Evaluation of Crushed Fine Materials

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ABSTRACT
As natural resources of sand and fine material used in the concrete industry are becoming more scarce, crushed materials are being used to a greater extent. Shape and texture of machine crushed materials differ from aggregates naturally ground, layered and sorted under the course of millions of years. It is assumed that crushed aggregates are more angular and of less smooth texture than natural aggregates, which will affect the flow behaviour of a suspension containing crushed material. In the mix design process of cementitious suspensions, an adequate rheology of the micro mortar (all constituents in the concrete being able to pass a 0.125 mm sieve, including the cement) is crucial. In this paper, the shape of fine particles is linked to the micro mortar plastic viscosity of the filler suspension including cement. The plastic viscosity here serves as an important quality
assessment of the filler, since the micro mortar workability features are vital for the final mix design quality of the concrete workability.

**Key words:** Instructions, layout, headings, figures and tables, references.

1. **INTRODUCTION**

Since there is more awareness on environmental friendlier production methods of concrete, natural resources of aggregates are more often spared and crushed rock material is more frequently favoured. Moreover, fillers can be used in combination with superplasticizers in order to expand the paste phase of concrete and thereby lower the cement consumption [1]. The trend towards the use of more crushed aggregate material and crushed fillers of different minerals and crushing techniques demands a simple filler quality assessment. It is assumed that crushed aggregates are more angular and that traditional grading curves used for mix design [2],[3],[4] are not simply applicable on irregularly shaped particles. This irregularity in shape affects the workability of the produced micro mortar. Furthermore, it is also assumed that both Bingham workability parameters, yield stress as well as plastic viscosity, are increased with an increased level of aggregate angularity [5]. Yield stress (τ₀, associated to the deformability of a material) might be lowered using superplastisizers whereas plastic viscosity (µpl, ease of material movement) might not [6], to the same extent. It is the increased plastic viscosity that may cause problems when designing the final concrete mix, which will inherit workability qualities that might not be adequate. Therefore, the fine particle fraction is here tested for angularity and plastic viscosity in order to establish a link between filler shape and plastic viscosity of the micro mortar suspension including the filler. Different test methods are shown, both in regard to the material properties as well as rheological measurements of the micro mortar paste phase.

2. **Features of fine rock particles**

2.1 **Particle shape**

Particle size and shape is defined in different ways. Particle size is here characterized by apperture, its shape is characterized by its aspect ratio, the ratio between the largest axis and the smallest axis. The grain shape of larger particles is to a large extent dominated by their crushing method. Preprint submitted to Elsevier February 10, 2015 They may be labeled rounded, irregular, flaky, angular, elongated, or flaky and elongated [7]. The authors would like to add the feature cubical as well, since some of the crushing methods result in rather cubical particles and some minerals resemble cubes in the finer fractions, see Fig. 1. The grain shape for small particles, such as fillers, is dominated by the particle mineralogy. Minerals are internally ordered according to their crystallization. This order is arranged in a repetitive three-dimensional array, as a patterned unit. These identical patterned units are found along a three-dimensional lattice in such a way that all have identical environments. The lattice is defined in three directions x, y, z and by the distance at which the pattern is repeated. It was demonstrated by Bravais 1848, [8] that it is geometrically
possible to have fourteen types of space lattices in order to keep a complete, repetitive pattern undestroyed. These patterns and compounds are connected in a way that is distinct for the particular mineral. Each mineral has both stronger and weaker bonds between its chemical compounds and repetitive patterns that differ from other minerals in distinct angles and directions according to Bravais' fourteen space lattices. Most minerals are made of ions or clusters of ions held together by electrical forces that arise between oppositely charged bodies.

Factors like the arrangement in space of these ions and ionic groups and the strength of the electrical forces that hold them together all comprise the structure of the crystal. When splitting a mineral, it will always split along one of the weaker bonds. Cleavage could be thought of as a discontinuous property reflecting the internal structure, since cleavage always takes place along those planes across which there exist the weakest electrical forces [9]. For this reason, particle shape is dominated by the crystal structure of the mineral. This particle shape becomes apparent as particles are sized below the so called free mineral limit. The free mineral limit is defined as the particular size limit below which the free mineral grains are exceeding the amount of rock fragments [10]. The free mineral limit differs from one rock type to another and presents a way to define the coarsity of the rock. Further cleavage results in a smaller particle breaking along the weakest bond/s. Crushing mica will result in even more flaky mica (weaker bonds in one direction and strong bonds in the two other directions), crushing a dolomite will result in smaller cubical particles (equally weak/strong bonds in all directions). This is further verified by the so called "Law of Constancy of Interfacial Angles". Danish scientist Niels Stensen in 1669, [11] found that identical faces on quartz crystals always included the identical angles 2 between faces. This law signifies that only the angles between faces are important, not the relative sizes of the faces. The law of constancy of interfacial angles was further confirmed in 1783 by Romé de L'Isle, [12] as a general valid law applying to all crystals [13].
2.2 Packing

Packing and packing rate of particles is another way to define particle quality. It is used widely in the field of geotechnology and has also become a common way to design a concrete recipe. Random (loose or poured) packing of monosized perfect spheres gives a packing ratio of approximately 62.5 %. Placing the spheres one by one to create a maximum packing grade will give a denser packing, a virtual packing density is defined by Larrard [14] as the perfectly attainable packing/placing of aggregates as possible. The virtual packing density of perfect monosized spheres is equal to $\pi/3 \sqrt{2} \approx 0.74 \%$, while the physical packing density that can be actually measured in the lab is almost identical to the loose packing of spheres. In order to pack aggregates as compact as possible, a number of methods are used: vibration, application of pressure or centrifugal forces. For fillers usually the maximum packing rate is also obtained using a wet method, sometimes including superplasticizer, in order to avoid particle charges to influence the maximum packing rate. Once smaller spheres are introduced into the monosized mix, perfectly filling the voids between the larger, original spheres, a denser packing is obtained. The span of particle sizes as well as how well the particles fit into one another affects the packing density. Other parameters that affect the particle density are shape and surface roughness of the particles. Flaky and elongated/rodlike particles may be compacted to a much larger extent than rounded particles and thus filling all the voids. However, it was also found that mix proportioning models minimizing the void ratio for maximum packing produces rather harsh concrete mixes. It is interesting to note that the flaky and rodlike aggregates produce a quite porous packing when random/loose packing is applied. For loose packing, perfect spheres render the highest packing rates whereas other aggregate shapes result in looser packing. For monosized particles of the same surface roughness (friction coefficient), loose packing as well as hard packing values are determined by particle shape.

3. PREDICTION OF VISCOSITY OF PARTICLE SUSPENSIONS

So far, plastic viscosity $\mu_{pl}$ was mentioned as a Bingham parameter. One also speaks of apparent viscosity $\eta$. For a moving material, it is the ratio between the stress level $\tau$ and the shear rate $\dot{\gamma}$:

$$\eta = \frac{\tau}{\dot{\gamma}}$$

(1)

with $\dot{\gamma} = \dot{x}/h$ for velocity $\dot{x}$ and material thickness $h$. As seen, the apparent viscosity changes as the shear rate changes, whereas plastic viscosity does not. Introducing yield stress $\tau_0$, plastic viscosity is defined as:

$$\mu_{pl} = \frac{(\tau - \tau_0)}{\dot{\gamma}}$$

(2)

Both apparent and plastic viscosity take the same value as the shear rate approaches infinity: $\dot{\gamma} \to \infty$ we obtain $\eta \to \mu_{pl}$. As can be read in [15], Einstein studied the increase in viscosity, adding to a Newtonian fluid a perfect sphere, as discussed in his papers dating back to 1906 and
1911. For an incompressible Newtonian liquid subjected to creeping flow a density neutral particle \( \rho_f = \rho_p \), density of fluid equals density of particle, increases viscosity by

\[
\eta = \eta_f (1 + 2.5\varphi)
\]

This holds true for a sufficiently small particle volume fraction, \( \varphi \), with no interaction of particles. The value 2.5 accounts for particle shape characteristics, it is the so called intrinsic viscosity \([\eta]\) for spherical particles. The relative viscosity \( \eta_r \) is defined as

\[
\eta_r = \frac{\eta}{\eta_f}
\]

with subscript \( f \) being the fluid without particles, whereas the specific viscosity is

\[
\eta_{sp} = \frac{(\eta - \eta_f)}{\eta_f}
\]

The intrinsic viscosity is written

\[
[\eta] = \lim_{\varphi \to 0} \frac{\eta_{sp}}{\varphi}
\]

Einstein's equation (3) can now be rewritten as \( \eta = \eta_f(1 + [\eta]\varphi) \) or \( \eta_{sp} = [\eta] \varphi \). Also note that the particle radius does not affect the viscosity, as long as the liquid volume is of an adequate amount. Years after Einstein's definition of the relation for viscosities of dilute suspensions, [16] published a relation introducing a self crowding factor to account for particle interactions of more concentrated suspensions. The crowding factor is today commonly replaced by the maximum solid fraction \( \varphi / \varphi_{max} \). Krieger and Dougherty's relation states that, for any kind of particle shape, the relation

\[
\eta_r = (1 - \frac{\varphi}{\varphi_{max}})^{-[\eta] \cdot \varphi_{max}}
\]

holds for concentrated suspensions. Particle asymmetry has a strong effect on the intrinsic viscosity and maximum packing fraction. Any deviation from the spherical shape results in an increase of the viscosity at the same volume concentration, [17],[18]. Equation (7) reduces to Einstein's equation for spherical particles in a dilute suspension. Krieger-Dougherty curves for Portland I cement with 0 % and 5 % superplasticizer are presented by [19].

4. OPEN CHANNEL FLOW TESTS

For slow open channel flow of given volume on a horizontal surface, the yield stress of a Bingham material is easily determined by a known relation between yield stress and spread length at flow stoppage. Once a known volume of a Bingham material is instead quickly released into the channel, the product of its final propagation at flow stoppage and propagation speed results in a plastic viscosity equation. It was previously shown that

\[
\mu_{pl} / (\rho / \rho_{water}) = A \cdot \ell \cdot t_x + B
\]

with \( (\rho / \rho_{water}) \) being the specific gravity of the material and parameters \( A = 0.0217 \) \( B = -0.0192 \) [20]. A promising R² value of 0.98 for previous lab test results with a MCR 300 rheometer
(Physica) equipped with a concentric measuring system, was obtained [21]. Fig. 2 shows the rheological measuring apparatus, developed to determine rheological characteristics. It consists of an open channel manufactured from frosted glass to ensure a low slip condition of the material. The channel is a 350 mm long horizontal open channel of 25 mm width. At one end of the channel, a $H = 50$ mm high gated column is attached to hold the fluid sample to be released. A volume of $19.75 \cdot 10^{-6}$ m$^3$ of fluid is filled into the container and then quickly released into the channel once the gate is quickly opened.

![Figure 2 – Geometry of the measuring box.](image)

Opening the container gate is performed by hand in one swift movement, the operator is the same for all laboratory experiments. A camera mounted straight above the measuring box is capturing the flow propagation of the released fluid. Gate opening time has been excluded from the duration of flow propagation by starting the clock at the first visible fluid exiting the gate. In addition to filming the spread in the transparent channel on graph paper and charting its propagation, time $t_x$ for the spread to reach a certain distance from the gate ($x = 70$ mm) is recorded as well. Final spread length $l$ is measured from the container gate to the center of the front line after flow stoppage. Open channel flow is utilized in favour of a rheometer to obtain plastic viscosity, since some of the filler mixes were rather viscous and of high concentration. In this case, segregation of the highly concentrated suspension sample in the rheometer cannot be ruled out.
5. EXPERIMENTAL DATA

5.1 Fine particle characteristics

The here presented fines in Table 1. are all cone crushed and wet sieved down to the fraction 63 - 125 µm according to [22] in order to eliminate the finest fraction so that particle shape analysis in the SEM is facilitated. Nevertheless, the fine materials still contain particles below the smallest sieve size to different amounts, since static electrical forces keep some particles clumped together.

Table 1 – Material data

<table>
<thead>
<tr>
<th>heightType of fines</th>
<th>$\rho$ [kg/m$^3$]</th>
<th>$&lt;63\mu$m %</th>
<th>Mica %</th>
<th>BET m$^2$/kg</th>
</tr>
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<tbody>
<tr>
<td>G1</td>
<td>2660</td>
<td>7.07</td>
<td>1.0</td>
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<td>MR</td>
<td>2700</td>
<td>8.43</td>
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<td>499</td>
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<td>G2</td>
<td>2610</td>
<td>11.06</td>
<td>3.0</td>
<td>503</td>
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<tr>
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<td>2650</td>
<td>8.51</td>
<td>1.5</td>
<td>873</td>
</tr>
<tr>
<td>GD$^\circ$</td>
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<td>8.51</td>
<td></td>
<td>1045</td>
</tr>
<tr>
<td>G4</td>
<td>2670</td>
<td>9.42</td>
<td>12.5</td>
<td>486</td>
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<tr>
<td>DB</td>
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<td>10.81</td>
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<td>1001</td>
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<tr>
<td>G5$^\circ$</td>
<td>2690</td>
<td>15.10</td>
<td>25.5</td>
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<td>14.19</td>
<td>22.3</td>
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</tr>
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<td>9.77</td>
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<td>42.5</td>
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<tr>
<td>CD$^\circ$</td>
<td>2750</td>
<td>4.39</td>
<td></td>
<td>174</td>
</tr>
</tbody>
</table>

$^\circ$ calculation of aspect ratio as the ratio between the largest axis to the smallest axis from SEM analysis

Notation G stands for a granite type of fine material

Notation MR stands for Metorhyolite

Notation GD stands for Granodiorite

Notation DB stands for Diabase

Notation IL is an Impure Limestone

Notation CD is the abbreviation of Crystalline Dolomite

Laser sieve curve and BET surface (Brunauer-Emmett-Teller, [23]) are carried out by Cementa AB, Sweden. The samples are all naturally dry and thoroughly mixed. Loose packing has been obtained according to [24], the material was poured into a 100 ml cylindric copper container of
height 85 mm and then weighed. The mica content of some samples was evaluated by point-counting thin sections, [25].

5.2 Cement paste mixes

A Swedish cement, type CEM II/A-L 42.5 R with a specific (BET) surface area of 1740 m²/kg was used in the experiments. The cement is co-ground with limestone, giving a limestone content of approximately 13 weight percent. The density of CEM II is ρ = 3080 kg/m³. The dry content of the polycarboxylate ether type of polymer superplasticiser is 35 percent. The paste was mixed in a Hobart mixer with the following mixing protocol:

1. All dry materials are mixed for 60 sec.
2. Water is poured during the following minutes during slow agitation (gear 1), 120 sec.
3. Superplasticiser is carefully added, 120 sec.
4. Scraping of the bowl is done manually, 60 sec.
5. High speed agitation of the mix (gear 2), 60 sec.
6. Resting of the paste in the bowl, 120 sec.
7. Scraping of the bowl is done manually, 60 sec.
8. High speed agitation, 60 sec.

Rheological measurements were performed with a MCR 300 rheometer (Physica) equipped with a concentric cylinder measuring system. The inner cylinder is profiled in order to avoid a slip surface. Part of the remaining paste is then mixed by hand with a spoon in a cup with one of the fine materials at a predefined concentration. Stirring is allowed to continue for several minutes to ensure a fully dispersed particle system. The plastic viscosity of the mix is then determined in the flow channel measuring system calibrated against the physica. Lastly, a sample from the remaining original cement paste is again measured in the rheometer. Time for the three measurements is clocked. Paste viscosity at the moment of filler mix measurement has been obtained by linear interpolation between the paste measurements before and after the fine material paste measurement, in order to account for any kind of workability loss. This way, a relative plastic viscosity µr of the filler mix can be obtained. The mix design of the cement paste is:

- 1000 g cement
- 350 g water
- 15 g superplasticiser

The amount of superplasticiser is rather high since the suspension is designed to hold high amounts of non-spherical filler particles.

6. RESULTS AND DISCUSSION

Fig. 3 confirms Krieger and Dougherty's relation, Eq. (7) for different types of fine material diluted in a predefined cement paste renders different curves. Fig. 3 shows the plastic viscosity of
different fine materials at different levels of volumetric concentration in paste mix. The volumetric concentration is calculated according to $V_f/(V_f + V_c)$ with $V_f$ being the volume of the fine material and $V_c$ being the volume of the cement paste. Data for a suspension containing perfect spheres was added, its intrinsic viscosity $[\mu]$ has a value of 2.5 according to Einstein and the maximum packing grade $\varphi_{\text{max}} \approx 0.74$ as stated in Section 2.2. This case is also found in Fig. 4, where the loose packing rate of a perfect sphere is 62.5 and the aspect ratio is at the minimum value 1.0. The same fine fraction materials are found in both aggregates, it is easily spotted that the perfect sphere has the lowest aspect ratio as well as the lowest plastic viscosity.

Figure 3 – Plastic viscosity of different fine materials at different levels of concentration in paste mix.

The coefficient of determination, [25] $R^2 = 0.94$ shows a high correlation between aspect ratio and loose packing. Without the point for the case of the perfect sphere, $R^2$ reduces to 0.85. It is still considered to be a strong correlation. Loose packing also shows a strong connection to plastic viscosity, as seen in Fig. 5. Since both intrinsic viscosity $[\mu]$ and packing $\varphi$ depend on particle shape, and both are mathematically connected to one another, one representative point on a plastic viscosity curve according to Krieger-Dougherty is sufficient to characterize a specific material.
In our case, a volumetric fine material addition of 60% of cement volume presents this characteristic point on the curve. The relation between the plastic viscosity with 60% fine material added to the paste and loose packing is shown in Fig. 4. This shows, that one simple mix of paste is sufficient to determine the quality of the fine material, such as aspect ratio and suitability for concrete mix design. Loose packing may also give an indication of mica content, which aspects aspect ratio to a great extent as seen in Fig. 2. A linear relation between loose packing and mica content is given by \( y = -0.1747x + 42.593 \) with an \( R^2 = 0.79 \). It is known that a mica content of more than 20% of the fine material is likely to give a harsh concrete mix. It is then easily seen, that a loose packing rate of less than 39.1% is likely to be due to particles of less favourable shape that will probably not produce a workable concrete. It is even more advantageous to test one fine material mix with the given cement paste design in order to evaluate the fines. Just one mix with a 60% fines by cement volume is sufficient to test for plastic viscosity. A plastic viscosity of more than 8.6 Pas indicate that the fines are not suitable to use in a concrete without modifications. Possible modifications include wind sieving, mixing with different type of fines or introducing smaller particles of better shape, such as silica fume.

Figure 4 – Aspect ratio and loose packing show a high correlation.
No correlation between BET-surface and plastic viscosity was found. Instead, a high BET-surface is usually associated with clays in the clay fraction < 4µm. These would instead affect the yield stress to higher values.

7. SUMMARY

A clear correlation between mica content and an unfavourable particle shape of the fine material has been established. The mica content can be determined by point counting thin sections. However, an easy-to-use quick rheological micro mortar test will provide an adequate quality assessment of the tested filler added to the cement paste. A testing volume as small as 19.75·10⁻⁶ m³ fluid is sufficient to obtain this important information about the filler quality.

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