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Test research on the effects of mechanochemically activated iron tailings on the compressive strength of concrete ☆

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HIGHLIGHTS

- High-silicon iron tailings were mechanochemically activated.
- Activated tailings were taken as a supplementary cementing material in concrete.
- The compressive strength of concrete may be as required at some mix proportions.
- The maximum cement substitution rate is 30% in common concrete.
- With a proper admixture, the cement substitution rate can be up to 40%.

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ABSTRACT

High-silicon iron tailings, which have been mechanochemically activated, was used for the preparation of concrete as a supplementary cementing material to substitute cement (by 10%, 20%, 30% and 40% respectively) based on their composition, particle sizes and pozzolanic activity. With the aid of tests, this paper discusses the effects of the tailings on the compressive strength of concrete. Tests show that with an increase in substitution rate of cement in the tailings, the compressive strength of concrete tends to decrease, and when the substitution rate is at 10%, 20% and 30% respectively, the compressive strength of concrete is measured up to the design requirement; if an appropriate amount of water reducing admixture is added while cement is substituted by tailings with a substitution rate of 10%, 20%, 30% and 40% respectively, the compressive strength of concrete is also measured up to the design requirement. The research result demonstrates that as far as the compressive strength concerned, it is feasible to use mechanochemically activated high-silicon iron tailings as a supplementary cementing material to partly substitute cement in concrete.

China is a country rich in mineral resources, and as one of important industrial solid wastes in China, a large amount of tailings are being discharged and deposited. At present, tailings are usually stored within tailings dams, which not only encroaches large amounts of land, causes environmental pollution, but also need large amounts of investment in their management each year, therefore, tailings have already been a heavy burden for mining enterprises. So, how to utilize tailings efficiently is of great significance to the sustainable development of the mining industry. So far, the comprehensive utilization of tailings involves four major aspects: first, reconcentration of tailings, that is, recycling valuable metals or minerals in tailings; second, used as a filler to backfill mined-out areas; third, used in building materials as a raw material; fourth, reclaiming farmland on the tailings dumping ground to plant trees or crops [1].

Tailings are of low-value raw material, so no economic benefit will be presented unless they are used on a large scale [2]. One of efficient approaches is to use them for building materials production [3], and much progress has been made over the years in this field [3–17]. For some types of tailings are rich in silicon, some researchers proposed to use them to prepare cementing materials [18–29]. However, to achieve this purpose, tailings are required to have a certain pozzolanic activity, while in practice, tailings usually have a lower activity, so activating tailings has become the focus of some researches. In literatures [18–20], the potential chemical reactivity of tailings is activated through high temperature

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calcination and rapid cooling; in literatures [21–26], tailings are mixed and ground together with cement clinker, gypsum and other gradients, so that tailings are activated and made into a new cementing material; in literature [28], a combination of high temperature and fine grinding is used to activate tailings; in literature [29], a combination of high temperature and chemical activator is used to activate tailings.

Compared with other types of industrial solid wastes with higher pozzolanic activity, such as fly ash, granulated blast-furnace slag powder and silica fume, etc., tailings, even though rich in silicon, cannot be available as supplementary cementing material for concrete, mainly because of its lower activity. In this research, mechanochemical method, an effective way to activate solid substance, was used to process silicon-rich iron tailings to decrease their particle sizes and raise their activity to a certain level. Literature research reveals that fewer studies published have focused on this area so far.

In consideration of the compositions of this type of tailings, and their particle sizes and chemical activity when they are activated, it is quite possible to use activated tailings as a supplementary cementing material for concrete preparation. This paper relates to the effects of mechanochemical activated iron tailings on the compressive strength of concrete. As we all know, concrete is one of building materials that are consumed largely in civil works, so it will be beneficial for the utilization of tailings as a resource if tailings can be used in concrete as an ingredient.

1. Mechanochemical activation of the tailings

Sampled from a mining enterprise in Liaoning province, China, the tailings present a particle shape as shown in Fig. 1, and the chemical component of the sample is as shown in Table 1, its XRD diffraction pattern as shown in Fig. 2, and its particle size distribution as shown in Fig. 3. As can be seen from Fig. 1, the tailings particles present smooth surface, just like irregular or angular gravels (Fig. 1). The chemical analysis shows (Table 1) that the main chemical composition of the sample is SiO_2, with an average mass fraction as high as 75.23%, and according to literature [30], the tailings are of high-silicon iron tailings; the phase analysis shows (Fig. 2) that the main mineral composition of the sample is quartz, followed by hematite. The particle size analysis shows that the particle size distribution of the tailings ranges from tens of microns to hundreds of microns (as shown in Fig. 3), with a medium value (d_{50}) of 124.727 μm and a specific surface area of 138 m^2/kg.

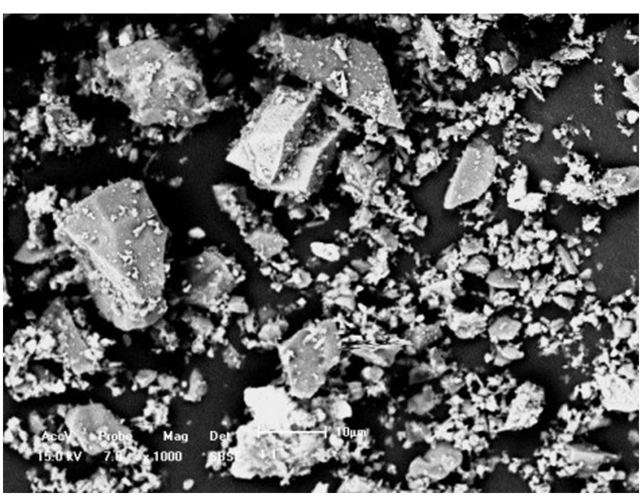

Fig. 1. Particle shape of the tailings (SEM1000×).

A mechanical force can change solid particles physically and chemically, of which their particle sizes may decrease, specific surface area may increase, and the internal structure and physical or chemical properties may change as well [31]. In this course, solid substance is often activated under the action of mechanical forces (such as grinding, impact force and pressure). According to literature [32], when there is no change in chemical compositions or structure, such an activation is called mechanical activation; when there is a change in chemical compositions or structure, such an activation is called mechanochemical activation.

In this research, grinding was adopted as the main means of mechanochemical activation to process the sampled high-silicon iron tailings. The grinding equipment, SYM-Cement Test Ball Mill, is manufactured by Shenyang Jinxin Detection Instrument CO., LTD, which has a cylinder size of φ500 × 500, a capacity of 5 kg and a rotating speed of 48 r/min, with steel balls and steel forging as grinding media.

When the grinding time is within 3.5 h, d_{50} decreases and specific surface area increases gradually over time; when the grinding time reaches 3.5 h, d_{50} is 9.429 μm and specific surface area is 2030 m^2/kg, and the particle size distribution is as shown in Fig. 4. Comparing Fig. 4 with Fig. 3, tailings particles below 10 μm make up an even higher proportion after grinding. As the grinding time reaches 4 h, d_{50} is 21.374 μm and specific surface area is 1400 m^2/kg, and d_{50} begins to rise and specific surface area begins to fall [33], which is because a particle aggregation happens as a result of an increase in surface energy of fine particles when tailings particles are ground to a certain degree. Fig. 5 shows that after grinding, the diffraction peak intensity of SiO_2 in the tailings declines somewhat, and the decline degree increases over time, which manifests that with the proceeding of mechanical grinding, the crystallinity of SiO_2 declines gradually.

Activity tests [34] show that the compressive strength (28d) ratio of cement-tailings mortar (when 30% of cement is substituted by tailings) is 88.9%, and the pozzolanic activity of the tailings is tested to be qualified. However, before grinding, the compressive strength (28d) ratio is 60.1% and the pozzolanic activity is tested not to be qualified. The activity tests were conducted by reference to *Test Method for Activity of Industrial Waste Slag Used as Addition to Cement* (National standard of the People's Republic of China, GB/T 12957-2005) and *Pozzolanic Materials Used for Cement Production* (National standard of the People's Republic of China, GB/T2847-2005). It follows that mechanochemical activation can improve the activity of tailings significantly.

Table 1
Chemical compositions of tailings, wt%.

	SiO_2	Fe_2O_3	Al_2O_3	MgO	CaO	K_2O	SO_3	Na_2O	TiO_2
1	75.11	11.26	2.78	2.08	1.42	0.42	0.078	0.49	0.06
2	75.31	11.40	2.53	2.12	1.48	0.39	0.079	0.46	0.04
3	75.26	11.28	2.61	2.09	1.50	0.40	0.075	0.51	0.07
Average	75.23	11.31	2.64	2.10	1.47	0.40	0.08	0.49	0.06

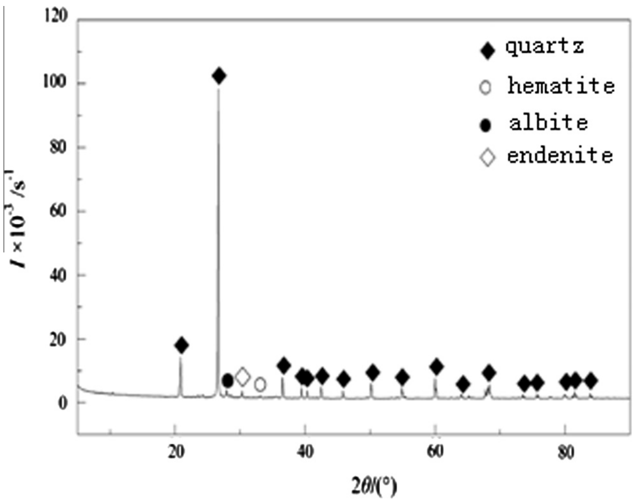

Fig. 2. XRD of tailings.

2. Test materials and mix proportion of concrete

2.1. Test materials

(1) Cement: ordinary Portland cement, produced by Dalian Xiaoyetian Cement Co., Ltd., and the mineral compositions of clinker, chemical compositions and main properties of cement are as shown in Tables 2–4.
(2) Coarse aggregate: natural gravel, with a density of 2930 kg/m^3 and particle size of 5–20 mm, continuous gradation.
(3) Fine aggregate: natural sand, with a density of 2630 kg/m^3 and fineness modulus of 2.71.
(4) Mechanochemically activated high-silicon iron tailings. See Section 1 of this paper for their properties.
(5) Water reducing admixture: polycarboxylic acid type naphthalene series high efficient water reducing agent, produced by Liaoning Kelong Fine Chemicals Co., Ltd.
(6) Water: drinking water.

2.2. Mix proportion of concrete

The mix proportion of concrete was designed as required in the *Specification for Mix Proportion Design of Ordinary Concrete* (National standard of the People's Republic of China, JGJ55-2011).

Two types of concrete, mixed and not mixed with water reducing admixture, were designed. In either type, there are five groups of concrete respectively, so there are 2 control groups (C-0, C′-0) and 8 groups of concrete mixed with tailings in total, with a cement substitution rate of 10% (TC-1, TC′-1), 20% (TC-2, TC′-2), 30% (TC-3, TC′-3), and 40% (TC-4, TC′-4) respectively.

The control concrete is designed with a strength grade of C30 and a slump of 70–90 mm. The mix proportions of concrete are as shown in Table 5.

3. Test results and analysis

3.1. Test results

Tests were conducted according to *Standard for Test Method of Mechanical Properties on Ordinary Concrete* (National standard of the People's Republic of China, GB/T50081-2002).

There were three specimens (150 mm × 150 mm × 150 mm) in each test group to test the compressive strength of the concrete at a curing time of 3 d, 28 d and 90 d respectively, and the average

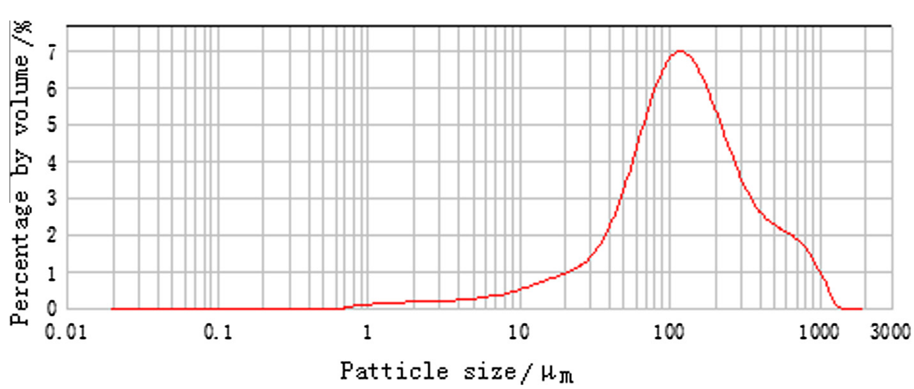

Fig. 3. Laser particle size distribution of tailings.

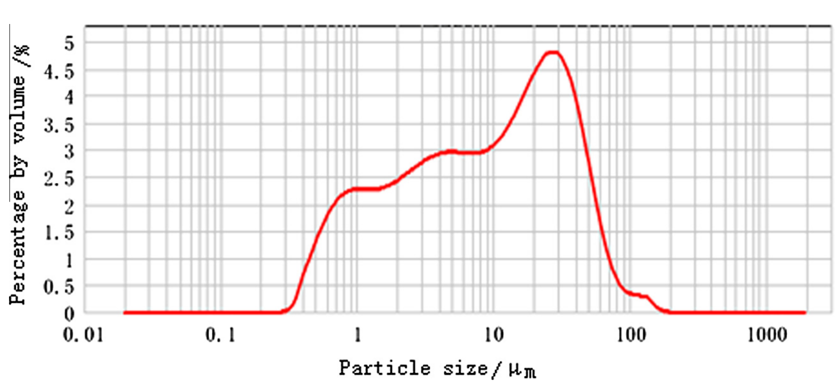

Fig. 4. Particle size distribution of tailings (grinding time: 3.5 h).

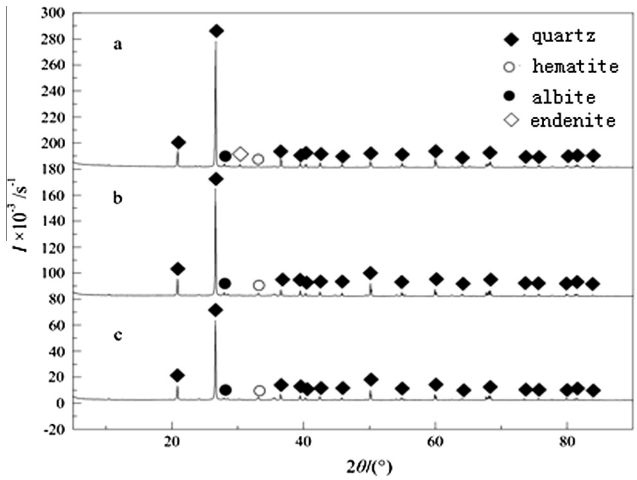

Fig. 5. XRD of tailings after grinding. a—grinding time: 0; b—grinding time: 3.5 h; c—grinding time: 4.0 h.

Table 2
Mineral components of cement clinker, wt%.

C_3S	C_2S	C_3A	C_4AF
52	22.4	8	10

Table 3
Chemical compositions of cement, wt%.

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	LOI
21.36	5.1	3.34	63.76	3.01	0.91	1.69

Table 4
Main technical data of cement.

Setting time/min		Soundness	Compressive strength/MPa		Flexural strength/ MPa	
Initial/min	Final/min		3 d	28 d	3 d	28 d
140	195	Qualified	27.6	50.1	5.8	8.5

Table 5
Mix proportion of concrete, kg/m^3.

Code	Cement	Water	Sand	Gravel	Tailings	Water reducing admixture
C-0	439	215	646	1100	0	0
TC-1	395.1	215	646	1100	43.9	0
TC-2	351.2	215	646	1100	87.8	0
TC-3	307.3	215	646	1100	131.7	0
TC-4	263.4	215	646	1100	175.6	0
C'-0	439	140	673	1147	0	1.3
TC'-1	395.1	140	673	1147	43.9	1.3
TC'-2	351.2	140	673	1147	87.8	1.3
TC'-3	307.3	140	673	1147	131.7	1.3
TC'-4	263.4	140	673	1147	175.6	1.3

value for three test pieces was taken as the test result. Test results are as shown in Table 6.

As can be seen from Table 6, the compressive strength of concrete has a development trend the same as the control group concrete, that is, it increases with the extension of the age of concrete; when the cement substitution rate is 10%, 20%, 30% and 40% respectively, and the compressive strength of the concrete is lower than that of the control group concrete, and the compressive strength of the concrete presents a downward trend with a increase in cement substitution rate. In the case no water reducing admixture is added, the compressive strength of the concrete meets the design requirement when the substitution rate is 10%, 20% and 30% respectively; in the case a proper amount of water reducing admixture is added, the compressive strength of the concrete meets the design requirement when the substitution rates is 10%, 20%, 30% and 40% respectively.

Table 6
Compressive strength of concrete, MPa.

Code	Curing time		
	3 d	28 d	90 d
C-0	25.50	44.06	48.80
TC-1	23.76	42.33	45.35
TC-2	20.42	38.87	40.54
TC-3	18.50	38.29	39.19
TC-4	15.10	32.34	35.36
C'-0	31.58	59.17	61.78
TC'-1	29.84	57.10	59.55
TC'-2	27.10	55.24	56.19
TC'-3	24.52	53.71	55.36
TC'-4	20.91	52.24	53.93

3.2. Analysis of test results

Mechanochemical activation endows tailings with potential activity (pozzolanic activity), which can induce a secondary hydration reaction of $Ca(OH)_2$ – a hydrate of cement and produce C–S–H, thus improving the strength of the concrete greatly.

3.2.1. Analysis of $Ca(OH)_2$ content

In the tailings-cement dual system, the secondary hydration reaction may reduce the content of $Ca(OH)_2$ in the system. This paper analyzed the changes of $Ca(OH)_2$ content in the hardened paste using TG-DTA technology and studied the effects of the use of tailings for substituting cement partly on $Ca(OH)_2$ content, which provides further evidence that a secondary hydration reaction is existent.

While the cement-based hardened paste specimens are being heated, various hydrates may decompose or dehydrate at different temperatures, so we can work out the contents of such hydrates through measuring their mass loss at specific phases of temperature [31]. Fig. 6 shows TG-DTA analysis curves of hardened paste specimen and cement–tailings hardened paste specimen at the age of 28d. As can be seen from the curves, there exist endothermic peaks and mass losses at 400–550 °C. According to literature [35], it is at 400–550 °C when $Ca(OH)_2$ decomposes and dehydrates, therefore, we can work out the contents of $Ca(OH)_2$ in the specimens accordingly.

Calculation of $Ca(OH)_2$ content [31]: because the loss in weight of $Ca(OH)_2$ is just the mass of water (ΔG_1) decomposed from $Ca(OH)_2$, as indicated on the TG curve corresponding to the DTA curve between 400 and 550 °C(from the initial temperature to the end temperature of the endothermic peak), thus based on the chemical reaction equation as below:

$$Ca(OH)_2 = CaO + H_2O$$

we can get the mass of $Ca(OH)_2$:

$$\Delta G_2 = 4.115 \times \Delta G_1$$

then the content of $Ca(OH)_2$ should be:

$$CH = \Delta G_2/\Delta G = 4.115 \times \Delta G_1/G$$

where ΔG – the mass of the specimen when heated to 900 °C.

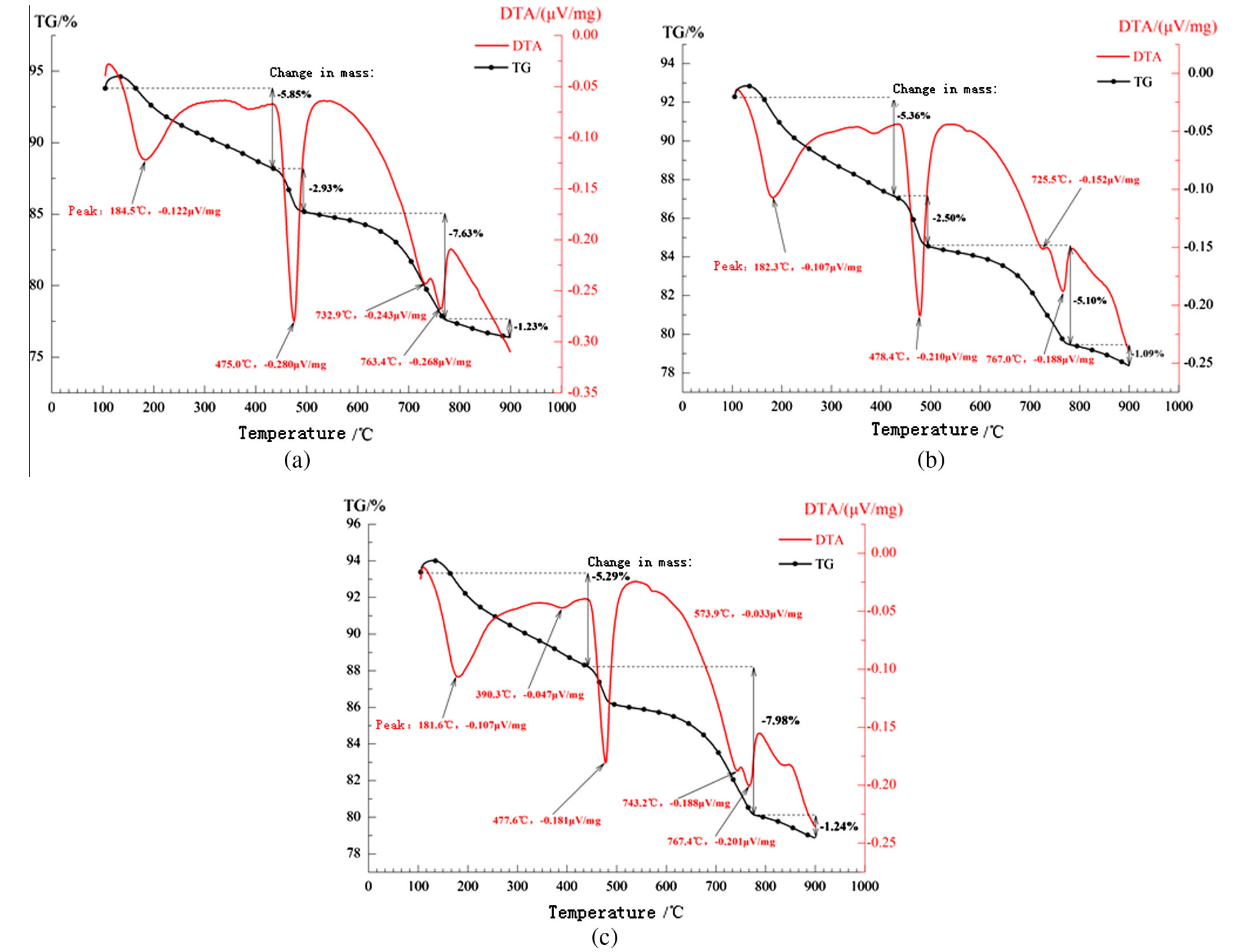

Fig. 6. TG-DTA analysis curve of hardened paste specimens (28 d). (a)—cement paste specimen; (b)—cement–tailings paste specimen (cement substitution rate: 20%); (c)—cement–tailings paste specimen (cement substitution rate: 30%).

The calculated contents of $Ca(OH)_2$ are as showed in Table 7. As can be seen from Table 7, when 20% or 30% of cement in tailings is substituted, the content of $Ca(OH)_2$ in the cement–tailings paste specimen is lower than that in the cement paste specimen, and the content of $Ca(OH)_2$ in the hardened paste specimen with a substitution rate of 30% is lower than that in the hardened paste specimen with a substitution rate of 20%. Thus we can conclude that it is the activity of tailings which causes the secondary hydration reaction, and the higher the substitution rate, the more $Ca(OH)_2$ is consumed during the secondary reaction.

3.2.2. EDS analysis

Several points were selected at random from the SEM diagram of the cement paste specimen (90 d) and cement–tailings paste specimen (90 d, with a substitution rate of 30%) to conduct EDS analysis for C–S–H, as shown in Fig. 7. Fig. 7 shows that in both of the above said paste specimens, Ca, Si, O and Al are all major elements, as manifests that in the two groups of specimens, C–S–H, C–A–H, and Aft or Afm grow in an interlaced way. But the Ca/ Si ratio in the cement–tailings paste specimen is lower than that in the cement paste specimen. It can be inferred from literature [36] that it is tailings which change the composition of C–S–H and result in the secondary hydration reaction.

In addition, mechanochemical activation can decrease the particle size of tailings greatly, so that fine particles may fill among unhydrated cement particles to improve the microstructure and compactness of concrete, thus ensuring the strength of concrete to some extent.

However, the activity of tailings is lower than that of cement after all, at the same time, with the increase in cement substitution rate, the amount of cement clinker decreases correspondingly, and cement hydrates decrease accordingly. This is one of the reasons why the compressive strength of concrete may decline with the cement substitution rate. Meanwhile, with the decrease in the amount of cement hydrates, the amount of $Ca(OH)_2$ involved in the secondary hydration reduces. This is another important reason why the compressive strength of concrete declines with the increase in cement substitution rate. But this research shows that

Table 7
$Ca(OH)_2$ content.

	ΔG_1/mg	ΔG_2/mg	ΔG/mg	CH/%
(a)	1.447	5.954	27.865	21.367
(b)	1.714	7.053	41.842	16.856
(c)	1.403	5.773	38.322	15.064

Note: (a)—cement paste specimen; (b)—cement–tailings paste specimen (cement substitution rate: 20%); (c)—cement–tailings paste specimen (cement substitution rate: 30%).

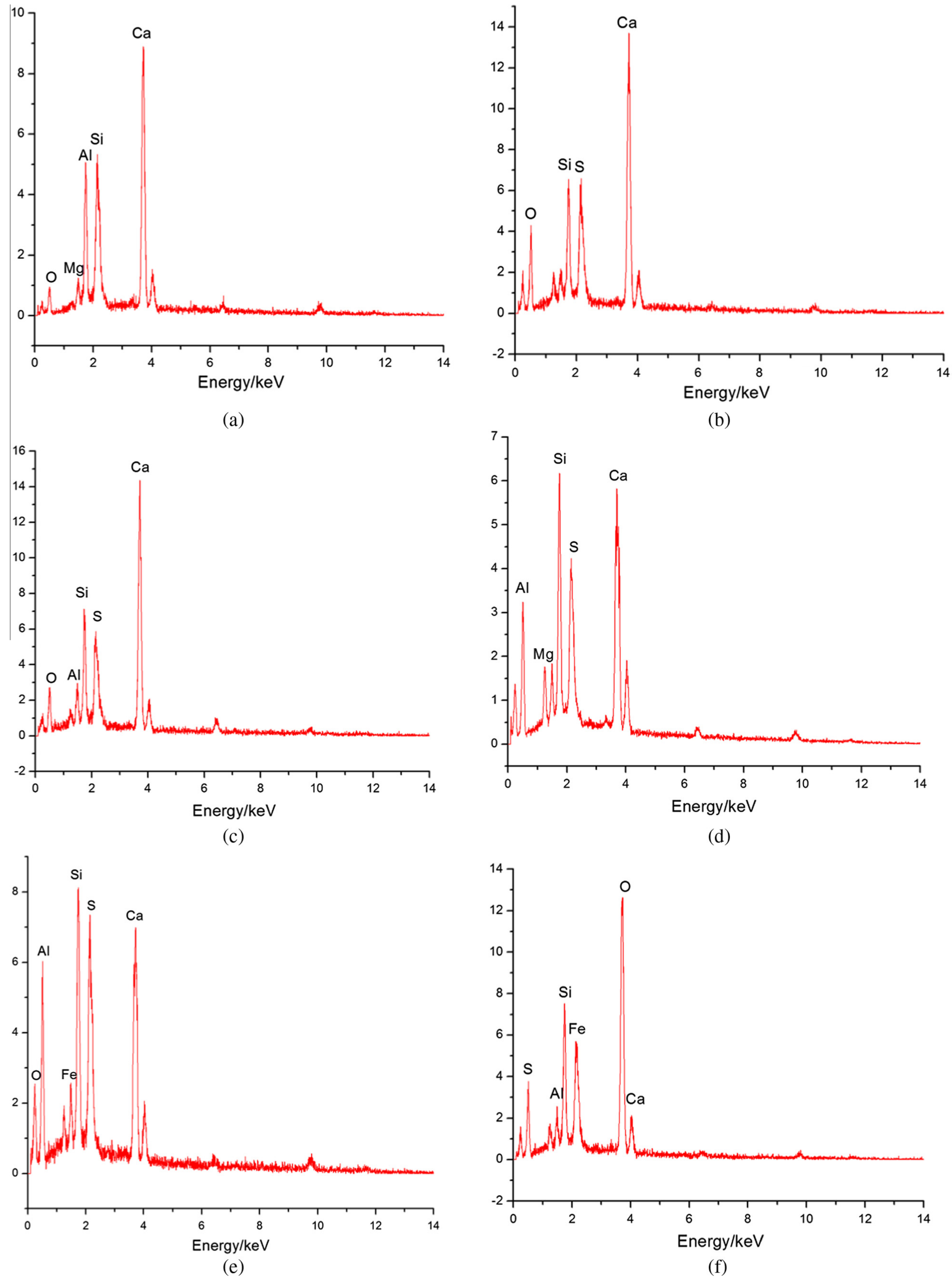

Fig. 7. EDS analysis. (a),(b),(c)—cement paste specimen; (d),(e),(f)—cement-tailings paste specimen.

a appropriate amount of water reducing admixture can increase the cement substitution rate to 40%, thus improving the efficiency of utilization of tailings in concrete works.

4. Conclusions

(1) When cement is substituted by 10%, 20% 30% and 40% of mechanochemically activated tailings respectively, the compressive strength of concrete shows a downward trend with the increase in cement substitution rate.
(2) Without water reducing admixture added, the compressive strength of concrete meets the design requirement when the substitution rates is 10%, 20% and 30% respectively; with a appropriate amount of water reducing admixture added, the compressive strength of concrete meets the design requirement when the substitution rates is 10%, 20%, 30% and 40% respectively.
(3) So far as the compressive strength of concrete concerned, it is feasible to use mechanochemically activated high-silicon iron tailings as a supplementary cementing material to prepare concrete.

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