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Effect of grinding time on the particle size distribution of gasification ash and Portland cement clinker

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In recent years the cement and concrete industry has reduced its environmental impact by increasing the use of waste materials as both cement extenders and fillers in concrete. Fly ash has been widely used as a cement extender in concrete for many years but the use of ash from other industries has been limited. In this study the use of ground coarse gasification ash as cement extender is investigated. The effect of grinding time on the particle size distribution (PSD) of gasification ash (GA) and Portland cement (PC) clinker was investigated. The PSD was determined for both blended GA and PC clinker that were first ground separately and interground GA and PC clinker. There appeared to be an optimum grinding time for the GA and interground of GA and PC clinker beyond which the fineness did not increase significantly. The particle size range was narrow after two hours' grinding and any increase in grinding time made it wider for GA and the blended cement. The fineness and Blaine specific surface area of GA and PC clinker increased with an increase in grinding time. However, this increase was less significant beyond two hours. The fineness had an effect on the rate of strength development of the blended cement. The compressive strength, particle size and Rosin-Rammler distribution parameters clearly indicated that grinding time should not be shorter than two hours for interblending and intergrinding of GA and PC clinker.

INTRODUCTION

Portland cement (PC) is an essential material in modern society because, as a major constituent of concrete, it forms a fundamental element of any housing or infrastructure development. The chemical process of making cement clinker produces CO_2, a major greenhouse gas contributing to climate change. For every tonne of PC produced, approximately 1,35 tonne of CO_2 is released into the atmosphere.

In recent years there has been a significant increase in the use of waste materials as both cement extenders (Erdogdu *et al* 1999) and fillers in concrete. The reason for this is a growing awareness of the engineering,

Figure 1 Coarse gasification ash before grinding

Table 1 X-ray fluorescence analysis of gasification ash

Constituent	Gasification ash (%)	Typical values of South African fly ash* (%)
Fe_2O_3	6,80	3,7–4,7
MnO	0,13	0
Cr_2O_3	0,63	0
V_2O_5	0,02	0
TiO_2	1,43	1,4–1,9
CaO	8,17	7,1–10,5
K_2O	0,83	0,5–1,2
P_2O_5	0,7	1,1–1,4
SiO_2	48,5	45–49
Al_2O_3	23,5	29–31
MgO	2,3	1,8–2,8
Na_2O	0,5	0,1–0,8
Cl	0	0
S	0,4	0
SO_3	0,49	0,5–1,0
Loss on ignition	5,18	5,0

* SANS 1491-2:2005 / SABS 1491-2:2005

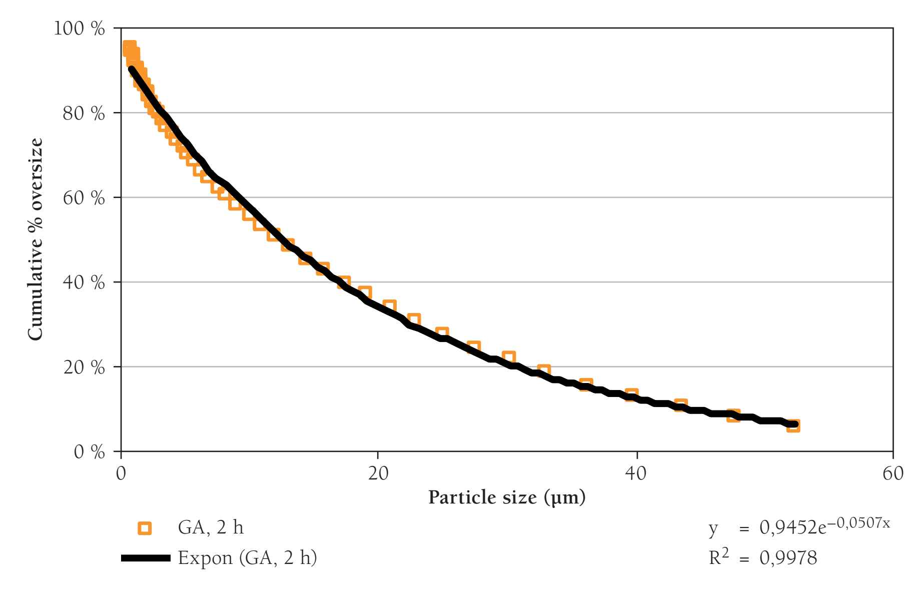

Figure 2 Exponential fit for cumulative % oversize particle size distribution

economical and ecological benefits that the use of waste materials has in the cement and concrete industries. Waste materials such as fly ash are used as supplementary major constituents of Portland cement and as extenders in concrete. Using waste from other industries as a cement extender is an opportunity for the cement industry to reduce its emission of CO_2. Currently pulverised fuel ash (also called fly ash) is widely used as a cement extender in concrete. Fly ash has been widely used as a cement extender in concrete for many years but the use of ash from other industries has been limited. In this study the use of ground coarse gasification ash as cement extender is investigated.

The Sasol Group of companies comprise diversified fuel, chemical and related manufacturing and marketing operations, complemented by interests in technology development, and oil and gas exploration and production. The feed to Sasol gasifiers principally consists of coarse coal (>5 mm) and extraneous rock fragments (stone). During the gasification of this coarse coal at elevated temperatures and pressure, a mixture of carbon monoxide and hydrogen (also referred to as synthesis gas) is produced. The coarse ash is formed at these elevated temperatures and pressure by the interaction of inert minerals present in the coal and stone. The coarse ash (as shown in figure 1) is removed from the gasifier and disposed as a by-product (Van Dyk *et al* 2005). As a coal by-product gasification ash has a similar chemical composition (see table 1) as fly ash, a cement extender commonly used in South Africa.

This study describes the experimental work done at the University of Pretoria to investigate not only the effect of grinding on the particle size distribution of blended and interground GA and PC clinker but also the strength development of cement containing GA.

EXPERIMENTAL DETAILS

Preparation of materials

In this project cement was manufactured in the laboratory using cement clinker obtained from a cement factory. The clinker was ground in a ball mill with 25 kg of round steel balls. The steel balls were individually measured and weighed and their diameters varied between 30 mm and 50 mm. All the milled samples were sieved through a 1,18 mm sieve and stored in airtight containers. Cement is normally manufactured by grinding clinker and a small percentage of gypsum (typically 2 % to 5 %) together. In this experiment 2,5 % fine gypsum, with an average particle diameter of 17,4 μm, was added to the cement after grinding.

Three sets of material were ground for different time intervals to establish an optimum grinding time. The sets consisted of cement clinker, GA and a blend of cement clinker and GA. An optimum grinding time was established by considering the specific surface area, particle size distribution and compressive strength development after grinding material for 30 minutes, 1 hour, 1 hour 30 minutes, 2 hours, 2 hours 30 minutes, and 4 hours.

Specific surface area

At most manufacturing plants a ball mill is used to grind the cement clinker. The principal test carried out at a cement mill is the fineness test in which the specific surface area is determined. The fineness of cement increases with an increase in the grinding time, but Bouzoubaâ *et al* (1997) found that this increase in fineness was less significant beyond two hours. An optimum grinding time of four hours was established by Bouzoubaâ *et al* (1997), beyond which the water requirement increased and the strength either decreased or did not increase significantly.

The fineness of cement is a major factor influencing its rate of hydration, since the hydration reaction occurs at the interface with water (Lea 1997). Greater cement fineness increases the rate at which cement hydrates and thus accelerates strength development. The effects of greater fineness on paste strength are manifested principally at early ages. Portland cement is usually

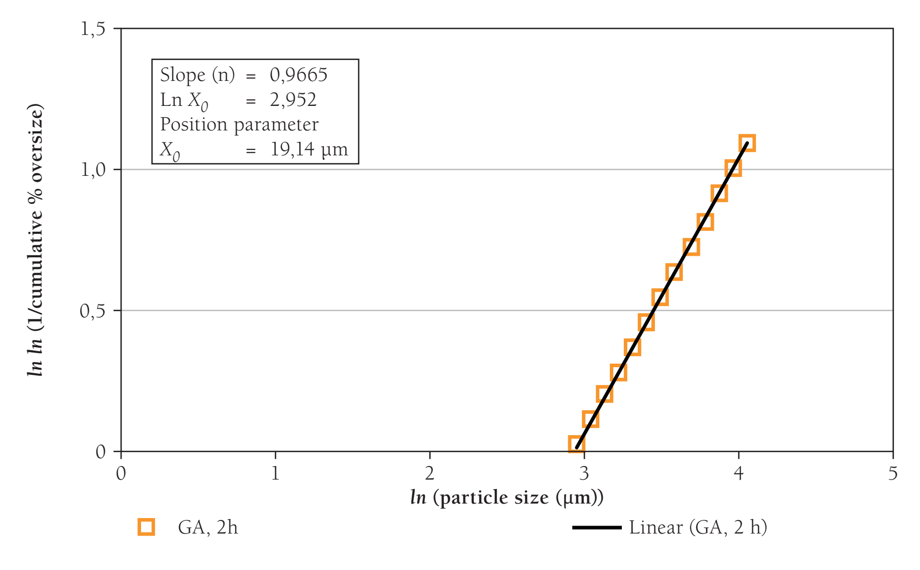

Figure 3 Rosin-Rammler distribution graph

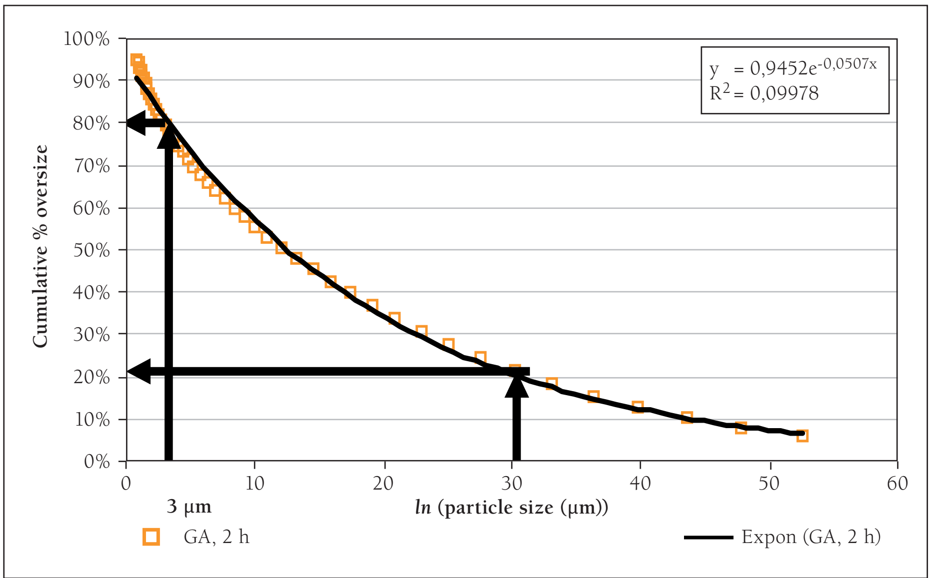

Figure 4 Cumulative % oversize intervals for <3 µm and 3 to 30 µm

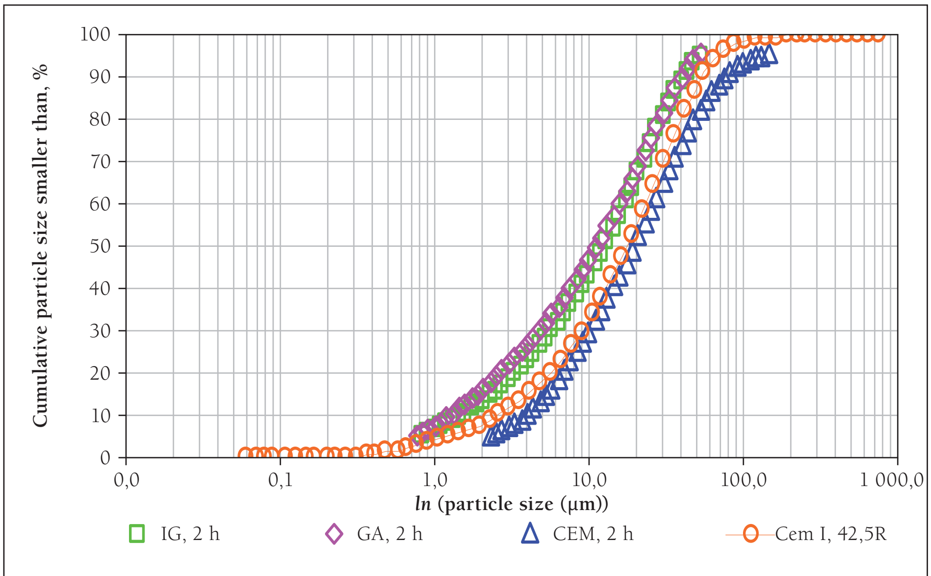

Figure 5 Graph of cumulative particle size distribution of cement and gasification ash (ground separately and interground)

ground to a Blaine specific surface area in the range 300–350 m^2/kg, and rapid hardening Portland cement to 400–550 m^2/kg.

As part of this experiment the Blaine surface area of ground cement and GA was determined after different grinding times to establish whether an optimum grinding time exists. The density of the GA and blended cement was established and the weight of the material used for testing was adjusted to ensure that the volume of the material placed in the Blaine cell remained constant.

Particle size distribution

Laser technology was used to determine the particle size distribution of materials after grinding. A Malvern Mastersizer 2000 wet system was used and the samples were dispersed in distilled water. It was assumed that the cement hydration that takes place during the couple of minutes required to complete the test would not result in a significant change in particle size distribution of the cement particles. The particle size distribution curve of a material provides a number of parameters such as the mean particle size, as well as the distribution of other sizes about the mean. The curve is usually drawn as the cumulative percentage values on the y-axis of particles smaller than the corresponding sizes on the x-axis. The x-axis is drawn to a log scale to accommodate large ranges of particle sizes. The shape of the curve gives an indication of the continuity in size distribution and the slope describes the wideness or range of the size distribution.

Approximately 95 % of cement particles are normally smaller than 45 µm, with the mean particle size around 15 µm. Bye (1999) supported the generally held view that the 3 µm to 30 µm fraction makes a major contribution to the 28-day strength. The range <3 µm is important for achieving high one-day strength.

Cement strength development

Mortar prisms were cast for the different grinding time intervals with a constant replacement of cement with GA of 35 % (m/m). The gypsum content was constant at 2,5 % replacement of the cement content.

A mixture of 450 g cementitious material, 250 mℓ water and 1 350 g standard reference sand was used to cast 40 mm x 40 mm x 160 mm prisms test specimens (SABS 50196 1994). Samples were cast in sets of three prisms and cured in water at 22–25 °C up to the time of testing. Strengths were measured at 2 and 28 days.

PRESENTATION OF PARTICLE SIZE DISTRIBUTION

Rosin-Rammler distribution function

In searching for a parameter which will provide a more representative description of the particle size distribution, the Rosin-Rammler function was introduced.

From a probability point of view Rosin and Rammler investigated the particle size distribution of crushed coal and developed a function that describes the distribution as (Rosin & Rammler 1933):

Table 2 Fitted function of oversize particle size distribution and Rosin-Rammler particle size distribution parameters

Sample	Fitted function y = a e(bx)		Fitted function y = cx + d		X_o	n
	a	b	c	d		
IG, 30 min	0,955	–0,0198	0,9743	–3,7795	47,90	0,9743
IG, 1 h	0,975	–0,0354	0,9848	–3,2664	28,01	0,9848
IG, 1,5 h	0,9929	–0,0514	0,9959	–2,9494	19,88	0,9959
IG, 2 h	0,9835	–0,05	0,9902	–2,9510	20,00	0,9902
IG, 2,5 h	0,9693	–0,0532	0,9813	–2,8498	18,20	0,9813
IG, 4 h	0,9822	–0,0608	0,9896	–2,7544	16,69	0,9896
GA, 30 min	0,9429	–0,0122	0,9653	–4,0996	78,35	0,9653
GA, 1 h	0,9329	–0,0284	0,958	–3,3470	33,05	0,9580
GA, 1,5 h	0,9331	–0,0382	0,9589	–3,0666	24,51	0,9589
GA, 2 h	0,9452	–0,0507	0,9665	–2,8296	19,14	0,9665
GA, 2,5 h	0,9254	–0,0554	0,9543	–2,6897	16,64	0,9543
GA, 4 h	0,8942	–0,0781	0,934	–2,2793	10,93	0,9340
Cem, 30 min	0,9675	–0,024	0,9816	–3,6309	37,71	0,9816
Cem, 1 h	0,7121	–0,0144	0,816	–3,1629	33,11	0,8160
Cem, 1,5 h	0,9411	–0,0249	0,9662	–3,5129	37,29	0,9662
Cem, 2 h	0,8794	–0,0237	0,9307	–3,3684	29,43	0,9307
Cem, 2,5 h	0,9077	–0,0247	0,9469	–3,4172	32,04	0,9469
Cem, 4 h	0,8179	–0,0179	0,8908	–3,4053	25,71	0,8908

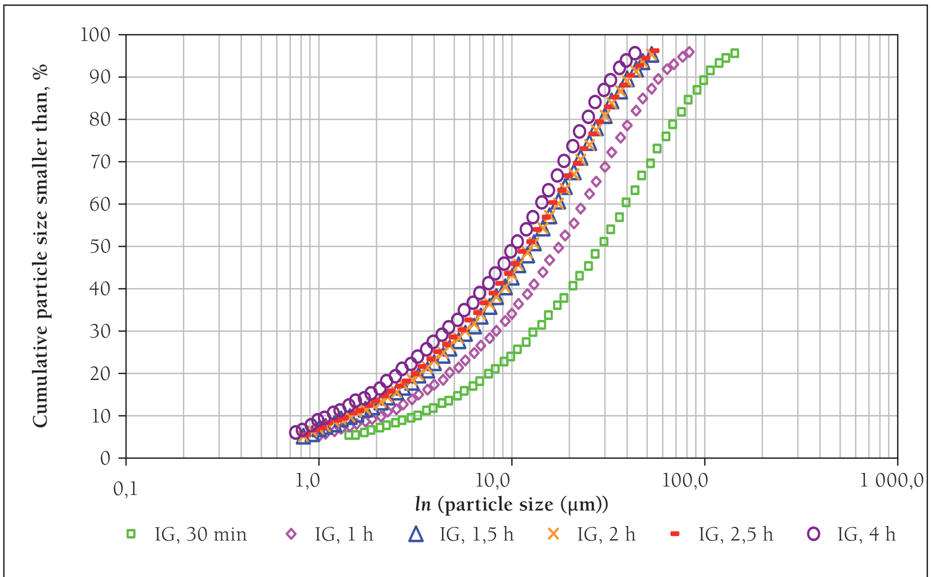

Figure 6 Particle size distribution of gasification ash and cement interground for different periods

$f(x) = exp(-bx^n)$

Where:

b = fineness characteristic measure of the material being analysed

n = a measure of the range of particle sizes

Rosin and Rammler also found that the function does not only apply to crushed coal but also to various other powered materials. The function was modified as follows:

$RR = exp -(x/X_o)^n$

Where:

the weight function $f(x)$ is denoted as RR

X_o = the absolute size constant or position parameter (it represents the particle size for which 36,8 % of the particles are coarser)

Taking the double logarithm of the above mentioned equation gives:

$lnln(1/RR) = n(lnx-lnX_o)$

This equation describes a straight line plot with a coordinate system made up of a log scale abscissa for the particle size x, and an ordinate with a double logarithm of the reciprocal of the residue RR. The slope of the straight line is n and the line intercepts the horizontal axis at a value describing the particle size X_o (Olorunsogo 1990).

Samples might have similar ranges of size distribution (denoted by equal n) but with possible varying degrees of fineness (indicated by different X_os). Samples might be of the same fineness (because of the same X_o) but possibly with different size ranges (denoted by different slopes) (Wainwright & Olorunsogo 1999).

By plotting the inverse of the cumulative percentage distribution, the cumulative % oversize particle distribution is obtained. Provision was made for statistical outliers by not taking the 5 % smallest diameters and 5 % largest diameters into account. For each sample a trend line is added to the cumulative % oversize graph (see figure 2).

The cumulative % oversize particle size distribution as discussed above can be represented as a Rosin-Rammler distribution. The Rosin-Rammler distribution graph in figure 3 is an example of how the values for the particle size distribution parameters were derived. The modified Rosin-Rammler distribution graph $lnln(1/y)$ versus lnx is plotted, with the fitted functions $y = a\ exp(bx)$ for the cumulative % oversize particle size distribution and x the particle size. A linear trend line and equation are also added to these graphs (see figure 3). The slope and interception with the horizontal axis of the line are taken as the n value and $ln\ X_o$ value of the modified Rosin-Rammler function respectively.

With this analysis 36,8 % of the particles are greater than the X_o value (position parameter in µm). This parameter is an indicator of the fineness. The n value (or slope of the graph) represents the range of the particle size distribution of the particle sizes greater than X_o.

Particle size parameters

Figure 4 indicates the 3 µm and 30 µm oversize particle size distributions. These particle sizes can give an indication of the % of the particle which lies in the <3 µm and 3–30 µm intervals, which correspond to values used by cement manufacturers to limit the fineness of cement.

RESULTS

Particle size distribution

The water demand and workability of cement paste is controlled by the particle size distribution. The cementitious activity of cement can be enhanced by controlling the particle size distribution.

The particle size distribution of material ground for two hours in comparison to that of a commercially available Cem I 42.5R

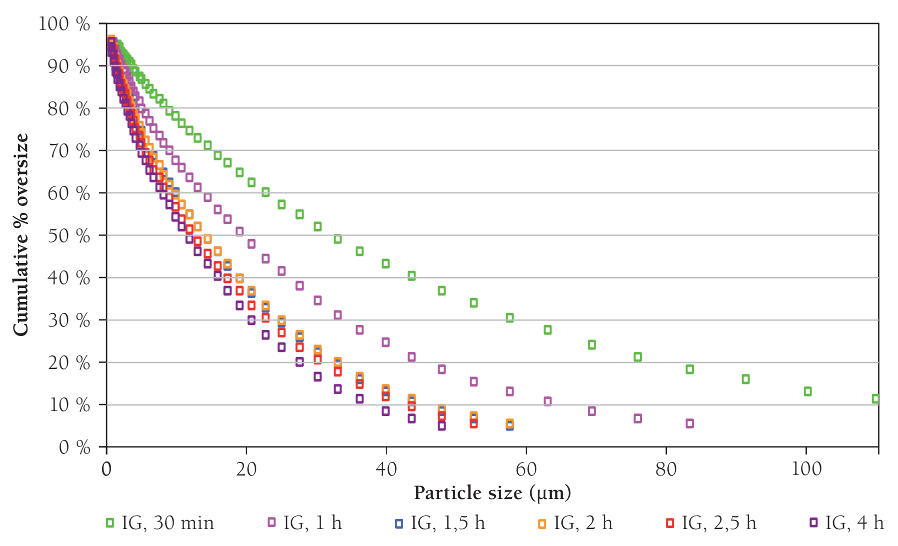

Figure 7 Summary of exponential fitted functions for the cumulative % oversize particle size distribution of gasification ash and cement interground for different periods

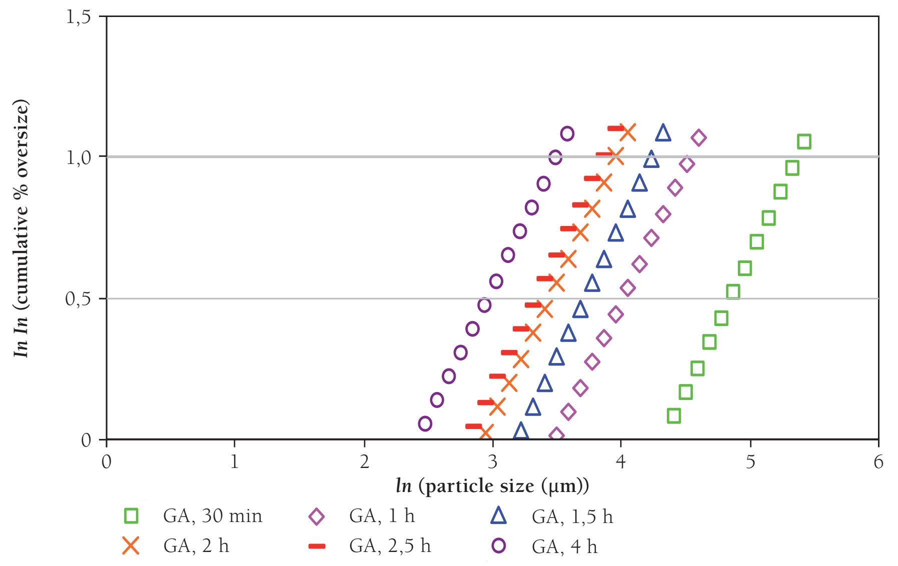

Figure 8 Summary of Rosin-Rammler distributions of gasification ash

cement can be seen in figure 5. Although the particle size ranges are in the same order of magnitude, the GA is significantly finer than the cement clinker after two hours of grinding. The GA had a particle size range between 0,08 µm and 50 µm, whereas the cement particles ranged between 2 µm and 110 µm. These results indicate that the PC clinker is more difficult to grind than the GA. It is however interesting to note that the particle size distribution of the blended material, where 35 % of the cement clinker had been replaced with GA before grinding (IG), is similar to that of the GA.

The particle size of the material that was interground shows that the particle size distribution for time intervals 1,5 hours and 2 hours was identical. Further grinding did not increase the fineness of the interground sample significantly (see figure 6). Thus, from the particle size distribution it is clear that an optimum grinding time can be established.

The relevance of the Rosin-Rammler particle size distribution parameters was evaluated by comparing the fitted functions for the cumulative % oversize particle size distributions for different grinding times. Both the constants in the equation vary with grinding time and therefore the constants as such cannot be used to compare the samples (see table 2).

The particle size distribution functions for GA are plotted in figure 7. The graph clearly indicates that the particle size of GA decreases as the grinding time increases. The fitted trend line that best describes the measured values is an exponential function. The statistical R-square value for all the samples of the exponential fit is between 0,995 and 0,999. The exponential equation is therefore a true representation of the cumulative % oversize particle size distribution.

The values of the slope n and the position parameter X_o for all the samples can be seen in table 2. The equations for the linear trend line fitted to the Rosin-Rammler distribution graph are also listed in table 2.

According to figure 7 the particle size distribution decreases as the grinding time increases for GA. It is clear that the grinding time has a significant influence on the range of the particle size distributions greater than the position parameter (X_o). The shorter grinding times seem to result in ground material with larger (coarse) particles. This trend can also be seen when comparing the Rosin-Rammler parameters (see figure 8).

As the grinding time is extended the particles become finer, resulting in the position parameter decreasing. This trend is prominent for the GA as well as the blended cement. The PC clinker alone has no clear trend, since the position parameter of the cement after 30 minutes is less than that of intergrinding of gasification ash, but after one hour the position parameters for all three are approximately the same. Hereafter the position parameter for both gasification ash and intergrinding continues to reduce as grinding time is extended, while the cement position parameter does not reduce further.

The position parameters for cement range between 25 µm and 38 µm, and there is no clear trend for the position parameter of cement reducing with increased grinding time. The position parameter of GA and interground GA and PC clinker is significantly smaller than that of cement. After two hours of grinding, the reduction in position parameter for both GA and the interground blend of GA and PC is not as noticeable as before. An exponential relation exists between particle size and grinding time and it seems as if an optimum grinding time can be established. For both the GA plus PC clinker and GA on its own this optimum seems to be in the region of two hours. Grinding for longer than this optimum time would not decrease the particle size considerably more and would only add to the cost of grinding.

The grinding time does not have a significant influence on the slope (n) value for GA plus PC clinker and GA on its own, which is an indication of the spread of particle sizes.

The percentage of particles >45 µm, between 30 and 3 µm, and <3 µm is listed in table 3. These results indicate that with increased grinding time the percentage of particles <3 µm increases. For cement the percentage remained almost constant between 52 and 58 %. For both the GA plus PC clinker and GA on its own the percentage particles <3 µm increased as grinding time increased. All of the results in the table indicate that with increased grinding time the particles become smaller.

Specific surface area

The Blaine surface-specific surface area for PC clinker, GA and interground PC clinker and GA is seen in figure 9.

Table 3 Oversize particle size distribution parameters

Sample	> 45 µm (%)	3–30 µm (%)	< 3 µm (%)	28-day compressive strength (MPa)	2-day compressive strength (MPa)
IG,30 min	35,12	41,73	9,18	27,3	11,8
IG, 1 h	17,19	55,2	13,76	32,9	13,3
IG, 1,5 h	7,66	62,05	18,01	40,1	17
IG, 2 h	7,89	62,18	18,48	43,2	21,5
IG, 2,5 h	7,05	62,74	19,65	41,6	19,5
IG, 4 h	4,09	64,44	22,07	43,6	19,2
GA, 30 min	50,87	31,78	7,34	–	–
GA, 1 h	23,59	48,51	14,82	–	–
GA, 1,5 h	14,70	56,3	16,55	–	–
GA, 2 h	7,32	59,57	22,01	–	–
GA, 2,5 h	5,89	59,79	25	–	–
GA, 4 h	3,94	58,34	32,48	–	–
Cem, 30 min	27,17	52,82	5,66	–	–
Cem, 1 h	24,86	57,08	6,32	–	–
Cem, 1,5 h	23,64	55,41	6,83	–	–
Cem, 2 h	21,92	57,94	7,13	44,9	17,6
Cem, 2,5 h	22,34	56,99	7,42	–	–
Cem, 4 h	26,40	53,25	7,97	–	–
IB, 30 min	35,46	43,14	5,62	30,8	10,1
IB, 1 h	24,05	52,01	8,25	32,1	11,9
IB, 1,5 h	20,50	52,62	9,95	33,1	11,2
IB, 2 h	16,81	56,40	11,30	45,1	20,1
IB, 2,5 h	16,59	56,02	12,43	45,4	18,4
IB, 4 h	18,54	53,62	16,97	43	17,6

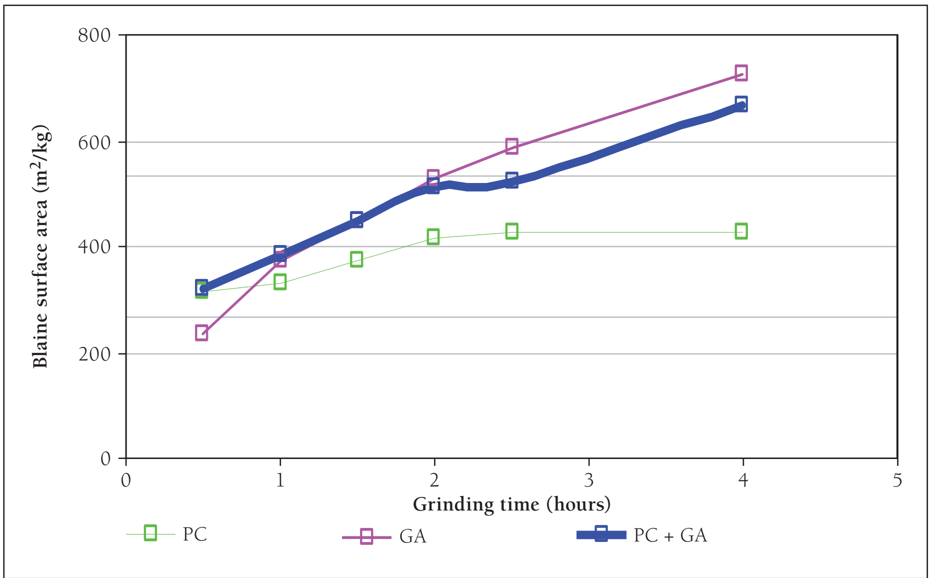

Figure 9 Graph indicating Blaine surface areas

From the graph it can be seen that cement has a surface area ranging between 300 m^2/kg and 450 m^2/kg. This range of surface area corresponds to commercial cement, as reported by Bhatty (2004). The surface area of cement increases for the grinding time periods for up to two hours, after which the surface area remains constant for 2,5 hours and slightly decreases for four hours of grinding. This indicates that longer grinding does not result in increased surface area. There seems to be an optimum grinding time resulting in the maximum surface area for the cement. Initially after 30 minutes, gasification ash has the lowest surface area, while the surface area of the interground sample is close to the cement sample surface area. From one hour of grinding, both the GA and interground samples have higher surface areas than the cement. The surface area for GA and interground PC clinker plus GA is approximately the same up to two hours of grinding. Thereafter the gasification ash's surface area increases more than the interground PC clinker plus GA. This indicates that the surface area of GA increases, while the PC clinker reached an optimum surface area. This is also true for the interground PC clinker plus GA.

Effect of grinding time on the strength development of cement

The compressive strength increased with decreasing position parameters (X_o) for interblended PC clinker and GA as well as interground GA and PC clinker in the ball mill. Thus finer particles resulted in greater strengths.

The percentage of particles in the 3 µm to 30 µm fraction used by cement manufacturers as a limit on fineness, effectively shows that as grinding time increased, the percentage of particles in the fraction increased. As the percentage of particles in the 3 µm to 30 µm fraction increased, the compressive strength increased. This trend is observed for both interblended GA and PC clinker and interground GA and PC clinker in the ball mill. For interblended GA and PC clinker it was however observed that the percentage particles <3 µm and in the 3 µm to 30 µm fraction did not significantly increase for increased grinding time.

For the <3 µm particle size fraction the percentage increased with increased grinding time for both interblending of gasification ash and cement ground separately in the ball mill and intergrinding of gasification ash and cement clinker in the ball mill. As the percentage of particles <3 µm of the interground PC clinker plus GA increased, the compressive strength increased. Thus, as the particles became finer the strength increased. As the ash is softer than the cement clinker, it will be ground preferentially, with the result that the interground material will have more fine gasification ash particles compared to the blended materials.

CONCLUSION

The GA ground separately and interground GA and PC clinker had similar particle size distributions. There is a considerable difference in particle size distribution between the GA and PC clinker ground separately for the same time interval. This indicates that the cement clinker is harder than the gasification ash.

As the grinding time was extended the particles became finer, resulting in the position parameter decreasing for the GA as well as the blended GA plus PC clinker. The PC clinker alone had no clear trend.

The specific surface area of PC clinker increased for grinding times for up to two hours, after which the surface area remained constant within the error of the method. This indicates that for the particular setup an optimum grinding time is achieved.

The surface area of GA and the interground blend of GA and PC was approximately the same for up to two hours of grinding, Thereafter the surface area of the GA increased more than that of the interground blend of GA and PC.

The compressive strength, particle size, and Rosin-Rammler distribution parameters clearly indicated that grinding time should not be shorter than two hours for interblending and intergrinding of GA and PC clinker. It seems that an optimum grinding time can be established. For both the GA and the interground blend of GA and PC this optimum seemed to be in the region of two hours.

The percentage particles in the <3 µm and 3 µm to 30 µm fractions increased with an increase in grinding time for both interblended GA and PC clinker ground separately in the ball mill and GA and PC clinker interground in a ball mill. Compressive strength increased with an increase in percentage particles in both the <3 µm and the 3 µm to 30 µm fractions in the case of interblended as well as interground of GA and PC clinker. Interblending of GA and PC clinker in the mixer showed that the percentage particles <3 µm did not increase significantly with an increase in grinding time of the individual constituents. The compressive strength for the interground material was slightly more after 28 days for a higher percentage particles <3 µm.

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