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Technology for producing ferroalloy from vanadium-bearing shale leaching residue

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Abstract: Black shale vanadium-bearing quartzites are predominantly processed by sulfuric acid leaching with the recovery of vanadium, molybdenum, uranium, and rare earth elements, resulting in the formation of vanadium-bearing shale leaching residue. Currently, cake of leaching vanadium black shale quartzite is mainly utilized in the construction industry for the production of building materials with low added value. This study investigates the production of ferrosilicon from a mixture of autoclave leaching residue of vanadium-bearing black shale quartzites and long-flame coal beneficiation tailings. The experiments were conducted using a second-order rotatable experimental design combined with electric smelting in an electric arc furnace to determine the optimal technological parameters. The results demonstrate that ferrosilicon grades FeSi45 and FeSi50 can be obtained from leaching residue and coal beneficiation tailings depending on their ratio and the proportion of steel shavings added to the charge. The optimal parameters for producing FeSi45 with a silicon recovery of 75.0-80.1% are a tailings-to-leaching residue ratio of 1.42:1-1.7:1 and 16.7-23.05% steel shavings. For FeSi50 with a silicon recovery of 75.0-79.4%, the optimal ratio is γ=1.44:1-1.7:1 with 14.3-17.8% steel shavings. Pilot-scale laboratory electric smelting of a charge containing 51.3% coal beneficiation waste, 30% leaching residue, and 18.7% steel shavings confirmed the feasibility of producing FeSi45 with a silicon content of 41.1-43.4% and a silicon recovery of 79% in the alloy.

Keywords: vanadium-bearing shale leaching residue, coal beneficiation tailings, Ferrosilicon, optimal technological parameters, electric smelting

1. Introduction

Vanadium is a strategic metal with growing demand in metallurgical and chemical industries. Around 18% of its production comes from primary ores, while up to 82% is obtained from secondary resources such as slags, petroleum residues, and spent catalysts (Gao et al., 2022). The conventional extraction technology is based on roasting followed by leaching, purification, and precipitation (Kunayev, 1971; Chepushtanova et al., 2024). Sodium salts (Na_2CO_3, Na_2SO_4, NaCl) are commonly used during roasting (Wang et al., 2008; Ye et al., 2012; Zhao et al., 2017; Wen et al., 2019), but due to toxic emissions and corrosive conditions, calcium-based additives are increasingly applied (Zhang et al., 1993; Wen, 2009; Li et al., 2016). Hydrometallurgical methods, particularly sulfuric acid and alkaline leaching, remain the most economically viable, although processes such as heap leaching show relatively low efficiency (Aimbetova, 2010; Oslopovsky et al., 2023). To improve extraction, various approaches have been studied, including the use of oxidizing agents, electrochemical and bioleaching methods, ion-exchange resins, and intensified leaching techniques (Lee et al., 2005; Zeng et al., 2009; Gao et al, 2018; Chen et al., 2020; Chen et al., 2021; Wang et al., 2022 a; Lu et al., 2024; Li et al., 2025; Tan et al., 2026).

A significant raw material potential for the production of vanadium and associated elements is represented by the black shale quartzites of the Greater Karatau basin, where large deposits are concentrated, including Balasauskandyk (>100 Mt (Feasibility Study, 1992; Ferro-alloy.com, 2025)), Kurumsak (133.5 Mt (Feasibility Study, 1992)), Zhabagly (102.6 Mt), and others (Kazakhstan National

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Encyclopedia, 2005). The average V_2O_5 content in the ores ranges from 0.5 to 1.3%, while associated components include 0.02-0.03% U, 0.03-0.04% Mo, 0.08-0.15% rare earth elements (REEs), 60-80% SiO_2, and 5-20% carbon (Yulusov, 2022). The black shale quartzites of the Greater Karatau have already been utilized as raw materials in several metallurgical processes. Vanadium-bearing quartzites containing 75-80% SiO_2 have been recommended as fluxing additives in phosphorus electric smelting. When smelted together with yellow phosphorus, an iron-phosphorus-vanadium alloy containing 4-5% V is formed. This alloy is subsequently subjected to pyrometallurgical-hydrometallurgical processing to obtain vanadium-enriched slag and ferrophosphorus (Oslopovsky et al., 2023; Sukharnikov et al., 2023).

During the agglomeration of fine phosphorite, the addition of up to 6% quartzite increases the mechanical strength of the sinter from 73.4% to 74.4%, enhances phosphorus productivity by 25-27%, reduces specific electricity consumption by 1.1%, and ensures vanadium recovery of 89.5-95.1% into the phosphorus-containing alloy (Alybaev, 1999).

In the production of high-carbon ferrochrome, the introduction of quartzites lowers the melting temperature of the charge by approximately 150 °C, reduces chromium losses to slag, and increases alloy yield by 0.5-0.9%, producing an alloy containing 0.1-0.2% vanadium (Davletov et al., 1995; Sukharnikov et al., 1995; Jamankulova et al., 2018a; Jamankulova et al., 2018b). The use of vanadium-bearing quartzites in slag-free smelting of ferrosilicochrome has enabled the production of complex Fe-Si-Cr-V master alloys containing 5.0-59.5% Cr, 22.0-29.0% Si, 0.12-0.23% V, and 0.17-7.7% C (Alybaev et al., 2011a; Alybaev et al., 2011b).

Replacement of conventional quartzite with black shale ore has enabled the production of high-silicon ferrosilicon containing 65.0-69.5% Si, with silicon recovery reaching 98.5-99.3%. At the same time, the process temperature decreases by 100-200 °C, specific electricity consumption is reduced by 8.8-9.8 kWh/kg, and $SiO_{(g)}$ losses decrease by 0.7-1.5% (Kozlov et al., 2018a; Kozlov et al., 2018b). Despite these advantages, the technology requires multistage raw material preparation and strict temperature control to prevent the formation of viscous silicates and silicon carbide.

Modern hydrometallurgical and combined processing technologies are being actively developed to enhance the recovery of valuable components from Kazakhstan's vanadium-bearing black shales. Roasting with ammonium bisulfate at 300-350 °C followed by sulfuric acid leaching at a solid-to-liquid ratio of 1:3 and 80 °C enables recovery of >98.0% U, 87.2% V, ≈80.3% rare earth elements, and 76.4% Mo, while producing commercial-grade potassium-aluminum or ammonium-aluminum alums (Kozlov et al., 2018c). A lower-temperature variant – roasting with ammonium bisulfate at 150-200 °C followed by sulfuric acid leaching at 80 °C, S:L=1:3 for 2 h – ensures extraction into solution of 81.7% V, 93.3% U, 82.2% Mo, and 78.3% REEs. The resulting solid residue, after washing, is directed to carbon enrichment, while the remaining material is recommended for use in ferrosilicon production (Baigenzhenov et al., 2019).

A hydro-electrometallurgical technology for processing vanadium-bearing black shale ores has also been developed (Jamankulova et al., 2018a; Dzhumankulova et al., 2020). Electrochemical oxidation in the anodic compartment of an electrolyzer in a sulfuric acid medium at a current density of 200 A/m^2, 65 °C, and a solid-to-liquid ratio of 1:4 enables the extraction of 59-70.1% V from non-roasted ore. Following preliminary roasting, vanadium recovery increases to 90.9-92.1%. The resulting solid residue is likewise proposed for utilization in ferroalloy production charge materials.

A comprehensive atmospheric-autoclave sulfuric acid process combined with sorption separation and the production of V_2O_5 (Grade 1 Vanadium Oxide) ensures recovery of 92-94% vanadium, 97-99% uranium, 68-80% molybdenum, and 75% rare earth elements. From the washed residue, flotation enables the recovery of 66% carbon with a carbon content of 35.1% in the concentrate (Komekova et al., 2016; Komekova et al., 2017; Seidakhmetova, 2020).

A technology has been proposed for the simultaneous recovery of vanadium, as well as the production of silicon carbide and sulfur from carbon-containing vanadium-bearing black shale (Miao and Yan, 2024). This approach is also applicable to vanadium-bearing shale leaching residue generated after sulfuric acid leaching, since both raw materials contain significant amounts of silicon and carbon. The shale, with the addition of 5% iron powder, is subjected to reductive roasting at 1400 °C, during which vanadium concentrates in the magnetic ferrosilicon phase. Simultaneously, SiC is formed according to the reaction $SiO_2+3C=SiC+2CO$, while sulfur transitions to the gas phase. Subsequent magnetic separation enables the recovery of vanadium-containing ferrosilicon from silicon carbide. The vanadium recovery into ferrosilicon containing 3.62% V reaches 80.16%.

The above analysis indicates that most existing technologies for processing vanadium-bearing black shales are primarily focused on the extraction of vanadium, uranium, and molybdenum, while being accompanied by the formation of substantial volumes of solid residues. As a result, the level of integrated utilization remains relatively low. Enhancing the degree of comprehensive utilization in the processing of black shale quartzites represents a global challenge and is relevant to all countries employing hydrometallurgical methods for vanadium recovery. For example, in China alone, vanadium mining and processing enterprises generate approximately 300,000 tons of leaching residue annually (with residue generation reaching 120-150 tons per 1 ton of vanadium produced), most of which is disposed of in tailings storage facilities (Yang et al., 2014; Meng et al., 2018). In addition, the large-scale accumulation of such waste materials imposes significant environmental burdens on mining regions. Therefore, the development of efficient approaches for converting leaching residues into commercially valuable products is of considerable technological, environmental, and economic importance.

Leaching residue derived from vanadium quartzites contains substantial amounts of SiO_2 and carbon, which makes it a potentially valuable secondary raw material for the production of competitive commercial products. In particular, VSLR (vanadium-bearing shale leaching residue) may be utilized in the manufacture of construction bricks, expanded clay aggregates, geopolymers, and other building materials, as well as for the production of white carbon black and thermal insulation materials.

For example, study (Liu et al., 2021) reports the use of VSLR from vanadium-bearing black shales as a raw material for manufacturing construction bricks by pressing a mixture containing up to 80% residue, followed by curing for up to 28 days. The resulting products exhibit porosity up to 15% and compressive strength reaching 32 MPa. Autoclaved bricks are produced by pressing followed by steam curing. A mixture composed of 60% residue, 25% lime, and 15% sand is subjected to autoclave treatment for 3 hours under saturated steam at a pressure of 1 MPa. Fired bricks are manufactured with the addition of corrective additives. Such bricks are characterized by compressive strength exceeding 15 MPa and water absorption of approximately 15%.

The utilization of vanadium-bearing shale leaching residue as a raw material for expanded clay production enables a reduction in firing energy consumption due to the presence of carbon in the shale-derived waste. In study (Wang et al., 2022 b), vanadium slag (wt.%: 76.7 SiO_2, 1.12 Al_2O_3, 1.16 Fe_2O_3, 4.18 CaO, 0.28 MgO, 0.46 K_2O, 0.19 Na_2O, and 6.18 loss on ignition (LOI)) was used as the main component. It was mixed with clay and fly ash in a ratio of 6:1:3. Granulation was carried out for 15 min at a charge moisture content of 18%. The granules were preheated at 400 °C for 30 min, followed by firing at 1160 °C for 20 min. The resulting expanded clay aggregate exhibited a compressive strength of 11.58 MPa, bulk density of 1014.7 kg/m^3, and water absorption of 5.61%, meeting the requirements for artificial lightweight aggregate grade 1100. The material can be used as an aggregate in construction concretes and in the production of lightweight expanded clay concrete.

VSLR in combination with red mud can also serve as a raw material for the production of lightweight aggregates without the use of clay. Mixtures of leaching residue and red mud are formulated based on the content of fluxing oxides, granulated, and sintered at 1080 °C, at which temperature granule bloating occurs due to gas evolution. The resulting lightweight aggregate is characterized by water absorption of 1.46% after 1 h, bulk density of 728.76 kg/m^3, and compressive strength up to 10.77 MPa (Song et al., 2021).

Based on residues from the processing of black shales, raw material formulations have been proposed for the production of thermal insulation materials. A mixture for lightweight porous aggregate has been developed containing 55.9% black shale residue, 9-10% electrostatic precipitator dust from aluminum production, and 34.1-35.1% liquid sodium silicate (modulus 4, ρ=1.44 g/cm^3) as a binder (Glebov et al., 2001a). For the production of granular thermal insulation aggregate, the authors of (Glebov et al., 2001 b) propose a mixture composed of 55.2-56.2% black shale residue (59.06% SiO_2, 16.55% Al_2O_3, 4.61% FeO, 1.26% C), 6.7-7.7% microsilica, and 37.1% liquid sodium silicate as a binder. Thermal treatment is carried out in two stages: rapid heating to 400 °C (133 °C/min) with a holding time of 30 min, followed by slow heating to 1175 °C (5 °C/min) with a 10 min holding period. The resulting material exhibits a bulk density of 143-151 kg/m^3. Due to the presence of carbon, black shale processing residues form a porous structure during thermal treatment, thereby reducing aggregate density. However, both the residues and electrostatic precipitator dust may contain hazardous impurities as well as valuable components that should preferably be recovered prior to their incorporation into construction mixtures.

Another promising application of leaching residue is the production of geopolymer-based construction materials – alkali-activated aluminosilicates capable of partially replacing Portland cement and characterized by high fire resistance due to their amorphous structure (Jiao et al., 2013; Bao et al., 2022). The leaching residue is ground, mixed with fly ash, and activated with liquid sodium silicate to obtain a heat-resistant, refractory geopolymer material (Meng et al., 2018). After thermal treatment, the samples retain their structure and strength, which may even increase after exposure to temperatures exceeding 900 °C without visible structural damage. A technology for the production of geopolymer binders from vanadium leaching residue has also been proposed (Lv et al., 2025). The process involves thermomechanical activation of a mixture of residue and Na_2CO_3 (17:6) at 450 °C, followed by grinding and subsequent mixing with manganese production slag (7:3) and 20% blast furnace slag. The compressive strength of the resulting material reaches 23.8 MPa and increases to 35.1 MPa after autoclave curing. The advantages of this approach include the utilization of leaching residue in the production of commercially demanded construction binders without the use of liquid caustic activators, as well as the incorporation of additional metallurgical wastes. However, the technology is associated with increased energy consumption for thermal activation and requires strict control of curing conditions.

Vanadium-bearing shale leaching residue is also considered as a technogenic raw material for the production of one-part geopolymers – dry mixtures consisting of aluminosilicate precursors and a solid alkaline activator, in which the geopolymerization reaction is initiated by the addition of water without the use of liquid caustic solutions (Luo et al., 2019). The leaching residue (83.47% SiO_2, 5.09% Al_2O_3, 3.20% Na_2O, 2.40% Fe_2O_3, 0.95% CaO, 0.23% V_2O_5) is blended with metakaolin (≈55.53% SiO_2, 40.09% Al_2O_3), obtained by calcining kaolin at 750 °C for 3 h, and 10% NaOH. The resulting paste is molded into specimens, sealed, and cured for 24 h. The optimal compressive strength is observed at a residue content of 65% in the mixture, reaching 23.9 MPa after 7 days and increasing to 31.55 MPa after 28 days.

Vanadium-bearing residues have also been investigated as mineral additives in cement production. Their incorporation allows a reduction in clinker firing temperature by approximately 50 °C, leading to noticeable decreases in energy consumption and production costs. Residual V_2O_5 exhibits a retarding effect, increasing the cement setting time (Yao et al., 2016).

The feasibility of utilizing vanadium leaching residues as secondary raw materials for the production of white carbon black – hydrated amorphous silica containing up to 90% SiO_2 – has also been demonstrated (Hu et al., 2020). White carbon black is widely used as a reinforcing additive and filler in the manufacture of rubber, polymers, paper, and various coatings. SiO_2 is extracted from black shale residues by alkaline leaching under the following conditions: residue-to-NaOH ratio of 1:1.15, temperature 98 °C, leaching time 4 h, and liquid-to-solid ratio (L:S) of 8:1. Subsequent precipitation of silica from the solution with sulfuric acid at pH 4-7 ensures more than 99% recovery into a product containing at least 95.5% SiO_2 (Kang et al., 2023; Kang et al., 2024).

A comprehensive processing technology for vanadium-bearing black shales has been developed based on stepwise hydrochloric acid leaching with the addition of NaF. Following the extraction of calcium, vanadium, and zinc, a solid residue is formed which, after calcination, contains up to 95% SiO_2. This product is considered a potential raw material for the production of high-purity silica (Liu et al., 2023).

In addition to applications in construction materials, another promising direction involves the recovery of valuable metals from black shale leaching waste. The feasibility of metal extraction from residues of a shale deposit in Hubei Province (China) has been investigated. Bioleaching using Ferriobacillus sulphuris at pH 2.0 with the addition of pyrite (15 g/l) enables the extraction of up to 83.4% vanadium and 87.8% copper within 10 days, which is 18-32% more efficient than conventional acid leaching (Wen, 2009). However, despite the high technological efficiency demonstrated, studies on metal recovery from vanadium leaching residues remain largely limited to laboratory-scale investigations.

Several studies have investigated iron recovery from leaching residues by direct reduction followed by magnetic separation. At a reduction temperature of 1200 °C and a holding time of 30 min, the degree of iron metallization reaches approximately 85% (Yang et al., 2013). Roasting followed by magnetic separation enables the production of an iron-bearing concentrate containing more than 90% Fe with an iron recovery exceeding 80%. Another approach is based on reduction with the addition of lime and lignite as a reducing agent. At a mass ratio of leaching residue : lime : lignite = 100 : 30 : 10, reduction at 1200 °C for 60 min followed by magnetic separation yields a concentrate containing 89.7-90.3% iron with an iron recovery of 83.88% (Du et al., 2024).

Studies have also been conducted on carbon recovery from the autoclave leaching residue of vanadium quartzites (Balasauskandyk deposit) (Efremov et al., 2024). According to the proposed technology, the residue (wt.%: 9.2 Fe_2O_3, 2.62 Al_2O_3, 75.12 SiO_2, 0.44 TiO_2, 0.53 V_2O_5, 14.19 LOI) is subjected to grinding in a ball mill followed by froth flotation (pulp density 2.8-3.3 g/cm^3, solid concentration 300-350 g/L). It was established that the optimal flotation reagents are a combination of kerosene and Flotol B, which enables the production of a carbon concentrate containing 39.5-40.0% carbon with a carbon recovery of 73% (concentrate yield 35%). The obtained product can serve as an alternative to commercial amorphous carbon for the production of sorbents, composites, and elastomers. The flotation tailings, containing 10.73% Fe_2O_3, 2.0% Al_2O_3, and 82.8% SiO_2, may be utilized as secondary raw materials in the production of construction materials.

The above review demonstrates that vanadium-bearing shale leaching residue is predominantly utilized in the construction industry. To a lesser extent, technologies have been developed for the selective recovery of silicon, carbon, and iron from this material. However, considering that vanadium-bearing black shale leaching cake contains substantial amounts of 65-80% SiO_2 and 15-20%carbon, its potential as a metallurgical raw material remains insufficiently explored. In our previous studies, the feasibility of producing FeSi45 ferrosilicon by replacing conventional quartzite with VSLR in the charge composition was demonstrated (Shevko et al., 2022; Badikova et al., 2025a; Badikova et al., 2025b). Therefore, the aim of this work is to develop an approach for the utilization of vanadium-bearing leaching cake in metallurgical processes to obtain commercially competitive products, ensuring minimal losses of valuable components and enhancing the degree of integrated raw material utilization. In particular, the study focuses on the smelting of ferroalloys from quartzite leaching residues with replacement of expensive coke by coal beneficiation tailings, containing 6.3–50% SiO_2 and 30–60% carbon. In contrast to previous studies (Badikova et al., 2025a; Badikova et al., 2025b), where the main focus was on the thermodynamic justification of the process for producing ferroalloys from a cake of quartzite leaching and coal beneficiation tailings, this article presents an experimental optimization of the process parameters for the electrosmelting. A distinctive feature of this work is the use of coal beneficiation tailings as a carbon-containing reducing agent instead of coke

2. Materials and methods

To determine the optimal technological parameters of electric smelting, a second-order rotatable experimental design (Box-Hunter design) was employed (Akhnazarova and Kafarov, 1985; Inkov et al., 2003; Ochkov, 2007; Shklyar, 2010; Grinfeld and Moiseev, 2014; Glukhikh et al., 2023; Liu et al., 2025). This approach enables the development of a mathematical model that accounts for the interaction between variables while minimizing the number of experimental runs and maintaining high statistical reliability. For a two-factor experiment, the total number of experimental runs (N) was determined using a formula that considers the number of independent variables and ensures data reproducibility:

$$N=2^K + 2\cdot k + n_0 , \quad (1)$$

where 2^K - Factorial Points, $2\cdot k$ - Axial Points and n_0 - Center Points.

The axial distance (star arm) α is determined from the expression: $\pm\alpha=2^{k/4}$. For a two-factor experiment (k=2), the axial distance is: $\pm\alpha=2^{2/4}=1.414$. Accordingly, the total number of experimental runs in the planned series is:

$$N=2^2+2*2+5=13. \quad (2)$$

The statistical significance of the regression coefficients was evaluated using Student's t-test. At a confidence level of P=0.95, the tabulated value of the t-criterion (t_{tab}) is 2.78 (SJSU Faculty, 2025). A regression coefficient was considered statistically significant if the condition $t_{calc} > t_{tab}$ was satisfied. The adequacy of the developed mathematical model with respect to the experimental results was verified using the Fisher F-test. For a confidence probability of α ≥ 95 %, the tabulated value of the Fisher criterion (F_{tab}) according to (SJSU Faculty, 2025) is 6.59. When $F_{calc} < F_{tab}$, the regression equation was considered adequate to describe the experimental data.

In all experiments, the total mass of the mixture consisting of coal beneficiation tailings (CBT) and vanadium-bearing black shale leaching residue (VSLR) was kept constant, while the mass of steel shavings was varied. The independent variables were the mass ratio of CBT to VSLR (γ) and the amount of steel shavings (St., wt.% relative to the total mass of tailings and residue). The optimization parameters (response variables) were the silicon recovery into the alloy (αSi(alloy), %) and the silicon concentration in the alloy (C Si(alloy), %). Based on the obtained regression equations, three-dimensional and contour (two-dimensional) response surface models were constructed to illustrate the effect of the independent variables on the optimization parameters. Geometric optimization of the process parameters was subsequently performed using the generated contour plots.

The chemical compositions of the raw materials used in this study were as follows:

- Vanadium-bearing shale leaching residue (VSLR) obtained after autoclave sulfuric acid leaching of vanadium-bearing quartzites from the Balasauskandyk deposit (wt.%): 72.5 SiO_2, 1.3 Al_2O_3, 1.4 Fe_2O_3, 1.0 CaO, 0.15 V_2O_5, 0.2 MgO, 3.0 H_2O, 0.15 BaO, 19.0 C.
- Tailings from dry (gravity) beneficiation of low-ash long-flame coal from the Shubarkol deposit (wt.%): 39.8 SiO_2, 7.32 Al_2O_3, 1.42 Fe_2O_3, 0.44 CaO, 0.5 MgO, 1.8 S, 48.72 C.
- Steel shavings (wt.%): 98.0 Fe, 0.9 C, 0.4 Si, 0.3 other elements.

Prior to smelting, the coal beneficiation tailings were crushed using a jaw crusher (A092, Matest, Italy) and screened on a sieve shaker A20 based on the VP30 T system (Vibrotechnik LLC, Russia). The tailings fraction of 1.0-1.5 cm was used for charge preparation. The leaching residue (VSLR), ground to a homogeneous particle size, was mixed with a 50% molasses solution as a binder and pelletized to obtain granules 1.0-1.5 cm in diameter. The wet pellets were subsequently dried in a laboratory drying oven (Binder, Germany) at 120 °C for 1.5 h. The compressive strength of the dried pellets ranged from 17.0 to 22.2 kg per pellet, while the impact strength exceeded 100 drops from a height of 2 m. Steel shavings was cut into spiral fragments measuring 3-5 mm.

Electric smelting of the vanadium-bearing shale leaching residue and coal beneficiation tailings was carried out using the laboratory setup shown in Fig. 1.

a) b) c)

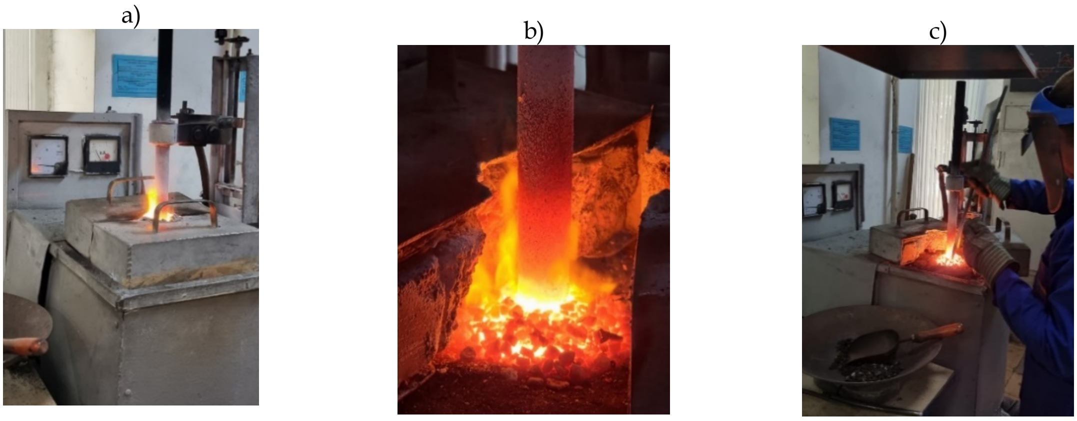


Fig. 1. Stages of electric smelting: furnace ignition (a); charge smelting (b); sealing of the furnace top (burden surface compaction) (c)

The main component of the experimental setup was a submerged arc electric furnace with adjustable power output. Power supply to the furnace was provided by a TDZhF-1002 transformer equipped with a thyristor control unit to enable smooth voltage regulation. The furnace lining was made of chrome-magnesite refractory bricks. Smelting was carried out in a graphite crucible installed on a graphite hearth, with an internal diameter of 8 cm and a height of 16 cm. The space between the graphite crucible and the chrome-magnesite lining was filled with graphite granules (0.1-1 mm). The diameter of the graphitized electrode was 3 cm. Vertical movement of the electrode was controlled by a dedicated mechanical positioning system. The furnace cover was fabricated from chamotte refractory material reinforced with iron. Electrical parameters were monitored using a TENGEN 42L6 ammeter and a CHNT 4226 voltmeter.

Prior to charge smelting, the furnace was preheated for 15 min by maintaining an electric arc between the electrode and the bottom of the crucible. The charge was introduced in portions into the preheated crucible and sequentially melted. After completion of smelting, the electrode was raised, the crucible was removed from the furnace, allowed to cool, and mechanically broken. The produced ferroalloy was separated from the slag.

The optimal technological parameters of electric smelting determined during the first stage of experiments were subsequently used for enlarged laboratory-scale tests involving tapping of the molten metal from the furnace. Electric smelting of the charge was carried out using a laboratory installation whose main component was a single-phase arc electric furnace equipped with one electrode. The electrode position was controlled by a screw-type mechanism that ensured vertical movement and stable positioning during operation.

Electrical power was supplied to the furnace from a TDZhF-1002 transformer equipped with a thyristor power regulator. The maximum transformer capacity was 56 kVA. The internal volume of the furnace bath was 23,750 cm3 (25x 25x 38 cm). Electrical parameters were monitored using ammeters and voltmeters (TENGEN 42L6, GB/T7676-1998, and CHNT 4226, China), installed on the transformer panel and the furnace control panel. The temperature beneath the furnace roof was measured using a TPP-0679 886 thermocouple, with data acquisition performed by a METAKON controller equipped with an RS-485 interface. Prior to charge loading, the furnace was preheated for 5 h. Preheating was performed by maintaining an electric arc between the electrode and the hearth, onto which coke was fed. During preheating, the current ranged from 500 to 750 A, and the voltage was maintained at 30-50 V. After completion of the preheating stage, the charge was introduced into the furnace bath and smelted for 2 h. The smelting process was conducted at a current of 700-900 A and a voltage of 20-35 V. Upon completion of smelting, the molten alloy and slag were tapped through the taphole into cast-iron molds (25x 8 x10 cm). Prior to tapping, the taphole was cleaned with a steel bar and opened using a thermal lancing device. After tapping, the molds were transferred to a cooling area, where they were kept for 5-6 h. After solidification, the contents of the molds were separated into two fractions: ferroalloy and slag.

The silicon recovery into the alloy was calculated as the ratio of the mass of silicon in the produced ferroalloy to its total amount in the initial charge, according to the following equation:

$$\alpha_{\mathrm{Me}} = \frac{\mathrm{M}_{alloy} \cdot \mathrm{C}_{\mathrm{Me}(alloy)}}{\Sigma G_{\mathrm{Me}(charge)}} \cdot 100; \quad (3)$$

where M_{alloy} is the mass of the produced alloy (g); $\mathrm{C}_{\mathrm{Me}(alloy)}$ is the content of the metal in the alloy (wt.%); $\Sigma G_{\mathrm{Me}(charge)}$ is the total mass of the metal in the charge (g).

The chemical composition of the raw materials and smelting products was determined using atomic absorption spectroscopy (AAS-1, Germany), scanning electron microscopy (SEM, JSM-6490LV equipped with an INCA Energy energy-dispersive X-ray microanalysis system, Japan), and X-ray fluorescence analysis (XRF; Thermo Scientific Niton XL3t XRF GOLDD+, USA).

In addition, the silicon content in the alloy was also determined by a pycnometric method based on alloy density (ρ, g/cm^3), using the following empirical relationship (Shevko et al., 2021; Shevko et al., 2025):

$$C_{\mathrm{Si(alloy)}} = 252.405-101.848\rho+18.209\rho^{2}-1.243\rho^{3} \quad (4)$$

Fragments of the electric smelting process carried out on the enlarged laboratory-scale installation are presented in Fig. 2.

3. Results and discussion

At the first stage of the study, exploratory electric smelting experiments were carried out to evaluate the influence of the mass ratio of coal beneficiation tailings to vanadium-bearing shale leaching residue (γ, CBT/VSLR) and the amount of steel shavings (St., wt.% relative to the total mixture mass). Fig. 3

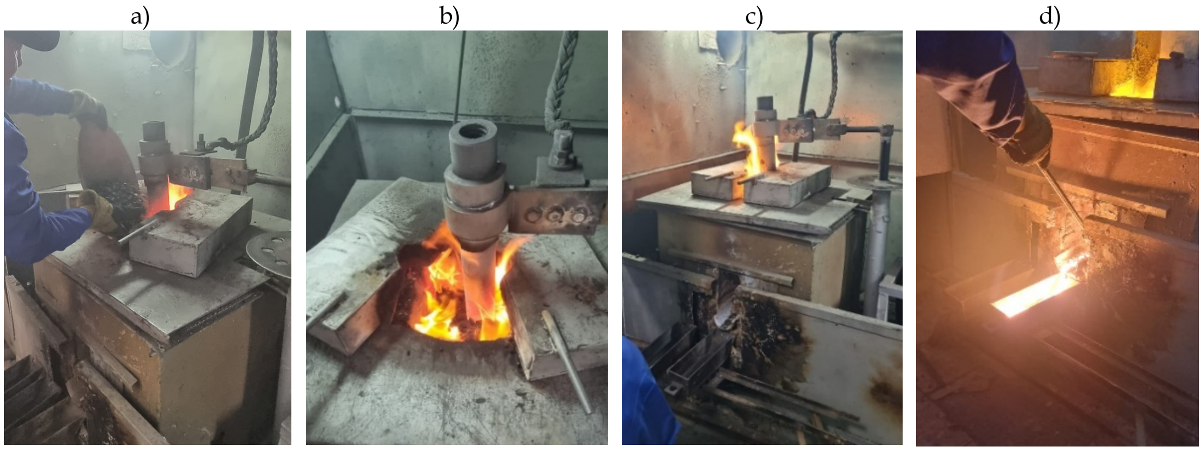


Fig. 2. Fragments of the enlarged laboratory-scale smelting process: (a) furnace charging with the prepared burden; (b) furnace top (burden surface); (c) electric smelting; (d) tapping of slag and molten alloy

illustrates the effect of γ and steel shavings content on silicon recovery into the alloy and silicon concentration in the alloy. The dependences of silicon recovery (αSi(alloy)) and silicon concentration (CSi(alloy)) on steel shavings content at γ=1:1 is described by the following regression equations:

$$\alpha Si(alloy) = 0.0204St^2 - 0.6511St + 73.944; \quad (R^2 = 1); \quad (5)$$

$$CSi(alloy) = 0.0676St^2 - 3.8489St + 89.956. \quad (R^2 = 1). \quad (6)$$

The dependences of αSi(alloy) and CSi(alloy) on γ at 20 wt.% steel shavings are expressed as:

$$\alpha Si(alloy) = 3.6735\gamma^2 + 5.9388\gamma + 59.788; \quad (R^2 = 1); \quad (7)$$

$$CSi(alloy) = 2.551\gamma^2 + 0.398\gamma + 37.051. \quad (R^2 = 1). \quad (8)$$

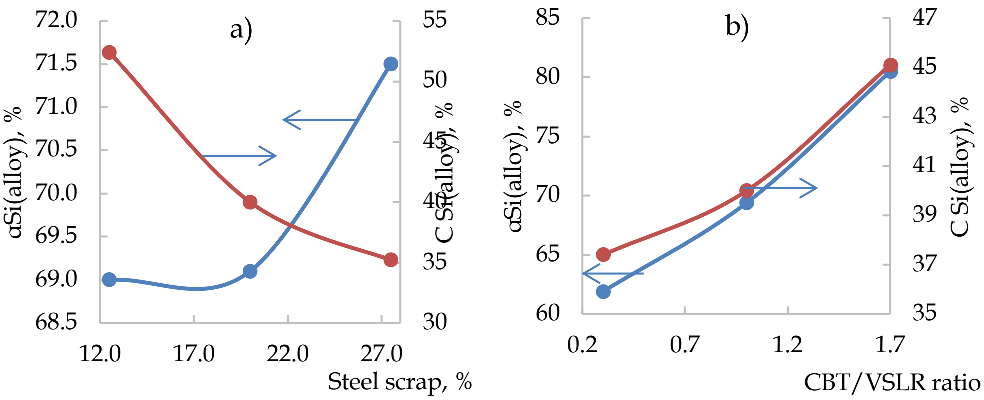


Fig. 3. Effect of the CBT/VSLR ratio (γ) and steel shavings content (St.) on silicon recovery and silicon concentration in the alloy:(a) γ = 1; (b) St. = 20 wt.%

As shown in Fig. 3, at γ=1, increasing the steel shavings content beyond 20 wt.% leads to an increase in silicon recovery into the alloy, accompanied by a decrease in its silicon concentration. At a constant steel shavings content of 20 wt.%, increasing the γ ratio results in both higher silicon recovery and increased silicon concentration in the alloy.

To determine the values of the independent variables at which silicon recovery and silicon concentration in the alloy reach their maximum values, further investigations were carried out using a second-order rotatable experimental design. The effect of the CBT/VSLR mass ratio (γ) and the amount of steel shavings (St., wt.%) on silicon recovery (αSi(alloy)) and silicon concentration (CSi(alloy)) was studied within the variable ranges presented in Table 1.

Table 2 presents the experimental design matrix and the results of ferrosilicon smelting from coal beneficiation tailings and vanadium-bearing shale leaching residue.

Based on the experimental results presented in Table 2, the following second-order regression equations were obtained in the form αSi = *f*(γ, St) and CSi = *f*(γ, St):

$$\alpha Si(alloy) = 65.137+4.759\ \gamma-0.627\ St+4.42\ \gamma2+0.0224\ St2-0.0849\ \gamma\ St; \quad (9)$$

$$CSi(alloy) = 81.99+3.16\ \gamma-3.467\ St+1.87\ \gamma2+0.062\ St2-0.1226\ \gamma \cdot St. \quad (10)$$

Table 1. Factor levels and variation intervals used to study the effect of component ratio and steel shavings content during smelting

Variable level	Coded value	Natural value	
		γ	St, %
Basic level	0	1	20
Variation interval	Δ	0.5	5.3
Upper level	+1	1.5	25.3
Lower level	-1	0.5	14.7
Upper axial ("star") point	+1.414	1.7	27.5
Lower axial ("star") point	-1.414	0.3	12.5

The obtained regression equations adequately describe the experimental data, since the calculated Fisher criteria for Equations (9) and (10) (6.2 and 5.32, respectively) are lower than the tabulated value (6.59). Using the developed regression models, three-dimensional response surface plots and contour maps illustrating the influence of the component ratio (γ) and steel shavings content (St.) on αSi(alloy) and CSi(alloy) were constructed in MathCAD software (Inkov et al., 2003; Ochkov, 2007; Glukhikh et al., 2023).

Table 2. Experimental design matrix and results of ferrosilicon smelting

No.	Coded variables		Natural variables		Technological parameters			
	X1	X2	γ	St, %	αSi(alloy) exp., %	αSi(alloy) calc., %	CSi(alloy) exp., %	CSi(alloy) calc., %
1	-1	-1	0.5	14.7	64.2	63.6	45.6	45.6
2	+1	-1	1.5	14.7	75.3	76.0	49.7	50.7
3	-1	+1	0.5	25.3	66.8	66.0	34.9	34.5
4	+1	+1	1.5	25.3	77.0	77.5	37.7	38.3
5	1.414	0	1.7	20.0	80.5	79.7	45.1	44.1
6	-1.414	0	0.3	20.0	61.9	62.8	37.4	37.8
7	0	1,414	1.0	27.5	71.5	71.7	35.2	35.2
8	0	-1414	1.0	12.5	69.0	68.9	52.4	51.8
9	0	0	1.0	20.0	69.4	69.0	40.0	40.0
10	0	0	1.0	20.0	68.8	69.0	39.8	40.0
11	0	0	1.0	20.0	69.0	69.0	40.6	40.0
12	0	0	1.0	20.0	69.5	69.0	40.3	40.0
13	0	0	1.0	20.0	68.5	69.0	39.5	40.0

As shown in Fig. 4, silicon recovery into the alloy exceeding 80% is achieved at γ = 1.6-1.7 and 22.6-27.5 wt.% steel shavings. The maximum silicon recovery reaches 81.7%. The highest silicon concentration in the alloy (56.5%) is observed at γ = 1.7 and 12.5 wt.% steel shavings.

To determine the optimal parameters for electric smelting of vanadium-bearing shale leaching residue and coal beneficiation tailings, a combined response map illustrating the variation of technological parameters as a function of γ and steel shavings content was constructed (Fig. 5). The technological parameters corresponding to the boundary points of the different regions are presented in Table 3. It can be seen that electric smelting of VSLR and CBT results in the production of two grades of ferrosilicon, FeSi45 and FeSi50 (Table 4).

Based on the data presented in Table 4, FeSi45 ferrosilicon with a silicon recovery of 75.0-80.1% is produced at a γ ratio of 1.42-1.7 and a steel shavings content of 16.7-23.05 wt.%. FeSi50 ferrosilicon with a silicon recovery of 75.0-79.4% is obtained at γ = 1.44-1.7 and 14.3-17.8 wt.% steel shavings.

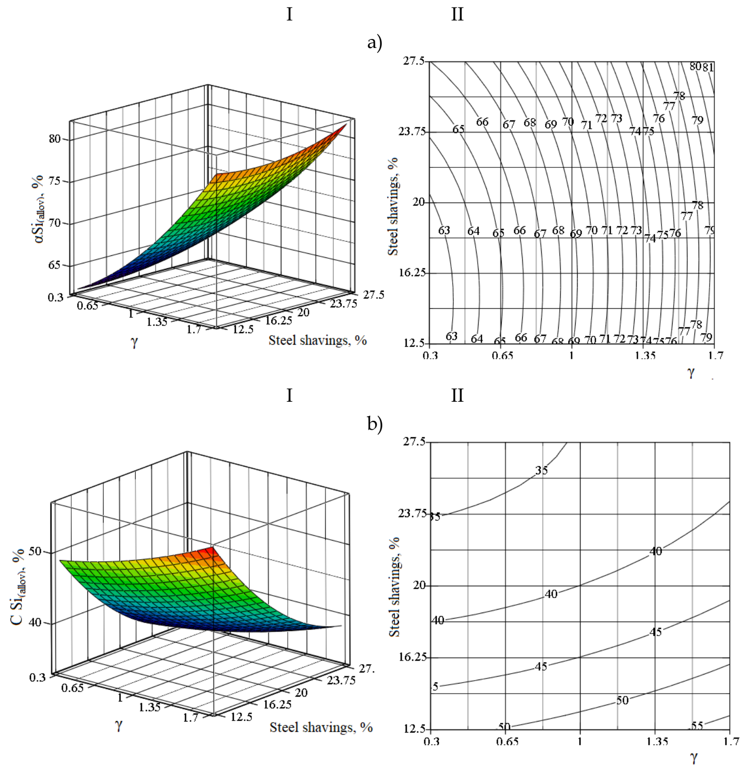


Fig. 4. Effect of the component ratio (γ) and steel shavings content (St.) on silicon recovery (f) and silicon concentration (b) in the alloy: (I) three-dimensional response surface; (II) horizontal contour section

Table 3. Technological parameters corresponding to the regions shown in Fig. 5

Points in Fig. 5	γ	St, %	αSi(alloy), %,	CSi(alloy), %
a	1.42	21.1	75.0	41.0
b	1.7	23.05	80.1	41.0
c	1.7	17.8	79.4	47.0
d	1.45	16.7	75	47
e	1.44	14.3	75.0	51.0
f	1.7	15.3	79.4	51.1

Table 4. Technological parameters of the regions shown in Fig. 5 and corresponding ferrosilicon grades

Region	γ	St, %	αSi(alloy), %,	CSi(alloy), %	Alloy grade
abcd	1.42-1.7	16.7-23.05	75.0-80.1	41-47	FeSi45
dcfe	1.44-1.7	14.3-17.8	75-79.4	47-51	FeSi50

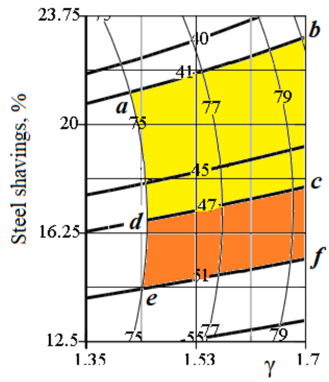


Fig. 5. Combined graphical representation of the effect of γ and steel shavings content (St.) on αSi(alloy) and CSi(alloy): (▬▬▬) - CSi(alloy), %, (———) - αSi(alloy), %

Fig. 6 presents, as an example, a photograph of the ferroalloy obtained during electric smelting of the charge (Experiment No. 5, Table 2). The silicon content in the alloy, determined using the empirical density-based relationship CSi(alloy)=f(ρ), was 45.1%. According to SEM-EDS analysis of the alloy (Fig. 7), the silicon content was 43.38%. Based on these results, the produced ferroalloy can be classified, in accordance with (State Standard 1415-93, 2011), as FeSi45 grade ferrosilicon. The silicon recovery into the alloy in this experiment was 79.7%.

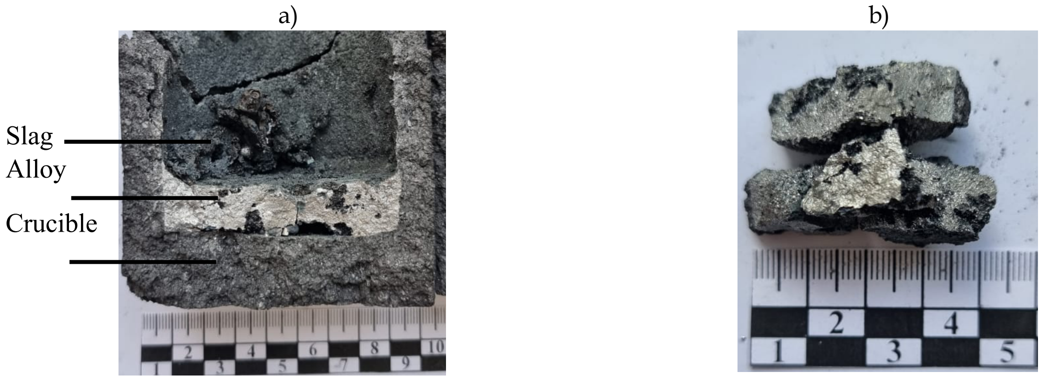
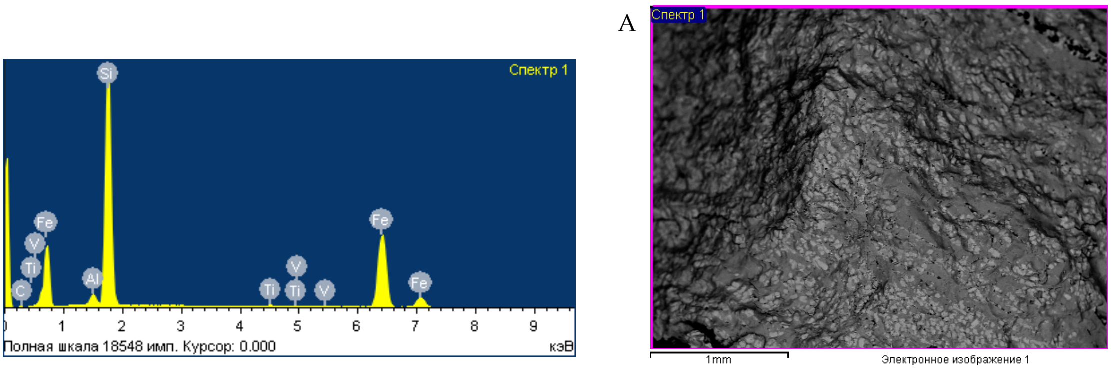


Fig. 6. Photograph of the produced ferroalloy: (a) crucible cross-section; (b) ferroalloy



Element	C	O	Al	Si	Ti	V	Fe
Content, wt. %	2.48	-	2.12	43.38	0.37	0.26	51.39

Fig. 7. SEM-EDS analysis of the produced ferroalloy

During enlarged laboratory-scale electric smelting of a charge composed of 51.3% coal beneficiation tailings, 30% vanadium-bearing shale leaching residue (VSLR) from hydrometallurgical processing of vanadium quartzites, and 18.7% steel shavings, the total charge mass was 30 kg, with $\gamma = 1.7$. The feasibility of producing FeSi45 grade ferrosilicon was experimentally confirmed. The obtained alloy contained 41.1-43.4% silicon. Silicon recovery into the alloy reached 79%, while the ferrosilicon yield was 46% of the total charge mass. The slag-to-metal ratio (slag factor) was 0.48. These results demonstrate the scalability of the proposed approach and confirm that coal beneficiation tailings can effectively replace conventional coke without deterioration of technological performance, enabling the production of commercially competitive ferroalloys from technogenic raw materials.

4. Conclusions

Based on the results of the conducted experimental and analytical studies, as well as the evaluation of existing approaches to the processing of vanadium-bearing black shale raw materials, the following conclusions can be drawn:

1. Vanadium-bearing black shale quartzites are predominantly processed by autoclave sulfuric acid leaching with recovery of vanadium, molybdenum, uranium, and rare earth elements (REEs),

resulting in the formation of vanadium-bearing shale leaching residue. This residue is mainly utilized in the construction industry for the production of low value-added building materials.

2. Electric smelting of a mixture of coal beneficiation tailings (CBT) and vanadium-bearing shale leaching residue (VSLR) in an arc furnace demonstrated that:
   - depending on the CBT/VSLR ratio (γ) and the steel shavings content, ferrosilicon grades FeSi45 and FeSi50 can be produced;
   - the optimal parameters for producing FeSi45 with silicon recovery of 75.0-80.1% are a CBT/VSLR ratio of 1.42-1.7 and 16.7-23.05 wt.% steel shavings;
   - the optimal parameters for producing FeSi50 with silicon recovery of 75.0-79.4% are γ = 1.44-1.7 and 14.3-17.8 wt.% steel shavings.
3. Enlarged laboratory-scale electric smelting of a 30 kg charge containing 51.3% coal beneficiation waste, 30% vanadium-bearing shale leaching residue, and 18.7% steel shavings confirmed the feasibility of producing FeSi45 grade ferrosilicon with a silicon content of 41.1-43.4% and silicon recovery of 79% into the alloy.

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