

## JOINT PROCESSING OF V-BEARING STEELMAKING SLAG AND V-BEARING BLACK SHALE FOR VANADIUM AND IRON SEPARATION

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### Abstract

To recycle vanadium from V-bearing steelmaking slag and V-bearing black shale, both were jointly roasted to generate vanadium-rich phase, and then vanadium was separated by magnetic separation in this study. The compositions of samples were determined by X-ray fluorescence meter and the phases in the samples were characterized using X-ray diffractometer. The experimental results showed that with increasing the ratio of CaO content to SiO<sub>2</sub> content in the samples the vanadium separation efficiency first decreased, increased, and then decreased again. With increasing roasting temperature from 1423 to 1623 K, the vanadium separation efficiency increased. With increasing roasting time from one hour to four hours, the vanadium separation efficiency increased. The optimum conditions for vanadium recycling are the basicity of 1.2, roasting temperature of 1623 K, and roasting time of 4 hours. Under this condition, the separation efficiency of vanadium reaches 71.6%, and the concentrate contains 3.67% V<sub>2</sub>O<sub>5</sub> and 46.9% Fe<sub>2</sub>O<sub>3</sub>. A flow for joint processing of V-bearing steelmaking slag and V-bearing black shale was proposed, which involves vanadium separating, and tailings utilizing.

**Keywords:** Vanadium-bearing steelmaking slag; Vanadium-bearing black shale; Joint concentration; Separation efficiency

### 1. Introduction

Coulsonite and V-bearing black shale are the two most important vanadium ores, with more than 95% of vanadium produced from these two ores. Different methods are used to extract vanadium from these two ores because of the differences in their properties.

For coulsonite, pyrometallurgy is generally used to extract vanadium [1]. In the process of blast furnace smelting, most of the vanadium oxide and iron oxide in the ore are reduced, producing V-bearing hot metal. When the vanadium content in the hot metal is below 0.2%, the hot metal is directly used to produce steel in a converter furnace. Most of the vanadium in the hot metal is transferred to the slag in the process of steelmaking, forming V-bearing steelmaking slag.

Steelmaking slag is typically highly basic. The slag containing vanadium and iron is a valuable resource. However, the high basicity leads to instability, which restricts utilization of the slag, and the vanadium in the slag can cause heavy metal pollution [2–6]. Some metallurgists have attempted to recycle vanadium from high-grade vanadium slag by

traditional methods, such as acid leaching [7–13], alkaline leaching [14–19], and bioleaching [20–22]. However, these methods do not apply to V-bearing steelmaking slag because of the low grade of vanadium.

Black shale containing 0.3–1.2% V<sub>2</sub>O<sub>5</sub> is a low-grade vanadium ore. The total reserves of vanadium are relatively large, and the reserves of vanadium in black shale are the second largest. It has promising prospects for development. Therefore, some researchers performed experimental studies and proposed the following methods for vanadium separation: the direct acid-leaching method [23–33] and roasting–leaching method [34, 35]. However, these methods still have a lot of issues because of the low grade of vanadium, such as large consumption of reagent, severe water pollution, and treatment of the leaching tailings.

From the above discussion, methods to extract valuable metals from the two types of vanadium resources and utilization of the remaining tailings are difficult problems, so it is necessary to investigate new methods.

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Many investigations indicate that black shale is an acidic ore mainly containing silica and alumina, and V-bearing steelmaking slag is a highly basic slag. The two are highly complementary in terms of chemical composition. Thus an assumption of combined treatment of the two at high temperature was proposed in our previous study [36]. If so, the carbon in the black shale would provide heat and maintain a reduction atmosphere, the valuable metals of iron and vanadium would form a V-bearing magnetic phase, and the high basicity of the steelmaking slag would decrease. The V-bearing magnetic phase and silicate phase in the roasted feed could be separated by magnetic separation to give a vanadium concentrate and tailings, which could be used as a vanadium source and to produce building materials, respectively. In the present work, the assumption was used for the first time, so the factors that influence vanadium concentration and separation, such as the shale to slag ratio and roasting temperature, were investigated.

## 2. Experimental

**Table 1.** Experimental conditions

No.	Ratio /mass %		Basicity	Temperature/K	Roasting time/hour
	Shale	Slag			
1	49.5	50.5	0.6	1473	2
2	44.3	55.7	0.7	1473	2
3	39.9	60.1	0.8	1473	2
4	36.0	64.0	0.9	1473	2
5	32.7	67.3	1.0	1473	2
6	27.2	72.8	1.2	1473	2
7	21.0	79.0	1.5	1473	2
8	17.8	82.2	1.7	1473	2
9	49.5	50.5	0.6	1573	2
10	44.3	55.7	0.7	1573	2
11	39.9	60.1	0.8	1573	2
12	36.0	64.0	0.9	1573	2
13	32.7	67.3	1.0	1573	2
14	27.2	72.8	1.2	1573	2
15	21.0	79.0	1.5	1573	2
16	17.8	82.2	1.7	1573	2
17	27.2	72.8	1.2	1523	2
18	27.2	72.8	1.2	1623	2
19	27.2	72.8	1.2	1623	3
20	27.2	72.8	1.2	1623	4
21	27.2	72.8	1.2	1623	1
22	27.2	72.8	1.2	1423	2

V-bearing black shale from Hunan Province, China, and V-bearing steelmaking slag from Maanshan Iron & Steel Company, Ltd. (Maanshan, China) were ground for 2 h in a ball grinder. The obtained shale powder was dried at 383 K for 12 h in a drying oven.

The steelmaking slag to black shale ratio was determined based on the metallurgical basicity principle (R), i.e., the ratio of the CaO content to the SiO<sub>2</sub> content in the sample. The molar ratio of ferric oxide to carbon was fixed at 0.4 by adding graphite. Dried shale powder, slag powder, and graphite powder were mixed for 2 h in the predetermined proportions in a blender mixer. The mixtures in industrial pure iron crucibles were placed in a sealed resistance furnace [36] and heated to the predetermined temperature at a rate of 5 K/min, roasted for a specified time at the temperature, cooled to room temperature at a rate of 5 K/min, and then the samples were removed from the furnace. Argon flow of 100 ml/min was maintained throughout the experiments. The experimental conditions for all of the samples are given in Table 1.

The roasted samples were ground into powder in a ball grinder. The powders were made into pulps by mixing them with tap water at a mass to volume ratio of 1:5. A magnetic tube (tube diameter: 50mm, head gap: 50mm, vibration frequency: 70times/min, magnetic field strength: 0-200KA/m) was used to process the pulps to obtain magnetic concentrates and tailings (magnetic intensity of 100KA/m was adopted). The obtained concentrates and tailings were dried at 383 K for 12 h in a drying oven.

The carbon content in the black shale was determined by loss on ignition. After grinding, the size distributions of the dried black shale powder, V-bearing steelmaking slag powder, and roasted samples were determined by the screening method. The chemical compositions of the dried black shale, V-bearing steelmaking slag, and dried concentrates and tailings were determined with an X-Ray fluorescence meter (ARL Advant'X Intellipower 3600). The phase compositions of the dried black shale, V-bearing steelmaking slag, and roasted samples were determined by X-ray diffractometry (Bruker D8 Advance X-ray diffractometer, Cu K).

Based on the compositions of the concentrates and tailings, the vanadium and iron separation efficiencies were calculated by the Hancock efficiency equation:

$$E = \frac{(\beta - \alpha) \times (\alpha - \theta) (\beta_i - \theta_i)}{(\beta_i - \alpha) (\alpha - \theta_i) (\beta - \theta)} \times 100\% \quad (1)$$

where  $E$  is the vanadium or iron separation efficiency (%),  $\alpha$  is the mass percentage of V<sub>2</sub>O<sub>5</sub> or Fe<sub>2</sub>O<sub>3</sub> in the roasted sample,  $\beta$  is the mass percentage of V<sub>2</sub>O<sub>5</sub> or Fe<sub>2</sub>O<sub>3</sub> in the concentrate (%),  $\theta$  is the mass



percentage of  $V_2O_5$  or  $Fe_2O_3$  in the tailings (%),  $\beta_i$  is the ideal mass percentage of  $V_2O_5$  or  $Fe_2O_3$  in the concentrate (%), and  $\theta_i$  is the ideal mass percentage of  $V_2O_5$  or  $Fe_2O_3$  in the tailings (%). Based on the assumption that all of the vanadium and iron enter the vanadium–iron spinel,  $\beta_i$  was obtained by calculation and  $\theta_i$  was considered to be zero.

### 3. Results

The chemical compositions of the black shale and the steelmaking slag are given in Table 2. The black shale mainly consisted of silica and alumina. The steelmaking slag was composed of calcium oxide, iron oxide, silica, and magnesium oxide.

The XRD patterns of the shale and the slag are shown in Figure 1. The shale mainly consisted of mica and silica. The slag was composed of tricalcium silicate, dicalcium silicate, calcium oxide, RO and dicalcium ferrite. Besides the aforementioned phases, according to our previous studies, the slag contained a supercooled glass. The glass phase formed from molten slag without crystallization, consisting of CaO, SiO<sub>2</sub>, FeOx, and MgO.

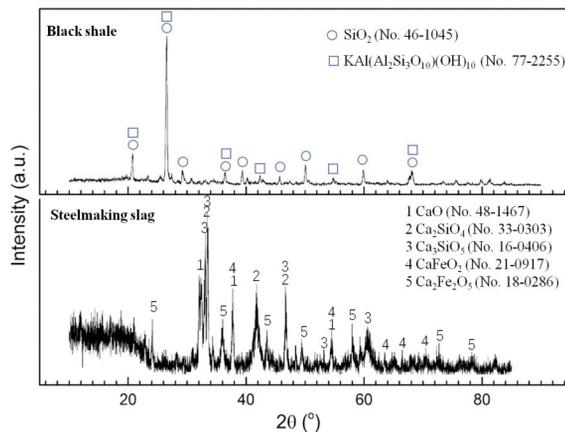


Figure 1. X-ray diffraction patterns of black shale and steelmaking slag

According to the results of X-ray diffractometry for the samples roasted at 1573 K, the pattern of the sample with a basicity of 0.6 is closely to that of the sample with a basicity of 0.7, the patterns of the samples with basicity of 0.8, 0.9, 1.0, 1.2 and 1.5 are similar, and the pattern of the sample with 1.7 is quite different from the others. Thus, the XRD patterns of the samples can be split into three groups. Three representatives from the three groups are shown in Figure 2. As seen in the Figure, the samples with

Table 2. Chemical compositions of the black shale and steelmaking slag /mass %

	SiO <sub>2</sub>	CaO	MgO	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	S	P <sub>2</sub> O <sub>5</sub>	V <sub>2</sub> O <sub>5</sub>	MnO	C	Others
Shale	65.61	6.86	2.45	2.86	7.70	2.27	1.52	0.83	0.98	-	7.59	1.33
Slag	9.06	39.97	10.36	31.14	2.24	-	-	2.33	0.86	2.49	-	1.56

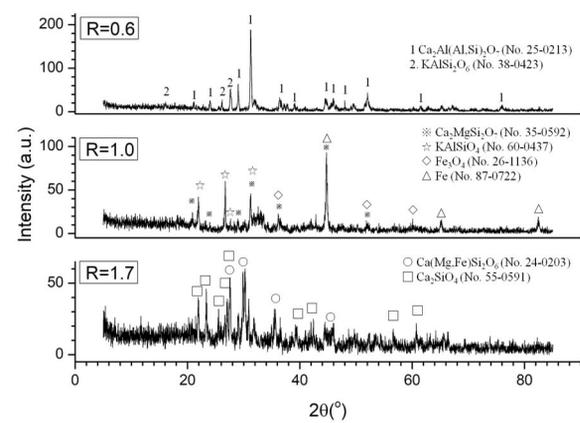


Figure 2. X-ray diffraction patterns of the samples roasted at 1573 K

lower basicities (0.6 and 0.7) mainly consist of aluminosilicate similar to muscovite, which indicates the original structure of mica isn't utterly destroyed, and the vanadium in the lattice of aluminosilicate is not released in full. The sample with a basicity of nearly 1.0 contains Ca<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>, KAlSi<sub>3</sub>O<sub>6</sub>, Fe and Fe<sub>3</sub>O<sub>4</sub> (V-bearing spinel phase). In the sample with a basicity of 1.7, two crystalline phases of dicalcium silicate and composite silicate occur. V-bearing spinel phase wasn't detected in the samples with low basicity and high basicity, which indicates that the formation of V-bearing spinel necessitates a moderate basicity.

The particle size distribution of the shale powder was as follows: the 75–150, 48–75, and <48 μm fractions accounted for 11.6%, 41.8%, and 46.6% of the total mass, respectively. In the steelmaking slag powder obtained by grinding, the 75–150, 48–75, and <48 μm fractions accounted for 26.1%, 18.7%, and 55.2% of the total mass, respectively. Although the roasted samples were grounded for the same time, the particle size distributions were different because of the mineral composition difference. This difference was relatively small, and the 75–150, 48–75, and <48 μm fractions accounted for 30%, 19%, and 51% of the total mass, respectively.

The V<sub>2</sub>O<sub>5</sub> and Fe<sub>2</sub>O<sub>3</sub> contents in the concentrates and tailings, and the calculated separation efficiencies are given in Table 3. The results indicate that the basicity, roasting temperature, and roasting time have large effects on the vanadium and iron separation efficiencies from the roasted samples.

**Table 3.**  $Fe_2O_3$  and  $V_2O_5$  contents and separation efficiencies

No.	Content /wt%				Original grade $\alpha$ /wt %		Ideal grade $\beta_1$ /wt %		Efficiency /%	
	Concentrates $\beta$		Tailings $\theta$							
	$Fe_2O_3$	$V_2O_5$	$Fe_2O_3$	$V_2O_5$	$V_2O_5$	$Fe_2O_3$	$V_2O_5$	$Fe_2O_3$	$V_2O_5$	$Fe_2O_3$
1	36.74	0.82	7.10	0.77	0.79	17.41	4.33	95.67	1.8	47.2
2	35.37	0.70	7.60	0.79	0.73	24.93	2.86	97.14	-3.9	35.2
3	35.37	0.67	11.38	0.81	0.74	23.88	2.99	97.01	-6.3	33.3
4	37.38	0.68	9.64	0.82	0.75	23.15	3.15	96.85	-6.1	39.3
5	35.19	0.67	8.96	0.8	0.72	24.28	2.90	97.10	-5.8	35.0
6	26.67	1.02	11.23	0.77	0.88	17.88	4.68	95.32	8.6	26.1
7	28.54	0.91	11.42	0.82	0.86	19.91	4.16	95.84	3.3	27.1
8	27.11	0.96	15.78	0.86	0.91	21.03	4.13	95.87	3.5	17.2
9	45.06	1.66	1.31	0.38	0.68	11.59	5.55	94.45	38.5	77.3
10	45.45	1.68	1.11	0.35	0.71	13.21	5.12	94.88	43.0	77.4
11	29.50	1.24	3.32	0.44	0.77	14.00	5.19	94.81	29.6	53.0
12	23.62	1.19	1.56	0.34	0.67	10.23	6.18	93.82	33.8	57.7
13	22.36	1.11	1.46	0.32	0.66	10.53	5.92	94.08	33.0	54.9
14	38.11	1.67	2.51	0.28	0.73	13.97	4.95	95.05	48.9	65.2
15	30.57	1.39	4.30	0.52	0.92	16.28	5.33	94.67	28.4	48.3
16	29.87	1.21	5.51	0.62	0.96	19.57	4.68	95.32	18.9	38.2
17	28.68	1.28	8.86	0.60	0.78	14.25	5.22	94.78	20.2	32.4
18	35.96	2.09	3.05	0.31	0.71	10.52	6.36	93.64	49.3	61.8
19	40.82	2.74	1.67	0.13	0.74	10.83	6.40	93.60	71.4	73.3
20	46.90	3.67	1.53	0.17	0.75	9.11	7.65	92.35	71.6	76.9
21	31.30	1.31	6.34	0.32	0.68	15.50	4.22	95.78	40.2	44.6
22	23.42	0.73	17.45	0.83	0.78	20.28	3.72	96.28	-4.0	9.3

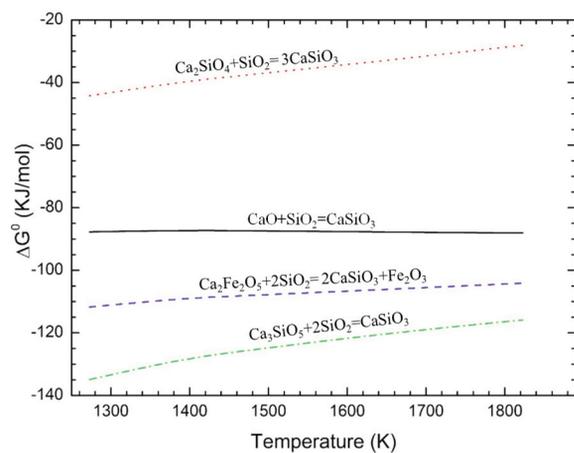
#### 4. Discussion

##### 4.1. Theoretical analysis of V-concentrating phase formation

According to our previous study [36, 37], the vanadium in the shale is mainly located in the mica, and the vanadium in steelmaking slag exists in a glass phase and dicalcium silicate. In the process of roasting, mica in the shale first broke down into silicate and water. The silicate and silica in turn reacted to calcium oxide, dicalcium silicate, tricalcium silicate, a glass phase mainly consisting of calcium ferrite and RO in the slag at higher temperature.

Standard Gibbs free energy for the possible reactions was plotted against temperature in Figure 3. The free energies are much less than zero, which indicates that the reactions may take place at 1273K to 1873K in term of thermodynamics. The reactions made the structures of mica, dicalcium silicate, and

the glass phase decompose. Vanadium oxide in the glass phase and the crystal lattice of mica and dicalcium silicate was released, and iron oxide got liberated from the glass phase and RO phase. In the



**Figure 3.** Variation of standard Gibbs energy for several reactions with temperature



study, the targeted compound of vanadium is V-bearing spinel ( $\text{Fe}_{3-m}\text{V}_m\text{O}_4$ ). The released vanadium oxide and iron oxide need further interaction with each other, forming V-bearing spinel ( $\text{Fe}_{3-m}\text{V}_m\text{O}_4$ ).

Vanadium and iron are the elements with variable valency and their valence is severely affected by oxygen partial pressure in atmosphere. Therefore, controlling oxygen partial pressure in atmosphere within an appropriate range is essential for obtaining the targeted compound. High pure argon gas can ensure the atmosphere [36].

Binary chemical basicity, i.e. the ratio of calcium oxide content to silica content, is another key factor influencing the occurrence state of vanadium. With increasing the basicity, vanadium attempts to form high valence oxides, combining to calcium oxide. Our previous studies demonstrated that vanadium was contained in dicalcium silicate ( $\text{Ca}_2\text{Si}_{1-x}\text{V}_x\text{O}_4$  or  $\text{Ca}_2\text{SiO}_4 \cdot n\text{Ca}_3\text{V}_2\text{O}_8$ ) in the slags with high basicity [36]. Lower basicity improves the transformation of iron element from high valence to low valence, reacting to acid oxides. In the studied system,  $\text{FeSiO}_3$  would be generated in substantial amounts, which restrains the generation of the spinel. Influences from the two aspects would lead to an optimal basicity.

#### 4.2. Effect of the basicity

The effect of the basicity on the vanadium and iron separation efficiencies is shown in Figure 4. For the samples roasted at 1473 K, with increasing basicity, the  $\text{V}_2\text{O}_5$  separation efficiency remained low (even less than 0), i.e., the  $\text{V}_2\text{O}_5$  content in the concentrate was less than that in the tailings. The  $\text{Fe}_2\text{O}_3$  separation efficiency decreased with increasing basicity, and it also maintained at low level. For the samples roasted at 1573 K, with increasing basicity, the  $\text{V}_2\text{O}_5$  separation efficiency first decreased, increased, and then decreased, reaching a maximum when the basicity was 1.2. This is consistent with the theoretical

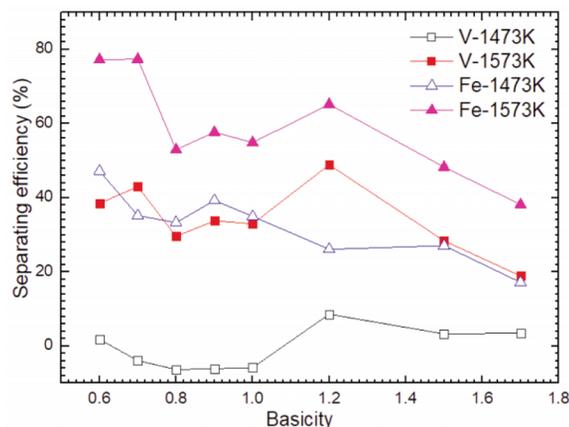


Figure 4. Effect of the basicity on separation efficiencies of vanadium and iron

analysis of V-concentrating phase formation. The variation trend of the  $\text{Fe}_2\text{O}_3$  separation efficiency was similar to that of the  $\text{V}_2\text{O}_5$  separation efficiency.

#### 4.3. Effect of the roasting temperature

The  $\text{Fe}_2\text{O}_3$  and  $\text{V}_2\text{O}_5$  separation efficiencies are plotted against the roasting temperature in Figure 5. With increasing roasting temperature from 1423 to 1623 K, the  $\text{Fe}_2\text{O}_3$  separation efficiency increased and then decreased, reaching a maximum at 1573 K. The  $\text{V}_2\text{O}_5$  separation efficiency greatly increased with increasing roasting temperature from 1423 to 1573 K, and it slightly increased when the roasting temperature increased from 1573 to 1623 K.

The reasons for the above trends are as follows. On the one hand, high temperature favors formation and growth of the vanadium-iron spinel phase, which is favorable for vanadium and iron separation. On the other hand, higher temperature leads to more liquid phase forming, and more vanadium oxide and iron oxide dissolve in the liquid phase, which results in an increase in the iron oxide content in the tailings.

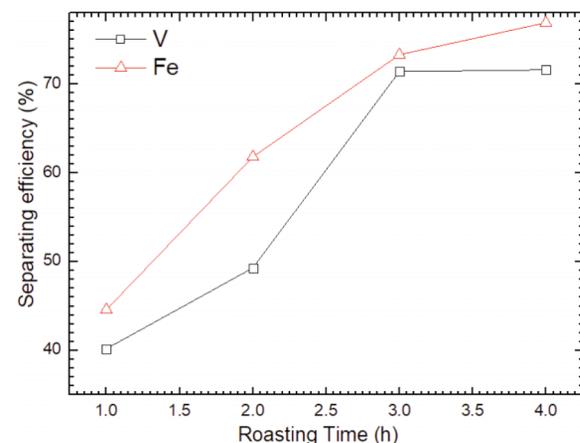


Figure 5. Effect of roasting temperature on separation efficiencies of vanadium and iron

#### 4.4. Effect of the roasting time

The vanadium and iron separation efficiencies are plotted against the roasting time in Figure 6. With increasing roasting time from 1 to 3 h, the vanadium separation efficiency dramatically increased from 40.2% to 71.4%, and slightly increased to 71.6% when roasting time increased from 3 to 4 h. With increasing roasting time from 1 to 4 h, separation efficiency of iron gradually increased from 44.6% to 76.9%.

Generally, a long roasting time promotes crystal grain growth. Large crystal grains are conducive to separation, resulting in high separation efficiency. The experimental results are consistent with this principle.

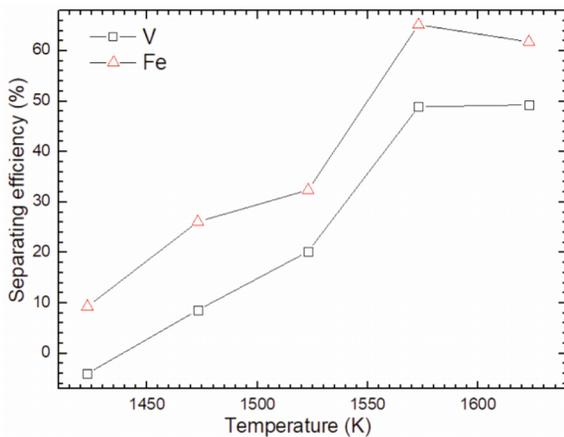


Figure 6. Effect of roasting time on separation efficiencies of vanadium and iron

#### 4.5. Processing flow

Joint processing of V-bearing black shale and V-bearing steelmaking slag can be performed in two steps. In the first step, the mixture of the two is roasted. In the second step, after grinding, vanadium and iron are separated from the roasted material by magnetic separation, producing a concentrate and tailings.

The obtained concentrate mainly consisting of iron and vanadium can be used to produce  $V_2O_5$  or vanadium-iron alloy. The tailings were mainly composed of calcium aluminosilicate and had high stability because of an appropriate basicity. Furthermore, the remaining vanadium in the tailings was present in the trivalent state in the form of spinel, which is relatively stable and non-toxic. Therefore, it can be used as building materials.

#### 5. Conclusions

Based on the investigation of joint roasting of V-bearing steelmaking slag and V-bearing black shale followed by magnetic separation, the effects of the roasting conditions on the vanadium and iron separation efficiencies can be summarized as follows.

1. With increasing basicity, in the samples roasted at 1573 K, the vanadium and iron separation efficiencies first decreased, increased, and then decreased, reaching a maximum when the basicity was 1.2. The vanadium and iron contents in the obtained concentrates showed a similar variation trend to the separation efficiencies.

2. Increasing the roasting temperature greatly affected the vanadium and iron separation efficiencies. The vanadium separation efficiency increased with increasing roasting temperature from 1423 to 1623 K. The iron separation efficiency increased with increasing roasting temperature from

1423 to 1573 K, and slightly decreased when the roasting temperature was increased from 1573 to 1623 K.

3. The roasting time had a significant effect on the vanadium and iron separation efficiencies. The vanadium separation efficiency increased from 40.2% to 71.4% when the roasting time was increased from 1 to 3 h, and it slightly increased from 71.4% to 71.6% when the roasting time was increased from 3 to 4 h. The iron separation efficiency increased from 44.6% to 76.9% when the roasting time was increased from 1 to 4 h.

4. Comprehensive evaluation of the vanadium and iron separation efficiencies and their contents in the concentrate showed that appropriate conditions are basicity of 1.2, roasting temperature of 1623 K, and roasting time of 4 h.

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## ZAJEDNIČKA PRERADA ČELIČNE ŠLJAKE I CRNOG ŠKRILJCA SA ZNAČAJNIM SADRŽAJEM VANADIJUMA SA CILJEM IZDVAJANJA VANADIJUMA I GVOŽĐA

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### Apstrakt

Da bi se reciklirao vanadijum iz čelične šljake i crnog škriljca sa značajnim sadržajem vanadijuma, u ovom istraživanju oba su zajedno pržena da bi se dobila faza bogata vanadijumom, a onda je vanadijum izdvojen uz pomoć magnetne separacije. Sastav uzoraka je određen rentgenskim fluorescentnim analizatorom a faze u uzorcima su okarakterisane uz pomoć rentgenskog difraktometra. Rezultati eksperimenta su pokazali da je sa povećanjem CaO sadržaja u odnosu na sadržaj SiO<sub>2</sub> u uzorcima efikasnost izdvajanja vanadijuma najpre padala, zatim rasla, i ponovo padala. Sa porastom temperature prženja sa 1423 na 1623 K, efikasnost izdvajanja vanadijuma je rasla. Sa porastom dužine prženja od jednog na četiri sata efikasnost izdvajanja vanadijuma je rasla. Optimalni uslovi za recikliranje vanadijuma su baznost od 1.2, temperatura prženja od 1623 K i vreme prženja od 4 sata. Pod ovim uslovima efikasnost izdvajanja vanadijuma dostiže 71.6%, a koncentrat sadrži 3.67% V<sub>2</sub>O<sub>5</sub> i 46.9% Fe<sub>2</sub>O<sub>3</sub>. Predložen je postupak zajedničke prerade čelične šljake i crnog škriljca sa značajnim sadržajem vanadijuma koji uključuje odvajanje vanadijuma i iskorišćenje jalovine.

**Ključne reči:** Čelična šljaka sa značajnim sadržajem vanadijuma; Crni škriljac sa značajnim sadržajem vanadijuma; Zajednička koncentracija; Efikasnost izdvajanja

