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The mechanochemical process and properties of Portland cement with the addition of new alkanolamines

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ABSTRACT

Grinding aids have been researched and used for decades to reduce energy consumption in the process of cement production. In this work, the effects of new alkanolamines, such as N,N-bis(2-hydroxyethyl)isopropanolamine (DEIPA), N,N-bis(2-hydroxypropyl)-2-aminoethanol (EDIPA) and N,N,N′,N′-tetrakis(2-hydroxyethyl)ethylenediamine (THEED), on the grinding, flowability and physical properties of Portland cement were investigated. The results indicated that DEIPA and EDIPA could significantly decrease the sieve residues (45 μm), and improve the flowability of the cement powder. They could decrease the compressibility and the magnitude of the pressure drop of the cement at a definite normal stress, thereby preventing cement particles from agglomerating. Notably, DEIPA and EDIPA increased both the early (3-day) and late (28-day) compressive strengths. DEIPA can be used as an accelerator to shorten the setting time. THEED just provides a greater contribution to the late compressive strength.

1. Introduction

Since 1824, cement has been one of the most important, irreplaceable and traditional building materials. The world cement production reached 4.18 billion tons in 2014. However, large amounts of CO_2 are released and limestone and energy are consumed during the cement manufacturing process. Approximately 0.98 tons of CO_2 is released during the manufacturing of 1 tons of clinker, the primary component of cement. Approximately 40% of the total electric energy (110 kW h/ton cement) is consumed for final cement clinker grinding [1–3]. Hence, the cement industry is facing severe challenges with respect to global warming and the present energy crisis.

Increasing amounts of chemical additives have been used in the process of cement grinding [4–6]. In the process of comminution and mixing, positive and negative charges are created on the newly fractured surfaces, leading to the agglomeration of particles due to van der Waals forces and electrostatic attraction. At the same time, coatings form on the surface of grinding media, which reduces the grinding efficiency [7,8]. On the one hand, chemical additives are generally adsorbed on the particle surfaces to decrease the hardness of the particle surfaces and better disperse fine particles by neutralizing charges and screening attractive forces [9,10]. On the other hand, some chemical additives can significantly affect the physical and mechanical properties of cement because of their physical and chemical interactions with hydrates and/or unhydrated phases rather than increasing the fineness of the particles [11–13]. The chemical additives used include alkylamines, alcohols, water-soluble polymers and inorganic salts. With developments in science and technology, alkanolamines have been synthesized and applied in the process of cement grinding. At present, the most widely used alkanolamines contain triethanolamine (TEA) and triisopropanolamine (TIPA). However, TEA increases only the early compressive strength, particularly before 3 days, and even decreases the strength in later stages [14]. TIPA improves only the late strength significantly [14–16]. Subsequently, new alkanolamines, such as DEIPA, EDIPA [17] and THEED [18] were developed.

The primary objective of this work is to investigate the effects of new alkanolamines on the grinding efficiency, flowability, phase structure, and physical and mechanical properties of Portland cement. The effects of DEIPA, EDIPA and THEED on the grinding efficiency were examined in terms of sieve residues (45 μm sieve size), specific surface area and particle size distribution. The changes in the angles of repose, the basic flowability energy (BFE) and specific energy (SE) were also examined to evaluate fluidity of the ground cement. The compressivity and air permeability of ground cement were measured to evaluate agglomeration during storage. The quantity of water required for normal consistency, setting time and strength were measured to evaluate the physical and mechanical properties of the ground cement.

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2. Materials and methods

2.1. Materials

2.1.1. Raw materials

The clinker and gypsum used in this work were obtained from a Chinese cement company. The chemical and mineralogical compositions of the clinker are shown in Table 1. TEA and TIPA were obtained from Jiaxing Jinyan and Fushun Jiahua, respectively, with purities of 85%. DEIPA, EDIPA and THEED were synthesized in the laboratory, and their molecular structures are shown in Fig. 1.

2.1.2. Sample preparation

Mixtures of clinker, gypsum and different amounts of the alkanolamines were ground using a laboratory batch ball mill with dimensions of 500 mm × 500 mm. The dosages of DEIPA, EDIPA or THEED were 0.01%, 0.015%, 0.03% and 0.05% by weight of the mixture of the clinker and gypsum. The dosage of TEA or TIPA was 0.015% as a reference. First, 4 kg of a mixture of 95% clinker and 5% gypsum was weighed and placed into the ball mill. Then, one of the chemical additives was added to the ball mill prior to initiating grinding of the mixture. Finally, the mixture was ground for 15, 20, 25, 30 and 35 min to prepare cement powder.

2.2. Characterization methods

2.2.1. Sieve residue of the cement

To investigate the effect of alkanolamines on the fineness of the cement, the sieve residue was measured using a negative pressure sieve analyzer with a sieve size of 45 μm and a pressure in the range of 4000–6000 Pa, according to the Chinese standard GB/T1345-2005.

2.2.2. Specific surface area of the cement

The specific surface area of the cement was measured using the Blaine method in accordance with the Chinese standard GB/T8074-2008.

2.2.3. Particle size distribution

The particle size distribution was measured by laser diffractometry in a Microtrac S3500SI.

2.2.4. Flowability of the cement

The angle of repose of the powder was measured using the injection method in FT-104B according to the Chinese standard GB11986–89. Two-hundred grams of cement was weighed and then dropped onto the experimental desk through a funnel to form a cone. The radius (R) and height (H) of the cone were measured. Finally, the angle of repose (α) was calculated according to Eq. (1).

$$\tan\alpha = \frac{H}{R} \tag{1}$$

Table 1
The chemical compositions for the clinker and gypsum and mineral compositions calculated by Bogue for the clinker.

Clinker	Loss	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O	K_2O
Chemical composition	0.46	65.70	21.60	5.51	3.39	1.65	0.41	–	–
Mineral	C_3S	C_2S	C_3A	C_4AF	f-CaO	KH	n	p	
composition	60.76	16.96	7.24	10.67	0.42	0.91	2.58	1.42	

Note: KH represents lime saturation factor, n represent silica ratio, P represent alumina ratio.

2.2.5. BFE, SE, compressibility and air permeability of the cement

BFE, SE, density and the value of the pressure drop of air through the ground powder were measured at a definite normal stress using a multifunctional powder tester in FT-4 (Freeman Technology). The compressibility was calculated according to Eq. (2), where ρ_0 is the density at a stress of 0 kPa and ρ_p is the density at a definite normal stress.

$$\text{Compressibility} = \frac{\rho_p - \rho_0}{\rho_0} \times 100\% \tag{2}$$

2.2.6. X-ray powder diffraction

The phase composition was investigated using a Rigaku SmartLab 3000A diffractometer with Cu K_α radiation ($\lambda = 0.154$ nm). The X-ray tube was operated at 35 kV and 30 mA. The optical configuration included a fixed divergence slit (1/2°) and a D/teX Ultra detector. The measurements were performed using a θ–θ reflection geometry. Data were collected from 10° to 70° in continuous mode.

2.2.7. Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FT-IR) was also used to analyze the phase compositions by studying the molecular vibrations for cement. FT-IR spectra were recorded on a Thermo Scientific Nicolet IS5 spectrometer over the range of 400 to 4000 cm^{-1}. KBr was used as a reference. The ratio of KBr to cement was 1:50. The cement and KBr were mixed and then pressed into a solid pellet before being measured.

2.2.8. Physical properties

The setting times and normal consistency of the cement paste were determined in accordance with the Chinese standard GB/T1346 using a Vicat apparatus. Cement paste was prepared from cement and water. The ratio of water to cement was 0.5.

2.2.9. Mechanical properties

The compressive and flexural strengths of mortars were measured according to the Chinese standard GB/T17671-1999. Mortars were prepared with Chinese standard sand, cement and water. A cement powder/sand/water weight ratio of 1:3.0:0.5 was employed.

3. Results and discussion

3.1. Effect of new alkanolamines on the fineness of the cement

The sieve residues (45 μm) are shown in Fig. 2 for cements without and with different alkanolamines ground for 15, 20, 25, 30 and 35 min. The sieve residues decreased with time for all samples. The new alkanolamines (DEIPA, EDIPA and THEED), particularly DEIPA and EDIPA, decreased the sieve residues for the cement under the same grinding conditions. The sieve residues decreased with increasing dosages of the alkanolamines at the same grinding time for DEIPA and EDIPA. Compared to DEIPA and EDIPA, THEED showed a slight effect on the sieve residue. The differences in sieve residues between the blank cement and cements containing EDIPA increased with increased grinding time. EDIPA had a greater effect on the grinding process at the later stages. Compared with TEA, the effects of THEED on the sieve residue were much less at the later age.

The specific surface area was also used to characterize the fineness of cement. The specific surface areas are shown in Fig. 3 for the cements without and with 0.015% alkanolamines. The specific surface area increased with grinding time for all samples. The specific surface area was lower for the cement containing DEIPA or EDIPA while it was higher for the cement containing THEED, compared to the blank cement ground for a given time.

In the initial stage of grinding, particles are shattered and new cracks, active sites and electrostatic charges occur on the fractured

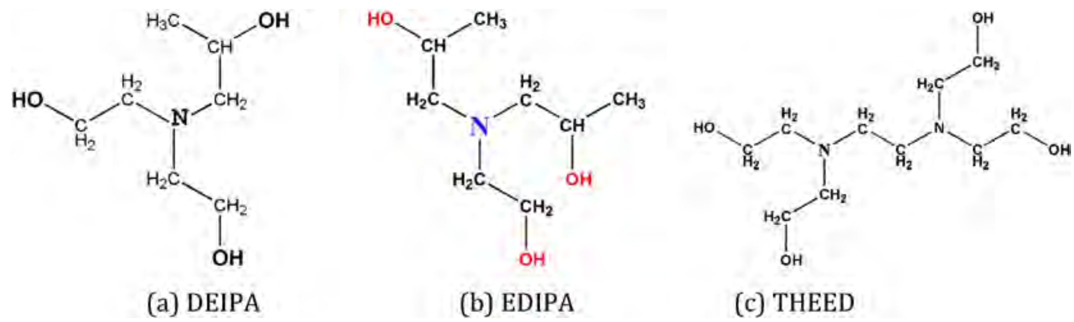

Fig. 1. The molecular structure of DEIPA, EDIPA and THEED.

surface under the mechanical force, which leads to the new cracks closure and trends toward particle aggregation. In the middle and later stages, particles are kept fine with reunion of fine particles due to electrostatic charges. DEIPA and EDIPA are trialkanolamines with their structures similar to that of TEA [14] (see Fig. 1). DEIPA and EDIPA have characteristics of polarity, an asymmetric structure and a charge center. On the one hand, DEIPA and EDIPA are adsorbed on the fractured surface and shield the active sites to decrease the external stress needed by cracks growth and to prevent particles from agglomerating in the initial stage of grinding. On the other hand, DEIPA and EDIPA inhibit the reunion of fine particles by decreasing electrostatic charges in the latter stage. THEED is an alkanolamine with special structural features of the two amines. Two nitrogen atoms at both ends of the molecule are adsorbed on the surfaces of two particles. The spatial dimension of THEED is much less than size range of powder particle. Hence, the grinding effect of THEED is less when compared to DEIPA and EDIPA.

3.2. Particle size distribution

The particle size distribution curves for cements without and with 0.015% alkanolamines ground for 25 min are shown in Fig. 4. The particle size distribution largely complies with a normal distribution for all samples. The particle size distribution curves largely overlap for the cement with alkanolamines, except for the blank cement. The volume of the particles with sizes in the range of 1 to 5 μm is greater for the blank cement, compared to the other samples. However, the sieve residues (45 μm) and specific surface area are lower for the cement containing DEIPA or EDIPA, compared to the blank cement. This result may be attributed to the aggregation of fine particles due to electrostatic charges.

3.3. Flowability of the ground cement

The angle of repose is primarily used to characterize the flowability of powders. A powder with a smaller angle of repose has greater

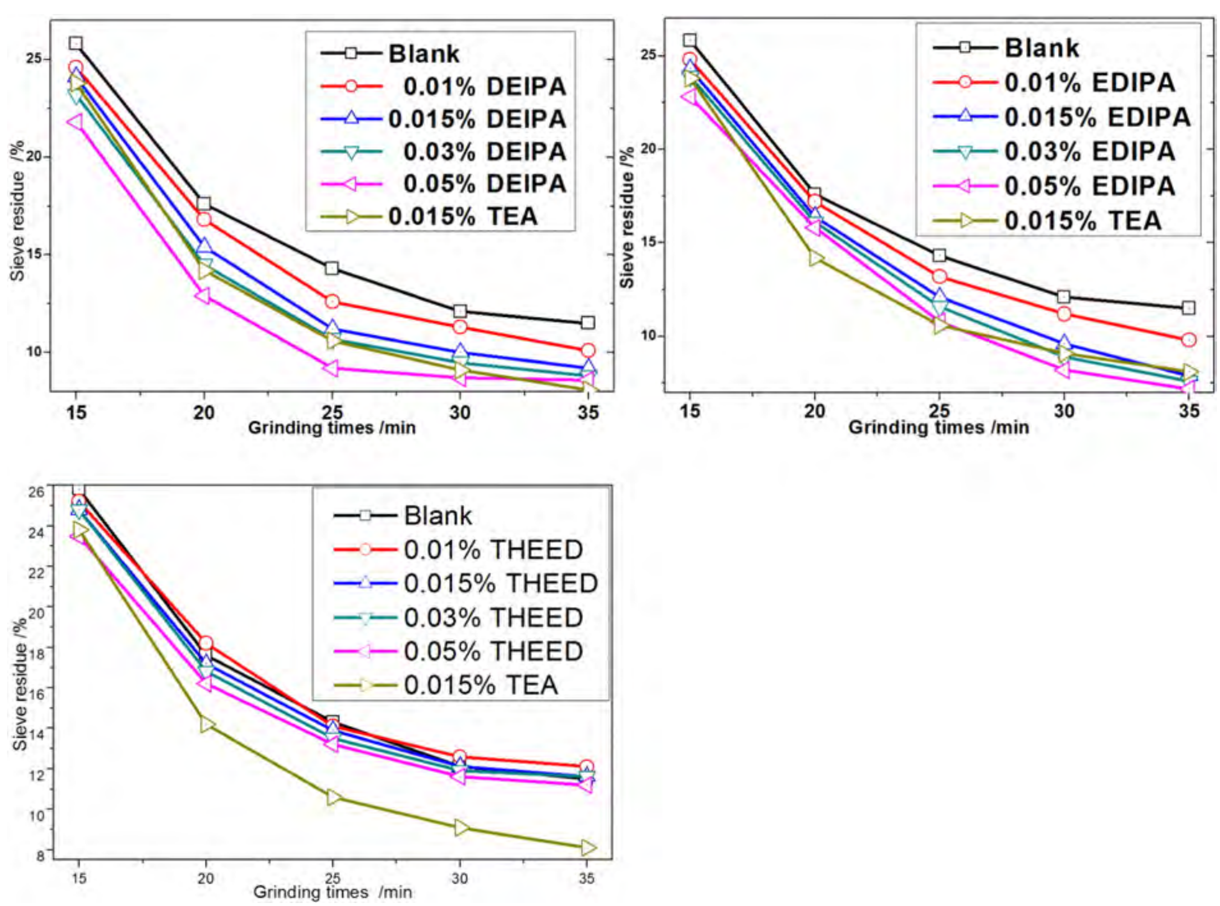

Fig. 2. The sieve residues (45 μm) of cements powder ground without and with alkanolamines for different times.

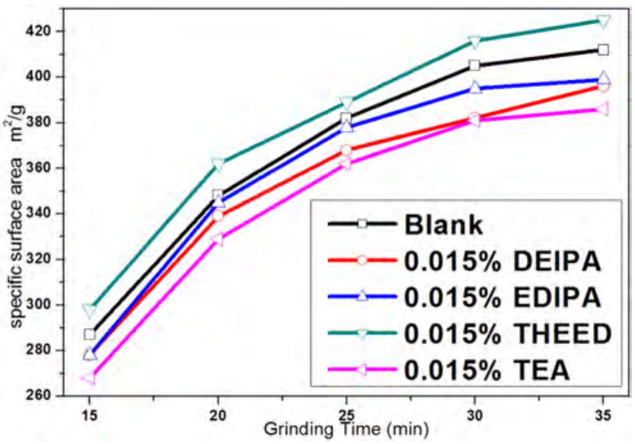

Fig. 3. Blaine specific surface area of cement powder without and with alkanolamines ground for different times.

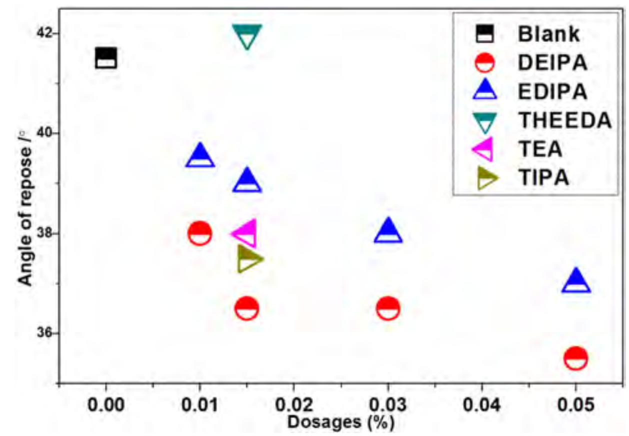

Fig. 5. The angles of repose of cements without and with different alkanolamines ground for 25 min.

flowability. Fig. 5 presents the angles of repose for cements without and with alkanolamines ground for 25 min. DEIPA, EDIPA, TEA and TIPA significantly reduce the angles of repose of the powder. The angles of repose decrease with the increase of alkanolamines, except for THEED which shows a small adverse effect on the angle of repose of the powder. For a given dosage of alkanolamines, the angle of repose is the smallest for the cement with DEIPA.

BFE is required to move pretreated and stable powder with a fixed flow mode and flow rate, and it is also an important parameter for characterizing changes in flowability. A powder with a lower BFE has greater flowability for pneumatic conveying, loading and packing of cement powder. New alkanolamines, such as DEIPA and EDIPA, result in a significant decrease in BFE, whereas THEED shows a slight effect (shown in Table 2) when these alkanolamines are at a dosage of only 0.015%. The BFEs of the cements with DEIPA and EDIPA were reduced by 26% and 16%, respectively, compared to the blank cement, illustrating that DEIPA and EDIPA favor the pneumatic conveying, loading and packing of cement powder.

SE is the shear flow of a unit mass of powder that is required to move a pretreated sample with a slight shear and lift mode. SE is primarily used to characterize the flowability of a powder in a loose heap, such as rubbing or mechanical interlocking between the particles under low stress. As shown in Table 2, the blank cement and cements with DEIPA and EDIPA have SEs of 5.74, 4.24 and 5.15 MJ/g, respectively, which indicates that DEIPA and EDIPA reduce the frictional resistance between the particles by 26.1% and 10.3%, respectively. The SE of the cement with THEED is close to that of the blank cement.

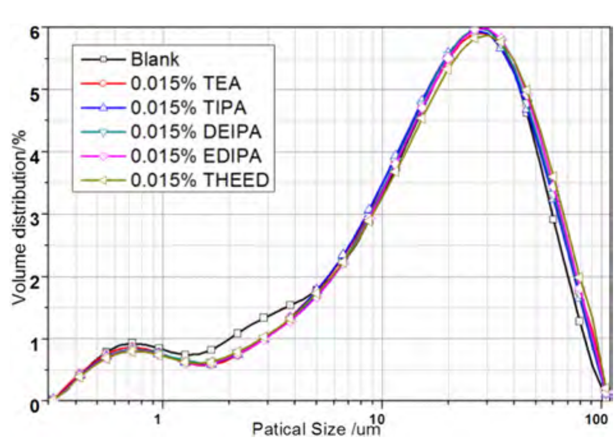

Fig. 4. Particle size distribution of cements powder without and with 0.015% alkanolamines ground for 25 min.

To achieve the flow of powder, the gravitational force on the powder particles must be larger than the resistance between the particles, such as frictional force, adhesion force and electrostatic force [19]. The above results show that the alkanolamines can decrease the internal resistance among particles to improve the flowability. As polar organics, the adsorption capacity of alkanolamines on the surface of cement particles depends on their molecular structure. Hence, the effect of THEED is different from the effects of DEIPA and EDIPA.

3.4. Compressibility and air permeability of the ground cement

Compressibility is the ability of plastic deformation of powder caused by external pressure. It is affected by the chemical composition, particle shape and structure, size and distribution, and bulk density of powder. In addition, compressibility is also related to the van der Waals forces and electrostatic attraction force between the particles [19]. The compressibility percentage values are shown in Fig. 6 for the cements without and with 0.015% new alkanolamines ground for 25 min. Compared to the blank cement, the compressibility percentage values are significantly lower for the cement with DEIPA or EDIPA at a definite normal stress (see Fig. 6). However, THEED has no effect on the compressibility. The addition of DEIPA and EDIPA is useful for preventing the agglomeration of particles during cement storage.

The air permeability of a powder reflects the pore sizes and distribution among the particles. A smaller pore size results in a higher pressure reduction due to pore resistance and thus a worse air permeability. Fig. 7 shows the values of the air pressure drop through the powder with a 2 mm/s air velocity at different applied normal levels of stress. The values of the pressure drop increase with increasing stress for all samples. When the stress is in the range of 6–8 kPa, the air permeability of the cement with the new alkanolamines is equal to that of the blank cement. However, the values of the pressure drop are lower for the cement with DEIPA and EDIPA compared to the blank cement if the stress is greater than 8 kPa. With respect to the cement containing THEED, its air permeability remains identical to that of the blank cement.

Table 2
Powder performances of cements without and with 0.015% different alkanolamines ground for 25 min.

Kinetics test	Blank	DEIPA	EDIPA	THEED
BFE (mJ)	111	87.8	95.3	108.9
SE (MJ/g)	5.74	4.24	5.15	5.65
Compressibility, CPS15 (%)	20.3	17.9	19.0	20.1
Pressure Drop, PD15, 0.5 (mbar)	25.5	22.9	24.4	25.3

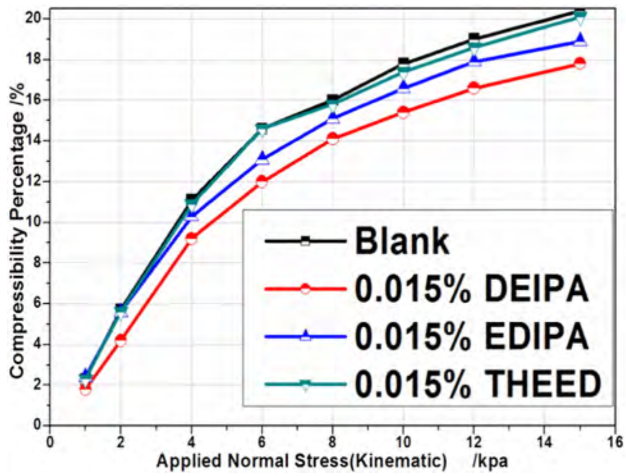

Fig. 6. Compressibility percent of different samples at different applied normal stresses.

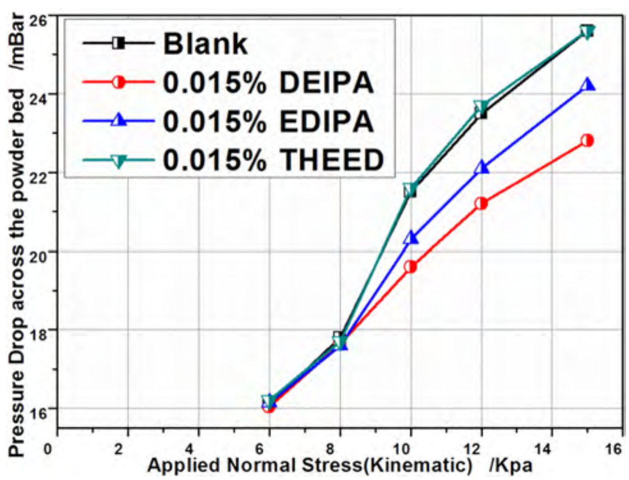

Fig. 7. The values of pressure drop across the powder bed with 2 mm/s air velocity at different applied normal stresses.

3.5. X-ray diffraction analysis

Figs. 8 and 9 present the full XRD patterns and characteristic patterns for the cements without and with 0.015% alkanolamines ground for 25 min. The phase compositions remain fairly constant for all cements. The phase compositions primarily consist of C_3S, C_2S, C_3A, C_4AF, gypsum($CaSO_4.2H_2O$) and f-CaO. However, the diffraction peak of C_3A at $2\theta = 26.68°$ disappears or weakens for the cement containing alkanolamines (Fig. 8(a)). The diffraction peak at $2\theta = 27.55°$ appears in the cement containing THEED due to β-C_2S (Fig. 8(b)). The characteristic peak of gypsum becomes broader and more intense for the cement with alkanolamines (Fig. 9). The characteristic peak of f-CaO also changes significantly for the cements with TIPA, THEED and EDIPA. The mechanochemical action means physical and chemical changes for samples under a mechanical force. The physical changes include apparent density, true density, and specific surface area, whereas the chemical changes include crystal defects, degree of crystallization, amorphization and dehydration [20,21]. The addition of additives was reported not only to inhibit the reunion of particles by balancing the unsaturated bond on the shattered particles but also to decrease the surface energy and degree of lattice distortion [22].

3.6. Fourier transform infrared spectroscopy analysis

FT-IR spectroscopy is one of the most useful techniques for characterizing molecular structures. FT-IR is typically used to monitor the hydration of cement. Fig. 10 presents the FT-IR spectra for the cements without and with 0.015% alkanolamines ground for 25 min. Three bands are observed in the range of 3700–3100 cm^{-1}. The band at 3639 cm^{-1} is attributed to the stretching vibration of O–H from $Ca(OH)_2$, with the band position approaching 3640 cm^{-1} as reported by M. M. Radwan [23], demonstrating that small portions of the fine powder appear to hydrate from the adsorption of water from the air. Gypsum shows two bands at 3411 cm^{-1} and 1624 cm^{-1} (3410 and 1623 cm^{-1} were reported in reference [24]) due to water molecules. The peak located at 1119 cm^{-1} (1120 cm^{-1} reported in [24]) is due to ν_3-$[SO_4]^{2-}$ from gypsum. The bands at 930 cm^{-1} and 519 cm^{-1} correspond to the Si–O asymmetric stretching vibration (ν_3) and the Si–O out-of-plane bending vibration (ν_4) of silicate, respectively, in good agreement with the values reported in the literature [25]. Compared to the blank cement, the intensities of the water molecular vibration bands, the $[SO_4]^{2-}$ vibration band from gypsum and the $[SiO_4]^{4-}$ vibration bands from silicates decrease for the cements with additives. The FT-IR and XRD results demonstrate that alkanolamines could accelerate the amorphization of gypsum and silicate phases.

3.7. Physical and mechanical properties of cement mortars

The fineness of cement is known to be a critical parameter for its properties [26,27]. The quantities of water required for a normal

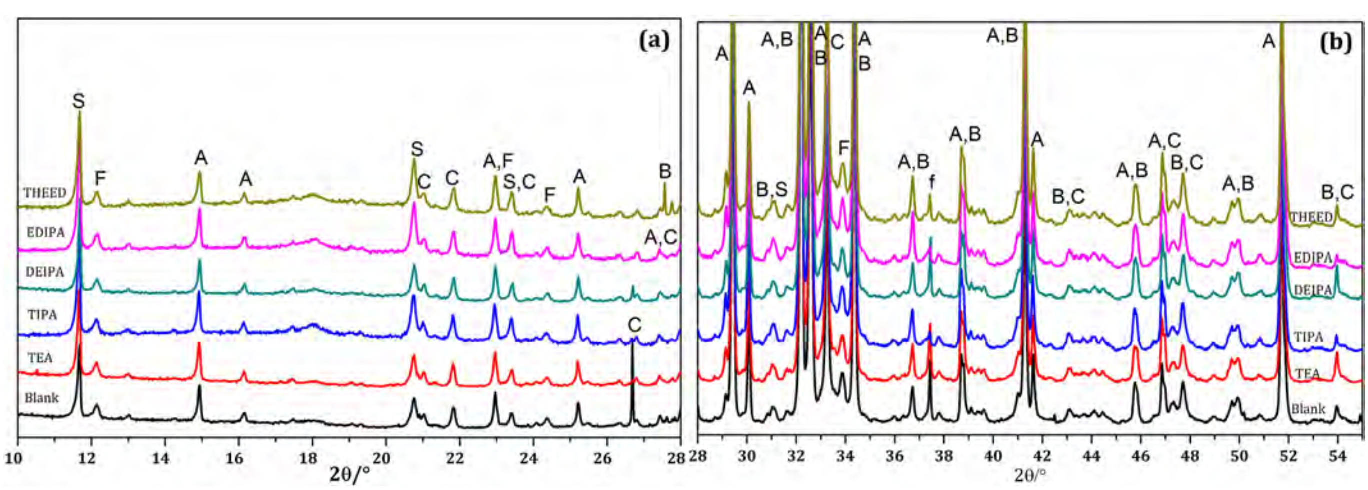

Fig. 8. XRD patterns of samples with different alkanolamines ground for 25 min at the windows of 10–28° 2θ (a) and 28–55° 2θ (b). The main reflection peaks of C_3A (C), Ferrite (F), gypsum (S), C_3S (A), C_2S (B) and f-CaO (f) are labeled.

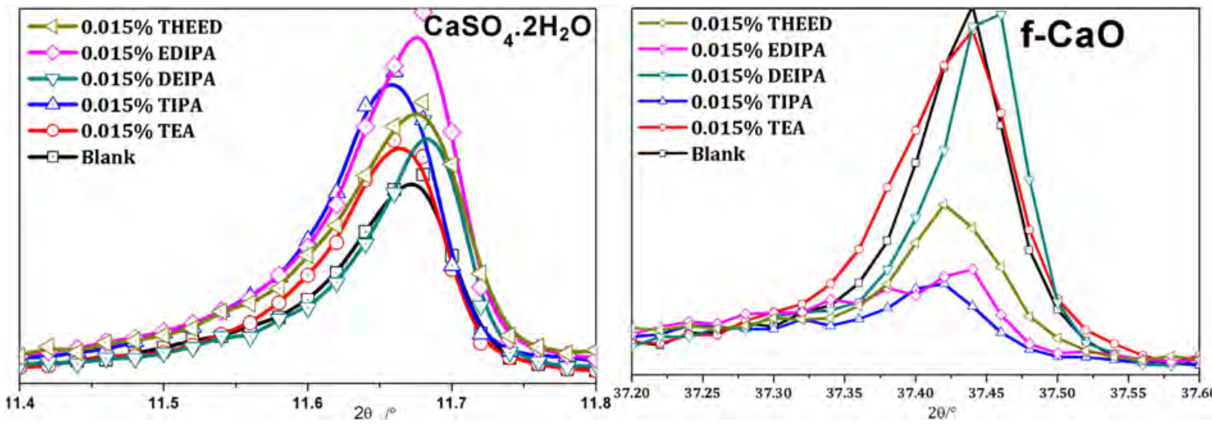

Fig. 9. Contrast of X-ray diffraction peak intensity of samples with alkanolamines ground for 25 min.

consistency, setting time and strength are shown in Table 3. As a result of the addition of alkanolamines, the quantity of water required increases for a normal consistency. Compared to the blank cement, the initial and final setting times are slightly decreased for the cement with alkanolamines, particularly TEA and DEIPA. TEA has been used as a component of admixtures for Portland cement for many years [28], typically being added at a low dosage as an accelerator due to its acceleration of the C_3A reaction. Hence, DEIPA can replace TEA as an accelerator for Portland cement. In the case of TIPA, it has been reported to increase the setting times by approximately 15% [28]. However, a small amount of TIPA slightly decreases the setting times in this work.

TEA significantly affects the compressive strength of the cement paste at 3 days, whereas TIPA notably improves the compressive strength at 28 days. TIPA can remain in solution for a sufficient period of time and catalyze the hydration of ferrite to form calcium sulfoaluminate hydrates after all of the free gypsum has been consumed [28,15]. TEA is mainly adsorbed on the portlandite surface within the first hours of hydration and accelerates the hydration of C_3A, but TIPA does not adsorb [28,29]. Hence, TEA provides a larger contribution to the early compressive strength, whereas TIPA mainly affects the late compressive strength. In addition, as with TIPA, THEED exhibits a significant effect on the late compressive strength. The new alkanolamines (DEIPA and EDIPA) are different from TEA, TIPA and THEED. The new alkanolamines enhance not only the early compressive strength but also the late compressive strength. However, the alkanolamines show only a slight effect on the flexural strength of the cement.

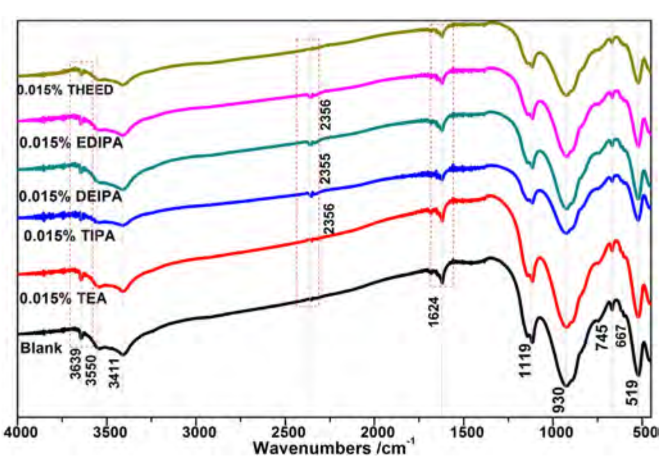

Fig. 10. Fourier transform infrared spectrum of different samples.

4. Conclusions

As with TEA, the new alkanolamines, such as DEIPA, EDIPA and THEED, are highly effective in the grinding of Portland cement, decreasing the sieve residues (45 μm). Compared to the blank cement, the alkanolamines significantly decrease the amount of superfine particles in the range of 1–5 μm.

Compared to the blank cement, the alkanolamines, except for THEED, significantly reduce the angle of repose of the ground cement. When the dosages of these alkanolamines are 0.015% by weight of the blank cement, they decrease the angle of repose in the order DEIPA > TIPA > TEA > EDIPA > THEED. To improve the powder flowability, the new alkanolamines, particularly DEIPA, decrease the BFE and SE. DEIPA decreases the BFE and SE by 20.9% and 26%, respectively. In addition, the compressibility and value of the pressure drop are enhanced with an increase in normal stress up to 15 kPa. At a constant normal stress greater than 8 kPa, DEIPA and EDIPA clearly reduce the compressibility and the value of the pressure drop to improve the agglomeration during the storage of the cement.

The mineral compositions of the cements do not change during grinding. The XRD and FT-IR results show that alkanolamines accelerate the amorphization of gypsum, being added at the same low dosages.

The amounts of water required for a normal consistency are slightly greater for the cements containing 0.015% alkanolamines. In addition, the initial and final setting times are decreased. DEIPA decreases the initial and final setting times by 20 and 25 min, respectively. As with TEA, DEIPA can be used as an accelerator. DEIPA and EDIPA enhance both the early and late compressive strengths, while THEED just provides a greater contribution to the late compressive strength. The alkanolamines exhibit only a slight effect on the flexural strength.

Acknowledgments

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Table 3
Physical and mechanical properties of cements without and with 0.015% different alkanolamines ground for 25 min.

Kinds of additive	Standard consistency water demand/%	Setting time/min		C/F strength/MPa			
		Initial	Final	3 days		28 days	
Reference	26.5	160	210	38.7	7.2	57.1	8.5
TEA	27.7	140	180	41.7	6.9	57.5	8.4
TIPA	27.2	150	205	39.4	7.3	61.2	8.6
DEIPA	27.3	140	185	41.3	7.3	62.4	8.5
EDIPA	27.6	145	190	40.7	7.6	61.1	8.8
THEED	27.7	155	195	39.9	7.1	61.3	8.6

Note: C/F—Compressive strength/Flexural strength.

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