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## Synchronous activation of Si and Al in vanadium-bearing shale leaching residue via sodium carbonate additive

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### HIGHLIGHTS

- The synchro activation of Si and Al in raw material was realized by sodium carbonate.
- Under the action of sodium carbonate, quartz crystals were transformed to nepheline.
- A safe and efficient activation process was proposed for VSLR.

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### ABSTRACT

With increasing academic interest in geopolymers, research into the activation of geopolymer raw materials has become necessary. In this study, a novel activator sodium carbonate was used to activate vanadium-bearing shale leaching residues (VSLR). Under conditions of 15 wt% Na<sub>2</sub>CO<sub>3</sub> addition, 450 °C activation temperature, and 60 min activation time, the (Si + Al) concentration in the activation residue leachate reached 1350.2 mg/L, which represents an increase of roughly 30% compared to that obtained by adding NaOH under the same conditions. Through thermodynamic and phase transformation analyses, it was found that the crystal minerals containing Si and Al in VSLR were synchronously activated at a lower temperature due to the presence of sodium carbonate. Therefore, the activation efficiency of Si and Al in VSLR was greatly improved and energy consumption reduced. The novel activator sodium carbonate is practically feasible, easy to operate, and highly efficient for VSLR activation compared to the traditional activator sodium hydroxide.

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## 1. Introduction

Vanadium-bearing shale leaching residue (VSLR) is produced as a by-product of the vanadium shale leaching process [1–5]. Roughly 120–150 tons of VSLR are produced when extracting 1 ton of vanadium product [6], as the vanadium grade in a vanadium shale is generally low [7,8]. Currently, the main disposal methods of VSLR are accumulation or tailings landfill, which occupies vast amounts of land, causes environmental pollution, and wastes resources. The use of VSLR for the preparation of high value-added products such as geopolymers was recently proposed by Jiao [9,10]. Geopolymers are excellent building materials due to their durability and thermal stability. This not only solves the problems of land use, environmental pollution, and resource wastage caused

by the improper disposal of VSLR, but also facilitates better resource utilization, resulting in economic and environmental benefits.

The silicon in VSLR mainly exists in the form of quartz, which has a low activity unless it undergoes activation. The low activity of VSLR makes it difficult to dissolve, which suggests that it does not participate in polymerization reactions that can be used to prepare geopolymers with low compressive strengths [11–14]. Therefore, it is necessary to activate VSLR and increase the dissolution rate of Si<sup>4+</sup> and Al<sup>3+</sup> in order to facilitate the subsequent preparation of desirable geopolymers. Alkali fusion [15–18] is an effective activation method for VSLR. The VSLR is usually heated in the presence of a certain amount of alkali additive, which can activate inert substances and contribute to the extraction of silicon and aluminum components.

At present, VSLR is mainly activated by either NaOH or Ca(OH)<sub>2</sub> [19–23], which have the disadvantages of a single activation effect,

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low activation efficiency, high risk of operation and causing serious corrosion of equipment, making using these additives problematic for large-scale applications.

Therefore, we need to find an environmentally friendly activator with multiple activation effects, a high activation efficiency, and good operability. By comparing our requirements with the activation process of fly ash, we found that  $\text{Na}_2\text{CO}_3$  [24–26] may be a suitable activator. This study used  $\text{Na}_2\text{CO}_3$  as an activator to investigate alkali fusion in VSLR in order to achieve safe and efficient activation.

## 2. Experimental

### 2.1. Materials

The VSLR used in this study was acid leaching residue of vanadium shale from Hubei province, China. Due to the low pH value of the leaching solution which leads to the VSLR, the pH value of the VSLR was only 2.75, which would result in an increased alkali dosage during the process of the alkali fusion and the preparation of geopolymers. Therefore, the VSLR was water-washed until its pH was approximately 7.0 before beginning the alkali fusion treatment process. The washed VSLR is hereafter referred to as raw material throughout this paper.

Chemical multi-elemental analysis of the VSLR was carried out by inductively coupled plasma atomic emission spectroscopy (ICP-AES), the results of which are listed in Table 1. It can be seen that the main chemical components of the VSLR are  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ , with contents of 64.41% and 3.87% respectively. By analyzing the phase of the VSLR (Fig. 1), it can be seen that the main mineral phases are quartz, gypsum and hematite, indicating that the  $\text{SiO}_2$  in the VSLR mainly exists in the form of quartz. Quartz is an extremely stable crystalline mineral, which results in the low activity of VSLR. It is therefore necessary to take certain measures to activate the quartz. Particle size analysis of the VSLR was carried out using a laser particle size distribution instrument. It was concluded that the predominant particle size of the VSLR was less than 60  $\mu\text{m}$ , accounting for more than 90 wt%.

### 2.2. Alkali fusion and activation effect evaluation

The VSLR and the sodium carbonate were mixed evenly at different mass ratios and ground for 2 min, then heated in a muffle furnace at predetermined conditions of temperature and time. In this study, the activation effects on the VSLR were evaluated by measuring the leaching concentrations of Si and Al: The higher the leaching concentrations of Si and Al, the better the activation effect of the VSLR. The leaching concentrations of Si and Al in the activation residue after undergoing alkali fusion treatment were determined by the following method. First, 1 g of activation residue was added to a 100 mL beaker containing 40 mL of 5 mol/L NaOH solution, which was then heated at 90 °C for 30 min. After this, 10 mL 37 wt% hydrochloric acid solution was added to the beaker and stirred for 30 min. Finally, the leachate was obtained by vacuum filtration and the concentrations of Si and Al in the leachate were measured using ICP-AES.

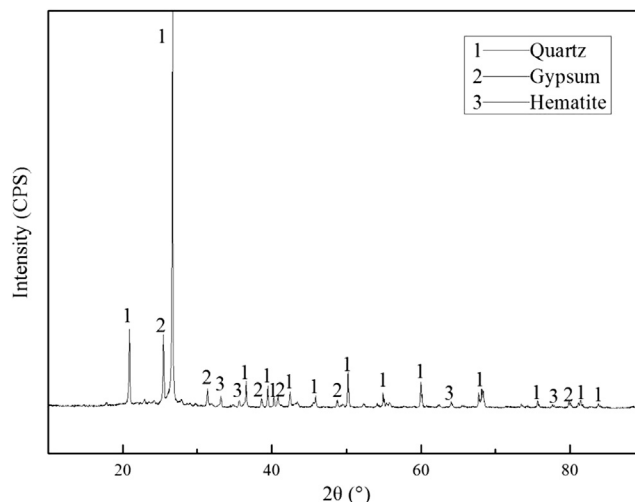


Fig. 1. XRD pattern of the VSLR.

### 2.3. Analytical methods

The phase compositions of the activation residues were tested using a D/MAX-RB X-ray diffractometer (Rigaku, Japan) using  $\text{Cu K}\alpha$  radiation. The infrared spectra of the samples were measured using a Nexus spectrometer (Thermo Nicolet, USA) and the KBr tableting method. A JSM-6610 scanning electron microscope (SEM, JEOL, Japan) equipped with a QUANTAX200-30 energy dispersive spectrometer (EDS, BRUKER, Germany) was employed for studying the surface morphology of the activation residues. The concentrations of Si and Al in the leachate and the chemical composition of the VSLR were obtained using ICP-AES conducted on an IRIS Advantage ER/S instrument (Thermo Elemental, USA). The standard Gibbs free energy ( $\Delta G^0(T)$ ) of the considered chemical reactions can be calculated using the “Reaction” module of FactSage 7.1 software.

## 3. Results and discussion

### 3.1. Thermodynamic calculation of alkali fusion reaction

The main chemical components of VSLR are  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and CaO. Therefore, the reactions between VSLR and sodium carbonate were dominated by the interactions between  $\text{Na}_2\text{CO}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$  and CaO [27,28]. Possible reaction equations are as follows:

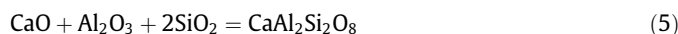
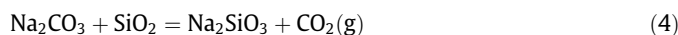
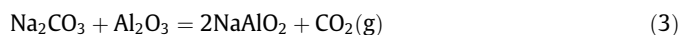


Table 1  
Chemical multi-elemental analysis of the VSLR.

Element	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	CaO	MgO	$\text{K}_2\text{O}$	$\text{Na}_2\text{O}$	$\text{TiO}_2$	$\text{SO}_3$	$\text{V}_2\text{O}_5$
Content,wt%	64.41	3.87	3.31	4.51	0.17	0.34	0.03	0.29	5.75	0.17

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