



Canadian Metallurgical Quarterly

The Canadian Journal of Metallurgy and Materials Science

ISSN: 0008-4433 (Print) 1879-1395 (Online) Journal homepage: www.tandfonline.com/journals/ycmq20

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To cite this article: Thuat Tien Phung, Thanh-Hai Pham, Toi Trung Tran & Luan Van Pham (24 Apr 2026): Recover nickel laterite from the tailing pond of chromite ore processing: the difference in using high-sulfur coal and anthracite coal, Canadian Metallurgical Quarterly, DOI: [10.1080/00084433.2026.2662798](https://doi.org/10.1080/00084433.2026.2662798)

To link to this article: <https://doi.org/10.1080/00084433.2026.2662798>



Published online: 24 Apr 2026.



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Recover nickel laterite from the tailing pond of chromite ore processing: the difference in using high-sulfur coal and anthracite coal

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ABSTRACT

This article presents a case study on nickel recovery from the tailings pond of a chromite ore processing plant in Vietnam. After chromite beneficiation, a significant amount of nickel remains in the tailings, requiring recovery to ensure the supply of this valuable metal for industrial development and to promote the rational, economical use of resources. Previous studies indicate that nickel in the sample occurs mainly as laterite. However, its low-grade, complex mineralogical characteristics and the limitations of conventional beneficiation techniques pose major challenges to effective recovery. A promising approach involves roasting – reduction followed by magnetic separation, in which laterite nickel is reduced using either anthracite or high-sulfur coal. Experimental results demonstrate distinct effects of the two coal types on the reduction behaviour of nickel laterite ore. After the reduction process, magnetic separation is applied to obtain nickel concentrates. The resulting concentrate contains 5.27% Ni with a recovery rate of 77.88%. The results highlight the potential of using high-sulfur coal as a viable alternative to anthracite as a reducing agent for nickel laterite ore. This approach not only lowers production costs but also enhances the value of sulfur-rich coal.

Cet article présente une étude de cas sur la récupération du nickel contenu dans le bassin de résidus d'une usine de traitement de minerai de chromite au Vietnam. Après l'enrichissement de la chromite, une quantité importante de nickel reste dans les résidus, exigeant une récupération pour garantir l'approvisionnement en ce métal de valeur pour le développement industriel et pour favoriser une utilisation rationnelle et économique des ressources. Des études précédentes indiquent que le nickel dans l'échantillon se présente principalement sous forme de latérite. Cependant, sa faible valeur, ses caractéristiques minéralogiques complexes et les limites des techniques d'enrichissement conventionnelles posent des défis majeurs pour une récupération efficace. Une approche prometteuse implique un procédé de grillage-réduction suivi d'une séparation magnétique, où la latérite nickélifère est réduite en utilisant soit de l'anthracite ou du charbon à haute teneur en soufre. Les résultats expérimentaux démontrent des effets distincts des deux types de charbon sur le comportement de réduction du minerai de latérite nickélifère. Après le procédé de réduction, on applique une séparation magnétique pour obtenir les concentrés de nickel. Le concentré obtenu contient 5.27% de Ni avec un taux de récupération de 77.88%. Les résultats soulignent le potentiel de l'utilisation du charbon à haute teneur en soufre comme choix viable par rapport à l'anthracite en tant qu'agent réducteur pour le minerai de latérite nickélifère. Non seulement cette approche diminue les coûts de production, mais elle améliore également la valeur du charbon à haute teneur en soufre.

ARTICLE HISTORY

Received 16 May 2025
Accepted 13 April 2026

KEYWORDS

High-sulfur coal; anthracite coal; nickel laterite; magnetic separation; recovery of nickel

Introduction

Nickel is a valuable metal that occurs in nature in two types of ores: sulfide ores and oxide ores (laterite ores), accounting for about 30% and 70% of reserves, respectively. However, 60% of the world's nickel is produced from sulfide ores [1,2]. The reason is that nickel laterite ores often have low nickel content and are difficult to process using conventional beneficiation and metallurgical methods. Sulfide ores that are easier to process are mined first, and their reserves are

increasingly depleted. While, in terms of benefits, laterite nickel ores have two significant development advantages: first, they are abundant in reserves; second, these ores are formed directly on the ground or in the tailing pond of other mineral processing plants and are easy to exploit [3–5]. This is a favourable condition for developing processing and purification technology for these ores [5–7]. To ensure the supply of nickel for other high-tech fields and to use mineral resources rationally and sustainably, it is essential to develop an efficient process for processing laterite nickel ores. Therefore,

studying options for treating laterite nickel ore is increasingly being considered [4,5,8–10].

In laterite ores, nickel is distributed among many different minerals, but is richest in nontronite, serpentine, and iron oxide minerals (goethite/limonite) [5,11,12]. This ore has a low nickel content (0.5–2.5% Ni) and is usually divided into three layers: limonite, saprolite, and garnierite [13,14]. Traditional processing methods (like flotation, gravity separation, etc.) to increase nickel content are ineffective [5,15].

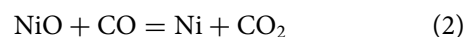
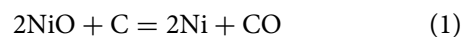
There are two preferred methods for recovering Ni from nickel laterite ore: hydrometallurgy and pyrometallurgy. In hydrometallurgy, the methods mentioned are (1) reduction roasting – ammonia leaching (Caron method) [2,16,17]; (2) high-pressure acid leaching [2] with the inorganic acids such as sulfuric acid [2,18–20], nitric acid [21] and hydrochloric acid [22]; (3) standard pressure acid leaching with the same types of acid above [23–28] or citric acid and organic acids. It can be seen that those methods have a low energy cost in advance, but are complex and time-consuming [29]. In addition, issues related to the recovery solvent and environmental treatment after processing are challenges.

Another method is pyrometallurgy, which includes (1) the production of ferronickel by reduction roasting in a rotary kiln-electric furnace smelting, and (2) the production of nickel mattes in reaction smelting [15,30,31]. This method is simple but energy-consuming and requires laterite ores with high nickel content.

To overcome the limitations of pyrometallurgical processes, scientists have recently proposed replacing the smelting step with magnetic separation to recover ferronickel particles generated during the reduction stage. Reduction-magnetic separation is a new and promising process for processing nickel laterite ores, with high potential for practical application, because the process is simple, consumes little energy, and can process different types of nickel laterite ores [16,32].

In the reduction-magnetic separation process, the reduction stage is a crucial step that determines the method's effectiveness. During this stage, the main chemical reactions are the reduction of metal oxides and the formation of Fe-Ni particles. The metals Fe and Ni are magnetic, and therefore will be separated from other non-magnetic impurities by the subsequent magnetic separation process. The sample composition consists primarily of metal oxide minerals. Besides Fe, Ni, and Co, the sample also contains other metals in significant quantities, including oxides of Al, Cr, Mn, Mg, Ca, and Ti. To reduce the metal oxides, coal is chosen as the fuel to provide carbon for the reduction reactions. The reduction reactions of the metals in the sample

have been described in previous studies [33–36]. Accordingly, the reduction reaction of nickel with carbon follows the following equations:



With iron and cobalt, the process mechanism is similar to that of nickel. The reduction reaction can occur directly by C or CO gas [35]. Reactions can occur to a very limited extent due to the mobility of C and metal oxides in the solid state, or more readily due to the mobility of CO gas in the gaseous state. The reduction process by carbon is essentially an indirect reduction process by CO gas produced from the gasification reaction of carbon (reaction 3). For oxides of other metals, such as CaO, Al₂O₃, Cr₂O₃, etc., these metals can also be reduced by CO [34]. To assess the possibility and extent of the reactions and under what conditions they occur, it is necessary to determine the Gibbs free energy (ΔG) change of those reactions. Gibbs free energy change is an important quantity in chemical thermodynamics, as it determines the reduction products and the conditions that favour reactions proceeding in the desired direction. In metallurgy, the Ellingham diagram is an effective tool for determining the equilibrium conditions of a reaction between a metal, its oxide, and oxygen. The Ellingham diagram is a graph of the change in Gibbs energy (ΔG) for each oxidation reaction as a function of temperature. The diagrams are very useful in predicting the conditions under which metal oxides will be reduced to metal [37].

After the reduction process, nickel and iron are in the form of strongly magnetic metals. And the recovery of concentrates containing Fe and Ni is carried out by magnetic separation. Therefore, the content and recovery of Ni depend heavily on the magnetic field strength. Recent studies have also implemented schemes combining thermal pretreatment and magnetic separation in the recovery of laterite nickel ore [38]. As mentioned previously, considerable research has been performed on the behaviour of the nickeliferous laterites during reduction roasting [36,39–53]. Several studies in the literature have used reduction roasting followed by magnetic separation to produce ferronickel ore. Factors such as ore composition, reduction conditions, nickel content, and nickel recovery efficiency are reported in each study.

In the studies mentioned above, no additional additives were added. Many inexpensive carbon-containing

reducing agents were used, including: charcoal, bituminous coal, spent carbon cathodes, lignite, and anthracite. Additionally, one study used hydrogen as a reducing agent. Operating temperatures and reaction times also varied. Typically, operating temperatures ranged from 1000°C to 1250°C, with an average of around 1150°C, while reduction times ranged from approximately 30 to 90 min, with an average of around 60 min. The reducing agent, operating temperature, and reduction time are all thought to influence nickel content and nickel recovery efficiency from the ore. Temperature and reduction time determine both the reduction kinetics and the degree of proximity to equilibrium, as well as the thermal activation development of ferronickel particles. These operating temperatures are significantly higher than predicted from existing thermodynamic models. Previous modelling studies have shown that to reduce most of the nickel in limonite ore effectively, temperatures of around 600°C are required [54]. Elliott et al. reported a nickel recovery rate of only 15% for limonite ore at 600°C [55]. The holding time at a given temperature is the energy consumption time; the longer the time and the higher the temperature, the higher the cost. However, there is little literature on the kinetics of subsequent processes, such as magnetic separation of nickel-containing limonite ore.

To increase the efficiency of the reduction-separation process, scientists worldwide have added additives. In the study of Jiang et al. (2013), it was reported that adding 10% sodium sulfate to the reduction process of nickel laterite ore (1.49% Ni and 34.69% Fe) at 1200 °C for 50 min, the reduced sample was magnetically separated and the product obtained had a nickel content of 10.86% and a recovery rate of 88.56% [56]. In 2015, in his doctoral thesis, Elliott experimented with sulfur additives and found that non-magnetic FeS phases appeared in the reduced product. This limited Fe from entering the magnetically separated concentrate, thereby increasing the Ni content in the obtained product [7]. Shiwei Zhou et al. (2015) compared three additives (Na_2SO_4 , Na_2S , and CaSO_4); Na_2S had a stronger effect on the phase transition of laterite nickel ore. Meanwhile, CaSO_4 also increased Ni content and recovery rate but required high temperatures above 1300°C, resulting in high energy consumption. Among the three additives, Na_2SO_4 was found to promote the growth of ferronickel and improve its nickel content the most. Increasing the dosage of Na_2SO_4 could promote the polymerisation of ferronickel and improve the nickel content in ferronickel [57]. In 2018, Xueming et al. used a reduction-separation process to treat laterite ore containing 1.8% Ni and 19.9% Fe, with the addition of 8% Na_2SO_4 [58,59]. The results obtained were

ferroniken products with a Ni content of 9.7% and a nickel recovery rate of 89.1% at a reduction temperature of 1420°C for 40 min [59]. In 2018, Shofi et al. conducted experiments comparing the additives NaCl , Na_2CO_3 , and Na_2SO_4 . Laterite nickel ore originating from Southeast Sulawesi, Indonesia, had a Ni content of 1.4% and a Co content of 0.07%. The results showed that, under optimal conditions at 1150°C, using Na_2SO_4 yielded the highest Ni concentration (4.6%) and a recovery rate of 73%. While using Na_2CO_3 , the Ni content in the concentrate was 2.5%, and the Ni content in the concentrate was lower than 2% when using NaCl . The addition of sodium sulfate produces ferronickel particles three times larger than those produced with sodium carbonate or sodium chloride [60]. Nurjaman showed that adding Na_2SO_4 to the selective reduction of low-grade laterite nickel ore with 1.4% Ni content can increase the nickel content to 6.09% at 1150°C for 60 min. In addition, the presence of Na_2SO_4 increases ferronickel particle size, thereby positively affecting the magnetic separation process and increasing nickel metal concentration. Meanwhile, the addition of the reducing agent reduces the nickel level in the concentrate by promoting the optimal reduction of iron oxide to metal [57]. In the comprehensive study of Jingcheng Dong et al. on the effects of additives, it was also shown that most studies highly appreciated Na_2SO_4 . The addition of additives increases the efficiency of iron and nickel reduction, creates a low-melting slag, enhances Ni diffusion into the iron phase, and forms larger ferronickel particles, thereby improving the efficiency of selective separation in the magnetic separation stage [58]. In another verification study by Suharno et al. in 2021 with additives Na_2SO_4 , NaCl and Na_2CO_3 , the results showed that sodium sulfate was better, with a mixing ratio of 15%, reduction at 1150°C, the highest nickel content in the concentrate was 15.06%, while the concentrate only reached 2.18% Ni with Na_2CO_3 additive, and 2.27% Ni with NaCl additive. The sulfidation mechanism, which can inhibit iron metallization, increases the concentrate's nickel content more effectively than the carbonate decomposition and chlorination processes. The sulfur content in the reducing agent also affects the selective reduction process. It promotes greater iron sulfidation, thereby reducing the amount of iron entering the concentrate and increasing the nickel content [61]. Thus, it can be seen that additives play an important role in the reduction process, improving the accumulation and growth of Fe-Ni particles, thereby increasing selectivity in the magnetic separation stage and helping the obtained concentrate product have higher content and recovery. Many additives have been studied by scientists

worldwide, including NaCl , CaCl_2 , CaSO_4 , Na_2CO_3 , S , Na_2S , and Na_2SO_4 .

According to some research, at high temperatures, Na_2SO_4 decomposes into Na_2S or S . These products then react with FeO and Fe to form FeS . FeS forms around FeO and prevents the reduction of Fe , thereby limiting Fe 's entry into the ore concentrate [58,59,62,63]. In addition, the sulfur produced in the mixture can promote the aggregation and growth of ferromagnetic particles by reducing the surface tension of Fe-Ni alloy particles [56,64]. Jingcheng Dong synthesised studies worldwide and reached a consensus that, during high-temperature reduction in a CO atmosphere, the Na_2SO_4 additive decomposes to produce Na_2S or S . These products then react with Fe and Ni oxides and metals to form the corresponding sulfides. Nickel sulfides are less stable and are reduced by iron [58].

Accordingly, during the reduction roasting stage, nickel and iron are reduced to metal at 1000–1200°C. The ore after reduction roasting is finely ground, and the ferromagnetic particles are separated by wet magnetic separation, with a nickel recovery rate of over 90%. The purpose of the magnetic separation process is to increase the nickel content in the ferromagnetic product. Nickel must be reduced to metal as much as possible, and iron must be reduced to a limited extent to achieve selective nickel reduction and obtain a ferromagnetic product with a high nickel content. One of the additives added during the process is coal (including high-sulfur coal and anthracite coal, with sulfur content below 0.5). Coal not only plays a role in energy supply but also supports the reduction process. The different types of coal result in varying Ni recovery efficiencies in the products due to sulfur present during the reduction roasting stage. However, the phenomenon has not yet been mentioned or researched before.

This article presents the results of recovering this type of nickel from the tailing pond of mineral processing plants (in the case study of Vietnam) using reduction roasting and magnetic separation with both types of coal. In addition, the research results show that reduction roasting with high-sulfur coal offers significant advantages over conventional coal. The result is not only to contribute to the prospects of recovering Ni efficiency but also to use high-sulfur coal effectively in laterite tailing treatment. It has significant meaning in the context of the global innovation trend and the circular economy.

Materials and methods

Samples

In the case study of Vietnam, nickel was discovered in 02 metamorphic chromite ore deposits: Nui-Nua

(Nong-Cong district), Mun village (Ngoc-Lac district), and Co-Dinh chromite placer (Thanh-Hoa province), with total reserves of about 3,067,020 tonnes of nickel [5,33,34]. The results of the sample composition analysis showed that the tailings sample from the chromite beneficiation process at Co Dinh, Thanh Hoa, contained 1.16% nickel. This metal occurs as oxide minerals, replacing Fe and Mg in chlorite minerals. The results of the mineral composition analysis are similar to those of Nguyen Khac Giang in 2020, who determined that the tailings from the chromite beneficiation process belonged to the laterite nickel ore type containing Ni . Clay and goethite minerals are also places where Ni is adsorbed [65]. In addition, according to the studies of Le Tien Dung and the research group of the Centre for Mineral Technology Development, Hanoi University of Mining and Geology, iron-manganese colloid particles were detected in the tailings, which also provide a Ni adsorption environment during the formation process [66]. However, Ni has not been considered a valuable metal in the chromite extraction process. Over 60 years of chromite mining activities in the mine areas have resulted in very large-scale sludge deposits containing Co and Ni , which can be considered valuable deposits of nickel-containing minerals. The main minerals in these ores are montmorillonite, antigorite, chlorite, quartz, goethite, talc, and illite. Nickel exists in the form of oxides, interspersed in the main mineral chlorite and possibly in the minerals montmorillonite, antigorite, goethite, etc. [5,33,34]. The ore type is low-grade laterite nickel ore, which is challenging to extract using conventional smelting and metallurgical technologies. Magnetic extraction schemes have also been tested on this type of ore, with low efficiency.

A combination of reduction roasting – magnetic separation is one of the methods considered more effective for separating nickel from laterite nickel ores. In particular, the reduction process serves as a pre-treatment and determines the quality of the concentrate obtained from magnetic separation [35–37]. Hard coal is the primary reducing agent used to reduce laterite ore. In addition, several additives are also included to improve reduction efficiency, notably sulfur-containing additives. Recent studies showed that sulfur or its compounds can form weakly magnetic iron sulfides during the reduction process, thereby limiting iron from entering magnetic products (concentrates containing Ni). Sulfur also increases nickel recovery efficiency in the magnetic separation stage by increasing the size of ferromagnetic particles formed during the reduction stage [37–39]. Thus, by using coal as a reducing agent in combination with sulfur or sulfur-containing compounds, it is effective for recovering nickel and cobalt from laterite

Table 1. Mineralogy composition of the tailing sample.

Mineralogy	Content (%)
Amphibole	2–4
Montmorillonite (Na,Ca) _{0.3} (Al,Mg) ₂ Si ₄ O ₁₀ (OH) ₂	21–23
Chlorite: (Mg,Ni)₃Si₂O₅(OH)₄	8–10
Goethite: Fe ₂ O ₃ ·H ₂ O	33–35
Talc: Mg ₃ [Si ₄ O ₁₀](OH) ₂	10–12
Antigorite: 3MgO·2SiO ₂ ·2H ₂ O	22–24
Illite: KAl ₂ [AlSi ₃ O ₁₀](OH) ₂	7–9
Quartz: SiO ₂	10–12
Feldspar: K _{0.5} Na _{0.5} AlSi ₃ O ₈	2–4

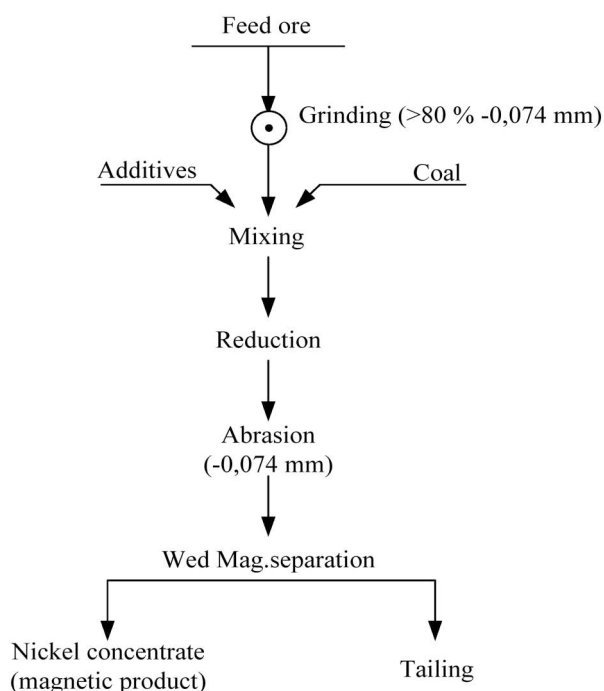
nickel ores. If high sulfur coals such as Na-Duong coals (Vietnam) (with the content of 7.66% S) can be used, then it can significantly reduce the cost of the process because Na-Duong coals are much cheaper than regular anthracites. The sulfur in coal benefits the reduction process, increasing the efficiency of nickel recovery.

The studies were conducted with tailings samples from a chromite ore mine in the Mau-Lam area, Thanh-Hoa province, Vietnam. The mineralogical composition of the sample was determined using an X-ray diffractometer (D8-Advance), and its chemical composition was analysed by ICP–MS. The tailings from the chromite ore beneficiation process are a nickel-containing laterite ore (as shown in Table 1); the samples contain 1.179% Ni (as shown in Table 2).

Test procedure

The initial tailings samples were dried, then finely ground (more than 80% of particles passing through a 0.074 mm sieve), thoroughly mixed, and divided into small samples for each experiment. The mass of the ore sample for each experiment is 50 grams. The coal used for the experiment was also dried and ground to a size of below 0.1 mm.

In each experiment, coal was mixed with the ore sample and placed in a graphite beaker with a lid. When the furnace temperature reached the required roasting level, the sample was brought into the furnace for reduction. According to the reference documents, 90 min is sufficient to achieve maximum reduction [35,37,40]. The reduced sample is allowed to cool in the same furnace until the furnace temperature is below 50°C, then ground to a size below 0.074 mm, and then separated in a wet magnetic separator. The magnetic field strength in these experiments was chosen to be 1000 Gauss. With this magnetic field strength, it is

**Figure 1.** Experiment procedure flowsheet.

possible to limit the entry of large amounts of low-magnetic impurities into the magnetic product, ensuring a high-grade nickel concentrate. The experimental procedure is shown in detail in Figure 1.

The samples used in the experiments, Na-Duong coal (high-sulfur coal, denoted as ND coal) and Vang-Danh anthracite (normal Vietnamese anthracite, denoted as VD coal), have the compositions shown in Table 3. The Na-Duong coal sample has a high sulfur content of 7.66%.

Results and discussion

Effect of temperature on the reduction process

In these experiments, the coal mixing ratio was 10% and the reduction time was 90 min. The reduction temperature was investigated in the range from 900 to 1200°C. The reduced product was magnetically separated at a magnetic field intensity of 1000 Gauss.

Experimental results shown in Figure 2 indicate that, in the investigated temperature range, increasing temperature will increase the actual recovery of Ni into the concentrate product. However, the Ni content in the concentrate increased significantly only in samples that were reduction-treated at temperatures between 900 and 1100°

Table 2. Results of the ore sample analysis.

Composition	Fe ₂ O ₃	Ni	Al ₂ O ₃	CaO	Co	Cr	MgO	MnO	Others
Content (%)	29.17	1.179	5.19	1.26	0.259	2.06	11.02	6.81	43.05

Table 3. Composition of the Na-Duong and Vang-Danh coals.

Composition	Na-Duong coal (ND coal)	Vang-Danh coal (VD coal)
Ash (A ^k), %	27.56	12
Volatile (%)	33.66	7.5
Sulfur (%)	7.66	< 0.5

C. As temperatures continue to rise, the nickel content in the concentrate remains almost unchanged. Another problem is that at 1200 °C, the sample has high fluidity, making subsequent size reduction more difficult. Research results also showed that at a reduction temperature of 1100°C, the product's nickel content and recovery degree are not much different from those at 1200°C.

When using Na-Duong coal, the magnetic concentrate product has a higher nickel content than when using Vang-Danh coal. This difference shows that the sulfur composition in Na-Duong coal has contributed to the formation of weakly magnetic iron sulfide compounds, which prevent them from entering magnetic products.

Effects of coal ratio in the reduction mixture

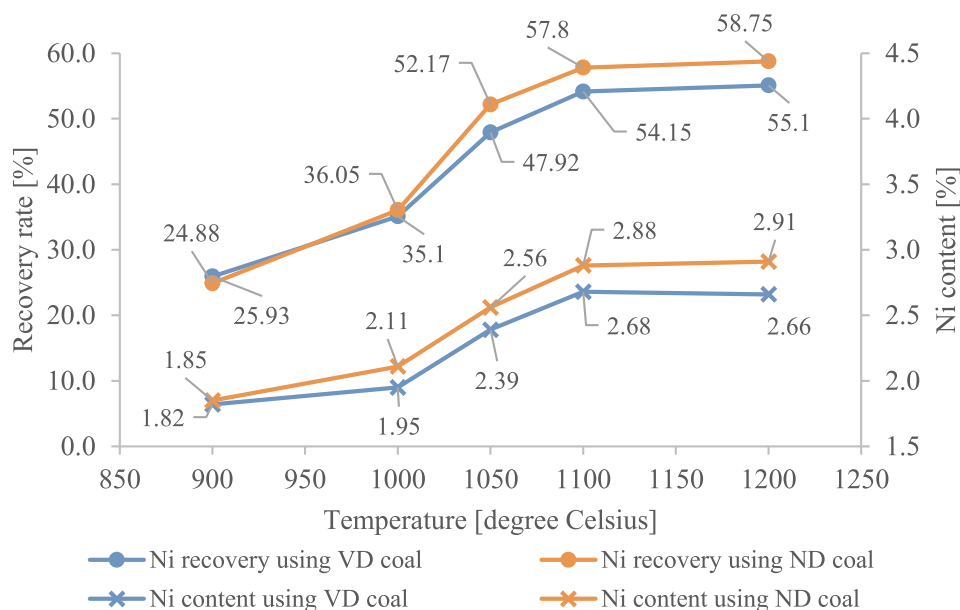
Coal is the primary source of carbon for the reducing reactions. In addition, Na-Duong coal contains high sulfur, so changing the amount of coal also changes the amount of sulfur supplied, supporting the limit on the reduction of iron. The studies on the coal ratios in the mixture were carried out at 1100°C, with coal ratios ranging from 5% to 12.5%; the reduction time was 90 min. The recovered samples were magnetically separated at a magnetic field strength of 1000 Gauss.

The research results shown in Figure 3 indicate that increasing the coal proportion from 5% to 10% increases the nickel reduction rate, thereby increasing the recovery of Ni in the magnetic concentrate after separation. However, increasing the coal proportion to 12.5% resulted in a maximum metal reduction, and thus the nickel content and recovery increased insignificantly.

Comparing the two types of coal, the difference is not significant, but they differ at various coal ratios. At a low coal ratio (<7.5%), using Na-Duong coal resulted in lower Ni content and recovery than using anthracite (Vang-Danh coal). This may be due to Na-Duong coal having higher volatile matter, ash content, and lower carbon content, which is insufficient to ensure metal reduction. Increasing the coal ratio to over 7.5% not only increased the carbon content but also increased the sulfur content, making the sulfur effect in the coal more pronounced. As a result, the concentrate quality improved compared to that obtained with anthracite coal. This was even more evident when the coal ratio was increased to 10% and 12.5%. Results show that the nickel recovery rate and nickel content in magnetic separation concentrates from the use of Na-Duong coal are higher than those from Vang-Danh coal.

Formation of iron sulfide in the reduced products

To assess the formation of iron sulfide in the reduced product, the samples after reduction at 1100°C with a coal ratio of 10% for 90 min were subjected to SEM-

**Figure 2.** Effect of temperature on recovery rate and Ni content in magnetised concentrate.

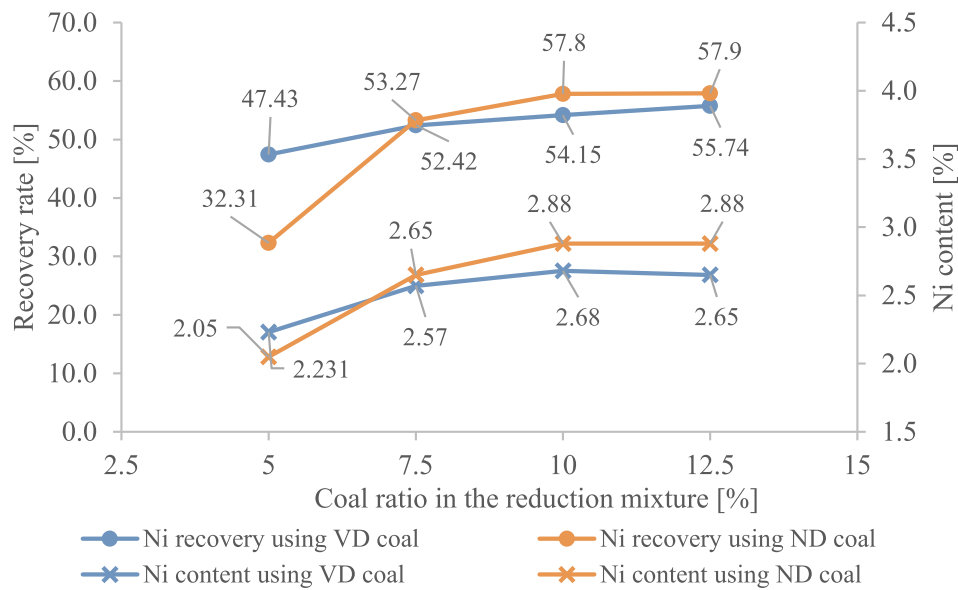


Figure 3. Effects of coal ratio on recovery rate and Ni content in magnetic concentrate.

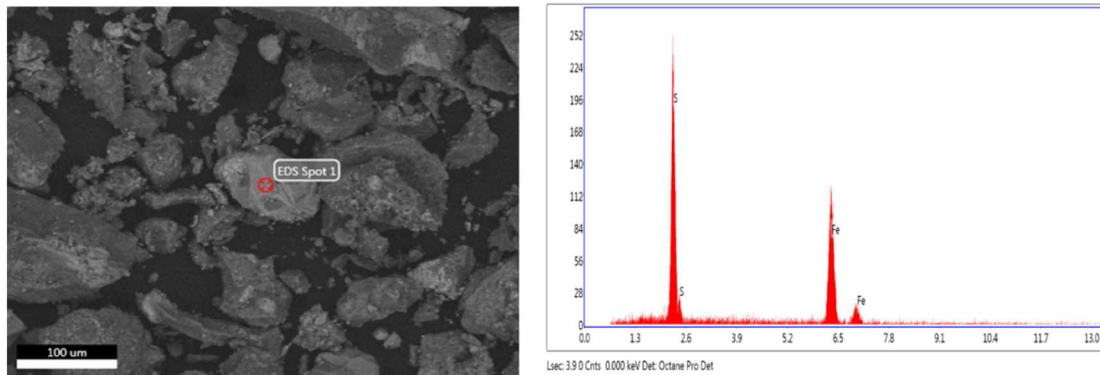


Figure 4. Results of the SEM-EDS analysis of the sample after reduction at 1100°C.

EDS analysis. These analyses were performed at the Centre for Excellence in Analysis and Experiment, Hanoi University of Mining and Geology. The study results shown in Figure 4 indicate that, in the reduced samples, particles containing the principal components Fe and S appear. This proves that sulfur in coal participates in the formation of iron sulfide.

The formation of iron sulfide has a positive effect in limiting iron from entering the concentrate product [37,38,40]. However, the number of iron sulfide particles found is not too much. The reason for this is that the sulfur content of the reduction mixture was only 0.766% (sulfur in the coal was 7.66%, while the total amount of mixed coal was only 10%). The sulfur in the reduction mixture was much less than the amount of iron in the ore (29% Fe_2O_3).

Comparing the morphological characteristics of the samples (Figure 5) under the same reduction conditions (1100°C, 10% coal mixing ratio, 90 min), melting was

observed during reduction with Na-Duong coal. In contrast, Vang-Danh coal exhibited a solidified, dense consistency and a lack of fluidity. This further demonstrates the effect of sulfur in forming the Fe-FeS eutectic system with a low melting point [41].

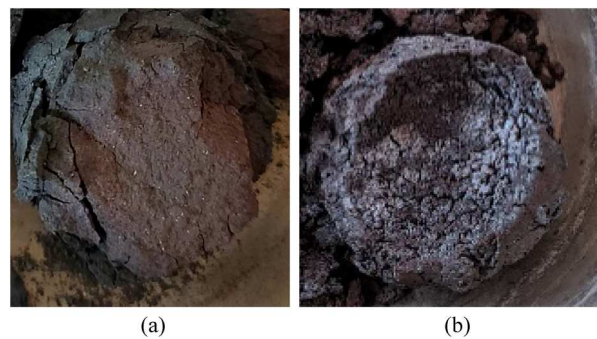


Figure 5. Sample image after reduction at 1100°C. a. Using the Anthracite coal (VD coal); b. Using the high-sulfur coal (ND coal).

Figure 6 shows the SEM image of the sample after reduction at 1100°C with a coal mixing ratio of 10% for 90 min. These are the optimal conditions determined from the survey experiments. It can be observed that the Fe-Ni particles formed when using Vang-Danh coal are very small (1–2 μm). In contrast, the Fe-Ni particles formed when reducing with Na-Duong coal are significantly larger (5–10 μm). However, the observed Fe-Ni particles (in both cases) are still relatively small. This indicates that the sulfur content of the coal has not significantly promoted the growth of Fe-Ni particles. To improve Ni recovery efficiency, the reduction process still requires an additional additive.

Reduction in the presence of additives

Many types of additives can be used for such a reduction process. This article presents the results of reduction tests with Na_2SO_4 . Previous publications have found this type of additive to be the most effective for the reduction of nickel laterite ores [37–40]. The tested

additive participation rates were 0, 3, 5, 8, and 10%, and the coal mixing ratio was 10%, reduced at 1100°C for 90 min. The additive forms iron sulfide, preventing iron from entering the magnetic concentrate. Sample images and experimental results are shown in Figure 7 and Figure 8.

The morphology of the sample after reduction (Figure 7) shows that samples reduced without additives exhibit a porous texture. In contrast, those reduced with additives exhibit a denser texture. It is demonstrated that additives had created a slag system with a lower melting temperature and lower slag consistency, which produced more favourable conditions for the diffusion of Ni and Fe, leading to the concentration of nickel to form ferronickel particles with strong magnetism and larger sizes [37–40].

The results of the study shown in Figure 8 also indicate that the obtained concentrates have higher recovery and content than in the case without additives. However, it should be noted that dense cakes are more challenging to size reduce. In addition, if

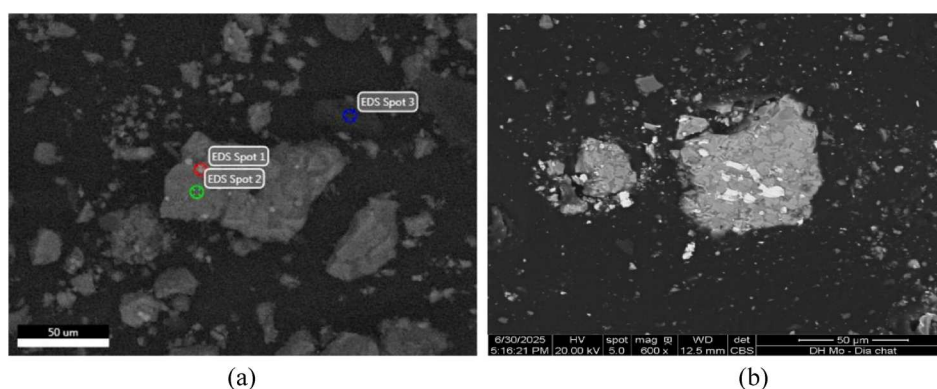


Figure 6. SEM image of magnetic concentrate after reduction at 1100°C. a. Using Vang-Danh coal; b. using Na-Duong coal.



Figure 7. Sample image after reduction at 1100°C. a. With no additives; b. With the use of Na_2SO_4 additive.

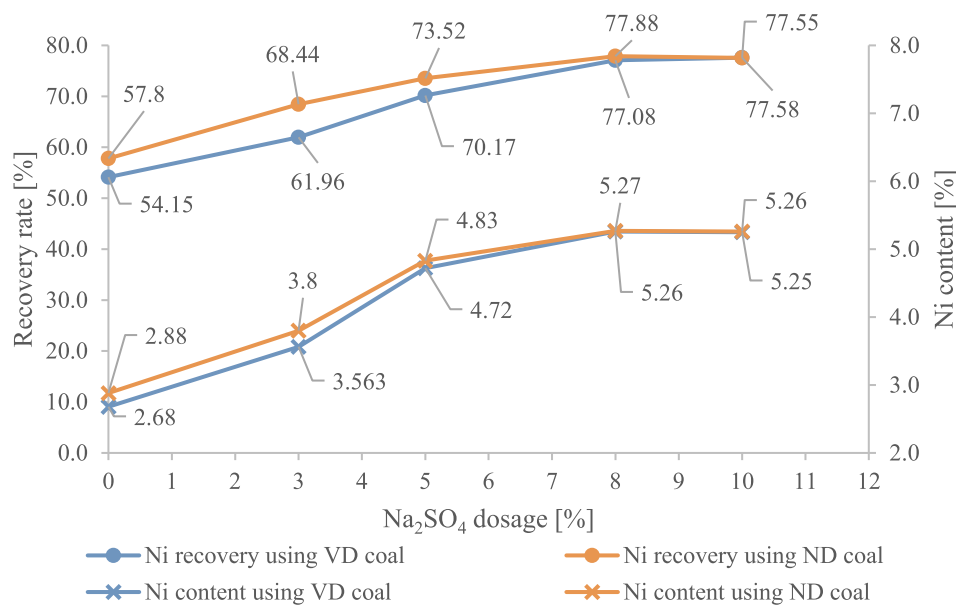


Figure 8. Effects of Na₂SO₄ on recovery rate and Ni content in magnetic concentrate.

the amount of iron sulfide formed is too much to cover the ore grains, then it has a reverse effect; it interferes with the process of reducing iron and nickel to form ferronickel particles. Moreover, nickel present in iron sulfides will not enter the concentrate during magnetic separation.

The research results also showed that the resulting concentrate (when reduced with additives) had a higher yield and content compared to the case without additives. Further comparison with the Vang-Danh coal case revealed that, with additives present, the differences in Ni content and yield were not significant. Meanwhile, when using Na-Duong coal, both Ni content and yield were higher than when using Vang-Danh coal. At a Na₂SO₄ ratio of 3%, the differences were 3.8–3.56 = 0.2% and 68.44–61.96 = 6.48%, respectively. This difference decreased with increasing Na₂SO₄ ratio and was nearly equal at 8% and 10%. The reason for this difference may be that at low additive ratios, where the additive's effect is low, the impact of S in the coal is more pronounced.

Conclusions

Analytical results show that the Na-Duong coal sample used in the experiment contains up to 7.66% sulfur. This sulfur content is considered to enhance the reduction process. The analysis also found particles containing the principal components Fe and S in the reduced product. Although there is no specific analysis of the form present, it can be confirmed that sulfur in the coal has participated in the formation of iron sulfides.

Compared to Vang-Danh coal, the use of Na-Duong coal allows the production of concentrate products with a similar nickel recovery rate but higher nickel content. Furthermore, the SEM images show the formation of larger Fe-Ni particles when high-sulfur coal is used for nickel reduction.

The additive Na₂SO₄ has significantly improved the reduction process. Compared to using only coal, the addition of 5% Na₂SO₄ during the reduction process increased the nickel content in the magnetic concentrate from 2.88 to over 4.83%, and the recovery rate reached over 73.52% (in the case of using high-sulfur coal). The result for using anthracite is lower, ranging from 2.68 to 4.72%, with a recovery of 70.17%.

For additive contents of 8% or more, the process-improvement role of high-sulfur coal compared to anthracite is negligible. However, since high-sulfur coal generally has low economic value as it cannot be used for conventional purposes such as thermal power, heating and fuel, chemical feedstock, other industrial processes, etc., its use is significant in the integrated use of resources, saving costs, and increasing the efficiency of mineral processing tailing treatment, aligning with the circular economy and green economy trends.

Research results have shown the prospect of using Na-Duong coal, a type of high-sulfur-containing coal, as a reducing agent in the reduction-magnetic separation process to recover nickel in low-content nickel laterite ores from tailing, especially in Vietnam. However, more research is needed on other technological parameters, such as reduction time, additive mixing

ratio, size-reduction parameters, etc., to provide a flowsheet and technological regime for applying high-sulfur coal in actual production.

Further research should be conducted: (1) Comprehensive chemical composition and characterisation analyses of the coal used, including anthracite and high-sulfur coal, are necessary to clarify the suitability of the coal under study; (2) The magnetic and non-magnetic fractions from magnetic separation need to be analysed for their chemical and mineral composition, to clarify phase formation after reduction and demonstrate the formation of iron silicate phases; (3) The influence of other minerals and their interactions on ferro-nickel formation and the overall process (including reduction, phase transformation, melting behaviour, or magnetic separation performance) should be investigated; (4) Phase analysis and phase transitions at different temperatures, coal reduction ratios, and additive amounts should be performed.

Acknowledgments

The authors would like to thank all colleagues at Hanoi University of Mining and Geology and Vinacomin for their analysis and discussion. Further, thanks to the companies Na-Duong coal mine, Vang-Danh coal mine, and Co-Dinh chromite joint stock company for their technical support and sample supply.

Declaration of generative AI and AI-assisted technologies in the writing process

During the preparation of this work, the authors used Grammarly to improve the language and readability. After using this service, the authors reviewed and edited the content as needed and take full responsibility for the publication's content.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Data availability statement

All data generated or analysed during this study are included in this published article.

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