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## Quality of fine materials from crushed rocks in sustainable concrete production

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### ABSTRACT

Crushed rocks are in general more flaky and irregular in shape than natural aggregates. Moreover the crushed rocks give more fine material. Fillers can in combination with superplasticizers be used to expand the paste phase of the concrete and thus be used to lower the cement consumption. To utilize the filler optimally you need to evaluate the filler quality.

There are several methods to evaluate the filler. In this article different methods both as regard the material properties and the behavior in mortar and paste tests are compared and evaluated. The analysis shows the importance of understanding the effect of flakiness and the properties of the finest fraction  $< 10 \,\mu$ m.

Keywords. Concrete aggregate, Crushed rocks, Fine material, Filler, Test methods

### INTRODUCTION

The main type of aggregates used in Sweden today is glaciofluvial material from eskers. Good natural aggregates are locally becoming scarce and the authorities are restricting their use for environmental reasons. The only other source with sufficient supply is crushed rocks. In Sweden most of the bedrock is granitic and most of the quarries are situated in this rock type. The quarries were not opened to produce aggregate for concrete and it is known that crushed granitic rocks can contain minerals, which are not desired and can cause problems especially as regard the fine aggregate.

The coarse aggregates in concrete production are already from crushed rocks but now the concrete producers also have to learn to use fine aggregate from crushed rock. The quarries have to learn how to produce suitable fine aggregates and the concrete producers have to learn how to use this aggregate.

To solve this problem a national research program was initiated. Crushed products from more than fifty different quarries have been analyzed for particle size distribution, particle shape and petrographic and mineralogical properties. These properties have been correlated to concrete mixes and their rheological properties (Lagerblad et al 2008).

The outcome showed a large variation, were some were appropriate and resulted in aggregate that could easily replace natural aggregates in concrete, while others were not suitable as concrete aggregate. In part the problems could be related to the crushing process

but the major problem was that some rocks gave to much fines and that the fines contained unsuitable components. This was especially the case with some granites and granitic gneisses. Massive limestone and basic rocks produced better materials but these rock types sometimes also gave unsuitable materials in the finer fractions.

The major difference between crushed rocks and natural aggregates, in both the coarse and fine aggregates, is the grain shape. The grain shape of the coarser fragments can, by attrition, be made more cubic (Bengtsson 2006). Most material was crushed in cone crushers but by using a vertical shaft impact crusher (VSI) the particles can be made more cubic, but the attrition in the VSI increases the amount of fines (Cepuritas 2011).

Below a certain size the particles are mainly mono-mineral why the effect of VSI decreases. The shape depends on crystallographic structure of the individual mineral. Mica minerals are flaky and free mica accumulates in the fine fractions (Loorents 2007). If the amount of free micas in the fine fractions is high the workability of concrete decreases and the water demand increases. Mica minerals (biotite, muscovite and chlorite) are common in granitoid rocks. Chlorites can often be found in basic rocks.

#### The effect of grain shape on the rheological properties of fresh concrete

Fresh concrete is a particle slurry of aggregate, cement and water. When cast, all the particles interact; they collide and/or glide on each other. Thus for flaky particles larger amounts of finer material is needed to get a good workability. A common practice in mix design, when the coarse aggregate is flaky, is to increase the amount fine aggregate. If the smaller particles are also flaky, more materials are needed in the finer fractions. This holds down to the finest particles. Thus the amount of free flaky mica minerals is high more paste is needed. This will increase the water demand and more specifically the amount of "micro paste". Micro paste contains cement, water and fillers.

Thus it is important to be able to proportion the micro paste so that the amount of water could be kept to a minimum to give good strength. Earlier high filler amounts caused high water consumption as the fine particles have a tendency to flocculate, but the modern superplasticizers have made it possible to better utilize the fillers. This, however, demands that fillers have a good quality. In this paper we regard all particles as fillers when they can pass a 125  $\mu$ m sieve.

#### The effect of fillers in the micro paste

Aggregates always contain fillers. In European standards particles smaller than 63  $\mu$ m are classified as fillers. In practice, however, particles smaller than 125  $\mu$ m are called fillers and commonly the micro mortar includes water and all particles including cement that are smaller than 125  $\mu$ m. The quality of and size distribution of the filler effect both the rheology of the fresh concrete as well as the physical properties of the hardened concrete.

Especially the ultrafine particles, those with a size smaller than the cement, have a profound effect on the strength of the concrete. The cements of today often include limestone coground with the cement clinker (in Sweden CEM II/A-LL, EN196-1). The limestone (calcite) is softer than the cement minerals and will thus form smaller particles in the cement mill. Thus the limestone is basically ultrafine filler. The CEM II gives a similar strength as CEM I at the same w/c ratio, which indicates that a better particle packing in the micro mortar is achieved. (Lagerblad & Vogt 2004, 2008) showed that the same effect can be achieved with almost any mineral if the particle size is smaller than that of the cement. Other experiments (Lagerblad & Vogt 2004) also showed that the amount of ultra filler could be increased further and replace even more cement without loss in either porosity or strength.

The ultra fillers have the most profound effect on concrete properties but the ordinary filler is also important. In an experiment (Westerholm & Lagerblad 2012) fillers were separated from crushed granite by air classification. This product was then added to a concrete mix. The material passed the 250  $\mu$ m sieve. It showed similar but not as pronounced effect as the ultra filler. By replacing cement with filler and keeping the w/c ratio constant, higher strength could be achieved. Like with the ultra filler, the addition of filler requires the use of superplasticizers and the concrete gets more viscous. This shows that one can minimize the cement content by careful proportioning of filler.

Much of the effect of the filler is related to particle packing and a more homogeneous paste structure. For the best result the sorting curve of the filler, including cement and ultra filler, should be continuous (Hüskens & Brouwers 2008, Vogt 2010).

### **QUALITY OF FINE MATERIAL**

The effect of the fine aggregate is related to the particle distribution and to the particle shape. An effect of this is shown in Figure 1. The rheology of a 0-2 mm mortar was investigated in a viscometer (Contec 4-SCC). The mortar is based on the fine fraction of a normal house construction concrete with a w/c of 0.57 (Westerholm 2006 Lagerblad et al 2008). It gives the result split into yield stress and plastic viscosity. In the experiment a fixed recipe was used. In the first series of experiments, the aggregate was tested as it arrived (original) and in a second series the grain size distribution was adjusted according to an optimized sorting (Mix 2). The results show that both the yield stress and plastic viscosity was lowered when adjusted to the optimized sorting. But even with this optimized reference sorting there is a large difference that is related to the characteristics of the grains. The samples with high yield stress have unsuitable fillers due to different types of micas and clays, mainly in the ultrafine filler fraction. The variation in the mixes with optimized sorting is mainly related to flakiness and can to a large extent be correlated to the amount of free mica particles.

The effect of the filler is related both to the amount of filler and the quality of the filler. Figure 2 shows an experiment in which the micro mortar was investigated in a paste viscometer (HAAKE CV 20). Like in the earlier described test the composition of the micro mortar was based on a basic recipe of a house construction concrete with a w/c of 0.57. The two test mixes, one with 13.3 and one with 26.1 volume % particles passing the 250  $\mu$ m sieve, were analyzed. The results show that with increasing amount of fine material the importance of quality increases. Some of the aggregates like K18, K6, and K9 have similar effect as the natural aggregate, while K4, K8 K10 and K1 give a bad rheology when added in larger amounts. Typically all the samples that give bad rheology contain large amounts of free mica in the fine fraction. Sample K8 contains sericite in the ultra fine filler fraction (will be described later). Sericite is a fine muscovite that forms due to hydrothermal or weathering of K-feldspar. When the sericite was washed away from aggregate K8 it resulted in good rheological properties (Westerholm 2006).

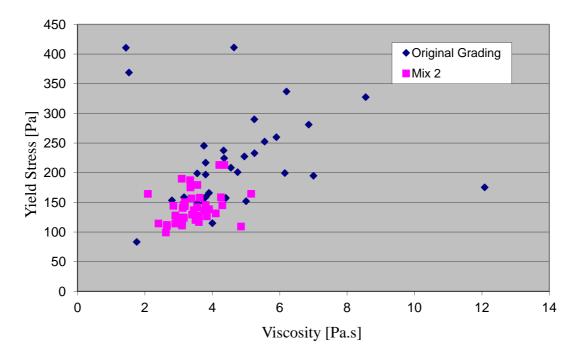


Figure 1. Yield stress and plastic viscosity of reference 0-2 mm mortars. The basic recipe contains 635 g. of cement (CEM II/A-LL), 1145 g. of 0-2 mm aggregate and 362 g of water per dm<sup>3</sup>. Original is as the aggregate arrived. Mix 2 is an optimized grading.

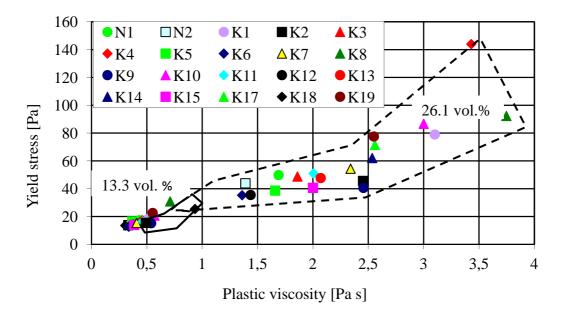
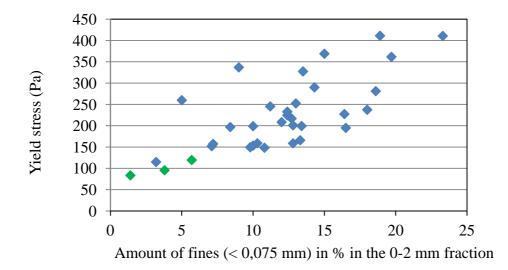


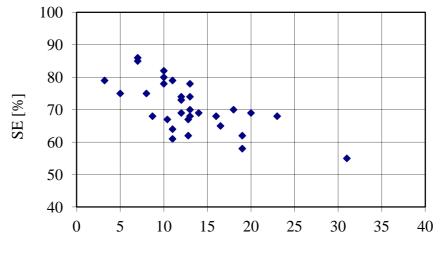
Figure 2. Yield stress and plastic viscosity of micro mortar containing 13.3 and 26.1 volume % of fines (<0.25 mm). From Lagerblad et al (2008). N1 and N2 are natural aggregates. K18 is a quartzite. The others are from granitic to granodioritic rocks. Data from K3 and K4, K6 and K8 are in Table 1.

Figure 3 shows the effect of the amounts of filler in the mix. The sorting is as it arrived. Typically the natural aggregates contain much less filler than the crushed rocks. With superplasticizer increased amounts of filler can be revoked.



# Figure 3. The diagram shows show the effect of different amounts of filler (< 75 $\mu$ m) on the yield stress of a 0-2 mm mortar. Green symbols are natural glaciofluvial aggregates and blue symbols crushed rocks. Original grading

The amount of fines can be estimated with the help of the sand equivalent test (EN 933-8). One problem here is that it only gives the amount. There is, however, no correlation between the amount of micas and the SE-value (Lagerblad et al 2008).



Amount of fines < 0,075 mm [%] in the 0-2 mm fraction

Figure 4. The effect of the amount of filler on the sand equivalent value.

## QUALITY AND ANALYSIS OF THE FILLER FRACTION IN AGGREGATES FOR CONCRETE

The filler and especially the fine filler are difficult to analyze, but basically the filler contains different free minerals. The type and amount of individual minerals depends on the rock type. The shape of the minerals is related to the crystallographic structure and properties of individual mineral. Micas and clays have a flaky crystal structure that is negative for the rheology and workability of the concrete. X-ray diffraction can give an indication on the mineralogy. The filler can also be analyzed in a scanning electron microscope. Normal stereomicroscope or thin section in polarizing microscope cannot unveil the finest particles. The methylene blue test (EN 933-9) sometimes unmasks bad components. Water absorbing clays like smectite give very large MB-values while sericite or large amount of ultrafine filler give a higher than average value. Thus a high value of the methylen blue test gives a high smectite content that is bad for both the fresh concrete and for the durability of the concrete (Lagerblad & Jacobsson 1997). A slightly increased MB-value indicates high contents of micas and clays in the filler (Lagerblad et al 2008).

The quality of the filler depends both on the size distribution and particle shape and presumably on the surface properties. The particle distribution can be analyzed by the sand equivalent test, but this test mainly gives the amount of filler and especially it reveals the amount of ultrafine material (Lagerblad et al 2008).

The size distribution can also be analyzed by laser sieve in combination with BET-surface. The laser sieve, however, assumes all particles are round and thus it does not give the full picture. Moreover, it does not fully reveal particles with sizes less than 1  $\mu$ m. Another method is the BET surface. With this method, the surface is measured. The surface is mainly given by the small particles, especially the ultrafine grains. By comparing laser sieve values with the BET surface (Table 1) some important information can be gained, but one must keep in mind that the laser sieve in contrast to the BET-surface does not take the finest particles into account.

Natural aggregates (N1 and N2) are from glaciofluvial eskers have high BET-surface but the amount of particles is small (see Figure 3). The origin of the natural aggregate is mainly granitic bedrock and the clays giving the high BET-surface are weathering products. The amount of ultra filler is relatively small and thus it does not cause any severe problems in concrete mixing.

In most crushed rocks the BET surface (<0.15 mm) is slightly more than 1000 m<sup>2</sup>/kg and the amount of particles with a laser sieve value of < 10  $\mu$ m is around 12 %. In general the amount of filler is higher in the crushed products. This is especially the case with samples that have passed a VSI-crusher.

Some of the quarries produce fillers with a much higher BET-surface than others. Two rock powders that give problems are E-VSI and another sample from the K8 quarry. Both give problems in concrete and in a rheology tests (see Figure 2). The sample from K8 is treated in Lagerblad et al (2008). Three different samples from different times and different parts of the K8 quarry were tested. They all gave high BET surface but image analysis and other tests showed that it was a good aggregate. When washed and the ultrafine material was removed it behaved very well showing that the properties of the ultra fine material are very important and the BET-surface is an important test. Further analysis in XRD (X-ray diffraction)

showed that the ultrafine part of the filler was enriched in sericite, fine-grained mica. Petrographic analyses showed that K-feldspar was altered due to sericitization. This is typical of either weathering or hydrothermal alteration. As the rock in the K8 quarry is not weathered it must be due to hydrothermal alteration. The petrography indicates that during crushing the sericite particles due to its small grain size in the rock was accumulated in the ultra filler fraction.

| Analysis < 125   |                    | BET surface | Laser sieve | Laser Sieve     |
|------------------|--------------------|-------------|-------------|-----------------|
| •                |                    |             |             |                 |
| μm               |                    | m2/kg       | < 10 µm     | < 63 µm         |
| N1               | Natural            | 6096        | 8.47        | 41.78           |
| N2               | Natural            | 3301        | 9.77        | 46.50           |
| ST-VSI           | Granite            | 1548        | 13.30       | 56.68           |
| ST-CC            | Granite            | 1261        | 12.16       | 51.30           |
| B-CC             | Granite            | 1069        | 9.31        | 46.53           |
| B-VSI            | Granite            | 1177        | 11.37       | 65.25           |
| D-VSI            | Granite            | 2161        | 11,47       | 56.26           |
| SK-VSI           | Granite            | 1552        | 13.64       | 54.84           |
| E-CC             | Granodiorite       | 1395        | 13.38       | 55.48           |
| E- VSI           | Granodiorite       | 3351        | 19.23       | 67.46           |
| L-VSI            | Gabbro             | 1265        | 11.14       | 55.59           |
|                  |                    |             |             |                 |
| Analyses < 250   |                    | Laser sieve | Laser sieve | Sand equivalent |
| μm               | m <sup>2</sup> /kg | %< 10 µm    | %< 63 µm    | 0-2 mm          |
| K4 CC (granite)  | 2490               | 5.3         | 27.4        | 64              |
| K2 CC (granite)  | 1315               | 8.1         | 35.9        | 72              |
| K3 CC (granite   | 840                | 6.6         | 26.3        | 77              |
| K8 CC (granite)  | 2588               | 10.4        | 33.2        | 62              |
| K6 old sample of | 610                | 5.5         | 24.2        | 69              |
| E-CC             |                    |             |             |                 |

## Table 1. Laser sieve and BET surface of a sequence of fillers. Analyzes on material < 125 μm. In the lower half of the table some old results from Lagerblad et al (2008) are included. These samples were analyzed on <250 μm.

The E-quarry is situated in a granodioritic rock type area. In earlier tests, marked as K6, it was one of the best aggregates and it had a low BET-surface and the amount of filler was low (See Figure 2). Petrographic analysis and XRD showed that the filler of the old sample was high in hornblende and low in chlorite. The new samples contained hornblende but were high in chlorites. Compared to cone crushing the VSI gave much more material < 10  $\mu$ m. Both the cone crushed and especially the VSI crushed rock gave bad rheology (see below). The problem with the G-quarry is probably similar to that of the K8 quarry. Chlorite is an alteration product from hornblende. When the rock and the hornblende are being crushed, the fine grained chlorites accumulate in the filler and especially in the ultrafine filler. In contrast to K8 the alteration is uneven and thus the product must be controlled and it must probably be washed to remove the ultrafine chlorite.

In most other cases the BET-surface lies around 1000-1500 m<sup>2</sup>/kg and when analyzed on the <250  $\mu$ m fraction around 800-1000 m<sup>2</sup>/kg. Some of them have slightly high values and in most cases these samples contain micas in the fine fraction.

There is also a range of indirect tests. One method is the Puntke test that basically is a water absorption test and gives an indication of surfaces that have to be wetted. The flow spread test (Hunger, M., and Brouwers 2009) relates the water/powder ratio to the spread (mini slump) and also gives an indication of the surface that needs wetting. Both the Puntke test and the flow spread test favors filler that are comparatively coarse but it gives good indications.

Both the Puntke and the flow spread test gives the amount of water needed to wet all grain surfaces. The Puntke test is easier to perform but the flow test is more accurate. The results from a set of tests are shown in Figure 5. Basically the good filler, like K3 (see Figure 2), needs little water to flow while bad filler, like in K4 (see Figure 2), needs more water. The results show that the test has a value but coarse fillers require less water than fillers with a lot of ultra fine material. Sample K 27 is from a crushed massive crystalline limestone with cubic calcite particles in the filler. Crystalline limestone in general gives more cubic grains and has better properties in concrete than granitic rocks. Sample K29 is crushed from a granodiorite with filler with low content of fine particles. Flow spread tests with K29 gives very good results as most of the particles are coarser than the cement particles. Both the Puntke and the flow spread test give results that can be used as quality criteria but one must first know the underlying fact to be able to use it as a standard test in the quarry.

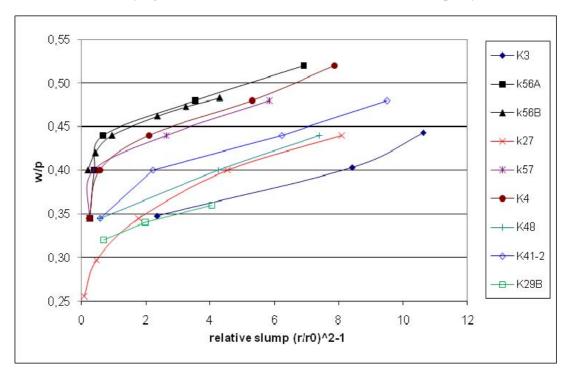


Figure 5. Flow spread test (Beta-p) for a set of fillers. 0-0.125 mm filler. w/p = water/powder. Relative slump =  $\left(\frac{r}{r_0}\right)^2 - 1$  where r = radius of spread, r<sub>0</sub> = flow cone diameter. The cone diameter is according to ASTM C-230.

The quality of the filler can also be tested mixed with cement and water. In a series of tests and standard mix we used a slump cone with a height of 57 mm an upper opening of 19 mm and a lower opening of 38 mm. The mixes are composed so that the amount of filler below and above 63  $\mu$ m is the same in all mixes. The mix contains 45 g CEM II/A-LL, 41.6 g filler

(<0.125 mm) and 36g water. The amount of filler is adjusted according to volume. Two test series were done, one with the filler as it arrived and one where it was split into 24.7 g < 63  $\mu$ m and 16.9 g 63-125  $\mu$ m. After testing the slump 9 g of water was added to the mix and a new slump test was made. This explains why the volume concentration of particles differs in the diagram. The measured flow was on a glass sheet.

The results show that the samples with the highest BET surface (E-VSI) also give the lowest spread. Sample E-CC and D-VSI that also have relatively high BET-surface also gives a relatively low slump flow. The other result is in much related to the amount of micas in sizes larger than  $10\mu m$ . More evaluations are needed.

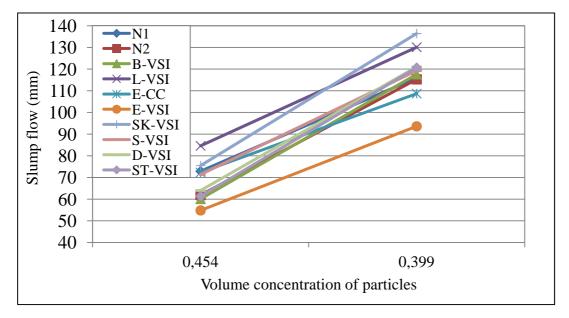


Figure 6. Micro mortar slump flow test. Explanation in the text.

### CONCLUSIONS

With crushed aggregates, due to the more irregular and flaky form, more paste is needed. Crushed aggregate normally contain more filler than natural aggregate. The filler fraction is together with cement and water a part of the paste. To expand the paste fraction it is necessary to have filler with good quality. The quality of the filler is the combined result of the sorting of the filler and the shape and surface of the filler particles. Filler particles are slowly soluble in the pore solutions of concrete but this will not affect the fresh concrete and will presumably not give expansive alkali silica reaction (Lagerblad 2012).

The quality of the filler has a profound effect on the workability of both mortar and concrete. In general crushing of rocks gives more filler than can be found in natural aggregate from glaciofluvial eskers. In old times it was difficult to make concrete with too much filler but today with modern effective superplasticizer it is possible incorporate higher contents into the concrete. The filler in combination with superplasticizer may even reduce the amount cement. This, however, requires that the filler is of good quality.

The filler in most cases contains free minerals that reflect the composition of the crushed rock. It is known that crushed granitic rocks give a more troublesome aggregate than for example limestone. One of the major problems is flaky particles especially free micas in the

finer fractions. Free mica is difficult to "cubisize" as the flakes rather bend than break. The micas seem to come out in the size they have in the rock. Micas in general shall be avoided. A special problem is the weathering or hydrothermal alteration products. Sericite alteration of K-feldspar or chloritisation of amphiboles can give accumulations in the ultra fine fraction. They are released to the ultra filler fraction during the crushing process.

There are several ways of analyzing the filler. The BET-surface is one of the best methods to reveal the quality of especially the ultrafine filler. X-ray diffraction and semi-quantitative analysis can give the type of minerals that in turn indicate the shape of the particles. The laser sieve indicate the particle size distribution. No specific method gives the full answers as regard quality thus a set of analysis are needed to evaluate the filler fraction of the aggregate. To find simple but yet efficient test methods for evaluation fillers for industrial purposes more research is needed.

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