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## Production of iron oxide pigment from the metallic component of ilmenite smelting

B.K. Kenzhaliyev, A.A. Ultarakova\*, N.G. Lokhova, K.K. Kassymzhanov, A.O. Mukangaliyeva

*Institute of Metallurgy and Ore Beneficiation, Almaty, Kazakhstan*

\*Corresponding author: [ult.alma@mail.ru](mailto:ult.alma@mail.ru)

**Abstract.** The smelting of off-grade ilmenite concentrates from the Obukhovskoye deposit results in the generation of significant quantities of reduced iron, which presents an opportunity for its utilization in the production of iron oxide pigments. The transformation of industrial waste into value-added materials aligns with contemporary trends in sustainable materials science. This study investigates the precipitation of divalent iron from sulfuric and hydrochloric acid solutions using ammonia to form goethite, focusing on the influence of magnesium impurities on the precipitation process. The presence of magnesium was found to inhibit the formation of goethite, leading to a significant reduction in pigment yield and quality. A ferrous solution was prepared by dissolving finely divided reduced iron in sulfuric acid, followed by the precipitation of iron oxides using a 25% ammonia solution. The synthesized iron oxide pigment was further refined through hydrogen peroxide treatment, ensuring a more uniform pigment composition and improved color stability. This approach offers a viable method for the recycling of industrial by-products while simultaneously addressing environmental concerns related to waste disposal. The findings contribute to the advancement of resource-efficient pigment synthesis techniques, demonstrating the potential for utilizing metallurgical waste as a precursor for high-quality iron oxide pigments suitable for various industrial applications.

**Keywords:** iron alloy, sulfuric acid, ferrous sulfate, precipitation, goethite, pigment.

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### 1. Introduction

Iron oxide-based pigments play a crucial role across various industries and scientific fields due to their unique physicochemical properties, high stability, and broad color spectrum, which can be derived from both natural and synthetic sources. However, synthetic pigments, particularly those based on iron oxides, offer several advantages that significantly expand their applications while ensuring consistent product quality [1,2].

Yellow iron oxide ( $\alpha$ -FeOOH), also known as goethite, is one of the most significant pigments in industrial applications. Its distinctive properties provide a stable yellow color, making it highly sought after in the production of paints, coatings, plastics, and even cosmetics [3]. An essential aspect of goethite's application is its thermal stability and chemical inertness, which render it preferable for use in environments exposed to external factors such as ultraviolet radiation and atmospheric conditions. In the construction industry, yellow iron oxide is extensively used for coloring concrete and bricks, owing to its superior resistance to fading and color stability under aggressive environmental conditions [4].

Red iron oxide ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>), commonly known as hematite, is one of the most widespread and in-demand pigments due to its vibrant red hue. In industrial applications, hematite serves not only as a pigment but also as a catalyst in chemical reactions and an active material in energy storage sys-

tems, including lithium-ion batteries and supercapacitors. From a chemical standpoint,  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> exhibits exceptional chemical stability, making it indispensable in the production of durable coatings and construction materials [5,6]. Furthermore, its use in cosmetics and pharmaceuticals is attributed to its non-toxicity and safety, which is particularly critical when developing products that come into direct contact with human skin.

Black iron oxide (Fe<sub>3</sub>O<sub>4</sub>), commonly known as magnetite, stands out among iron oxide-based pigments due to its intrinsic magnetic properties. This characteristic expands its potential applications in high-tech industries, including the production of magnetic data storage devices, such as magnetic tapes and hard drives [7-10]. Additionally, Fe<sub>3</sub>O<sub>4</sub> is widely used in the manufacturing of toners for printing devices, attributed to its ability to achieve uniform pigment particle distribution on surfaces.

Magnetite has also found extensive applications in medicine, particularly in magnetic resonance imaging (MRI), where it serves as a contrast agent. Moreover, it is utilized in targeted drug delivery systems, where its high biocompatibility and surface functionalization capabilities enable its integration into nanomedicine, opening new prospects for diagnostics and therapy [11-13].

Synthetic iron oxide pigments offer distinct advantages over their natural counterparts [14]. Firstly, they ensure color

purity and stable, reproducible quality, which is particularly crucial for manufacturing processes requiring precise color control. Secondly, the ability to fine-tune the physicochemical properties of synthetic pigments allows for the development of materials with tailored characteristics, including high-temperature stability, chemical resistance, and fade resistance. This makes synthetic pigments highly preferable in industries such as high-performance paint and coating production, especially for applications under extreme operating conditions [15-18].

The incorporation of iron oxides into construction materials is driven by their high resistance to atmospheric factors and ultraviolet radiation. Concrete, bricks, and building mortars pigmented with iron oxides exhibit long-lasting durability and color retention over extended periods. This aspect is particularly important in architectural projects and urban infrastructure development, where both longevity and aesthetic appeal are critical factors [19].

In pharmaceutical and cosmetic industries, iron oxide pigments are employed due to their non-toxicity and safety for human health. These pigments are widely used in decorative cosmetics, such as eyeshadows, blushes, and lipsticks, where high purity and color stability are essential. In pharmaceuticals, iron oxides are used for tablet and capsule coloring, facilitating medication identification and enhancing consumer appeal [20-24].

Particular attention should be given to the use of iron oxides in specialized fields, such as ceramics, magnetic coatings, and toners. In ceramic manufacturing, iron oxide pigments are employed to create durable-colored glazes and enamels that retain their hues even after high-temperature firing [25,26]. In the production of magnetic coatings, iron oxides play a key role due to their ferromagnetic properties, making them indispensable for hard drives and magnetic tapes. Furthermore, in toner production for laser printers and copiers, iron oxides are utilized for their uniform distribution properties and strong adhesion to paper surfaces.

In summary, iron oxide-based pigments, both natural and synthetic, have a vast range of applications across multiple industries, including construction, chemical, pharmaceutical, and cosmetic sectors [27]. However, synthetic pigments, due to their high purity, stability, and reproducibility, open new avenues for the development of high-performance materials. Future research on iron oxide pigments will focus on the development of novel functional materials with enhanced properties, ensuring even broader applications in science and technology [28,29].

Investigating and developing new methods for obtaining iron-containing pigments from chemical and metallurgical waste is a highly relevant and pressing challenge for modern science and industry. In recent years, significant attention has been given to utilizing industrial waste as raw materials for the synthesis of high-quality pigments, which not only reduces production costs but also addresses waste disposal and recycling issues, contributing to environmental sustainability. Iron-containing pigments, such as hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) and other iron oxides, exhibit high stability, corrosion resistance, and a broad spectrum of applications, making them indispensable across multiple industrial sectors [30].

The utilization of ferrous sulfate ( $\text{FeSO}_4$ ) waste, accumulated during chemical and metallurgical processes, for the synthesis of iron-containing pigments is a promising research direction. It is well established that the two-stage synthesis

method for iron red pigment from long-term stored ferrous sulfate waste involves the stages of purification and precipitation [31]. The first stage-purification of ferrous sulfate waste is a crucial procedure to obtain high-quality raw materials suitable for further use. Modern purification methods, including the application of microwave reactors, significantly accelerate and enhance the efficiency of iron salt purification, representing a key step in achieving high-purity final products.

The use of a microwave reactor for the synthesis of iron oxide-based pigments allows for a substantial reduction in the phase transition temperature of goethite ( $\alpha\text{-FeOOH}$ ) to hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ). While traditional methods require temperatures of approximately  $500^\circ\text{C}$ , the microwave method reduces this threshold to  $170^\circ\text{C}$ , thereby eliminating the need for additional thermal processing or calcination. This represents a significant breakthrough not only in terms of energy efficiency but also in improving the physicochemical properties of the synthesized materials. Lowering the synthesis temperature reduces the formation of pigment agglomerates, directly influencing its quality and coloration ability. As a result, the synthesized materials exhibit more stable and homogeneous characteristics compared to commercial analogs, as confirmed by several studies [32].

The synthesis of pigments from ferrous sulfate waste has proven to be highly efficient and serves as an effective solution for industrial waste recycling. This approach offers broad prospects for the industrial-scale production of iron-based pigments, which can be applied in construction materials, paint and coating industries, and several other sectors. Laboratory studies indicate that synthesized pigments exhibit notable advantages over commercial products, making their utilization highly attractive from both technological and economic standpoints [33].

Another important development is the production of anti-corrosion pigments based on hematite derived from steel industry waste. A specific method has been developed for obtaining pigments from powdered residues formed during the rolling of reinforcement steel, including the use of rust and mill scale [34]. These waste materials typically contain a high percentage of iron oxides, primarily  $\text{Fe}_2\text{O}_3$ , along with other oxide impurities such as  $\text{SiO}_2$ ,  $\text{CaO}$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{MnO}$ , which may influence the final pigment properties. Notably, the initial waste material contains various crystalline phases of iron oxides, including magnetite, maghemite, lepidocrocite, wüstite, goethite, and hematite. The presence of these phases highlights the complex structure and rich composition of the raw materials, necessitating precise control over temperature conditions and retention time during calcination to produce a homogeneous pigment with high hematite content.

The pigment synthesis process involves calcining the raw material at different temperatures, allowing for the regulation of hematite content in the final product. The highest hematite concentration ( $\alpha\text{-Fe}_2\text{O}_3$ ) is achieved by calcination at  $850^\circ\text{C}$  for 1 hour, emphasizing the need to optimize processing conditions to obtain the highest-quality pigment. This method is highly efficient, enabling the production of anticorrosion pigments with enhanced properties. This is particularly critical for applications in aggressive environments where corrosion resistance is essential.

Thus, modern methods for producing iron-containing pigments from industrial waste represent a critical advancement in the development of recycling technologies and the utilization of secondary resources. These methods not only address

waste disposal issues but also contribute to the creation of new materials with superior performance characteristics, making them highly valuable across various scientific and industrial fields. The implementation of such methods can significantly reduce production costs, improve product quality, and minimize environmental impact, making these innovations highly promising for future industrial applications.

## 2. Materials and methods

### 2.1. Precipitation of pure components from iron-containing acidic solutions

Currently, the processing of titanium-containing concentrates (titanomagnetite, ilmenite, rutile) with high chromium, vanadium, and manganese content is gaining increasing relevance in global industrial practice [14].

The object of this study was model iron-containing acidic solutions.

Analysis Methods: X-ray phase analysis of the sludge was performed using a D8 Advance diffractometer (BRUKER) with Cu-K $\alpha$  radiation. The obtained diffraction data were processed and interplanar spacing calculations were carried out using EVA software. Sample decoding and phase identification were performed using the Search/Match program with the ASTM Card Database.

X-ray fluorescence analysis was conducted using a wavelength-dispersive spectrometer Venus 200 (PANalytical B.V., Netherlands).

The chemical composition of the samples was determined by optical emission spectrometry with inductively coupled plasma (ICP-OES) using an Optima 2000 DV spectrometer (Perkin Elmer, USA).

Experimental Procedure: The following equipment was used for the experiments: pH meter Mark 901 (Russia), Laboratory hot plate Arc Velp (Italy), Magnetic stirrer Stirrer ES Velp (Italy). Model solutions of 0.1N hydrochloric acid (pH 1.2) and 0.1N sulfuric acid (pH 1.34) were prepared. Ferrous sulfate heptahydrate (FeSO $_4$ ×7H $_2$ O) was added to the solutions in the required quantity to achieve an iron concentration of 50 g/L. After the addition of ferrous sulfate heptahydrate, the pH of the hydrochloric acid solution increased to 1.61, while the sulfuric acid solution reached 1.7. The hydrochloric acid solution was acidified to pH -1.0 using 10% HCl, and the sulfuric acid solution was adjusted to pH -1.0 with 10% H $_2$ SO $_4$ .

The precipitation of iron oxides from these solutions was carried out by the gradual addition of a 25% ammonia solution at temperatures ranging from 25°C to 55°C. A 250 mL heat-resistant beaker containing the prepared model solution was heated to the required temperature using a laboratory hot plate, while stirring at 200 rpm. Ammonia was added dropwise using a glass pipette until the pH reached 3. The resulting solution with the precipitate was filtered, and the solid residue (cake) was dried at 105°C for 2 hours.

## 3. Results and discussion

The results of precipitation experiments from hydrochloric acid solutions are presented in Table 1. Fe $^{3+}$ O(OH). When the temperature was increased to 55°C, somalnokite (syn.  $\epsilon$ -Fe $_2$ O $_3$ , iron (III) oxide) was formed. Similar precipitation experiments were conducted using sulfuric acid solutions, adjusting the pH to 4.5. The results of iron oxide precipitation using a 25% ammonia solution from sulfuric acid solutions in the temperature range of 25-55°C are presented in Table 2.

According to the X-ray phase analysis results, the precipitates obtained over the entire temperature range were identified as bernallite Fe $^{3+}$ (OH) $_3$ .

The third series of experiments involved the precipitation of iron oxides from an iron-hydrochloric acid solution, in which MgCl $_2$  was added to obtain a magnesium concentration of 10.1 g/dm $^3$ , with an initial solution pH of -1. The precipitation process was conducted at temperatures ranging from 25 to 55°C, with the addition of a 25% ammonia solution to adjust the pH to 4.5. The experiment lasted for 4 hours. The solution containing the precipitate was filtered, and the precipitate was dried at 105°C for 2 hours. The results of these experiments are presented in Table 3.

Table 4 shows the changes in X-ray phase analysis as the temperature of iron oxide precipitation increases.

Due to the high content of the amorphous component, X-ray fluorescence analysis could not be performed on the precipitates obtained at 25°C and 35°C. In the precipitate obtained at 45°C, goethite Fe $^{3+}$ O(OH) and magnesium sulfate hydrate MgSO $_4$ ×2H $_2$ O were identified. The precipitate obtained at 55°C contained goethite Fe $^{3+}$ O(OH) and iron oxide Fe $_2$ O $_3$ , with traces of MgSO $_4$ ×1.25H $_2$ O also detected. The presence of magnesium sulfate hydrate is attributed to insufficient washing of the precipitate and the presence of an amorphous phase.

Table 1. Results of iron oxide precipitation from hydrochloric acid solutions

Name	Temperature, °C	Cake weight, g	Volume, ml	Fe		Mg	
				g/dm $^3$	wt.%	g/dm $^3$	wt.%
Initial Solution			1000	50	100	0.11	100
25% Ammonia Solution	25		36				
Obtained							
Precipitate		3.28		36.1	2.4	0.03	0.9
Solution			1000	48.8	97.6	0.107	99.1
25% Ammonia Solution	35		36				
Obtained							
Precipitate		3.48		42.12	2.9	0.028	0.9
Solution			1000	48.53	97.1	1.07	99.1
25% Ammonia Solution	45		36				
Obtained							
Precipitate		3.24		40.8	2.6	0.019	0.6
Solution			1000	48.68	97.4	0.1073	99.4
25% Ammonia Solution	55		36				
Obtained							
Precipitate		3.2		43.83	2.8	0.014	0.4
Solution			1000	48.6	97.2	1.075	99.6

**Table 2. Results of iron oxide precipitation from sulfuric acid solutions**

Name	Temperature, °C	Cake weight, g	Volume, ml	Fe		Mg	
				g/dm <sup>3</sup>	wt.%	g/dm <sup>3</sup>	wt.%
Initial solution			1000	50	100	0.128	100
25% ammonia solution	25		30				
Obtained							
Precipitate		3.72		32.94	2.5	0.018	0.5
Solution			1000	48.8	97.5	0.128	99.5
25% ammonia solution	35		30				
Obtained							
Precipitate		3.44		34.7	2.4	0.023	0.6
Solution			1000	48.8	97.6	0.131	99.4
25% ammonia solution	45		30				
Obtained							
Precipitate		6.64		33.22	4.4	0.014	0.12
Solution			1000	47.8	95.6	0.192	99.88
25% ammonia solution	55		30				
Obtained							
Precipitate		4.44		31.6	2.81	0.142	0.7
Solution			1000	48.6	97.19	0.054	99.3

**Table 3. Results of iron oxide precipitation from hydrochloric acid solutions in the presence of magnesium chloride**

Name	Precipitation, temperature, °C	Precipitate weight g	Volume, ml	Fe		Mg	
				g/dm <sup>3</sup>	wt.%	g/dm <sup>3</sup>	wt.%
Initial solution			10000	50	100	10.1	100
25% ammonia solution	25	14	-	-	-	-	-
MgCl <sub>2</sub>	40	-	-	-	-	-	-
Obtained	-	-	-	-	-	-	-
Precipitate	-	3.28	-	27.35	1.9	2.60	0.9
Solution	-	10000	-	49.0	97.1	10.3	99.1
25% ammonia solution	35	14	-	-	-	-	-
MgCl <sub>2</sub>	40	-	-	-	-	-	-
Obtained	-	-	-	-	-	-	-
Precipitate	-	3.48	-	34.3	2.7	2.96	1.2
Solution	-	10000	-	48.6	97.3	10.08	98.8
25% Ammonia solution	45	14	-	-	-	-	-
MgCl <sub>2</sub>	40	-	-	-	-	-	-
Obtained	-	-	-	-	-	-	-
Precipitate	-	3.24	-	25.9	1.3	3.87	2.25
Solution	-	10000	-	48.5	96.9	9.89	97.75
Initial solution	-	-	10000	50.0	100%	10.1	100%
25% ammonia solution	25	14	-	-	-	-	-
MgCl <sub>2</sub>	40	-	-	-	-	-	-
Obtained	-	-	-	-	-	-	-
Precipitate (wt.%)	-	3.28	-	27.35	1.9	2.60	0.9

The precipitation process was carried out within the temperature range of 25-55°C, with the addition of ammonia to adjust the solution pH to 4.5. The duration of the experiment was 4 hours. The solution containing the precipitate was filtered by gravity, and the precipitate was dried at 100°C for two hours. The results of these experiments are presented in Table 5.

**Table 4. Results of X-ray phase analysis of precipitates in the presence of magnesium chloride in the temperature range of 25-55°C**

Precipitation temperature, °C	Amorphous phase content, %	Crystalline phase content, %
25	78.6	21.6
35	74.7	25.3
45	50.9	49.1
55	58.8	41.2

**Table 5. Results of iron oxide precipitation from sulfuric acid solutions in the presence of magnesium sulfate**

Name	Precipitation temperature, °C	Precipitate weight, g	Volume, ml	Fe		Mg	
				g/dm <sup>3</sup>	wt.%	g/dm <sup>3</sup>	wt.%
Initial solution			10000	50.0	100	10.0	100
25% ammonia solution	25	50	-	-	-	-	-
MgSO <sub>4</sub>	50	-	-	-	-	-	-
Obtained	-	-	-	-	-	-	-
Precipitate	-	3.24	-	30.7	2.0	1.27	0.9
Solution	-	10000	-	48.08	96.2	9.89	98.1
25% ammonia solution	35	50	-	-	-	-	-
MgSO <sub>4</sub>	50	-	-	-	-	-	-
Obtained	-	-	-	-	-	-	-
Precipitate	-	3.08	-	34.5	2.3	0.97	0.7
Solution	-	10000	-	47.9	97.8	9.86	99.3
25% ammonia solution	45	50	-	-	-	-	-
MgSO <sub>4</sub>	50	-	-	-	-	-	-
Obtained	-	-	-	-	-	-	-
Precipitate	-	3.24	-	28.6	2.0	1.27	0.9
Solution	-	10000	-	47.90	97.3	9.99	99.2
25% ammonia solution	55	50	-	-	-	-	-
MgSO <sub>4</sub>	50	-	-	-	-	-	-
Obtained	-	-	-	-	-	-	-
Precipitate	-	4.32	-	30.6	2.2	0.95	0.6
Solution	-	10000	-	47.7	97.3	9.94	99.8

Table 6 shows the changes in X-ray phase analysis as the temperature of iron oxide precipitation increases. The precipitates obtained within the temperature range of 25-55°C contain goethite  $Fe^{3+}O(OH)$  with an admixture of magnesium sulfate hydrate  $MgSO_4 \times 1.25H_2O$ .

**Table 6. Results of X-ray phase analysis of precipitates in the presence of magnesium sulfate in the temperature range of 25-55°C**

Precipitation temperature, °C	Amorphous phase content, %	Crystalline phase content, %
25	64.5	35.4
35	63.0	37.0
45	52.2	47.8
55	50.5	49.5

Thus, from pure model acidic iron-containing solutions, within the temperature range of 25-55°C, crystalline goethite and iron oxide is precipitated. However, in the presence of magnesium ions, the precipitation process results in a significant portion of amorphous precipitate, while the crystalline fraction consists of goethite and iron oxide.

### 3.1. Production of iron oxide pigment from the magnetic fraction of roasted titanium-containing product

To investigate the possibility of obtaining pigment, a batch of reduced iron obtained through the pyrometallurgical processing of ilmenite concentrate was selected. Table 7 presents the X-ray fluorescence composition of the metal. The material consisted of variously shaped droplet-like iron ingots, which were crushed and sieved through four meshes with apertures of 2 mm, 1 mm, 0.5 mm, and 0.1 mm. The results of the fractional analysis are shown in Table 8.

**Table 7. X-ray fluorescence composition of reduced iron**

Element	Content, %	Error Margin, $\pm 3\sigma$
Fe	95.74	0.14
Cr	0.17	0.086
P	0.329	0.043
Si	0.192	0.063
Mn	0.162	0.071
Cu	0.064	0.021
V	0.039	0.023
Nb	0.024	0.004
Sn	0.021	0.02

**Table 8. Fractional analysis of crushed metal**

Class	Content, %
+2 mm	3.6
-2+1 mm	4.9
-1+0,5 mm	18.0
-0,5+0,1 mm	73.5
Total:	100.0

The fine fraction was retained for pH adjustment, while the coarse fraction (+1–0.5 mm, 20 g) was placed in a beaker, and 200 mL of 15% sulfuric acid solution was added. The mixture was leached at room temperature until the complete dissolution of metallic iron. Throughout the mixing process, pH measurements were conducted and maintained at 1. If the pH increased, sulfuric acid was added dropwise, whereas if the pH dropped to 0, fine iron fraction was supplemented.

The total sulfuric acid consumption amounted to 12 mL. The solution was filtered, yielding 2.52 g of solid residue (cake) and 250 mL of solution with an iron content of 67.3 g/L. From this solution, 3.12 g of iron was precipitated using

6 mL of ammonia. A light brown goethite was obtained with impurities, and the composition of its main components is provided in Table 9. Figure 1 presents a photograph of hematite derived from the dried goethite.

**Table 9. Chemical composition of goethite, wt. %**

Component wt. %	Fe	Mg	Al	Si	Mn	V	Cr	Co	Ga	As	O
	43.4	0.033	0.013	0.06	0.05	0.13	0.1	0.083	0.012	0.007	56.11

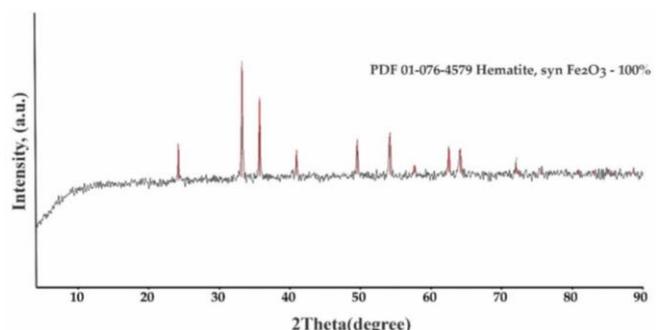


**Figure 1. Light brown hematite**

Thus, goethite with impurities was obtained, serving as a precursor for pigment production.

The practical synthesis procedure for yellow iron oxyhydroxide pigment from hematite using hydrogen peroxide involves several critical stages. First, 30 g of hematite powder is suspended in 600 mL of distilled water and heated to 35-40°C. Maintaining a precise temperature range is crucial for optimal reaction kinetics. Subsequently, 60 mL of 30% hydrogen peroxide is slowly added over 10-15 minutes under continuous stirring to ensure uniform oxidation and prevent rapid gas evolution. The mixture is then held at the target temperature for 30-60 minutes, allowing for the formation of iron oxyhydroxide, indicated by the gradual yellowing of the suspension. Upon completion of the reaction, the mixture is slightly cooled, and the solid pigment is separated from the liquid phase by filtration. The collected yellow pigment is dried at 120°C for two hours to remove residual moisture and stabilize its structure. This controlled drying process is essential to achieve the desired crystalline and chemical stability of the final product.

To ensure the scientific validity of the procedure, the dried pigment undergoes X-ray diffraction (XRD) analysis to confirm its crystalline phases, verifying the successful formation of iron oxyhydroxide. The results of the X-ray structural analysis of the iron oxide pigment are presented in Figure 2, while Figure 3 provides a photograph of the obtained pigment.



**Figure 2. X-ray diffraction pattern of hematite pigment**

Currently, the primary consumers of iron oxide pigments are the construction materials industry. Established production processes include bulk-colored paving tiles manufactured using the «Besser» technology, colored wall blocks, colored bricks, colored chalk, gypsum, plasters, and other related products.



Figure 3. Yellow iron oxide pigment

Considering that classical methods of pigment production are energy-intensive due to the high cost of reagents and the complex processing schemes for iron oxides, there is an increasing demand for simplified technologies. These alternative methods facilitate the production of pigments with a diverse color palette by utilizing various industrial waste streams as raw materials, making them highly relevant in the current industrial landscape.

#### 4. Conclusions

The results of X-ray fluorescence analysis of the precipitates obtained during iron oxide precipitation from hydrochloric acid solutions in the temperature range of 25-45°C indicate that the precipitate consists of goethite  $\text{Fe}^{3+}\text{O}(\text{OH})$ . With an increase in temperature to 55°C, somalnokite ( $\epsilon\text{-Fe}_2\text{O}_3$ , iron(III) oxide) is formed.

According to the X-ray phase analysis of the precipitates obtained from sulfuric acid solutions, bernallite  $\text{Fe}^{3+}(\text{OH})_3$  was the dominant phase throughout the entire temperature range.

During iron oxide precipitation from hydrochloric acid solutions in the presence of magnesium chloride, an increase in temperature led to a decrease in the amorphous fraction and an increase in the crystalline phase content.

Due to the high amorphous content, X-ray fluorescence analysis could not be performed on precipitates obtained at 25°C and 35°C. The precipitate obtained at 45°C was identified as goethite  $\text{Fe}^{3+}\text{O}(\text{OH})$  and magnesium sulfate hydrate  $\text{MgSO}_4 \times 2\text{H}_2\text{O}$ . The precipitate obtained at 55°C contained goethite  $\text{Fe}^{3+}\text{O}(\text{OH})$  and iron oxide  $\text{Fe}_2\text{O}_3$ , with traces of  $\text{MgSO}_4 \times 1.25\text{H}_2\text{O}$  also detected. The presence of magnesium sulfate hydrate is attributed to insufficient washing of the precipitate and the presence of an amorphous phase.

In iron oxide precipitation from sulfuric acid solutions in the presence of magnesium sulfate, a temperature increase also led to a decrease in the amorphous fraction and an increase in the crystalline phase content. The precipitates obtained in the temperature range of 25-55°C contained goethite  $\text{Fe}^{3+}\text{O}(\text{OH})$  with an admixture of magnesium sulfate hydrate  $\text{MgSO}_4 \times 1.25\text{H}_2\text{O}$ .

From pure model acidic iron-containing solutions in the temperature range of 25-55°C, crystalline goethite and iron oxide was precipitated. However, in the presence of magnesium ions, a significant portion of the precipitate was amorphous, while the crystalline fraction consisted of goethite and iron oxide.

The dissolution of the +1-0.5 mm fraction of reduced iron in a 15% sulfuric acid solution at room temperature and pH 1 was followed by iron oxide precipitation using a 25% ammonia solution. The resulting product was goethite with impurities, serving as a precursor for pigment production.

To obtain yellow iron oxide pigment, hematite powder was suspended in distilled water and heated to 35-40°C. Once the target temperature was reached, 30% hydrogen peroxide was slowly added under constant stirring, followed by a 30-60-minute reaction period. The precipitate was then separated by filtration and dried at 120°C for two hours.

#### Author contributions

Conceptualization: B.K.K., A.A.U.; Data curation: B.K.K., A.A.U.; Formal analysis: A.A.U., A.O.M., N.G.L.; Funding acquisition: B.K.K., A.A.U.; Investigation: A.A.U., N.G.L.; Methodology: A.A.U., A.O.M.; Project administration: B.K.K., A.A.U.; Resources: K.K.K., A.O.M.; Software: K.K.K., A.O.M.; Supervision: B.K.K., A.A.U.; Validation: B.K.K., A.A.U.; Visualization: K.K.K., A.O.M.; Writing – original draft: B.K.K., A.A.U.; Writing – review & editing: B.K.K., A.A.U. All authors have read and agreed to the published version of the manuscript.

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#### Conflicts of interests

The authors declare no conflict of interest.

#### Data availability statement

The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

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## Ильменит балқымасының металл құрамдас бөлігінен темір оксидті пигмент алу

Б.К. Кенжалиев, А.А.Ультаракова\*, Н.Г. Лохова, К.К. Касымжанов, А.Ө. Мұқанғалиева

Металлургия және қен байыту институты, Алматы, Қазақстан

\*Корреспонденция үшін автор: [ult.alma@mail.ru](mailto:ult.alma@mail.ru)

**Андатпа.** Обухов кен орнының кондициялық емес ильменит концентраттарын балқыту қалпына келтірілген Темірдің едәуір мөлшерінің пайда болуына әкеледі, бұл оны темір оксиді пигменттерін өндіруде қолдануға мүмкіндік береді. Өнеркәсіптік қалдықтарды қосылған құны бар материалдарға айналдыру тұрақты материалтану саласындағы қазіргі тенденцияларға сәйкес келеді. Бұл зерттеу магний қоспаларының тұндыру процесіне әсеріне ерекше назар аудара отырып, гетит түзу үшін аммиакты қолдана отырып, күкірт және тұз қышқылдарының ерітінділерінен екі валентті Темірдің тұндырылуын зерттейді. Магнийдің болуы гетиттің түзілуін тежейтіні анықталды, бұл пигменттің өнімділігі мен сапасының айтарлықтай төмендеуіне әкеледі. Құрамында темір бар ерітінді күкірт қышқылында ұсақ ұнтақталған қалпына келтірілген темірді еріту арқылы, содан кейін 25% аммиак ерітіндісін пайдаланып темір оксидтерін тұндыру арқылы дайындалды. Синтезделген темір оксиді пигменті сутегі асқын тотығымен өңдеу арқылы одан әрі тазартылды, бұл пигменттің біртекті құрамын және түс тұрақтылығын жақсартты. Бұл тәсіл қалдықтарды жоюға қатысты экологиялық мәселелерді шеше отырып, өнеркәсіптік жанама өнімдерді қайта өңдеудің өміршең әдісін ұсынады. Нәтижелер металлургиялық қалдықтарды әртүрлі өнеркәсіптік қолданбаларға жарамды жоғары сапалы темір оксиді пигменттері үшін прекурсор ретінде пайдалану әлеуетін көрсете отырып, пигментті синтездеудің ресурс тиімді әдістерін дамытуға ықпал етеді.

**Негізгі сөздер:** темір қоспасы, күкірт қышқылы, темір купоросы, тұндыру, гетит, пигмент.

## Получение железооксидного пигмента из металлической составляющей плавки ильменита

Б.К. Кенжалиев, А.А. Ультаракова\*, Н.Г. Лохова, К.К. Касымжанов, А.О. Муканғалиева

Институт металлургии и обогащения, Алматы, Казахстан

\*Автор для корреспонденции: [ult.alma@mail.ru](mailto:ult.alma@mail.ru)

**Аннотация.** Плавка некондиционных ильменитовых концентратов Обуховского месторождения приводит к образованию значительных количеств восстановленного железа, что открывает возможность его использования в производстве железооксидных пигментов. Трансформация промышленных отходов в материалы с добавленной стоимостью соответствует современным тенденциям в области устойчивого материаловедения. В данном исследовании изучается осаждение двухвалентного железа из растворов серной и соляной кислот с использованием аммиака для образования гетита, при этом особое внимание уделяется влиянию примесей магния на процесс осаждения. Было обнаружено, что присутствие магния ингибирует образование гетита, что приводит к значительному снижению выхода и качества пигмента. Железосодержащий раствор готовили путем растворения тонкоизмельченного восстановленного железа в серной кислоте с последующим осаждением оксидов железа с использованием 25%-ного раствора аммиака. Синтезированный железооксидный пигмент дополнительно очищали путем обработки перекисью водорода, что обеспечивало более однородный состав пигмента и улучшенную стабильность цвета. Этот подход предлагает жизнеспособный метод переработки промышленных побочных продуктов, одновременно решая экологические проблемы, связанные с утилизацией отходов. Результаты способствуют развитию ресурсоэффективных методов синтеза пигментов, демонстрируя потенциал использования металлургических отходов в качестве прекурсора для высококачественных пигментов оксида железа, подходящих для различных промышленных применений.

**Ключевые слова:** сплав железа, серная кислота, железный купорос, осаждение, гетит, пигмент.

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