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Utilization of waste glycerin, industry lignin and cane molasses as grinding aids in blended cement

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HIGHLIGHTS

- Waste glycerin (WG), industry lignin (IL) and cane molasses (CM) are used as grinding aids.
- Grinding efficiency of WG, IL and CM on the blended cement are different.
- Performance of blended cement with composite grinding aids are evaluated.

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ABSTRACT

This paper investigates the viability of using waste glycerin (WG), industry lignin (IL) and cane molasses (CM) used as grinding aids in blended cement, performance of the blended cement with single and composite grinding aids were researched. The structure characterization of the grinding aids was carried out by Fourier transform infrared spectroscopy (FT-IR); the particle size distribution, Blaine specific surface area, setting times, water requirement of normal consistency, flexural and compressive strengths at 3 and 28 days of the blended cement were researched, the experiments results indicate that the addition of grinding aids results in an improvement of the fineness and strength of the blended cement. Compared with the cement mixture without grinding aids, waste glycerin has a better optimization effect on the particle size distribution, industry lignin and cane molasses perform a better retarding effect, and enhancement effect of cane molasses on the strength of blended cement is more significant. For the blended cement with composite grinding aids (0.0065% WG, 0.0095% IL, 0.024% CM), the compressive strength at 3 days and 28 days are 2.6 MPa and 10 MPa higher than those of the control cement respectively. The hydration products and microstructure of blended cement with composite grinding aids were analyzed by X-ray diffraction (XRD), thermo-gravimetric and differential scanning calorimetry (TG-DSC) analysis and scanning electron microscopy (SEM), the research shows that for the blended cement, the addition of grinding aids can accelerate the hydration reaction degree and improve the compactness and uniformity of the products structure.

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1. Introduction

Cement is the most important and traditional building materials, the world cement production reached 4.18 billion tons in 2014, and China contributes most of the product capacity that about 2.5 billion tons, the resource and energy consumption of cement production is giant during cement producing process [1,2]. Approximately 40% of the total electric energy is consumed on the final cement clinker grinding, and much of them is transformed into heat and waste, and a serious of environment problems are brought such as the emission of greenhouse gas CO_2 (0.98 ton/ton clinker), toxic gas (SO_2, NO_X), and suspended fine ash particles [3,4]. Therefore, a small improvement in grinding efficiency not only have a large impact on the operating cost of a cement company, but also make a reduction of pollution source emission.

Admixture of grinding aids in cement grinding process can save mechanical energy and affect the variations of fineness characteristics [5], the action of grinding aids is governed by mechanochemical activation that has been discussed [6]. Various of material have been used as grinding aids which can be classified by liquid and solid grinding aids. Liquid grinding aids such as aliphatic amines, amine alcohols and glycol compounds, including triethylenetetramine (TETA), etraethylenepentamine (TEPA), triethanolamine (TEA) and triisopropa-nolamine (TIPA), *etc.* Solid

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grinding aids such as metaphosphoric and sulfonate, including sodium tripolyphosphate (STPP), dodecyl benzenesulfonic acid (DBA) and calcium lignosulfonate (CL), *etc* [7]. The plurality of functional groups in grinding aids made them has a strong adsorption that can adsorbed on the surface and micro-crack of cement particle, forming absorption film, reducing the free energy of particle surface and promoting the propagation of cracks [8,9]. TEA is one of the most commonly used grinding aids that has proved to have an obvious effect on the strength enhancement, TEA could accelerate the reaction of C_3A with calcium sulfate in Portland cement [10,11], but the addition of TEA will lead to a higher cement manufacturing cost and it seems not as effective as expected for the grinding production of blended cement [12,13], so the advantages of various grinding aids are need to be considered in the cement grinding process.

Industry lignin is a by-product of paper industry, about 0.5 ton of industry lignin is generated from the production of per ton of paper pulp, owing to its advantages including easily available, low cost and environmental friendly, lignin and its derivatives can be used as concrete additives that has a super retarding performance [14,15]. Cane molasses is a by-product of the cane sugar production, it is a thick, dark brown and semi drifting material with 40–56% sugar content, molasses has proved to have a water reducing performance that could used as an admixture in concrete durability [16], now the usage of molasses in the cement concrete construction in China is gradually expanding [17,18]. Waste glycerin is acquired from the biodiesel production, the utilization of this glycerin is limited since it is considered an unrefined raw material that must be refined for its further use [19].

The purposes of this paper are to investigate the effect of waste glycerin, industry lignin and cane molasses on the grinding efficiency, mechanical strength, hydration products and microstructure of Portland cement. The grinding efficiency was examined from the particle size distribution and Blaine specific surface area, the water requirement of normal consistency, setting time and mechanical strength were tested to evaluate the physical performance of the ground cement, the chemical structure of the grinding aids and the hydration products and microstructure of the blended cement were also researched.

2. Experimental procedure

2.1. Materials

Portland cement clinker, gypsum, fly ash, granulated blast-furnace slag (GBFS) were provided by Wuhu Hailuo cement Co., Ltd in Anhui province, China; sand, China ISO standard sand, produced by Xiamen ISO Standard Sand Co., Ltd. The chemical compositions of the raw materials are shown in Table 1.

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

Chemical composition	Clinker	Gypsum	Fly ash	GBFS
SiO_2	19.83	7.69	52.03	35.06
Al_2O_3	4.35	1.88	30.66	15.26
CaO	64.1	34.69	6.27	34.62
Fe_2O_3	2.59	0.69	5.73	1.78
MgO	1.33	0.96	1.07	9.89
SO_3	1.01	32.07	0.29	0.05
Loss	4.25	20.68	0.86	0.10
Total	97.46	98.66	96.91	96.76

Waste glycerin (WG), a by-product of forestry bio-diesel oil, light yellow transparent liquid; industrial lignin (IL), a by-product of cellulose pulp from paper production, brownish yellow powder, the determination content of carbon, hydrogen and nitrogen elements element are 42.09%, 4.23% and 0.78%, respectively, both the waste glycerin and industry lignin are provided by the National Engineering & Technology Research Center of Forest Chemical industry. Cane molasses (CM), brown viscous liquid, provided by Liuchen Sugar company, Guangxi, China, the main chemical components of cane molasses are given in Table 2.

Table 2
Chemical composition of cane molasses (wt.%).

Molasses	Brix	Purity	Sucrose	Invert sugar	Colloid	pH	Ash	Nitrogen
Components	76.14	48.42	40.07	8.75	6.42	6.85	7.72	0.35

2.2. Test Methods

2.2.1. Cement test mill

The control cement mixture was selected with the weight ratio of clinker: fly ash: GBFS: gypsum: 80:10:5:5. A laboratory ball mill was used to grind blended cements, clinker, gypsum and GBFS were crushed into small particle less than 6–7 mm diameters prior to grinding procedure, the total feed weight was 5 kg per mill and the grinding time was kept the same as 20 min for each ground cement. The type of the ball mill is 500 mm × 500 mm, 48 r/min and closed circuit, and the grinding media is composed by 60 kg steel balls (Φ40 mm, Φ50 mm, Φ60 mm and Φ70 mm) and 40 kg small steel forgings (Φ25 mm × 35 mm).

2.2.2. Grinding efficiency test

The fineness of cement was evaluated from the Blaine surface measurement and particle size distribution [20,21]. Blaine surface of the blended cement with and without grinding aids were tested according to the Chinese national standard GB/T8074-2008 (Testing method for specific surface of cement-Blaine method). Particle size distribution of the blended cement were tested according to the Chinese national standard JC/T 721-2006 (Test method for particle size of cement, Laser based methods), a Malvern Zetasizer 3000HSA laser diffraction particle size analyzer was used in aqueous liquid module mode.

2.2.3. Physical performance test

The water requirement of normal consistency and setting time of the ground cement were tested according to the Chinese national standard GB/T1346-2011 (Test method for water requirement of normal consistency, setting time and soundness of the Portland cement). Mechanical strength of the mortars were tested according to the Chinese national standard GB/T 17671-1999 (Method of testing cements-Determination of strength), mortar prisms (40 × 40 × 160 mm) were cast according to the same weight ratio of cement:sand:water:1:3.0:0.5, after 24 h in a moist cabinet, they were removed from the mold and cured in water at room temperature (20 ± 1 °C). At the ages of 3 and 28 days, flexural strength of every mixture was measured on three prismatic specimens and then the compressive strength test was tested on six pieces of prisms.

2.2.4. Fourier transform infrared spectroscopy (FT-IR) analysis

Chemical structure of the grinding aids were characterized by a Thermo-Nicolet Nexus 670 FT-IR, in the range of 4000–400 cm^{-1} with 200 successive scans. The spectra rationed against a potassium bromide (KBr) background. For the solid IL powder, 3 mg of

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