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THE EFFECT OF GRINDING ON THE PHYSICAL PROPERTIES OF FLY ASHES AND A PORTLAND CEMENT CLINKER

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ABSTRACT

The effect of the grinding on the physical properties of three ASTM Class F fly ashes and a Portland cement clinker were investigated. The specific gravity and the fineness of the fly ashes increased with an increase in the grinding time. However, this increase was less significant beyond 2 h. The morphology of the fly ashes was changed by grinding. Most of the plerospheres and large, irregular-shaped particles were crushed after 2 h of grinding. However, the number of the spherical particles reduced with increased grinding. There appears to be an optimum grinding time of approximately 4 h for the fly ashes beyond which the water requirement increased and the strength activity indices either decreased or did not increase significantly. © *1997 Elsevier Science Ltd*

Introduction

The production of Portland cement contributes significantly to the CO_2 emissions. For every tonne of Portland cement produced, approximately one tonne of CO_2 is released to the atmosphere (1). Therefore, it is imperative that technologies be developed to reduce the production of Portland-cement clinker in rotary kilns while maintaining the target production of cements and meeting the demand of the construction industry.

In the 1980s, CANMET developed high-volume fly ash concrete, in which 55–60% of the Portland cement is replaced by low calcium fly ash, a by-product of thermal-power plants. This type of concrete has demonstrated excellent mechanical and durability properties, and is slowly getting acceptance worldwide (2–19). However, at present fly ash has to be added at the ready-mixed concrete batching plants to produce high-volume fly ash concrete; this necessitates additional storage silos and quality control at ready-mixed concrete plants, thus raising the cost of the concrete.

In 1995, CANMET undertook a project to develop a blended cement incorporating high volumes of ASTM Class F fly ash. The blended cement is made by grinding together 55% of low-calcium fly ash, 45% of ASTM Type III cement clinker, and a small amount of

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gypsum. Previous studies show that the grinding crushes the cenospheres and the large particles of fly ash resulting in higher specific gravity and fineness, and consequently higher pozzolanic reactivity of the fly ash (20–22). According to Cornelissen et al. (22), the use of ground fly ash as a cementitious material for mortars and concretes results in higher strengths with no negative effect on the workability. This paper presents the results of the effect of the grinding on the physical properties of three fly ashes and a clinker. The production of the blended cements and their performance will be presented in future papers.

Materials and Methods

Cement

ASTM Type I Portland cement was used as a control for the determination of the strength activity indices, and its chemical analysis and physical properties are given in Table 1.

Portland Cement Clinker

A Portland cement clinker ASTM Type III was used, and its specific gravity and chemical composition are also given in Table 1.

Fly Ash

Three fly ashes from Lingan (Nova Scotia), Sundance (Alberta), and Genesee (Alberta) were used in this study. Their physical properties and chemical composition are given in Table 1, and their mineral compositions are given in Table 2. The particle size distribution of the fly ashes is illustrated in Figure 1. The particle size distribution of the materials was determined by a Microtrac ×100 particle size analyzer using scattered light from laser beams projected through a stream of particles suspended in isopropanol. The amount and direction of the light scattered by the particles were then measured by an optical detector, and analyzed by a computer.

Lingan fly ash, a typical ASTM Class F ash, contained 3.68% CaO and a high Fe_2O_3 content of 35.05%. The equivalent alkali content (Na_2O + 0.658 K_2O) of the ash is 1.85%. The ash had a Blaine fineness of 2590 cm^2/g and a specific gravity of 2.82. The relatively high specific gravity of the fly ash is, to a large extent, related to its high iron content.

Sundance fly ash, which met the general requirements of ASTM Class F ash, had a relatively high CaO content of 13.38% and alkali content (Na_2O equivalent) of 3.98%. The Blaine fineness of the ash was 3030 cm^2/g and the specific gravity was 2.14.

Genesee fly ash had a relatively low Blaine fineness of 1770 cm^2/g, and the amount of ash retained when wet-sieved on a 45-μm sieve was 36.5%, thus this ash fails to meet the fineness requirements of ASTM C 618 (ASTM C 618 requires that the amount retained when wet-sieved on a 45-μm sieve be less than 34%). The ash had a relatively low specific gravity, which is, to a large extent, related to the larger amount of plerospheres, which are hollow particles filled with smaller spheres, in the sample compared with the fly ashes from Lingan or Sundance (Fig. 2). The ash had a CaO content of 5.83%, and an alkalai content (Na_2O equivalent) of 3.64%. This ash meets the general chemical requirements of ASTM Class F fly ash.

TABLE 1
Physical Properties and Chemical Analysis of the Materials Used

	ASTM Type I Cement	Clinker	Fly ash Lingan	Fly ash Sundance	Fly ash Genesee
Physical Tests					
Specific gravity	3.13	3.23	2.82	2.14	2.03
Fineness					
passing 45 μm, %	93.1	-	87.4	83.6	63.5
specific surface, Blaine, cm^2/g	4000	-	2590	3030	1770
median particle size (μm)	-	-	14	12.4	21.2
Compressive strength of 51 mm cubes, MPa					
7-day	29.6	-	-	-	-
28-day	40.3	-	-	-	-
Water requirement, %	-	-	97.9	99.2	95.9
Pozzolanic Activity Index, %					
7-day	-	-	84.1	94.5	81.7
28-day	-	-	88.6	106.9	84.5
Chemical Analysis, %					
Silicon dioxide (SiO_2)	21.36	22.25	36.85	52.35	62.56
Aluminum oxide (Al_2O_3)	3.98	4.51	18.35	23.35	20.85
Ferric oxide (Fe_2O_3)	3.15	3.41	35.05	4.65	4.45
Calcium oxide (CaO)	62.41	65.54	3.68	13.38	5.83
Magnesium oxide (MgO)	2.57	2.85	1.04	1.28	1.45
Sodium oxide (Na_2O)	0.20	0.38	0.77	3.60	2.51
Potassium oxide (K_2O)	0.80	0.82	1.64	0.58	1.72
Equivalent alkali ($Na_2O + 0.658\ K_2O$)	0.73	0.92	1.85	3.98	3.64
Phosphorous oxide (P_2O_5)	0.21	0.22	0.26	0.20	0.12
Titanium oxide (TiO_2)	0.19	0.20	0.87	0.75	0.67
Strontium oxide (SrO)	0.27	-	-	-	-
Sulphur trioxide (SO_3)	3.43	<0.01	1.76	0.21	0.08
Loss on ignition	1.72	0.01	2.36	0.32	0.26
Bogue Potential Compound Composition					
Tricalcium silicate C_3S	51	-	-	-	-
Dicalcium silicate C_2S	23	-	-	-	-
Tricalcium aluminate C_3A	5	-	-	-	-
Tetracalcium aluminoferrite C_4AF	10	-	-	-	-

The particle size distribution curves in Figure 1 indicate that the Genesee fly ash is the coarsest of the three ashes tested. However, the Lingan fly ash contains more larger particles above 100 μm than the Sundance and Genesee fly ashes.

Grinding Mill

A grinding mill, 420 mm in length and 500 mm in diameter, was used for grinding the fly ashes and the clinker and for producing the blended products. According to past experience,

TABLE 2
Mineral Composition of the Fly Ashes

	Phases identified				
Type of fly ash	Glass	Quartz	Mullite	Magnetite	Hematite
Lingan	x	x	x	x	x
Sundance	x	x	x		
Genesee	x	x			

this mill works well when approximately 45% of its volume is occupied by the material to be ground and the grinding ball charge. The grinding efficiency of the mill is dependent primarily upon the amount of the materials to be ground, the ratio of the material to the grinding balls, and the size of the grinding balls. Figure 3 shows the effect of the mass and the size of the grinding balls on the Blaine fineness of a sample of 10 kg of Sundance fly ash. According to the results, a combination of 35 kg of large balls (30 mm thick and 30 mm diameter) and 35 kg of medium balls (20 mm thick and 20 mm diameter) was selected for the study on the grinding of the cement clinker and the fly ashes.

Grinding Procedures and the Determination of the Physical Properties of the Materials

For determining the grinding efficiency of individual materials, the fly ashes and the cement clinker were ground separately for up to 10 h. Before grinding, the cement clinker was crushed and sieved so that all the particles were less than 0.6 mm. Samples were taken after 2, 4, 6, 8, and 10 h of grinding, and the specific gravity, specific surface area (Blaine fineness), and particle size distribution were determined. The morphology of the selected fly ash samples were examined. The water requirement and the pozzolanic activity index of the

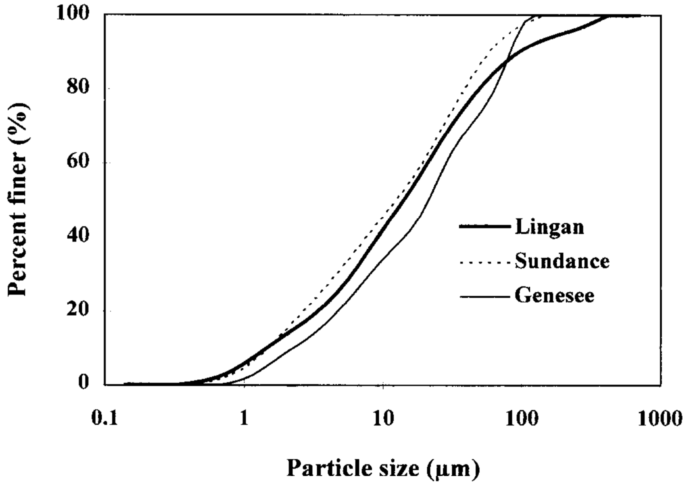

FIG. 1.
Particle size distribution of the fly ashes as received.

FIG. 2.
A plerosphere in Genesee fly ash.

original unground fly ashes and the fly ashes after 2, 4, and 10 h of grinding were also determined.

The specific gravity of the original fly ashes was determined according to ASTM C 188, whereas the specific gravity of the ground fly ashes was determined with an air comparison pycnometer 930 (Beckman). Past experience indicates that the specific gravity determined by the above two methods was comparable.

The specific surface of the fly ash samples was determined by an air permeability apparatus according to ASTM C 204. The particle size distribution of the materials was determined by the Microtrac ×100 particle size analyzer, and the morphology of the fly ash samples was examined under a Jeol JSM 820 scanning electron microscope. The water requirement and the pozzolanic activity of the fly ashes were determined according to ASTM C 311.

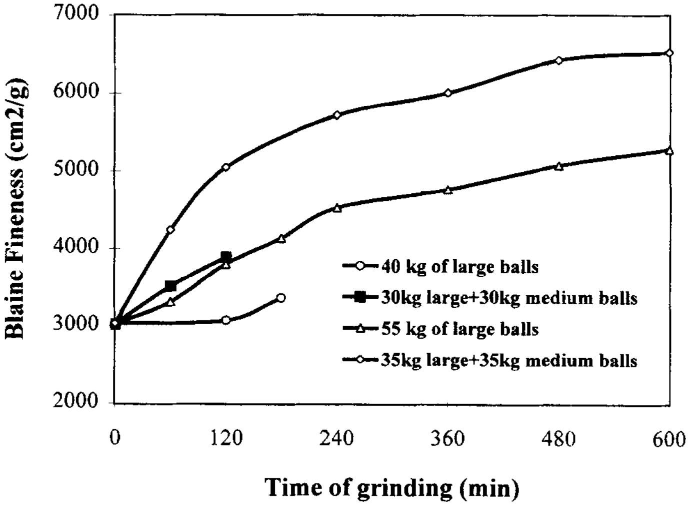

FIG. 3.
The effect of the amount of the grinding balls in the mill on the Blaine fineness of the Sundance fly ash (10 kg fly ash was in the grinding mill).

TABLE 3
Effect of the Grinding on the Physical Properties of the Fly Ashes

		Type of fly ash		
		Lingan	Sundance	Genesee
Specific gravity	before grinding	2.82	2.14	2.03
	after 2 h grinding	2.99	2.53	2.44
	after 4 h grinding	3.03	2.59	2.50
	after 10 h grinding	3.15	2.63	2.54
Specific surface area, Blaine, cm^2/g	before grinding	2590	3030	1770
	after 2 h grinding	3970	5045	4000
	after 4 h grinding	4920	5720	4770
	after 10 h grinding	6380	6540	6580
Median particle size, μm	before grinding	14.0	12.4	21.2
	after 2 h grinding	6.7	6.5	7.7
	after 4 h grinding	5.2	5.1	6.7
	after 10 h grinding	3.8	4.1	3.8
Water requirement, %	before grinding	97.9	99.2	95.9
	after 2 h grinding	97.1	97.1	95.0
	after 4 h grinding	97.1	96.7	95.0
	after 10 h grinding	97.9	98.8	96.7

Results and Discussion

Tables 3 and 4 summarizes the physical properties of the fly ashes as received, and after 2, 4, and 10 h grinding.

Specific Gravity

Figure 4 shows the effect of the grinding on the specific gravity of the fly ashes. The specific gravity of the Genesee, Sundance, and Lingan fly ashes increased from 2.03, 2.14, and 2.82 to 2.44, 2.53, and 2.99, respectively, after 2 h of grinding. After that, however, the increase in the specific gravity was not significant. This indicates that most of the plerospheres and cenospheres, which usually have relatively low specific gravity, had been crushed after 2 h of grinding. Figure 4 also shows that the increase in the specific gravity of the fly ashes from Sundance and Genesee after 2 h of grinding was more than that for the fly ash from Lingan. This indicates that Lingan fly ash either contained less plerospheres and cenospheres or that the plerospheres in the sample were more difficult to crush compared with the Sundance and Genesee fly ashes.

Fineness

Specific Surface (Blaine). The effect of the grinding on the specific surface of the fly ashes and the cement clinker is presented in Figure 5. The specific surface of the fly ashes and the clinker increased with an increase in the grinding time, but high specific surface can be reached only at the expense of more energy and time. The specific surface of the clinker after 2 h of grinding was 2830 cm^2/g, and increased to 5290 cm^2/g after 10 h of grinding. The

TABLE 4
Effect of the Grinding on the Strength Activity Indices of the Fly Ashes

			Control mixture	Test mixtures		
				Lingan	Sundance	Genesee
Compressive strength of 51-mm cubes, MPa	before grinding	7-day	29.6	24.9	28.0	24.2
		28-day	40.3	35.7	43.1	34.1
	after 2 h grinding	7-day	32.4	30.4	35.1	31.7
		28-day	41.1	42.1	50.3	46.7
	after 4 h grinding	7-day	32.4	31.4	36.5	33.5
		28-day	41.2	42.7	52.9	48.6
	after 10 h grinding	7-day	32.9	30.2	35.3	32.5
		28-day	44.1	44.3	57.8	52.1
Strength activity index, %	before grinding	7-day	-	84.1	94.5	81.7
		28-day	-	88.6	106.9	84.5
	after 2 h grinding	7-day	-	93.8	108.3	97.8
		28-day	-	102.5	122.5	113.7
	after 4 h grinding	7-day	-	96.9	112.6	103.4
		28-day	-	101.9	126.1	115.9
	after 10 h grinding	7-day	-	91.8	107.0	98.8
		28-day	-	100.4	131.0	118.0

specific surface of the Genesee, Lingan, and Sundance fly ashes increased from 1770, 2590, and 3030 to 4000, 3970, and 5045 cm^2/g after 2 h of grinding, and to 6580, 6380, and 6540 cm^2/g after 10 h of grinding. It appears that the first 2 h of grinding was more effective in increasing the fineness of the fly ashes, and such an increase for the Sundance and Genesee

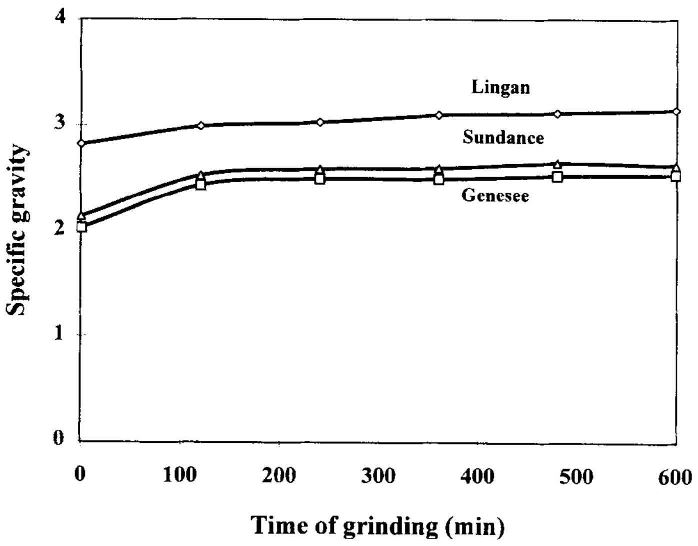

FIG. 4.
The effect of grinding on the specific gravity of the fly ashes.

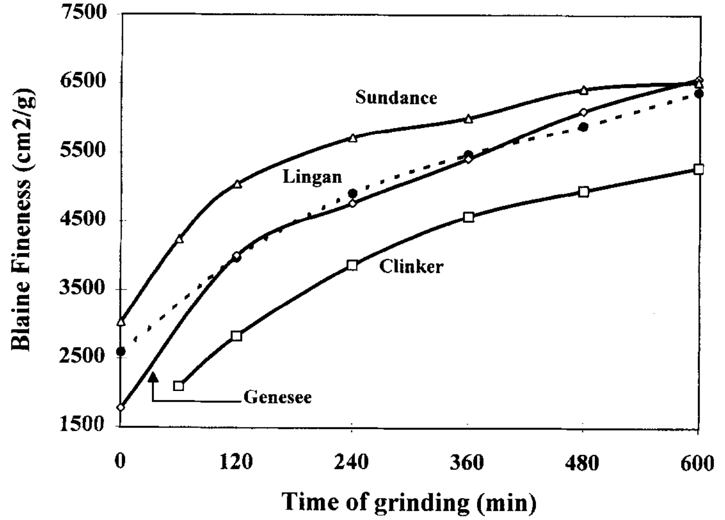

FIG. 5.
The effect of grinding on the Blaine fineness of the clinker and the fly ashes.

fly ashes was more significant than that for the Lingan fly ash. This is in line with the data obtained for the specific gravity of the ashes.

Figure 5 indicates that the grinding was most effective for the increase in the specific surface of the coarse Genesee fly ash.

Particle Size Distribution. Figures 6 through 9 show the changes in the particle size distribution of the cement clinker and the fly ashes after up to 10 h of grinding.

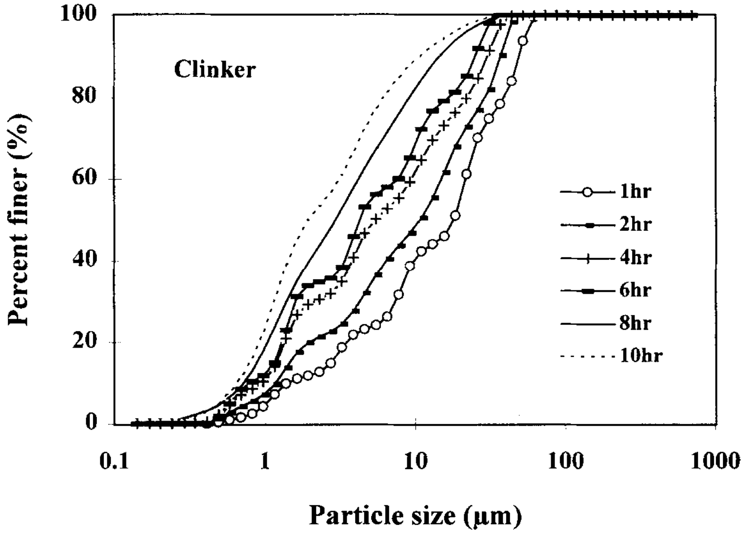

FIG. 6.
The effect of grinding on the particle size distribution of the clinker.

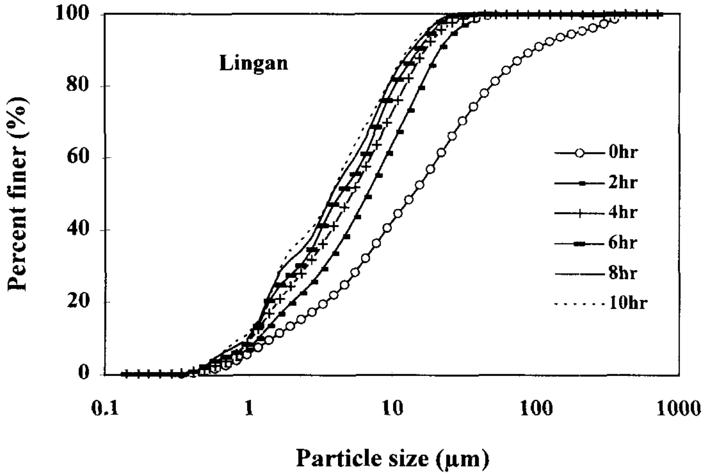

FIG. 7.
The effect of grinding on the particle size distribution of Lingan fly ash.

The particle size of the cement clinker and the fly ashes decreased with increasing grinding time. The median particle size for the cement clinker after 2 h of grinding was 13.0 μm, and decreased to 4.0 μm after 10 h of grinding. The median particle size for the Genesee, Lingan, and Sundance fly ashes decreased from 21.2, 14.0, and 12.4 μm to 7.7, 6.7, and 6.5 μm after 2 h of grinding, and further decreased to 3.8, 3.8, and 4.1 μm, respectively, after 10 h of grinding. For the fly ashes, the particle size decreases were most significant during the first 2 h of grinding. After 2 h of grinding, most of the large particles had been crushed so that all the particles were less than 65 μm, and more than 60% of the particles were less than 10 μm. Further increase in the grinding time was less effective in increasing the particle

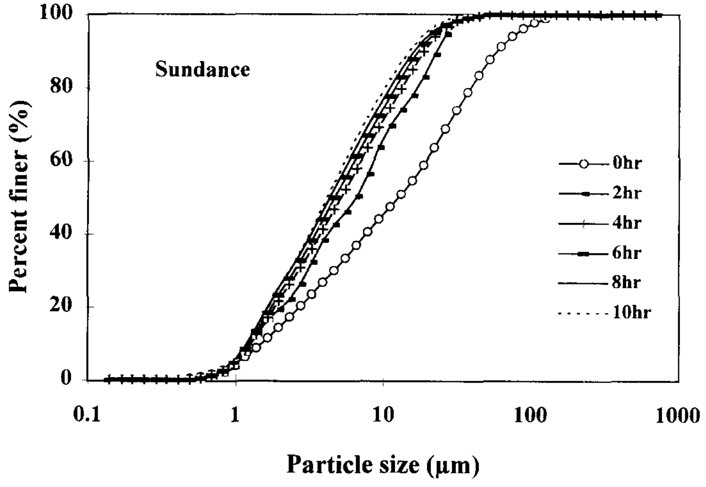

FIG. 8.
The effect of grinding on the particle size distribution of Sundance fly ash.

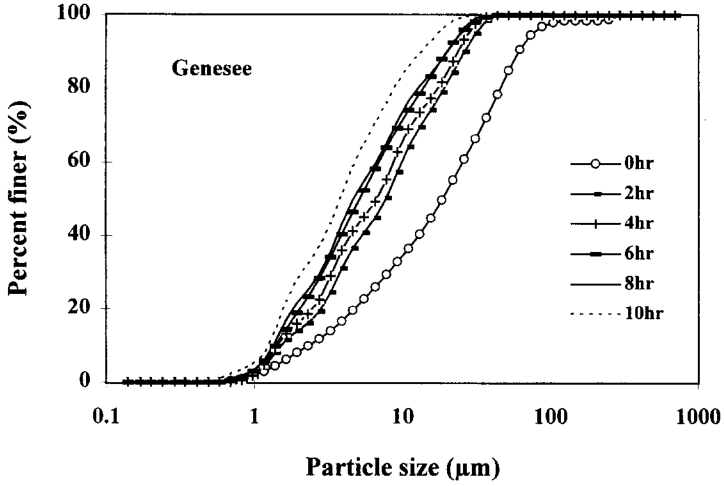

FIG. 9.
The effect of grinding on the particle size distribution of Genesee fly ash.

fineness. This is in line with the results of the specific gravity and specific surface as discussed above.

Morphology of Fly Ash Particles

Figures 10 through 12 show the typical micrographs of the original unground fly ash (Lingan) and the fly ash after 2 and 10 h of grinding.

The fly ashes "as received" contain mostly spherical particles with a small amount of plerospheres and irregular-shaped particles. After 2 h of grinding, most of these plerospheres and large, irregular-shaped particles had been crushed, but there were still some left in the sample (Fig. 11). After 10 h of grinding, the fly ashes contained particles of which were either

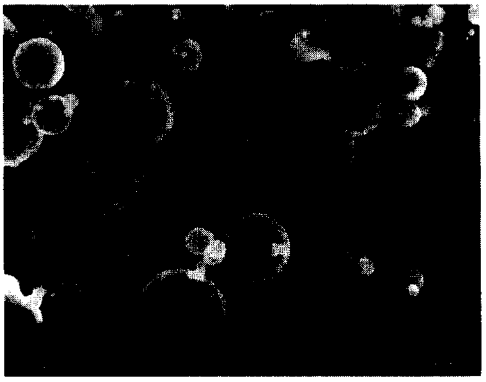

FIG. 10.
Scanning electron micrograph: Lingan fly ash (as received).

FIG. 11.
Scanning electron micrograph: Lingan fly ash after 2 h of grinding.

spherical and of irregular shape. However, the number of spherical particles reduced with increased grinding.

Water Requirement and Strength Activity

Figure 13 shows the water requirement of the fly ashes as a function of the grinding time. After subjecting to grinding for 2 h, the water requirement of the fly ashes decreased somewhat primarily due to the break up of the large plerospheres. Increasing the grinding time from 2 to 4 h did not affect the water requirement substantially. Further increasing the grinding time from 4 to 10 h increased the water requirement because of the increase in the irregular-shaped particles.

The strength activity indices at 7 days are shown in Figure 14. The highest strength activity indices were obtained using the fly ashes that had been subjected to 4 h of grinding, and these indices were 15 to 27% higher than those of the unground fly ashes. When the grinding time for the fly ashes was increased from 4 to 10 h, there was a significant drop in the strength activity indices at 7 days; the indices at 28 days were not affected significantly. The lower

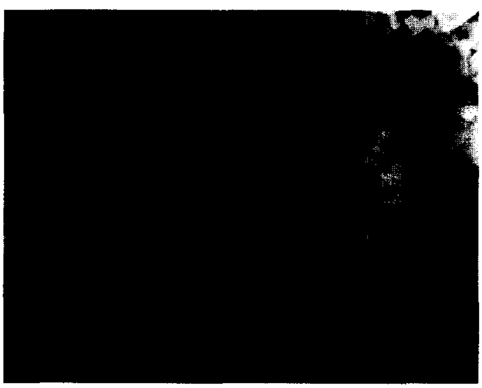

FIG. 12.
Scanning electron micrograph: Lingan fly ash after 10 h of grinding.

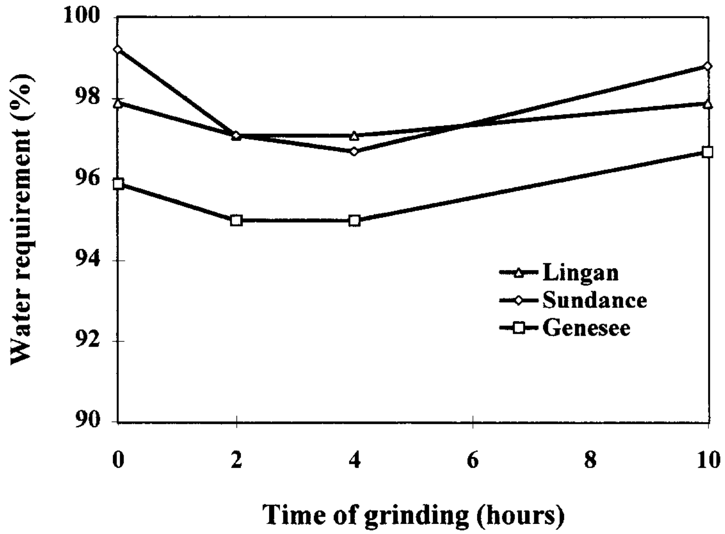

FIG. 13.
The effect of grinding on the water requirement of the fly ashes.

values of the 7-day strength activity indices for the fly ashes after 10 h of grinding may be due to the higher water demand because of the high specific surface of the fly ashes. This was, however, overcome at 28 days because of the increased pozzolanic activity of the fly ashes between 7 and 28 days.

Based on the above results, it appears that there is an optimum grinding time of approximately 4 h beyond which the water requirement increased and the strength activity indices either decreased or did not increase significantly.

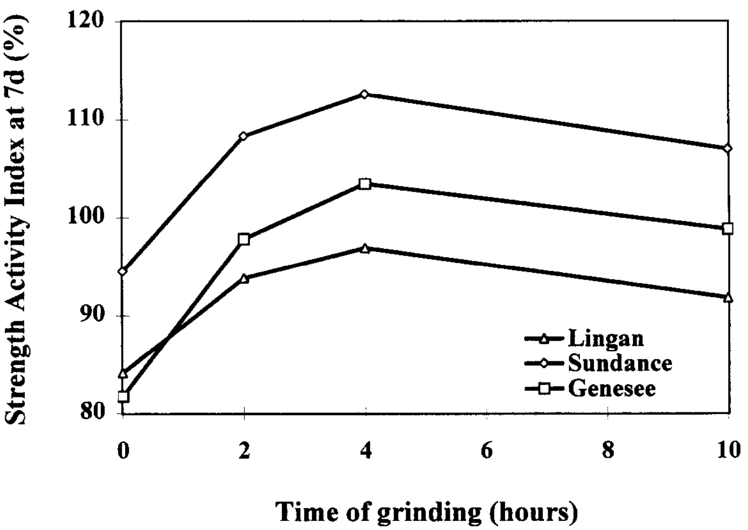

FIG. 14.
The effect of grinding on the strength activity index of the fly ashes at 7 days.

Conclusions

Based on the test results obtained in this investigation, the following conclusions can be drawn:

The specific gravity and the fineness of the fly ashes increased with an increase in the grinding time. However, this increase was less significant beyond 2 h.

The morphology of the fly ashes was changed by grinding. Most of the plerospheres and large, irregular-shaped particles were crushed after 2 h of grinding. However, the number of the spherical particles reduced with increased grinding.

The strength activity indices of the three fly ashes investigated increased with increasing grinding time. There appears to be an optimum grinding time of approximately 4 h for the fly ashes beyond which the water requirement increased, and the strength activity indices either decreased or did not increase significantly.

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