Analysis of wide diffuse halo formation in the x-ray diffraction spectrum during the reduction of Fe$_2$O$_3$ by waste-activated sludge (WAS)

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Abstract

A simulation of the diffuse experimental x-ray halo obtained when waste-activated sludge (WAS) was used as the Fe$_2$O$_3$ reducing agent made it possible to explain the large width of the experimental halo and the change in its shape as a result of the change in the amorphous phase content. The halo consists of different sets of amorphous phase convolutions. The synthesized product was a composite consisting of amorphous iron with inclusions of amorphous oxides of aluminum and silicon, amorphous carbon, and amorphous Si, Al, and C alloys on the surface of the Fe particles. The shift in the halo makes it possible to qualitatively estimate the contributions of oxide and carbon components or alloys to the halo of amorphous iron.

Introduction

Due to the significant contamination of clean water with domestic and industrial waste on the one hand, and the depletion of water resources, on the other hand, activated sludge (AS) has now become widely used for final wastewater treatment. Many microorganisms that make up the activated sludge of a biological treatment plant, being in the waste liquid, absorb pollutants into the cell, where they undergo biochemical transformations under the influence of enzymes. At the same time, organic and some types of inorganic pollutants are used by the bacterial cell in two directions: (a) biological oxidation in the presence of oxygen to harmless products of carbon dioxide and water; (b) synthesis of new cells (formation of biomass). The effectiveness of the AS is assessed by a hydrobiological analysis of the water-silt mixture by microscopy, which makes it possible to assess the degree and shelf life of the bioproduct as an adsorbent. When there is a deviation from the normal course of the technological process of biological wastewater treatment, AS is classified as waste (WAS), removed from the treatment basins, and stored at designated sites. Due to the large volume of exported WAS and the contamination of soil and water sources in places where they are stored, environmentalists are faced with the task of disposing of WAS. To date, various WAS processing technologies have been developed and are being developed. Among the simplest technology is WAS pyrolysis in an oxygen-free environment, which makes it possible to transform a bioproduct into a carbon material. Pyrolysis is a simple and exceptional technology that neutralizes environmentally harmful compounds present in WAS. The waste activated sludge (WAS) study showed [1–5] that sand and clay are present along with biocomponent. The composition of WAS used by us is presented in table 1. In addition, other impurities may be present in the WAS, but in small quantities. The presence of inorganic impurities in the WAS depends on several factors. The main sources are as follows: from what sources the polluted water comes to the city water purification stations, which way the polluted water passes before it enters the purification pools, and what elements and compounds are adsorbed by activated sludge (AS).
Table 1. Composition and type of WAS.

| Elements            | Content, % |
|---------------------|------------|
| Organic material    | 80.20      |
| Clay + sand         | 19.80      |

Because pyrolysis at 800 °C–1000 °C under oxygen-deficient conditions leads to the formation of poorly ordered reactive carbon [1], the process of WAS pyrolysis at 1000 °C is associated with the carbothermal reduction of oxide-containing compounds [6]. In this regard, the resulting carbonizate must have a complex composition (reactive carbon and oxide reduction products), and its further use as a metal oxide reducer may differ from the direct reduction of oxides with carbon. To confirm this assumption iron oxide was chosen as the reduction object since the Fe₂O₃-C system occupies a key place in ferrous metallurgy and has been studied in detail in numerous works [7, 8]. Our studies have shown that during the joint thermal treatment of Fe₂O₃-WAS mixtures at 1000 °C, an amorphous product is formed, the main component of which is Fe [9].

The purpose of this work was to elucidate the reasons for the formation of a wide diffuse halo during the reduction of Fe₂O₃ to amorphous Fe by waste-activated sludge (WAS) [9]. In [9], it was found that during the temperature treatment of the Fe₂O₃-WAS mixture in the temperature range of 800–1000 °C, the hematite reduction process proceeds according to the scheme: Fe₂O₃ → Fe₃O₄ → FeO → Fe. However, if the reduction at 800 and 900 °C is accompanied by the formation of reduction products in the crystalline state, then the temperature treatment of the mixtures at 1000 °C by ‘jumps’ leads to the formation of an amorphous material, which is characterized by a wide halo in the diffraction patterns.

The solution to this problem is supposed to be found by modeling a diffuse halo, which consists of several amorphous components included in the WAS.

Materials and methods

Compact samples were prepared from mixtures of Fe₂O₃—WAS with different contents of hematite (from 40 to 60 wt %) and WAS (from 40 to 40 wt %). The samples were subjected to temperature treatment at 1000 °C for 1, 3, and 5 h under oxygen-deficient conditions. At the end of the sintering regime, the samples were cooled to room temperature in a furnace in free mode.

The obtained specimens were investigated by x-ray diffraction (XRD) using Cu Kα radiation (Siemens D-500 diffractometer). Scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS) microanalyses were performed using Schottky FE-SEM (SU5000 HITACHI) and an LEO 1450 VP scanning electron microscope.

Two types of samples were studied in the work: in the form of thin sections and faults. In the first case, sections were made using epoxy glue. The samples were polished to remove the epoxy adhesive and the surface layer of the samples, which can contain contamination from molds, oxidation products, and macro defects in the form of pores and microcracks. The thickness of the removed layer was ~1.5 mm for a primary sample thickness of 5 mm. Manual polishing of thin sections was carried out to a mirror finish. The EDS study of the fracture surface of the samples in the topography mode was carried out for 1 month, which led to some oxidation of the fracture surface. To analyze the formation of anomalously wide halos recorded by the XRD method, we simulated superposition halos consisting of sets of amorphous compounds containing elements detected by the EDS method. A simulation of the total diffuse halo was performed for the components that were part of the Fe₂O₃—WAS mixtures, namely, Fe, Al₂O₃, SiO₂, and C. The summation was conducted for lines of Gaussian shape with a width of 2θ ~ 40°. The first intensity of the lines was considered 100%. The step-by-step summation of the two lines was conducted such that the intensity of the sum line was 100%. For example, the intensity of line 1 is 100% + the intensity of line 2 is −0%; the intensity of line 1 is 90% + the intensity of line 2 is 10%, and so, on up to the intensity of line 1 is 0%, the intensity of line 2 is 100%.

Results and discussion

X-Ray and SEM/EDS results

First, x-ray phase studies of the products of the joint treatment of Fe₂O₃—WAS mixtures prepared as described in [9], were carried out, and it was found that the main phase was an amorphous material (figure 1). The shape of the halos was dependent on the composition of the mixtures and sintering time. Moreover, as shown in (figure 2), the maxima of diffuse halos in the samples are often found at different values of 2θ.
According to the EDS analysis (table 2), the most representative element in the samples was Fe. In addition, C, Si, Al, and O were detected. The presence of these elements shows the incomplete reduction of oxide compounds by the formed carbon even at $t_{tr} = 5$ h. As shown in table 2, the contents of Si, Al, and Fe were consistent with the «trends» in the contents of WAS and Fe in the mixtures.

The SEM and EDS images presented in figures 3 and 4 show that Fe formed large dense and less dense accumulations. Between them are smaller dense accumulations consisting of elements such as Si, Al, O, and C.

### Table 2. Content of elements in specimens after temperature treatment of WAS–Fe$_2$O$_3$ mixtures at 1000 °C during 5 h according to EDS on the square 80 × 80 µm.

| Type of mixture, wt% | Fe  | C   | O   | Si  | Al  |
|---------------------|-----|-----|-----|-----|-----|
| 60 WAS–40 Fe$_2$O$_3$ | 54.11 | 15.26 | 24.01 | 1.33 | 5.28 |
| 50 WAS–50 Fe$_2$O$_3$ | 61.46 | 15.72 | 19.91 | 0.78 | 2.13 |
| 40 WAS–60 Fe$_2$O$_3$ | 76.11 | 3.53 | 18.97 | 0.74 | 1.02 |

Figure 1. X-ray diffraction patterns of treated mixtures WAS + Fe$_2$O$_3$ at 1000 °C during different time.

Figure 2. The position of the diffuse halo maximum depending on the sintering time ($t_{tr}$) of various mixtures (a) and the content of components in mixtures (b).
Their presence shows the incomplete reduction of silica and aluminosilicate formed from clay. Figures 3, and 4 draw attention to the following: around dense accumulations of elements such as Si and Al, there are ‘rarefied’ areas of these elements (these areas are marked with an oval in figure 4). Moreover, Si, Al, and C elements are also registered on the ‘rarefied’ accumulations of Fe elements. The absence of large dense areas of concentration of iron elements, but only their ‘nuclei,’ located among a more rarefied Fe medium, indicates that we are observing a disordered system that consists of poorly ordered iron as well as silicon, aluminum, and carbon.

Figure 3. Distribution of elements on a thin section of the sample obtained from mixtures 40 wt% Fe2O3–60 wt% WAS at Ttr = 1000 °C during ttr = 1 h, 3 h, and 5 h in the map mode.
Thus, the synthesized material, in which amorphous iron with Si, Al, and C impurities predominate, is a composite containing amorphous oxides (SiO₂, Al₂O₃, SiO₂–Al₂O₃) and oxygen-free compounds (C and Al-Si-C, Fe-Al-Si-C alloys) as macro impurities.

Simulation
Since for amorphous alloys, oxide compounds, and iron, the halo maxima in the diffraction patterns are found at different values of 2\(\theta\) [10–21], which should have a large width and change the shape and position of the maximum depending on the content of certain amorphous components.

With a short processing time and a high clay and sand/silica content in the initial mixtures, the superposition halo should consist of amorphous phases such as Fe, C, SiO₂, Al₂O₃, and SiO₂–Al₂O₃. With a long processing time and high carbon content in the initial mixtures, owing to the further development of the reduction processes, compounds such as Al–Si, Al–Si–C, Fe–Si, Fe–Si–Al alloys, and more complex compounds/alloys can be obtained.

With the prevalence of amorphous oxide compounds, the main halo of amorphous Fe with a maximum

Figure 4. Distribution of elements in the zone of destruction of samples, obtained from different mixtures Fe₂O₃ - WAS at \(T_m = 1000°C\) and \(t_m = 1\) h in the map mode.
intensity at $2\theta \sim 45^\circ$ [11, 22] should be superimposed by a complex-shaped halo with maxima at $2\theta \sim 23^\circ$ (main peak) and $2\theta \sim 63^\circ$ (very weak peak) [23–25]. For amorphous carbon, the main and weaker peaks are found at 25° and 45°, respectively [19, 20, 26]. When metallic alloys predominate, the main halo of amorphous Fe should be superimposed by a halo of complex shape with maxima at $2\theta \sim 37^\circ$–45° (main peak) and 65°–80° (weak peak) [27–31]. Thus, depending on the content of oxide compounds, alloys of elements, and the presence of amorphous carbon, the shape of the composite halo should change, and its maximum should shift slightly.

The simulation of binary compositions showed that when the content of oxides is more than 30% in a mixture of amorphous Fe-(Si + Al) oxides (figure 5(a)), the maximum of the total halo shifts to the region of lower values of $2\theta$ (figure 6(a)), the shape of the halo and its width change. However, when the oxide content was more than 70%, the total halo narrowed, and there was a gradual transition to the form of a halo of amorphous oxide compounds (see figure 5(a)).
When halos of Fe-C mixtures are summed (figure 5(b)), the nature of changes in the total halo is similar to the case presented in figure 5(a): at a C content of more than 20%, the halo maximum shifts to the lower values of $2\theta$ (see figure 6(b)), the shape of the halo and its width change (see figure 5(b)). At a carbon content of more than 80%, the halo shape and maximum approach that of amorphous carbon.

For the C-oxide mixtures, there was only a small difference between the maxima of the respective compounds (figure 5(c)). The shift in the total maximum compared to the maximum for amorphous carbon is noticeable only at an oxide content of more than 25% (see figure 6(c)). In this case, the width of the total halo did not change significantly. With an increase in the oxide content of more than 50%, a narrowing of the total halo is noted, and a gradual manifestation of the second maximum, characteristic of oxides, is noted.

For the Fe-alloy mixtures (figure 5(d)), the shift of the total maximum towards higher values of $2\theta$ becomes noticeable at an alloy content of more than 10% (see figure 6(d)) and is accompanied by a broadening of the total amorphous band up to an alloy content of 70%–80% (see figure 5(d)).

For the Fe alloy composition (figure 5(e)), the shift of the total maximum to lower values of $2\theta$ becomes noticeable only when the alloy content is more than 50% (see figure 6(e)) and is accompanied by a broadening of the total halo (see figure 5(e)).

For oxide–alloy mixtures (figure 5(f)), the position of the component maxima is significantly different. As the content of alloys in the binary system increases, the shape of the total halo, its maximum, and its width change significantly (see figures 5(f), 6(f)). When the alloy content was more than 60%, a wide bell-shaped halo

![Figure 6. The maximum position of simulated diffuse halos depending on composition of mixtures: Fe:(Si-Al-O) oxides (a); Fe: carbon (b); carbon:alloys (c); carbon:alloys (d); Fe: alloys (e); oxides: alloys (f).](image-url)
was formed.

Based on the simulation results, it can be concluded that the greatest influence on the change in the shape, width, and position of the superposition halo maximum is exerted by the components for which the shape of the individual halos and the meaning of their maxima are significantly different. These include binary mixtures of iron-oxides, iron-carbon, carbon-alloys, and oxide-alloys. Because there are only a few differences between the total halos of iron-oxide mixtures and oxide-alloy mixtures, they can be considered to coincide. In turn, the total halos for the iron-carbon mixtures and carbon-alloy mixtures were similar. As a result, it is possible to proceed to the modeling of ternary iron-(oxides + carbon)-carbon systems or iron-oxides’-alloys systems.

Ternary mixture halo modeling is based on the halo summation of iron-oxide mixtures (see figure 5) with different contributions of carbon and alloy halos. Simulation results are shown in figure 7. The change in the position of the maximum total halo is shown in figure 8.
The obtained simulation results showed that with the simultaneous presence of different amorphous compounds in the specimens, wide halos of complex shapes were observed in the XRD patterns. The shift of the total halo maximum to the range of small values of $2\theta$ compared to $2\theta = 45^\circ$ (for amorphous iron) indicates the presence of oxide compounds and carbon in specimens. The shift of the halo maximum to the region $2\theta = 45^\circ$ indicates the appearance of alloys. But the manifestation of a weaker peak in the region $2\theta \sim 65^\circ$–$80^\circ$ for alloys makes it possible to establish their presence in the synthesized samples. Let us turn to the diffraction patterns of the synthesized samples (shown in figure 1). Given that the main component of the composites is Fe (see table 2), and then followed in descending order by the content of elements such as C, Al, and Si, it can be concluded that a comparison of experimental halos should be carried out with simulated halos in which the iron content lies in the range 80%–50%, carbon 15%–5%, and oxides 5%–1% (see figures 7(a), (b), (d), (e)). In this case, the position of the halo maxima should be considered (figures 2 and 8).

From the type of simulated superposition halos consisting of amorphous iron, oxides, and alloys (some of them are shown in figure 7), it can be concluded that the shape of the halos and the position of its maximum change significantly if the content of amorphous Fe prevails over the content of oxides or/and carbon, or their combined value (see figures 7(a)–(c)). The shape of the total halo and the position of its maximum in the case of an increase in the presence of alloys, even in the presence of amorphous oxides and carbon, are largely determined by the content of amorphous iron (see figures 7(d)–(f)). The presence of the alloys themselves can be judged by the appearance of a weak shoulder in the region $2\theta \sim 70^\circ$–$80^\circ$.

Thus, we can proceed to modeling a ternary mixture consisting of amorphous (iron+alloys)-(oxides +carbon)-alloys $\rightarrow$ iron-oxides-alloys. This implies that in the diffraction patterns at $2\theta \sim 45^\circ$ the intensity of the iron halo is actually the sum of the intensities of iron and alloys. In the region $2\theta \sim 25^\circ$, the intensity of halo oxides is the sum of the intensities of oxides and carbon. Only in the region $2\theta \sim 80^\circ$ does the intensity of the weak peak reflect the actual content of the alloys. This simplification allows us to move on to modeling a superposition diffuse halo consisting of three components (see figure 9).
Simulation of superposition diffuse x-ray halos consisting of three components shown in figure 9 made it possible to establish such a ratio of amorphous phases that fully correlated with experimentally detected halos (see figure 10). The performed simulation made it possible to a rough estimate of the contribution of each of the amorphous phases to the experimental halos (see table 3).

**Conclusions**

A study of the products of sintering (heat treatment) of Fe₂O₃—WAS mixtures under conditions of oxygen deficiency showed that the synthesized product is an amorphous material, the main component of which is iron. Along with iron, the synthesis product contains inclusions in the form of aluminum and silicon oxides, SiO₂—Al₂O₃ compounds, carbon inclusions, and complex alloys. The absence of crystalline phases in the corresponding compounds indicated their amorphous state. It is suggested that the formation of a wide diffuse halo is due to the superposition of various amorphous states.
### Table 3. Simulated halo shape and its components.

| Type of initial mixtures \( t_{\text{ox}} \text{h} \) | 60 wt.%\( \text{Fe}_2\text{O}_3 \)–40 wt.%\( \text{WAS} \) | 50 wt.%\( \text{Fe}_2\text{O}_3 \)–50 wt.%\( \text{WAS} \) | 40 wt.%\( \text{Fe}_2\text{O}_3 \)–60 wt.%\( \text{WAS} \) |
|---|---|---|---|
| 60 wt.%\( \text{Fe}_2\text{O}_3 \)–40 wt.%\( \text{WAS} \) | 31 | 71.7 | 27.3 | 35 | 47 | 18 | 34.5 | 55 | 10.5 |
| 50 wt.%\( \text{Fe}_2\text{O}_3 \)–50 wt.%\( \text{WAS} \) | 34.2 | 58.87 | 14.9 | 33 | 34 | 32 | 34.5 | 33.3 | 32.1 |
| 40 wt.%\( \text{Fe}_2\text{O}_3 \)–60 wt.%\( \text{WAS} \) | 15.6 | 57.8 | 26.5 | 41 | 28 | 31 | 44.84 | 35.77 | 19.37 |

Note: the contributions of amorphous phases in relative units were estimated based on the area \( S \) under the corresponding halos. \( S = I \times B \), where \( I \) is the halo amplitude/intensity, \( B \) is the halo width. The square/content of different amorphous phases normalized to \( S \).
Simulation of a superposition of diffuse x-ray halos, consisting of different sets of halos (amorphous phases) belonging to iron, Si, and Al oxides, carbon, and alloys based on Fe–Si–Al–C and located at different $2θ^°$, made it possible to identify three main areas, the overlap of which predetermines the formation of wide halos. Namely, the amorphous phases of oxides and carbon are the basis for the formation of a common main halo at $2θ^°\sim(23–25)^°$, and the amorphous phases of iron and alloys are the basis for the formation of a common main halo at $2θ^°\sim(37–45)^°$. However, the amorphous phases of the alloys additionally form a common less intense halo in the region $2θ^°\sim(65–80)^°$. It has been established that the shape of the superposition halos and the position of their maxima depends on the contribution of individual amorphous components.

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Ethical Compliance: All procedures performed in this study involving human participants were in accordance with the ethical standards of the institutional and/or national research committee and with the 1964 Helsinki Declaration and its later amendments or comparable ethical standards.

Conflict of Interest Declaration: The authors declare that they have no affiliations with or involvement in any organization or entity with any financial interest in the subject matter or materials discussed in this manuscript.

Data availability statement
The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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