



# Effect of energy and temperature on performance of alkanolamine processing additions



Joseph J. Assaad

Professor of Civil Engineering, Holderchem Building Chemicals, Baabda, PO Box 40206, Lebanon

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## ABSTRACT

The effect of tertiary alkanolamines including triethanolamine (TEA) and triisopropanolamine (TIPA) on clinker processing and cement hydration reactions is well documented in literature. Yet, the results obtained cannot be directly transposed to real-scale industrial grinding, given that additive degradation due to attrition and heat are substantially different from those experienced under laboratory conditions. This paper is part of a research project undertaken to bridge the gap between laboratory and industrial grinding. It mainly seeks at quantifying the effect of specific energy consumption ( $E_c$ ) and elevated temperature on degradation (i.e., loss in performance) of alkanolamine-based additive used at 350 and 460 g/ton of cement. A series of grinding tests was performed using 50-l laboratory mill at various  $E_c$  values and materials temperature. Tested properties included water demand, setting time, heat of hydration, and strength development. Laboratory results are validated using data collected from real-scale closed-circuit tube mill operating at 90 ton/h.

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## 1. Introduction

The incorporation of small amounts of tertiary alkanolamines such as triethanolamine (TEA), triisopropanolamine (TIPA), diethanol-isopropanolamine (DEIPA), methyl-diethanolamine (MDEA) is common in the cement clinker processing industry. In addition to energy savings due to reduced agglomeration of cement grains, the practical consequences of such organic compounds include an increase in early/late age compressive strength and reduction of clinker factor while maintaining similar mechanical performance (Engelsen, 2008; Katsioti et al., 2009; Assaad et al., 2009, 2010). The former direction is relevant when producing cement possessing given fineness yet with high strength requirements (i.e., ASTM C150 Type III (ASTM C150, 2012)), while the latter is more and more demanded with today's constraints regarding the reduction of  $CO_2$  emissions and usable energy that could result from clinker production. Typical addition dosages vary from 200 to 1000 g/ton of the manufactured cement mass.

Scientific literature is abundant with laboratory studies assessing the effect of TEA and TIPA on cement hydration processes and altered properties such as heat release, rheology, setting time, and compressive strength. For instance, Ramachandran (1976) reported that TEA accelerates dissolution of  $C_3A$  and  $C_3S$  to form hydration products (Aft, CH and C-S-H) at early stages, which results in faster setting time and increased early strength.

However, with ongoing hydration, TEA becomes adsorbed on CH surface due to its low steric hindrance and precipitates along with CH when the concentration of  $Ca^{2+}$  reaches saturation (Perez et al., 2003; Heinz et al., 2010), thereby reducing its strength action at later stages. In contrast, the concentration of TIPA in solution does not decrease due to its higher steric hindrance, which forms  $Fe^{3+}$  complexes that accelerate hydration of  $C_4AF$ , even when the gypsum has been fully consumed (Gartner and Myers, 1993; Caijun et al., 2011). Gartner and Myers (1993) reported that the increase in 28-days compressive strength due to TIPA could reach 22.8% if  $C_4AF$  content is more than 7.6%. Sandberg and Doncaster (2004) showed that the average strength enhancement could be 10% for hydrated Portland cement pastes and 9% for mortars; tests were done using 10 different cements while TIPA added at 200 g/ton to the mixing water. Aggoun et al. (2008) found that the combination of calcium nitrate with either TEA or TIPA accelerated by up to 40% setting times and increased compressive strength by 35%.

Although such studies contributed largely to better understanding of altered cement properties due to alkanolamine additions, major discrepancies have been reported when transposing the results obtained under laboratory conditions to real-scale industrial mills (Mejeoumov, 2007; Fidan, 2011; Assaad, 2015). In fact, most of published data was concluded following clinker grinding in laboratory mills, whereby grinding parameters and additive degradation due to attrition and heat are substantially different from those encountered in reality. Also, it is to be noted that several researchers added their compounds as post-addition in

E-mail address: [jassaad@ndu.edu.lb](mailto:jassaad@ndu.edu.lb)

mixing water to simplify testing protocols (Engelsen, 2008; Caijun et al., 2011; Sandberg and Doncaster, 2004; Aggoun et al., 2008), which further enlarged the gap between laboratory and real-scale test results.

In earlier study, Assaad (2015) reported that the discrepancies in cement hydration reactions and properties sampled from industrial vs. laboratory testing could partly be related to differences in cement particle size distribution (PSD) curves, mostly the material percentages finer than 5  $\mu\text{m}$  or coarser than 90  $\mu\text{m}$ . Hence, industrial grinding generated narrow cement PSD curves as a result of enhanced screening, ventilation, and circulating load; while the PSD widened during laboratory grinding. Tests realized under industrial conditions required around 10% more specific energy consumption ( $E_c$ ) than those ground in laboratory; testing was realized with glycol-based grinding aid composed of diethylene glycol and propylene glycol at 280 and 530 g/ton of produced cement. Assaad (2015) and Assaad and Issa (2016) proposed a laboratory locked-cycle grinding approach that mimics real-scale clinker processing, and resulting cement properties including water demand, setting time, and compressive strength matched fairly well those obtained from industrial grinding. The matching of properties was mainly attributed to a physical effect (i.e., similar fineness and PSD), given that the glycol organic compounds function essentially as reducers of electrostatic forces during comminution with negligible effect on cement hydration and strength gain (Engelsen, 2008; Mejeoumov, 2007; Assaad, 2015; Jolicoeur et al., 2007).

Limited information exists in literature regarding TEA and TIPA degradation during clinker processing, and most importantly the extent to which this could alter cement hydration processes and properties following real-scale grinding. In 1998, Jeknavorian et al. (1998) evaluated the recovery rates of TEA, TIPA, glycol, and phenol-based additives following clinker grinding by pyrolysis gas chromatography-mass spectrometry (pyr-GC/MS). The cement samples were obtained by inter-grinding clinker with 200 g/ton of each additive using 13-L rotating ball mill with temperature control. The recovery rate was found to be highly dependent on the grinding process and binding capacity of various additives. Hence, when using relatively short grinding process conducted at ambient temperature, the additives were recovered at different rates varying from 30% to 85% of their originally added concentration. In contrast, in the case of a longer, more intense grinding conducted at 105  $^{\circ}\text{C}$ , the alkanolamine and glycol-based additives yielded extremely low recovery rates less than 25%. This was attributed to a combination of phenomena including (a) polymer decomposition during the milling process; (b) formation of complexes or salts at the fractured cement particles undetectable by the pyr-GC/MS; (c) thermal polymer degradation favored by elevated mill temperature and partial evaporation; and (d) irreversible adsorption of the grinding aid on the heated steel chamber and balls (Jeknavorian et al., 1998).

It is important to note that no attempts were made by Jeknavorian et al. (1998) to determine whether the drop in recovery rate is associated with equivalent decrease in additive functionality during grinding or changes in cement properties. In other words, it is not clear whether the discrepancies between

laboratory and industrial mills could be attributed to alteration in additive performance as a result of intense energy and/or elevated temperature. For indication, the materials temperature during real-scale grinding should not exceed 105  $^{\circ}\text{C}$ , in order to avoid dehydrating too much gypsum and causing false set (Alsop, 2001). Cement manufacturers attribute particular attention to limit temperature rise by cooling the exterior of the mill with water or fresh air, and sometimes pulverizing water inside the mill.

Generally, the boiling points and decomposition temperatures of majority of organic compounds used in commercial additives are substantially higher than the cement grinding temperature; for example, the boiling points for TEA and TIPA are 325 and 355  $^{\circ}\text{C}$ , respectively. However, earlier studies conducted by Mishra (2012) showed that the thin molecular additive layers adsorbed onto newly fractured cement particles could decompose irreversibly in presence of oxygen during grinding at temperatures lower than 100  $^{\circ}\text{C}$ , forming volatile secondary compounds. Such decomposition could occur at temperatures as low as 50  $^{\circ}\text{C}$  when the cement is stored for weeks in silos filled with hot products (Mishra, 2012). Assaad and Asseily (2011) reported that capability of water reducing chemicals to preserve efficiency after being inter-ground with clinker depends on grinding parameters (i.e.,  $E_c$  and temperature) along with the molecule type, dosage rate, and whether addition is made in liquid or powder form. On average, the loss in water reduction due to  $E_c$  registered under ambient temperature varied from 4% to 12% for lignosulphonate and polynaphthalene sulphonate polymers, while this increased up to 16% for polycarboxylate-based ones. An additional loss varying from 1% to 3.5% was noted when the cement was ground at elevated temperature of 95  $^{\circ}\text{C}$  (Assaad and Asseily, 2011).

This paper is the continuation of a research project undertaken to bridge the gap between laboratory and real-scale grinding of cement clinker. It is divided into two main phases; the first compares the data collected during real-scale clinker grinding in closed-circuit tube mill operating at around 90 ton/h to that obtained under laboratory conditions. Tests were run using commercially available alkanolamine-based additive at 350 and 460 g/ton of produced cement. Given the remarkable discrepancies obtained, the second phase aims at proposing various charts that facilitate predicting the effect of  $E_c$  and/or elevated temperature on degradation (i.e., loss in performance) of additive used. A series of grinding tests was performed using 50-l laboratory mill at different cement fineness, and properties evaluated included water demand, setting time, heat of hydration, and strength development. This paper can be of particular interest to cement manufacturers and technologists dealing with polymer degradation during clinker processing as well as variability of cement hydration reactions and properties due to organic additions.

## 2. Materials, grinding mills, and testing procedures

### 2.1. Materials

Industrial clinker used for the production of ASTM C150 Type I cement (ASTM C150, 2012), natural pozzolan meeting the

**Table 1**  
Chemical composition of clinker, pozzolan, and gypsum materials.

	SiO <sub>2</sub> ,%	Al <sub>2</sub> O <sub>3</sub> ,%	Fe <sub>2</sub> O <sub>3</sub> ,%	CaO,%	MgO,%	SO <sub>3</sub> ,%
Clinker	20.6	6.32	4.3	64.2	1.86	0.22
	C <sub>3</sub> S = 54.8%; C <sub>2</sub> S = 17%; C <sub>3</sub> A = 9.4%; C <sub>4</sub> AF = 12.5%; Loss on ignition = 1.06%; Na <sub>2</sub> O <sub>eq</sub> = 0.4%; Free lime = 0.3%; Specific gravity = 3.14					
Pozzolan	73.2	15.3	4.4	2.8	2.25	0.13
	Loss on ignition = 3.22%; Moisture content = 0.06%; Na <sub>2</sub> O <sub>eq</sub> = 1.05%; Pozzolan activity index with Portland cement at 28 days = 82.4%					
Gypsum	2.7	0.53	0.39	32.5	1.5	43.1
	Free water (T < 45 $^{\circ}\text{C}$ ) = 0.03%; Combined water (T < 230 $^{\circ}\text{C}$ ) = 15.6%; Carbon dioxide = 3.7%					

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