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Jet mill grinding of portland cement, limestone, and fly ash: Impact on particle size, hydration rate, and strength

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ABSTRACT

While the majority of commercial ordinary portland cement (OPC) is ground using a ball mill or a vertical roller mill, other industries have shown that jet mill grinding can be an alternative approach for grinding materials. This paper investigates the potential application of jet mill grinding for two systems. The first system is a blend of OPC and 15% limestone, and the second system is a blend of OPC and 40% fly ash. It was observed that when jet mill grinding is used, the average particle size of the powders is decreased to approximately 4 μm or less with a narrower particle size distribution than that achieved using ball milling. In addition to evaluating the size and shape of the particles obtained from the jet mill grinding process, this paper focuses on evaluating, using isothermal calorimetry, the effect these changes in particle size and distribution have on the extent and rate of hydration as well as their effect on the compressive strength of cement pastes or mortars.

This study also investigated differences between inter-grinding and blending separately ground materials to form an OPC/limestone mixture. Both inter-ground and separately ground OPC/limestone mortars demonstrated an accelerated hydration at early ages accompanied by an increase in early age strength. This appears to be primarily due to the increased surface area of the finer particles that provides more available surface for the hydration reaction. The inter-grinding appeared to be more effective than grinding the materials separately because an improved graded particle size distribution was obtained. The inter-ground OPC/limestone mixture shows accelerated initial hydration at water to powder ratios (w/p, where powder = cement + limestone) of 0.50 and 0.35 when compared with the samples before grinding. At the lower w/p of 0.35, the OPC/limestone mixture appears much more efficient. In the OPC/fly ash mixture, jet mill grinding also accelerates the rate of hydration and strength development.

1. Introduction

Several different techniques are available to control the fineness of cement-based materials. These include a series of non-destructive approaches (i.e., sieving [1–3], air classifying [2], or magnetic-extraction [3] methods) as well as destructive approaches (i.e., mechanical grinding methods [3–12]). The cement industry typically uses ball-mill grinding as the preferred method to reduce the size of clinker in cement manufacturing [3–9]. Vertical roller mill grinding [13] is being increasingly used. Roller milling [14] and airflow milling [15] have also been used to grind cements with the particle gradation and shape change dependent on the specific milling procedure. Research described in this paper examines the use of an alternative milling method, jet mill grinding, and in particular, the effects that changes in particle size and distribution resulting from jet mill grinding have on the extent and rate of hydration.

Jet mill grinding works by using multiple air jets or air streams to accelerate cement (or fly ash and limestone) particles from a very low velocity to the sonic velocity range. As collisions occur between cement and/or supplementary cement particles, the cement and any supplementary cementitious materials are ground. Jet mill grinding has been used in other industries as a method to modulate the particle size distribution [16–24]. When the jet mill is compared to the ball mill, the jet mill is able to grind materials to a smaller particle size (1–10 μm) with a narrower particle size distribution [5]. The fluidity of jet mill ground cement/limestone mixture and shape of cement particles have been examined

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[5], and it was found that after jet mill grinding, the fluidity of OPC/limestone pastes increased. The jet mill ground particles showed similar dimensions in orthogonal directions. Furthermore, when ball milling is used, the final product may be contaminated with fragments of the ball-milling media that are ground off or broken off during the milling process. Phase transitions may also occur in the material due to heat generated in the grinding process. This can be avoided with jet milling because the material being ground is itself the milling media, and minimal additional heat will be generated during the process because of the cooling effect of the jets [25]. The jet mill grinding process also has the capability to produce blended powders since multiple streams of material can be introduced into the jet mill simultaneously [26].

The potential for using jet-mill processing may be of particular interest now due to the growing interest in reducing the clinker content per ton of cement as a method to reduce CO_2 production. Blended cements that incorporate mineral powders are being increasingly developed due to their economic and environmental protection benefits [3]. In addition to the benefit of reducing CO_2 production, the use of cement/limestone or fly ash blends can improve performance [27]. For example, finer fly ash may improve mechanical properties and the hydration reaction without sacrificing workability [1–3,6–9,27,28]. Limestone fines have also been found to be effective in accelerating the rate of hydration or helping to control viscosity for self-consolidating concrete [5,10–12,27,29–31]. The jet milling approach may even open up the possibility of using previously land-filled materials as supplemental materials since regrinding may make these materials more chemically active.

2. Experimental methods

Research described in this paper focuses on measuring particle size distributions obtained by jet mill grinding of two cement-based mixtures: (1) OPC/limestone and (2) OPC/fly ash, and assessing the impact of the particle size distribution on the rate of hydration (using isothermal calorimetry) and on compressive strength of standard cylinder specimens. The following section describes how the materials were prepared and tested.

2.1. Jet mill grinding

A jet mill works by using multiple air jets or air streams to accelerate particles from low velocity to a very high velocity (sonic). Collisions between particles that are induced in this high-velocity environment occur in the center of the chamber and cause the grinding. An illustration of a typical particle path of a jet mill is shown in Fig. 1a (adapted from Ref. [26]). The material is fed into the milling device through a hopper at a constant rate set by the operator. As the material is fed into the system, it passes through the jet mill. The air streams accelerate the particles causing them to collide with one another. The collision causes the particles to fracture, and thereby to reduce their size. Unlike ball milling, there is a reduced risk of contaminating the product with milling media because the material itself is the milling media. The interior walls of the chamber can also be lined with materials that are highly resistant to abrasion to minimize powder contamination [26].

A Micron-Master Jet Pulverizer jet mill (0.61 m in diameter)was used in this study to grind the materials as shown in Fig. 1a and b. The materials to be ground were blended for 60 min in a 0.19 m^2 V-blender (this step is only conducted for blends). After blending, approximately 45 kg of the pre-blended material was transferred into the hopper for jet mill grinding. The feed rate was set at 9.07 kg/h. The pressure of air feeds to the mill was 552 kPa. Once all of the material passed through the jet mill, the materials were defined as having completed one-pass of grinding.

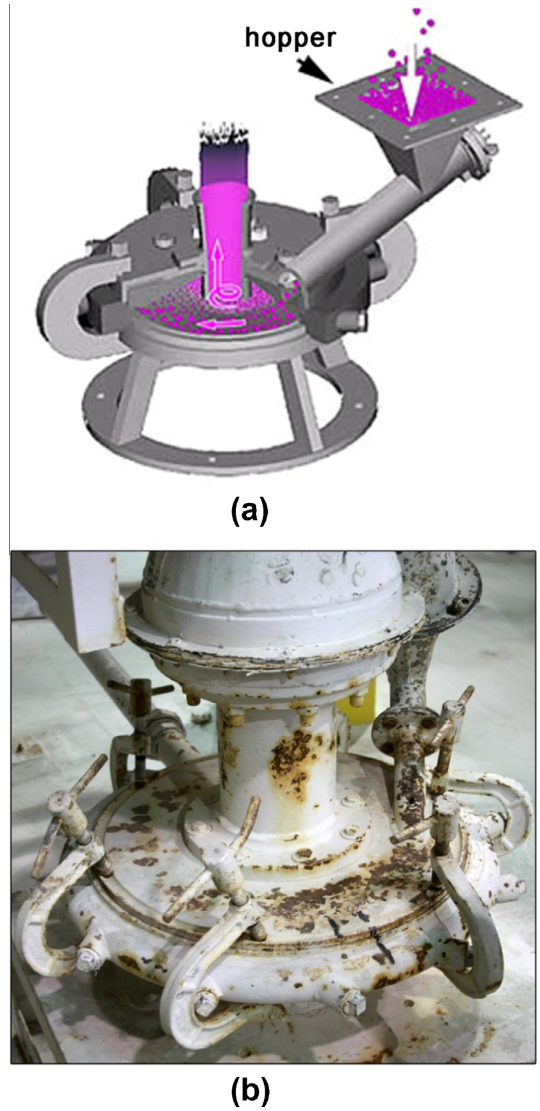

Fig. 1. (a) A conceptual illustration of a typical jet mill adapted from Ref. [26]. (b) A picture of the jet mill used to grind materials.

In this study, a maximum of three passes through the jet mill were performed to determine the appropriate grinding level for the OPC/limestone mixtures.

2.2. Constituent materials

Two samples of blended cements were investigated. One sample consisted of a blend of ordinary portland cement (OPC1) and a limestone powder. The second sample contained a different ordinary portland cement (OPC2) blended with Class C fly ash. The chemical compositions of the materials are listed in Table 1. The mineral compositions of the two cements were obtained from X-ray diffraction and the Rietveld fitting method.

The mixture proportions for the two samples are shown in Tables 2 and 3. The following notations were used: (1) C, L, and F were used to identify cement, limestone, and fly ash, respectively; (2) G in front of C, F, and L denotes a material ground by jet mill grinding; (3) the mass percentage of limestone or fly ash in each mixture was noted after C, F, and L except for cement reference (C); and (4) I and S denote whether the material was inter-ground (I) or ground separately (S). The inter-ground materials were blended and ground in the jet mill, whereas the separate-ground

Table 1
Compositions of OPCs, limestone, and fly ash.

	OPC1 (used in cement/limestone mixture)	OPC2 (used in cement/fly ash mixture)	Limestone		Fly ash	
C_3S (%)	69.17	55.52	$CaCO_3$ (%)	>99	SiO_2 (%)	38.71
C_2S (%)	0	26.45	SiO_2 (%)	0.8	Al_2O_3 (%)	19.15
C_3A (%)	4.72	4.47			Fe_2O_3 (%)	6.49
C_4AF (%)	18.86	8.05			CaO (%)	23.51
					MgO (%)	5.29
					SO_3 (%)	1.36
					Na_2O (%)	1.64
					K_2O (%)	0.58
					LOI (%)	0.30

Table 2
Mixture proportions used in cement/limestone mixture.

	C		C85/L15		GC85/L15		S-GC85/GL15		I-GC85/GL15	
w/p	0.5	0.35	0.5	0.35	0.5	0.35	0.5	0.35	0.5	0.35
OPC1 (g)	100	100	85	85	85	85	85	85	85	85
Limestone (g)	0	0	15	15	15	15	15	15	15	15
Water (g)	50	35	50	35	50	35	50	35	50	35
Silica sand (g)	100	100	100	100	100	100	100	100	100	100
SP (g)	0	0.9	0	0.9	0	0.9	0	0.9	0	0.9

Table 3
Proportions used in cement/fly ash mixture.

	C	C60/F40	GC60/F40	I-GC60/GF40
w/p	0.35	0.35	0.35	0.35
OPC2 (g)	100	60	60	60
Fly ash (g)	0	40	40	40
Water (g)	35	35	35	35
Silica sand (g)	100	100	100	100

materials were individually ground in jet mill and then blended outside of the jet mill.

The OPC/limestone blends contained 15% (by mass) limestone and were cast with water to powder ratio (w/p, herein p = cement + limestone) of 0.50 and 0.35. Specimens with the higher w/p of 0.50 were cast with water and the powder material only, while specimens with the lower w/p of 0.35also included polycarboxylate-based Type F high-range water reducing admixture, referred to as superplasticizer (SP). The OPC/fly ash blends containing 40% (by mass) fly ash were designed for testing at a w/p (herein p = cement + fly ash) of 0.35. In all the mortars, silica sand (ASTM Graded, U.S. Silica Corp.) was used as fine aggregate with a sand to powder ratio of 1:1 by mass.

2.3. Experimental methods

X-ray diffraction (XRD) characterization of powders was conducted using a Bruker D8 instrument with a Cu Kα source at 40 kV and 40 mA. Scanning electron microscopy (SEM) of OPC/limestone powders was performed using an FEI Quanta 3D with a field emission gun working at 15 kV. Specimens were Pd-coated prior to SEM observation to obtain a conductive surface. Particle size distributions (PSDs) were measured by Coulter LS32 laser sizer (Beckman Coulter Inc.) with a high reproducibility (<1%) [32]. Methanol was used as a dispersant. The densities required for PSD calculation were measured by a Micromeritics Accupyc 1330 pycnometer.

All mortars were prepared in a Hobart mixer following ASTM C 305. After casting, the specimens were prepared for testing. Approximately 15 g of each sample was placed in a glass vial with a temperature of 23 ± 0.1 °C for isothermal calorimetry using a TAM Air isothermal calorimeter with a stable baseline [33]. The compressive strength was measured using an FX700 with cylindrical specimens (approximately 50 mm diameter and 100 mm height) in accordance with ASTM C-39. Each test reported consists of the average of two specimens. All specimens were sealed until the desired age of testing, which were 1, 3, and 7 days for the cement/limestone mixture and 1, 3, 7, and 28 days for the cement/fly ash mixture.

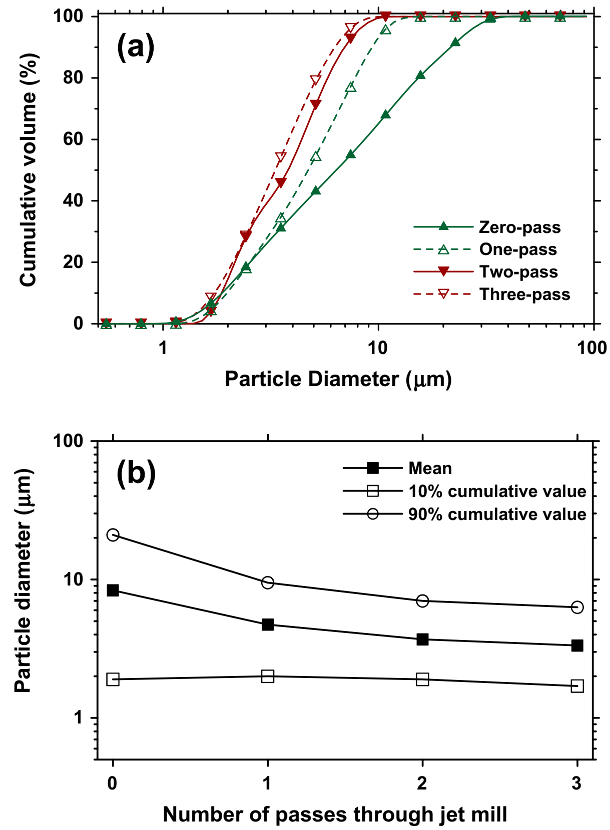

Fig. 2. (a) Particle size distribution and (b) corresponding mean particle diameter of cement/limestone blends with particle size corresponding to 10% and 90% cumulative volume after zero to three passes through jet mill grinding. The sample labeled zero-pass refers to the one only blended by V-blender with no grinding.

3. Results and discussion

3.1. Jet mill grinding

The extent of grinding was controlled by the number of passes the material made through the jet mill. Fig. 2a shows an example PSD of an OPC/limestone blend as a function of the number of jet mill passes. Fig. 2b and Table 4 shows the change in mean particle diameter and size distribution. The distribution is represented here as the difference between particle sizes corresponding to 10% and 90% cumulative volume limits, known here as the 80% limit.

Before grinding, the OPC/limestone mixture has a broad size distribution with 10% smaller than 1.9 μm in diameter and 90% smaller than 21.0 μm, i.e. with an 80% limit of 1.9–21.0 μm and a mean particle diameter of 8.3 μm. The first pass through the jet mill reduced the mean diameter to 4.7 μm with a reduction in the 80% limit to 2.0–9.5 μm. The second and third passes resulted in finer grinding to mean sizes of 3.7 and 3.3 μm and further narrowing of the 80% limit to 1.9–7.0 μm and 1.7–6.3 μm, respectively. The small changes in the mean particle diameter and distribution between 2 and 3 jet mill passes led to the selection

Table 4
Mean particle sizes and distribution over 0–3 passes through jet mill indicated by the difference between particle size corresponding to 10% and 90% cumulative volume.

Passes through jet mill	Particle size corresponding to 10% cumulative volume (μm)	Particle size corresponding to 90% cumulative volume (μm)	Mean particle size (μm)
0	1.9	21.0	8.3
1	2.0	9.5	4.7
2	1.9	7.0	3.7
3	1.7	6.3	3.3

of 3 jet mill passes for cement/limestone blends. Similarly due to small changes after 2 passes, 2 jet mill passes was used for cement/fly ash blends for the remaining experiments.

Typical shapes of the ground particles are shown in Fig. 3, demonstrating the finer particles sizes, narrower particle size distribution, and irregular shapes of the jet milled OPC/limestone powders. Similar grinding effects were observed in OPC/fly ash mixture, as described in Section 3.3.2.

3.2. Series I – OPC/limestone blends

3.2.1. XRD characterization

The XRD spectrum of inter-ground I-GC85/GL15 mixture in Fig. 4 is a combination of spectra from reference cement (C) and limestone (L) before grinding, demonstrating that jet mill grinding produces no discernible phase transitions.

3.2.2. Comparing inter-grinding and separate-grinding

Inter-grinding was compared with separate-grinding using the GC85/GL15 specimens. The mean particle size of separately ground cement and limestone S-GC85/GL15 was 2.9 μm, slightly lower than the mean particle size of the inter-ground material I-GC85/GL15 which was 3.3 μm. The particle size distributions of separately ground portland cement (GC) and limestone (GL) are shown in Fig. 5a for comparison.

When pastes were made using these materials, no significant difference was observed in the extent of the hydration reaction as indicated from the cumulative heat development (Fig. 5b) or the rate of hydration as indicated from the rate of heat development (Fig. 5c). The inter-ground specimens were observed to have an average 1-day strength that was 20% higher than specimens cast from materials ground separately (even higher than the control cement) (Fig. 5d). The difference in strength is likely due either to the preferential grinding of limestone resulting in smaller particles that are better distributed as nucleation sites or to an improvement in particle packing, as similar heat is released in both cases.

3.2.3. Effect of OPC/limestone binder in the w/p = 0.5 mixture

The influence of inter-grinding of the constituents in the OPC/limestone mixture is shown in Fig. 6. The mean particle sizes of C, C85/L15, and I-GC85/GL15 are 7.8, 8.3, and 3.3 μm, respectively. The un-ground C85/L15 has a slightly larger mean particle size than C reference. After grinding, the mean particle size of I-GC85/GL15 decreased to less than half of that of C and C85/L15. Since limestone is softer than OPC, it is expected that limestone was preferentially ground as compared with the OPC.

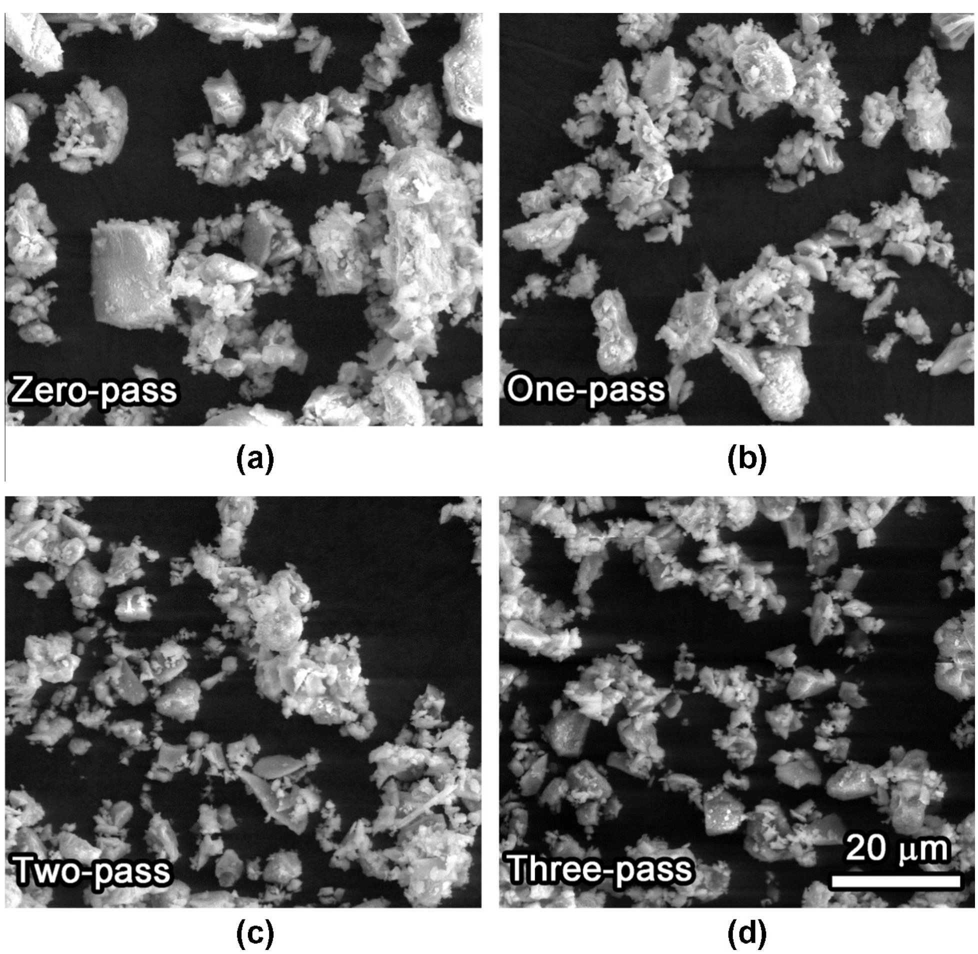

Fig. 3. SEM images of cement/limestone blends with zero to three passes grinding. (a) Zero-pass grinding. (b) One-pass. (c) Two-pass. (d) Three-pass.

The heat evolution rate and cumulative heat evolution were evaluated as shown in Fig. 6b and c, respectively. The high initial rate of heat release after the first several minutes is attributed to wetting and a rapid dissolution, which contributes to a small percentage of the total heat evolved [34]. The following period characterized by low heat evolution is considered an induction period [35] which is followed by a large exothermal peak that is dominated by the hydration of C_3S and growth of C–S–H.

The replacement of 15% of the cement with limestone resulted in a reduction in the maximum rate of heat evolution (i.e., the decrease of 19% at approximately 7 h as seen in Fig. 6b. This reduction is due to dilution of the cement by the limestone. No appreciable acceleration of hydration was observed with coarse limestone addition (the term 'coarse limestone' in this paper refers to the as-received limestone without any grinding) presumably due to the relatively large particle size, as indicated by the similar cumulative heats of C and C85/L15 mortars in Fig. 6c. After jet mill grinding, I-GC85/GL15 showed a more rapid rate of reaction with a maximum rate of heat evolution 0.0074 W/g of powder in Fig. 6b and thus increased cumulative heat in Fig. 6c, indicating accelerated hydration, although the induction period was slightly longer. The inter-ground material also had a higher cumulative heat release, corresponding to around 14% more cement hydration at 7 days (as estimated from the degree of hydration based on heat release).

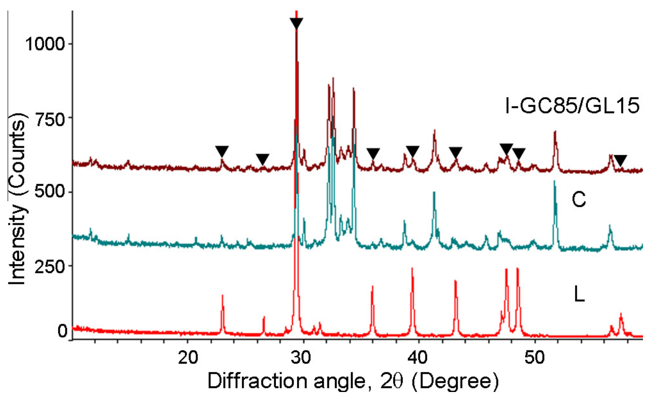

Fig. 4. XRD patterns of fly ash (F), cement (C), and I-GC85/GL15 (inter-ground cement and limestone mixture) powders. Markers indicate the peaks contributed by the limestone in the mixtures although overlapping with the peaks of C reference exists.

The compressive strength of cement/limestone binder up to 7 days is shown in Fig. 7a. In Fig. 7b the strength was normalized by dividing the strength of the specimen by the strength of the reference specimen cured to the same age [6]. Fig. 7 shows that when coarse limestone is combined with OPC (C85/L15), the lowest strength was observed. The addition of coarse limestone results in a reduction of strength at 1 and 7 days by 12.8–19.2 MPa, that is, a 47–62% strength ratio relative to C reference. This is believed to be attributable to the dilution effect.

Inter-ground I-GC85/GL15 mortar displays higher 1-day strength (29.2 MPa) than both C and C85/L15 mortars, with a strength ratio of 121%. Afterwards, the strength of I-GC85/GL15 mortar was 60–75% less than the OPC reference mortar at 3 days and 7 days. The inter-ground cement/limestone blends show rapid early strength development due to the decreased particle size that exposes more surface area of the cement for reaction and increases limestone nucleation sites. The lower compressive strength measured after 3 days for I-GC85/GL15 may be attributable to a less well-graded cement PSD [36]. Another hypothesis that at the relatively higher w/p of 0.5, there was a large volume of water

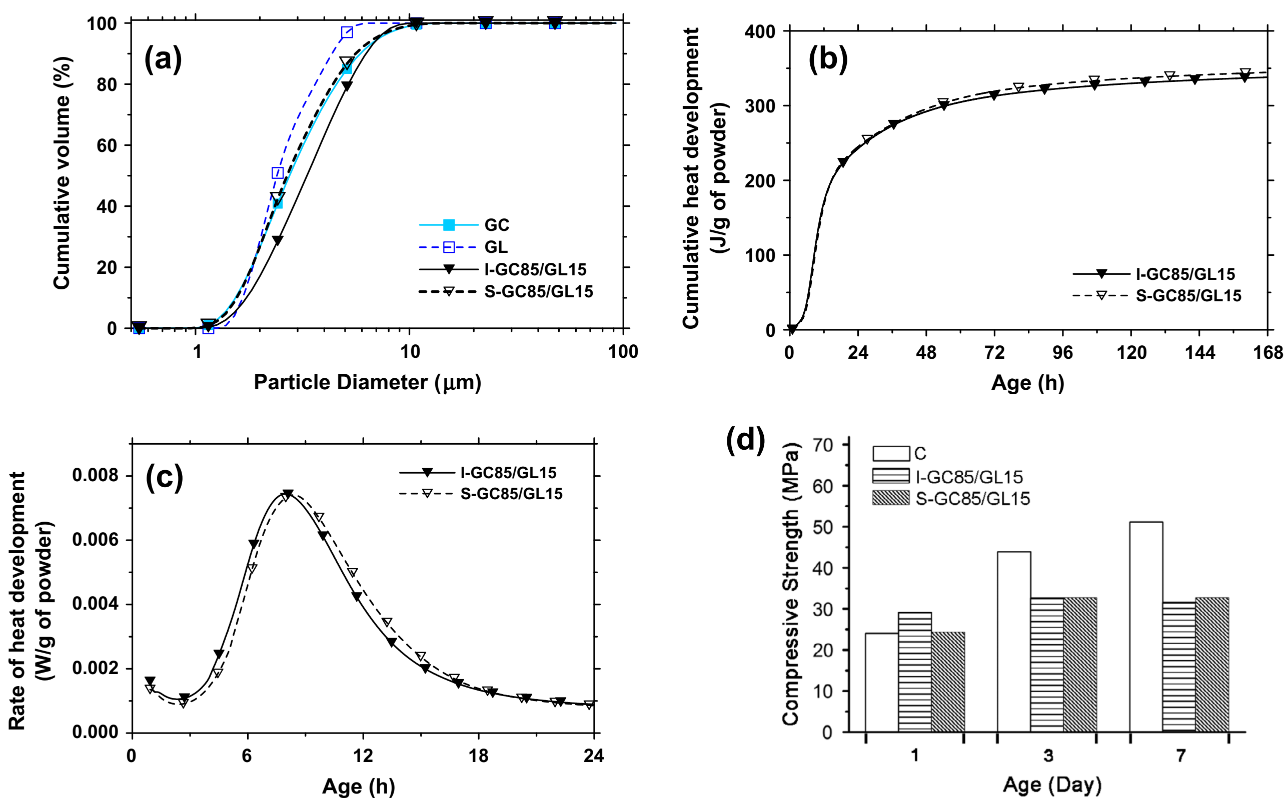

Fig. 5. Influence of inter-grinding and separate-grinding on the C85/L15 mixture. (a) Particle size distribution. (b) Cumulative heat evolution. (c) Rate of heat development. (d) Compressive strength as comparing with that of C reference mortar.

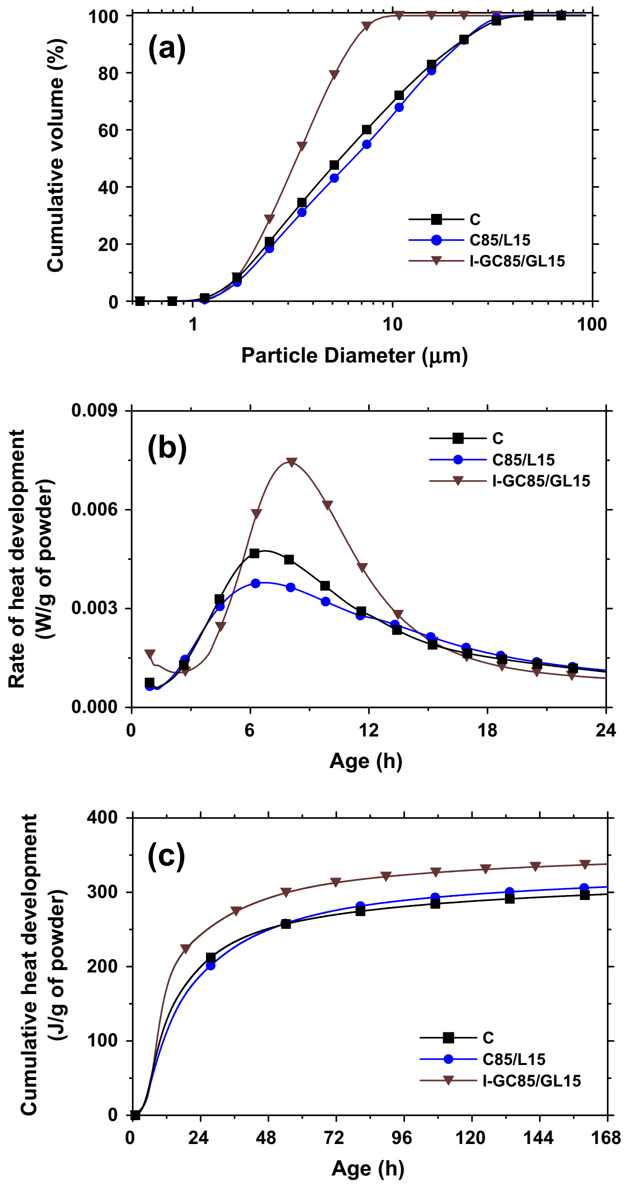

Fig. 6. Influence of grinding on cement/limestone binder at w/p of 0.50. (a) Particle size distribution. (b) Rate of heat evolution. (c) Cumulative heat.

sufficient to hydrate a large proportion of the cement. To investigate this potential benefits of using the limestone in a lower water to cement ratio (w/c) system where enhanced cement hydration could occur, additional testing was performed as described in the following section [38].

3.2.4. Effect of water to powder ratio (w/p = 0.35)

To evaluate the potential benefit of using limestone in mixtures with different w/c, specimens were prepared with a w/p of 0.35 (SP% = 0.9%). At a w/p of 0.35, the induction periods of blended cement mortars C85/L15, S-GC85/GL15, and I-GC85/GL15, were shortened by 1.1, 2.3, and 7.8 h, respectively, relative to C reference mortar as seen in Fig. 8a. After jet mill grinding, both S-GC85/GL15 and I-GC85/GL15 had faster rates of reaction shown as higher exothermal peaks with maximum heights of 0.0065 and 0.0088 W/g of powder, respectively, compared to about 0.0045 W/g of powder for C reference mortar, and an even lower value of 0.0036 W/g of powder for C85/L15 mortar. Accordingly, the cumulative heat of ground materials also increased (Fig. 8b). Both the reduced induction period and the improved heat evolution indicated the acceleration of hydration by grinding, where inter-grinding performed better than the separate-grinding process. Ground S-GC85/GL15 and I-GC85/GL15 had increased surface area, exposing more surface area to water to further enhance the hydration process relative to C reference. I-GC85/GL15 performed better than S-GC85/GL15 because the better dispersed limestone particles by jet mill grinding enhance nucleation.

Compressive strengths of C, C85/L15, and separate-ground S-GC85/GL15 were measured and are reported in Fig. 8c, and the normalized compressive strengths are reported in Fig. 8d. Unfortunately inter-ground I-GC85/GL15 displayed a rapid loss of workability and could not be cast as cylinders for compression test after the mixing stopped. However, the increased heat evolution of I-GC85/GL15 compared with S-GC85/GL15 in Fig. 8a and b implies the possibility of producing even higher very early strength for inter-ground I-GC85/GL15 mortar. At a lower w/p of 0.35, a 1-day compressive strength of 43.3 MPa was obtained from S-GC85/GL15, a strength ratio of 166% with C reference mortar (26.0 MPa). For longer curing time the trend reversed, with the strength ratio reduced to 84% (45.5 MPa relative to 54.3 MPa of reference) at 3 days and 91% (59.1 MPa relative to 64.6 MPa of reference) at 7 days when the benefits from the accelerated hydration cannot compensate for the dilution effect of the limestone (Fig. 8c and d). The strength of C85/L15 was always below the value of C reference, with 94% of the C reference at 1 day, 88% at 3 days, and 91% at 7 days.

The strengths observed for cement/limestone blends in lower w/p mixtures are higher than that observed in the higher w/p mixtures. This can be explained by a combination of two factors. First,

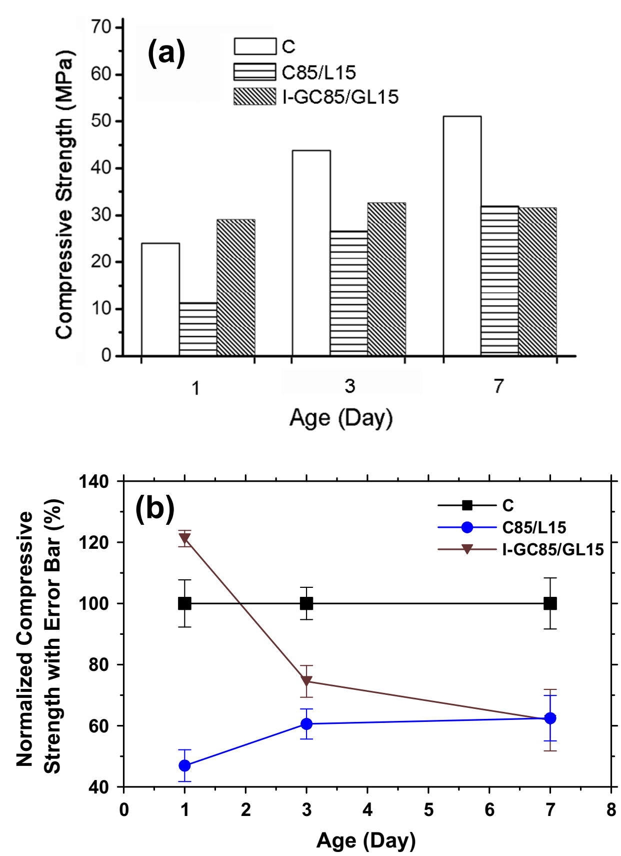

Fig. 7. Compressive strength (a) and normalized strength gain rate with error bar (b) of ground and unground cement/limestone mortars in comparison with C reference mortar. The w/p used here is 0.50.

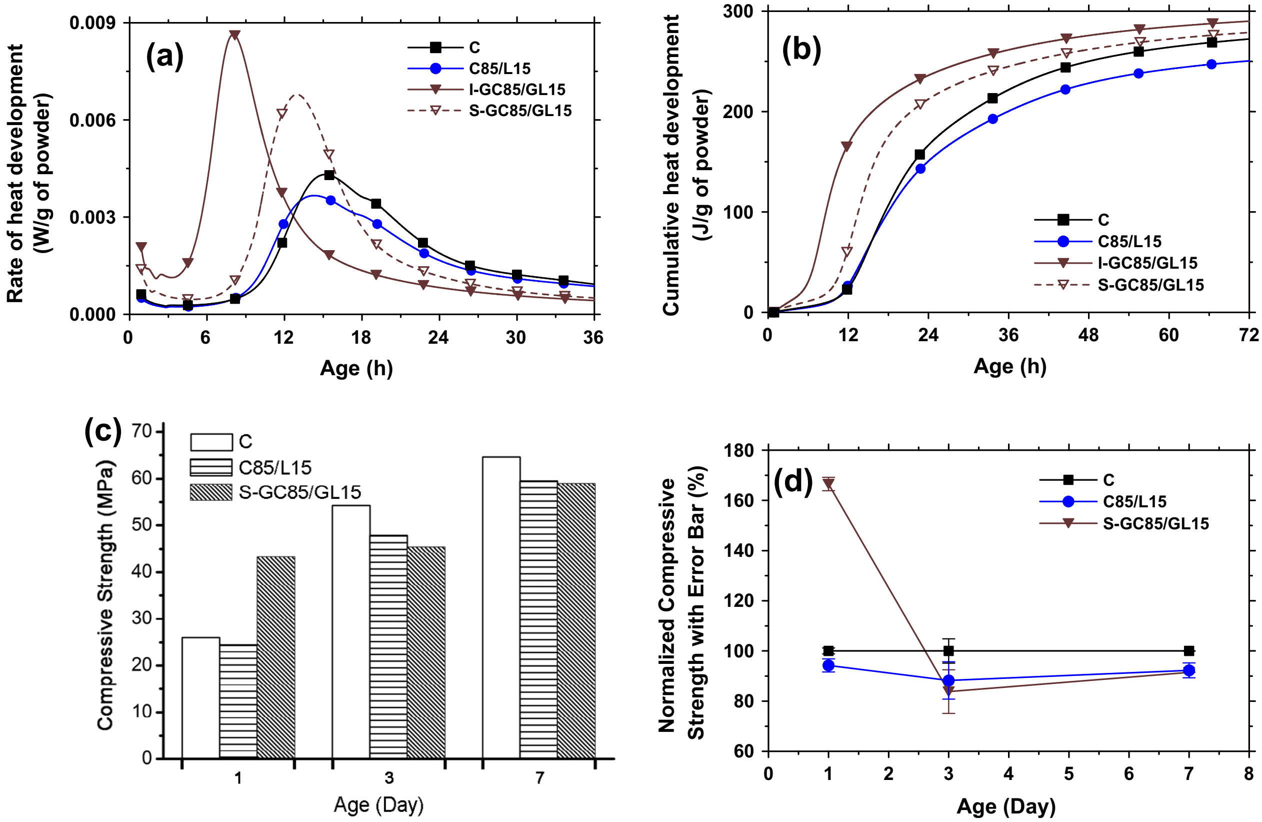

Fig. 8. Influence of grinding on cement/limestone binder at w/p of 0.35. (a) Rate of heat evolution. (b) Cumulative heat. (c) Compressive strength. (d) Normalized compressive strength with error bar.

the higher strength results from improved packing density due to a better powder dispersion with the addition of SP [37]. Second, at the lower w/p, e.g. 0.35, there is insufficient water in the pure cement mixture to enable the original cement to react to a very high degree of hydration. This occurs since the system essentially 'runs out of water'. Thus part of the cement acts as inert filler. When a limestone is used, a portion of the limestone replaces a portion of the cement thereby enabling the cement in the low w/p limestone mixture to hydrate to a greater extent [38].

3.3. Series II – cement/fly ash blends

3.3.1. XRD characterization

For inter-ground cement and fly ash, XRD results shown in Fig. 9 indicate that inter-grinding of this mixture is again only a physical process, producing no discernible phase transitions.

3.3.2. Effect of cement/fly ash binder

The mean particle sizes of C, C60/F40, and I-GC60/GF40 are6.9, 12.5, and 4.7 μm, respectively. The as-received fly ash has a larger particle size than the cement reference and thus leads to a larger mean particle size in the C60/F40 mixture with a wider distribution as shown in Fig. 10a. After jet mill grinding, I-GC60/GF40 displays a significantly narrower distribution. Therefore, jet mill grinding can significantly decrease the mean particle size and distribution of cement/fly ash blends, similar to the cement/limestone blends results.

The induction period was prolonged and less heat was released by replacing 40% of portland cement with fly ash due to the dilution of cement with the more slowly reacting fly ash (Fig. 10b and c). However, jet mill grinding partly compensated for this less reactive material by accelerating the hydration rate. It is believed that this occurs primarily due to the higher surface area for reaction [39].

The 28-day compressive strength is shown in Fig. 11a with the normalized strength relative to C reference mortar in Fig. 11b. The incorporation of supplementary fly ash (C60/F40) reduced the strength of mortar specimens from 27.7 MPa to 12.5 MPa at 1 day, which is about 45% of the reference cement specimen. After jet mill grinding, the strength of I-GC60/GF40 was higher than the un-ground material with a 1-day strength of 22.3 MPa (which is 81% of the C mortar reference specimen). Over the first 7 days, the inter-ground I-GC60/GF40 mortar benefits in terms of strength development relative to un-ground C60/F40 mortar although the strength is still lower than that of C reference. However, the difference between blends and C reference mortar becomes smaller with time.

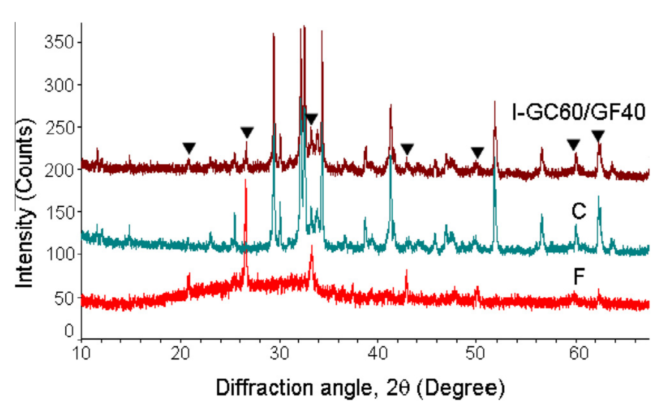

Fig. 9. XRD patterns of fly ash (F), cement (C), and I-GC60/GF40 (inter-ground cement and fly ash mixture) powders. Markers indicate the peaks contributed by the fly ash in the mixture.

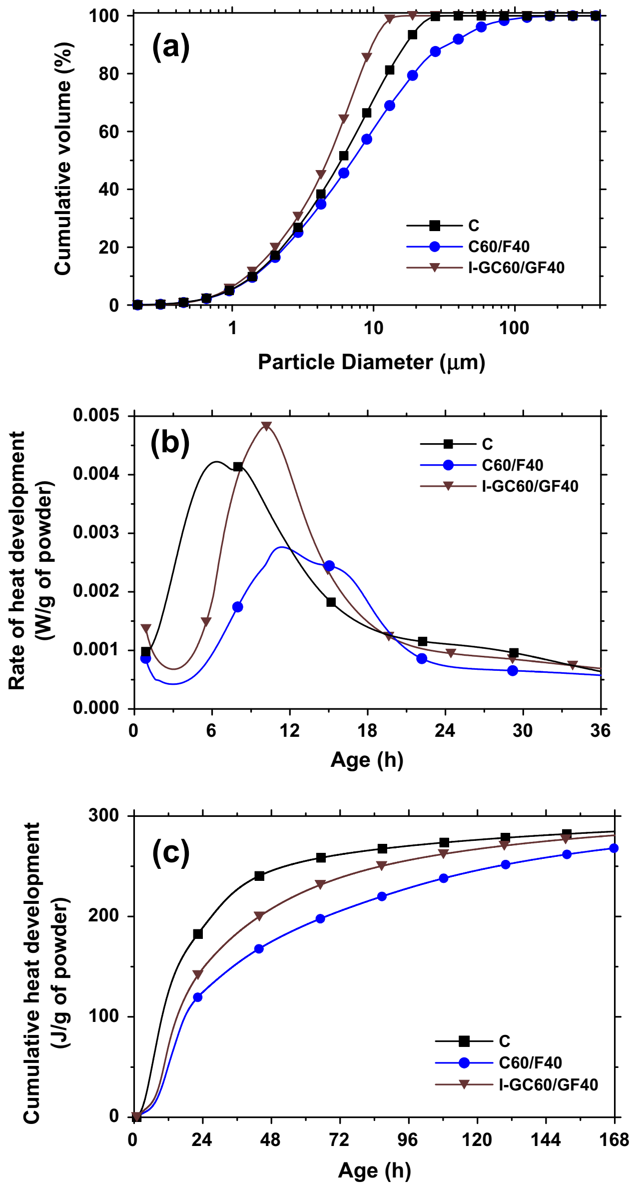

Fig. 10. Influence of grinding on fly ash and cement binder. (a) Particle size distribution. (b) Rate of heat evolution. (c) Cumulative heat.

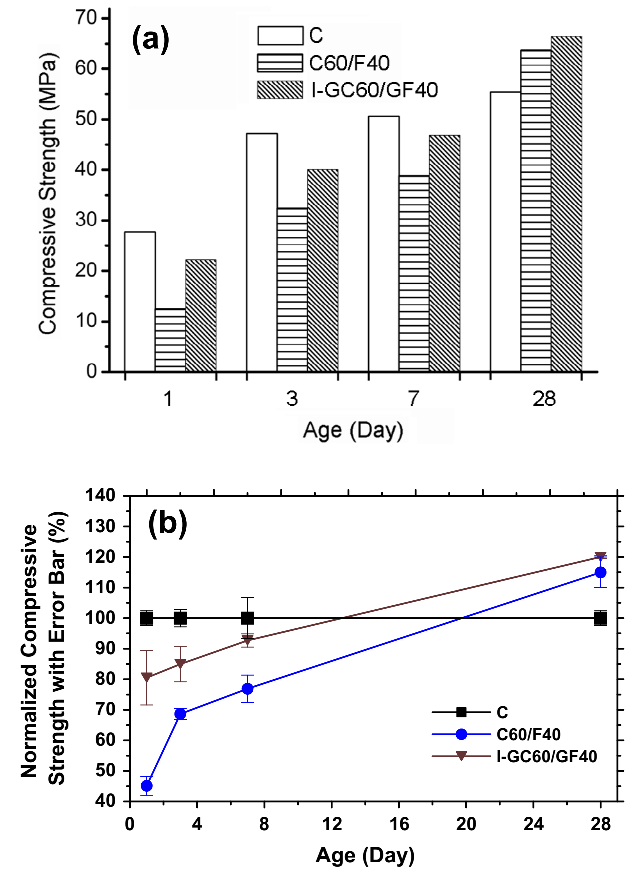

Fig. 11. Compressive strength (a) and normalized data with error bar (b) of cement/fly ash mortars in comparison with C reference mortar. The w/p used here is 0.35.

The 28-day strength of C60/F40 mortar (63.7 MPa) exceeded that of the C reference mortar (55.4 MPa) presumably due to the pozzolanic reactivity of the fly ash. At 28 days, the strength of the I-GC60/GF40 mortar was 20% higher than the reference. Fig. 11b shows that the strength ratio of C60/F40 increases from 45% at 1 day to 115% at 28 days, while I-GC60/GF40 shows a normalized strength of 80% at the age of 1 day to 120% at 28 days, implying the increased surface area and associated nucleation effects can improve the early hydration [1]. This indicates that although one shortcoming of using higher volumes of fly ash is a delay in setting and a reduction in early age strength gain, the inter-grinding method may provide an approach to overcome this delay in set and slow strength gain.

4. Conclusion

This study investigated the effect of jet mill grinding on OPC/limestone and OPC/fly ash blends. Jet mill grinding produces irregular cementitious particles with an average particle size of approximately 4 μm or less with a narrowed size distribution range. XRD indicated that jet mill grinding is a physical process only and results in no discernible phase transformations.

For OPC/limestone mixtures, inter-grinding and separate-grinding were compared. The results imply that jet mill grinding produced smaller particles that can accelerate early-age hydration reactions. Jet mill ground materials displayed improved early age strength development which is attributed to increases in surface area and the number of nucleation sites. Inter-grinding appeared to be more effective than combining separately ground materials; however, further research is likely needed to explore these differences. When investigating the OPC/limestone, the grinding effect was more pronounced at lower w/p of 0.35 than at higher w/p of 0.50. This was due to the fact that in the lower w/p mixtures, the replacement of cement by limestone allows the remaining cement to hydrate more completely, resulting in a higher degree of hydration.

For the cement/fly ash mixture, jet mill grinding also accelerated the rate of hydration. Finely ground blended I-GC60/GF40 mortar had greater surface area and was more reactive, thus resulting in the increase in hydration and higher compressive strength. At the age of 28 days, the strength of GC60/GF40 even exceeded that of the C reference mortar. This could overcome construction concerns with slow strength development.

Overall, jet mill grinding was found to provide benefits in early age hydration and compressive strength for both cement/limestone and cement/fly ash mixtures. The grinding process also provides a new efficient approach for industrial application in adding limestone and fly ash supplement materials to cement blends.

While this study has shown that jet mill grinding may have significant benefits, additional studies on the economics and energy efficiency of jet mill grinding are needed to assess its economic viability for commercial applications. Additional research would be needed to investigate the influence of jet mill grinding and optimal particle size distribution on rheological properties or volume change.

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References

[1] Erdogdu K, Turker P. Effects of fly ash particle size on strength of portland cement fly ash mortars. Cem Concr Res 1998;28(9):1217–22.
[2] Chindaprasirt P, Homwuttiwong S, Sirivivatnanon V. Influence of fly ash fineness on strength, drying shrinkage and sulfate resistance of blended cement mortar. Cem Concr Res 2004;34(7):1087–92.
[3] Paya J, Borrachero MV, Monzo J, Peris-Mora E, Bonilla M. Long term mechanical strength behaviour in fly ash/portland cement mortars prepared using processed ashes. J Chem Technol Biotechnol 2002;77(3):336–44.
[4] Osbaeck B, Johansen V. Particle-size distribution and rate of strength development of portland-cement. J Am Ceram Soc 1989;72(2):197–201.
[5] Sakai E, Masuda K, Kakinuma Y, Aikawa Y. Effects of shape and packing density of powder particles on the fluidity of cement pastes with limestone powder. J Adv Concr Technol 2009;7(3):347–54.
[6] Kiattikomol K, Jaturapitakkul C, Songpiriyakij S, Chutubtim S. A study of ground coarse fly ashes with different finenesses from various sources as pozzolanic materials. Cem Concr Compos 2001;23(4–5):335–43.
[7] Sengul O, Tasdemir MA. Compressive strength and rapid chloride permeability of concretes with ground fly ash and slag. J Mater Civ Eng 2009;21(9):494–501.
[8] Jaturapitakkul C, Kiattikomol K, Sata V, Leekeeratikul T. Use of ground coarse fly ash as a replacement of condensed silica fume in producing high-strength concrete. Cem Concr Res 2004;34(4):549–55.
[9] Paya J, Monzo J, Borrachero MV, PerisMora E, GonzalezLopez E. Mechanical treatment of fly ashes. 2. Particle morphologies in ground fly ashes (GFA) and workability of GFA-cement mortars. Cem Concr Res 1996;26(2):225–35.
[10] Kakali G, Tsivilis S, Aggeli E, Bati M. Hydration products of C(3)A, C3S and portland cement in the presence of $CaCO_3$. Cem Concr Res 2000;30(7):1073–7.
[11] Liu JH, Song SM, Sun YM, Wang L. Influence of ultrafine limestone powder on the performance of high volume mineral admixture reactive powder concrete. New Mater Adv Mater 2011;152–153:1583–6 [Pts 1 and 2].
[12] Sezer GI, Copuroglu O, Ramyar K. Microstructure of 2 and 28-day cured Portland limestone cement pastes. Indian J Eng Mater Sci 2010;17(4):289–94.
[13] Ito M, Sato K, Naoi Y. Productivity increase of the vertical roller mill for cement grinding. In: 1997 Ieee/Pca cement industry technical conference – Xxxix conference record; 1997. p. 177–94.
[14] Folsberg J. The air-swept ring roller mill for clinker grinding. In: 1997 Ieee/Pca cement industry technical conference – Xxxix conference record; 1997. p. 157–76.
[15] Wan HW, Shui ZH, Lin ZS. Analysis of geometric characteristics of GGBS particles and their influences on cement properties. Cem Concr Res 2004;34(1):133–7.
[16] Sotome I, Nei D, Tsuda M, Hossen MS, Takenaka M, Okadome H, et al. Decontamination effect of milling by a jet mill on bacteria in rice flour. Biocontrol Sci 2011;16(2):79–83.
[17] Palaniandy S, Azizli KAM. Microstructure characterization of mechanically activated talc in jet mill using XRD line broadening technique. In: International conference on advancement of materials and, nanotechnology 2007, vol. 1217; 2010. p. 437–41.
[18] Palaniandy S, Kadir NA, Jaafar M. Value adding limestone to filler grade through an ultra-fine grinding process in jet mill for use in plastic industries. Miner Eng 2009;22(7–8):695–703.
[19] Palaniandy S, Azizli KAM. Mechanochemical effects on talc during fine grinding process in a jet mill. Int J Miner Proc 2009;92(1–2):22–33.
[20] de Vegt O, Vromans H, den Toonder J, Maarschalk KV. Influence of flaws and crystal properties on particle fracture in a jet mill. Powder Technol 2009;191(1–2):72–7.
[21] Kalman H, Katz A. Preliminary experimental analysis of a spiral jet mill performance. Part Part Syst Charact 2007;24(4–5):332–8.
[22] Uvarov VA. The use of a countercurrent jet mill in the production of insulating ceramics. Glass Ceram 2006;63(7–8):274–6.
[23] de Vegt O, Vromans H, Faassen F, Maarschalk KV. Milling of organic solids in a jet mill. Part 2: Checking the validity of the predicted rate of breakage function. Part Part Syst Charact 2006;22(4):261–7.
[24] de Vegt O, Vromans H, Faassen F, Maarschalk KV. Milling of organic solids in a jet mill. Part 1: Determination of the selection function and related mechanical material properties. Part Part Syst Charact 2005;22(2):133–40.
[25] http://www.jetpulverizer.com/faq.php?open=milling-equipment.
[26] http://jetpulverizer.com/how-jet-mills-work.php.
[27] Kadri EH, Aggoun S, De Schutter G, Ezziane K. Combined effect of chemical nature and fineness of mineral powders on portland cement hydration. Mater Struct 2010;43(5):665–73.
[28] Chindaprasirt P, Jaturapitakkul C, Sinsiri T. Effect of fly ash fineness on compressive strength and pore size of blended cement paste. Cem Concr Compos 2005;27(4):425–8.
[29] Pera J, Husson S, Guilhot B. Influence of finely ground limestone on cement hydration. Cem Concr Compos 1999;21(2):99–105.
[30] Sato T, Beaudoin JJ. Effect of nano-$CaCO_3$ on hydration of cement containing supplementary cementitious materials. Adv Cem Res 2011;23(1):33–43.
[31] Bentz DP, Sato T, Varga IDL, Weiss WJ. Fine limestone additions to regulate setting in high volume fly ash mixtures. Cem Concr Compos 2012;34(1):11–7.
[32] Keck CM, Muller RH. Size analysis of submicron particles by laser diffractometry – 90% of the published measurements are false. Int J Pharm 2008;355(1–2):150–63.
[33] http://www.lp-clickfair.de/module/exhibitor/uploads/907930/de/TAM_Air.pdf.
[34] Gruyaert E, Robeyst N, De Belie N. Study of the hydration of portland cement blended with blast-furnace slag by calorimetry and thermogravimetry. Journal of Thermal Analysis and Calorimetry. 2010;102(3):941–51.
[35] Bullard JW. A determination of hydration mechanisms for tricalcium silicate using a kinetic cellular automaton model. J Am Ceram Soc 2008;91(7):2088–97.
[36] Celik IB. The effects of particle size distribution and surface area upon cement strength development. Powder Technol 2009;188(3):272–6.
[37] Kwan AKH, Chen JJ, Fung WWS, Li LG. Improving packing density of powder in cement paste for production of high-performance concrete. Adv Build Mater 2011;168–170:1640–7 [Pts 1–3].
[38] Bentz DP, Irassar EF, Buchler B, Weiss WJ. Limestone fillers to conserve cement in low w/cm concretes: an analysis based on powers' model. Concr Int 2009;31(11):41–6.
[39] Bentz DP, Ferraris CF, Varga DLI, Peltz MA, Winpigler JA. Mixture proportioning options for improving high volume fly ash concretes. Int J Pavement Res Technol 2010;3(5):234–40.