed [70, 71],

and the surface of cathode materials was modified [72]. In this regard, the attention of researchers is directed to the creation of different types of reactors with high oxygen reduction rates, and the most promising solution is to supply oxygen to the electrochemical reactor at different rates [73, 74].

In this study, a perforated tubular graphite cathode was developed through which oxygen was bubbled into the electrochemical reactor. To increase the rate of hydroxyl radical generation, the graphite electrode was modified with  $\text{Fe}_2\text{O}_3$  by electrochemical deposition. Rhodamine B (RhB) was chosen as a model pollutant. The impact of various operating parameters such as air pressure on the removal efficiency of RhB was investigated.

## 2. Experimental

### 2.1. $\text{Fe}_2\text{O}_3/\text{GT}$ electrode preparation

The graphite electrode was modified by electrochemical reduction of iron oxide (III) from a pyrophosphate solution. Graphite tubes, previously sealed hermetically on both sides to prevent iron deposition on the inner surface of the tube, were used to apply  $\text{Fe}_2\text{O}_3$ . Electrolysis was preliminarily carried out in a solution containing  $\text{FeSO}_4$  with a concentration of 2.0 g/l,  $\text{K}_4\text{P}_2\text{O}_7$  with a concentration of 10 g/l and  $\text{NaOH}$  – 1 g/l. The resulting electrode was annealed at a temperature of 450 °C. After this, the tubular graphite electrode was perforated.

### 2.2. $\text{Fe}_2\text{O}_3/\text{GT}$ characterization

The morphology of the samples was characterized using an Aspek ExPress VP scanning electron microscope-microanalyzer (FEI, USA). The structures were characterized by Raman spectroscopy using an Ntegra Spectra research complex of probe-based confocal laser microscopy (ZAO NTI, Russia) with excitation by a laser of  $\lambda = 532$  nm.

### 2.3. Electro-Fenton process efficiency

Graphite tubes (China) were used as electrode materials. The diameter of the graphite tube was 1 cm, the length was 25 cm. A thread was cut on the graphite cathode for connecting the nozzle for supplying air under pressure. The graphite tubes were placed in a polyethylene vessel with a wall thickness of 10 mm and a length of 15 cm, where four perforated graphite tubes were placed vertically. The distance between the electrodes was 0.5 cm. Compressed atmospheric air was passed through the perforated graphite tubes. The principle of electrolysis under pressure is described in detail in our work [75].

The efficiency of the electro-Fenton process was estimated by oxidation rhodamine B in 0.1 M  $\text{Na}_2\text{SO}_4$  solution. Oxygen was bubbled into the electrochemical reactor using a compressor. The dye concentration was determined using a pre-built calibration curve. The optical density of RhB was measured using a scanning UV/Vis

spectrophotometer of the SF-2000 series (Russia). After the measurement, the solution was poured back into the electrochemical reactor, and the process was continued.

The calculation of energy consumption was carried out using the following equation:

$$EC = \frac{U \cdot I \cdot t}{\Delta C}, \quad (4)$$

where  $U$  – voltage, V;  $I$  – current, A;  $t$  – electrolysis time, h;  $\Delta C$  – the concentration change, mg.

### 3. Results and Discussion

The morphology of the electrode was characterized by investigated various areas of the electrode surface. Figure 1 shows an image of the surface, EDX and Raman spectra of the selected area of the cathode surface.

As can be seen from Figure 1a, spherical particles with a size of about 1.85  $\mu\text{m}$  are formed on the surface of the graphite electrode. These particles according to the EDX spectra correspond to iron (III) oxide. The EDX spectra shown in Figure 1b correspond to the area of the electrode surface marked in the SEM image. The iron content in this area is 7 wt.% (Figure 1b). The presence of oxygen and iron in this area indirectly confirms the formation of iron oxide on the electrode surface. White inclusions on the electrode surface apparently correspond to iron oxides. Thus, during the electrochemical reduction of iron from a pyrophosphate electrolyte with subsequent heating at 450  $^{\circ}\text{C}$  in atmospheric air, iron is deposited on the surface of the graphite electrode in the form of iron (III) oxide microparticles.

The Raman spectra were obtained for the determination of the phase modification of  $\text{Fe}_2\text{O}_3$  on the graphite surface.

As can be seen from Figure 1d, two bands at 1347  $\text{cm}^{-1}$  and 1589  $\text{cm}^{-1}$  are attributed to the  $\text{sp}^3$  and  $\text{sp}^2$  states of carbon in graphite [76]. The peaks at 342.3  $\text{cm}^{-1}$  and 738.9  $\text{cm}^{-1}$  are characteristic bands for maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) [77]. The mode at 1020  $\text{cm}^{-1}$  is characteristic of hydrated phosphate bonds (P-OH bonds) [78] and shows the absorption of the pyrophosphate electrolyte by graphite.

The obtained electrode was used to study the process of electrochemical reduction of oxygen to hydrogen peroxide. The reduction of oxygen to hydrogen peroxide plays an important role in the electro-Fenton process [79]. In this

case, iron (III) oxide deposited to the graphite surface is a catalyst for the decomposition of hydrogen peroxide formed due to the reduction of oxygen. In this case, highly active hydroxyl radicals are formed, participating in the oxidation of organic compounds.

The initial electrical resistance of the graphite tube was 4.6 Ohm; the deposition of  $\text{Fe}_2\text{O}_3$  leads to an increase in resistance to 1500 kOhm when measuring the contact between  $\text{Fe}_2\text{O}_3$  and the graphite substrate. However, such a high resistance does not affect the overall energy consumption of the electro-Fenton process using the resulting electrode, since  $\text{Fe}_2\text{O}_3$  was not applied to the entire surface of the electrode. The internal part of the graphite tube remains uncoated, and  $\text{Fe}_2\text{O}_3$  was deposited only on some areas of the graphite.

As shown in Figure 2 by the cathodic voltammetry curves on  $\text{Fe}_2\text{O}_3$  modified graphite in 0.1 M  $\text{Na}_2\text{SO}_4$  solution, oxygen reduction occurs at the potential range starting from 0.1 V. The primary electrochemical process is the reduction of oxygen at the electrode.

The operating characteristics of a graphite cathode were studied during oxidation of RhB in 0.1 M  $\text{Na}_2\text{SO}_4$  solution at different air pressures at neutral pH. Figure 3a shows the kinetic curves of oxidation of the RhB using different cathode materials. As can be seen from the obtained data, the highest RhB oxidation rate is observed when using  $\text{Fe}_2\text{O}_3$  modified graphite as the cathode material with simultaneous air supply through a graphite tube under a pressure of 0.1 MPa.

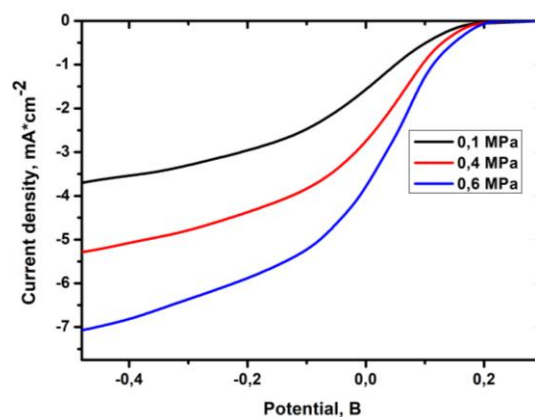


Figure 2 Oxygen reduction voltammetry curves on a graphite electrode in 0.1 M  $\text{Na}_2\text{SO}_4$  solution at different air pressures (MPa).

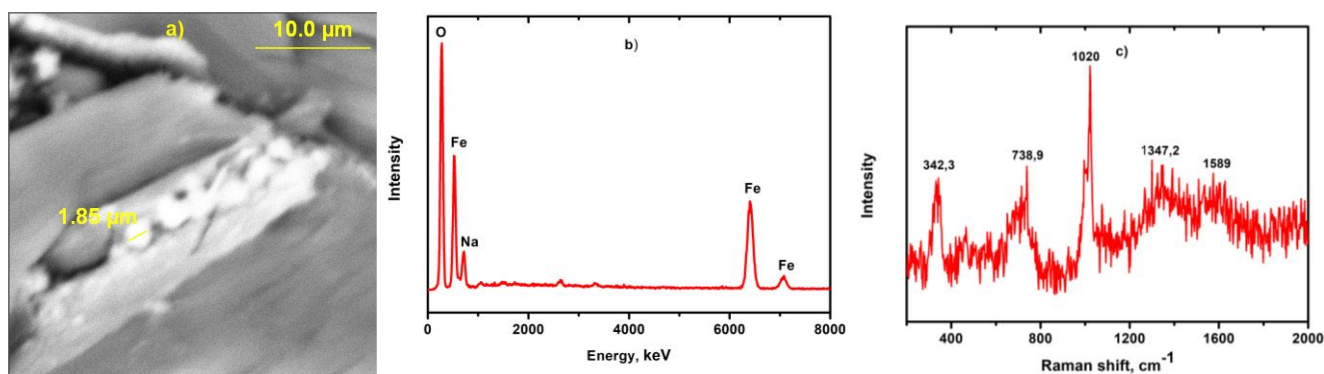
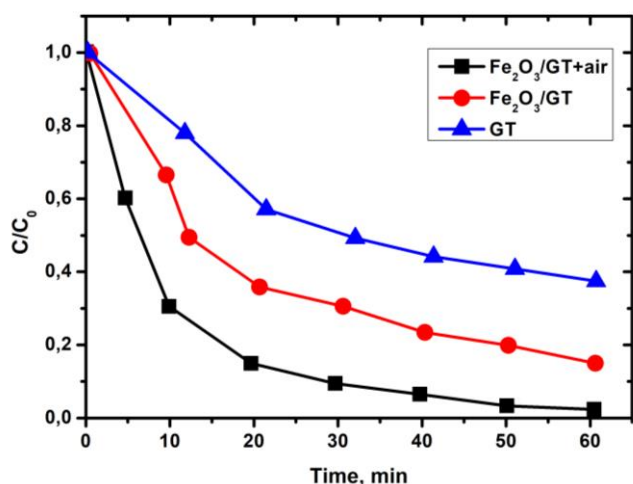


Figure 1 SEM image (a), EDX (b) and Raman spectra (c) of the selected area of the tubular graphite cathode surface, modified with  $\text{Fe}_2\text{O}_3$ .



**Figure 3** Kinetic curves of RhB oxidation during electrochemical generation of hydrogen peroxide using different cathode materials ( $C = 0.1 \text{ M Na}_2\text{SO}_4$ , anode - graphite tube,  $P = 0.1 \text{ MPa}$  with air supply;  $C = 100 \text{ mg/l}$ ).

Although the dye oxidation rate increases when using a  $\text{Fe}_2\text{O}_3$  modified graphite electrode, the  $\text{H}_2\text{O}_2$  accumulation rate is still low compared to the oxygen reduction rate. This is due to the low solubility of molecular oxygen in aqueous solutions under normal conditions ( $0.1 \text{ MPa}$  pressure and ambient temperature). The solubility of oxygen under these conditions is approximately  $40 \text{ mg}\cdot\text{l}^{-1}$  when pure oxygen is supplied and  $8 \text{ mg}\cdot\text{l}^{-1}$  when air is used [80, 81]. In addition, it should be noted that, simultaneously with the formation of hydrogen peroxide, a parasitic reaction of hydrogen evolution proceeds at the cathode, and at the anode, in addition to dye oxidation, a reaction of oxygen evolution from water occurs.

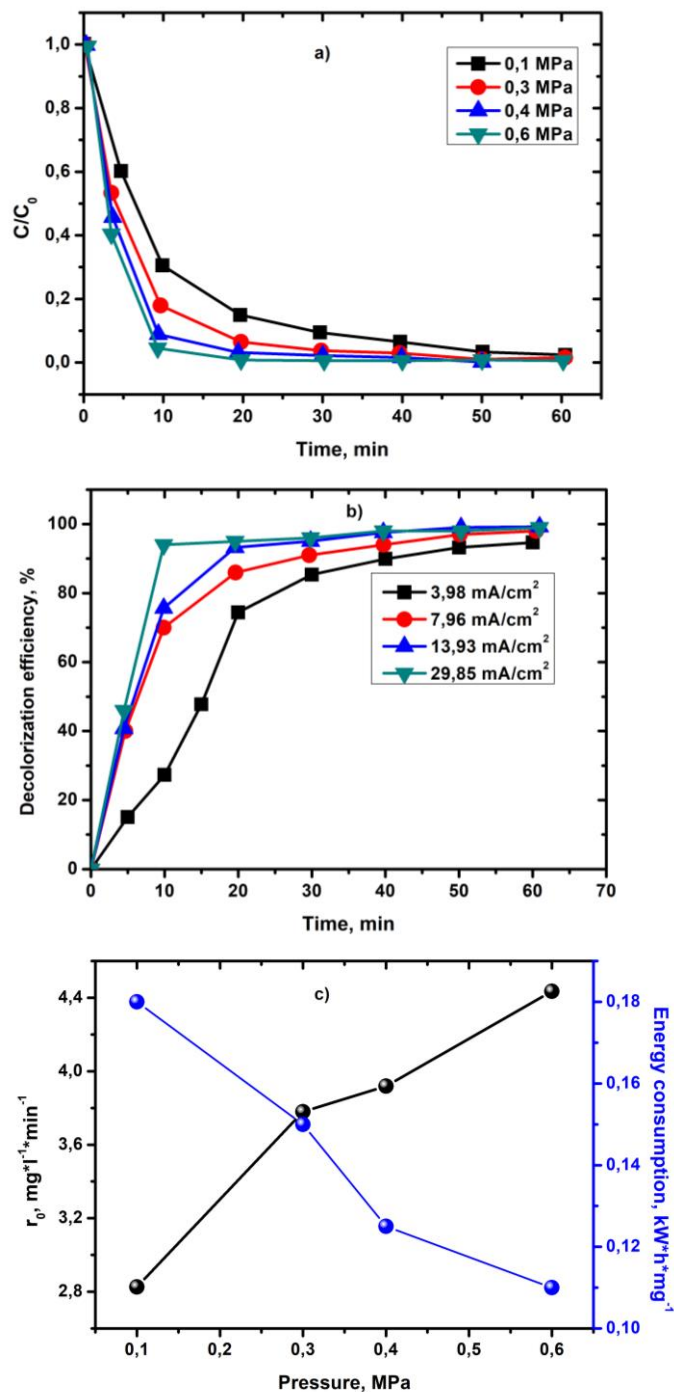
Since air supply to the electrochemical reactor through a  $\text{Fe}_2\text{O}_3$  modified tubular graphite cathode promotes the intensification of the oxidation process, we investigated the effect of the supplied air pressure on the oxidation rate of RhB due to the electro-Fenton process. The obtained experimental data in the form of kinetic curves of RhB oxidation at different pressures and the effect of pressure on the rate of the electro-Fenton process are shown in Figure 4a.

As shown in Figure 4b, the decolorization efficiency of RhB solution using  $\text{Fe}_2\text{O}_3/\text{GT}$  electrode and passing air under  $0.1 \text{ MPa}$  pressure reached 94% after only 10 minutes of treatment at a current density of  $29.85 \text{ mA/cm}^2$ . Increasing the current density leads to an increase in the rate of anodic oxidation of RhB on the graphite anode [19]. However, further increase in the current density leads to the destruction of the graphite anode.

An increase in the air pressure from  $0.1 \text{ MPa}$  to  $0.6 \text{ MPa}$  leads to an increase in the oxidation rate of the RhB by 1.6 times. Air supply at higher pressures through a tubular  $\text{Fe}_2\text{O}_3/\text{GT}$  cathode leads to an increase in the yield of hydrogen peroxide due to increasing solubility of oxygen, which promotes the rate of the electro-Fenton process

(Figure 4c). The use of high pressures leads to increased requirements for the reactor design for the electro-Fenton process. Based on this, the low air pressures ( $0.1\text{--}0.6 \text{ MPa}$ ) were used in this investigation.

The comparative data of the rate constant, decolorization of RhB and energy consumption using different cathode materials are presented in Table 1.



**Figure 4** Kinetic curves of RhB oxidation by electro-Fenton process at different air pressures ( $i = 7.96 \text{ mA/cm}^2$ ) (a); decolorization of Rhodamine B solution at different current densities (b) ( $P = 0.1 \text{ MPa}$ ) and the effect of oxygen pressure on the electro-Fenton process rate and energy consumption (c) ( $C = 0.1 \text{ M Na}_2\text{SO}_4$ , anode - graphite tube, cathode -  $\text{Fe}_2\text{O}_3/\text{GT}$ ;  $C = 100 \text{ mg/l}$ ,  $i = 29.85 \text{ mA/cm}^2$ ,  $P = 0.6 \text{ MPa}$ ).

**Table 1** Effect of cathode material on the rate constant, decolorization and energy consumption of the electro-Fenton process.

Electrode	Rate constant, $\text{min}^{-1}$	Decolorizations, %	Energy consumption, $\text{kW}\cdot\text{h}\cdot\text{mg}^{-1}$
GT	0.016	62.6	0.15
$\text{Fe}_2\text{O}_3/\text{GT}$	0.031	85.1	0.19
$\text{Fe}_2\text{O}_3/\text{GT}+\text{air}$	0.056	99.6	0.11

As can be seen from Table 1, the rate constant of the process and the degree of bleaching of the RhB dye solution increase in the range of cathode materials used GT  $\text{Fe}_2\text{O}_3/\text{GT}$   $\text{Fe}_2\text{O}_3/\text{GT}+\text{air}$ . This is due to the decomposition of hydrogen peroxide generated on the cathode to hydroxyl radicals due to the heterogeneous Fenton process on the  $\text{Fe}_2\text{O}_3$  surface [82, 83]. In this case, the supply of air under pressure leads to an increase in the yield of hydrogen peroxide and a corresponding increase in the efficiency of the RhB oxidation process. The power consumption does not change significantly when using any of the cathode materials under consideration. A slight increase when using  $\text{Fe}_2\text{O}_3/\text{GT}$  is due to an increase in resistance at the boundary between graphite and the applied  $\text{Fe}_2\text{O}_3$ . And the decrease in energy consumption with increasing pressure is associated with a decrease in the size of gas bubbles and a corresponding decrease in the resistance of the electrolyte [81].

The oxygen bubbled through the tubular cathode is reduced to hydrogen peroxide, with subsequent formation of hydroxyl radicals on the electrode surface due to the heterogeneous Fenton-like process involving  $\text{Fe}_2\text{O}_3$  [39, 40]. Hydroxyl radicals subsequently participate in the oxidation of dye molecules, ultimately forming carbon dioxide and water (Figure 5).

Carrying out the electro-Fenton process under a pressure of 0.6 MPa also allows decreasing the energy costs and energy consumption is reduced by 0.07  $\text{kW}\cdot\text{h}\cdot\text{mg}^{-1}$ . Ultimately, this can lead to decrease the cost of the

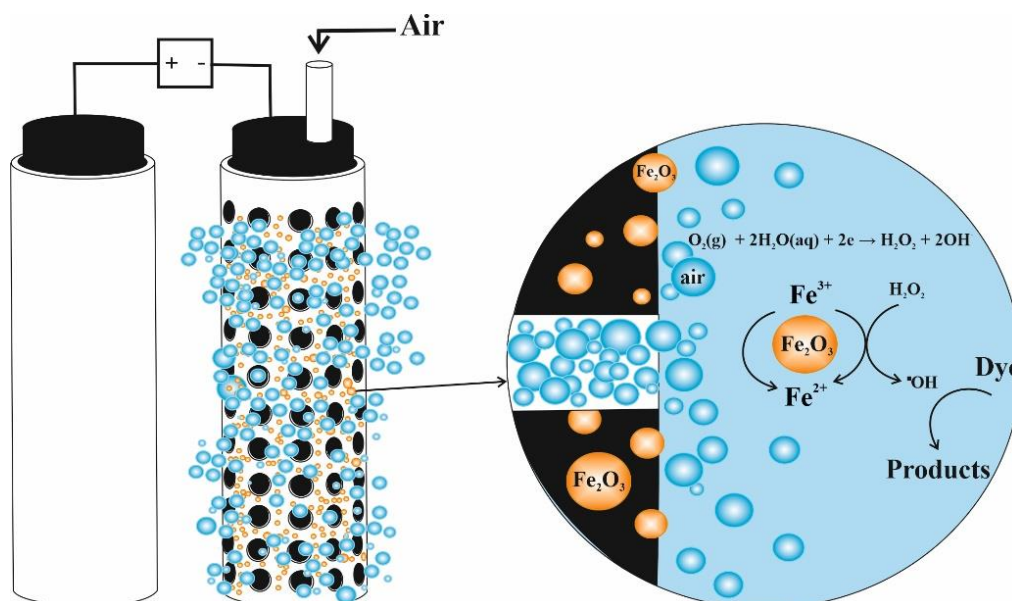
electrochemical treatment of wastewater containing various organic compounds.

#### 4. Limitations

On a graphite electrode, oxygen reduction occurs at a low rate. The use of high air pressures in the electro-Fenton process partially solves this problem, but at the same time, the hardware design of the process is complicated, associated with the use of high pressures. In addition, there is a problem of the determining the proportion of oxidized dye on the anode and due to the electro-Fenton process. In the future, we will investigate the electro-Fenton process with use a  $\text{Fe}_2\text{O}_3$  modified hydrophobized graphite electrode with air bubbled at atmospheric pressure. For the determination of the anodic and cathodic processes contribution in the oxidation of RhB, we will study the electro-Fenton process in an electrochemical reactor with separated chambers.

#### 5. Conclusions

In this study  $\text{Fe}_2\text{O}_3$  particles were deposited on the surface of a perforated tubular graphite electrode by electrochemical deposition from a pyrophosphate electrolyte. The resulting  $\text{Fe}_2\text{O}_3/\text{GT}$  electrode was used as a cathode in the electro-Fenton process. The efficiency of the electro-Fenton process was evaluated in the oxidation of Rhodamine B dye. To increase the efficiency of the electro-Fenton process, air was bubbled through the  $\text{Fe}_2\text{O}_3/\text{GT}$  perforated tubular graphite electrode at different pressures. At air pressures from 0.1 to 0.6 MPa, the electrode demonstrated an increased rate of the rhodamine B oxidation in the electro-Fenton process. Complete decolorization of the rhodamine B solution was achieved in 20 min of electrolysis at a pressure of 0.6 MPa. Carrying out the electro-Fenton process under air pressure also reduced the energy consumption.



**Figure 5** The scheme of the electro-Fenton process under air pressure using the  $\text{Fe}_2\text{O}_3/\text{graphite}$  perforated tubular electrode.

## Supplementary materials

No supplementary materials are available.

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## Data availability statement

Data will be made available on request.

## Author contributions

Conceptualization: A.I., Z.A.

Data curation: A.I.

Formal Analysis: A.I., Z.A., M.I.

Funding acquisition: A.I.

Investigation: A.I., Z.A., M.I.

Methodology: A.I., Z.A., M.I., T.A.

Project administration: A.I.

Resources: A.I., Z.A.

Software: A.I.

Supervision: A.I.

Validation: A.I., M.I.

Visualization: A.I., M.I.

Writing – original draft: Z.A., M.I., T.Kh.

Writing – review & editing: A.I., T.Kh.

## Conflict of interest

The authors declare no conflict of interest.

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