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Preparation of alinite cement from municipal solid waste incineration fly ash

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ABSTRACT

The feasibility of partially substituting ordinary raw materials with municipal solid waste incineration (MSWI) fly ash in alinite cement production was investigated by X-ray diffraction (XRD), X-ray fluorescence spectrometry (XRF) and scanning electron microscopy (SEM). The physical properties and leaching behavior of the produced cement were also evaluated. Experimental results show that good quality clinkers can be obtained by firing the raw mixes, in which the replacement of MSWI fly ash reaches to 30%, at 1200 °C for 2 h. Alinite cements have higher early strengths at all gypsum additions, while the best result having acceptable early and 28-day strengths is obtained at 5% of gypsum addition. Results also show that the leaching toxicity of heavy metals is far lower than that of the regulatory limit at all testing ages. Based on this study, MSWI fly ash is viable as an effective, alternative raw material in alinite cement production.

1. Introduction

All over the world large amounts of MSWI fly ash are produced from municipal solid waste incineration plants every year. In China, the output of MSWI fly ash has been over 300 thousand tons per year and is increasing year by year [1]. Due to the high levels of dioxins, soluble salts and heavy metals present in MSWI fly ash, fly ash is classified as hazardous waste in China and its disposal may pose a significant risk to the environment [2–4]. Over the past three decades, although the stabilization technology for MSWI fly ash has advanced markedly [5–7], certain problems with conventional stabilization technologies cannot be ignored, such as long-term stability and the cost of stabilization chemicals. Therefore the development of a more reliable and economical technique to treat MSWI fly ash is urgently needed.

Recently, reusing of municipal wastes as raw materials in cement manufacturing has been investigated substantially [8–12]. There are advantages of using a cement kiln for MWSI fly ash treatment. The high temperature and high retention time in cement kilns will effectively destroy the dioxins present in the ash, and capture most of heavy metals in the product [13–15]. Therefore, production of different types of cement clinker is one promising technique to treat MSWI fly ash reliably and economically, especially in the production of chlorine bearing alinite cement [16–18].

Alinite has a crystal structure similar to alite in which oxygen ions are partially substituted by chlorine and silica by alumina, and it can be represented by the general formula: $Ca_{10}Mg_{1-x/2}V_{x/2}[(SiO_4)_{3+x}(AlO_4)_{1-x}]O_2Cl$ (V: a lattice vacancy, $0.35 < x < 0.45$) [19–21]. The formation of alinite phase is promoted by chloride ions, MgO addition, and low alkali content [22]. The clinkering temperature for alinite was reported to be as low as 1100–1200 °C, and the energy consumption for grinding alinite cement is lower than ordinary Portland cement (OPC) [23,24]. Alinite cement requires a lower w/c ratio than OPC, and it set more quickly [25]. Additionally, the hydration products of alinite have a capability to fix heavy metals in the crystal lattice, which can be used to immobilize hazardous waste materials [16,21,26]. The work done by Pradip et al. [27] showed the possibility of producing good quality alinite cements from industry/mining wastes such as waste materials from an integrated steel plant and gold tailing sands.

The chemical composition analysis indicates that MSWI fly ash contains a certain amount of heavy metals, which are known to have remarkable effects on the clinkering process [18,28,29]. Thus it is worth investigating the effect of MSWI fly ash content on the phase formation of alinite cement clinker. Additionally, the composition analysis of raw materials also shows the presence of magnesium salts. It is reported that MgO plays an important role in the alinite formation as well as stabilization [20,22]. The higher the MgO and chloride content, the more is the alinite formation,

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Abbreviations: MSWI, municipal solid waste incineration; XRD, X-ray diffraction; XRF, X-ray fluorescence spectrometry; SEM, scanning electron microscopy; EDS, energy disperse spectroscopy; ICP-AES, inductively coupled plasma atomic emission spectrophotometer; *KH*, lime saturation moduli; *a*, silica-alumina ratio; *c*, silica-chloride ratio.

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and the magnesium salts when mixed with $CaCl_2$ are more effective as a mineralizer than $CaCl_2$ alone [30]. However, when a high MgO content is present in the raw mix, some other phases tend to form with a corresponding reduction in the amount of CaO, SiO_2 and MgO available for the formation of alinite [17]. Consequently, the right amount of MgO in the raw mix needs to be determined.

This paper describes the feasibility of partially substituting ordinary raw materials with MSWI fly ash in alinite cement production. In order to control the quality of the fired clinkers, certain Cement Moduli (namely lime saturation moduli, Silica–alumina ratio, silica–chloride ratio) were defined. Based on that, the effects of different process parameters like composition of raw mix and firing temperature on the phase formation were investigated. The alinite cement obtained from the optimized clinker was subjected to compressive strength, setting time and water demand for normal consistency tests in order to evaluate its physical properties. Additionally, to assess the impact of the produced cement on environment, the heavy metal contents trapped in the clinkers and the leaching toxicity of hydrated alinite cement were also evaluated.

2. Materials and methods

2.1. Materials

The MSWI fly ash was originated from Suzhou Wastes Incineration Plant in China. A class P.O42.5 Portland Cement from Anhui (province in PR china) Conch Cement Co., Ltd. was used in this study. The chemical compositions of MSWI fly ash and limestone are given in Table 1. The XRD study on the MSWI fly ash sample (Fig. 1) indicates that it is comprised of a mixture of predominantly Ca-rich compounds, amorphous glass and silicates. The fly ash is also rich in chloride compounds (NaCl, KCl, CaClOH). CaClOH is an intermediate phase formed during the absorption of HCl. The presence of $CaCO_3$ may have been caused by partial, earlier weathering of the ash [1]. Additionally, Reagent grade chemicals, $CaCl_2$, $CaSO_4{\cdot}2H_2O$, SiO_2, Al_2O_3 and MgO, were also used in this work. All the materials were ground and sieved through a 200-mesh sieve.

2.2. Raw mix preparation

The Cement Moduli employed in the investigation were defined as follows (Eqs. (1)-(3)) [31]. Typically, the parameters are controlled at *KH* value around 1.0, *a* value around 4.5 and *c* value around 2 [31]. To investigate the effects of MSWI fly ash and MgO on the phase formation, nine sets of compositions were prepared and fired at 1200 °C for up to 2 h. The effects of firing temperature (1050–1250 °C) were studied on the representative sample of the nine sets. Table 2 gives the compositions of these raw mixes along with the parameters, where *m* is the total amount of MgO (wt.%) in the raw mixture.

$$\text{Lime saturation moduli}(KH), KH = \frac{\text{CaO(wt.\%)} - 0.7\text{Fe}_2\text{O}_3\text{(wt.\%)}}{2.608\text{SiO}_2\text{(wt.\%)}} \quad (1)$$

$$\text{Silica—alumina ratio}(a), a = \frac{\text{SiO}_2\text{(wt.\%)}}{\text{Al}_2\text{O}_3\text{(wt.\%)}} \quad (2)$$

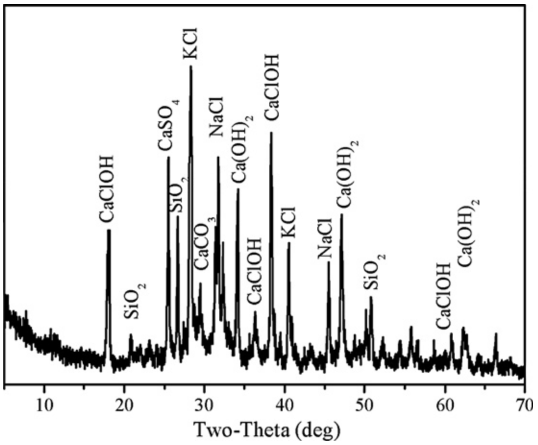


Fig. 1. XRD pattern of MSWI fly ash.

$$\text{Silica—chloride ratio}(c), c = \frac{\text{SiO}_2\text{(wt.\%)}}{\text{CaCl}_2\text{(wt.\%)}} \quad (3)$$

The mix proportions were prepared as follows: 30 g of the raw materials in predetermined proportions were taken and then mixed in isopropyl alcohol thoroughly. The resultant mixtures were dried in an oven at 65 °C for 24 h, then pressed and moulded into cylindrical samples of $\Phi 30 \times 5\ \text{mm}^3$. The cylinders were fired in a Nabertherm furnace at different temperature for up to 2 h and an average heating rate of about 30 °C/min was employed. Finally, the clinkers so prepared were cooled at ambient temperature and then ground to a fineness of about 400 m^2/kg Blaine surface area for future analysis.

2.3. Testing methods

The phases formed on firing of the raw mix were determined by X-ray diffraction (XRD) technique using Cu Kα radiation at 40 kV and 25 mA. The chemical composition of the prepared clinker was determined by X-ray fluorescence spectrometry (XRF). The morphology of the clinker was performed by scanning electron microscopy (SEM).

In order to study the effect of gypsum on the physical properties, the setting time and water demand tests were performed according to Chinese National Standard GB/T 1346-2001, and the compressive strength tests were carried out as follows: the optimized clinkers were interground with appropriate amount of gypsum to make alinite cement. The cement samples were mixed with water with W/C ratio of 0.3 and the paste was put into 10 mm × 10 mm × 10 mm cube moulds with vibration. The molded pastes were kept at 20 ± 2 °C and relative humidity exceeding 90% for 24 h, and then removed from the molds. The demoulded samples were cured in a saturated lime water at 20 ± 2 °C for 1, 3, 7 and 28 days and then tested for compressive strength.

The heavy metals concentration in the clinker made from the optimized composition was determined by inductively coupled plasma atomic emission spectrophotometer (ICP-AES). To prepare the solution for ICP-AES analysis [13], approximately 0.5 g clinker was successively digested by HNO_3, $HClO_4$ and HF acids in a Teflon beaker and on a hot plate. The final volume of the solution

Table 1
Chemical composition of raw materials (by wt.%).

	CaO	SiO_2	$CaCl_2$	SO_3	Al_2O_3	K_2O	Na_2O	Fe_2O_3	MgO	LOI
MSWI FA	29.8	13.9	10.8	6.8	3.7	3.5	3.2	1.5	1.5	25.2
Limestone	55.3	0.7	–	–	0.1	–	–	0.2	1.8	41.9

Table 2
Composition (by wt.%) of the raw mix (Set 1: A1–A5).

	MSWI fly ash	Limestone	SiO_2	Al_2O_3	$CaCl_2$	MgO	Cement Moduli			
							KH	*a*	*c*	*m*
A1	0.00	73.50	15.00	3.43	7.84	0.10	1.02	4.5	2.0	1.4
A2	15.22	63.18	12.42	2.74	6.27	0.00	1.02	4.5	2.0	1.4
A3	30.33	52.87	9.90	2.06	4.38	0.00	1.02	4.5	2.0	1.4
A4	45.28	42.84	7.30	1.37	2.44	0.00	1.02	4.6	2.0	1.4
A5	60.26	32.48	4.86	0.73	0.65	0.00	1.01	4.5	2.0	1.5
B1	30.33	52.87	9.90	2.06	4.38	0.00	1.02	4.5	2.0	1.4
B2	30.31	52.33	9.80	2.04	4.34	1.00	1.02	4.5	2.0	2.4
B3	30.24	51.82	9.70	2.02	4.3	2.10	1.02	4.5	2.0	3.4
B4	30.26	52.26	9.59	2.00	4.23	3.10	1.03	4.5	2.0	4.4

obtained after dissolution of the residue in 10 mL of concentrated HNO_3 was made up to 100 mL with distilled water. For analysis by ICP-AES, the solutions were again diluted 50 times. The weight percentage of the heavy metals in the clinkers was deduced from the concentration obtained. The toxicity leaching characteristics of heavy metals present in the clinkers were studied through leaching test. The leachate was prepared based on Chinese National Standard HJ 557-2009 (solid waste-extraction procedure for toxicity of solid waste-horizontal vibration method). The hydrated samples were crushed until the maximum size of particle was below 5 mm. After that, 30 g coarse grains were weighed accurately and then placed into a 600-mL-lidded polythene bottle containing 300 mL of distilled water. The suspension was fixed onto a horizontal vibrator and shaken at the frequency of 130 ± 4 times per min with an amplitude of 40 mm. Finally, the solution was filtrated with a medium-speed quantitative filter paper. The concentrations in the filtrate (pH adjusted to 5.8–6.3 with sodium hydroxide or hydrochloric acid) were determined by ICP-AES.

3. Results and discussion

3.1. Effect of composition

Five samples (A1-A5) were prepared to study the effect of MSWI fly ash on the formation of the clinker minerals. XRD patterns of the sintered clinkers (A1–A5) are shown in Fig. 2. It is seen that A1 has the structure of a typical alinite cement clinker. The dominant phases (alinite and β-C_2S) along with some minor phases $C_{11}A_7{\cdot}CaCl_2$, f-MgO, f-CaO were well crystallized in clinker A1, giving peaks at the corresponding 2θ values. With different replacement of MSWI fly ash, from 15% to 60%, the phases formed in the clinkers are the same as that of A1, and no detection of new phases is recorded. With an increase in MSWI fly ash content up to 30%, the amount of alinite and β-C_2S produced remains constant. However, the intensity of the alinite peak decreases rapidly when the amount of MSWI fly ash increased from 30% to 60%. The fall in the formation of alinite might be attributed to the joint effect exerted by heavy metals, anions and alkalis present in the raw mix, and this effect might be presented as an adverse one when the MSWI fly ash content exceeds a certain value. Therefore, for the investigated MSWI fly ash samples the maximum allowable content for the mixtures to achieve desirable clinkers is 30 wt.%, and composition A3 was identified as the most promising one for further studies.

Fig. 3 shows the XRD patterns of the products obtained from B1–B4 compositions. The XRD study of the sample B1 shows the formation of alinite and β-C_2S as the major phase and $C_{11}A_7{\cdot}CaCl_2$, f-CaO as the minor phases. With an increase in *m* (from 1.4 to 4.4) by adding extra amount of MgO, there is a slight increase in the formation of alinite, a decrease in the formation of β-C_2S, but a significant increase in the formation of f-MgO. It is generally known that f-MgO has an adverse effect on the soundness of the produced cement. Therefore, the optimum amount of MgO in the raw mix is about 1.4%. This value is slightly less than that required for the pure alinite, and the possible reason for this could be due to the partial replacement of MgO by other oxides (such as ZnO) that are present in the MSWI fly ash [17].

From the experiments carried out, it is concluded that alinite cement clinkers can be successfully prepared by controlling the Cement Moduli at *KH* value around 1.0, *a* value around 4.5, *c* value around 2 and *m* value around 1.4, and the replacement could be taken up to 30% of MSWI fly ash in the raw mix.

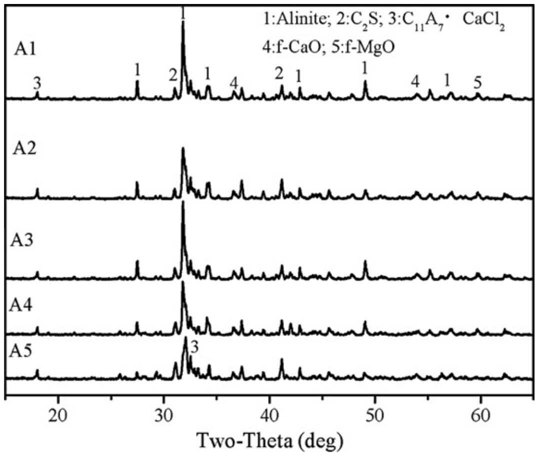


Fig. 2. XRD patterns of alinite cement clinkers (A1–A5) fired at 1200 °C for 2 h.

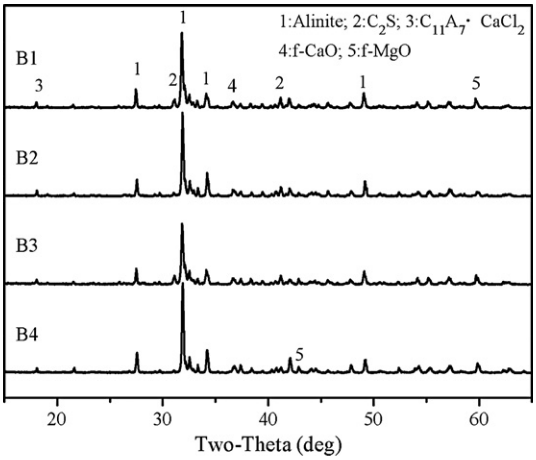


Fig. 3. XRD patterns of alinite cement clinkers (B1–B4) fired at 1200 °C for 2 h.

3.2. Effect of firing temperature

To study the effect of firing temperature on the quality of the produced clinker, the selected composition A3 was fired at different temperature (1050 °C, 1100 °C, 1150 °C, 1200 °C and 1250 °C) for 2 h. XRD patterns of the fired samples are shown in Fig. 4. It is seen that the formation of $C_{11}A_7{\cdot}CaCl_2$ is not dependent upon the temperature in range of 1050–1250 °C. The effect of firing temperature is noticed in the relative proportion of alinite and β-C_2S. The formation of alinite increases with increase in the firing temperature (from 1050 °C to 1250 °C), with a corresponding decrease in the formation of β-C_2S.

This work shows that good quality clinker can be fired at a temperature range of 1200–1250 °C. These results, however, are not in agreement with those of Chang et al. [32] who found that the optimum condition for the formation of alinite is 1375 °C for 2 h. The difference for this may be attributed to the higher amount of minor elements in this work, which promote the formation of clinker compounds and lower the clinkering temperature.

3.3. XRF analysis

The chemical composition of clinker A3 is given in Table 3. Based on the XRF results, the quantity of clinker minerals can be estimated by using Bogue formulas: Alinite = 83.4%, β-C_2S = 9.1%, $C_{11}A_7{\cdot}CaCl_2$ = 3.7%.

3.4. SEM–EDS analysis of clinkers

The SEM photos of A3 sintered at 1200 °C for 2 h are shown in Fig. 5. The clinker appeared as irregular particles (Fig. 5a and b) [33]. Since alinite was the major phase in clinker A3, Alinite could be observed by Energy Disperse Spectroscopy (EDS) in Fig. 5c.

From Fig. 5c and Table 4, it is seen that the real value of composition of alinite is very close to the theoretic value, and the average proportion is $Ca_{9.34}Mg_{0.9}(SiO_4)_{3.71}(AlO_4)_{0.72}O_{0.24}Cl$.

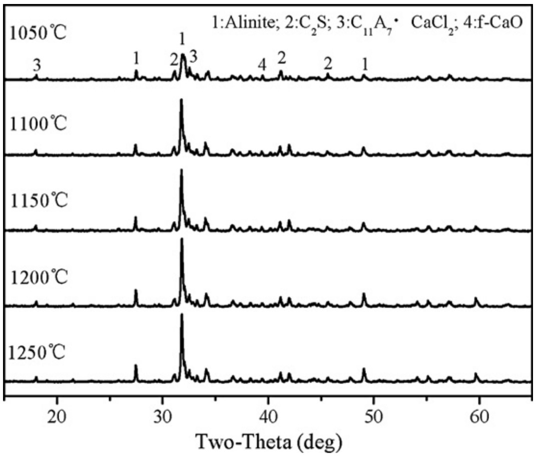


Fig. 4. XRD patterns of A3 fired at different temperatures for 2 h.

Table 3
Chemical composition of clinker A3 (by wt.%).

CaO	SiO_2	$CaCl_2$	Al_2O_3	Fe_2O_3	MgO
58.8	21.9	11.6	4.8	0.7	2.1

3.5. Physical properties

3.5.1. Compressive strength

Four kinds of alinite cement were prepared by grinding the A3 clinker with 0%, 3%, 5% and 10% $CaSO_4{\cdot}2H_2O$ separately to a fineness of 400 m^2/kg (Blaine). Fig. 6 shows compressive strength development of the alinite cements and Portland Cement (P.O42.5). It is seen that the addition of gypsum as additive to alinite cement causes an enhancement in compressive strength, and the strengths of all the alinite cements at 1 day and 3 day are greater than those of the P.O42.5. However, the compressive strengths of all the alinite cement samples develop slowly between 3 day and 28 day. At 28 days, the alinite samples have slightly higher strengths when the gypsum replacement is over 3%, but much lower strengths under 3% compared with the P.O42.5. It is worth mentioning that there is an increase in the early day strengths (1 day and 3 day) with increase in the gypsum replacement (from 3% to 5%), but the early strength decreases significantly when the gypsum replacement reaches to 10%. Therefore, the optimum content of gypsum in the alinite cement is about 5% by weight.

3.5.2. Setting time and water demand

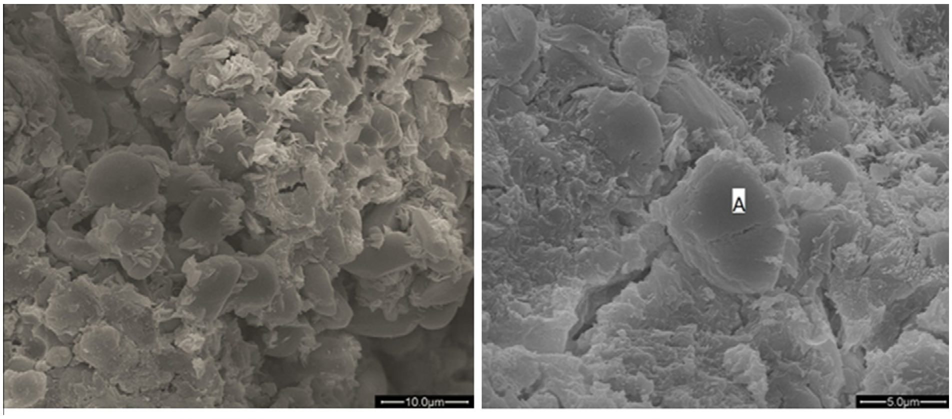
The setting time and water demand of alinite cements and P.O42.5 samples are given in Table 5. It is seen that alinite cement presents less water demand than the Portland cement to acquire normal consistency. The addition of gypsum leads to a slight increase of the water demand that is disproportionate to the gypsum content. The normal consistency varies from 22% to 24% in all the alinite cement samples.

The addition of gypsum exerts a significant influence on the setting time of alinite cement. Both the initial and final setting times are shortened significantly with an increase in the gypsum content. Moreover, the alinite cement has shorter setting time compared with Portland cement, which is a significant characteristic of alinite cement. The initial setting time of C-3 cement with an acceptable early and later days strength is not more than 20 min while the final setting time is within 60 min.

3.6. Volatilization during clinkerization and toxicity leaching characteristics of heavy metals

According to the previous studies [34], minor elements could be divided into three groups based on their volatility, high volatile (<20% remained in clinker, like Pb and Cd), moderate volatile (such as Ni and Cr), and low volatile (almost 100% remained in clinker, like Cu). The determined heavy metals concentrations in the MSWI fly ash samples and the optimized clinkers (A3) were measured as shown in Table 6. It can be found that a large amount of Cd, Zn and Pb (more than 85%) are volatilized during the clinkerization. Meanwhile, over 70% of the Ni, Cr and Cu are trapped in the clinker. Heavy metals like Cd, Zn and Pb might be present as $CdCl_2$, $ZnCl_2$ and $PbCl_2$ due to high amounts of chloride in the MSWI fly ash. These compounds would be volatilized at high temperature during the clinkerization process because of their relatively low boiling point (the boiling points of $CdCl_2$, $ZnCl_2$ and $PbCl_2$ are 960, 732 and 950 °C, respectively) [13]. Heavy metals like Ni, Cr and Cu are not volatilized during the clinkerization, because these metals react with other constituents in the raw mix and form some stable compounds.

Since the clinker contains high amount of Zn, Cr, Cu along with considerable amounts of Cd, Pb and Ni, these elements may be leached in an acid environment or washed out by water. To investigate this possibility, leaching tests were performed on hydrated samples (A3 with 5% gypsum) at different curing ages. The experimental results are shown in Table 7. It is observed that the con-



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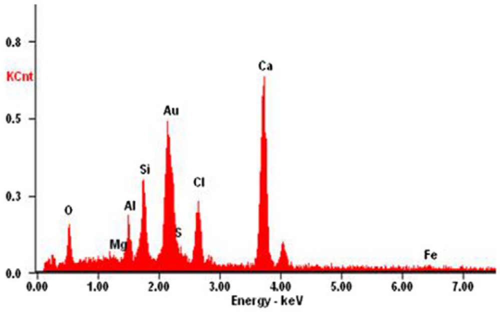

(c) EDS of point A

Fig. 5. SEM photos and EDS pattern of cement clinker (A3 fired at 1200 °C for 2 h).

Table 4
Atomic proportion of point A in Fig. 4b (at./%).

Element	Mg	Al	Si	Cl	Ca	O	Total
EDS value of point A	2.68	2.15	11.03	2.97	27.74	53.42	99.99
Theoretic value	2.35	1.76	10.00	2.94	29.41	52.94	99.40

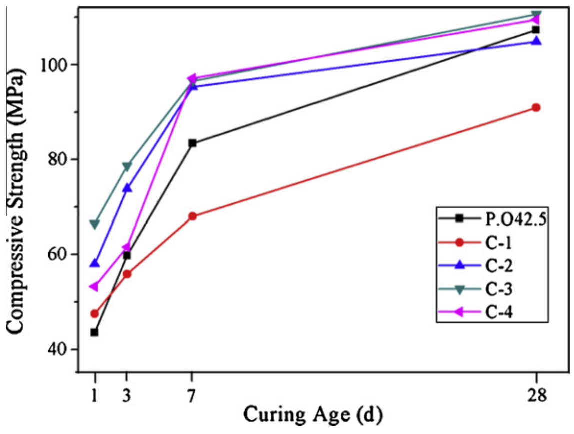

Fig. 6. Compressive strengths of alinite cements and P.O42.5, (C-1): 100% A3 clinker; (C-2): 97% A3 clinker with 3% $CaSO_4{\cdot}2H_2O$; (C-3): 95% A3 clinker with 5% $CaSO_4{\cdot}2H_2O$; (C-4): 90% A3 clinker with 10% $CaSO_4{\cdot}2H_2O$.

Table 5
Setting time and water demand of the alinite and Portland cements.

Sample	Water demand (%)	Initial setting (min)	Final setting (min)
C-1	22	45	135
C-2	22	26	85
C-3	23	17	54
C-4	24	14	50
P.O42.5	26	65	210

Table 6
Heavy metals in MSWI fly ash and A3 clinker (mg/kg).

	Cd	Cr	Cu	Zn	Ni	Pb
MSWI fly ash	68.3	451	1122	3269	70.5	1607
A3 clinker	4.3	316.8	976.4	413.5	54.0	32.5

Table 7
Concentrations of heavy metals in leachates at different curing ages (mg/L).

	Cd	Cr	Cu	Zn	Ni	Pb
Regulatory limit[a]	<1	<15	<100	<100	<5	<5
1 day	ND	0.013	0.051	0.039	0.011	0.009
3 day	ND	ND	0.037	0.021	ND	ND
7 day	ND	ND	0.023	0.014	ND	ND
28 day	ND	ND	0.014	ND	ND	ND

ND: not detected (<0.005 mg/L).

[a] Chinese National standard GB 5085.3, 2007 (Identification standards for hazardous wastes-Identification for extraction toxicity).

centrations of all the investigated elements in the leachates are far below the regulatory limits at all curing ages. This indicates that most heavy metals are trapped in the clinker and well fixed by the hydration products. Thus the produced cements do not present an immediate threat to environment.

4. Conclusions

Results of this study show that it is feasible to produce good quality alinite cement using MSWI fly ash as a raw material. The following conclusions may be drawn from the present investigations:

(a) In order to produce good quality alinite cement clinker, the percentage of MSWI fly ash in the raw mix should be not more than 30 wt.%, and the total amount of MgO in the raw mix is about 1.4%;
(b) From the XRD patterns and energy consumption, it can be seen that good quality clinker was obtained by firing the raw mix at 1200 °C for 2 h;
(c) With an increase in the addition of gypsum content, there is a slight increase in water demand and a reduction in setting times. Compared with the compressive results for P.O42.5, alinite cements have higher early strengths at all gypsum additions, while the best result having acceptable early and 28 day strengths was obtained at 5% of gypsum addition.
(d) During the clinkerization and hydration process, heavy metals such as Cr, Pb, Cd, Zn, Ni and Cu are trapped in the clinker and well fixed by the hydration products, and do not present a leaching hazard to the environment.

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