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A study of intergrinding and separate grinding of blast furnace slag cement

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Abstract

Grinding-related parameters of blast furnace slag cements (BFC), such as Bond grindability, specific rate of breakage and breakage distributions were determined employing separate and intergrinding modes. Strength tests were performed on mortar specimens made by BFC prepared by these modes of grinding to the same fineness. Overall results favor the use of separate grinding mode in view of lower specific energy consumption, ease of manufacture, higher addition of slag on top of more flexible product quality arrangement.

Keywords: Breakage; Kinetics; Granulated blast-furnace slag; Work index; Strength

1. Introduction

Since the first discovery of cementitious properties of blast furnace slag (BFS) by Emil Langen in 1862 and particularly after the oil crisis in 1970s, manufacture of blast furnace slag cement (BFC) has substantially increased due to greater emphasis on energy conservation, utilization of waste materials and certain technical advantages over ordinary Portland cement (OPC), such as higher resistance to aggressive conditions. Today, BFC accounts for nearly 15% of all cement used in Western Europe [1] and 15% of total cement production in India [2].

Turkey is one of the leading manufacturers of cement and steel in Europe, producing around 35 Mt of cement and 13 Mt of crude steel annually [3]. However, the manufacture of BFC was about 0.75% and 2.2% of the total Turkish cement production in 1996 and 1998, respectively, which is now fast increasing with installation of new slag grinding mills around the country utilizing all slag production from the three integrated steel works in Turkey.

BFC has been traditionally produced by intergrinding cement clinker with slag in tube mills. A recent and more popular concept is to grind clinker and slag separately and mix them according to market requirements. Separate grinding can be performed in vertical mills, roller presses and even Horomills as well as tube mills which still remain as the main milling system in cement grinding [4].

In recent years, there have been some interests in studies to compare the intergrinding and separate grinding of BFC, and various advantages/disadvantages of each mode have been put forward [5–7]. In this study, the same issue was investigated from the point of view of their grindability, grinding kinetics and strength properties. Bond grindability tests revealed higher specific energy consumption for intergrinding. Breakage properties as determined by Austin et al.'s BII model [8] on clinker and slag samples as well as on their various mixtures revealed considerable differences between clinker and slag; those for the mixtures lying in between the two.

In this paper, the results of kinetic tests will be given as a summary for it will be subject of another paper.

Strength tests on BFC produced by separate grinding gave higher values for 2, 7 and 28 days as compared to the BFC produced by simultaneous grinding to the same Blaine specific surface.

Based on overall results, it was concluded that separate grinding should be preferred in view of lower specific energy consumption, ease of manufacture, higher addition of slag (i.e., less environmental hazards) on top of higher flexibility in product quality arrangement according to market requirements.

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Table 1
XRF analysis of clinker and slag

	Clinker	Slag
Chemical composition (%)		
SiO_2	20.50	42.29
Al_2O_3	5.66	10.56
Fe_2O_3	3.87	0.32
CaO	64.99	37.35
MgO	2.43	6.71
K_2O	0.83	0.94
TiO_2	0.29	0.45
Mn_2O_3	0.06	1.67
P_2O_5	0.09	0.011
Cr_2O_3	0.023	0.012
Na_2O	0.04	0.18
Loss on ignition	0.5	0.55
Free lime	–	–
Insoluble residue	–	–
SO_3	–	–
Main composition (%) Bogue		
C_3S	65.16	–
C_2S	9.82	–
C_3A	8.46	–
C_4AF	11.78	–

2. Material characterization

Clinker and slag samples were obtained from Oysa-Iskenderun cement grinding plant. They were characterized for their chemical, mineralogical and mechanical properties by XRF, XRD, SEM and Bond grindability tests. XRF analysis are given in Table 1, where clinker phases present are also shown as calculated by Bogue's formula.

XRD results shown in Fig. 1 confirms the presence of main mineral phases in the clinker sample and the presence of high quality amorphous material in the slag sample. Examplary SEM photographs of both clinker and slag are presented in Fig. 2.

3. Experimental work

3.1. Bond grindability tests

The ball mill grindability tests were conducted in a standard Bond mill for 90-μm test sieve. As received, the slag was finer and hence the clinker was also reduced down to similar fineness by crushing in a roll crusher and −3.36 mm samples were used as feed materials for the tests. Grindabilities of various blends of clinker and slag were also determined. The Bond grindability test procedure is well documented in the literature and hence the repetition is avoided here [9].

3.2. Kinetic tests

The kinetic characterization of grinding involves two functions, namely breakage rate and breakage distribution.

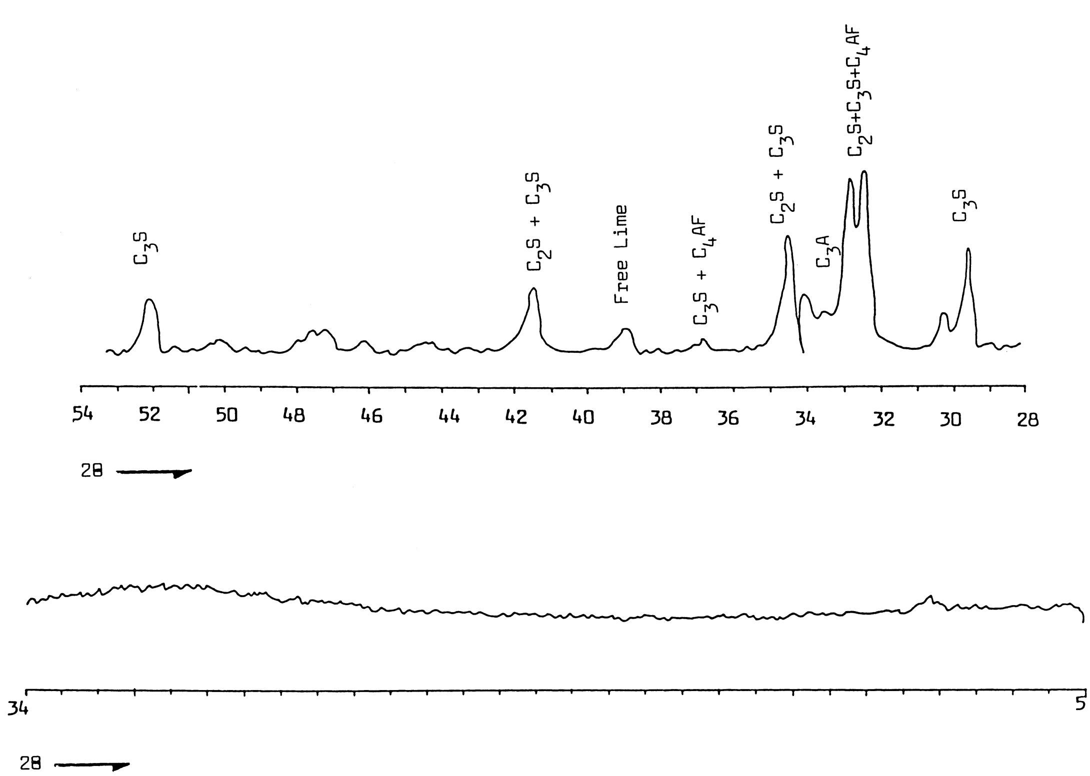

Fig. 1. XRD patterns of clinker and slag.

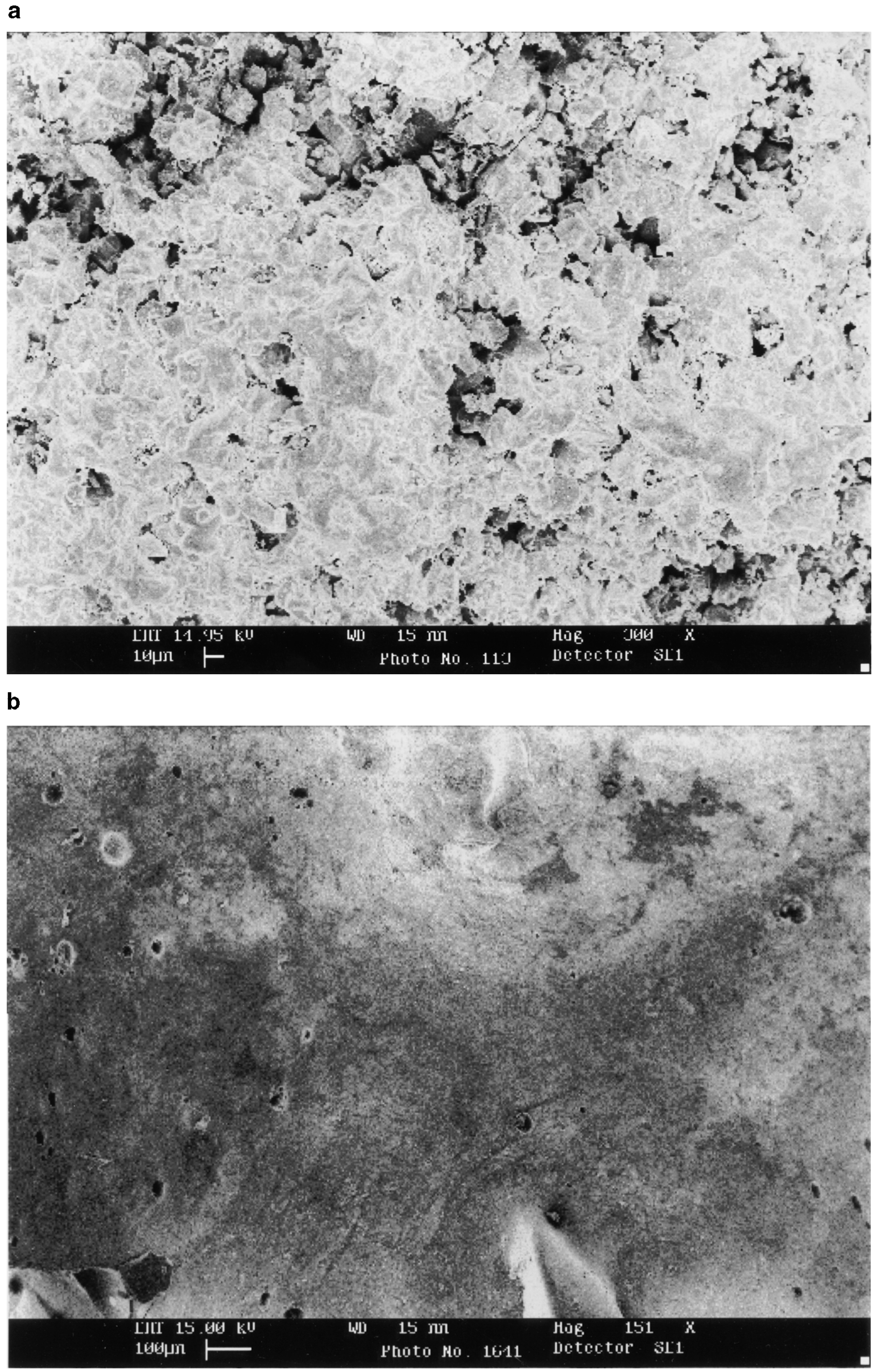

Fig. 2. SEM photographs of clinker (above) and slag (below).

Breakage rate function refers to the disappearance rate of individual particle size fraction while breakage distribution defines how the broken fragments are distributed among the finer fractions. For batch grinding, both functions can be determined experimentally by carrying out kinetic tests.

For a given size fraction the rate or specific rate of breakage, S_i, is determined using the data generated by

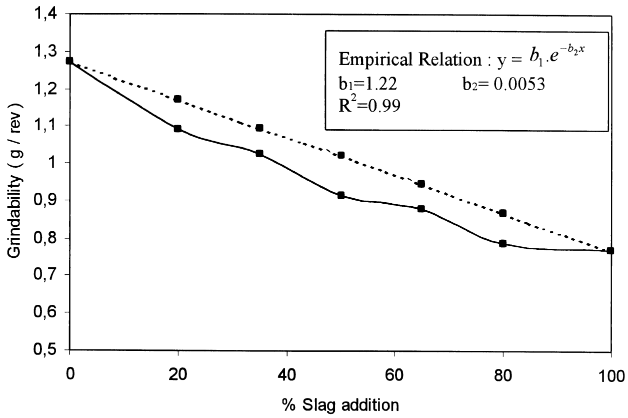

Fig. 3. Variation of grindabilities of clinker, slag and their mixtures.

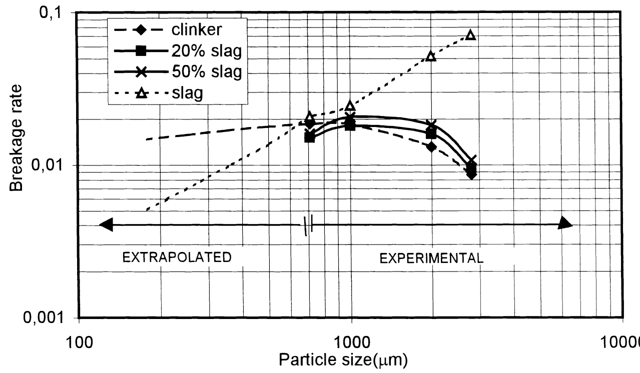

Fig. 5. Variation of specific breakage rate vs. particle size.

laboratory kinetic tests on monosize fractions. The data are expected to fit the following equation when the breakage is first order.

$$\log W_i(t) = \log W_i(0) - S_i t/2.3 \quad (1)$$

where W_i is the weight fraction of material of size i, S_i is the specific rate of breakage of size i and t is the time.

As Eq. (1) implies, S_i, is determined from the slope of line passing through the points of percent oversize vs. time, plotted on a log vs. linear scale.

The kinetic tests were conducted in a standard Bond ball mill under standard conditions on four monosize fractions of clinker and slag samples, namely $-2.8+2.0$; $-2.0+1.4$, $-1.0 + 0.71$, and $-0.71 + 0.05$ mm fractions, and on their various mixtures having 20%, 35%, 50%, 65% and 80% slag. The monosize fractions were prepared by roll crushing followed by Russel screening. Fractions separated by Russel screening were further hand screened in Tyler sieves to have at least 95% of the material in the specified monosize fractions [8].

The product size distribution from a mono feed size at a short grinding time (0.25 min) was used to determine the breakage distribution function, B, values according to Austin et al.'s BII method [8]. Size distributions were determined by dry screening on Tyler sieves.

3.3. Strength tests

The mortar specimens of OPC and BFC with varying slag content and fineness were prepared according to the Rilem–Cembureau method in a laboratory of 20 ± 2% °C and 50 ± 5% relative humidity. Test specimens were of 40 ×

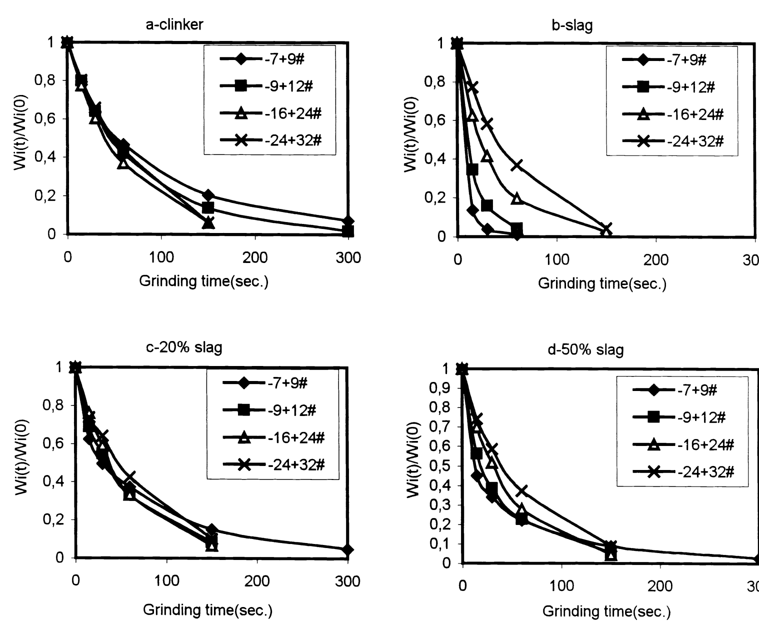

Fig. 4. Rate of disappearance of various monosize fractions of clinker, slag and their mixtures.

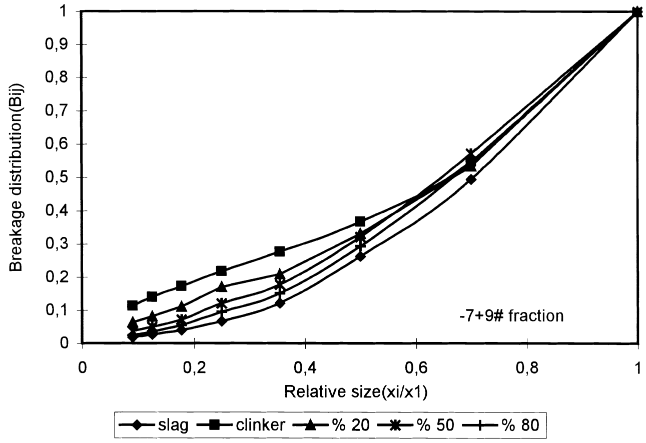

Fig. 6. Variation of B values for $-7+9$ # fraction for clinker, slag and their various mixtures with relative particle size.

40 × 140 mm in size, and following demolding after 24 h, were kept in water until they were broken.

4. Results and discussion

The results of Bond grindability tests are given in Fig. 3. It is clear from Fig. 3 that, as expected, grindability of slag is lower than clinker, i.e., it is more resistant to grinding. A more interesting point highlighted in the results is the change of grindability with the amount of slag added to the mixture. When different mixtures of materials with varying grindabilities are ground, the grindability of the mixture is commonly taken as the weighed average of the component grindabilities. If it had been the case, the grindabilities of the mixtures would have been presented by the linear dotted line as seen in Fig. 3. However, as Fig. 3 shows, this is not the case and the grindability of the mixtures is always lower than this average. Bond test simulates closed-circuit grinding, and this would be due to the harder slag comprising the circulating load to a greater extent [10]. Nevertheless, the grindabilities of the mixtures can be expressed by an empirical equation given in Fig. 3, where model constants of the relation are also presented.

Kinetic behavior of clinker, slag and their mixtures having 20% and 50% slag are graphically presented in Fig. 4. Results for three other mixtures have similar trends and are not depicted here. The separate grinding behavior of both materials (Fig. 4a and b) may generally be classified as first order. Only the coarsest fraction ($-7+9$ #) of slag displays appreciable deviation from first order. This may be attributed to the ball size being improper for this size. Nevertheless, it was assumed that both materials displayed first-order breakage, and specific rate of breakage of each size fraction was calculated using a non-linear regression algorithm and presented in Fig. 5.

However, intergrinding the mixtures resulted in mostly non-first-order breakage rate. This is the expected behavior when two materials exhibit independence when they are interground. As expected, larger deviations from first order occurred in size fractions at which clinker and slag had most divergent S_i values.

Experimental breakage distribution functions for clinker, slag and their mixtures having 20%, 50% and 80% slag are shown in Fig. 6. It appears that the primary breakage of

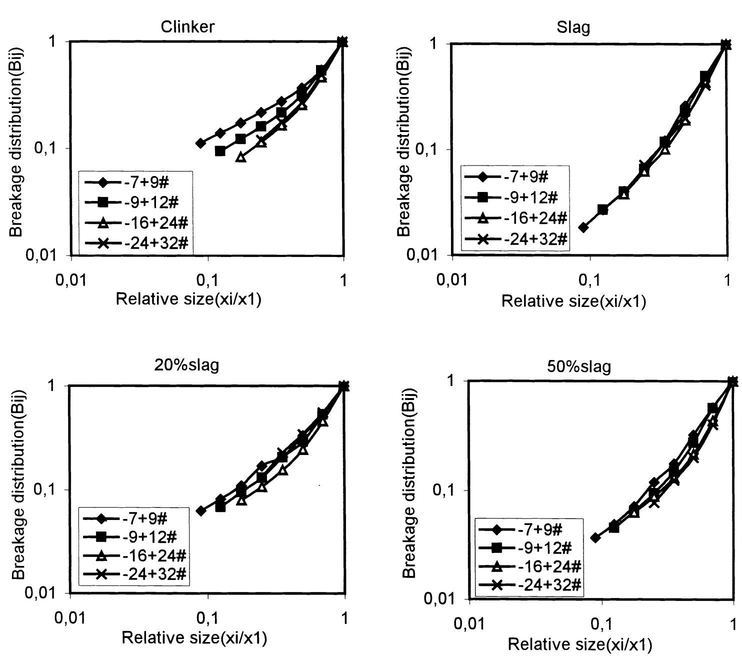

Fig. 7. Variation of breakage distribution for various feed sizes of clinker, slag and their two mixtures with relative particle size.

clinker produces finer size distribution as compared to that of slag. The primary breakage distributions for the mixtures are in between the two, systematically shifting towards slag with increase in slag ratio. Although clinker has lower rates of specific breakage, the breakage distributions show that it produces a finer product when it is broken. This eventually leads to a higher grindability.

Variation of breakage distributions for various feed sizes of clinker, slag and of their various mixtures are presented in Fig. 7, where clinker and slag exhibit non-normalizable (dependent on the initial size) and normalizable distribution values, respectively. The mixtures show intermediate behavior depending on the slag addition.

When the results of Bond grindability and kinetic tests are compared, the striking point is the fact that the clinker, which has a higher Bond grindability, has lower specific rate of breakage within the size range studied. This gives an impression that the two results are contradictory. A close evaluation of the available data provides some explanation for such behavior. First, the specific rate of breakage for both materials become very similar at the finest fractions studied. If the breakage function curves are extrapolated towards the fine-size range, the two curves may be expected to intercept reversing the position of the two materials. The two sets of specific rate of breakage data were fitted separately to a commonly used breakage rate function given in Eq. (2), and plausibility of the above hypothesis was investigated. The breakage rate function is given below,

$$S_i = A\left({X_i}/{X_0}\right)^{\alpha}\left(\frac{1}{1+\left({X_i}/{\mu}\right)^{\Lambda}}\right) \quad (2)$$

where X_i is the lower limit of the interval i (mm), X_0 is the initial particle size and A, α, μ and Λ are model parameters that depend on the grinding conditions and the properties of the material being ground.

The parameters of the function, A, α, μ and Λ, were calculated using a non-linear regression algorithm and are given in Table 2. With the use of these parameters, grinding rate curves for slag and clinker were extrapolated as seen in Fig. 5, which indicates that slag would have lower rate below approximately 650 μm. This explains why slag has an overall lower grindability although it needs experimental verification of grinding rates for these fine sizes below 650 μm.

Table 2
Model parameters for clinker, slag and their various mixtures

	Breakage rate parameters				Breakage distribution parameters			
Material	A	α	μ	Λ	φ	γ	β	δ
Clinker	0.0213	0.2127	2.107	2.565	0.385	0.667	3.732	0.081
20% slag	0.0197	0.697	2.000	3.518	0.513	1.325	14.561	0.466
50% slag	0.0231	0.955	1.804	3.536	0.226	1.000	4.162	0.453
Slag	0.053	0.9587	1.875	0	0.099	1.184	13.521	0.7

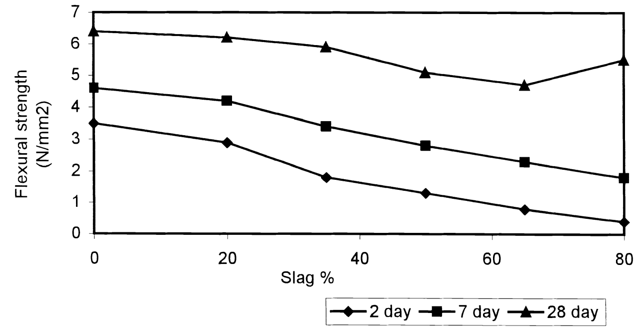

Fig. 8. Variation of flexural strength with slag addition.

Secondly, as shown by the primary breakage distributions (Fig. 6), despite its higher breakage rate, slag produces a coarser product when it is broken. This is also an important factor determining the Bond grindability of a given material.

Strength tests were conducted in order to evaluate the effect of following parameters on strength properties of the cements:

- slag ratio,
- slag fineness,
- separate and simultaneous grinding.

Figs. 8 and 9 illustrate flexural and compressive strength of the OPC and of the BFC having varying amounts of BFS that were produced by simultaneous grinding of the components to the Blaine fineness of 3000 cm^2/g. The results show considerable decrease in both strengths with the increase in slag content of BFC for all three curing ages studied. This is probably due to the low reactivity of slag in hydration reactions in this fineness range which implies that the slag should be ground finer to achieve the required strength [11]. To verify this finding, additional tests were performed on BFC samples produced by grinding clinker + gypsum to the Blaine surface of 3000 cm^2/g and mixing it with equal proportions of separately ground slag having varying fineness between 3000 and 6000 cm^2/g. The results presented in Figs. 10 and 11 show that the flexural strength of BFC is not

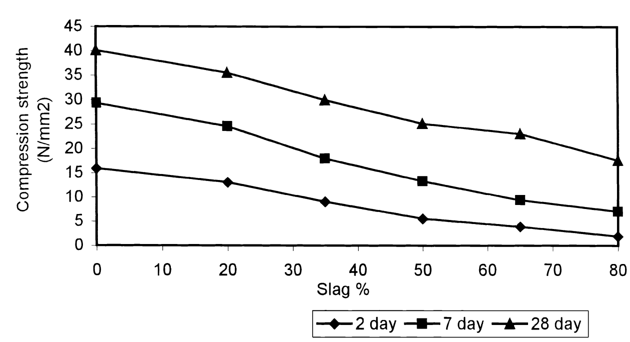

Fig. 9. Variation of compression strength with slag addition.

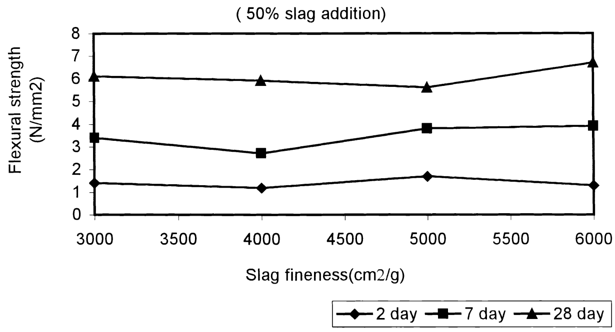

Fig. 10. Variation of flexural strength with slag fineness.

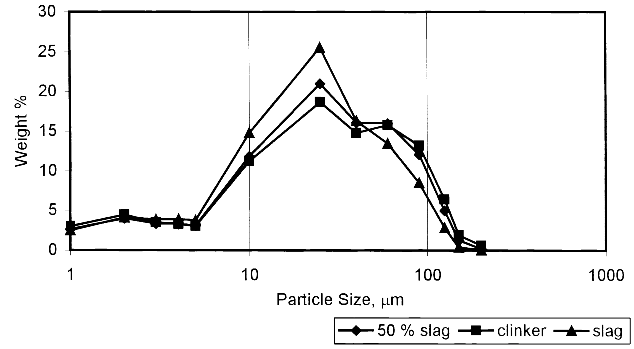

Fig. 12. Laser diffraction particle size analysis.

improved with the increasing fineness of slag, but the compressive strength values increase with increasing fineness of slag, this being more pronounced with 28-day curing age.

Another significant point can be reached when the strength values for the two BFC each having 50% slag and 3000 cm^2/g Blaine-specific surface but produced differently, i.e., by co-grinding and by separate grinding are compared. The relevant flexural (Figs. 8 and 10) and compressive (Figs. 9 and 11) strength values for intergrinding are lower than the values for separate grinding for all the curing ages, the effect being more pronounced for longer curing ages. It is believed that this is because of the differences in grindabilities of clinker and slag. In case of intergrinding, the overall size distribution of the mixture is the summation of the separate distributions representing the two components. When grindabilities of the components differ, their individual distributions are also different. Due to preferential grinding, the harder component, slag, tends to accumulate in coarse fractions having narrower size distribution, higher mean size and lower specific surface area compared to the separately ground blends. The softer component, clinker, being ground at a faster rate would accumulate in finer size fractions having wider size distribution, lower mean size and higher specific surface area compared to the blend. With this reasoning, although the specific surface areas of the two BFC are the same, the slag in the interground BFC is relatively coarser than the slag ground separately. Since the coarser slag would not take part in hydration reactions as fast as the fine slag it would decrease the strength values of the BFC produced by intergrinding.

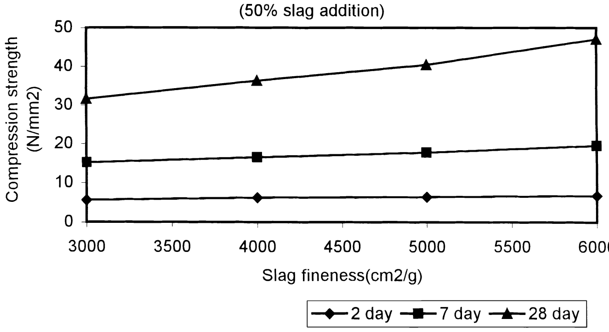

Fig. 11. Variation of compression strength with slag fineness.

To verify this argument, weight fractions retained on 38- and 45-μm sieves were determined by Alpine sieving apparatus for BFC samples interground and separately ground to 3000 cm^2/g fineness. The weight fractions retained on both sieves were higher in case of intergrinding, indicating accumulation of slag in coarse fractions. The relevant figures were 35%, 30% for 38 μm and 28%, 23% for 45 μm.

Particle size distributions for the clinker and slag ground separately to 3000 cm^2/g Blaine fineness as determined by Malvern laser diffraction analyzer are presented in Fig. 12, where it is seen that the harder component, slag, has a narrower distribution than the softer component, i.e., clinker, as also suggested by other researchers [12]. The distribution for a mixture having 50% slag interground to the same fineness lies in between.

5. Conclusions

Based upon evaluation of the results following conclusions can be drawn.

- Clinker and BFS have considerably different grindabilities, with the slag being harder to grind.
- Clinker and BFS have differences in their grinding kinetics, with the clinker having lower rate in coarse fractions and probably higher rate in fine fractions as compared to slag.
- Breakage distribution values for clinker and slag are normalizable and non-normalizable, respectively.
- Bond grindabilities of mixtures are lower than the weighed average of the grindabilities of the components for all slag additions. This indicates that the specific grinding energy per specific surface necessary to produce BFC is greater when the components are interground.

- Slag being harder than clinker gives relatively narrow size distribution when it is ground to the same fineness.
- In case of intergrinding, slag having lower grindability accumulates in coarser fractions; with the clinker having higher grindability accumulates in finer fractions.
- Interground BFC has lower strength values, particularly at late curing ages compared to BFC produced by separate grinding to the same Blaine fineness.
- The increasing amount of slag has a detrimental effect in the strength of BFC when fineness is kept constant.
- When BFC is produced by separate grinding, increase in slag fineness improves the strength while the clinker fineness is kept constant.

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