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Steel-slag as filler material in concrete

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In cement-based products, such as concrete and mortars, a balance between the particle sizes of the components must be kept in order to obtain the required material properties, such as workability and strength. Sometimes the aggregates lack the necessary amount of fines, hence fine particles, i.e., filler materials, have to be added. Large quantities of filler material are needed in the production of modern concrete types; one principle of self-compacting concrete (SCC) is, for instance, the use of large amounts of fillers to reduce the friction between the coarser aggregates. Use of by-products such as slag, dust or sludge from the metallurgical industries as filler materials in concrete may help to conserve natural resources and at the same time be an economically positive option. However, to be able to successfully use the available materials they must be suitable for the planned purpose, i.e., they must be compatible with cement. In this paper three different experimental studies of steel-slags will be discussed. The aim of the studies has been to investigate if it is possible to improve the steel-slags properties, by selective screening, fine wet grinding or remelting, so that the steel-slags can be used as mineral-addition/filler material in concrete.

Keywords: concrete, filler, steel-slag, modification

Introduction

In cement-based products, such as concrete and mortars, a balance between the particle sizes of the components must be kept in order to obtain the required material properties, such as workability and strength. Sometimes aggregates lack the necessary amount of fines, or the aggregates may even have been washed at the quarry or gravel pit—in order to remove fine material that may cause problems with dusting, or humus that will cause durability problems in concrete. In order to optimize the quality of the concrete, usually fine particles have to be added. Large quantities of filler material are needed in the production of modern concrete types. One example is self-compacting concrete (SCC) where large amounts of fine material is used to reduce the friction between the larger aggregates.

Use of by-products such as slag, dust or sludge from the metallurgical industries as filler materials in concrete may help to conserve natural resources and at the same time be an economically positive option. However, to be able to successfully use the available materials they must be suitable for the planned purpose—to be mixed with cement. Earlier investigations[1] of slag, dusts and sludges have shown that some materials give rise to durability problems due to their content of reactive (free) calcium oxide (f-CaO) or magnesium oxide (MgO). When f-CaO comes in contact with water it starts to hydrate and a large amount of heat is released. The heat may cause the cement paste to crack and thus reduce the strength of the concrete. MgO decreases the concrete's long-term durability, since the formation of brucite causes an expansion, and subsequent cracks in the cement paste. Other materials have shown a low reactivity with cement, resulting in decreased strength and durability.

The experiments described in this paper have been conducted in order to see if it is possible to solve the durability and activity problems of three steel-slags so that they can be used as filler in concrete. Three different studies were conducted:

1. The effect of the fines of disintegrating AOD-slag on concrete strength was examined[2].
2. The effect of wet ground EAF- and AOD-slags on cement pastes' heat development, concrete strength and shrinkage/expansion were examined.
3. Remelting and granulation of AOD-, EAF- and ladle-slags and their effects on cement hydration were examined (part of a joint MiMeR*-project).

The chemical content of the steel slags and cement used in the three projects is shown in Table I.

Fine fractions of disintegrating AOD-slag as filler in concrete

Earlier investigations[1] show that ground slags can be used as filler in concrete. It would be a more economically attractive prospect to use them as fillers if no further treatment was needed. Thus, the idea is to use fine particles of disintegrating slag from the stainless steelmaking process as filler in concrete.

Materials

Fresh AOD-slag of the disintegrating type was collected; it had not been subject to any form of quenching or grinding. The slag was sieved and the -45 μm fraction gathered for this study. Quartz with the maximum particle size of 45 μm was used as reference material in the same proportions as

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Table I
Major chemical content of the EAF- AOD- and ladle-slag, and cement (wt-%)

	EAF	AOD	LD	Cement
SiO_2	34.0	27.0	14.3	22.3
CaO	47.0	54.0	42.5	64.8
MgO	6.0	6.0	12.7	0.8
Al_2O_3	2.3	4.9	22.8	3.4
FeO	2.0	2.6	1.5	4.3

the slag. The cement used in these experiments is an ordinary Portland Cement (CEM I 42.5 BV/SR/LA). The particle size distribution of the sand (granite and quartz) and cement used in the experiments are shown in Figure 1.

Experiments

Mortars of cement, slag or quartz and water were combined in a laboratory mixer and the specifications for mixing, stipulated in EN 196-1, were followed. The water/solid ratio (w/s) was kept at 0.50. Cement was replaced by 10, 20 and 30 weight percent of slag and quartz, respectively. The receipt is shown in Table II.

To test the compressive and flexural strength, mortar prisms of 4x4x16 cm^3, with the w/c-ratio 0.50, were cast in moulds according to the procedure in SS 13 11 12. Demoulding took place after 48 hours and the prisms were cured and stored in 100 percent relative humidity (RH). The compressive strength was tested with uniaxial compression at 2, 7 and 29 days. The press used for the compressive strength tests is in accordance with SS 13 11 10. The flexural strength was tested after 29 days. The rate of mid span displacement was kept at 0.01 mm/second.

Results

The compressive strength for the samples that contain AOD-slag is slightly higher than the corresponding references at all the three replacement levels. This is an indication that the fine particles of the AOD-slag may have a positive influence on concrete strength.

The flexural strength for the samples containing 10 and 20 percent slag are higher than the quartz containing references, see Table II. However, at 30 percent replacement level, quartz has the higher strength. These flexural strength values are, however, only indications since only two samples of each mixture have been tested. The standard deviation is low, less than ±2.5 percent for all samples. The diagram in Figure 2 shows the compressive strength as a function of the filler amount. The results from the investigation by Magnelöv and Drugge[2] have been supplemented by additional strength tests.

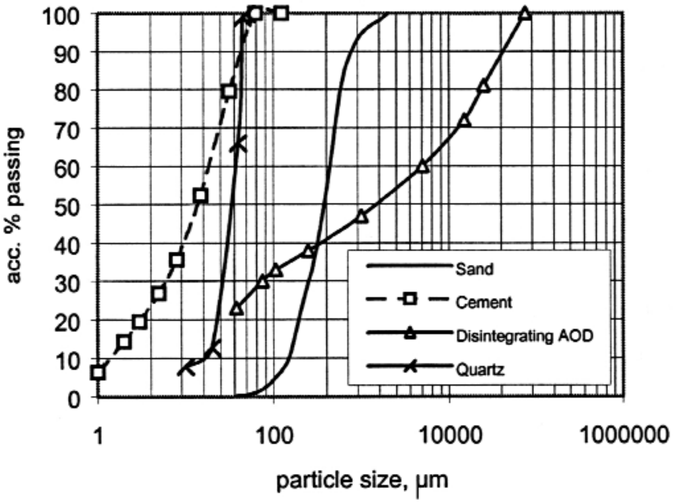

Figure 1. The particle size distribution of the sand and cement used in the experiments (the particle size distribution for the unsieved AOD-slag is included)

Discussion

The results of this investigation imply that slag addition compared to quartz addition gives a stronger concrete. This may be due to the filler effect, i.e., fine particles fill the voids between the larger cement grains and aggregates and make the cement paste more homogenous and dense, and thus increase the strength. Another explanation may be due to the fact that slag contains calcium silicates that may take part in the cement reactions and thus increase the strength by increasing the amount of binder/cement gel.

Long-term tests must be made in order to clarify the slags' influence on concrete strength. However, it is clearly possible to use the fine AOD-slag particles as filler material in concrete as far as strength is concerned. The slags' content of chromium and other metal ions will probably not affect its usability as filler since the levels are low.

The effect of wet grinding on steel slags, and their subsequent use as filler in concrete

Many steel-slags contain free calcium oxide and magnesium oxide that may cause durability problems. The hydration of calcium oxide is an exothermal process that will cause cracking of the cement paste if it has hardened. Magnesium oxide hydrates slowly into brucite, which has a larger volume and causes the cement paste to crack, thus reducing the strength. The aim of these experiments is to investigate if the durability problem can be avoided if the CaO and MgO, i.e., the slags, are very fine particulate and evenly distributed in the cement paste. Cement paste can withstand rather high internal forces if they are evenly distributed.

Table II
Receipt (kg), and flexural strength of cement pastes after 29 days

Replacement level	Filler	Cement	Sand	Water	Quartz	Slag	Flexural strength MPa
10%	Quartz	0.405	1.35	0.225	0.045	-	2.40
10%	Slag	0.405	1.35	0.225	-	0.045	2.99
20%	Quartz	0.360	1.35	0.225	0.090	-	2.44
20%	Slag	0.360	1.35	0.225	-	0.090	2.82
30%	Quartz	0.315	1.35	0.225	0.135	-	2.21
30%	Slag	0.315	1.35	0.225	-	0.135	2.09

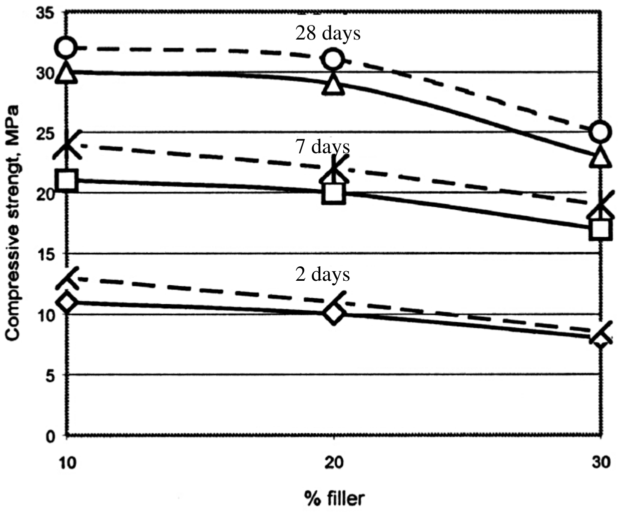

Figure 2. Compressive strength as a function of the amount of filler. The solid lines represent quartz, and the broken lines the AOD-slag

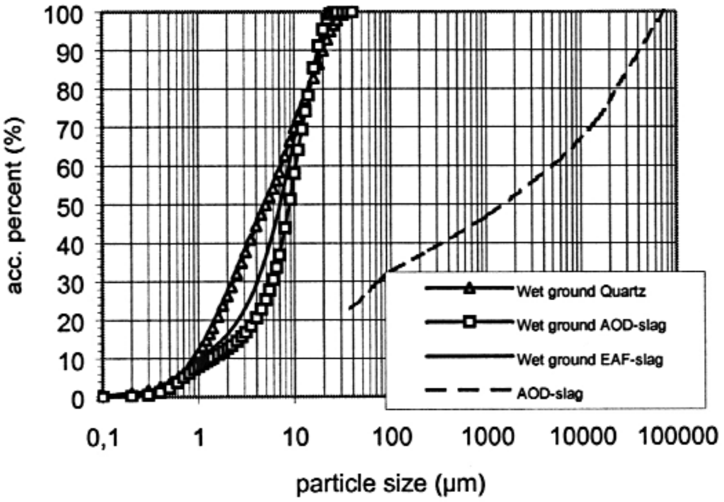

Figure 3. Particle size distributions of the ground slags and quartz-sand. The original particle size distribution of the AOD-slag is included, but not the EAF-slag since it was lumpy and therefore not sieved

Materials

In order to evaluate the possibility of modifying slags and thus their effect on concrete properties, wet grinding of two steel-slags was performed. Electric arc furnace slag (EAF) and stainless steelmaking slag (AOD) were investigated. Quartz sand (sand from the Baltic sea) was used as reference material. A 7.5 kW Sala Agitated Mill (SAM) was used in the experiments.

Experimental

Each material was fed into the mill together with water. They were ground for 20 minutes and then the slurry was poured into plastic buckets, and left to allow hydration of reactive constituents to take place. One hour later, the slurries were homogenized and their water/solid-content analysed. The ground materials' particle size distributions are shown in Figure 4.

Isothermal calorimetric measurements

Isothermal calorimetric measurements were performed in order to see how the materials affect the cement hydration when mixed into cement paste. Twenty weight-percent of the cement was replaced by the various slags (dry weight). The instrument has an accuracy of ±5 percent. Four minutes elapsed between the addition of the water to the cement/filler mixture and the placement of the paste in the calorimeter. The mixing procedure is in accordance with the SS-ENV 206. The water/solid-ratio was kept at 0.40 for all samples. Mortar and pestle were used to (dry) grind samples of the original slags so that their heat development curves could be determined as well as the wet ground slags. The material was sieved and the -45 µm fraction used. A sample each of the wet ground materials was dried, deagglomerated and thereafter mixed into cement paste and analysed with the calorimeter. The water content in the slurries was analysed and the amount of mixing water adjusted to obtain the correct water/solid ratio.

According to Murat and Sadok[3], hydration kinetics, i.e., the nucleation rate V_n, and the growth rate V_g, can be approximated by the following formulas:

$$V_n = \frac{1}{t_0} \qquad [1]$$

$$V_g = \frac{1}{(t_{\max} - t_0)} \qquad [2]$$

where: t_0 is the time at which the transition between the dormant period and the accelerating period occurs, and t_{max} is the time when the maximum heat, dQ/dt, is obtained (the second peak).

The hydration is accelerated when the peak occurs earlier and similarly retarded when it occurs later.

Compressive strength

The concrete was based on a receipt containing 433 kg cement and 1672 kg aggregates per cubic metre. Mixtures of concrete, where 20 and 40 percent cement were replaced with AOD-slag, EAF-slag and quartz, respectively, were mixed in accordance to SS EN-206-1; the references without filler were made in the same way. Concrete cubes, 0.10 x 0.10 x 0.10 m^3 were cast to determine the filler materials' influence on the concrete strength. To facilitate calculations of efficiency factors, concrete with three different water/cement-ratios (w/c-ratio) was mixed. The samples with w/c-ratios of 0.61 and 0.81 correspond to the clinker content in the mixtures with 20 and 40 percent slags.

Shrinkage and expansion

In order to test the effect of the ground slags on shrinkage and expansion, prisms of 2.5 x 2.5 x 25 cm^3 with the w/s-ratio of 0.40 were cast in steel moulds. Steel knobs were attached to the short sides to enable measurements of length alterations. Demoulding took place 24 hours after casting, and the prisms were cured in 100 percent RH. The prisms for shrinkage and expansion measurements were stored in constant temperature, 20ºC, and in 50 and 100 percent RH respectively. The lengths were measured immediately after demoulding and then again after 91 days. A standard measuring-rod was used as a reference.

Efficiency factor

In order to be able to measure the relative effect of filler material compared to cement, an efficiency-factor (k-factor) can be calculated[4, 5]. This factor represents the amount of

Table III
Nucleation and growth rates, 20% replacement

	t_0 (min)	t_{max} (min)	V_n (min^{-1})	Vg (min^{-1})
EAF dry ground	140	760	7.14 E-3	1.61 E-3
EAF wet ground	150	640	6.67 E-3	2.04 E-3
EAF dried, wet ground	150	720	6.67 E-3	1.75 E-3
AOD dry ground	170	710	5.88 E-3	1.85 E-3
AOD wet ground	120	500	8.33 E-3	2.63 E-3
AOD dried, wet ground	180	730	5.56 E-3	1.82 E-3
Quartz	140	650	7.14 E-3	1.96 E-3
Quartz wet ground	100	520	10.0 E-3	2.38 E-3
Quartz dried, wet ground	170	760	5.88 E-3	1.69 E-3
Cement (pure)	140	650	7.14 E-3	1.96 E-3

Table IV
Compressive strength (MPa) and efficiency factor

	w/s	w/c	1 day	7 days	28 days	7 days k-factor	28 days k-factor
Cement	0.48	0.48	12.3	37.2	52.9	-	-
Cement	0.61	0.61	9.4	36.0	43.4	-	-
Cement	0.81	0.81	6.9	23.3	28.4	-	-
Quartz 20%	0.48	0.61	9.1	28.3	48.0	0.39	0.73
Quartz 40%	0.48	0.81	5.1	16.8	31.4	0.30	0.40
EAF 20%	0.48	0.61	7.9	28.4	46.3	0.40	0.63
EAF 40%	0.48	0.81	5.7	20.5	35.8	0.43	0.53
AOD 20%	0.48	0.61	6.3	30.5	44.7	0.54	0.55
AOD 40%	0.48	0.81	4.4	21.2	34.1	0.45	0.48

filler that can be considered equal to the cement, in regard to the compressive strength.

$$S_c = K\left(\frac{1}{W/C} - a\right) \qquad [3]$$

Where S_c is the compressive strength, W/C the water/cement-ratio in the initial mixture, K is a parameter depending on the cement type, and a is a parameter depending on time and curing.

For cement containing filler material, the following formula should be used, where P is the filler content in the concrete and k the efficiency factor.

$$S_c = K\left(\frac{1}{W/(C+kP)} - a\right) \qquad [4]$$

The lower the *k*-factor, the lower the level of activity the material has. The *k*-factor for cement is 1.

Results and discussion

Material

The particle size distributions of the wet ground materials can be seen in Figure 3.

Calorimetry

The cement pastes' hydration kinetics was investigated by isothermal calorimetry and the result evaluated in accordance with the formulas 1 and 2 (Table III). The nucleation rate and growth rate are increased for the wet ground materials in comparison to both the dried and original materials. The growth rate, V_g, is decreased for the dried wet ground materials, compared to the dry ground ones. The same relationship is found for the nucleation rate, V_n. These results suggest that the wet fine grinding activates the materials, or the particle surfaces.

Compressive strength

The concretes with the lower water/solid-rate (w/s-ratio 0.48) have overall a higher strength than the ones with higher w/s-ratio (0.81), regardless whether filler material has been added or not (Table IV). Comparisons between the samples with a w/s-ratio of 0.48 show that the reference, without filler addition, has the highest strength, and the sample containing 20 percent quartz the second highest. However, the samples with 40 percent addition of EAF or AOD slag have a higher strength than both the reference and the sample with 40 percent quartz after 28 days. The samples with filler addition obtain a lower short-term strength (1 to 7 days), but the long-term strength is positively affected, especially the samples containing the higher amount of filler.

Efficiency factor

In accordance with Papadakis' *et al.*[4, 5] findings, an efficiency factor (*k*-factor) has been calculated. Concretes without additions but varying water/cement-ratios were made and the constants, *K* and *a*, calculated (see Table IV). The *k*-factor for the 28 days samples are higher than the 7 days values. The *k*-factor for quartz is 0.56, for EAF and AOD 0.58 and 0.51, respectively. The *k*-factor for cement is 1.0. This factor will give only an indication of the added materials' effect on concrete strength, since it does not distinguish between filler effect and/or chemical reactions.

Shrinkage- and expansion measurements

The shrinkage and expansion measurements (Table V) show that the samples containing fine ground slags have approximately the same shrinkage and expansion as the

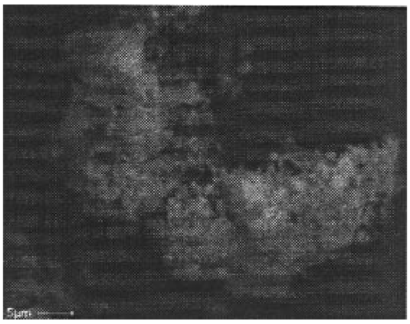

Figure 4. Micrograph of dried wet ground quartz

pure cement paste. The prisms containing untreated slag show a higher shrinkage and expansion. The AOD-slag has higher expansion than both the reference and the EAF-slag.

Scanning electron microscopy analyses

SEM analysis of the dried wet ground quartz show that the particle surfaces are covered with a fluffy coating consisting of very small particles; there are no free quartz surfaces. It seems that the particles are fused together in agglomerates by a gel-like mass (Figure 4). Chemical analysis shows that the main constituent of the ‘gel’ is SiO_2 with traces of iron. The iron is probably from the cypelbs used as grinding media in the mill.

Discussion and conclusions

Wet fine grinding of EAF and AOD slags seems to be a feasible way of making it possible to use the slags as filler material in cement-based products. The wet grinding appears to increase the slags’ activity; the heat of hydration is increased and accelerated. Hydration and fine grinding of the f-CaO and MgO reduce the risk of expansion and subsequent strength loss: either the oxides have time to hydrate or the smaller particle size makes it possible for the cement paste to resist the forces from expansion. The compressive strength and the calculated efficiency factor show that the fine particulate slags have a positive influence on the strength development. It is not possible to pinpoint the reason for this since the *k*-factor gives only an indication of the added materials’ effect on concrete strength—it does not distinguish between filler effect and/or chemical reactions.

The dried fine ground fillers have a retarding effect on the cement hydration. This may be due to the fine particles and the precipitate of dissolved material that coat the particles after drying. The cement and dry fine ground material are mixed thoroughly before the water is added and the calorimetric measurements take place. Thus, it is possible that the particle flocks are deagglomerated and very fine particles attach themselves to the larger cement grains and retard the cement hydration by physical means. The stored EAF-slag slurry started to gel one day after grinding. This is probably due to hydroxide precipitation (f-CaO reacts with water). According to Lind[6], this is a known phenomenon: when slags that not are completely dry are ground, the pH will increase. This may be a part of the explanation as to why the slags have an efficiency factor above 0. One known way of activating slag-cements is to add NaOH, which will start the cement reactions.

Table V
Measurements of shrinkage and expansion after 91 days
(* Dry ground)

	Shrinkage %	**Expansion %**
Cement	3.08	0.30
20% EAF	3.21	0.30
40% EAF	3.28	0.32
40% EAF *	3.33	0.41
20% AOD	3.19	0.29
40% AOD	3.24	0.27
40% AOD *	3.42	0.48

Lidström[7] found that quartz surfaces were activated due to changes in the lattice structure by grinding, i.e., mechanical activation. The surface of the crystalline material is disrupted and obtains, to varying degrees, an amorphous structure. Amorphous SiO_2 is more soluble and has a higher surface activity than the crystalline material. This is probably the reason that the ground materials in this investigation obtain a higher reactivity and that it is decreased after drying.

Modification by remelting and granulation of steel slags

Steelmaking slag is not a normal additive in concrete, in contrast to blast furnace slag, which is commonly used. However, one way of increasing the reactivity of steel slags is to increase their glassy content by remelting and rapidly cooling, i.e., granulation of the slag. The aim of this study is to improve reactivity and compatibility with cement. Another benefit of the granulation process may be a limited leaching by the glassy material. Isothermal calorimetry was used to determine the slags’ effect on cement pastes’ heat development.

Material

The chemical content of the three used steel slags, AOD, EAF and ladle-slag, can be found in Table I.

Experimental

Melting and granulation of slags

A slag granulation system has been assembled, at the University of Technology in Luleå, close to the laboratory induction furnace used for the slag melting[8], Figure 5.

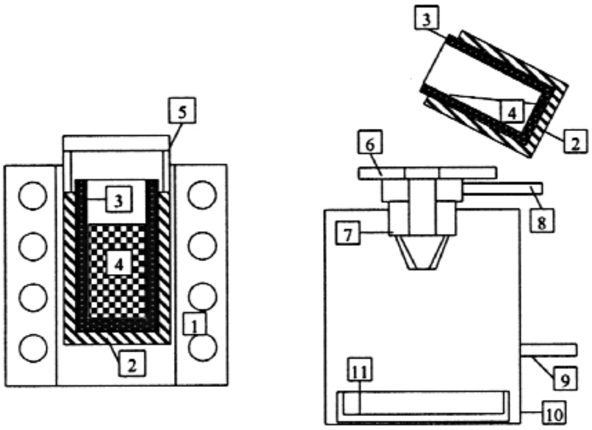

Figure 5. The slag melting and granulation equipment[8]: 1. Induction coil, 2. Refractory crucible, 3. Graphite crucible, 4. Slag, 5. Refractory cover, 6. Steel plate, 7. Granulation head, 8. Water inlet, 9. Water outlet, 10. Water tank, 11. Granualated slag container

The AOD and ladle-slag with low contents of iron oxides were melted in a system that contains an induction coil and a graphite crucible, inserted in a refractory crucible. The thermal energy generated inside the graphite by induction power was transferred directly to the slag, which made it possible to heat and melt it quickly. After melting the charged slag, solid slag (*ca.* 0.5 kg) was charged on top of the molten slag. In order to facilitate the slag melting an iron rod was used to mix the solid and liquid slag. This procedure was repeated until the amount of molten slag reached a total weight of 2–2.5 kg. The EAF slag was melted in an MgO crucible.

The refractory crucible and the crucible holder were manually lifted and placed above the slag granulation system. The water supply was started, and the crucible tilted to pour the molten slag into the granulation head. The water jets hit the slag stream and the granulation took place. The granules fell down into a slag container at the bottom of the water tank. The water outlet regulated the water level in the tank. The duration for the slag tapping and granulation was about 30 seconds.

X-ray diffraction analysis

The states of the original and the granulated slags, i.e. if they were crystalline or amorphous, were examined with X-ray diffraction analysis. The slags were ground, and then sieved with a 60 μm screen before analysis. A Philips PW1710 X-ray Diffractometer was used for the diffraction studies, a PW1729 X-ray Generator produced the copper radiation, Cu Kα. Scans were run with the X-ray source set at 40 kV and 30 mA. The measurement were made for the 2Θ range 5º–70º with a step size of 0.020º and a measuring time per step of 1.00 s.

Calorimetric measurements

The heat development curves were determined with an isothermal calorimeter. Cement pastes, where 20 weight-percent of the cement was replaced with the respective slags, were analysed. Pure cement was used as reference material.

Results

X-Ray diffraction analysis

The XRD analysis shows that the original ladle-slag is crystalline and that the granulated ladle-slag is almost wholly amorphous, and it contains a small amount of crystalline magnesium oxide. The granulated EAF-slag contains the same compounds as the original slag, the peaks are, however, slightly lower. The granulated AOD-slag has fewer peaks than the original slag, and its structure has clearly changed towards a more glassy state. The crystalline compounds consist mainly of calcium oxides and calcium silicates and a small amount of magnesium oxide.

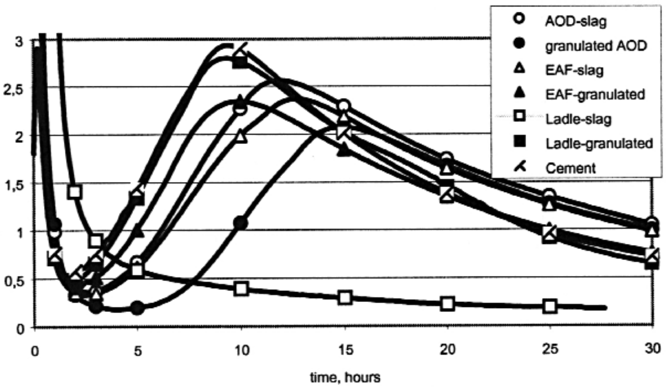

Figure 6. Heat development curves for cement pastes containing 20 percent slag (the diagram shows untreated and granulated slags). Cement without additions was used as reference material

Table VI
Nucleation and growth rate, 20% slag

	t_0 (min)	t_{max} (min)	V_n (min^{-1})	V_g (min^{-1})
EAF-slag	140	760	7.14 E-3	1.61 E-3
EAF-granul.	110	600	9.09 E-3	2.04 E-3
AOD - slag	170	710	5.88 E-3	1.85 E-3
AOD-granul.	260	930	3.85 E-3	1.49 E-3
Ladle-slag	-	-	-	-
Ladle-granul.	130	560	7.69 E-3	2.32 E-3
Cement	130	560	7.69 E-3	2.32 E-3

Calorimetric measurements

The calorimetric measurements of the ladle-slag show that the untreated slag retards the heat development severely. However, the granulated slag has clearly become reactive: the nucleation and growth rates are significantly higher. In fact they are almost the same as the pure cements. The maximum heat developed is slightly lower though. The heat development for granulated AOD-slag is lower and retarded in comparison to both the reference and the original slag. The granulated EAF-slag is slightly accelerated compared to the untreated slag; the amount of developed heat has not changed (Figure 6 and Table VI).

Discussion and conclusions

The activity and compatibility of the ladle-slag is positively affected by remelting and granulation. The EAF-slag obtains a higher activity—it is slightly accelerated compared to the original slag, while AOD-slag becomes retarding and has lower activity. This may be due to the glassy content of the slags[9]. The glass content of the original ladle-slag was 18 percent, and after granulation 98 percent. The glassy content of the AOD-slag was not increased after the granulation process although the materials changed in appearance. The granulated EAF- slag contains 17 percent glass compared to the original slags' 2 percent. The residual stainless steel in the original slags appears as droplets in the granulated. Figure 7 shows metal droplets in the granulated EAF-slag; the size of the droplets is approximately 1.5 mm in diameter.

Preliminary results from leaching tests[9] indicate that the slags have obtained lower leachability. Conductivity measurements of the granulated slags indicate a decrease compared to the untreated slags.

Concluding remarks

- The compressive strength for mortar containing fines (-45 μm fraction) of disintegrating AOD-slag obtains a slightly increased strength compared with the reference samples containing quartz. This effect may be due to the filler effect or because a positive chemical effect takes place.
- Wet grinding of AOD and EAF-slags appears to be a feasible way of increasing the slags' activity. The durability problem, expansion caused by reactive MgO and CaO, seems to be overcome by the finely ground

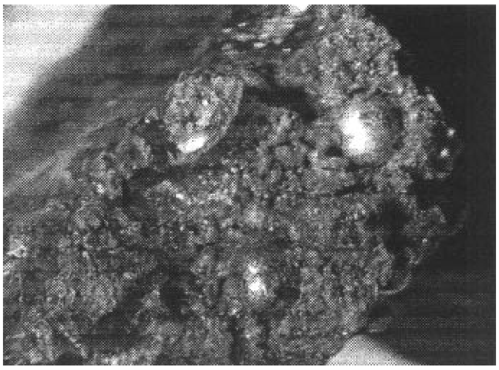

Figure 7. Granulated EAF-slag, surface of a larger granule with metal droplets

slags, they have the same volume changes as the cement reference. The ground material must, however, be used in slurry form since drying destroys the obtained surface activation of the particles.

- Remelting and granulation of slags had a positive effect on the examined ladle- and EAF-slag. The granulated AOD-slag, on the other hand, had decreased activity and retarded the cement reactions. The granulated slags' effects on cement hydration are positive when the glassy phase is increased and vice versa.

The results from the three investigations show that steel-slags can be used as filler in cement-based products since the problems of activation (effect on cement hydration) and durability can be overcome.

Acknowledgements

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