

Study on The Performance and Hydration Mechanism of Hybrid Activation of Vanadium-Titanium Iron Ore Tailings

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Abstract

This paper investigates the activation of vanadium titanium iron ore tailings (VTIOT) to enhance their cementitious potential. Building upon previous studies, it explores two hybrid activation methods, thermal and chemical activation, to analyse how calcination temperature affects the physical and chemical properties of VTIOT and the performance of resulting cementitious materials, and how different activators affect the performance of VTIOT cementitious materials, and to analyse the mechanism of hydration. Through various analyses, including TG-XRD, SEM, alkali leaching, mechanical testing, hydration heat analysis, and TGA, the hydration mechanism of VTIOT cementitious materials is examined. Results indicate minimal changes in micro-morphology and mineral phase of VTIOT calcined within a range of 550°C to 950°C for 2 hours. Moreover, mortars doped with 30% of VTIOT from different calcination temperatures exhibit comparable compressive strength and activity index to uncalcined VTIOT. Notably, a 0.2‰ dosage of Diethanolisopropanolamine (DEIPA) yields an 85% activity index, outperforming other dosages and activators. Additionally, DEIPA prolongs the induction period of hydration heat and increases the exothermic peak and hydration exotherm amount within 7-d compared to other activators. These findings shed light on the efficacy of hybrid activation methods and the influence of activators on the hydration kinetics of VTIOT cementitious materials.

1. Introduction

Vanadium-titanium magnetite (VTM) is an essential strategic mineral resource in China [1],[2], with estimated reserves of more than 18 billion tons [3],[4]. During the beneficiation process of the mineral, a large amount of solid industrial waste, vanadium-titanium iron ore tailings (VTIOT), is generated [5],[6], which not only occupies a large amount of land resources, but also forms severe environmental pollution [7] and safety hazards [8]. For

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example, heavy metals' Cr, Zn, Ni, and Cu content in the soil around the VTIOT pond exceeds the soil background value [9]. To solve the environmental problems caused by tailings and to adapt to the new requirements of green and low-carbon development, the Chinese government has proposed that tailings be used as raw materials to develop new types of cementitious materials, low-carbon concrete and other low-carbon building materials [10]. However, since most of the VTIOT are the low-silica and low-calcium tailings [11]-[13], which are not conducive to the preparation of cementitious materials [14], there are fewer reports of related studies.

To realise the use of VTIOT as a mineral admixture instead of cement, the research team of this paper investigated various activation methods [15] such as mechanical [16], chemical [17], thermal [18], and mechanical-chemical [19],[20], mechanical-thermal [21], and thermal-chemical [22]. Mechanical activation was used in the previous stage to obtain the VTIOT powder with an activity index of 75%, which realised the tailings in the application of cementitious materials. In this paper, based on mechanical activation, the hybrid activation technology of thermal activation and chemical activation is further adopted to study the effect of calcination temperature on the physical and chemical properties and activity of VTIOT, and the impact of the type of activators and the amount of doping on the activity and hydration mechanism of VTIOT, which provides support for the advancement of the research and development of VTIOT as a green and low-carbon building material.

2. Experimental Materials and Methods

2.1 Materials

The materials used in this study include Vanadium-Titanium Iron Ore Tailings (VTIOT), sand, cement, water, diethanolisopropanolamine (DEIPA), sodium silicate (SS), carbide slag (CS), and desulfurization gypsum (FGDG). The tailings used in the experiment were taken from the VTIOT pond of Jingcheng Mining Group in Kancheng County, Chengde City, Hebei Province. Its chemical composition is shown in Table 1, with 43.55% SiO₂, 15.95% calcium oxide (CaO), 12.04% iron oxide (Fe₂O₃) and 10.99% aluminium oxide (Al₂O₃). The main mineral composition is shown in Fig. 1, containing diopside, chlorite, hornblende, mica, quartz, ilmenite, epidote and plagioclase. The density of VTIOT is 3.133 g/cm³. From Fig. 2, it can be found that the raw VTIOT particles exhibit irregular shapes and are not uniform in size.

The cement used in the experiment was P.I 42.5 reference cement. Its chemical composition is shown in Table 1. Standard sand, following the China ISO standards, was also incorporated. The water used in the experiment was tap water sourced from Shijingshan District, Beijing. DEIPA, SS, CS, and FGDG were taken from the State Key Laboratory of Solid Waste Reuse for Building Materials.

Table 1 Chemical composition of VTIOT and reference cement (wt.%)

Materials	SiO ₂	CaO	Fe ₂ O ₃	Al ₂ O ₃	MgO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	BaO
VTIOT	43.55	15.95	12.04	10.99	9.28	1.92	1.22	1.12	0.62	0.24
P.I 42.5 cement	21.81	63.55	2.78	5.00	2.28	0.15	0.56	0.25	-	-
Materials	SO ₃	MnO	CeO ₂	SrO	Pr ₂ O ₃	Cl	Cr ₂ O ₃	NiO	LOI	
VTIOT	0.24	0.22	0.16	0.14	0.12	0.08	0.05	0.04	2.01	
P.I 42.5 cement	2.13	-	-	-	-	-	-	-	1.69	

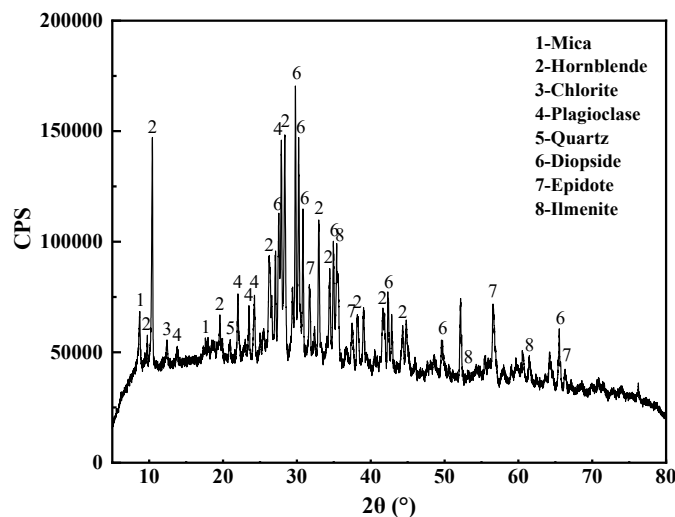


Fig. 1 XRD pattern of VTIOT

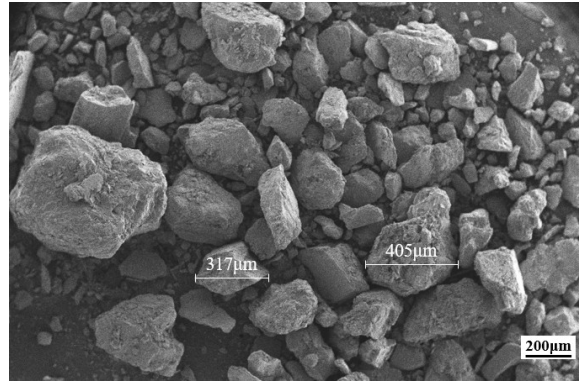


Fig. 2 SEM image of the raw VTIoT

2.2 Experimental Methods

2.2.1 Specimen Preparation

The previous mechanical activation experiments by Tian et al. [23] have confirmed that by grinding the VTIoT with a 500 mm cement test small ball mill with each load set at 5 kg for 50 minutes, the resulting VTIoT powder has the optimal activity index. Therefore, in this study, the VTIoT that had been grounded for 50 minutes was further used for activation experiments. The procedures are as follows:

- (i) VTIoT powder in the CINITE TSX1600 muffle furnace calcination, placed in the amount of 500 g/time, thickness ≤ 1.5 cm, calcination temperature was set to 550°C, 650°C, 750°C, 850°C, 950°C, 5°C per minute to increase the temperature, starting from room temperature, take with the furnace to the corresponding temperature, holding 2 h, and then with the furnace to cool down to room temperature, take out.
- (ii) The FGDG was calcined in the muffle furnace, placing 250 g/time, taking with the furnace to 800 °C, holding for 2 h, and then with the furnace to cool down, take out.
- (iii) The CS was calcined in the muffle furnace, placing the amount of 250 g/time, taking with the furnace to 850°C, holding for 2 h, and then with the furnace to cool down, take out.
- (iv) SS was diluted with tap water at a concentration of 13.52%.
- (v) DEIPA was diluted with tap water alone at 0.64%.

2.2.2 Calcination Analysis of VTIoT

The procedures of calcination analysis of VTIoT are as follows:

- (i) Mineral composition and morphology analysis of VTIoT. The mineral composition of the specimens was analysed by X'Pert Power X-ray diffractometer (XRD), and the morphology of the specimens was observed by ZeissSupra55 scanning electron microscope (SEM) produced by Carl Zeiss, Germany.
- (ii) An alkali leaching method was employed to assess the active components within the VTIoT powder. Firstly, 1g of the original tailings powder or grinding VTIoT powder was added to 40 mL of sodium hydroxide (NaOH) solution with a 5 mol/L concentration. The mixture underwent magnetic stirring for 2 h at room temperature, followed by filtration. Subsequently, 10 mL of hydrochloric acid with a mass fraction of 37% was added to the filtrate and stirred for 30 min. Finally, the content of Si and Al elements in the solution was determined using ICP-OES.

2.2.3 Strength Activity Index Analysis of VTIoT Mortar Specimens

To assess the strength activity index in VTIoT mortar specimens, the evaluation was conducted following the guidelines outlined in GB/T1596-2017 "Fly Ash Used in Cement and Concrete." The study specifically focused on determining the 7-d and 28-d compressive strength of VTIoT mortar subjected to varying calcination temperatures and activators. Using the following formula, the strength activity index was subsequently determined based on the compressive strength.

$$H_{28} = \frac{R}{R_0} \times 100 \quad (1)$$

where H_{28} is the strength activity index (in %), R is the 28-d compressive strength of the test mortar (in MPa), and R_0 is the 28-d compressive strength of the compared mortar (in MPa). The 7-d activity index can be calculated accordingly.

The specimen size was 40mm×40mm×160mm. When evaluating mortar specimens of VTIoT at different calcination temperatures, the cementitious material was mixed according to the mass ratio of 3:7 between the VTIoT powder and reference cement. When evaluating mortar specimens of the effect of different activators, the cementitious material was mixed according to the mass ratio of 3:7 between the VTIoT powder and reference cement, and in equal proportions by deducting the corresponding proportion of the mass of the activators. W/C was 0.5. Experimental sand is standard sand, and the experimental apparatus is a cementitious mortar flexural and compressive testing machine. To the appropriate age, all specimens were soaked and cured in a standard curing box.

2.2.4 Chemical Activation Paste Experiments of VTIoT Specimens

The activator with the highest activity index in each group was selected for the paste experiments. The cementitious chemical activation materials were prepared the same way as above, 4g in the paste experiment. W/C was 0.5, added and stirred well. The paste comparison tests of cementitious materials were carried out to determine the 7-d heat of hydration, SEM and TGA. SEM equipment was as above. The heat of hydration was tested by an 8-channel thermostatic calorimeter (TAM Air, Thermometric, Sweden), and the TGA was performed with the Q500 TA Thermogravimetric Analyser of Shanghai Lairui Scientific Instruments Co.

3. Results and Discussion

3.1 Thermal Activation

3.1.1 Effect of Calcination Temperature on The Micro-Morphology

The VTIoT specimens show different colours at different calcination temperatures. From Fig. 3, with the temperature increase, the VTIoT specimens' colour gradually changed from greenish grey to earthy yellow, and the Fe₂O₃ caused this colour change in the tailings at high temperature. The amount of Fe₂O₃ content and the calcination temperature determined the colour change of VTIoT. In addition to the colour change of the sample, in the five temperature nodes from 550°C to 950°C, VTIoT did not occur sintering or melting phenomena, basically kept the loose state unchanged, only at 950°C, there is a slight sintering, but it is still touchable to disperse. According to related research, when the calcination temperature of VTIoT reaches 1050°C, the tailings particles have a certain degree of bonding between the particles, and there will be a small amount of glassy phase generation [24].



Fig. 3 VTIoT specimens colour comparison: (a) The VTIoT at different calcination temperatures; (b) The uncalcined VTIoT; and (c) The VTIoT after calcination at 950°C

From Fig. 4, further observation of the micro-morphology of the uncalcined VTIOT and the calcined VTIOT at different temperatures, it is found that there is no significant change in the micro-morphology of the uncalcined VTIOT, and it remains the same.

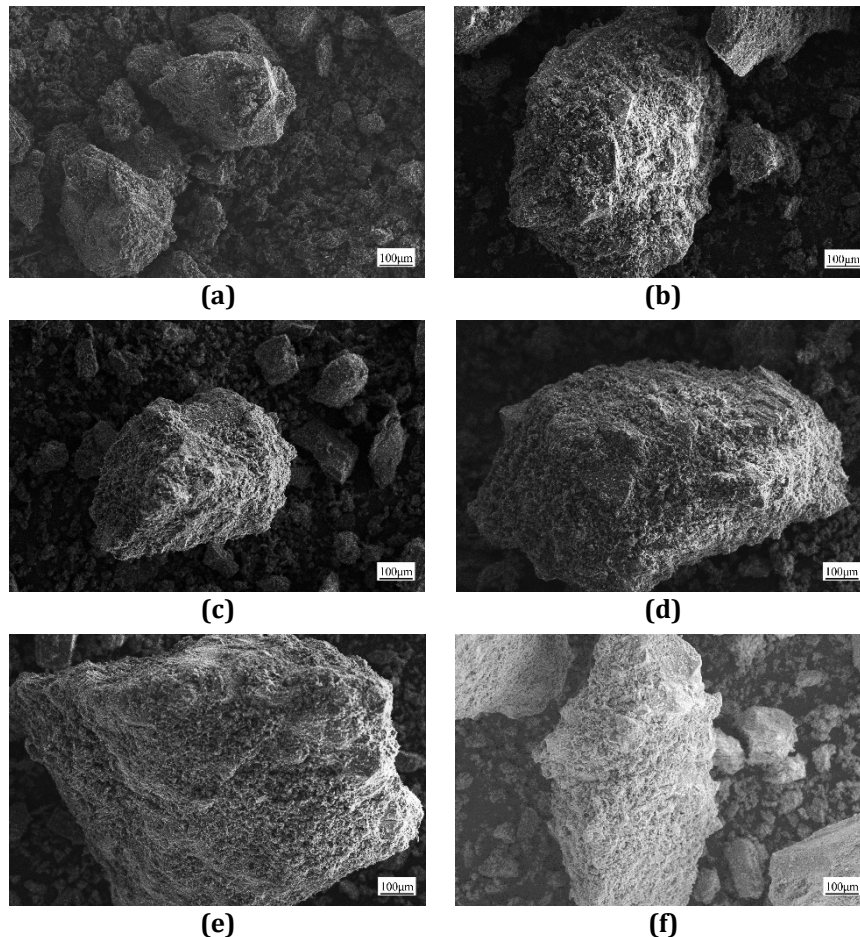


Fig. 4 SEM images of VTIOT at different calcination temperatures: (a) The uncalcined VTIOT; and (b) to (f) Calcined VTIOT at 550°C, 650°C, 750°C, 850°C, and 950°C, respectively

3.1.2 XRD Analysis at Different Calcination Temperatures

From the XRD of VTIOT at different calcination temperatures in Fig. 5, it can be seen that the mineral phase of VTIOT is similar at the three nodes of calcination temperatures of 550°C, 750°C and 950°C, and only the diffraction peak of mica has decreased at 950°C. This is consistent with the mica decomposition's temperature nodes at high temperatures [25]. The study shows that the starting deformation temperature of tremolite is 1170°C, the softening temperature is 1280°C, and the melting temperature is 1390°C [26]; the liquid phase line of plagioclase is about 1215°C [27]; the decomposition temperature of chlorite is about 970°C [28]; and the melting point of quartz is about 1750°C [29]. The diffraction peaks of chlorite in the raw ore disappeared after 50 min of mechanical grinding and, therefore, did not appear in the mineral phases at each temperature node. From the TGA-DTG of raw VTIOT (Fig. 6), it can be found that the total weight loss from 20°C-900°C is only 1.81%, and there is only one heat-absorption peak at 550°C-750°C. The mass loss is pronounced, and only the clay material in the mineral phase of VTIOT, chlorite, decomposes in this temperature interval [30]. The above study shows that the VTIOT has good thermal stability at 900 °C. There is no change in the active components of the tailings after calcination. Only a slight decomposition of mica, which has a small content in the tailings, occurs at 950°C, which has a limited role in enhancing the activity of the tailings.

3.1.3 Effect of Calcination Temperature on The Active Elements

The alkali leaching method was used to test the active Si^{4+} and Al^{3+} content in the VTIOT, and the test results are shown in Table 2. The results show that the calcination of VTIOT at five different temperature nodes did not cause any abnormal changes in the active Si^{4+} and Al^{3+} content in the tailings. Compared with the active element content of the VTIOT before calcination, there was also no significant increase in the active element content. This shows

that the calcination of VTIoT did not cause substantial changes in the crystal structure of the minerals, and did not change the active element content of VTIoT.

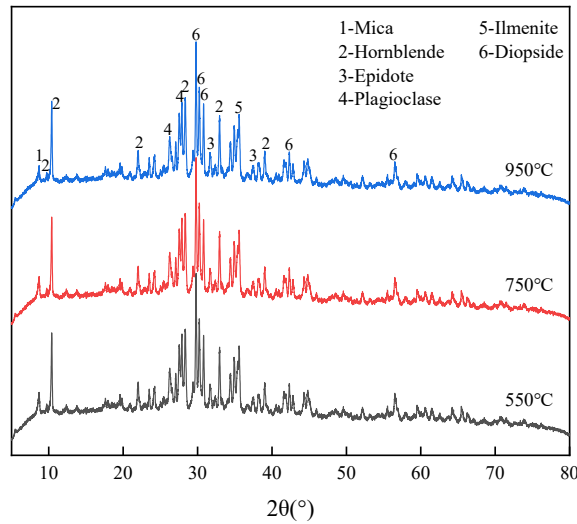


Fig. 5 XRD patterns of VTIoT at different calcination temperatures

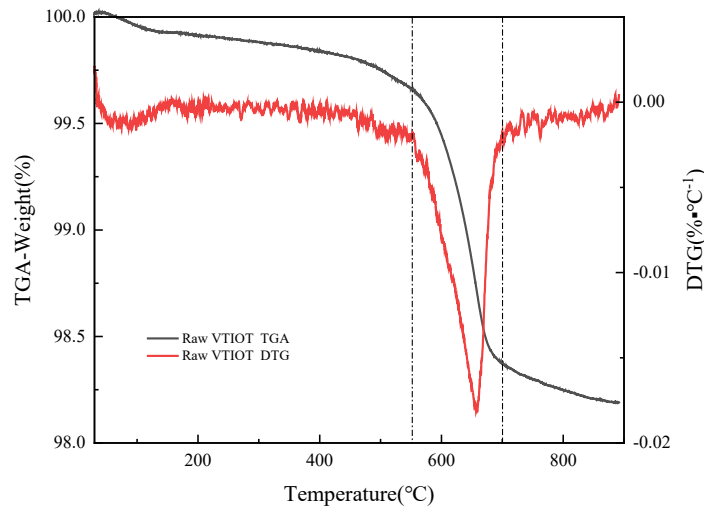


Fig. 6 TGA-DTG curves of raw VTIoT

Table 2 Content of active Si and Al at different grinding times

Grinding time	VTIoT (50min)	VTIoT (550°C)	VTIoT (650°C)	VTIoT (750°C)	VTIoT (850°C)	VTIoT (950°C)
Si ⁴⁺ , Al ³⁺ content (mg/L)	41.6	42.8	39.4	38.4	43.8	37.3

3.1.4 Effect of Calcination Temperature on the Strength Activity Index

The compressive strengths of VTIoT mortar specimens at 7-d and 28-d were tested. The activity index was calculated, and the results are shown in Fig. 7 and Fig. 8. From Fig. 7, the compressive strengths of VTIoT mortar specimens calcined at different temperatures are close to that of the uncalcined VTIoT mortar specimen, the results of the compressive strengths at 7-d and 28-d are close to each other, and the value of the compressive strengths at 28-d is floating up and down in the range of 38MPa. Fig. 8 Activity index also shows that the activity index of calcined VTIoT mortar is close to that of the uncalcined specimen. The result shows that calcined VTIoT cannot improve the activity in this temperature range. Therefore, it is recommended that the calcined tailings should not be used in the activation process. If high-temperature calcination is to be used, it is recommended that the calcination temperature be further increased or the method of rapid cooling be adopted to improve the activity index of VTIoT.

The VTIoT of this experiment contain mineral components such as ore, chlorite, hornblende, tremolite, mica, ilmenite, etc., and there are not many studies on the hardness of the ore, melting temperature, chemical decomposition, and surface energy corresponding to these mineral components, respectively. The authors suggest that not only the tailings types to divide the research object, but also to the mineral components as the object of research, such as pyroxene, chlorite, etc., the study of a single mineral component of the grinding characteristics, melting temperature, chemical reactions, etc., from a single mineral component of the study to a particular kind of tailings of all the mineral components of the study to the tailings of the mineral components of the basic theoretical research to find the tailings suitable for the direction of the application. In this way, not only will there be a better explanation of tailings green building materials utilisation, but also a more precise grasp of its suitable application and promotion direction, to improve the level of comprehensive utilisation of tailings green resources.

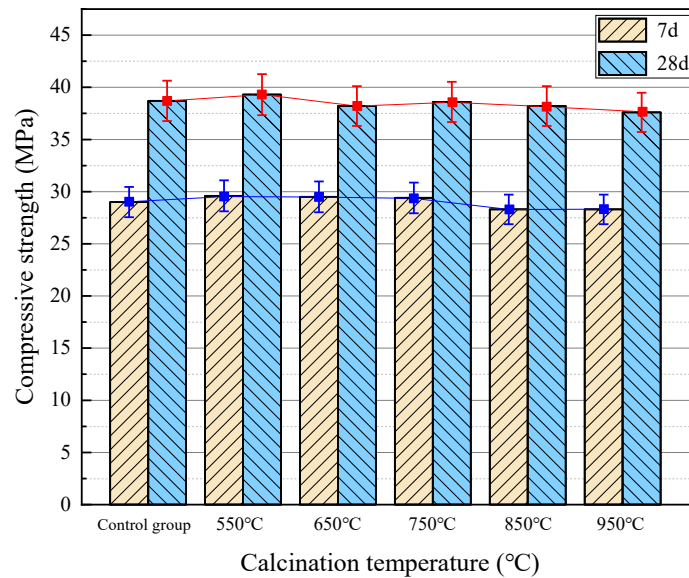


Fig. 7 Compressive strength of VTIoT mortar specimens under different calcination temperature

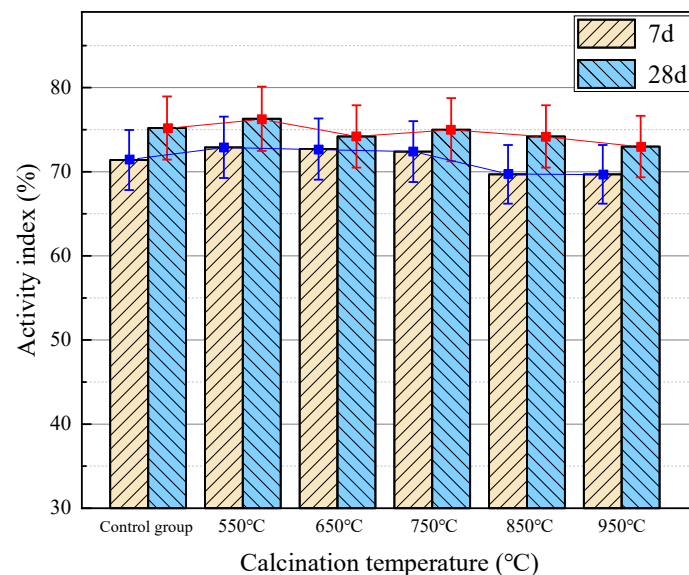


Fig. 8 Activity index of VTIoT under different calcination temperatures

3.2 Chemical Activation

3.2.1 Effect of Activator Type and Dosage on Strength Activity Index

To further improve the activity index of VTIoT mortar, DEIPA, SS, CS, and FG DG were used as chemical activators to test the compressive strength and activity index of VTIoT mortar specimens at 7-d and 28-d. The dosage of activators is shown in Table 3, and the experimental results of compressive strength and activity index are shown in Fig. 9 and Fig. 10. It can be seen that the type and dosage of the activators have an essential effect on the

compressive strength and activity index of VTIoT mortar specimens. The 7-d and 28-d compressive strength of 0.1‰ DEIPA VTIoT mortar specimens were 34.1MPa and 41.8MPa, respectively; the 7-d and 28-d compressive strength of 0.2‰ DEIPA was 36.1MPa and 47.6MPa, respectively; the 7-d and 28-d compressive strength of 0.3‰ DEIPA was 33.5MPa and 42MPa, respectively; which indicated that the dosage of 0.2‰ DEIPA promoted the hydration reaction of cementitious materials, and better improved its 7-d and 28-d activity index, and reached 78.3% and 85%, respectively. The 7-d and 28-d compressive strengths of 2.5‰ SS VTIoT mortar specimens were 29.7MPa and 41.7MPa, which were close to the compressive strength of doped with 5‰ SS, but with 7.5‰ SS, the results were a little lower compared to the other two. From the experimental results, the 7-d and 28-d compressive strengths of the VTIoT mortar specimens doped with different contents of CS and FG DG are close to each other, lower than those doped with DEPIA and SS activators, and their activity index is also lower than that of VTIoT without activators. This indicates that calcined FG DG is anhydrous gypsum [31], calcined CS with increased CaO content [32], the hydration reaction process did not improve the compressive strength of the mortar specimens, consistent with the results of Poon et al. [33], the 7-d, 28-d compressive strength of VTIoT mortar specimens doped with FG DG decreased.

Table 3 Amount of activators in cementitious materials

Types of activators	DEIPA			SS			CS			FGDG		
Amount	0.1‰	0.2‰	0.3‰	2.5‰	5‰	7.5‰	0.5%	1%	2%	0.5%	1%	2%

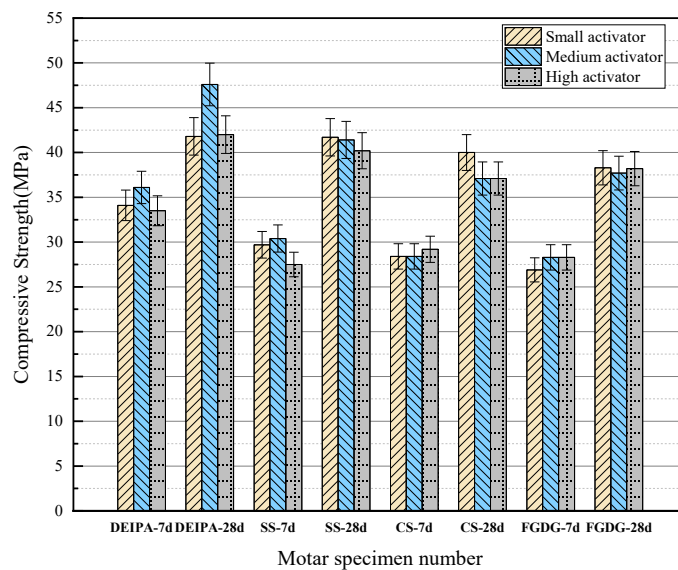


Fig. 9 Compressive strength of VTIoT mortar specimens under different activators

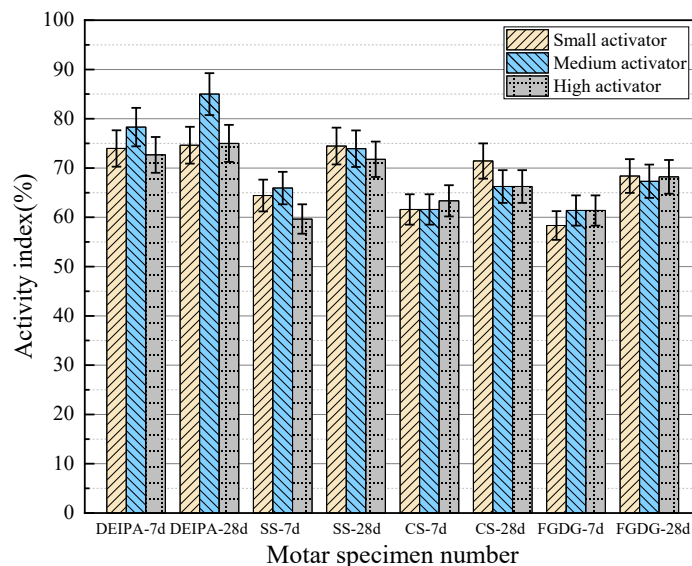


Fig. 10 Activity index of VTIoT mortar under different activators

From the test results, it can be found that the compressive strength and activity index of VTIoT are further improved through the hybrid activation technology, which provides powerful technical support for the use of low-silica and low-calcium tailings as a new type of cementitious materials, low-carbon concrete, high-performance concrete, and filling of air-mining zones, which helps to improve the utilization rate of such tailings, alleviate the pressure of environmental pollution and high energy consumption in the cement industry, and is of great significance to the realization of the dual-carbon strategy of China.

3.2.2 Hydration Mechanism Pure Paste by Chemical Activation

3.2.2.1 Effect of Activators on the Heat of Hydration

The activators have a significant effect on the pre-hydration reaction of the paste. Fig. 11 shows the 7-d heat of hydration of the reference cement (control group) and the paste with activators. From Fig. 11, with time, the exothermic amount of the control group is significantly higher than that of the cementitious material doped with the activators; the induction period of the control group is shorter than that of the cementitious material doped with the activators; and the exothermic peak of the control group is also higher than others. The exothermic peak of the DEPIA-doped cementitious material is higher than that of the other activators. The time to reach the exothermic peak is also significantly delayed, with a more pronounced and steeper drop after reaching the peak, and then at 0.6-d, the exothermic curve had a nearly 90-degree turn. The peak exothermic value of SS, CS, and FG DG pastes is relatively close, and the exothermic rate after the peak has a more obvious difference. The exothermic rate of SS doping decreases faster. It indicates that the hydration kinetic mechanism of cementitious materials under the action of different activators has changed differently, affecting the hydration induction period, acceleration period and subsequent hydration process of cementitious materials, and slows down the hydration process of cement.

Fig. 11 shows that the hydration reaction of cementitious materials after doping with activators and VTIoT significantly reduces the cumulative exothermic amount and slows the hydration process compared with the control group. The total exothermic amount of cementitious materials doped with different activators is the same in about 1-d, except for the lower exothermic amount of CS. After 1-d, the heat released by the cementitious material doped with SS is higher than that with other activators, and DEIPA is the next highest. At about 2.25 d, the total exothermic amount of DEIPA and SS crosses, indicating that the total of the two is the same. After that, the exothermic amount of DEIPA is always higher than that of other activators.

3.2.2.2 SEM Analysis of Cementitious Materials with Different Activators

Fig. 12 shows the SEM morphology of cementitious materials doped with different activators for 7-d, a is doped with 0.2% DEIPA, b is doped with 5% SS, c is doped with 05% CS, and d is doped with 2% FG DG. From Fig. 12, there are many intertwined elongated acicular AFT crystals and agglomerated C-S-H gel generation in the hydration products, and the structure is relatively dense. The tailings particles not involved in the hydration reaction are distributed in the hydration products, which play a better physical filling role.

3.2.2.3 TGA-DTG Analysis of Cementitious Materials with Different Activators

Relevant studies by Yunli [34] have shown that in the DTG curve of cementitious materials composed of silicate cement and fly ash, slag, iron ore tailings, etc., the weight loss peaks in the temperature interval of 50 to 200°C are the mass loss of calcium alumina (AFT), C-S-H gel, and alumina, ferric oxide, monosulfate (AFm), losing bound water. The weight loss peaks in the temperature interval 400 to 500°C represent the mass loss of calcium hydroxide $\text{Ca}(\text{OH})_2$, losing bound water. The corresponding weight loss peaks in the temperature range 600 to 800°C indicate the mass loss of carbon dioxide from the high-temperature decomposition of calcium carbonate (CaCO_3). From Fig. 13(a), it can be found that the DTG of the cementitious material doped with activators also conforms to the above characteristics. However, the doped materials with different activators have some effect on the hydration products formed by the cementitious material. For example, the weight loss peak of C-S-H gel is the smallest when DEIPA is doped, while the weight loss peak of C-S-H gel formed by FG DG is the largest; however, the weight loss peak of AFm of DEIPA is the largest, while the weight loss peak of AFm of SS and CS is the smallest. The peak weight loss of $\text{Ca}(\text{OH})_2$ under the effect of different activators is closer, except for the control group, which indicates that the above four activators have a weak impact on forming $\text{Ca}(\text{OH})_2$ hydration products after 7-d.

From Fig. 13(b), it can be found that the control group has the most considerable mass loss, followed by the mass loss of the doped DEIPA cementitious material. This indicates that the hydration reaction of DEIPA cementitious material is better promoted than that of other activators.

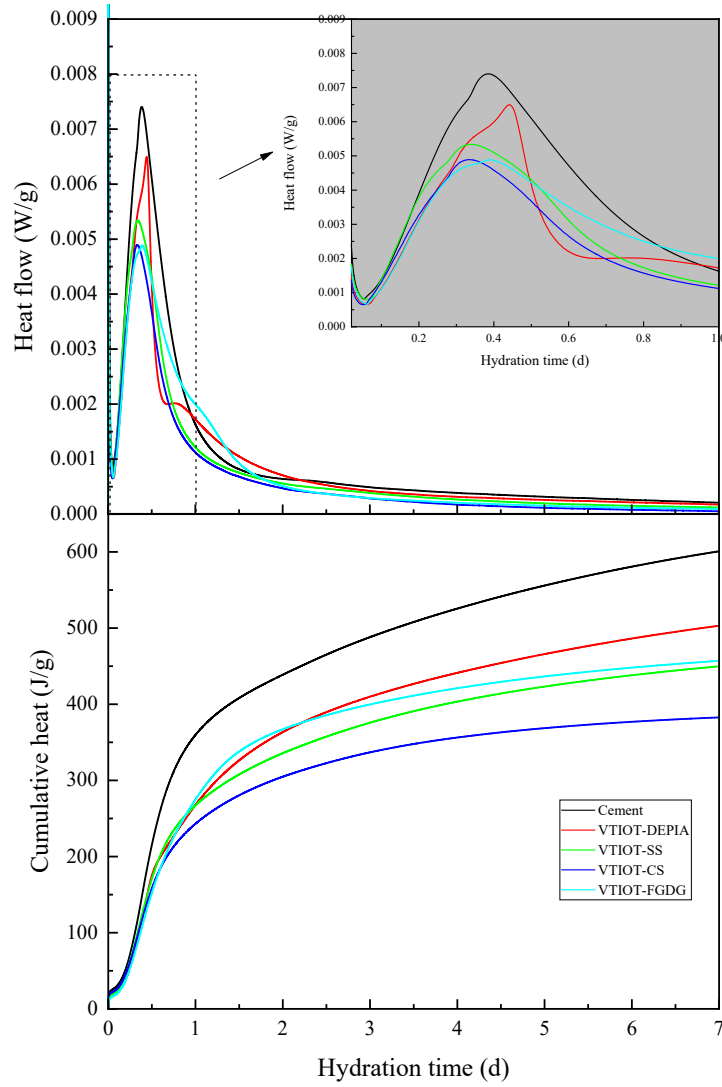


Fig. 11 7-d heat of hydration of paste specimens

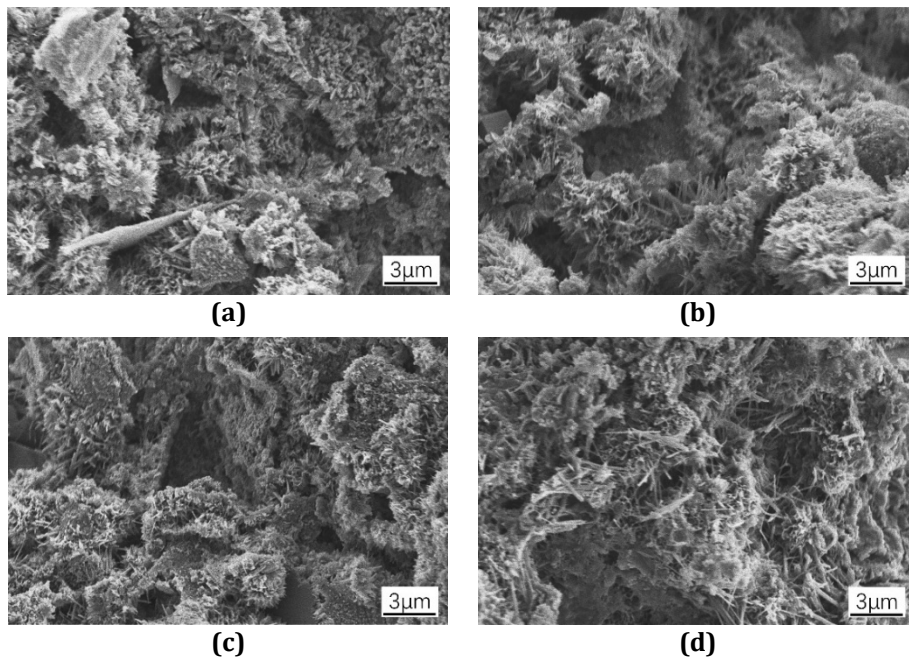


Fig. 12 SEM morphology of VTIOT cementitious materials hydration products for 7-d: (a) VTIOT-DEPIA; (b) VTIOT-SS; (c) VTIOT-CS; and (d) VTIOT-FGDG

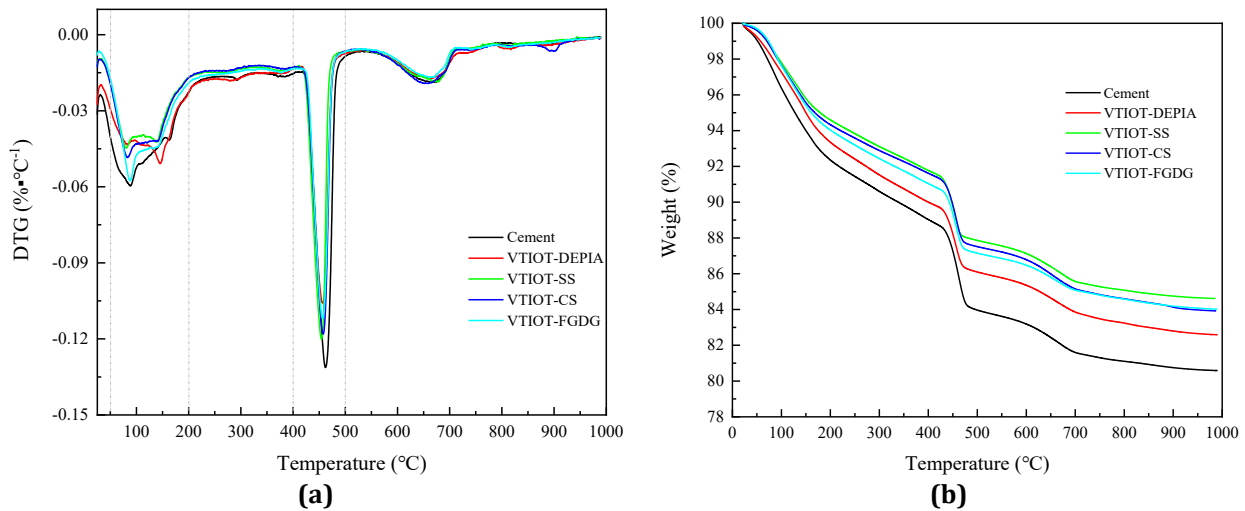


Fig. 13 TGA-DTG thermograms of hydrated pasts: (a)-DTG; and (b)-TGA

4. Conclusion

Based on all the test results analysis, the effects of using hybrid activation techniques, thermal activation or chemical activation, on the physical and chemical properties, strength activity index and hydration mechanism of VTIIOT are as follows:

- Calcined VTIIOT at 550°C-950°C, the tailings' micro-morphology, mineral phase, and active Si⁴⁺ and Al³⁺ elements did not change significantly. The oxidation of Fe₂O₃ in the tailings at high temperature caused the tailings colour to gradually change from greenish grey to earthy yellow; calcining VTIIOT at 950°C slightly melted the mica.
- Mechanical-thermal activation failed to improve further the compressive strength and activity index of VTIIOT mortar. The 7-d compressive strength mixed with 30% of different calcination temperatures of VTIIOT fluctuated 5% above and below 29 MPa, and the 28-d compressive strength fluctuated 5% within 38.6MPa. Its strength activity index is close to the experimental results of mechanical VTIIOT mortar.
- Using mechanical-chemical activation, the type and dosage of activators have a greater effect on the compressive strength and activity index of VTIIOT mortar. Doped with 0.2 ‰ DEPIA mortar 28-d compressive strength of 47.6 MPa, the activity index of 85%, compared with the unadded activator mortar, the activity index has been greatly improved, which will help VTIIOT used in the preparation of green low-carbon concrete, high-performance concrete, ultra-high-performance concrete and new wall materials and other aspects, to promote better energy conservation and emission reduction to achieve the dual-carbon goals.
- Under the action of different activators, the hydration heat induction period of the paste of VTIIOT cementitious materials is prolonged. The exothermic peaks have a big difference, and the hydration exothermic amount of paste doped with DEPIA for 7-d is significantly higher than that of paste doped with others. The TGA-DTG and the SEM results confirmed that the hydration products of the pastes with different activators are Ca(OH)₂, C-S-H, Aft, and AFm.

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Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **Study conception and design:** Tian Xiaoping, Wang Changlong, Hidayati Asrah, Lim Chung Han; **Data collection:** Tian Xiaoping, Zheng Yongchao, Huang Tianyong, Wen Xiaoqing; **Analysis and interpretation of results:** Tian Xiaoping, Wang Changlong; **Draft manuscript preparation:** Tian Xiaoping, Wang Changlong, Hidayati Asrah, Lim Chung Han. All authors reviewed the results and approved the final version of the manuscript.

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