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Composition of tailings after selective reduction of laterite

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Abstract. The selective reduction process generates products in the form of concentrates and tailing/by-products. There is high percentage of iron and other elements in the tailings that are not extracted in selective reduction process. Properties of by-products of selective reduction were investigated using X-ray diffraction (XRD), inductively coupled plasma optical emission spectroscopy (ICP–OES), ultraviolet-visible (UV–VIS), and scanning electron microscopy energy dispersion spectroscopy (SEM–EDS). Based on the results of this study, the properties of iron-sulfur, iron-magnesium-aluminium, and silica phases in the tailings can be interpreted experimentally. For future research, it can be the reference for such processes as acid and base leaching. Pure iron extracted from tailings can be used for metal fuel in the future. The tailings composition data will help future researchers to find optimal processes for the tailings.

Keywords: composition, tailings, phase, microstructure

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Состав хвостов при избирательном восстановлении латерита

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Аннотация. В результате избирательного восстановления образуются концентраты и пустая порода (хвосты). В хвостах содержится высокий процент железа и других элементов, которые не извлекаются в процессе селективного восстановления. Свойства хвостов после избирательного восстановления исследовались методами рентгеноструктурного анализа, оптико-эмиссионной спектрометрии с индуктивно связанной плазмой, оптической, УФ- и сканирующей электронной микроскопии, а также энергодисперсионного микроанализа на растровом электронном микроскопе. Экспериментально установлено, что хвосты содержат сульфиды железа, соединения железа с алюминием и магнием, а также диоксид кремния. На основании полученных данных в дальнейшем возможна разработка технологий кислотного и щелочного экстрагирования минералов с целью получения чистого оксида железа, применяемого в качестве металлического топлива.

Ключевые слова: состав, хвосты, фаза, микроструктура

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Introduction

Extraction of nickel from the widely occurring laterite ores has become an important task of research [1 – 9]. Previous studies have shown that direct reduction roasting process followed by magnetic separation is an effective method for recovering nickel from laterite nickel ores [10 – 17]. To obtain nickel alloy powder with high nickel content, iron reduction needs to be controlled in the direct reduction roasting process. Selective reduction can be achieved by adjusting the reducing atmosphere [18 – 19] and the number of additives [20 – 22]. Li et al. [10] found that FeS is produced by direct reduction with the Na_2SO_4 additive. The formation of the Fe/FeS eutectic promotes the growth of Ni/Fe particles; simultaneously, in the magnetic separation process the nonmagnetic FeS will go to the tailings thus achieving the purpose of selective reduction. Also, the formation of FeS promotes the growth of metal particles. Jiang et al. [25] found that Na_2SO_4 reacts with silicates producing low melting point nepheline and suppressing FeO reduction by inhibiting the diffusion of the reducing gas; it can also promote the growth of nickel-iron particles through the formation of FeS. In the FeO reduction process, the diffusion of the reducing gas was impeded due to the increase in the amount of liquid phase in the roasting system. All of the above studies found that FeS plays an important role in the selective reduction of laterite nickel ore. In direct selective reduction of laterite nickel ore, FeS also serves as a paramagnetic film covering the FeO surfaces. That thin layer blocks the contact between the reducing gas and FeO suppressing the reduction process. Iron-rich, it can be used as a nanoparticle's precursor in food technology, biomedicine, energy and fuel production, etc. [26, 27]. The best possible application of the iron nanoparticles precursor is for metal fuel which is illustrated in Fig. 1 [27]. Iron-rich by-product shall be seen as a primary source for the extraction processes to be used in the future.

Material and Methods

Data on tailings/by-products is taken from selective reduction process at the Research Unit for Mineral Technology, National Research and Innovation Agency of Indonesia, South Lampung, Lampung, Indonesia. First, the by-product was brought through a 200-mesh shaker sieve. After that the sample was dissolved in aqua regia for 5 days, diluted 50 and 1000 times, and analysed first by the ICP-OES Analytika Jena PQ9000 (with the resulting data converted in excel from ppm to weigh percent); and then by the XRD PANalytical X'Pert3 Powder (in the 200 mesh sample size, the 2θ is in the range of 10 – 80°, step size 0.05, and analysis data by High Score Plus) (Fig. 3). For SEM–EDS Thermo-scientific Quatro 6 with magnification 5000× was used and Bruker for EDS.

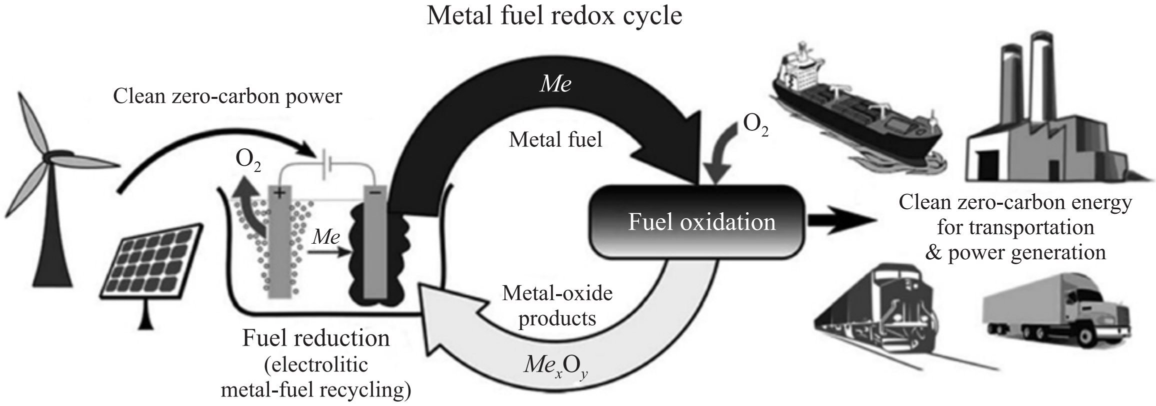

Fig. 1. Metal fuel in the future [27]

Рис. 1. Примеры применения металлического горючего в будущем [22]

RESULTS

ICP and UV–VIS Study

Table 1 gives tailing specific chemical composition: 0.74 wt% Ni, 39.45 wt% Fe, 5.1 wt% Mg. Fig. 2 gives Fe, Ni, Mg, Mn, Al, and Co ratios in the tailings according to their absorbance and wave lengths [28]; therefore, the same elements are detected by UV–VIS and ICP. The agreement of the ICP and UV–VIS result shows that the aggregate amounts of components in both tests are the same; for example, iron is the greatest ingredient while cobalt, nickel, and manganese are the least ones.

XRD Study

In the sulfide phase, iron occurs in a higher percentage than in magnesioferrite, forsterite, and quartz phases, where there is less iron which occurs together with constituent elements magnesium or aluminium. The Rietveld refinement calculation results agree with the XRD results in Table 2. Significantly, it is the first time that the tailing product is addressed as raw material.

SEM–EDS Study

This study gives the same results for the elements and phases of the tailings which can be further identified in microstructure. The morphology of tailings, as shown in Fig. 4, convincingly proves that the major elements in the tailing are iron-sulfur, iron-magnesium-silica-oxide, natrium, and quartz extracted in selective reduction with sulfur appearing in XRD. From Table 3, the magnesium-iron-aluminium is appearing in spots *2* and *4* indicating the magnesioferrite and forsterite phases.

Table 1

Chemical composition of tailing

Таблица 1. **Химический состав хвостов**

Element	Fe	Ni	Mg	Mn	Al	Co
Amount, wt%	39.45	0.74	5.1	0.55	2.63	0.074

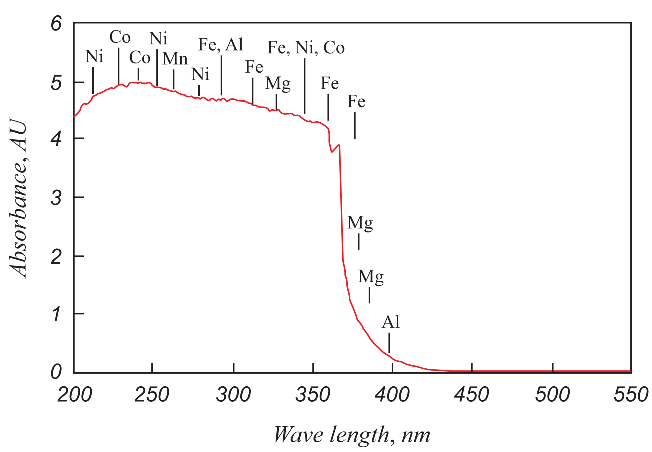

Fig. 2. UV–VIS analysis of tailing

Рис. 2 Результаты УФ- и оптической микроскопии хвостов

Table 2

Rietveld refinement calculations of tailing

Таблица 2. **Фазовый состав хвостов по Ритвельду**

Compound	Total, %
Pyrite	32.5
Wuestite	24.3
Magnesioferrite	21.5
Forsterite	16.3
Quartz	5.4

Table 3

Chemical composition of tailing in EDS

Таблица 3. **Химический состав хвостов, полученный с помощью энергодисперсионного микроанализа**

Sampling spots	Element (%wt)					
	Fe	Ni	Mg	Al	Mn	Co
1	57.48	–	1.37	–	0.63	–
2	47.77	–	3.77	1.70	0.97	–
3	37.64	–	1.99	5.13	1.03	–
4	20.45	–	6.47	14.50	1.01	–
5	0.98	–	–	–	–	–

CONCLUSIONS

Based on the results, the tailing includes such phases as iron-sulfur, iron-magnesium-aluminium, and silica which can be interpreted so that the tailing is iron-rich and not

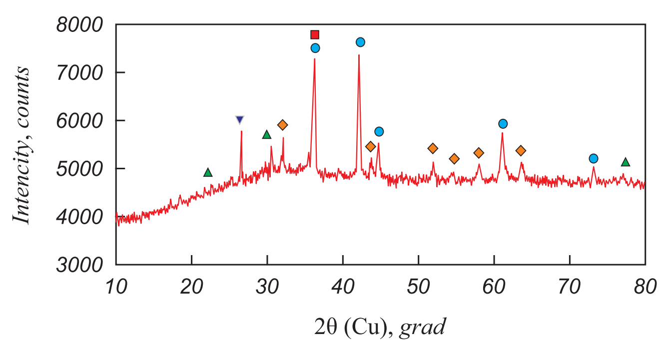

Fig. 3. XRD analysis of tailing:
● – pyrite, ■ – wuestite, ◆ – magnesioferrite, ▲ – forsterite, ▼ – quartz

Рис. 3. Результаты рентгеноструктурного анализа хвостов:
● – пирит; ■ – вюстит; ◆ – магнезиоферрит; ▲ – форстерит; ▼ – кварц

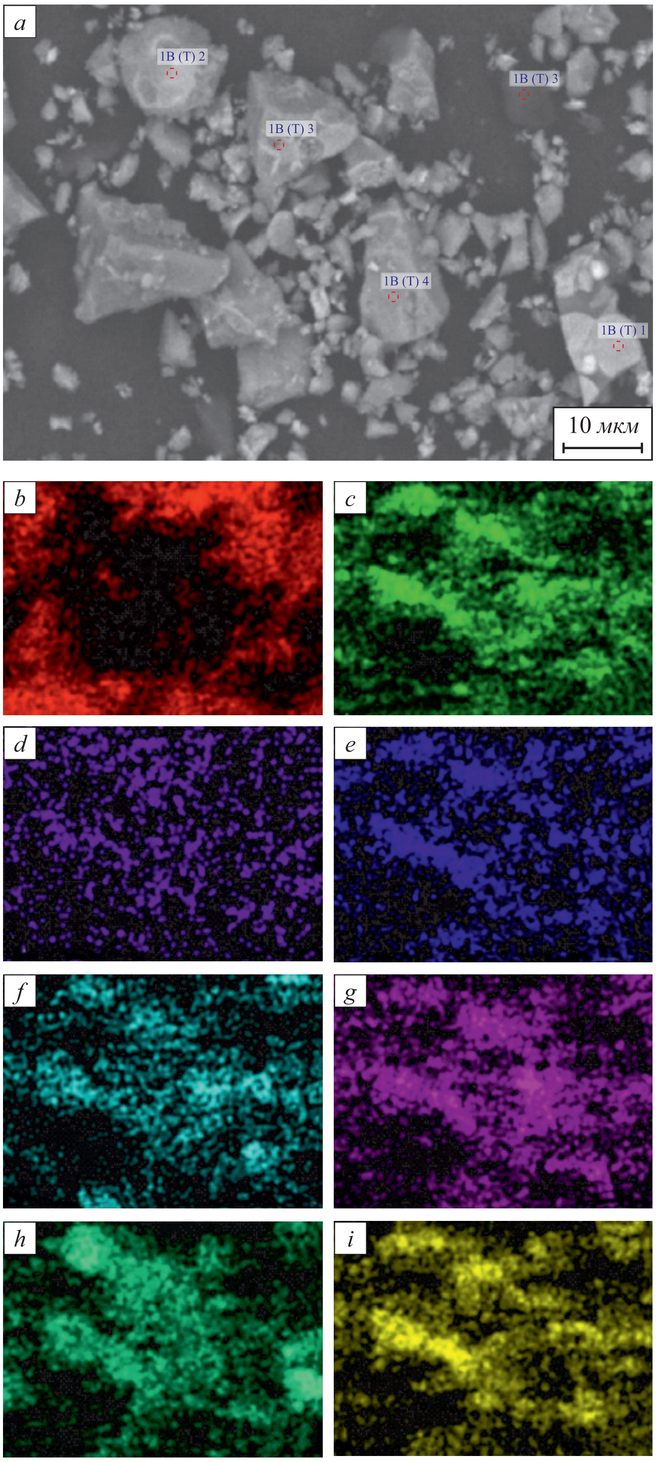

Fig. 4. SEM EDS of tailing in ×5000 (*a*) area magnification mode: *b* – carbon, *c* – oxygen, *d* – sulfur, *e* – natrium, *f* – magnesium, *g* – aluminium, *h* – silica, *i* – iron

Рис. 4. Результаты энергодисперсионного микроанализа хвостов на растровом электронном микроскопе: *a* – ×5000; *b* – углерод; *c* – кислород; *d* – сера; *e* – натрий; *f* – магний; *g* – алюминий; *h* – двуокись кремния; *i* – железо

usable after process. The process under consideration can serve as reference for further processes such as acid and base leaching. Pure iron extracted from tailings can be used for metal fuel in the future.

References / Список литературы

1. Li Y.J., Yu H.C., Wang D.Q., Yin W.X., Bai Y.S. The current status of laterite ore resources and its processing technology. *Jinshu Kuangshan/Metal Mine*. 2010; 11: 5–9.
2. Rao M.J., Li G.H., Jiang T., Luo J., Zhang Y.B., Fan X.H. Carbothermic reduction of nickeliferous laterite ores for nickel pig iron production in China: a review. *JOM*. 2013; 65(11): 1573–1583. https://doi.org/10.1007/s11837-013-0760-7
3. Wang C.Y., Yin F., Chen Y.Q., Wang Z., Wang J. Worldwide processing technologies and progress of nickel laterites. *Chinese Journal of Nonferrous Metals*. 2008; 18(S1): 1–8.
4. Dalvi A.D., Bacon W.G., Osborne R.C. The past and the future of nickel laterites. *PDAC 2004 Int. Conf. Trade Show and Investors Exchange, Toronto, 7-10 March, 2004*: 1–27.
5. Bahfie F., Manaf A., Astuti W., Nurjaman F., Prasetyo E., Sumardi S. Characterization of thermal upgrading of nickel from limonite by selective reduction. *Izvestiya. Ferrous Metallurgy*. 2022; 65(7): 471–478. https://doi.org/10.17073/0368-0797-2022-7-471-478

   Бахфи Ф., Манаф А., Астути В., Нурджаман Ф., Прасетио Э., Сумарди С. Термическое обогащение никеля из лимонита методом селективного восстановления. *Известия вузов. Черная металлургия*. 2022; 65(7): 471–478. https://doi.org/10.17073/0368-0797-2022-7-471-478
6. Bahfie F., Manaf A., Astuti W., Nurjaman F. Studies on reduction characteristics of limonite and effect of sodium sulphate on the selective reduction to nickel. *Journal of the Institution of Engineers (India): Series D*. 2020; 102(1): 149–157. https://doi.org/10.1007/s40033-020-00240-3
7. Bahfie F., Manaf A., Astuti W., Nurjaman F., Prasetyo E. Studies of carbon percentage variation and mixing saprolite-limonite in selective reduction. *Materials Today: Proceedings*. 2022; 62: 4156–4160. https://doi.org/10.1016/j.matpr.2022.04.679
8. Bahfie F., Murti D.U., Nuryaman A., Astuti W., Nurjaman F., Prasetyo E., Sudibyo S., Susanti D. Kinetic properties of nickel leaching by ANOVA method. *Progress in Physics of Metals*. 2022; 23(3): 476–88. https://doi.org/10.15407/ufm.23.03.476
9. Nurjaman F., Handoko A.S., Bahfie F., Astuti W., Suharno B. Effect of modified basicity in selective reduction process of limonitic nickel ore. *Journal of Materials Research and Technology*. 2021; 15: 6476–6490. https://doi.org/10.1016/j.jmrt.2021.11.052
10. Li G.H., Shi T.M., Rao M.J., Jiang T., Zhang Y.B. Beneficiation of nickeliferous laterite by reduction roasting in the presence of sodium sulphate. *Minerals Engineering*. 2012; 32: 119–26. https://doi.org/10.1016/j.mineng.2012.03.012
11. Kim J., Dodbiba G., Tanno H., Okaya K., Matsuo S., FujitaT. Calcination of low-grade laterite for concentration of Ni by magnetic separation. *Minerals Engineering*. 2010; 23: 282–288. https://doi.org/10.1016/j.mineng.2010.01.005
12. Zheng G.L., Zhu D.Q., Pan J., Li Q.H., An Y.M., Zhu J.H., Liu Z.H. Pilot scale test of producing nickel concentrate from low-grade saprolitic laterite by direct reduction-magnetic separation. *Journal of Central South University*. 2014; 21: 1771–1777. https://doi.org/10.1007/s11771-014-2123-0

13. Li B., Wang H., Wei Y.G., The reduction of nickel from low-grade nickel laterite ore using a solid-state deoxidisation method. *Minerals Engineering*. 2011; 24: 1556–1562. https://doi.org/10.1016/j.mineng.2011.08.006

14. Liang W., Wang H., Fu J.G., He Z.X. High recovery of ferronickel from low grade nickel laterite ore. *Journal Central South University (Science and Technology)*. 2011; 42(8): 2173–2177.

15. Li G.H., Rao M.J., Jiang T., Huang Q.Q., Tang T.M., Zhang Y.B. Innovative process for preparing ferronickel materials from laterite ore by reduction roasting-magnetic separation. *The Chinese Journal of Nonferrous Metals*. 2011; 21(12): 3137–3142.

16. Huang D.H., Zhang J.L., Lin C.C., Mao R. Production of ferro-nickel granules from nickel laterite ore/coal composite briquettes by direct reduction. *Chinese Journal of Engineering*. 2011; 33(12): 1442–1447. https://doi.org/10.13374/j.issn1001-053x.2011.12.015

17. Cao Z.C., Sun T.C., Yang H.F., Wang J.J., Wu X.D. Recovery of iron and nickel from nickel laterite ore by direct reduction roasting and magnetic separation. *Chinese Journal of Engineering*. 2010; 32(6): 708–712. https://doi.org/10.13374/j.issn1001-053x.2010.06.004

18. Sun T.C., Ji Y.N., Jiang M. Influence mechanism of different types of coal on selective nickel reduction in nickel laterite reduction roasting. *Chinese Journal of Engineering*. 2011; 33(10): 1197–1203. https://doi.org/10.13374/j.issn1001-053x.2011.10.015

19. Jiang M., Sun T.C., Liu Z.G., Kou J., Liu N., Cao Y.Y., Zhang S.Y. Effects of coal types and additives on selective direct reduction of nickel laterite. *Mining and Metallurgical Engineering*. 2012; 32(5): 77–81.

20. Li G.H., Rao M.J., Jiang T., Shi T.M., Huang Q.Q. Reduction roasting-magnetic separation mechanisms of nickelferous laterite ore in presence of sodium salts. *The Chinese Journal of Nonferrous Metals*. 2012; 22(1): 274–278.

21. Sun T.C., Jiang M., Liu Z.G., Liu N., Zhang S.Y., Kou J., Xu C.Y. Research on the effect of additive on selective reduction of the laterite ores with low nickel and high iron content. *Zhongguo Kuangye Daxue Xuebao/Journal of China University of Mining and Technology*. 2013; 42(5): 838–844.

22. Zhu D.Q., Cui Y., Vining K., Hapugoda S., Douglas J., Pan J., Zheng G.L. Upgrading low nickel content laterite ores using selective reduction followed by magnetic separation. *International Journal of Mineral Processing*. 2012; 106–109: 1–7. https://doi.org/10.1016/j.minpro.2012.01.003

23. Valix M., Cheung W.H. Effect of sulphur on the mineral phases of laterite ores at high temperature reduction. *Minerals Engineering*. 2002; 15: 523–530. https://doi.org/10.1016/S0892-6875(02)00069-9

24. Lu J., Liu S.J., Shangguan J., Du W.G., Pan F., Yang S. The effect of sodium sulphate on the hydrogen reduction process of nickel laterite ore. *Minerals Engineering*. 2013; 49: 154–164. https://doi.org/10.1016/j.mineng.2013.05.023

25. Jiang M., Sun T.C., Liu Z.G., Kou J., Liu N., Zhang S.Y. Mechanism of sodium sulfate in promoting selective reduction of nickel laterite ore during reduction roasting process. *International Journal of Mineral Processing*. 2013; 123: 32–38. https://doi.org/10.1016/j.minpro.2013.04.005

26. Ali A., Zafar H., Zia M., ul Haq I., Phull A. R., Ali J. S., Hussain A. Synthesis, characterization, applications, and challenges of iron oxide nanoparticles. *Nanotechnology, Science and Applications*. 2016; 2016(9): 49–67. https://doi.org/10.2147/NSA.S99986

27. Julien P., Bergthorson J. M. Enabling the metal fuel economy: green recycling of metal fuels. *Sustainable Energy & Fuels*. 2017; 3: 1–11. https://doi.org/10.1039/C7SE00004A

28. William F. M., Charles H. C., Bourdon F. *Tables of Spectral-Line Intensities. Part I: Arranged by Elements*. U.S. Department of Commerce-National Bureau of Standards; 1975: 456.

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