Construction and Building Materials 75 (2015) 163–168

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Preparation of polymer-based cement grinding aid and their performance on grindability

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HIGHLIGHTS

- The process of preparing polymer-based grinding aid was introduced.
- The structure of polymer-based grinding aid was characterized by FT-IR.
- The performance of polymer-based grinding aid was determined.
- The change of shape of ground cement particles was determined by SEM.
- Polymer-based grinding aids increased the fluidity of ground cement.

ARTICLE INFO

Article history:
Received 21 July 2014
Received in revised form 24 September 2014
Accepted 30 October 2014

Keywords:
Polymer
Grinding aid
Specific surface area
Particle size distribution
Repose angle

ABSTRACT

This paper described a chemical process called water-based free radical polymerization to synthesize a polymer-based grinding aid (PGA), which was characterized by infra-red spectroscopy (FT-IR) and used for the production of Portland cement (ground in a ball mill at a laboratory stage). For this purpose, reference cement was also produced without using PGA. All the cement mixtures were tested for specific surface area and particle size distribution. The changes of shape of particles and repose angles of ground cement were also tested to evaluate the flow characteristics of ground cement. Compared to a reference cement admixture, the addition of PGA led to significant gains in specific surface area, a fact that was attributed to the PGA's ability to prevent agglomeration and powder coatings of ball. The results also indicated that PGA had obviously influences on particle size distribution of ground cement, which showed a narrower grading spectrum as the PGA dosage increased. Test results by SEM and repose angle method showed that PGA resulted in improvement of roundness and fineness of cement grains and fluidity of ground cement.

1. Introduction

Cement world production currently increases up to approximately 4 billion tons/year. The electrical energy consumed in cement production is 110 KW/tone and about 30% of which is used for raw material preparation and about of 40% for final cement clinker grinding [1–3]. This leads to important cost increase of the final product, especially under the present energy scarce environment [4].

During comminution of clinkers, highly reactive positive and negative charges are created on the newly fractured surfaces. As a result, the agglomeration phenomenon of solid particles occurred due to Van der Waals force and electrostatic attraction, which results in the formation of coatings in the grinding media and reduces the grinding efficiency [5–7]. In order to improve the energy efficiency of cement production process, one of successful directions is extensive use of grinding aid in comminution process. Because of highly organic polar nature, grinding aid is generally absorbed on particle surface through physical or chemical interaction with cement salts [8]. Due to particle surface state transformation, new charges are neutralized and attractive surface forces are screened, leading to lower grain surface hardness and better dispersion ability of particles [9].

Nowadays, the most commonly used grinding aid generally include highly purified chemicals [10–14] such as diethanolamine (DEA), triethanolamine (TEA), ethylene glycol (EG) triisopropanolamine (TIPA), diethyleneglycol (DEG), inorganic salts as well as industry by-products such as molasses, corn syrups, polymeric alcohol mixtures. Many researches have already been carried out for the grinding effect of these grinding aids on the productivity

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http://dx.doi.org/10.1016/j.conbuildmat.2014.10.046
0950-0618/

Table 1
Chemical analysis and compositions of Portland cement clinker used.

Oxides	Chemical analysis (%)	
	Clinker	Gypsum
Na_2O	1.30	–
MgO	2.50	0.44
K_2O	0.66	–
CaO	54.87	34.55
TiO_2	0.23	–
Cr_2O_5	0.03	–
Fe_2O_3	2.63	0.33
MnO_2	0.03	–
Al_2O_3	7.89	0.01
SiO_2	22.55	0.30
P_2O_5	5.36	–
SO_3	1.91	45.23
LOI	–	17.43

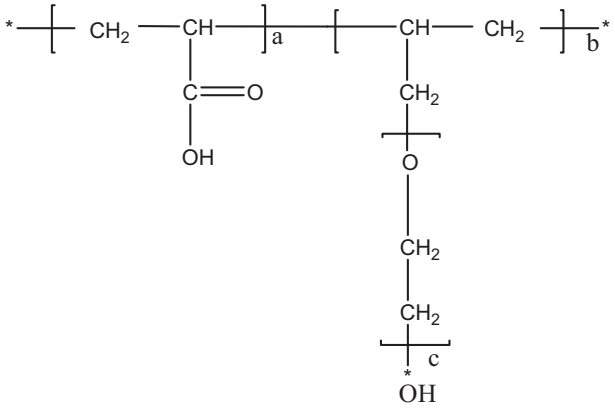

Fig. 1. Chemical structure of polymer-based grinding aids.

increase and gains in mill output. For example, M. Katsioti, etc. studied the characterization of six commercial grinding aids and their impact on grindability [15]. According to their test results, TEA and TIPA presented in grinding aid as main components increased the grindability index up to 14% and 26%, respectively. In addition, Beet molasses used as a grinding aid in blended cements was also evaluated [11]. It was found that beet molasses used as grinding aid provided better size distribution for blended cement which contributed to the development of strength of cement at 3 days and 28 days. Abdulkader Rashwani, etc. studied the grindability of industrial waste of soapstock during cement production and found the cement particles were finer and particle size distribution program shifted toward finer diameter [16].

Generally, the concentration range of grinding aid added is of great concern, which may cause the variability of cement performance. For example, at an addition of 0.02% to Portland cement, TEA acts as a set accelerator, at 0.25% it acts as a mild set retarder, at 0.5% TEA acts a severe retarder, and at 1% it is a very strong accelerator [17,18]. Moreover, a large quantity of inorganic salts used can obviously affect the durability, life and safety of concrete. In order to develop new grinding aid with more stable quality and higher cost performance, intensive study has been done on polymer-based grinding aid.

The aim of this paper was the preparation of polymer-based grinding aid which was measured by FT-IR. The grinding aptitude of PGA was examined by specific surface and particle size distribution of ground cement at different dosages and grinding durations in compared with the reference cement. The changes of shape of cement particles and repose angles of ground cement were also examined to evaluate the fluidity of ground cement.

2. Material and methods

2.1. Materials

The following chemicals were used for synthesis: acrylic acid was obtained from Liyang Zhongshan chemical Ltd., china; allyl polyoxyethylene ether was supplied by Nanjing Yangzi Oxiran chemical Co., Ltd, china; hydrogen peroxide was obtained from Wuxi Ruibo chemical Co., Ltd. China; vitamin was obtained from Shengxu chemical Co., Ltd. China; triethanolamine (TEA) was obtained from Oriental petrochemical corporation; dodecyl mercaptan was obtained from Nanjing Huachen fine chemical Co., Ltd. Portland cement clinker and gypsum were from Hailuo cement Co., Ltd. in Anhui province PR. China. Their chemical and mineralogical compositions are shown in Table 1. The influences of PGA on cement are presented in Table 2.

2.2. Synthesis process

PGA is regarded as water soluble copolymers. They are composed of carboxyl groups and long side chains of polyoxyethylene ether. Hydrogen peroxide and vitamin are used as initiators, while dodecyl mercaptan as chain transfer agent. In each process, the polymerization was carried out in a jacked reactor equipped with a stirrer and a reflex condenser. First of all, 240 g of Allyl polyoxyethylene ether (APE) and 0.25 g of dodecyl mercaptan (DM) were dissolved in water in reactor. Secondly, 20 g of Acrylic acid, 30 g of hydrogen peroxide, and 5.6 g of vitamin were dissolved in water, respectively, which were named as PI, PII and PIII. Then, the reactor was heated to the reaction temperature. When the solution in reactor became clear, PI, PII and PIII were fed into the reactor with constant flow pumps. During the feeding process, the reaction temperature was kept at 50–55 °C, and the feeding time was lasted for 3.5 h. The reaction continued for another 20 min at 50–55 °C, when the feeding finished. After cooling, the PH value of solution was adjusted by addition of 35 wt.% aqueous NaOH solution. Finally, the copolymer-based grinding aid was obtained with solid content of 30% and PH value of 4–5. Fig. 1 shows the structure of copolymer-based grinding aid. The process flow chart of synthesis was presented in Fig. 2.

2.3. Methods

2.3.1. Physical and mechanical properties of cement

The water requirement of normal consistency and the setting times of cement pastes were determined according to the European Standard EN 196-3 [19]. The normal mortar flow was determined according to ASTM C1437 [20]. The compressive strength at the ages of 3, 7 and 28 days were conducted in accordance with the Chinese Standard GB/T17671 [21].

2.3.2. Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR was performed with a Bio-Rad FTS 6000 FTIR using KBr pellet techniques to measure the energy absorption of the ancient samples. Here, 3 mg of the finely ground <80 m specimen powder was homogeneously ground with 300 mg of KBr powder until the mixture had the consistency of fine flour, and then pressed into a thin 15 mm diameter disc.

Table 2
Setting time, standard consistency, flow of normal mortar and compressive strength of ground cement.

Cement index	PGA ratios (%)	Setting time (min)		Water requirement of normal consistency (% w/w)	Flow of normal mortar (%)	Compressive strength (MPa)		
		Initial	Final			3 d	7 d	28 d
1	0	250	272	26.1	100	27.90	38.10	43.80
2	0.02	253	273	26.1	105	28.20	38.05	44.50
3	0.06	255	273	26.0	110	29.06	38.20	46.18
4	0.10	260	280	25.9	120	29.41	39.37	46.73

Note: grinding duration = 45 min.