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Quantifying the effect of clinker grinding aids under laboratory conditions

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ABSTRACT

The effect of clinker grinding aids (GAs) on mill performance and cement properties is currently quantified in real-scale grinding plants. In fact, laboratory-grinding mills operated for given time interval do not consider the effect of circulating load, thereby leading to increased specific energy consumption (Ec) with excessively wide cement particle size distribution (PSD) curves. The main objective of this paper is to develop and validate a laboratory locked-cycle approach that mimics the industrial grinding operations occurring in closed-circuit tube mills. Test results have shown that Ec and its rate of decrease due to GA additions can be adequately predicted using this approach, when compared to those resulting from a real mill operating at 90 ton/h. The addition of GA led to narrower PSD curves and shifting towards smaller diameters, just like what happens in industrial grinding. Also, the cement properties including water demand, setting time, and compressive strength matched to a large extent within each other.

1. Introduction

Laboratory-grinding mills are widely used by cement researchers and companies to assess the effect of grinding aids (GAs) on processing of clinker and energy savings. The grinding is realized either for fixed specific energy consumption (Ec) or Blaine fineness, and results compared to those of a control mix ground without GA. In case the grinding is performed for fixed Ec (i.e., mill rotating for given time interval), the incorporation of GA is typically associated with an increase in cement Blaine fineness and compressive strength at various ages (Assaad et al., 2009; Sverak et al., 2013). Practically, this could be relevant when producing cement possessing increased Blaine values necessary for high early strength requirements (such as complying with ASTM C150 Type III (ASTM C150, 2012). From the other hand, tests realized for fixed Blaine fineness are often used to evaluate the effect of GAs on Ec and resulting cement properties such as water demand, setting time, and compressive strength (Assaad et al., 2010; Katsioti et al., 2009). Nowadays, GAs are increasingly used to reduce Ec and improve mill productivity for given cement fineness, thereby meeting with today's constraints regarding the reduction of usable energy (Schneider et al., 2011).

Because of different operational modes, data obtained from laboratory-grinding mills operated for fixed Ec or Blaine values cannot be directly transposed to industrial mills. In fact, the clinker processing in real-scale mills is a continuing process, whereby the grinding forces are applied to the coarse particles while the fine ones are discharged as soon as they have been reduced to the required cut size (Bhatty et al., 2004; Mejeoumov, 2007). All particles larger than the cut size would be sent to a reject stream, therefore creating a so-called circulating load (CL). The CL is defined as the average number of times that the material circulates through the grinding system before becoming the product. Typical CL values reported in the cement industry can vary from 1 to 3.5 depending on several factors including targeted fineness, separators efficiency, mill type and configuration, and clinker hardness (Mejeoumov, 2007; Benzer et al., 2001; Sottili and Padovani, 2002).

Unlike industrial mills, laboratory-grinding mills operated for fixed Ec or Blaine values do not account for CL, therefore leading to significantly different cement particle size distribution (PSD) curves. The PSD is often approximated using the Rosin–Rammler–Sperling–Bennett (RRSB) function where the particle size (d_0) reflects fineness and spread factor (n) evaluates the narrowness or wideness of PSD; the higher n means a narrower PSD (Delagrammatikas and Tsimas, 2004; Ferraris et al., 2002). The PSD differences between laboratory and industrial mills were acknowledged by ASTM C465 Standard Specification for GAs; these were related to changes in cement flowability and mill retention time (MRT) during grinding (ASTM C465, 2010). The MRT can be defined as the average time necessary for the bulk material to pass through the tube mill (Bhatty et al., 2004; Sottili and Padovani, 2002; Schnatz, 2004). Hence, to properly evaluate GA effects on

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Ec and cement properties, the standard recommends the realization of full-scale tests over enough time to ensure reaching equilibrium conditions and stable CL (ASTM C465, 2010). Fredvik (2005) found that laboratory mills lead to significantly wide cement PSD curves with *n* parameter lower than 1.0, when compared to those normally obtained in real mills. This, in its turn, alters cement properties such as water demand and strength development. Maxson et al. (1934) noticed the same phenomenon in the mineral grinding industry, as the ore exhibiting maximum resistance to grinding was found to accumulate in the CL. The authors concluded that a testing method that does not account for CL would yield considerably different results from those experienced in practice (Maxson et al., 1934).

The cement PSD is vital for water demand, rheology, and hydration processes such as heat release, setting time, volume change, and strength development (Katsioti et al., 2009; Zhang and Napier-Munn, 1995; Bentz et al., 1999; Assaad, 2015; Assaad and Issa, 2014). For example, Katsioti et al. (2009) reported that GAs added during clinker processing in industrial mills lead to narrow PSD, given the reduced percentage of very fine particles (below 1 μm) that mainly influence the setting time. This is why the resulting cement strengths at equal Blaine are higher than those obtained when grinding is realized without such additives. Conversely, when grinding is realized in laboratory mills, the percentage of very fine particles increases due to reduced cement agglomeration resulting from the presence of GAs (Katsioti et al., 2009; Assaad, 2015). It is to be noted that the Blaine measurement cannot effectively represent the entire PSD, given that two cements with different ratios of fine to coarse particles and described by different PSD can possess the same Blaine value (Delagrammatikas and Tsimas, 2004; Ferraris et al., 2002; Assaad and Issa, 2014).

2. Context and paper objectives

Currently, the quantification of GA effects on mill performance and cement properties is realized in industrial grinding mills. Yet, this task is quite cumbersome for both GA suppliers and cement companies, as it extends over several days and requires thorough planning ahead of time. For instance, the addition of GA especially at high rates should not abuse the grinding system, and requires close monitoring of grinding parameters such as the fresh feed rate and CL, mill and elevator motors power, mill sound and temperature, and separator rotor speed and air flow (Mejeoumov, 2007; Benzer et al., 2001; Sottili and Padovani, 2002; Fidan, 2011; Alsop, 2001). Normally, several hours are required to ensure uniformity of grinding parameters, and samples of cement are taken at regular time intervals for ensuring consistent cement fineness and residue.

The industrial testing becomes particularly complicated if a GA is already being used in the plant, as this necessitates stopping of GA dispensing to "clean" the mill from residual polymers and obtain the control cement mix. This stage extends over several hours, throughout which the grinding parameters should be continuously adjusted over time. For mills operating at several dozens of tons of cement per hour, this induces considerable drops in mill productivity and large amounts of finished products with highly fluctuated quality (Sottili and Padovani, 2002; Alsop, 2001; Fuerstenau and Han, 2003). It is to be noted that the interpretation of industrial data is often disputed between GA suppliers and cement companies, especially when testing is realized over several days due to inevitable changes in clinker quality, grinding parameters, and sampling procedures for cement testing.

This research study was initiated following an industrial grinding test undertaken to quantify the effect of a glycol-based GA on mill performance and cement properties. The first phase presents the data collected over 2 consecutive days of clinker grinding in a real-scale closed-circuit tube mill operating at around 90 ton/h. Tests were run without or with GA at different dosage rates of 280 and 530 g/ton of produced cement. The objective of the second phase seeks to develop a laboratory testing method that takes the CL into account and mimics the continuous grinding encountered in real mills. The method was inspired from the standard Bond method that was originally proposed to evaluate grindability of ores in the mineral industry (Maxson et al., 1934). Also, grinding tests realized over fixed time interval were realized. The comparison of mill performance including cement PSD and properties due to GA additions achieved under industrial and laboratory conditions is discussed in the third phase. Such data can be of particular interest to cement manufacturers, GA suppliers, as well as researchers dealing with processing and testing procedures of a variety of solid-mineral materials.

3. Materials used and testing methods

3.1. Materials

Industrial clinker used for the production of ASTM C150 Type I cement (ASTM C150, 2012), natural pozzolan meeting the requirements of ASTM C618 Class N (ASTM C618-12a, 2012), and gypsum materials were employed. Their ratio in the cement produced was maintained at 86.5%, 8.5%, and 5%, respectively. The materials chemical composition and PSD are presented in Table 1.

Commercially available glycol-based GA was used; it is referred to as clinker grinding improver and pack-set reducer in the cement industry. Its active chemicals determined by the Karl Fisher method, specific gravity, and pH were 64%, 1.08, and 7.8, respectively. The main components of this additive included diethylene glycol and propylene glycol.

3.2. Testing methods

The fineness of cement was evaluated using the Blaine measurement, sieve residue, and PSD curve. The Blaine was determined using the air-permeability apparatus as per ASTM C204 Test Method (ASTM C204, 2011), while the 45- and 90-μm residues refereed to as R-45 and R-90, respectively, were determined using a mechanical shaker.

A laser particle-size diffraction analyzer (Fritsch, Analysette 22 NanoTec Plus) capable of measuring particle sizes varying from 0.01 to 2000 μm was used for plotting the PSD curves. The apparatus complies with ISO 13320 technical requirements, with reproducibility (d_{50}) less than 1%. The equipment generates high-speed

Table 1
Chemical composition and grading of clinker, pozzolan, and gypsum.

	Clinker	Pozzolan	Gypsum
SiO_2, %	20.6	73.2	2.7
Al_2O_3, %	6.32	15.3	0.53
Fe_2O_3, %	4.3	4.4	0.39
CaO, %	64.2	2.8	32.5
MgO, %	1.86	2.25	1.5
SO_3, %	0.22	0.13	43.1
Percent retained on			
9.51 mm	1.2	3	0
4.76 mm	7.3	5.6	0.6
2.38 mm	3.7	6.2	3.5
1.19 mm	26	12.3	16.8
0.595 mm	11.5	13	24.4
0.297 mm	26.5	25.6	22.1
0.149 mm	14	20.5	14.5
0.074 mm	7.8	8.8	16
Pan	2	5	2.1