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Selective agglomeration of magnetite in entlandite-serpentine system and implication for their separation

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Abstract: In nickel sulfide processing, magnesium silicates (serpentines) can easily form slime coatings or hetero-aggregation on pentlandite surfaces, and hence decrease the pentlandite flotation rate and recovery. In this work, magnetic separation of pentlandite from serpentine using selective magnetic coating through adding magnetite fines as magnetic seeds was investigated. Interactions of magnetite-pentlandite and magnetite-serpentine were calculated by the DLVO (Derjaguin-Landau-Verwey-Overbeek) theory. The results show that the interaction of magnetite-pentlandite was obviously stronger than that of magnetite-serpentine with an external weak magnetic field (4776 A/m^{-1}). Therefore, fine magnetite fractions selectively adhered to the pentlandite surfaces and enhanced its magnetism, resulting in being separated from serpentine by magnetic separation, which was further verified by magnetic coating-magnetic separation and SEM observations.

Keywords: *serpentine, pentlandite, magnetite, selective magnetic agglomeration, magnetic separation*

Introduction

There are two major sources to extract nickel that is sulphide ores and laterites in nature, and sulphide ores are still the main source of Ni (pentlandite), which are typically floated by employing combinations of xanthate used as a collector, MIBC as a frother, soda ash or acid as pH modifier, and carboxy-methyl-cellulose (CMC) or sodium hexametaphosphate (SHMP) as a dispersant (Wellham et al., 1992; Peng and Seaman, 2011). In the nickel sulphide ores, the predominant sulphide minerals are pentlandite, chalcopyrite and pyrrhotite. The gangue minerals consist mainly of MgO silicates, serpentine, pyroxene, chlorite, and talc and so on. Relatively high levels of MgO silicates often report to the froth phase resulting in Ni grade decrease during flotation, affecting the smelter efficiency and lifetime. Many studies have revealed mechanisms of magnesium silicates interference as follows: *i*) entrainment or

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entrapment of fine MgO silicates (Pietrobon et al., 1997; Patra et al., 2012), *ii*) heterocoagulation or slime coating (Edwards et al., 1985), *iii*) transport of MgO silicates via composite particles having hydrophobic sites (Bremmell et al., 2005), and *iv*) inadvertent activation of these MgO silicate minerals by Cu^{2+} or Ni^{2+} species (Fornasiero and Ralston, 2005).

In the past 10 years, many experiments have been conducted to depress MgO minerals and improve the Ni recovery. A new process was implemented at Mt. Keith to increase the Ni recovery, which contained a two-stage cyclone classification for different rougher flotation cells. The underflows from the two-stage cyclone both passed to two parallel rougher scavenger flotation, and then were cleaned, while the overflows of the only second stage cyclones passed to the flotation column. As a result, a substantial increase in the nickel recovery was achieved (Senior and Thomas, 2005). Other approaches proposed to improve flotation of pentlandite minerals include: high intensity conditioning (HIC) (Chen et al., 1999), thermal pretreatment (Bobicki et al., 2014), and disintegrating fibers by chemical dissolution (H_2SO_4) and mechanical activation (grinding) (Uddin et al., 2012). Finally, pentlandite minerals were well separated through flotation.

In this work we propose selective magnetic coating to separate pentlandite from serpentine. With the addition of fine magnetite particles and using external magnetic field, elective agglomeration and magnetic separation of fine pentlandite particles which the magnetic property was enhanced by being coated of the magnetite fines, was achieved. This method has the following advantages: without or a small amount addition of reagents and the relatively simple system of two phases of the liquid-solid. Therefore, the purpose is to utilize magnetic separation to recover pentlandite and provide an alternative method for further separating nickel sulphide ores using selective magnetic coating.

At present, the magnetic coating technology has been used widely in many fields, such as treatment of effluent, separation of biological cells, coal desulphurization and mineral processing. The essence of this method is to incorporate a discrete magnetic phase (mainly fine magnetite) into either weakly or nonmagnetic target particles to increase their magnetism and recover these agglomerates by magnetic separation (Parsonage, 1988; Feng et al., 2000). Prakash et al. (1999) discussed separation of hematite from mixtures of hematite, quartz and corundum with a recovery of 90~96% using the magnetic coating method at a magnetic field of 620 $kA \cdot m^{-1}$. Singh et al. (2015) adopted this process to recover iron minerals from Indian iron ore slimes by the addition of oleate colloidal magnetite. An iron concentrate of 62.6% Fe with a recovery of 72% was obtained under optimum conditions of pH 7.0, colloidal magnetite concentration 10~40 g/Mg and a magnetic intensity 950 $kA \cdot m^{-1}$. The magnetic coating of other minerals, for instance chromite/serpentine, quartz/ magnesite, and calcite/quartz and so on, were also described in details (Prakash et al., 1999; Anastassakis, 2002; Ucbas et al., 2014). In spite of the fact that many minerals have been successfully separated using the magnetic coating technology, very little literatures have been published to process sulphide ores, particularly separation of pentlandite from serpentine (Lu et al., 2015). Thus, the present paper discusses the application of magnetic coating process for separation of pentlandite from serpentine minerals and aims to demonstrate the possibility of sorting copper-nickel sulfide ores.

Experimental

Samples and methods

Pentlandite, serpentine, and magnetite minerals used in this study were obtained from Jinchuan (Gansu Province, China), Xiuyan (Liaoning Province, China), and Nanfen (Liaoning Province, China), respectively. Each lump ore sample was handpicked to prepare pure minerals. The pentlandite sample was crushed with a hammer to –2 mm before being further separated to remove gangue. The –2 mm sample was then ground in a ceramic ball mill for 8 min to -75 µm, and then concentrated on a Wilfrey shaking table. The concentrate was separated to remove the strong-magnetic minerals by a low-intensity magnetic separator. The weakly magnetic product was further enriched on the shaking table to obtain pure pentlandite minerals. The serpentine sample was crushed with a hammer to –2 mm, and then was ground in a ceramic ball mill for 5 min to obtain pure serpentine minerals of –75+20 µm. The magnetite sample was first hammer crushed to –2 mm, and then was ground in a stirred mill for 45 min. The products concentrated on a shaking table and the concentrates subsequently were sorted by a weak magnetic separator. Then, the magnetic concentrate was reground for 45 min to -10 µm.

The XRD patterns and chemical multi-element analyses of each mineral are listed in Figs. 1–3 and Tables 1–3. The grain size distributions of samples were measured by a Malvern particle size analyzer model 2000 (Table 4). The data in Table 4 reveal that the median diameters D_{50} of pentlandite and magnetite particles were 56.01 and 2.01 µm, respectively.

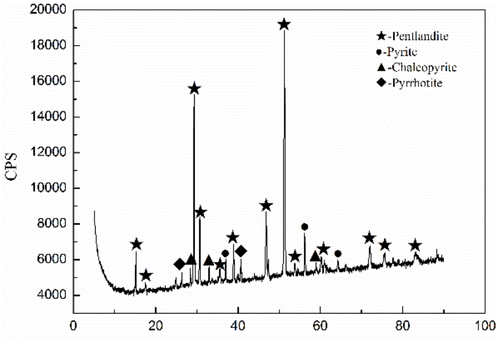

Fig. 1. X-ray diffraction spectra of pentlandite sample

Table 1. Chemical multi-element analyses of pentlandite sample

Chemical compositions	Ni	Fe	S	Cu	Al_2O_3	SiO_2	MgO
Content, %	26.25	32.03	34.50	5.08	0.37	0.33	0.05

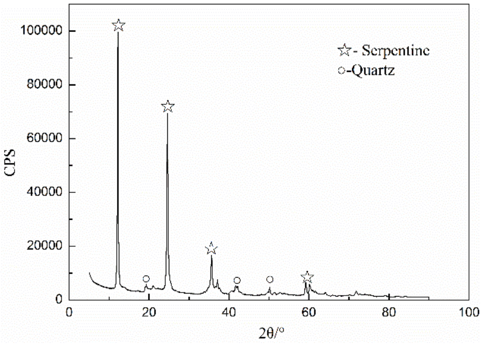

Fig. 2 X-ray diffraction spectra of serpentine sample

Table 2. Chemical multi-element analyses of serpentine sample

Chemical compositions	MgO	SiO_2	Fe	Al_2O_3	CaO	S
Content, %	35.02	41.89	0.41	0.38	0.20	0.16

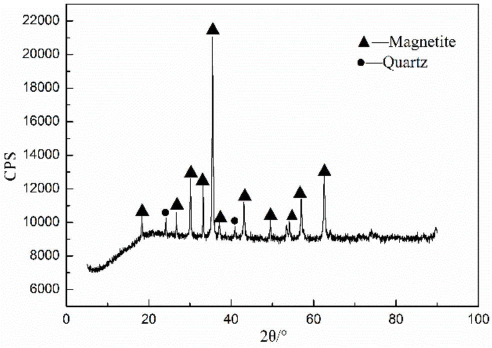

Fig. 3. X-ray diffraction spectra of magnetite sample

Table 3. Chemical multi-element analyses of magnetite sample

Chemical compositions	Fe	SiO_2	Al_2O_3	CaO	MgO	S	P
Contents, %	69.54	2.85	0.062	0.01	0.029	0.007	0.008

Table 4. Particle size distribution results of samples

Mineral	D_{10}, µm	D_{50}, µm	D_{90}, µm
Pentlandite	24.26	56.01	96.87
Magnetite	1.98	4.01	11.63

Magnetic coating tests

Magnetic coating tests were carried out in a 200 cm^3 XFGC batch flotation cell which was similar to the flotation cell employed in previous publications (Gao et al., 2016a, 2016b; Wang et al., 2016). A magnet provided by a magnetic stirrer was placed on the bottom of the flotation cell for producing an magnetic field of around 4776 $A\cdot m^{-1}$. For single mineral tests, the mineral suspension was prepared by adding 10.0 g pentlandite (or serpentine) minerals to 100 cm^3 deionized water and conditioned for 2 min. Fine magnetite fractions were then added into the suspension and stirred for another 3 min at a stirring speed of 2400 $r\cdot min^{-1}$. For mixed minerals tests, 10.0 g of pentlandite was mixed with the desired amount of serpentine and added to 100 cm^3 deionized water and conditioned for 2 min. Then, magnetite fines were added and stirred for further 3 min at a stirring speed of 2400 $r\cdot min^{-1}$.

Magnetic separation tests

The above coated suspension was subjected to magnetic separation using a XCSQ-50×70 wet high intensity magnetic separator (WHIMS). Magnetic and nonmagnetic products collected by the WHIMS were filtered, dried and weighted. For single mineral tests, the recovery of magnetic separation was determined with respect to the solid mass distributions between magnetic and nonmagnetic products. For mixed mineral tests, the magnetic and nonmagnetic products obtained were analysed for the Ni and MgO content. The recoveries of Ni and MgO were calculated based on the yield and grade of each product.

SEM analysis

A Zeiss Ultra Plus scanning electron microscope equipped with an energy dispersive X-ray spectrometer was used for the SEM-EDS studies. The magnetic concentrates and tailings of mixed minerals were mounted in a carbon conductive resin, and then gold sprayed for the SEM-EDS observation.

Interactions between particles in suspension

Selective adsorption of fine magnetic fractions on minerals surfaces are controlled by the total energy of interaction between particles in the suspension. If it is a net repulsive interaction, the particle will tend to be dispersed, and hence no magnetic coating occurs. In contrast, a net attractive interaction between a mineral and magnetite will favour conglutination and form a magnetic coating on the mineral surface. The dominating interactions between particles are determined by the van der Waals interactions U_A, electrical interactions U_E, and magnetic interactions U_M (existing between two magnetic particles, when the magnetic field is added) in the deionized water. If it is assumed that these interactions are additive each other, the total interaction U_T is given by Lu (2003):

$$U_T = U_A + U_E + U_M. \tag{1}$$

The van der Waals interaction U_A between spherical mineral particles is calculated according to the following expression:

$$U_A = -\frac{A_{132}}{6}\left[\frac{2R_1R_2}{H^2-\left(R_1+R_2\right)^2}+\frac{2R_1R_2}{H^2-\left(R_1-R_2\right)^2}+\ln\frac{H^2-\left(R_1+R_2\right)^2}{H^2-\left(R_1-R_2\right)}\right] \tag{2}$$

where, for $H << \min(R_1, R_2)$, the expression is simplified as follow:

$$U_A = -\frac{A_{132}R_1R_2}{6H\left(R_1+R_2\right)}. \tag{3}$$

The critical parameter in the van der Waals interaction is the Hamaker constant:

$$A_{132} = \left(\sqrt{A_{11}}-\sqrt{A_{33}}\right)\left(\sqrt{A_{22}}-\sqrt{A_{33}}\right) \tag{4}$$

where A_{11} and A_{33} are the Hamaker constant of mineral 1 and mineral 2, respectively; A_{22} is the Hamaker constant of water in the vacuum is equal to 3.68×10^{-20} J.

The interaction U_A between a spherical particle and a layered particle is usually determined by the following equation:

$$U_A = -\frac{A_{132}}{6}\left[\frac{2R}{H}+\frac{2R}{H+4R}+\ln\left(\frac{2R}{H+4R}\right)\right] \tag{5}$$

where H is the distance between two mineral particles, R is the radius of the spherical particle.

It must be noted that the particle size of magnetite is extremely fine (<4 μm), while the serpentine belongs to be phyllosilicates. Therefore, magnetite and serpentine are assumed into a sphere and a layer, respectively, and the interaction between them should be calculated by the Eq. (5). The Hamaker constants of pentlandite, serpentine and magnetite are 22.8×10^{-20}, 10.6×10^{-20}, and 24.0×10^{-20} J, respectively. The radii of magnetite and pentlandite are 2.0 and 28 μm, respectively.

The electrical interaction under constant potential U_E between two different spherical particles was described by Hogg et al. (1965) as follows:

$$U_E = \frac{\pi\varepsilon_0\varepsilon R_1R_2}{R_1+R_2}\left(\phi_1^2+\phi_2^2\right)\left[\frac{2\phi_1\phi_2}{\phi_1^2+\phi_2^2}\ln\frac{1+e^{-\kappa H}}{1-e^{-\kappa H}}+\ln\left(1-e^{-2\kappa H}\right)\right] \tag{6}$$

where ε_0 and ε are the permittivity of vacuum and solution ($\varepsilon_0\varepsilon = 7.17\times10^{-20}$ F·m^{-1}), respectively; φ_1 and φ_2 are the zeta or Stern potentials of minerals 1 and 2, respectively, κ is the reciprocal of Deby-Huckel parameter of 10.4×10^{-20} m^{-1}, R is the radius of particle 1 and 2.

The interaction U_E between a spherical particle and a plate-like particle is usually determined by the following equation:

$$U_E = \pi\varepsilon_0\varepsilon R\left(\phi_1^2+\phi_2^2\right)\left[\frac{2\phi_1\phi_2}{\phi_1^2+\phi_2^2}\ln\frac{1+e^{-\kappa H}}{1-e^{-\kappa H}}+\ln\left(1-e^{-2\kappa H}\right)\right]. \tag{7}$$

Equations (6) and (7) hold exactly for φ_1 or φ_2 of less than 25 mV and the particle radii should be greater than $10/\kappa$. Zeta potentials of magnetite, serpentine and pentlandite are -4.21, 8.08 and -18.01 mV, respectively, measured by the Nano-ZS900 Zeta Plus potential meter.

Wang et al. (1992) described the magnetic interaction U_M between magnetite and weakly magnetic minerals (such as hematite, pentlandite) due to the remanent magnetization of magnetite, which is originated by the ambient geomagnetic field, when no external magnetic field is added:

$$U_M = -\frac{2V_P\rho_P\chi_P R_M^6\sigma_0^2}{9\left(R_M+R_P+H\right)^6} \tag{8}$$

where V_P is the volume of a pentlandite particle and ρ_P its density, σ_0 is the permanent magnetization of magnetite (1040 $\mathrm{A\cdot m^{-1}}$), χ_P is the specific susceptibility of pentlandite (1130.4×10^{-8} $\mathrm{m^3\cdot kg^{-1}}$).

The interaction between two magnetic mineral particles was presented by Song (1988) in presence of an external magnetic field:

$$U_M = -\frac{2}{27}\pi R_M^3 R_P^3\chi_P\rho_P M\left[\frac{3B}{\left(R_P+R_M+H\right)^3}+\frac{10\pi R_M^3 M\mu_0}{\left(R_P+R_M+H\right)^6}\right] \tag{9}$$

$$M = \chi_M\rho_M\frac{B}{\mu_0} \tag{10}$$

where M is the magnetization intensity, B is the magnetic field intensity, μ_0 is the permeability of vacuum ($4\pi\times10^{-7}$ $\mathrm{H\cdot m^{-1}}$), ρ_M is the density of magnetite, χ_P and χ_M are the specific susceptibility of pentlandite and magnetite, respectively. The values of χ_M and χ_P are 364.2×10^{-6} and 527.5×10^{-8} $\mathrm{m^3\cdot kg^{-1}}$, respectively, under the external magnetic field intensity of 4776 $\mathrm{A\cdot m^{-1}}$ measured by the Gouy method.

Results and discussion

Interactions between minerals

In this study, the fine magnetite fractions were chosen. Because of the extremely fine particle size, it is assumed to be a small sphere, while serpentine belongs to the phyllosilicate mineral. Therefore, the interaction between them was calculated by the Eqs. (5) and (6) as shown in Fig. 4, mainly including the van der Waals U_A and electrical U_E interactions in the deionized water.

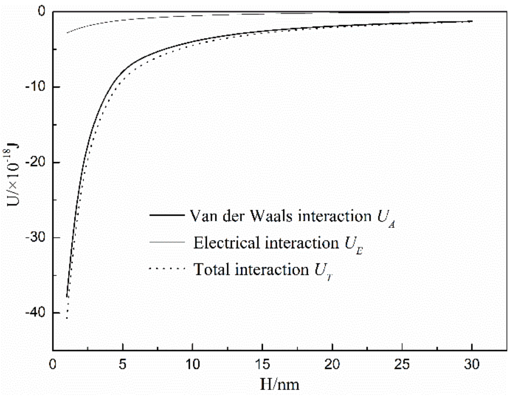

Fig. 4. Interaction of magnetite-serpentine particles as a function of distance between two mineral particles *H* in deionized water

As seen in Fig. 4, the total interaction between magnetite fines and serpentine particles was slightly attractive. Compared to the electrostatic attraction U_E, the van der Waals attraction U_A was much stronger and dominant between them. The closer the separation distance of particles, the stronger the attraction between them. The attractive interaction U_T for magnetite/serpentine occurred around 6 nm.

Other than the electrical U_E and van der Waals U_A interactions, there exists the magnetic interaction U_M between magnetic minerals, which has been confirmed by different reseراchers (Zhang et al., 1986; Wang and Forssberg, 1992). Therefore, the interaction was calculated using Eqs. (3) and (6) for the van der Waals U_A and electric U_E interactions, respectively, Eqs. (8) and (9) for the magnetic interaction U_M under the ambient geomagnetic field and an external magnetic field, respectively. The typical interaction curves of the magnetite/pentlandite particles are shown in Fig. 5.

From Figure 5, the electrostatic interaction U_E between magnetite fines and pentlandite particles was weakly repulsive due to their slightly smaller opposite surface charge, which was almost offset by the magnetic interaction U_{M1} from the remanent magnetization of magnetite by the ambient geomagnetic field. Compare to the van der Waals interaction U_A of magnetite-serpentine particles in Fig. 4, the interaction of magnetite-pentlandite particles was slightly stronger. After adding an external magnetic field of 4776 A·m^{-1}, the attractive interaction between fine magnetite particles and pentlandite particles became much stronger and almost unaffected by the separation distance between particles. Therefore, the total interactions U_T between them became much stronger than that between magnetite fines and serpentine particles. Thus, it indicated that there existed selective agglomeration between magnetite and pentlandite particles, and hence pentlandite could be separated from serpentine through the magnetic coating method, which was further confirmed by magnetic coating tests.

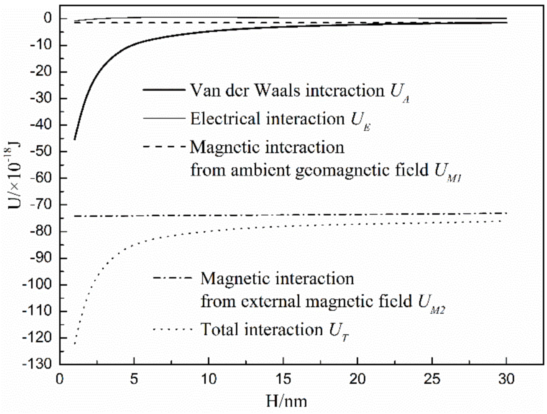

Fig. 5. Interactions of magnetite-pentlandite particles as a function of distance between two mineral particles *H* in deionized water

Results of the magnetic coating tests

Results of single mineral tests of pentlandite and serpentine are summarized in Fig. 6 as a function of magnetic field intensities. The amount of magnetite fractions added, as a percentage of the mineral, is in the range of 0–3%.

From Figure 6, it is clearly seen that the recovery of pentlandite was much higher than that of serpentine with or without the addition of magnetite. To obtain the same recovery, the required magnetic field intensity changed significantly with increasing amounts of magnetite. For example, the magnetic field intensity of 320 $kA\cdot m^{-1}$ achieved the pentlandite recovery of 85% without any addition of magnetite. When 1% and 3% magnetite fractions were added, the magnetic field intensities of achieving the same recovery reduced to 167 and 76 $kA\cdot m^{-1}$, respectively. In addition, it is found that the pentlandite recovery increased with increasing magnetite addition. Comparing no magnetite addition with magnetite addition of 3% at a magnetic field intensity of 200 $kA\cdot m^{-1}$, the recovery of pentlandite increased from 75.22 to 95.31%. However, the recovery of serpentine only slightly increased with increasing the magnetic field intensity and magnetite addition. Thus, it can be concluded that it is possible to separate pentlandite from serpentine using the magnetic coating method.

On the basis of above results, under a magnetic field intensity of 200 $kA\cdot m^{-1}$, the magnetic coating tests were carried out with the mixed minerals of serpentine and pentlandite of the mass ratio of 2:1 (the total mass was 15 g). When no magnetite fractions were added, the Ni grade of magnetic concentrates reached up to 24.81% and the corresponding MgO content was as low as 0.94%, whereas the Ni recovery reduced to 67.14%. This suggests that some pentlandite phases lost in the magnetic tailings. Further, with adding 3% magnetite fractions, although the Ni grade slightly decreased to 22.48% and the MgO content was up to 5.39%, the Ni recovery increased up to 86.01%. This clearly indicated that pentlandite particles were strongly coated by magnetite fines, which were congruent with the theoretical calculation results.

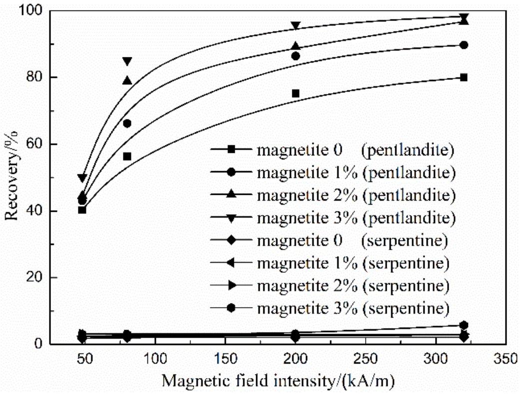

Fig. 6. Influence of magnetite amount added on magnetic coating under different magnetic intensities

Changes of surface morphology of pentlandite and serpentine particles by magnetic coating tests

Results from calculations of DLVO theory and magnetic coating tests showed that pentladite obtained a much stronger magnetic coating, and hence enhanced its magnetic property, resulting in being separated from serpentine minerals by magnetic separation. Therefore, the SEM study was undertaken to verify the selective coatings of magnetite between pentlandite and serpentine minerals and the results are shown in Fig. 7.

The images in Fig. 7 show that morphologies of magnetic concentrate (d) treated with magnetite fines are completely different from that of the pentlandite (b). It indicates that the coarse pentlandite surfaces were covered by magnetite ultrafines, whereas no surface magnetic coating was observed on the surfaces of serpentine (c), which still kept the same as the previous morphologies of the serpentine (a). From Figure 8 it can be seen that the nonmagnetic products were mainly serpentine particles. However, it can be found that there existed minor amounts of fine pentlandite particles (white particles, seen in Fig. 7 (c)) in the nonmagnetic products due to the weak interaction with the magnetite fines and lost in magnetic tailings (nonmagnetic products). EDS analyses of the magnetic products in Fig. 8 suggest that the coated particle was the pentlandite mineral, while the coating particle was magnetite fractions. Hence, it can be concluded that pentlandite minerals can be selectively coated by the magnetite fines, and then separated from serpentine minerals through the magnetic separation due to its magnetic property being enhanced.

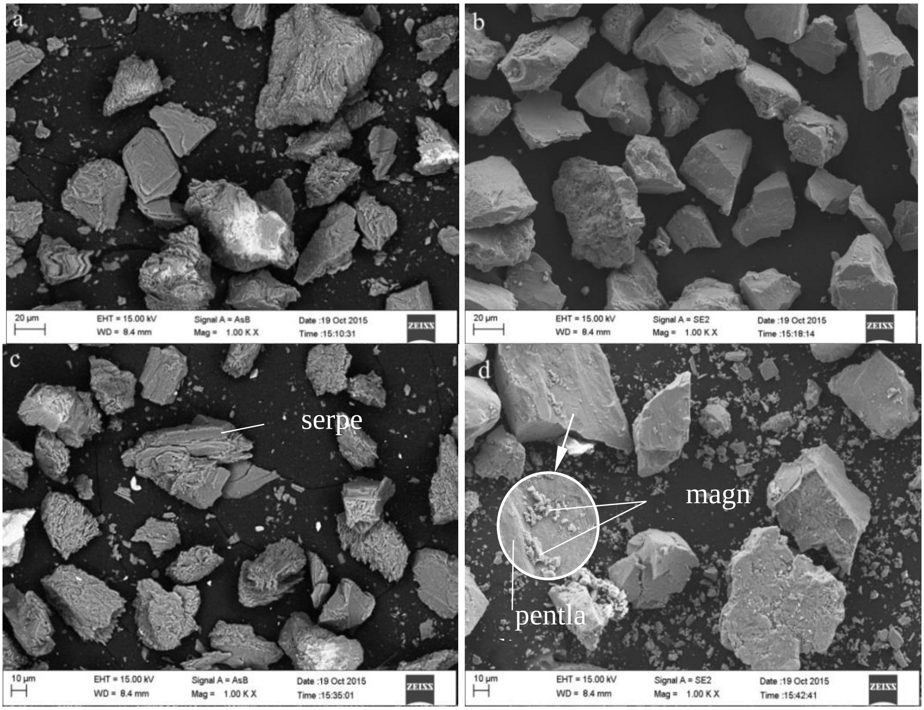

Fig. 7. SEM pictures of (a) serpentine, (b) pentlandite, (c) nonmagnetic products, and (d) magnetic products

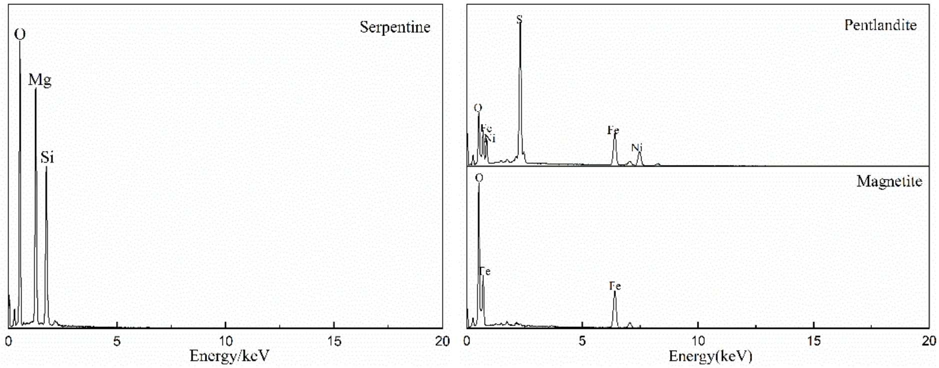

Fig. 8. EDS of nonmagnetic products and magnetic products

Conclusions

In this study separation of pentlandite from serpentine was attempted by the selective magnetic coating method, aiming to enhance the magnetic susceptibility of pentlandite minerals by incorporating small quantities of magnetite fractions for further magnetic separation. The major conclusions are as follows.

Calculations of DLVO theory show that when magnetite fines were added in the pentlandite/serpentine suspension system, the interaction between minerals (serpentine, pentlandite) and magnetite was mainly determined by the van der Waals interaction. The interaction of pentlandite-magnetite particles was slightly stronger than that of serpentine-magnetite particles. However, with applying an external magnetic field of 4776 $A \cdot m^{-1}$, interaction between magnetite and pentlandite particles became even stronger to lead selective agglomeration due to the magnetic interaction.

Theoretical calculations were further confirmed by magnetic coating tests and SEM analyses, which clearly showed that surfaces of pentlandite were coated by the magnetite fractions. Thus, pentlandite was easily separated from serpentine by magnetic separation.

Acknowledgments

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