



## Innovative methodology for comprehensive utilization of iron ore tailings Part 2: The residues after iron recovery from iron ore tailings to prepare cementitious material

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### ABSTRACT

In order to comprehensive utilization of iron ore tailings, this experimental research was to investigate the possibility of using the residues after iron recovery from iron ore tailings as raw materials for the preparation of cementitious material, abbreviated as TSC, including analyses of its mechanical properties, physical properties and hydration products. The TSC1 was prepared by blending 30% the residues, 34% blast-furnace slag, 30% clinker and 6% gypsum. Meanwhile, the raw iron ore tailings (before iron recovery) with the same proportion of TSC1 were selected to compare the cementitious activity of raw tailings and the residues after magnetizing roasting, denoted by TSCO. The hydration products of them were mostly ettringite, calcium hydroxide and C–S–H gel, characterized by XRD, IR and SEM. It was found that ettringite and C–S–H gel were principally responsible for the strength development of TSC mortars with curing time. The results showed that the kaolinite of the tailings was decomposed completely after magnetizing roasting, which promoted the cementitious property of TSC1. Moreover, the mechanical properties of TSC1 are well comparable with those of 42.5 ordinary Portland cement according to Chinese GB175–2007 standard.

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### 1. Introduction

The recovery of iron from iron ore tailings was studied in Part 1 (Innovative methodology for comprehensive utilization of iron ore tailings Part 1: The recovery of iron from iron ore tailings using magnetic separation after magnetizing roasting), and the results indicate that the technology is feasible using magnetizing roasting. But it should be noted that the residues, after the recovery of iron, are up to 70% of total amount of iron ore tailings, and they still face the problem of storage and secondary pollution. Therefore, in order to realize zero-emission of iron ore tailings wastes, we use the residues to prepare cementitious material.

It is well known that various industrial solid wastes, as partial replacement of clinker, have been successfully used in the production of blended cement, which has positive effects on resource conservation, reducing energy consumption and minimizing CO<sub>2</sub> emission [1]. Fly ash, granulated blast-furnace slag and calcined clay are mostly used for blended cement [2–8].

In addition, other industrial wastes are also used for preparing cementitious materials. Zhang et al. [9] investigated the possibility

of incorporating red mud and coal gangue to prepare red mud–coal gangue cementitious material, with 50% red mud–coal gangue mixtures, 24% blast-furnace slag, 20% clinker and 6% gypsum, and found it had a high early stress and the hydration products were mostly ettringite, calcium hydroxide and C–S–H gel.

However, there is only a little information about iron ore tailings in the production of cementitious materials. On one hand, the iron ore tailings are used as admixture in cement. On the other hand, they are used to produce clinker, but the consumption of iron ore tailings is only about 5% [10,11]. In this paper, we use the residues after iron recovery from iron ore tailings, granulated blast-furnace slag, clinker and a small amount of gypsum to prepare a new kind of cementitious material (TSC). The aim of the present study was to investigate the possibility of the residues as raw materials for the preparation of TSC, including analyses of its chemical composition, physical and mechanical properties of hydration products.

### 2. Experiment

#### 2.1. Materials

In optimum conditions mentioned in Part 1, the residues from magnetic separation process were collected for reuse of TSC. The XRD pattern of the residues was shown in Fig. 1. The main mineral phase was quartz. In addition, calcite, mica and a spot of magnetite

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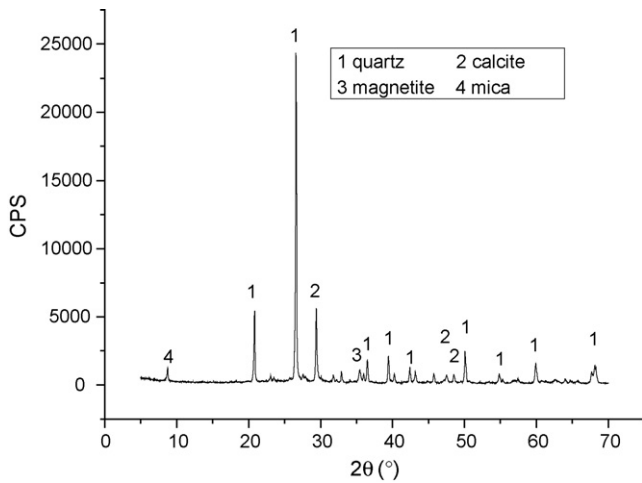


Fig. 1. XRD pattern of the residues.

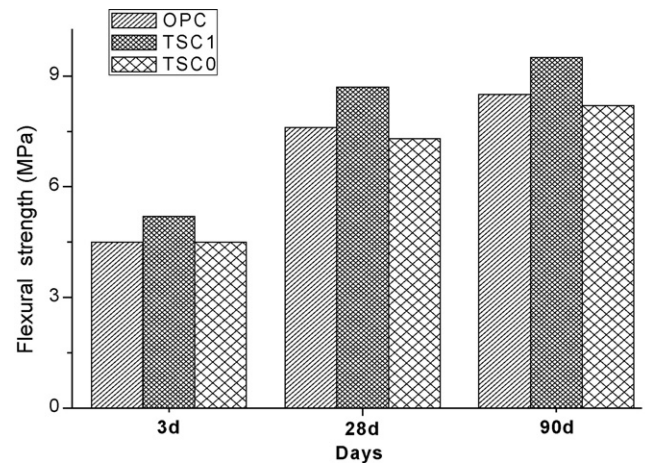


Fig. 2. Flexural strength of TSC comparing with that of OPC (42.5).

Table 1

Chemical composition and physical properties of raw materials.

Oxides (%)	Residues	Slag	Clinker
SiO <sub>2</sub>	63.40	33.59	21.94
Al <sub>2</sub> O <sub>3</sub>	9.92	14.37	5.27
CaO	13.29	38.32	66.09
Fe <sub>2</sub> O <sub>3</sub>	6.09	1.11	3.84
Na <sub>2</sub> O	0.32	0.18	0.3
K <sub>2</sub> O	1.23	0.11	0.7
P <sub>2</sub> O <sub>5</sub>	2.31	–	–
MgO	1.50	8.43	0.88
TiO <sub>2</sub>	0.31	0.85	–
SO <sub>3</sub>	0.85	2.26	0.31
LOI	0.78	0.44	0.67
Specific gravity	2.65	2.78	3.16
Specific surface, blaine (m <sup>2</sup> /kg)	420	375	400

were also existed. Granulated blast-furnace slag was supplied by Tangshan steel refining plant, with 0.080 mm sieve residue 5.0% and Blaine's specific surface area of 375 m<sup>2</sup>/kg. A sample of clinker was employed for this investigation from Beijing Xingang. The chemical compositions and physical properties of raw materials were shown in Table 1.

## 2.2. Preparation of TSC

The residues after iron recovery of the tailings were blended with granulated blast-furnace slag, clinker and gypsum, in appropriate proportions to prepare TSC. The designed proportion of TSC1 is listed in Table 2. Meanwhile, the raw iron ore tailings (before iron recovery) with the same proportion of TSC1 were selected to compare the cementitious activity of raw tailings and the residues after magnetizing roasting, denoted by TSC0.

The TSC pastes with water to solids ratio of 0.3 were prepared and molded in 20.0 mm × 20.0 mm × 20.0 mm test cubes that were cured in a moist cabinet at 95% humidity and 20 °C. After one day of hydration, the pastes were demoulded and transferred to isothermal curing cabinet at the previously mentioned humidity and temperature until mechanical testing was performed. The hydration of the pulverized and sieved specimens was terminated

Table 2

Designed proportion of TSC1 (%).

Residues	Slag	Clinker	Gypsum
30	34	30	6

by alcohol drenching at the desired testing ages, then dried at 80 °C in a vacuum oven for 24 h for further characterization.

## 2.3. Testing conditions

- (1) The chemical analysis was performed with the X-ray fluorescence (XRF-1700) analyzer.
- (2) Water requirement of normal consistency and setting time of TSC pastes were determined by Vicat probe and Vicat needle apparatus according to Chinese Standard GB/T 1346-2001 [12], as well as the expansion of cement paste was tested by Le Chatelier method.
- (3) Fluidity of TSC mortar with a cement/sand/water ratio (by weight) of 1:3:0.5 was measured in accordance with Chinese Standard GB/T 2419-2005 [13].
- (4) Mechanical tests were carried out according to Chinese Standard GB/T17671-1999 [14], and the mix ratio (by weight) for these tests was also 1:3:0.5 cement/sand/water. Mortar specimens in size of 40.0 mm × 40.0 mm × 160.0 mm were cured in a moist cabinet at 95% humidity and 20 °C for 24 h, and then demoulded and placed in the isothermal curing cabinet at the previously mentioned humidity and temperature until mechanical testing (3, 28, and 90 days of curing) was performed.
- (5) XRD analysis was conducted by Rigaku D/max-RB X-ray diffractometer with Cu-Kα radiation, voltage 40 kV, current 200 mA and at the scanning rate of 8°/min from 5° to 70°.
- (6) FTIR (Spectrum GX, PE) was used to analyze the chemical structure of hydrated products.
- (7) The morphology and microstructure of hydrated products were investigated using scanning electron microscope (JSM-6460LV SEM).

## 3. Results and discussion

### 3.1. Mechanical and physical properties of TSC

In order to compare the mechanical properties of TSC, a 42.5 ordinary Portland cement (OPC) in accordance with Chinese Standard GB175-2007 was selected as a reference in the test of strength development.

The flexural and compressive strength results of TSC1, TSC0 mortars comparing with OPC are presented in Figs. 2 and 3, respectively. For all samples, the flexural and compressive strength values increased with curing time. Additionally, both the values decreased with increasing residues percentage in TSC, regardless of curing

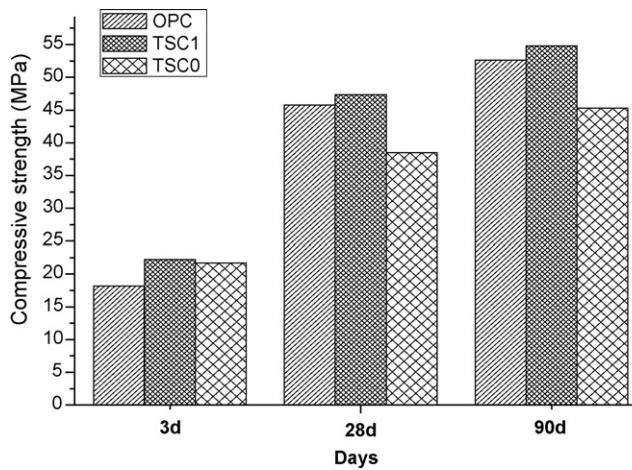


Fig. 3. Compressive strength of TSC comparing with that of OPC (42.5).

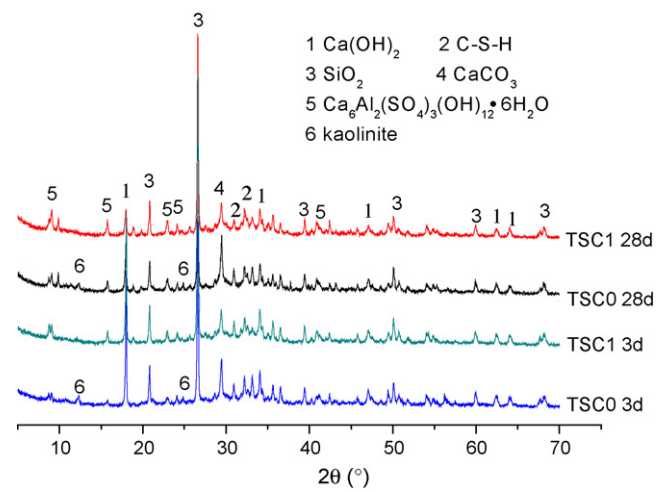


Fig. 4. XRD patterns of hydrated TSC pastes at different curing ages.

time. According to the results, both the flexural and compressive strength values of the TSC1 mortars at 3, 28 and 90 days were higher than those of the reference OPC. On the other hand, TSC0 mortars yielded lower values than those of OPC. This behavior is attributed to the moderated pozzolanic activity of the tailings. In addition, from the difference of strength development between TSC1 and TSC0, it is noted that magnetizing roasting is significant to promote its cementitious activity.

As a result, for these three types of cementitious materials studied, the mortars prepared with TSC1 meet the requirements of the Chinese GB175-2007 standard in terms of the flexural and compressive strength.

The general mechanical and physical properties of TSC1 are presented in Table 3.

From Table 3, it is noted that the setting time of TSC1 satisfied the requirement in Chinese Standard GB 175-2007 for ordinary Portland cement (initial setting time  $\geq 45$  min, final setting time  $\leq 600$  min). In addition, water demand for normal consistency of TSC1 was higher than OPC, which is in a range of 25–28% [15,16]. It could be mainly due to the large specific surface of residues. According to the Le Chatelier method, the expansion was well below the maximum accepted value of 5 mm.

It can be inferred from the mechanical and physical properties that it is feasible to use the residues after iron recovery from iron ore tailings together with granulated blast-furnace slag to prepare cementitious material, and the amount of the residues can be used up to 30% of raw materials. According to this technology, the residues of tailings can be utilized significantly. Moreover, this environmental friendly cementitious material can also save a lot of natural resources, such as limestone, and finally decrease the production cost of cement.

### 3.2. XRD analysis of the TSC pastes

The XRD patterns of the TSC pastes hydrated at 3 and 28 days are presented in Fig. 4. It can be seen that the diffraction peaks of ettringite ( $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}$ ),  $\text{Ca}(\text{OH})_2$  and C–S–H phases

appeared in all samples obtained during hydration. The main obvious change between different hydration ages is the peaks of  $\text{Ca}(\text{OH})_2$  diminished, whereas those of ettringite slightly increased. In addition, the kaolinite still existed in TSC0 pastes, but did not appear in TSC1 pastes.

At later ages, C–S–H gel and the stable ettringite are the major hydration products. Konsta-Gdoutos et al. [17] reported that the remaining unreacted crystalline  $\text{Ca}(\text{OH})_2$  may show a slight decrease on the strength development. It can be deduced that C–S–H gel and ettringite make contribution to the strength development with curing time of TSC mortars. This is in agreement with the results of Zhang et al. [9] from studies in red mud–coal gangue cementitious material.

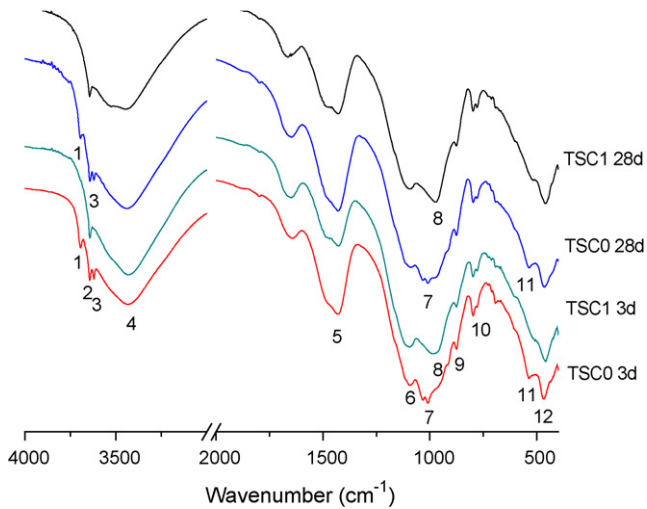
In comparison with the patterns of 3 days and 28 days curing time of TSC1 and TSC0, respectively, both of the ettringite peaks of TSC0 are lower than TSC1, which illustrated that the strength of TSC1 mortars were higher than TSC0. It should be noted that, as mentioned in Part 1, the kaolinite of tailings after magnetizing roasting at  $800^\circ\text{C}$  was decomposed completely, while existed in the raw iron ore tailings. Many researches have been carried out investigating the phase transformation of calcite kaolinite [18–20], and after dehydroxylation of kaolinite the amorphous metakaolin generated, which increased its pozzolanic activity. Murat [21] and De Silva and Glasser [22] reported that during the hydration process, ettringite was firstly generated by metakaolin,  $\text{Ca}(\text{OH})_2$  and gypsum. So, it confirms that the higher strength of TSC1 mortars due to more ettringite generated in the hydration process than TSC0.

Meanwhile, crystallized minerals in all of the hardened pastes, such as quartz ( $\text{SiO}_2$ ) and calcite ( $\text{CaCO}_3$ ), typically associated with the residues of tailings and considered as nonreactive were observed. It can be inferred that the cementitious properties of residues at the process of magnetizing roasting were not promoting adequately, especially for the inactive  $\text{SiO}_2$ . So, our next research focus is on how to improve the cementitious activity of quartz effectively in the process of roasting.

Table 3  
Mechanical and physical properties of TSC1 (%).

Sample	Specific surface, blaine ( $\text{m}^2/\text{kg}$ )	Setting time (min)		Water of normal consistency (%)	Expansion (mm) (Le Chatelier)	Compressive strength (MPa)	
		Initial	Final			3 days	28 days
TSC1	397	154	247	30	1.6	22.2	46.9





**Fig. 5.** Infrared spectra of TSC pastes after 3 days and 28 days curing (1 = 3694  $\text{cm}^{-1}$ , 2 = 3645  $\text{cm}^{-1}$ , 3 = 3620  $\text{cm}^{-1}$ , 4 = 3434  $\text{cm}^{-1}$ , 5 = 1428  $\text{cm}^{-1}$ , 6 = 1096  $\text{cm}^{-1}$ , 7 = 1009  $\text{cm}^{-1}$ , 8 = 972  $\text{cm}^{-1}$ , 9 = 878  $\text{cm}^{-1}$ , 10 = 800  $\text{cm}^{-1}$ , 11 = 540  $\text{cm}^{-1}$ , 12 = 468  $\text{cm}^{-1}$ ).

### 3.3. IR analysis of the TSC pastes

The IR patterns of hydration products of TSC pastes after 3 days and 28 days curing time are shown in Fig. 5. It is clear that, in TSC0 3 days and 28 days patterns, the bands at 3694  $\text{cm}^{-1}$ , 3620  $\text{cm}^{-1}$ , 1009  $\text{cm}^{-1}$ , and 540  $\text{cm}^{-1}$  are due to kaolinite. In all of the patterns, the band at 3645  $\text{cm}^{-1}$  which is due to the stretch vibration of OH group of  $\text{Ca}(\text{OH})_2$  [23] decreases with the progress of hydra-

tion. The peak at 3434  $\text{cm}^{-1}$  is due to water [24], and the bands at 1428  $\text{cm}^{-1}$  and 878  $\text{cm}^{-1}$  are attributed to presence of  $\nu_3\text{-CO}_3^{2-}$  and  $\nu_2\text{-CO}_3^{2-}$  of calcite, respectively [25]. Meanwhile, the IR frequencies at 1096  $\text{cm}^{-1}$ , 800  $\text{cm}^{-1}$  and 468  $\text{cm}^{-1}$  are typical bands, assigned to quartz. The results are in harmony with XRD analysis. It should be noted that the band at 972  $\text{cm}^{-1}$  region is due to Si–O stretching vibration of C–S–H [23], and increases with the stage of curing indicating increasing the formation of C–S–H gel.

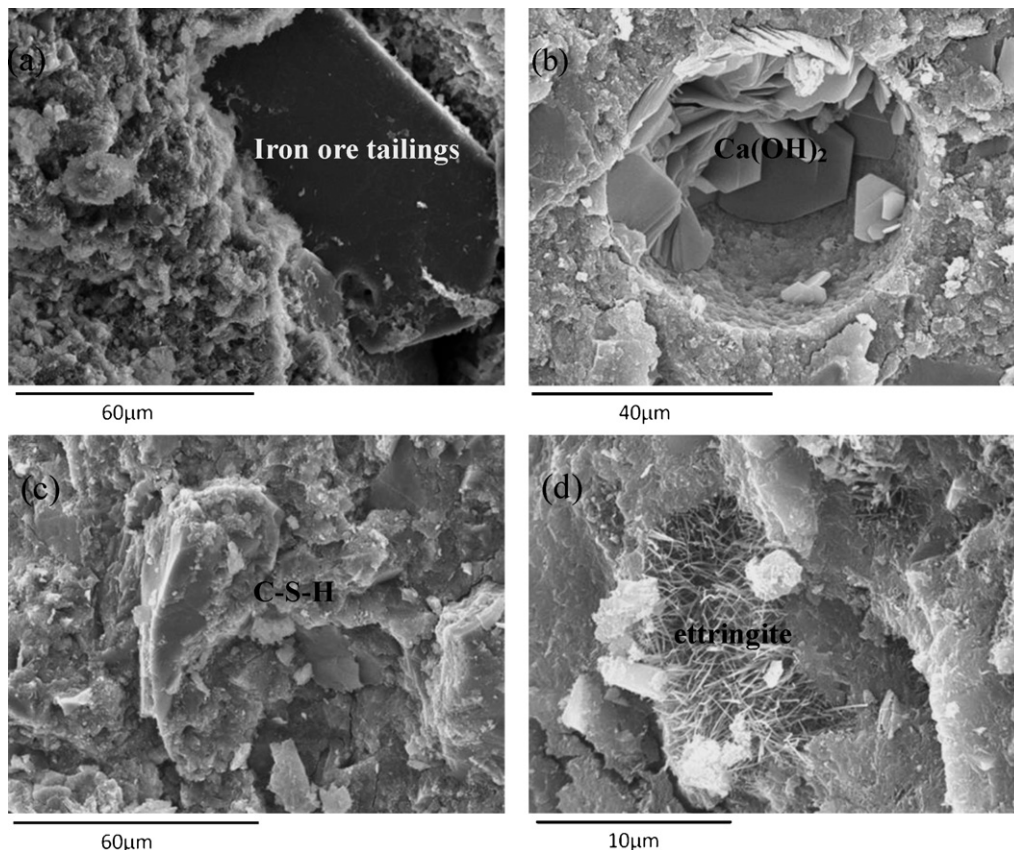
### 3.4. Morphology and microstructure of the TSC pastes

The microstructures of the TSC0 and TSC1 pastes hydrated at 3 and 28 days are shown in Figs. 6 and 7. In Fig. 6(a), there was an obvious particle of unhydrated iron ore tailings. However, at the age of 28 days, amorphous C–S–H gel coated on the surface of the unhydrated grain, seen in Fig. 6(c). Therefore, it can be inferred that the inactive iron ore tailings were filled in the hydrated products as inert aggregates, which influenced the strength value. At the same time, crystallized  $\text{Ca}(\text{OH})_2$  and rod-like ettringite phase can be found in Fig. 6(b) and (d).

In the hydration progress of TSC1 pastes, there were many ettringite and C–S–H gel generated at 3 days curing, seen in Fig. 7(a). The microstructure of rod-like ettringite and amorphous C–S–H gel are shown in Fig. 7(b) and (d). From Fig. 7(c), we can see the hydrated product was presented a more dense structure and lower porosity than Fig. 6(c).

### 3.5. Discussion

In this study, it is found that TSC1 made by the residues after iron recovery from iron ore tailings, blast-furnace slag, gypsum and



**Fig. 6.** Microstructure of the hydrated TSC0 pastes at different curing ages: (a) and (b) 3 days; (c) and (d) 28 days.

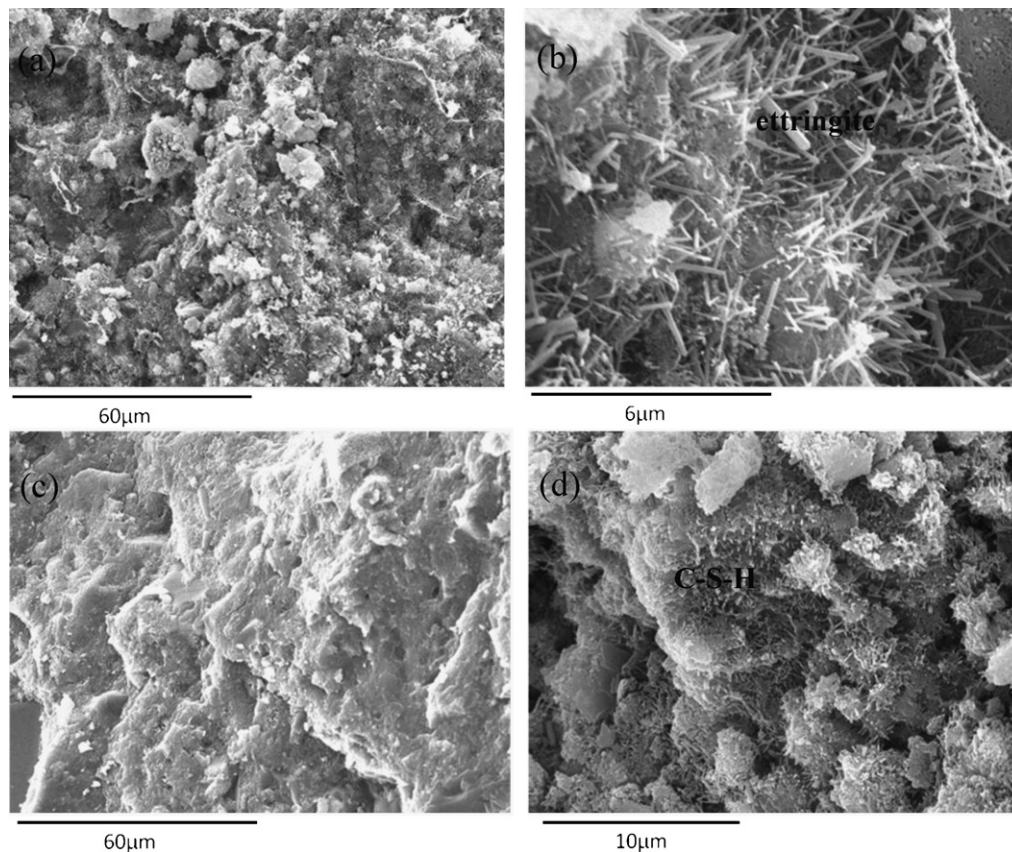


Fig. 7. Microstructure of the hydrated TSC1 pastes at different curing ages: (a) and (b) 3 days; (c) and (d) 28 days.

clinker meet the requirements of the Chinese GB175-2007 standard.

A large part of the investigations about blast-furnace slag have been carried out, known as “alkali-slag cement” or “alkali-activated slag cement” [26–29]. Blast-furnace slag is a by-product of iron production industry, having a high content of calcium. Being a low performance cementitious material, it can achieve high compression strength when an alkaline activator is used. The most used alkaline activators are sodium or potassium hydroxide and water glass, and the main reaction product of alkali activation of blast-furnace slag is C–S–H gel confirmed by Wang and Scrivener [27]. But in this current work, it is quite different from traditional alkali-activated slag cement. In TSC material, the clinker not only supports the strength development in curing progress, but also can be regarded as activator of slag. Because of its low alkalinity and combination with sulfates provided by gypsum, the hydration products are mainly ettringite and C–S–H gel, which is also confirmed by Zhao et al. [3].

However, an important problem is put forward: what is the role of the residues of iron ore tailings in this cementitious material? By contrast with the strength development and hydration property of TSC1 and TSC0, the clay minerals in the tailings, such as kaolinite, have positive influence on the strength development after magnetizing roasting. Other components, especially quartz, are unhydrated in the progress of curing, which can be seen from SEM images, and negatively influence the strength. So, in order to promote the residues' activity, we need to do more to improve the quartz's activity in the process of magnetizing roasting.

The physical and mechanical properties confirm the usefulness of TSC, which indicates that it is feasible to use the residues after iron recovery from iron ore tailings to replace up to 30% of the raw

materials to produce cementitious material, which is much more than used as admixture or producing clinker [10,11]. Combined with Part 1, the whole technology of comprehensive utilization is scientific and feasible. Not only have good economic benefit (iron recovery), but also the iron ore tailings can be utilized significantly (the amount is 30%).

This technology is supported by other researchers. Liu et al. [30] use Bayer red mud for iron recovery and building material production from aluminosilicate residues. In their research, the iron recovery rate is 81.40% and the compressive strength of cured brick samples achieved the maximum peak of about 24.10 MPa at the content of hydrated lime of 13%. As a result, this kind of wastes can be comprehensive utilized by this technology and realize the goal of zero-emission.

#### 4. Conclusions

In this study, the chemical composition, mechanical and physical properties and hydration products of TSC were investigated. From the studies carried out, the following conclusions can be drawn:

- (1) The mechanical and physical properties confirm the usefulness of TSC1, which indicates that it is feasible to use the residues after iron recovery from iron ore tailings to replace up to 30% of the raw materials to produce cementitious material, and the mechanical properties of TSC1 are well comparable with those of 42.5 ordinary Portland cement according to Chinese GB175-2007 standard. Meanwhile, possessing important environmental and economic significances, the produced TSC1 cannot only consume large quantities of iron ore tailings, but also save a lot of nature resources for the cement clinker, such

as limestone and iron source in particular, and finally decrease the production cost of cement.

- (2) The hydration products of TSC are mostly calcium hydroxide, ettringite and C–S–H gel. Calcium hydroxide decreases with the curing age, whereas those of ettringite slightly increased. C–S–H gel and ettringite make contribution to the strength development with curing time of TSC pastes.
- (3) By comparing the strength and development and hydration property of TSC1 and TSC0, the clay minerals in the tailings, such as kaolinite, have positive influence on the strength development after magnetizing roasting. This is due to more ettringite formed from  $\text{Ca}(\text{OH})_2$ , metakaolin (generated by dehydroxylation of kaolinite at magnetizing roasting) and gypsum, whereas those of kaolinite in TSC0 was unhydrated in the progress of curing.
- (4) Combined with Part 1, the comprehensive utilization of iron ore tailings is scientific and feasible. Using this technology, it cannot only realize zero-emission of iron ore tailings wastes, save a lot of nature resources, but also create maximum economic benefit.

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