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Thermal upgrading of nickel from limonite by means of selective reduction

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Abstract. X-ray diffraction (XRD), X-ray fluorescence (XRF), scanning electron microscopy energy dispersion spectroscopy (SEM–EDS), and differential thermal analysis (DTA) measurements were used to investigate the mineralogical characteristics and distribution of data set in limonite soil from Indonesia. The findings point to a complicated inner core in laterite ore. Ni, Fe, Mg, Al, and Si levels in limonite are 1.4, 50.5, 1.81, 4.86 and 16.5 wt %, respectively. The iron oxide/oxyhydroxide content of limonite is 94.4 and 5.6 % silicate. DTA shows that limonite has a phase transition in the low temperature (200 – 300 °C) with the goethite transformation to hematite. This phase is good optimalization for nickel diffusion in the iron. Moreover, for this limonite, the thermal upgrading was used as a good method.

Keywords: characterization, limonite, phase, microstructure, thermal gravimetry, thermal upgrading

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Оригинальная статья

Термическое обогащение никеля из лимонита методом селективного восстановления

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Аннотация. Для изучения минералогического состава и параметров лимонитовых руд из Индонезии применялись методы рентгеновской дифракции (XRD), рентгеновской флуоресценции (XRF), энергодисперсионного микроанализа на растровом электронном микроскопе (SEM–EDS) и дифференциального термического анализа (DTA). Полученные данные указывают на наличие сложного внутреннего ядра в латеритной руде. Содержание Ni, Fe, Mg, Al и Si в лимоните составляет 1,4; 50,5; 1,81; 4,86 и 16,5 % (вес.) соответственно, оксидов/гидроксидов железа – 94,4 %, силикатов – 5,6 %. Термический анализ показывает, что при низкой температуре (200 – 300 °C) в лимоните происходит фазовый переход, при этом гетит замещается гематитом. Эта фаза является оптимальным вариантом для диффузии никеля в железо. Более того, для данного лимонита в качестве подходящего метода было выбрано термическое обогащение.

Ключевые слова: характеристика, лимонит, фаза, микроструктура, термическая гравиметрия, термическое обогащение

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Introduction

Given that nickel laterites represent 60 – 70 % of the world's Ni resources, they have been mined for around 140 years, generating less than 40 % of global Ni output until 2000, with the following exceptions from sulfide ores [1, 2]. The discovery and mining of laterite nickel ores has increased with the fall in the availability of sulfide nickel ore deposits [1]. Laterite nickel resources provide several advantages, including abundant supply, inexpensive investment costs, simple transportation, and a range of products like ferronickel, nickel matte, and nickel oxide [1]. Nickel laterite deposits were divided into two categories based on their chemical and physical characteristics: saprolite and limonite ores [3]. Limonite ores contain goethite, gibbsite, and chromite, along with undersid stages such cristobalite and protoenstatite [4 – 8]. Nevertheless, due to their low crystallization and tiny particle size, mechanical procedures are ineffective for these nickel laterites. As a result, the use of physical treatment techniques to properly upgrade these ores is generally difficult. Pyrometallurgical extraction techniques are used to extract nickel from laterite ores. They may yield significant nickel recoveries counter this challenge [9 – 22]. Furthermore, the laterite's complicated mineral makeup will have a significant impact on the reducing roasting process. As a result, knowing the disintegration of laterite is critical for laterite pre-reduction [13]. Since nickel is found in goethite and silicate minerals, an additive must be used to dissolve the silicate and nickel connections. This can easily be done by means of a solvent [13 – 15, 19 – 21]. This research, will study saprolite in material characterization to establish the optimal process for extracting it.

Material and Methods

Limonite ore is from Torobulu, Southeast Sulawesi, Indonesia. In the first step, limonite was sieved by shaker sieve until the size of 100 mesh. This is the optimal size for laterite and was weighed at 10 and 1 grams for the experiment and DTA test. The test involved annealing at the temperature variation of 150 and 300 °C for 1 hour for experiment test in furnace with 1 atm air pressure. The test for the DTA Linseis – PT1600 involved annealing at temperature 200 to 1200 °C with deviation 10 °C/minute and 1 atm air pressure and analysis data using Origin 8.1. It was then analysed by XRF Bench Top PANalytical Epsilon 3XLE (in the sample size 200 mesh – raw after homogenization in ball mill for 8 h – concentrate sample after magnetic separation and drying). The data was analysed in Microsoft Excel 2016, XRD PANalytical X'Pert3 Powder (in the sample size 200 mesh, the 2θ is in the range 10 – 80° with step size 0.05 and analysis data by High Score Plus) for SEM-EDS Thermo-scientific Quatro 6 with magnification 5000× and Bruker for EDS. The chemical reaction can be seen in (1) for dihydroxylation goethite and (2) water evaporation. After the characterization test, the sample was mixed with a reductor and additive at step *1* to *2*. Step *1* is without holding temperature and goes direct to reduction at 1150 °C. Step *2* is with a holding temperature at 300 °C and goes direct to reduction at 1150 °C.

$$4FeO(OH)(s) \xrightarrow{\text{Heating at 300 °C}} 2Fe_2O_3(s) + 2H_2O(aq); \quad (1)$$

$$2H_2O(aq) \xrightarrow{\text{Heating at 120 °C}} 4H^+ + O_2^{2-}. \quad (2)$$

Raw material analysis

As can be seen in Table 1, the specific chemical composition in saprolite ore is obtained at 1.4 wt % Ni, 50.5 wt % Fe and 1.81 wt % Mg. Fig. 1 shows that saprolite ore contains element goethite ($FeHO_2$) and quartz (SiO_2). All the substances present in the limonite ore is an oxide element and it is mineraloid structure. Nickel content in the nickel oxide largely forms and associates with iron in the goethite structure. The Rietveld refinement calculation results are obtained similar to the XRD results in

Table 1

The chemical composition of limonite ore

Таблица 1. **Химический состав лимонитовой руды, % (вес)**

Ni	Fe	Si	Mg	Al	Ca	Cr	Mn	Co
1.4	50.5	16.5	1.81	4.86	0.177	2.68	0.847	0.0662

Table 2

Rietveld refinement calculations of limonite ore

Таблица 2. **Расчеты лимонитовой руды методом Ритвельда**

Compound	**Total (%)**
Goethite	94.4
Quartz	5.6

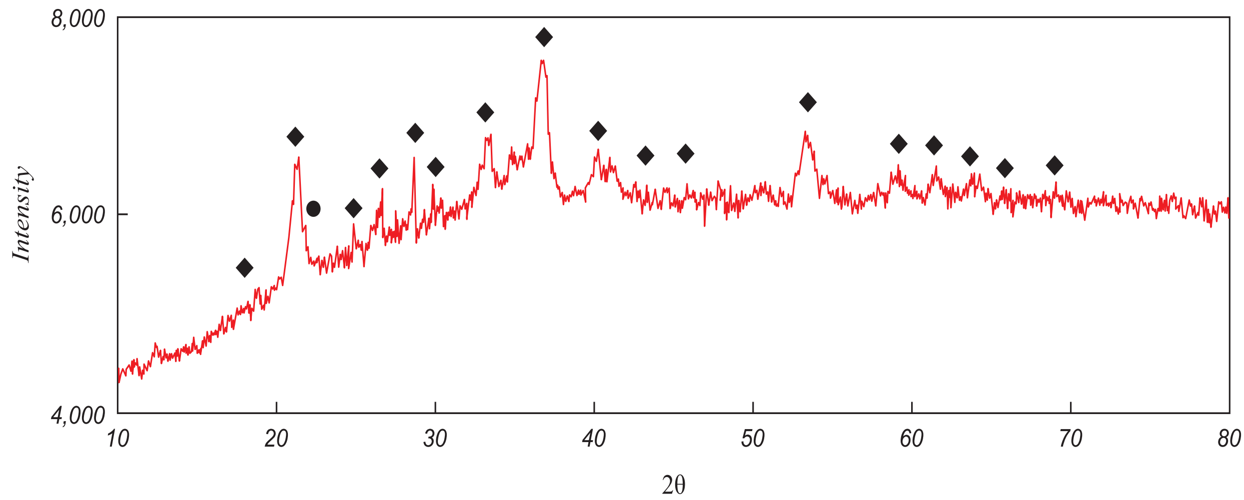

Fig. 1. XRD analysis of limonite:
◆ – goethite and ● – quartz

Рис. 1. Анализ лимонита методом рентгеновской дифракции:
◆ – гетит; ● – кварц

Table 2, indicating that the raw material is limonite ore. Fig. 2 and 4 show that curve *1* is the water evaporation and curve *2* is the dihydroxylation of goethite.

This study shows the thermal gravimetry and phase of limonite with the steep curves at each temperature (120 and 300 °C) according to Fig. 2 and 4, indicating change to the goethite phases and the water evaporation. The result of the experiment explains the transition of phases in limonite. The morphology of limonite is shown in Fig. 3. The result shows convincingly that the major element in the limonite are iron-magnesium-silica-oxide and nickel strongly associated with iron.

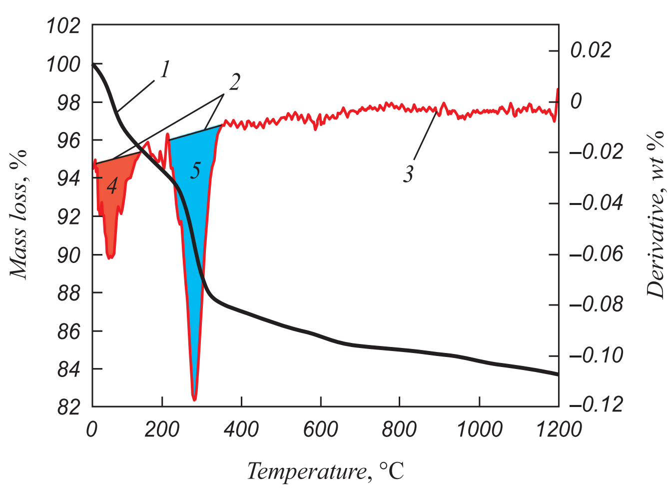

Fig. 2. DTG analysis of limonite:
1 – limonite; *2* – TG; *3* – DTG; *4* – water evaporation;
5 – dihydroxylation of goethite

Рис. 2. Анализ методом производной термогравиметрии лимонита:
1 – лимонит; *2* – термогравиметрия; *3* – производная термогравиметрии; *4* – испарение воды; *5* – дегидроксилирование гетита

Experiment Analysis

Effect of temperature variation on the phase transition

According to the raw material characterization, the phase transition is indicated at 120 and 300 °C. In this result, at 120 °C, the phases are still same with the raw material which show a good process of water evaporation. This process should happen for limonite at this temperature. On the other hand, the phase changes acutely from goethite to hematite and the quartz is still in the same condition as raw at 300 °C. Table 3 shows that the percentage of goethite fell drastically to zero. The OH structure has changed to oxide structure and the DTG results also show convincingly the transition of goethite to hematite with high mass loss at 300 °C in Fig. 2.

Effect of temperature variation on the morphology

The effect of heating at 120 °C separates the iron-nickel from the raw condition in Figure 3 and is comparable with

Table 3

Rietveld refinement of calculation of the temperature variation
Таблица 3. **Расчет изменения температуры в зависимости от процентного содержания фаз методом Ритвельда**

Total percentage, %			Temperature, °C
goethite	hematite	quartz	
94.4	0	5.6	120
0	94.4	5.6	300

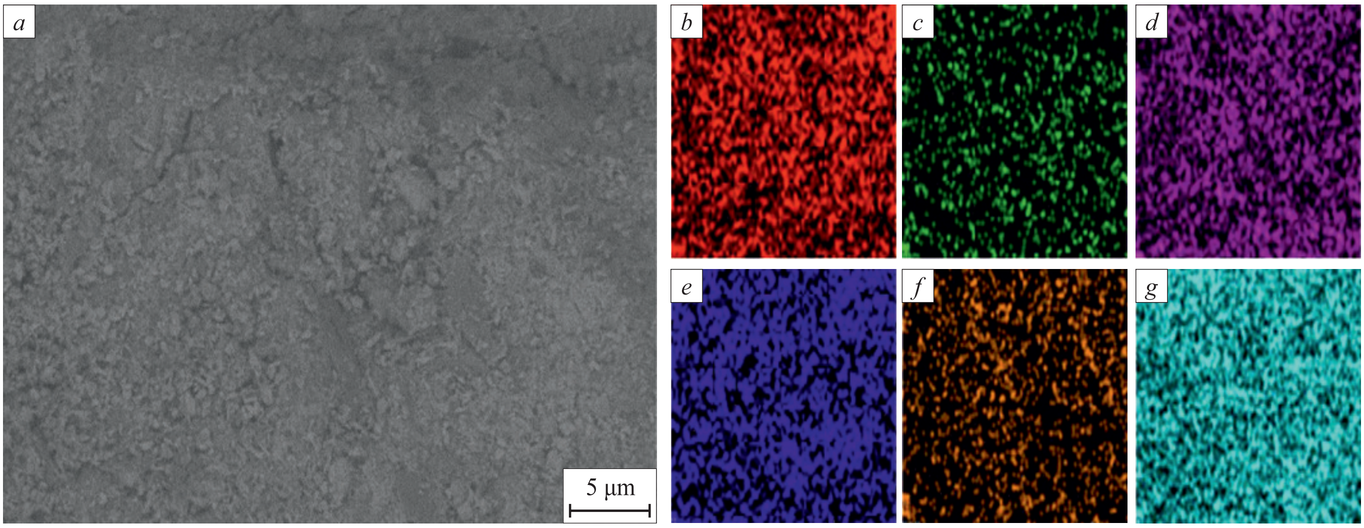

Fig. 3. SEM EDS raw limonite with area mode magnification 5000× (*a*):
b – magnesium; *c* – aluminum; *d* – iron; *e* – silica; *f* – nickel; *g* – oxide

Рис. 3. Снимок энергодисперсионного микроанализа на растровом электронном микроскопе лимонита с увеличением 5000 (*a*):
b – магний; *c* – алюминий; *d* – железо; *e* – кремний; *f* – никель; *g* – оксид

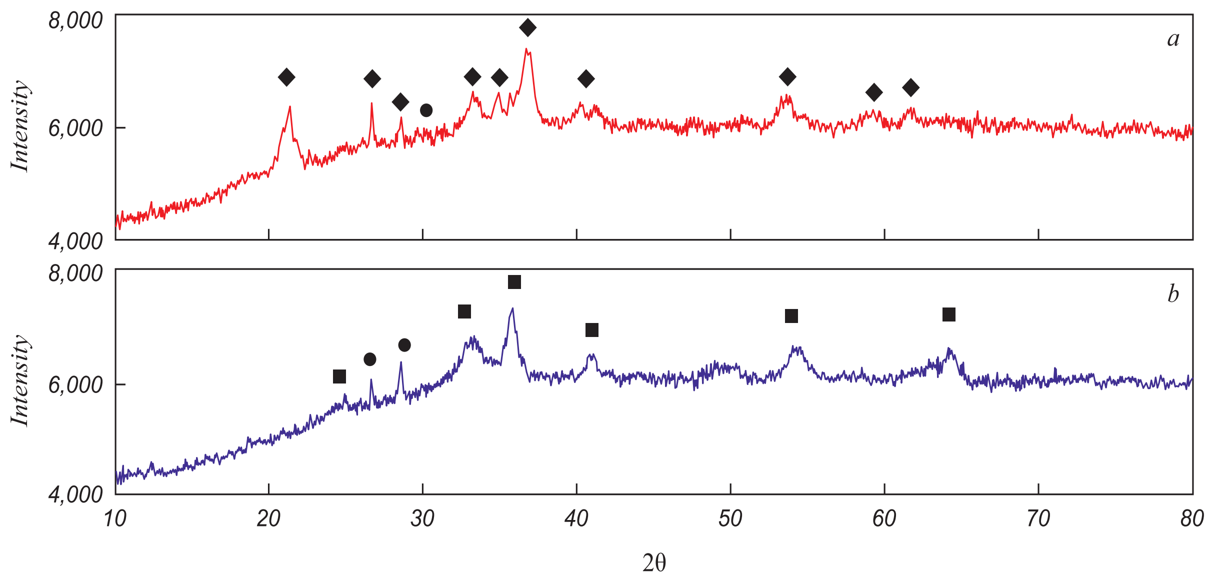

Fig. 4. XRD pattern of 120 °C (*a*) and 300 °C (*b*):
■ – hematite; ◆ – goethite; ● – quartz

Рис. 4. График данных рентгеновской дифракции при температуре 120 °C (*a*) и 300 °C (*b*):
■ – гематит; ◆ – гетит; ● – кварц

Fig. 5. It happens when the water evaporation has the initial effect for phase transition. On the other hand, magnesium-aluminum-silica is associated with strong binding. The initial stage can change the structure even though the XRD result has no phase transition.

The heating process at 300 °C results in greater merging of the iron-nickel, according to the spreading of element in Fig. 6. Thus 300 °C is the optimal temperature for changing the phase of goethite to hematite. The shaping and joining of the transition magnesium-aluminum-silica results in a connection of the phase transition of goethite to hematite perfectly going well. This stage can change the structure and the XRD result has convincing phase transition.

Effect of thermal upgrading on the phase transition, nickel grade, and microstructure

Indication of thermal upgrading has a major effect on phase transformation. Step *2* is the optimum condition with a high nickel percentage which Ni and Fe are 6.079 and 67.893 wt % in Fig. 7. The saturation of nickel was high with increasing temperature and time. Fig. 8

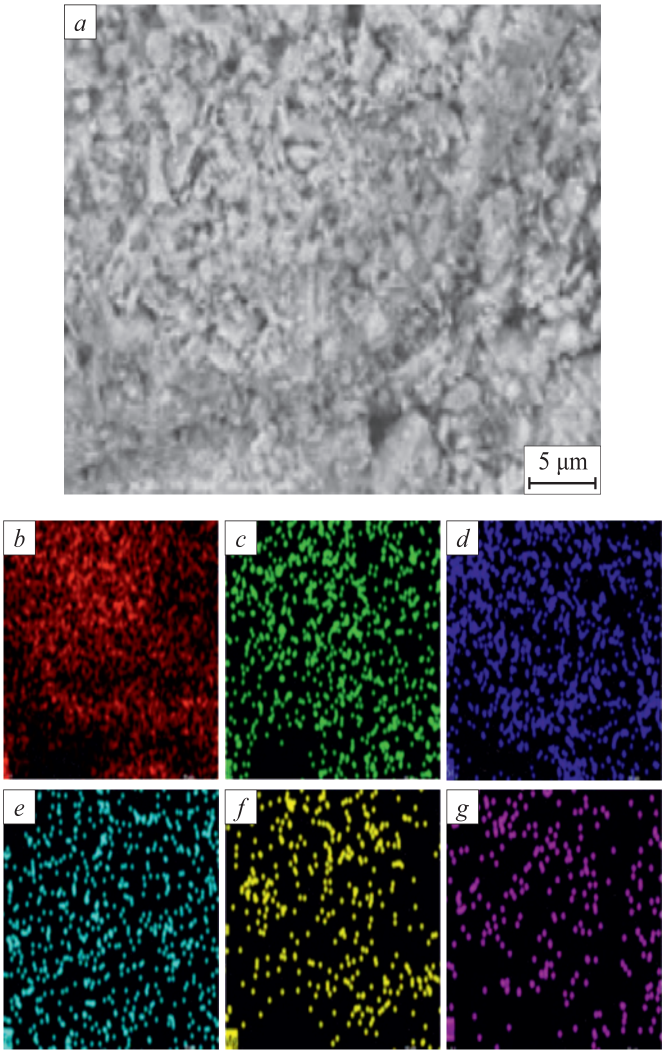

Fig. 5. SEM EDS limonite at 120 °C with area mode magnification 5000× (*a*):
b – oxide; *c* – aluminium; *d* – silica; *e* – iron; *f* – magnesium; *g* – nickel

Рис. 5. Снимок энергодисперсионного микроанализа на растровом электронном микроскопе лимонита при температуре 120 °C с увеличением 5000 (*a*):
b – оксид; *c* – алюминий; *d* – кремний; *e* – железо; *f* – магний; *g* – никель

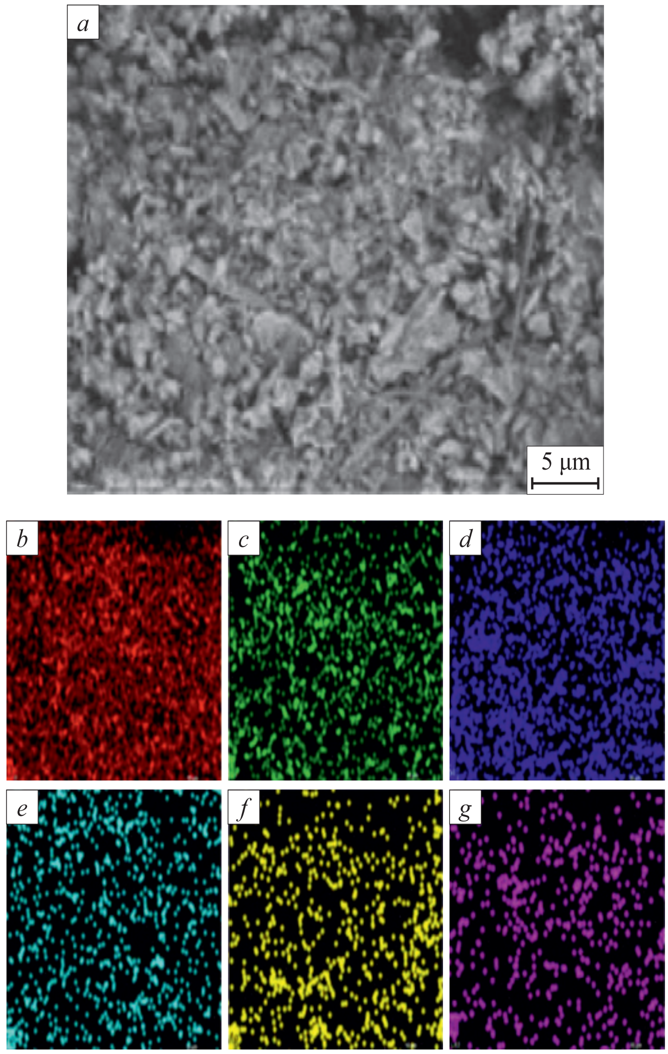

Fig. 6. SEM EDS limonite at 300 °C with area mode magnification 5000× (*a*):
b – oxide; *c* – aluminium; *d* – silica; *e* – iron; *f* – magnesium; *g* – nickel

Рис. 6. Снимок энергодисперсионного микроанализа на растровом электронном микроскопе лимонита при температуре 300 °C с увеличением 5000 (*a*):
b – оксид; *c* – алюминий; *d* – кремний; *e* – железо; *f* – магний; *g* – никель

shows appearance of wustite due to the composition of Fe on the 250 – 350 °C. The iron-nickel phase appears more in step *2* than another step. Tthe grain size average is 10 – 15 µm and indication iron-nickel appears the optimal percentage in the small grain size. Thermal upgrading at a low temperature has a major effect on increasing the percentage of nickel until 6 wt % causing the dihydroxylation of goethite.

Conclusion

Based on the results, the phase transition of goethite to hematite is optimal at a temperature of 300 °C. The changing of phases causes saturation of the goethite phase and

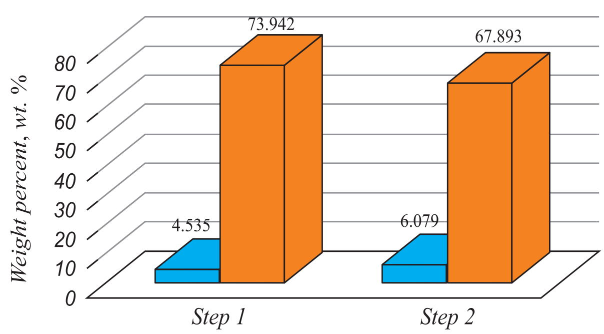

Fig. 7. The nickel (■) and iron (■) percentage after thermal upgrading of limonite

Рис. 7. Процентное содержание никеля (■) и железа (■) после термического обогащения лимонита

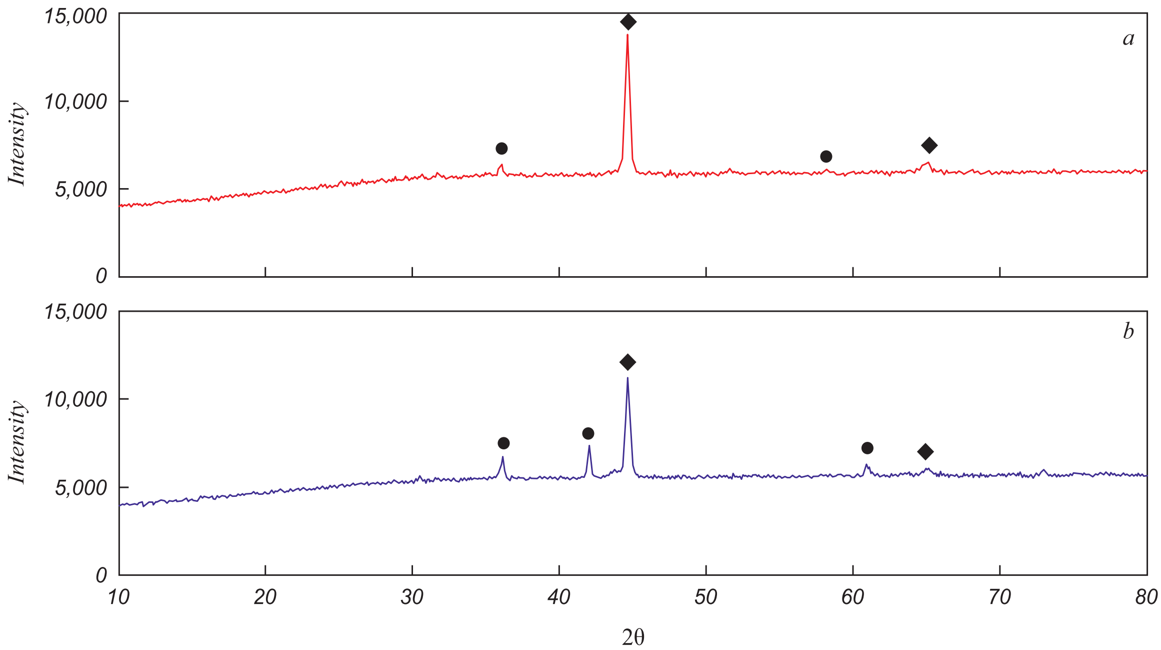

Fig. 8. The phases of step *1* (*a*) and step *2* (*b*) selective reduction of limonite:
● – wustite; ◆ – iron/nickel

Рис. 8. Селективное восстановление лимонита, фазы ступени *1* (*a*) и ступени *2* (*b*):
● – вюстит; ◆ – железо/никель

the change to hematite. As a result of the thermal upgrading experiment, step 2 at 300 °C before the reduction process ensures the optimal nickel percentage and recovery. Thermal upgrading at a low temperature has a major effect on increasing the percentage of nickel until 6 wt % causing the dihydroxylation of goethite. In future research, such characterization could serve as a reference for further process as a selective reduction experiment. By using it for initial stage before the reduction process will provide the optimal nickel percentage and recovery.

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