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Measurement of particle size distribution and specific surface area for crushed concrete aggregate fines

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ABSTRACT

Different methods for measuring particle size distribution (PSD) and specific surface area of crushed aggregate fines (\leq 250 µm), produced by high-speed vertical shaft impact (VSI) crushing of rock types from different quarries in Norway, have been investigated. Among all the methods studied, X-ray sedimentation is preferred because it has adequate resolution and requires fewer and more reliable input parameters. This combination makes it suitable for practical applications at hard rock quarries. X-ray microcomputed tomography (μ CT) combined with spherical harmonic analysis was applied to estimate the actual error introduced when PSD measurements were used to calculate the specific surface area of the VSI crushed rock fines. The µCT results, to the limit of their resolution, show that the error in the calculated surface area caused by assuming spherical particles (a common assumption in PSD measurements) is of unexpectedly similar magnitude (-20% to -30%) over the entire investigated particle size range, which was approximately 3-200 µm. This finding is important, because it simplifies relative surface area determination and is thought to be quite general, since the crushed aggregate fines investigated were produced from 10 rock types that had a wide range of mineralogies.

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50 1. Introduction

The concrete aggregate industry has historically limited particle 52 size distribution (PSD) analysis, for fine particles, to simply determining the mass fraction of particles passing a sieve with square 53 openings of minimum edge length 0.063 mm (according to EN 54 933-1 [1]) or 0.075 mm (according to ASTM C136 [2]). The Euro-55 pean industry standard method intended for analysing the grading 56 of filler aggregates, namely EN 933-10 [3], is similar. This standard 57 58 only describes a method of more precisely determining the amount 59 of particles that are smaller than 0.063 mm, but not differentiating 60 the particles beyond that. On the other hand, natural and manufac-61 tured concrete fine aggregates (sand) have been reported to include particles down to the sub-micrometer size range [4-7]. 62

The importance of a more detailed fine particle analysis has become more evident during the last few decades, with the need to replace the use of depleting natural sand materials, which normally contain little of the fine material that passes a 0.063 mm sieve, with manufactured crushed sands that generally include a much higher fine material content [8]. Accurate determination of the particle size distribution (PSD) of this material in the size range <0.063 mm is expected to provide valuable information for concrete proportioning [4,5,9–11,6]. Fines have a significant influence on most concrete properties, both in fresh and in hardened concrete. The PSD and specific surface area are the main parameters used to describe fines. Furthermore, the influence of fines is even more pronounced for modern high-workability concrete such as self-compacting concretes [11,6,7].

As there is no standard procedure covering the whole range of concrete aggregate PSD, different researchers [12,4,13,10,14,5, 11,6,7] have used widely different measurement methods. It is, however, well-established from research within the geological 80 sciences on analysing natural sediments of similar grain size distri-81 butions [15–18] that different measurement methods can yield

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Table 1

Crushed rock fines used for the study.

Rock type Rock type designation		Mylonitic quartz diorite T1	Gneiss/granite T2	Quartzite T3	Anorthosite T4	Limestone T5	Limestone T6	Dolomite T7	Basalt T8	Aplite T9	Granite/ gneiss T10
Fraction	Nominal size ^a [µm]	Designation of crushed aggregate fines									
Fine Medium Coarse	4–25 20–60 40–250	T1-1 T1-2 T1-3	T2-1 T2-2 T2-3	T3-1 T3-2 T3-3	T4-1 T4-2 T4-3	T5-1 T5-2 T5-3	T6-1 T6-2 T6-3	T7-1 T7-2 T7-3	T8-1 T8-2 T8-3	T9-1 T9-2 T9-3	T10-1 T10-2 T10-3

^a The nominal size is approximate given in terms of the d_{10} and d_{90} diameters, which means that each size range can include up to about 10%, by mass, smaller and larger particles.

Table 2

Mineralogical composition of 4-25 µm powder fractions determined with quantitative XRD.

Rock type	Mylonitic quartz diorite	Gneiss/granite	Quartzite	Anorthosite	Limestone	Limestone	Dolomite	Basalt	Aplite	Granite/ gneiss
Rock type designation	T1	T2	T3	T4	T5	T6	T7	T8	T9	T10
Tested fraction	4–25 μm									
Mineral or group of minerals	Mass %									
Quartz	27.9	20.9	90.0	6.5	2.3	2.5	1.1	8.9	36.2	17.8
Carbonate minerals	4.4	-	3.6	10.6	97.7	95.0	95.0	8.3	-	5.0
Epidote minerals	8.4	-	-	24.4	-	-	-	7.6	-	-
Feldspar minerals	37.7	63.9	3.9	33.1	_	0.4	0.6	26.5	58.2	58.8
Sheet silicates	8.0	8.1	1.5	20.4	-	1.5	0.7	5.2	2.7	9.2
Chlorite	11.3	1.4	1.0	2.6	-	0.6	1.6	20.2	1.7	0.5
Inosilicate minerals	1.0	3.9	-	2.3	-	-	1.1	11.0	1.2	8.7
Iron oxide minerals	-	-	-	-	-	-	-	3.5	-	-
Other minerals	1.3	1.9	-	0.2	-	-	-	8.8	-	-

very different results depending on the properties of the analysed
materials. A recent study [7] suggested that this can also be true for
crushed concrete aggregate filler materials. Therefore, a variety of
measurement techniques has been investigated in this paper to
better understand how the size and surface area of fine particles
can be determined and how the results can be interpreted in terms
of particle size, surface area, and shape.

90 2. Materials and methods

91 *2.1. Materials*

Fine aggregate powder (filler) materials used for the study were 92 produced from 10 different blasted and crushed rocks with an 93 94 original size range of about 4-22 mm. Further processing included 95 Vertical Shaft Impact (VSI) crushing to generate fines and airclassification into three distinct size fractions with approximately 96 the following d_{10} to d_{90} ranges: 4–25 µm, 20–60 µm and 97 40–250 μ m (Table 1). The size parameter d_N is the maximum 98 diameter of the smallest N % of the particles by mass. Thirty differ-99 ent fine powder samples were produced: three particle size ranges 100 101 for each of the 10 rock types with different mineralogical compositions (Table 2). The finest of powder fractions (4–25 µm) included 102 all the particles smaller than 4 µm generated during the crushing 103 104 and afterwards extracted by air-classification. Mineralogical com-105 position of the powders was determined by quantitative X-ray diffraction (XRD) analysis. The samples were first ground using a 106 micronizing mill with agate grinding elements to a fineness of 107 108 d_{50} approximately equal to 10 μ m, using ethanol as a grinding 109 fluid, and subsequently dried overnight at 85 °C in a covered petri 110 dish. After drying, the sample material was put in a poly(methyl

methacrylate) (PMMA) specimen holder following minor 111 adaptations of standard procedures [19]. XRD data were collected 112 in a Bruker¹ X-ray Diffractor D8 Advance, using 40 kV, 40 mA and 113 Cu K α radiation of wavelength K α 1 = 0.15406 nm and 114 $K\alpha 2 = 0.154439$ nm and a $K\alpha 1/K\alpha 2$ ratio of 0.5. Diffractograms were 115 recorded at diffraction angles (2 θ) from 3° 2 θ to 65°, in 0.009° 116 increments with 0.6 s counting time per increment. The total 117 analysis time per sample was 71 min. Further analysis was based 118 on the X-ray powder diffraction results and the minerals in the ICDD 119 database implemented in the software Bruker EVA[®]. The first step 120 was mineral identification, and then the peaks of each mineral were 121 scaled manually to give the best fit to the observed XRD 122 diffractogram. The semi-quantitative mineralogy found based on 123 2θ -intensity data analysed by the XRD instrument was passed to 124 the software Topas Rietveld XRD, which was used to perform a struc-125 tural refinement. The results of the analysis (Table 2) are provided 126 only for the $4-25 \,\mu m$ fractions, but the mineralogical composition 127 was in fact determined for all three size ranges of the fillers. The 128 compositional variation among different particle sizes of the same 129 rock type was relatively small, which is why all of the results have 130 not been reported here. The uncertainty in the mineralogical compo-131 sitions presented in Table 2 is estimated to be about ±1.6% out of the 132 mass% for a single mineral phase at the 95% confidence level, as also 133 demonstrated for rock material by Hestnes and Sørensen [20]. The 134 groups of minerals used in Table 2 can include up to three different 135 individual minerals. 136

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¹ Commercial equipment, instruments, and materials mentioned in this paper are identified in order to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology (NIST), nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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