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Preparation of Portland cement with sugar filter mud as lime-based raw material

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ABSTRACT

The objective of this study was to assess the preparation of Portland cement with sugar filter mud (FM) as lime-based raw material. Burnability of raw mix, SEM characteristic and phase component of clinker, compressive strength, setting time and hydration characteristic of cement are investigated. The results show that FM can improve the raw mix burnability, and increase liquid phase amount. Less than 20% FM is helpful to promote the C_3S formation, and heighten the C_3S content in clinker. More than 20% FM will result in the new phase formation. Compressive strength, setting time and hydration characteristic of cement all are related to the replacement ratio of limestone with FM. It is found that the proper FM amount can raise the compressive strength, cut down the setting time, and promote the cement hydration in the initial and acceleration period. Excessive FM will lead to the decrease of compressive strength, the delay of setting time and prolongation of cement hydration.

1. Introduction

Solid waste (SW) is generated in the industrial production activities such as manufacturing, energy production, water supply, chemical engineering and food processing et al., and it generally is associated with hazardous constituents such as toxic heavy metal, bacteria and harmful organic substance et al., and has a high public health and environmental risk. But if it is disposed with the reasonable technology or approach, it may be changed valuable material or fuel. Public health and environment will be prevented from its potential threat. The appropriate disposal method for SW consequently has been the expectance, which the enterprise, researcher and government have given their great efforts to make come true.

Sugar filter mud (FM) is produced after sugar juice clarified, and is the main solid waste in sugar industry. Its safe disposal always has been the hot topic for random stacking occupies land and pollutes the air, landfill pollutes the underground water. Although it is fairly rich in inorganic and organic nutrients (Yaduvanshi and Yadav, 1990), it finds that little is used to produce agriculture fertilizer (Elsayed et al., 2008). The major reason for this is the insoluble and imbalance nature of the nutrient in it. Besides, it takes long time to decompose, and the intense heat and foul smell are generated (Sen and Chandra, 2006). It is reported that FM can be reused to desulphurize the fuel gases (Dolignier and Martin, 1997), and to prepare the octacosanol (Qu et al., 2012). Unfortunately, other kinds of SW are produced, and also have to be disposed. Lime can be prepared with FM (Nikolaos, 2004). But, it is necessary to avoid the presence of unsuitable substances. Sarka et al. (2008) state FM can be used as raw material to produce the breezeblock. The concern is that its high calorific value will not be exploited to the full in this process. There have been many approaches for FM utilization, but all of them still have various drawbacks. In China, more than 16,000,000 t of sugar (in 2011) is produced annually, and about 16,000,000 t of FM also is discharged every year. Serious environment problems have happened because it is stacked around the factory without safe disposal. It is pressing to find a new method to reuse FM reasonably. This method should be the better one, and the sugar industry and environment all can be developed sustainably.

In recent years, sustainable development and natural resources reservation have become global issues (Sabine, 2013). The cement industry, which is known as one of the important consumptive industry for raw material and energy, has integrated these issues to its development policy (Schneider et al., 2011). Many industrial SW are reused to replace the traditional materials or fuel in the production process (Li et al., 2012; Rodriguez et al., 2013). Alternative

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raw materials mainly provide the necessary chemical compounds, such as CaO, SiO_2, Fe_2O_3 and Al_2O_3 for cement raw mill. The main chemical component of FM is CaO (Neha et al., 2011), which is also the main chemical compound of lime-based cement raw materials. Thereby, at least in theory, it can be used as the alternatives of lime-based materials for clinker production. There are also several advantages of utilizing FM in the cement clinker production. The high temperature (above 1400 °C) in kiln can decompose the toxic organic matters and the bacteria; fuel can be also saved due to high calorific value of FM (Ribbing, 2007). But, FM also contains several higher contents of impurities except for the CaO, such as MgO, SO_3 and P_2O_5 as compared with the traditional lime-based raw materials. Minor content of MgO, sulfur compounds and phosphates normally are used as mineralizers to decrease the viscosity of the interstitial melt, stabilize different polymorphs of tricalcium silicate (C_3S) and dicalcium silicate (C_2S), and improve the Portland cement strength, alone or together with other minor components (Stanek and Sulovsky, 2002; Kolovos et al., 2001). In practical production, the content of sulfate and magnesium added to the raw mix are limited for the restrictions on the SO_3 and MgO content in the clinker. Additionally, high content of P_2O_5 will decrease the C_3S: C_2S rate, and give rise to the formation of α-C_2S (Lin et al., 2009).

Although there is lot of research efforts as reported in these literature, it is difficult, through them, to evaluate the preparation of Portland cement clinker with sugar filter mud as lime-based raw material because of the varieties of materials used in the previous researches. There still remain some important points needed to systematically clarify. They are crucial to make effective use of FM in Portland cement production. Therefore, the raw mix burnability is discussed, the morphology and composition characteristics of the clinker are presented, and the physical performance and the hydration characteristic of cement prepared with FM are investigated in this paper.

2. Experimental

2.1. Materials

Limestone and gypsum were industrial materials. Powders of aluminum sesquioxide, silicon dioxide and ferric oxide were used to adjust the contents of Al_2O_3, SiO_2 and Fe_2O_3 in the raw mixes, and all of them are chemical reagents. FM was obtained from a sugar manufacture corporation. FM and limestone were dried to constant weight at 105 °C, crushed by jaw crusher and ground to ASTM 200 mesh size with a centrifugal ball mill. Chemical compositions of FM and limestone are shown in Table 1.

2.2. Clinker preparation

The compositional parameters in cement chemistry are listed as follows (Eqs. (1)–(3)).

$$\text{Lime saturation ratio (KH)} = \frac{CaO - 1.65Al_2O_3 - 0.35Fe_2O_3}{2.80SiO_2} \quad (1)$$

$$\text{Silica ratio (SM)} = \frac{SiO_2}{Al_2O_3 + Fe_2O_3} \quad (2)$$

Table 1
Chemical compositions of FM and limestone (wt %).

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	Na_2O	K_2O	MgO	SO_3	P_2O_5
Limestone	3.62	0.65	0.35	50.37	0.05	0.03	0.88	–	–
FM	2.30	0.39	0.37	48.40	0.08	0.49	1.32	2.85	2.5

$$\text{Alumina ratio (IM)} = \frac{Al_2O_3}{Fe_2O_3} \quad (3)$$

All raw materials were blended with a similar set of parameters (KH = 0.90, SM = 3.0, IM = 1.7). The difference was that the limestone was replaced with different percentages of FM. FM1, FM2, FM3, FM4 and FM5 represent respectively the mixes in which FM substitutes for 5, 10, 15, 20 and 40 wt % the limestone. The chemical composition and parameter of all mixes are given in Table 2.

All mixes were prepared with appropriate water, put into the cylindrical mold, and pressed to a slice with a pressure of 200 MPa. Then these slices were heated to 1450 °C with the rate of 25 °C/min, kept the temperature for 2 h in the furnace, and cooled rapidly in the air to room temperature. With 3 wt % gypsum, the clinkers were ground to ASTM 200 mesh, and the cements were obtained.

2.3. Testing methods

The contents of free lime (f-CaO) in clinkers were analyzed by the glycerol–ethanol method.

Scanning electron microscope (SEM) was carried out on a Quanta 200 FEG to observe the microcosmic characteristics of the obtained clinkers. The accelerating voltage was 20 kV, and the magnification was 2000.

Mineral phases of clinkers and pastes were identified by a D/max 2550 X-ray powder diffractometer (XRD), and the 2θ range was 20°~60°, in 0.02° steps, counting by 4 s per step. The radiation was CuKα at wavelength of 0.1541 nm (40 kV).

Compressive strength tests were carried out according to the Chinese National Standard GB/T 17671-1999. Mortars were prepared by mixing cements with drinking water at a water-to-cement weight ratio of 0.5 and cement-to-sand ratio of three, casted in 40 mm × 40 mm × 160 mm molds and vibrated at the time of casting to remove air bubbles. The molded pastes were kept at 20 ± 20 °C and relative humidity exceeding 90% for 24 h, and then removed from the molds. The demoulded samples were cured in a water tank at 20 ± 2 °C for the set ages and then their strengths were measured.

Setting time of cement was examined according to the Chinese National Standard GB/T 1346-2001.

An isothermal heat-conduction calorimetry (TAM air C80, Thermometric, Sweden) was used to measure the hydration heat evolution of cements. The water-cement ratio was 0.5 and experimental temperature was 20 ± 0.1 °C. Cement and water were tempered for several hours before mixing, then the water was injected into the reaction vessel and the samples were stirred in the calorimeter for several minutes. This procedure allowed monitoring the heat evolution from the very beginning when water was added to cement. Data logging was continued for about 3 days.

Table 2
Chemical composition and parameter of all raw mixes (wt %).

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	Na_2O	K_2O	MgO	SO_3	P_2O_5
Control	24.08	4.91	2.96	70.04	0.07	0.04	1.23	–	–
FM1	24.08	4.91	2.96	70.04	0.07	0.07	1.26	0.21	0.18
FM2	24.08	4.91	2.96	70.04	0.07	0.11	1.30	0.41	0.36
FM3	24.08	4.91	2.96	70.04	0.08	0.15	1.33	0.62	0.55
FM4	24.08	4.91	2.96	70.04	0.08	0.17	1.36	0.82	0.72
FM5	24.08	4.91	2.96	70.04	0.09	0.30	1.50	1.65	1.45
LSF					0.90				
IM					1.70				
SM					3.0				