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Comparison of separate and co-grinding of the blended cements with the pozzolanic component

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Abstract

The main difference between separate and co-grinding of blended cement is the fact that co-grinding occurs interaction among the milled components. These interactions may speed up milling process, or on the contrary to slow it down. It depends on grindability of the components. Separate grinding and subsequent homogenization is more common. High speed disintegrator appears to be a promising technology for the homogenization of blended cements after separate milling process in traditional mills, which can be associated with final grinding. The aim of the work was to compare the effect of separate and co-grinding, and their combinations on the properties of the blended cement. At first the samples of pure glass, pure Portland cement and its mixture were pre-ground in the ball mill to the specific surface area of 400 m^2/kg according to Blaine. The material was subsequently ground either in a ball mill or disintegrator by the various combinations of separate or co-grinding. All the samples were subjected to granulometric and morphological analysis and the analysis of the technological properties. In the case of the combination of cement and glass the co-grinding appeared to be more advantageous than the separate grinding with homogenization. The high speed disintegrator has produced sharp-edged grains with narrower particle size distribution curve than traditional ball mill. Technological properties of the cement have been also influenced by high speed disintegrator. Compressive strength in early age was higher than in the case of ball mill type cement, however, the final strength were essentially the same.

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1. Introduction

Secondary raw materials represent an ever more frequent replacement for primary raw materials in the production of cement. In current practice the Portland clinker is replaced by hydraulically active compounds or agents with pozzolanic properties [1,2]. Glass is chemically and mineralogically very close to traditional pozzolans and various authors have described the behavior of finely ground glass in cement composites [3,4,5]. However, due to considerable ability to agglomerate, the recycled glass is not usually enough reactive and acts only physically-mechanically as a filler [4]. A common production process of the blended cements is separate grinding of the individual components and their subsequent homogenization. This procedure is very common especially when blast furnace slag is used. And it works in this case, because Portland cement clinker and blast furnace slag have very different grindability and it is therefore preferable to grind them separately, and subsequently to homogenize [6]. As noted above, fine glass powder exhibits significant ability of the aggregation, which greatly complicates homogenization with the Portland cement. Therefore, the traditional approach of separate grinding and subsequent homogenization seems to be less suitable in the case of glass-cement system. An interesting option to prevent the formation of agglomerates with pure glass is co-grinding of the glass and clinker because the grindability of both components are very similar [7]. But the question is if this income brings any benefits to final cement properties than separate grinding and subsequent homogenization.

2. Materials and methods

The laboratory-prepared blended cement was used to compare the effects of a separate and co-grinding of the final cements properties. The samples of the blended cement should always have the same composition. The ratio between the components were 80 wt. % of Portland cement and 20 wt. % of the pozzolanic ingredient. The recycled glass was used as a pozzolanic ingredient. Portland cement was prepared also in the laboratory from the Portland cement clinker at a dose of 95 wt. % and from gypsum PREGIPS at a dose of 5 wt. %. Five samples of the blended cement by different technology of separate and co-grinding were prepared in total.

The jaw crusher Retsch with the size of the exit slit 3 mm and two types of mills were used to samples preparation. The laboratory ball mill OM BRIO 20 was used as the primary mill. Mill rotation frequency was 45 rpm. The secondary mill was the disintegrator DESI 11, which is a high speed pin mill with two counter-rotating rotors. The total installed output of the mill is 4.1 kW. Rotor rotation frequency is up to 12000 rpm and maximum speed of impact is 240 $m \cdot s^{-1}$. The material is fed by a continuous feeder and enters the grinding chamber through the middle of the left rotor. The construction of mill allows for choice of working tools. For the evaluation of milling process, CR type rotors were used. The rotors were designed and manufactured by the company FF servis s.r.o. The left rotor has two rows of pins and right rotor has three rows. The pins have a square cross section. The raw material for the blended cement production was ground in a jaw crusher at first and subsequently was milled in a ball mill to achieve specific surface area of 400 $m^2 \cdot kg^{-1}$ according to Blain. Cement components were ground together and separately as well. Three of the five samples were then ground by one pass through the mill DESI 11. Remaining two samples were then ground in a ball mill to achieve the same specific surface area as samples obtained from the disintegrator DESI 11. The Samples which were ground separately had to be then homogenized in a laboratory homogenizer. The process of grinding and marking the samples is summarized in Table 1.

Table 1. The samples marking and the description of the grinding process.

Sample	Pre grinding 400 ($m^2 \cdot kg^{-1}$) Ball mill		Final grinding DESI 11		Final grinding Ball mill		Homogenization
	Separate	Co-grind.	Separate	Co-grind.	Separate	Co-grind.	
A	X	-	-	X	-	-	-
B	-	X	-	X	-	-	-
C	-	X	-	-	-	X	-
D	X	-	X	-	-	-	X
E	X	-	-	-	X	-	X

All samples were subjected to specific surface area according to Blaine and particle size distribution measurement by laser granulometry. For the measurement of Blaine specific surface area, a PC-Blaine-Star automatic device was used with a measurement cell capacity of 7.95 cubic centimeters. The determination was performed three times to eliminate errors, and the resultant value was the average of these determinations. Laser granulometry was performed on a Malvern Mastersizer 2000 in dry conditions. The effect of the milling technique on the morphology of the grains was observed and assessed by electron microscopy (SEM). A Tescan MIRA 3 XMU SEM with a secondary electron detector was used. For each sample the water/cement ratio, initial and final setting time according to CSN EN 196-3 were determined. All the mechanical tests were elaborated on the samples prepared from cement pastes. The dimensions of the cement bars were 20 × 20 × 100 mm. Water cement ratio of each mixture has corresponded to the previous results. The increases of the compressive strength at time intervals of 1, 3, 7, 28 and 56 days were observed on these samples.

3. Results

3.1. The specific surface area and particle size distribution

The specific surface area according to Blaine of all samples is shown in Table. 2.

Table 2. Specific surface area of all the samples.

Sample	Pre grinded ($m^2 \cdot kg^{-1}$)	Final specific surface area ($m^2 \cdot kg^{-1}$)
A	402	493
B	400	487
C	406	486
D	403	490
E	408	483

All samples achieved virtually the same specific surface area. The fineness of the material so did not affect the resulting technological properties. For the comparison of the effects of the selected mill on the particle distribution, all the results were summarized in figure 1a. For the results of the very fine particles size distribution, see Fig. 1b.

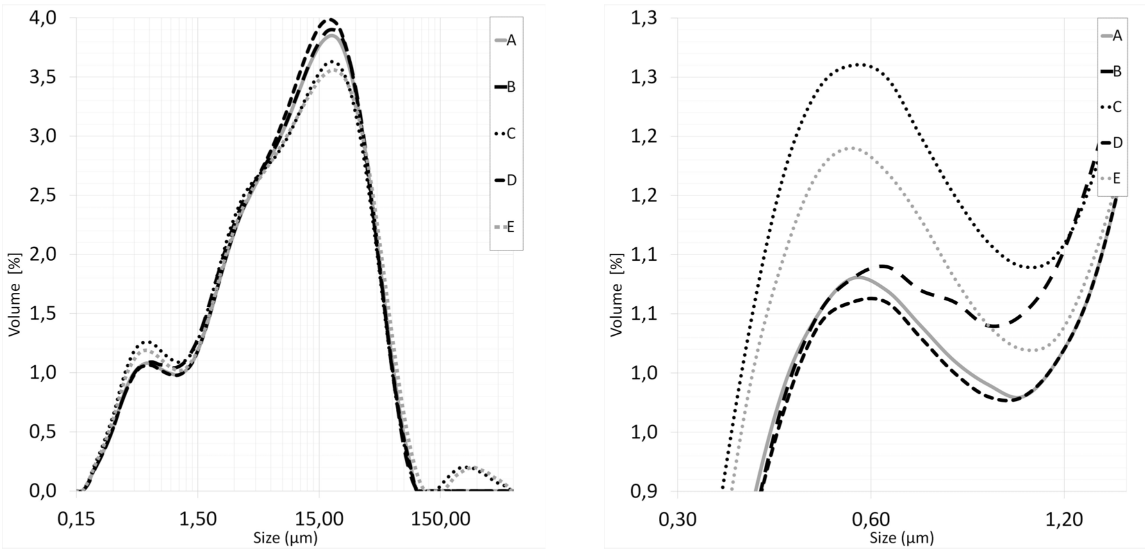

Fig. 1. (a) Particle size distribution of all the samples; (b) particle size distribution of very fine particle size (0.3–1.5 µm).

The results indicate a narrower particle size distribution curve and a slightly smaller percentage of very fine particles below 1 micron in samples which were finally milled in the high speed disintegrator DESI (A, B, D). In the case of a laboratory ball mill, a small content of very coarse particles still remained (C, E).

When comparing the various milling procedures, it can be observed in the case of the mill DESI (A, B, D) that in the area of particles size over 10 microns was higher content of the particles observed in case of separate grinding procedure (D). Conversely, in the area of very fine particles below 1.5 microns, their content increased in case of the samples A and B, i.e. co-grinded samples. Above 1.5 microns was the curve of particle size distribution of samples A and B essentially identical. The similar trend was observed in case of the samples which were ground only in traditional ball mill (C, E). This trend was even more pronounced for particles above 30 microns. The high proportion of particles in this size was observed for the separately ground sample (E).

3.2. Morphological changes

For the assessment of the effects of various methods of preparation on the morphology, the co-ground and separate ground samples were compared to each other. These samples were prepared in the ball mill and in the high speed mill as well. The effects of the technology on the morphology is demonstrated in the Figures 2 and 3.

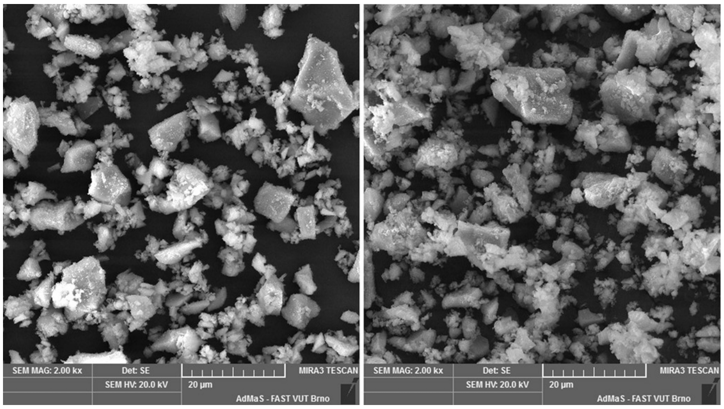

Fig. 2. (a) SEM co-grinding DESI; (b) SEM co-grinding ball mill.

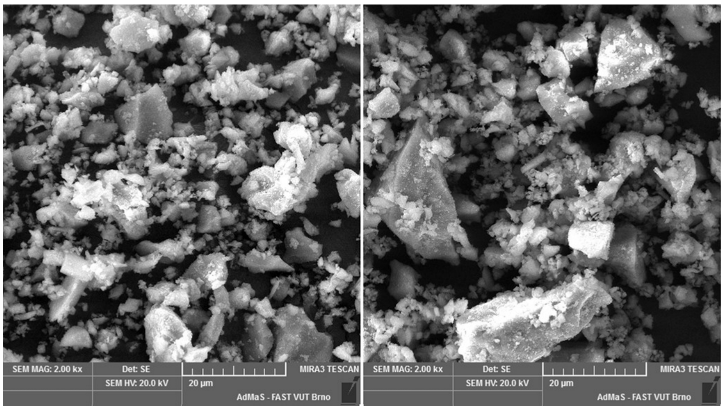

Fig. 3. (a) SEM separate grinding DESI; (b) SEM separate grinding ball mill.

In both cases the ball mill produced rounded grains. In contrast, the grains from the high-speed mill are sharp and angular. The agglomeration occurred in all cases. It was very difficult to distinguish clinker and glass grains in the agglomerates without use of EDX.

3.3. Technological properties

The water cement ratio, initial and final setting time were determined according to ČSN EN 196-3+A1. Results are in the Table 3.

Table 3. Water cement ratio and the setting time.

	A	B	C	D	E
W/C ratio	0.280	0.280	0.275	0.275	0.267
Initial setting time (*min*)	160	180	160	150	135
Final setting (*min*)	200	230	215	180	195

Separately ground samples (C, E) always showed lower water cement ratio than the co-ground samples milled by the same technology. Initial and final setting time for the co-ground samples (A, B, C) were longer than for the samples milled separately (D, E).

The higher strengths in early age were achieved with the samples which were finally milled in the high speed mill. Co-ground cement, regardless of the selected technology, also showed higher increase in strength in early age than the separately ground samples. However the strength after fifty-six days were similar. For the results of the compressive strength, see Table 4.

Table 4. Compressive strength.

	Compressive strength (*MPa*)				
	1 day	3 days	7 days	28 days	56 days
A	54.2	55.7	60.4	60.6	64.8
B	48.2	49.6	59.4	63.3	68.1
C	45	48.5	55.1	60.8	68.6
D	50.5	50.8	51.4	55.9	57.6
E	37.8	48	54.1	61.6	62.9

4. Discussion

Based on the results it can be stated that the particle size distribution was influenced by the type of mill and by the method of preparation as well. When comparing the influence of the type of mill it can be said that the samples ground in a high speed mill contained higher proportion of particles of 5–30 micron, than the samples from the ball mill. However, for the very fine particles below 1 micron the situation was reversed. A small amount of very coarse particles was also observable in the samples from the ball mill (C, E), which were not refined during grinding process. In the case of high-speed mill, smaller amounts of very fine particles can be explained by the intensive surface charging particles and their agglomeration, which occurs due to high milling intensity. The agglomeration occurs in the ball mill too, but because of the slower milling process there is a longer time to partially disintegrate agglomerates again. Coarse particles cannot pass through the disintegrators milling chamber and therefore they do not occur in the samples A, B, and D.

When comparing the results obtained from co-ground and separate ground samples it can be said that in the case of the cement - glass combination better results were achieved by co-grinding, especially for the very fine particles. The better results were achieved by co-grinding for this materials combination regardless of the selected type of mill. Results are valid for the cement and recycled glass combination, of course. It seems that cement and glass particles act synergistically in the co-grinding. This synergistic effect can be explained by the influence of agglomeration. The pure glass particles readily form agglomerates due to grinding process which are very difficult to disintegrate. The presence of the cement particles in the agglomerates leads to partial collapse of the agglomerates and to the increase of the finest particles amount. The effect on the morphology of the grains can be explained by different milling principle of these mills. The high-speed mill grinds particularly by the impact of particles on the working tools and by the mutual collisions among the particles. The milled material stays in the mill in the order of seconds and the energy content per volume in the mill chamber is extremely high. In contrast, ball mill grinds mostly by friction. Particles are disintegrating slowly and gradually. Therefore, the shape of the grains is more angular in the case of samples milled in a high speed mill. Samples ground only in a ball mill had rounded grains. The grains shape then has an impact on water cement ratio. The higher water cement ratio was achieved by the samples which were prepared in the high speed disintegrator. The higher water cement ratio was always achieved for co-ground samples (B, D) compared to the separately ground cements (C, E). This may be because of the better homogeneity of the material. The glass particles homogeneously present in the agglomerate facilitates the penetration of water into the agglomerates and wetting of the cement particles that would not be in contact with water otherwise. Better homogeneity and higher water cement ratio can explain a longer initial and final setting time of the co-ground samples. The advantages of the co-grinding for the cement-glass system is then reflected in the results of the compressive strength. Co-ground samples achieved better parameters than the samples which were ground separately and then homogenized. Compressive strength in early age was higher for disintegrator type cement than in the case of ball mill type cement, however, the final strength was essentially similar. This phenomenon could be caused by mechanical activation of the binder. [8,9,10]

5. Conclusion

The question of the blended cement preparation by the separate or co-grinding is very important. Unfortunately, the answer cannot be generalized for all combinations of materials. It is always necessary to describe a specific combination of the cement ingredients. In this case it was a combination of Portland clinker and recycled glass. The recycled glass was chosen as potentially useful pozzolanic component. The comparison of the influence of separate or co-grinding this two components was performed on two types of mill. In both cases, the co-grinding seemed to be preferable choice for combination of Portland clinker and recycled glass, because of the homogeneity of the resulting material. In this case, co-grinding has prevented forming of the pure glass agglomerates. The glass should then operate in the cement paste as the active ingredient, and not only as a filer. This was reflected on the mechanical properties of the hardened material.

Acknowledgements

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References

[1] C. Meyer, Y. Xi, Use of recycled glass and fly ash for precast concrete, J. Mater. Civil Eng. 11 (1999) 89–90.
[2] D. Gazdič, Slag-sulphate binder preparation, Adv. Mat. Res. 818 (2013) 68–71.
[3] T. Melichar, J. Přikryl, P. Matulová, Substituce pojiva v cementových kompozitech jemně mletou recyklovanou sklovinou s ohledem na životní prostředí, Beton TKS 9 (2009) 50–55.
[4] A. Khmiri, B. Samet, M. Chaabouni, A cross mixture design to optimise the formulation of a ground waste glass blended cement, Constr. Build. Mater. 28 (2012) 680–686.
[5] A.M. Rashad, Recycled waste glass as fine aggregate replacement in cementitious materials based on Portland cement, Constr. Build. Mater. 72 (2014) 27–52.
[6] M. Öner, A study of intergrinding and separate grinding of blast furnace slag cement, Cement Concrete Res. 30 (2000) 473–480.

[7] K. Dvořák, D. Dolák, D. Všianský, D. Dobrovolný, Evaluation of the grindability of recycled glass in the production of blended cements, Materiali in technologije 5 (2016).
[8] P. Baláž, Mechanochemistry in Nanoscience and Minerals Engineering, Springer-Verlag Berlin Heidelberg, 2008.
[9] I.A. Massalimov, Materials processing in a disintegrator and their use for the improvement of chemical technologies, the Abstract of doctoral thesis, Ufa, 2005.
[10] K. Tkáčková, Mechanical activation of minerals, Miner. Eng. 11 (1991) 185.
[11] CSN EN 196-3+A1 2009 Methods of testing cement – Part 3: determination of setting time and soundness.