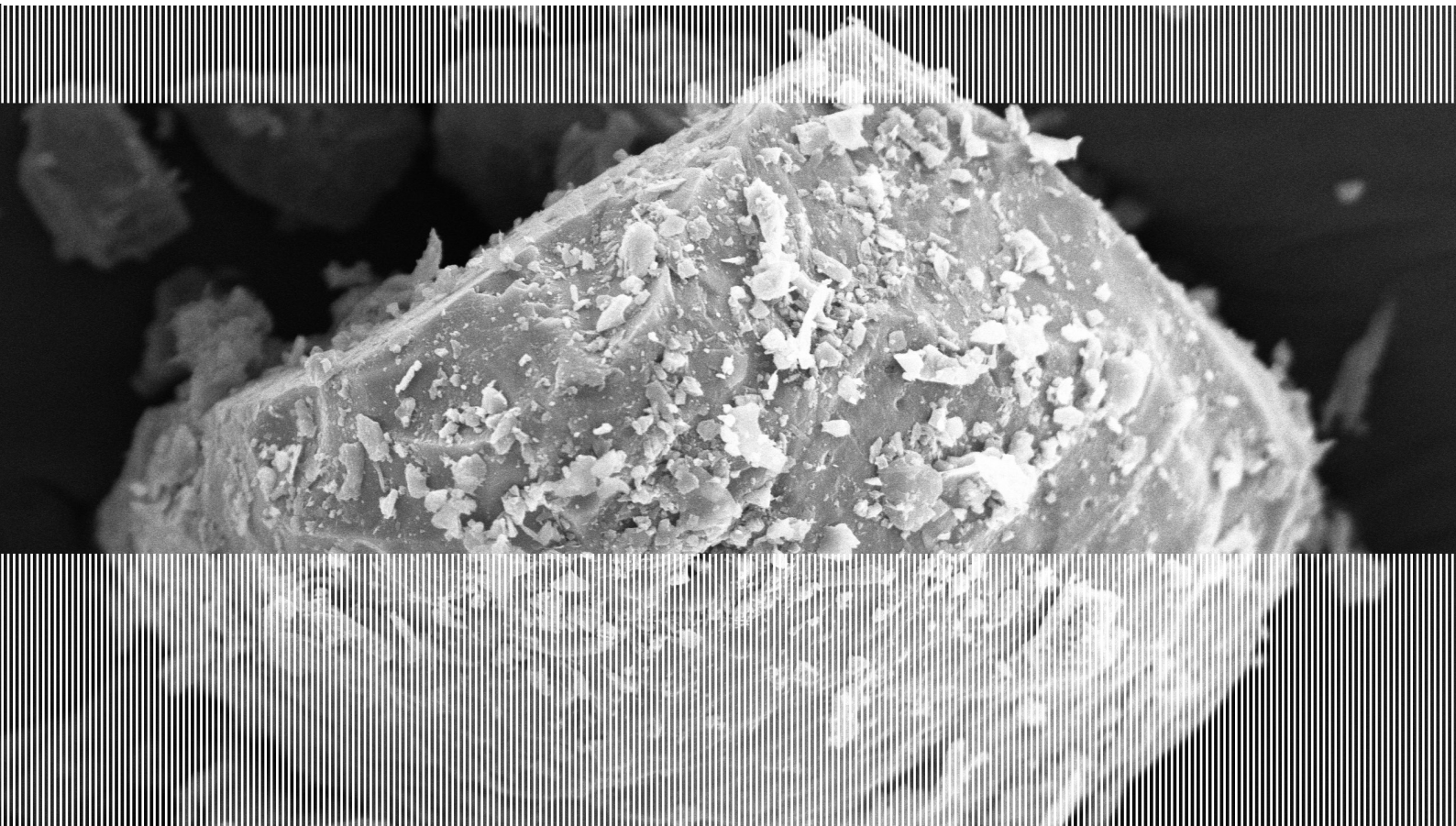


SINTEF Building and Infrastructure Rolands Cepuritis (Norcem AS)

Physical properties of Norwegian mineral fillers investigated by different methods

COIN Project report 43 – 2012



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FA 2 Competitive constructions

SP 2.1 High quality manufactured sand for concrete

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Keywords:

Manufactured sand; filler; water absorption; porosity; specific surface

Project no.: 33D005940

Photo, cover: Rolands Cepuritis

ISSN 1891-1978 (online)

ISBN 978-82-536-1296-6 (pdf)

ISBN 978-82-536-1297-3 (printed)

13 copies printed by AIT AS e-dit

Content: 100 g Scandia

Cover: 240 g Trucard

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Glossary of notations and terms

Terms:

Apparent particle density = ratio of the oven dried mass of a sample of aggregate to the volume it occupies in water including any internal sealed voids but excluding water accessible voids.

Filler = all fine mineral aggregate particles less than 125 μm if not stated otherwise.

Matrix = The matrix phase of a concrete mix is defined to consist of free water, admixtures and all solid particles having particle size less than 0.125 mm, i.e. binder and filler of the aggregate.

Particle density on an oven dried basis = ratio of the oven dried mass of a sample of aggregate to the volume it occupies in water including both internal sealed voids and water accessible voids.

Particle density on a saturated and surface-dry basis = ratio of the combined mass of a sample of aggregate and the mass of water accessible voids to the volume it occupies in water including both internal and water accessible voids when present.

Notations:

BET (method) = is a specific surface area determination method based on a theory published by Brunauer, Emet and Teller (BET) in 1938.

PSD = particle size distribution.

RH = relative humidity.

SSD = saturated surface-dry.

SEM = scanning electron microscope.

VSI = vertical shaft impactor.

1. Introduction

1.1. Background

As described in the State-of-the-art report by Wigum et al (2009), there is an increasing miss balance between the need for aggregates in the society and the available geological sources traditionally used for concrete. Some of the glaciofluvial deposits in Norway, best suited for concrete purposes, have an expected lifetime of less than 10 years. Transportation distances from the good resources to the urban areas increases and consequently also the environmental impact. The need to develop technology for 100 % use of manufactured aggregate in concrete is then obvious.

Naturally weathered sand differs from most fine crushed aggregates (manufactured sand) by grading, particle shape and surface texture. Typically crushed fine aggregate would incorporate *a lot more fines (filler)*, different particle size distribution (than natural sand) and be more angular with rougher surface. Due to these differences, concrete with crushed aggregate often displays higher water demand and lower workability than the corresponding concrete with glaciofluvial aggregate (Ahn 2000, Quiroga 2003, Westerholm 2006a, Kim et. al. 2008, Cepuritis 2011). The differences are more expressed if the crushed fine aggregate is a by-product (also known as “waste sand” or leftover rocks from quarrying) of coarse aggregate production and no special processing techniques are utilized to improve the characteristics.

The changes in the fine aggregate market described above have renewed interest focusing on the production of manufactured sand for use in concrete. As a result, rather extensive studies on crushed fine aggregate have been carried out and are still in progress within several research programmes accompanied with research in universities. Such as ECOserve¹ established by the European Commission in 2002 (completed in 2006) and COIN² established by the Research Council of Norway in 2007 (planned to be completed in 2015). Extensive Master level study has just (2011) been accomplished by Cepuritis at Norwegian University of Science and Technology, four Licentiate theses have also lately been delivered at Chalmers and Luleå Universities of Technology in Sweden (Esping 2004, Westerholm 2006b, Johansson

¹ <http://www.eco-serve.net>

² <http://www.coinweb.no>

2008 and Johansson 2011) along with two Doctoral theses at The University of Texas in Austin, USA (Ahn 2000, Quiroga 2003).

1.2. Significance of the fine particles

The most significant results of the research on crushed fine aggregate done so far within the COIN project (Cepuritis 2011) indicate that the filler part (fines) of the manufactured sand is mainly responsible for the effect on rheology of fresh concrete. This is with the condition that the PSD of the coarse fractions in the sand is not extremely altered or the particles of the sand are not very flaky due to improper crushing technique or unusual mineralogy of the parent rock.

It must be noted here that the particle size definition of fines is diverse. According to the EN-product standard EN 12620 (2008) for concrete aggregates, fines are all material less than 63 μm . ASTM standard C33 / C33M – 11 (2011) has a similar limit of 75 μm . For practical concrete proportioning purposes (Mørtzell 1996, Smeplass and Mørtzell 2001) in Norway it is quite common that all material less than 125 μm is referred to as fines. This definition is also used in this report.

1.3. Previous research and needs for further studies

It has recently been proven (Cepuritis 2011) that by utilizing different crushing techniques (VSI crushing and cone crushing) it is possible to alter the shape of particles smaller than 125 μm . Then a proper choice of crushing machinery in combination with a suitable classification technique (air sieving and/ or washing) would allow us to produce high quality manufactured fine aggregate. This would then include the designed amount of filler particles with optimum grading and good shape.

In order to proceed with the above mentioned, we would need to have a deep understanding of how different physical properties of fillers (PSD, specific surface, shape, surface texture, mineralogy, porosity etc.) influence rheological properties of fresh matrix and concrete.

The effect of very different mineral fillers on the rheology of the fresh cementitious materials (both concrete and matrix) has been studied by a wide variety of authors (Nehdi, Mindess and Aitcin 1997; Zhang and Han 2000; Ferraris, Obla and Hill 2001, Pedersen 2004, Cepuritis 2011, Cepuritis et. al. 2011). Most of the authors relate a large part of rheology differences due to different fillers to the particle size distribution (Nehdi, Mindess and Aitcin 1997, Zhang and Han 2000, Ferraris, Obla and Hill 2001, Pedersen 2004) or specific surface area (Bigas and Gillias 2002, Esping 2004, Westerholm 2006b) of the fines. Finer fillers normally tend to give higher flow resistance while coarser tend to improve the flowability.

None of the above mentioned authors has given a clear relationship between the filler properties and flow properties. One reason for that is of course the complexity of such a correlation, the other perhaps is that too many different parameters (particle shape, PSD, mineralogy etc.) have been studied at a time. However, another important factor affecting on such studies is simply the lack of knowledge regarding the determination of the physical properties of the fillers. For example, in order to properly proportion matrix or concrete mixes we would need to know the actual water absorption and SSD density of the filler particles. Up to now, no research has been carried out in order to investigate the porosity of fillers that is accessible to the water and thus affects the water absorption and rheology of the tested mixes. In addition, in the same way as for the well-known discussion regarding cement paste (water vapour adsorption BET specific surface vs. nitrogen adsorption BET specific surface) (Pane and Hansen 2004), different mineral filler particle characterization techniques (Blaine, BET, Laser Diffraction, Sedimentation, etc.) can easily give us results that differ ten-fold (Esping 2004 and 2008, Cepuritis 2011). So the question of the “real” mineral filler surface is still not resolved. While, on the other hand, as previously noted due to the large surface area, quantification of fillers is proven to be very essential for the ability to control rheological parameters of fresh concrete and matrix mixes.

1.4. Objectives

The principal objective and scope of this project work is to obtain a clearer knowledge about the porosity and water absorption properties of the most widely used Norwegian mineral fillers. This would hopefully allow us to discuss the results of specific surface measurements that have been performed with different methods in order to find the “real” surface area of the fine materials.

2. Materials and methods

2.1. Fillers used for the study

A total of 7 fillers (≤ 0.125 mm) from 5 different quarries in Norway have been used for the study (see Table 2-1). Six of the samples were produced by crushing (or grinding in case of limestone) of different types of rocks in various processes while one was produced originally from natural deposits. Description and flow sheets of the production process for most of those materials can be found in (Cepuritis 2011).

Table 2-1: Fillers used for the study

No	Quarry (type)	Deposit	Source material*	Producer
1.	Årdal (natural)	Granite/ gneiss glaciofluvial and moraine aggregate	0/8 mm	NorStone AS
2.	Årdal (crushed/ unwashed)			
3.	Årdal (crushed/ washed)			
4.	Tau	Mylonite rock	0/2 mm washed manufactured sand	Norsk Stein AS
5.	Jelsa	Gneissgranite rock		
6.	Hokksund	Gneiss rock	0/4 mm unwashed manufactured sand	Hokksund Pukkverk AS
7.	Brevik (limestone)	Limestone rock	Initial product	Norcem AS

* The source material that the fine aggregate (≤ 0.125 mm) was obtained from by dry sieving at laboratory conditions

Typical petrographic composition values (will be variable) provided by the Norwegian Geological Survey and from a separate report prepared at SINTEF, in case of fillers from Ådal, is given in Table 2-2. It must be noted that none of the data has been obtained exactly from the tested fillers and not even from the crushed aggregate that the fillers were sieved out of. Thus, it has a highly informative nature. To give impression of a general physical appearance and structure of the rocks mentioned in Table 2-2 some general illustrative pictures are given in Figures 2-1 to 2-2. Even though, the mineralogical composition given in Table 2-2 does not indicate the highest mica content for the Hokksund material, practical experience, i.e. visual

inspection of the coarse aggregate fraction from Hokksund has revealed comparably very high amounts of flaky mica particles. This is illustrated in Figure 2-3.

Table 2-2: Typical petrographic composition values (will be variable) of the tested fillers

No	Quarry/ type	Deposit	Rock type	Typical petrographic composition values provided by Norwegian Geological Survey*							
				Quartz	Feldspar	Pyroxen	Mica	Epidote	Chlorite	Calcite	Other minerals
				[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]
1	Årdal (natural)	Granite/ gneiss glaciofluvial and moraine aggregate	Igneous / metamorphic	Petrographic analysis with thin section/ rock-mineral composition according to SINTEF Building and Infr. test report No 25675 (2010) : Feldspathic rock/ feldspar particles 47%; granite 40%; dark rock 6%; quartzite, coarse grained/ quartz particles 5%; quartz rich rock 1%; mylonite/ cataclasite 1%							
2	Årdal (crushed/ unwashed)										
3	Årdal (crushed/ washed)										
4	Tau	Mylonite rock	Metamorphic	55	30	-	6	4	4	-	1
5	Jelsa	Gneissgranite rock	Igneous / metamorphic	30	40	-	18	5	-	2	5
6	Hokksund	Gneiss rock	Metamorphic	35	40	20	5	-	-	-	-
7	Brevik (limestone)	Limestone rock	Sedimentary	-	-	-	-	-	-	100	-

* <http://www.ngu.no>.



Fig. 2-1: Rock types of the fillers used for the study: *granite* (source: unknown)



(a)



(b)



(c)

Fig. 2-2: Rock types of the fillers used for the study: (a) *mylonite* (Bentley 2009); (b) *limestone* (Mineral Information Institute 2012); (c) *gneiss* (<http://geology.com/rocks/gneiss.shtml>)



Fig. 2-3: Flaky mica particles in oversize feed material 22/40 mm from Hokksund Pukkverk AS (Cepuritis 2011)

2.2. Filler characterization

All of the 7 fillers from Table 2-1 have been characterized by various methods. Filler particle size distribution was determined using Backman Coulter LS 230 laser diffraction device and Micrometrics SediGraph 5100, specific weight was measured by Micrometrics AccuPyc 1330 Pycnometer and specific surface tests were performed with a Micrometrics FlowSorb II 2300 nitrogen adsorption device by utilizing BET approach and a Blaine apparatus for the Blaine method. The measurements have been performed within the COIN project FA 2.3 “*High quality manufactured sand for concrete*” by SINTEF Materials and chemistry – Report No 11014 (2011).

Scanning electron microscopy (SEM) pictures of all the fillers have also been taken by the author after a standard training course given by the laboratory personnel.

In addition SSD particle density and water absorption of corresponding sand fractions (0/2 mm or 0/8 mm) containing the fillers (except of the limestone filler) was also determined by the “*pycnometer method for aggregate particles between 0.063 and 4 mm*” according to EN 1097-6 (2002). The water absorption and SSD density tests were carried out at Norstone AS aggregate laboratory in Årdal by an experienced operator.

In the following chapters a short description of the test methods used is given. Much of the information is taken from a report by Wigum (2010). Only very basic description and most important parameters of the test methods are discussed. Broader explanation of all the test methods can be found in the original report by Wigum (2010) or in the other provided references.

2.2.1. PSD and specific surface area using laser diffraction by Beckman Coulter LS 230 laser diffraction device

The Beckman Coulter LS 230 measures particle sizes from 40 nm to 2000 μm (0.04 – 2000 μm) by laser diffraction. It is based on the principle that particles scatter and diffract light at certain angles based on their size, shape, and optical properties. A 750 nm diode laser is used for analysis in the size range from 400 nm to 2 mm. The beam passes through filters as well as projection and Fourier lenses and is spatially recorded onto 126 photodiode detectors. The particle size, shape, and optical properties of the particles control the spatial variation of the diffracted beam. The calculations assume the scattering pattern is due to single scattering events by spherical particles. The advantages of this technique include ease of operation, large range of detectable particle sizes, and accuracy in the micron and submicron range. More information can be found in a report by Wigum (2010) and from the producer (<https://www.beckmancoulter.com>).

From the grading test results specific surface area has been calculated. Specific surface area was calculated following the suggestions by Erdem, Khayat and Yahia (2009). They proposed a way of calculating surface area of aggregate particles using sieve analysis and assuming that the particles are spherical in shape. An example calculation, including the assumed spherical particle diameter steps, is presented in Appendix A.

2.2.2. PSD and specific surface area using sedimentation by Micrometrics SediGraph 5100

The particle size distribution was first measured with the Coulter LS 230 and then the sample was sieved through a 63 μm sieve in order to analyse the fine particle grading again with the aid of the SediGraph. The results were then mathematically combined in order to obtain the full (0-125 μm) grading curve.

The Micrometrics SediGraph 5100 particle size analyser measures the sedimentation rates of particles in suspension and automatically presents this data as a cumulative mass % distribution in terms of the Stokesian or equivalent spherical diameter in micrometres (μm). The instrument determines, by means of a finely collimated beam of X-rays, the concentration of particles remaining at decreasing sedimentation depth

as a function of time. The instrument typically yields a particle diameter distribution over the range 50 to 0.18 μm . More information can be found in a report by Wigum (2010) and from the producer (<https://www.micrometrics.com>).

From the grading test results specific surface area has been calculated in the same way as described in the last paragraph of the previous chapter.

2.2.3. Specific surface area using permeametry (air permeability) by the Blaine apparatus

Blaine method is the most widely used for specific surface measurements in the cement industry. This simple method measures the time for a specific volume of air to flow through a known volume of compacted powder and together with the density of the substance, this is used to calculate the specific surface area of the sample. The main advantages of this technique are that it is simple and rapid. However, it is not very accurate and suffers from a number of weaknesses, e.g. variable particle shape and become extremely unreliable at surface areas greater than 500 m^2/kg (Potgieter and Strydom 1996 cited in Wigum 2010).

2.2.4. Specific surface using the BET (gas adsorption) method by Micrometrics FlowSorb II 2300 nitrogen adsorption device

In situation where very accurate actual particle surface measurements are required, one of the most common methods to measure surface area is the BET method (Brunauer, Emmett and Teller introduced this method through a publication in 1938). This method relies on a mathematical formula that describes the adsorption of a monolayer of a particular gas on the finely divided material to calculate its surface area. In this case, nitrogen (N_2) was used as the absorbed gas. Opposite to the other specific surface determination methods described above this is the only method that measures both the *internal* and the *external* surface area of a material. More information regarding the BET method and application of the approach (by utilizing different gasses) to various porous building materials and fine fillers can be found in selected references (Fagerlund 1968, Pane and Hansen 2004 and Esping 2008).

2.2.5. Specific weight by gas pycnometer Micrometrics AccuPyc 1330 Pycnometer

The Micrometrics AccuPyc 1330 Pycnometer works by measuring the amount of displaced gas (helium or He). The pressures observed upon filling the sample chamber and then discharging it into a second empty chamber allow computation of the sample solid phase volume. Gas molecules rapidly fill the tiniest pores of the sample; only the truly solid phase of the sample displaces the gas. This leads to that the Micrometrics AccuPyc 1330 Pycnometer determines density and volume by measuring the pressure change of helium in a calibrated volume. More information on

gas pycnometers can be found in a paper by Tamari (2004) or from the producer (<https://www.micrometrics.com>).

2.2.6. Micrographs by Hitachi S3400 SEM

A scanning electron microscope (SEM) is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern. Electron beams that are emitted by the microscope interact with the atoms of the sample producing signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity. The information can then be converted to an image (Reed 2005). For the current study a Hitachi S3400 instrument was used. More information on SEM imaging can be found in the book by Reed (2005) or from the equipment producer (<http://www.hitachi-hita.com>).

The preparation of the filler samples for the SEM imaging was as follows:

- First a sample holder was cleaned carefully with alcohol;
- Then a piece of double sided adhesive carbon tape was applied to the sample holder;
- A sample of the filler was sprinkled carefully on the tape, the not adhered leftover filler was blown away;
- Sample was placed in a coater and a micron range thin layer of gold was applied to the sample in order to make it conduct;
- Sample was placed in Hitachi S3400 and the SEM micrographs were then taken.

2.2.7. SSD density and water absorption according to EN 1097-6

So far there is no CEN or widely known other standard/ test method that is used in order to determine the water absorption of mineral fillers. As a result values obtained from the sand (containing the 0.063-0.125 mm fraction of the particular filler) water absorption tests are usually used. The EN standard that is used for this purpose is EN 1097-6 (2002). The standard hasn't been a success from the beginning since a lot of corrigendum errors in some of the fundamental equations could be found in the first version of the standard (2000). Even though they have been corrected now, one particular method in this standard still remains among one of the most miss-trusted within those that are used in the concrete and aggregate production industry (personal communication with Børge Wigum (NorStone AS), Torstein Riskedal (NorStone AS), Sverre Smeplass (Skanska AS) and many more).

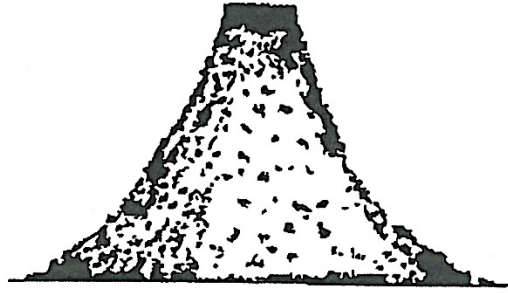
The EN 1097-6 actually contains three different methods that describe how to determine three types of densities (apparent particle density, particle density on an oven-dried basis and particle density on a SSD basis, see glossary of notation and terms) for three different particle size ranges:

- Wire-basket method for aggregate particles between 31.5 mm and 63 mm;
- Pycnometer method for aggregate particles between 4 mm and 31.5 mm;
- Pycnometer method for aggregates between 0.063 mm and 4 mm.

In particular, the determination of the SSD density for the sand fraction (between 0.063 mm and 4 mm) has been questioned a lot. The problem lies in the fact that it is hard to determine when the SSD condition for this fraction has been reached. While for the coarser particles it can be easily observed visually, a special method has to be used for the particles between 0.063 and 4 mm. According to this method, in order to check, if after some drying, the material has reached the SSD state, the 0.063 mm to 4 mm fraction is loosely filled in a truncated metal cone ($d_{\max}=90$ mm; $d_{\min}=40$ mm; height=75 mm), tamped 25 times with a metal tamper, the cone is lifted and the nature of the sand fraction shape is then observed. The truncated test cone and metal tamper are illustrated in Figure 2-4. Then, according to the standard, if the cone has not collapsed the aggregate is still in the moist state (Figure 2-5 (a) and (b)), and the drying should be continued. However, if a situation as in Figure 2-5 (c) can be observed the aggregate has reached the SSD condition and the mass of the sample at this state can be used in order to calculate the SSD density. Figure 2-5 is taken from the Annex F of the EN 1097-6 (2002).



Fig. 2-4: Truncated steel cone and tamper that are used according to EN 1097-6 (2002) in order to assess if a sand sample has reached the SSD state (UTEST 2012)



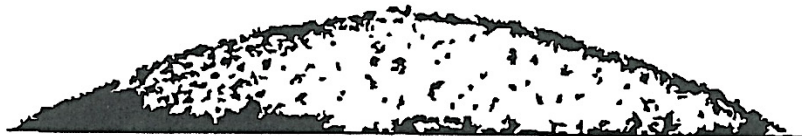
a) Aggregate moist; almost retains complete shape of metal mould



b) Aggregate slightly moist; appreciable slump observed



c) Aggregate saturated surface-dry; almost complete collapse but definite peak still visible and slopes are angular



d) Aggregate nearly oven dry; no distinct peak, surface outline close to being curvilinear

Fig. 2-5: Guidance on the saturated and surface dry condition of fine aggregate from the Annex F of EN 1097-6 (2002)

First of all, the described test method of determining the SSD state of the sand particles (0.063 mm to 4 mm) can be criticized simply by the fact that it is very operator dependant. Because with real aggregates choosing between (b) to (d) states in Figure 2-5 is most likely a very subjective decision.

Also the sand fraction moisture states described in Figure 2-5 have been subjected to doubts. In their suggestions to the CEN/ TC 154/SC (the Technical Committee “Aggregates” within CEN) the Norwegian Concrete and Aggregate Laboratory (NBTL 2003) states: “According to clause 9.3 water saturated and surface dry state is

obtained by successive drying of the test portion and repeating of the cone test until “the collapse situation occurs at moulds” removal. Annex F gives guidance on how saturated and surface dry condition looks like. However, figure c which should illustrate the saturated and surface dry condition is not correct because this figure shows the oven dry condition (see enclosed documentation). Moreover, figure d illustrating nearly oven dry condition can not be obtained on any European aggregate types. The curvilinear form is not possible to obtain by the procedure given in the standard. The most correct illustration of saturated and surface dry state is figure b which should be used, but with another text.” Then they suggest the following amendment to the Annex F (Figure 2-5) of the standard:

- Figure a) no changes;
- Figure b) replace old text with “Aggregate saturated surface dry; appreciable slump observed”;
- Figure c) replace old text with “Aggregate nearly oven dry; complete collapse of cone and slopes are angular”;
- Figure d) delete/ remove figure d.

NBTL has carried out several tests to determine the SSD condition of aggregates (NBTL 2003, 2004). Their experience is illustrated in Figures 2-6 to 2-8, where the successive drying and cone test procedure of Norwegian standard sand (0-4 mm) is shown. Figure 2-6 shows wet condition with intact metal moulds shape, Figure 2-7 is the situation when the cone has collapsed accurately, and should be the SSD condition according to the normative text of EN 1097-6 (2002) while Figure 2-8 is the shape of oven dried aggregates. The same shape of the oven dried aggregates (Figure 2-8) has also been obtained by testing Danish natural sand dried for 24 hours at 105 °C (NBTL 2003).



Fig. 2-6: Aggregate moist; complete shape of the metal moulds; the height is reduced for about 2-3 cm due to tamping with the steel tamper (NBTL 2003)



Fig. 2-7: Aggregate SSD; the cone shape has just collapsed; the height is reduced for about 0.5-1 cm due to tamping with the steel tamper (NBTL 2003)



Fig. 2-8: Aggregate dry; the cone shape has totally collapsed and slopes are angular – a “new cone” with a clear peak has occurred; the aggregate has been oven dried for 24 hours at 105 °C (NBTL 2003)

If the differences between the Annex F of EN 1097-6 (2002) and the observations with real Norwegian and Danish aggregates are true, this would mean that significantly lower water absorption values are measured. Figure 2-9 (NBTL 2003, 2004) then shows water absorption values of Norwegian fine aggregates assessed according to EN 1097-6 (2002) and as suggested by NBTL – when the cone moulds have accurately collapsed on x-axis (the shape shown in Figure 2-7) and according to Figure (c) of Annex F in EN-1097 (2002). As expected, the water absorption value obtained by Figure (c) in the Annex F is generally about half the value compared to using the accurately collapsed cone shape shown in Figure 2-7.

Moreover, according to NBTL (2003, 2004) testing fine and coarse aggregate from the same deposit according to EN-1097 (2002) then gave significantly lower water absorption for the fine one. They claim that (NBTL 2003): “Because the surface area of fine aggregate is significant higher compared to coarser aggregate the water absorption of fine aggregate should not be lower (but higher) than the water absorption of coarse aggregate which is the case by using figure c in annex F.” Some other opinions, however, co-exist regarding the comparison of water absorption

values obtained on different particles sizes. It has been proposed (personal communication with Bård Pedersen, Norwegian Public Roads Directorate (previously NorStone AS)) that when the aggregate particle size is considerably reduced, like from the maximum grain size of coarse aggregate (usually approx. 22 mm) to the sand and filler fractions, the porosity of the aggregate should also be reduced. This is because when a particle is crushed in the crusher cavity the fracture surface would follow the weakest points, i.e. the biggest pores in the particle. Thus, the total porosity of a small particle should be lower than for a bigger one. However, even if this phenomenon is measurable, the specific surface of the sand would anyway be considerably higher than that of the coarse particles. Meaning that there would still be much more surface that is open to water penetration than for the same volume of coarse aggregate. So it is actually rather hard to support one or another opinion.

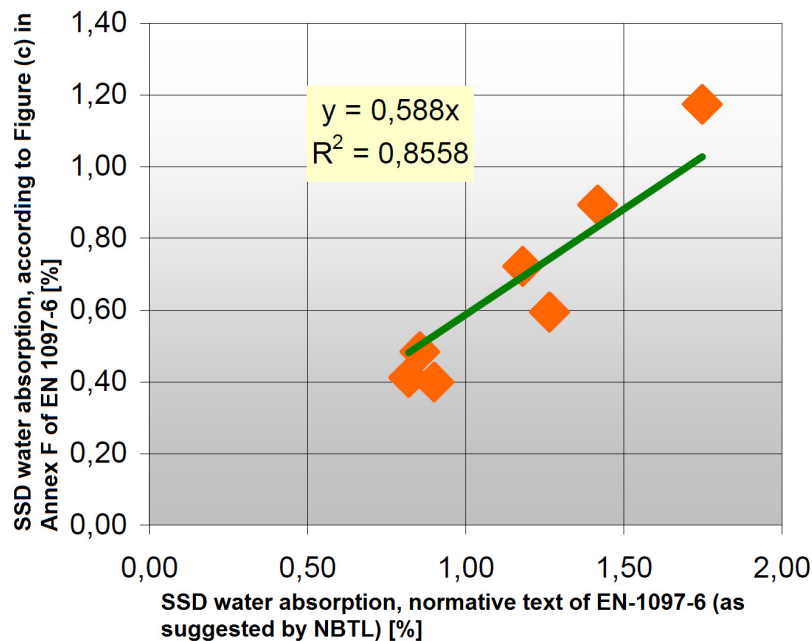


Fig. 2-9: SSD water absorption obtained according to the normative text of EN 1097-6, i.e. “collapse has accurately occurred” (x-axis) and by Figure (c) in the Annex F of EN 1097-6 (y-axis)

Some other data regarding the water absorption measurements of different particle fraction sizes is presented in Table 2-3. It can be seen from there that even if the Annex F of EN 1097-6 (2002) is used in order to determine the SSD water absorption of the aggregate from the same source, the values for the fine fraction can still be higher than those obtained for coarser ones. This is in fact measured on particles from the same deposit as the Norwegian standard sand shown on Figures 2-6 to 2-8. Since this is opposite to what has been reported by NBTL (2003, 2004) the source of the difference might just simply be the repeatability or the operator dependence of the cone test method itself.

Table 2-3: SSD water absorption and density values of different fractions from the same source measured according to EN 1097-6 (2002)

Material deposit	Tested fraction	NS-EN 1097-6:2002	
		Saturated surface-dried (SSD) density	Water absorption
	[mm]	[kg/m ³]	[%]
Årdal (NorStone AS)	0/8	2670	0.6
	2/8	2670	0.6
	8/16	2690	0.4

3. Results and discussion

3.1. Filler characterization results

Results of the filler PSD analysis are illustrated on Figures 3-1 and 3-2. All the other determined physical properties of the fines are presented in Table 3-1.

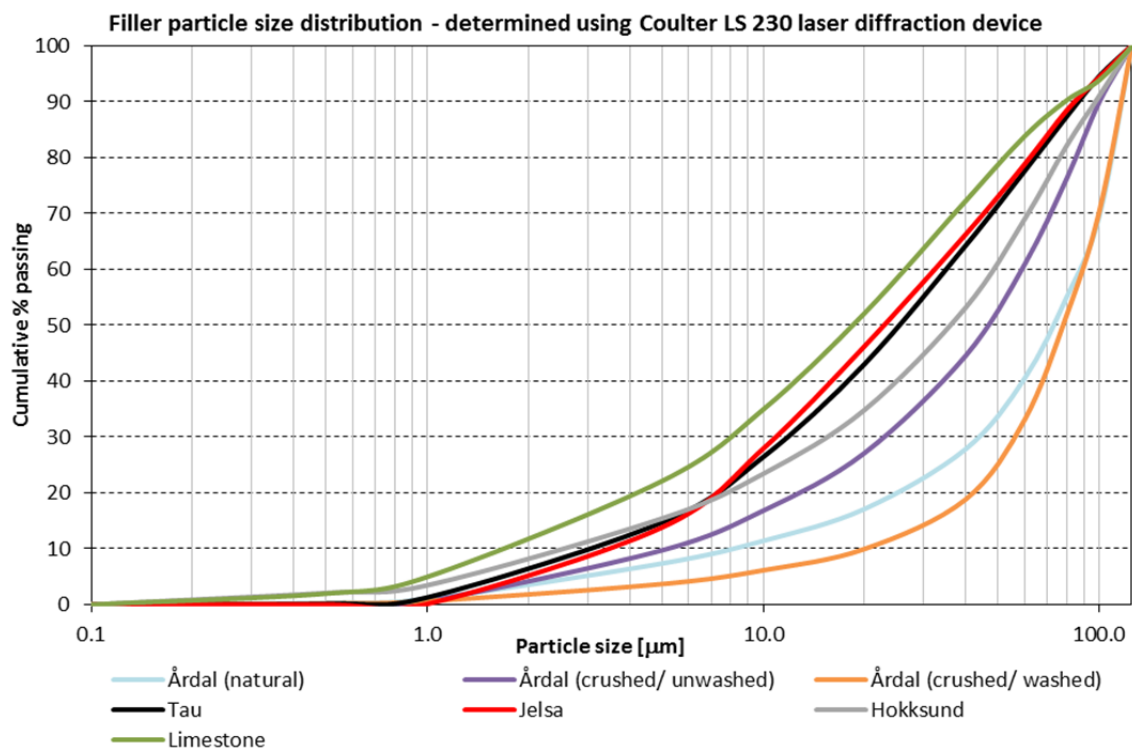


Fig. 3-1: Particle size distribution of the fillers used for the study measured with Backman Coulter LS 230 laser diffraction device

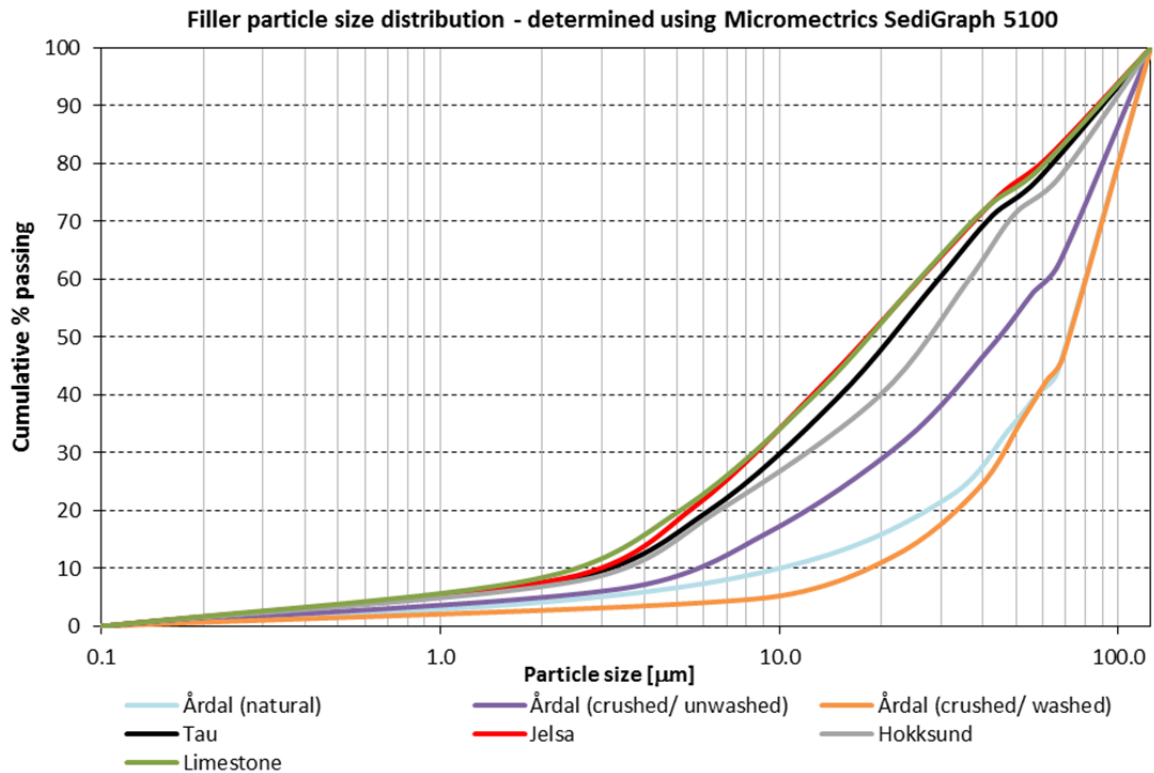


Fig. 3-2: Particle size distribution of the fillers used for the study measured with Micromeritics SediGraph 5100

Table 3-1: Determined specific weight, SSD density, water absorption and specific surface measurements/ calculations of the fillers

No	Quarry/ type	Specific weight of fillers	Saturated surface-dried (SSD) density	Water absorption	Tested fraction	Specific surface area					
		AccuPyc 1330 helium pycnometer	NS-EN 1097-6:2002			Blaine	BET	LS Particle Size Analyzer*	Micromeritics SediGraph 5100*	Ratio of BET/ LS PSA	Ratio of BET/ SediGraph
		[g/cm ³]	[g/cm ³]	[%]	[mm]	[m ² /kg]				[1]	[1]
1	Årdal (natural)	2.710	2.670	0.3	0/8	131.1	2140	144	357	15	6
2	Årdal (crushed/unwashed)	2.720	2.670	0.3	0/8	165.8	1600	153	364	10	4
3	Årdal (crushed/washed)	2.730	2.670	0.3	0/8	64.5	870	93	209	9	4
4	Tau	2.790	2.750	0.3	0/2	229.5	1750	225	283	8	6
5	Jelsa	2.810	2.750	0.3	0/2	309.8	1520	192	302	8	5
6	Hokksund	2.860	2.860	0.2	0/2	225.5	3760	361	421	10	9
7	Brevik (limestone)	2.740	-	-	-	413.3	1170	470	584	2	2

* The surface area of the aggregate was calculated according to Erdem, Khayat and Yahia (2009) using sieve curves and assuming that particles are spherical in shape.

3.2. SEM micrographs

Some selected micrographs of all the fillers are given in Tables 3-2, 3-3 and 3-4. More SEM images of all the fillers and in addition also of the most commonly used cement in Norway, i.e. Standard Fly-ash (STD FA; CEM II/A-V) cement from Norcem AS, can be found in Appendix B.

Table 3-2: SEM micrographs of the tested fillers

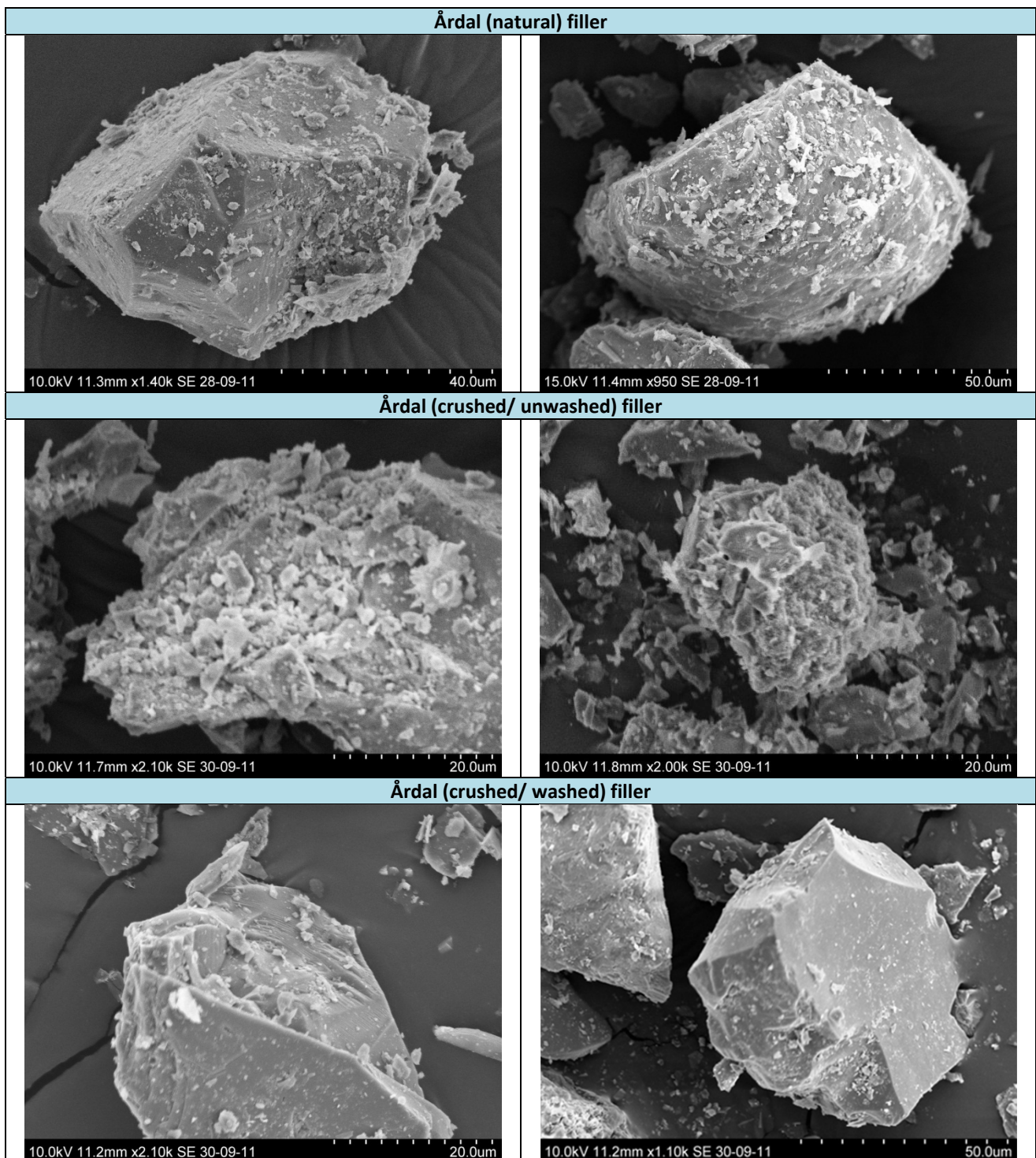


Table 3-3: SEM micrographs of the tested fillers

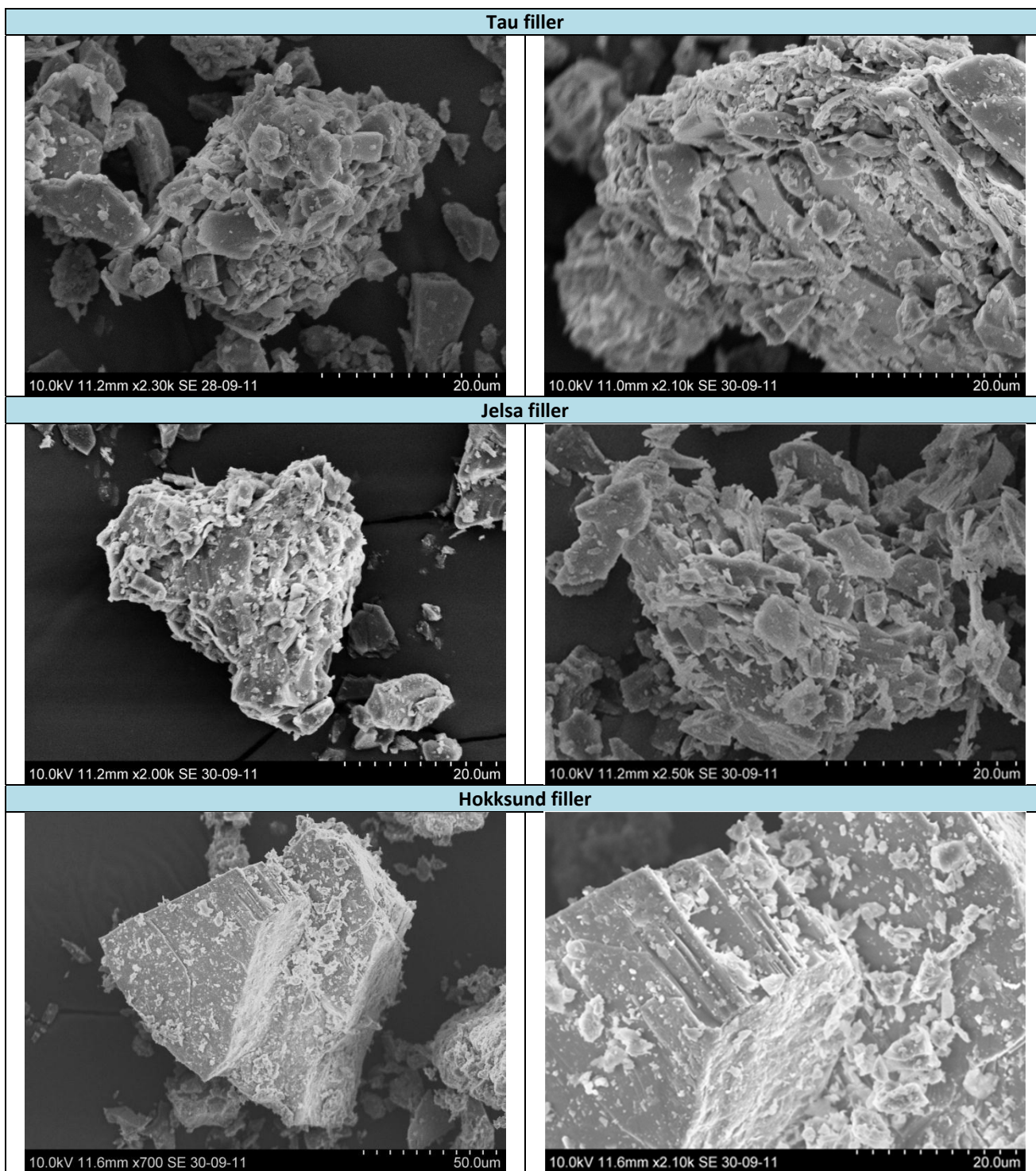
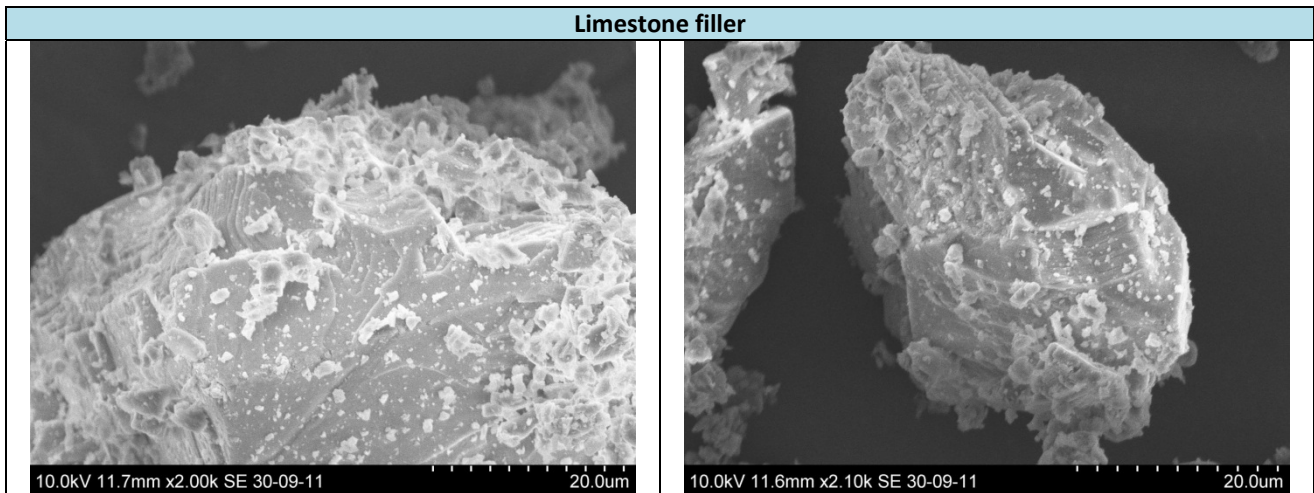


Table 3-4: SEM micrographs of the Limestone filler



3.3. Effect of PSD on SSD water absorption of fine aggregate

The theoretical assumptions (if any) regarding the fact that the 0/0.063 mm particles are not included in the SSD measurements of the sand fraction according to EN 1097-6 (2002) are not known. Some practical experience of the author with draying of fine mineral materials of a wide grading and high very fine (< 0.063 mm) particle content suggests that they would allow such a material sticking together (in order to resemble a not collapsed cone as in Figure 2-5) at much dryer conditions, i.e. relative humidity (RH) within the aggregate sample. Perhaps, this can be explained by the bridging effect (Figure 3-3) due to capillary condensation, and then theoretically described by the Kelvin-LaPlace equation (Hunter 2001):

$$-\frac{RT}{v^l} \cdot \ln RH = 2 \cdot \frac{\sigma^{lg}}{r} \quad (3-1)$$

where

R = ideal gas constant; R = 8.3144621(75) J/(mol·K);

T = temperature, K;

v^l = molar volume of liquid (in this case water); $v^l = 18.05 \cdot 10^{-6} \text{ m}^3/\text{mol}$ at 20 °C;

RH = relative humidity above the capillary meniscus;

σ^{lg} = surface tension of liquid (in this case water)-gas interface; $\sigma^{lg} = 0.073 \text{ N/m}$;

r = radius of the capillary meniscus that is formed between two fine particles.

If we now assume that the contact angle α between the capillary condensed water and the fine particle surface is zero, i.e. half of the particle size R is thus equal to the radius of the meniscus ($R = r \cdot \cos\alpha = r \cdot \cos 0 = r$), then by using Equation 3-1 and solving for r we get:

$$R = r = -\frac{2 \cdot \sigma^{lg} \cdot v^l}{RT} \cdot \frac{1}{\ln RH} \quad (3-2)$$

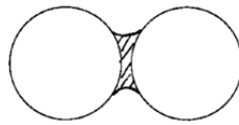


Fig. 3-3: Figure demonstrating the bridging between two spheres due to capillary condensation (Hunter 2001)

The temperature and RH in the bulk of the sand particles after drying and putting the sample in the steel cone (Figure 2-4) is not known. Thus the calculation was performed at two temperatures (20 °C and 40 °C) and variable RH. The results are presented in Figure 3-4.

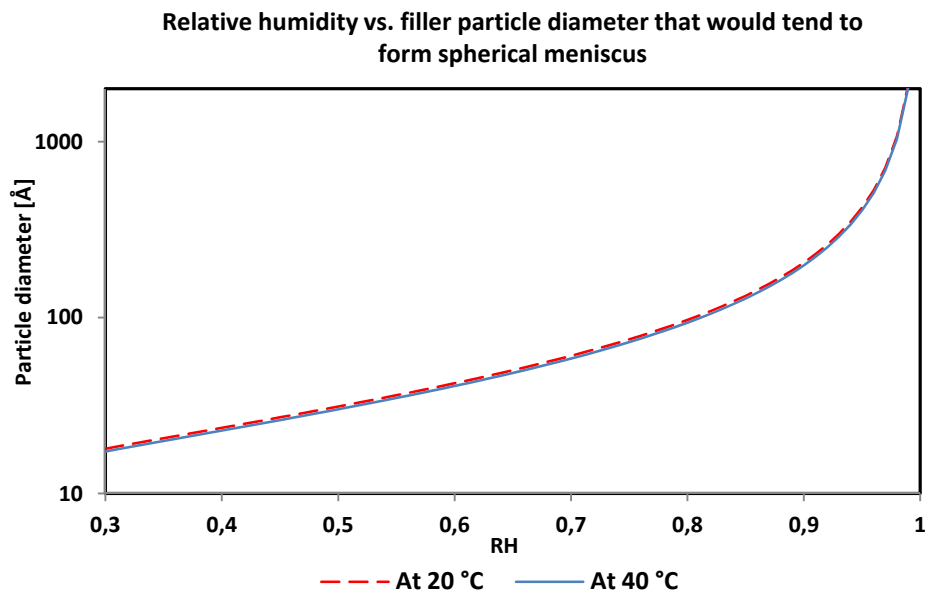


Fig. 3-4: Relative humidity vs. filler particle diameter that would tend to form spherical meniscus

It can be seen from Figure 3-4 that only filler particles in the size range 0.002-0.2 μm would tend to stick together due to the capillary condensation at different RH. The temperature actually seems to have negligible effect on this phenomenon. From the filler PSD determination results presented on Figures 3-1 and 3-2 it is clear that only up to 2% of the filler grains are smaller than 0.5 μm, thus most likely only around 1% falls in the 0.2 μm range. This means that the PSD of the filler will have no noticeable effect on the water absorption and SSD density measurements of the corresponding sand. It also means that capillary condensation can't be used to theoretically justify why the 0/0.063 mm fraction is removed from the sample when determining the SSD density and water absorption of the sand particles according to EN 1097-6 (2002).

3.4. Density and water absorption of the fillers

There have been discussions (see at the end of chapter 2.2.7) that SSD density and water absorption of the fillers should be both reduced and increased when compared to coarse particles from the same source. As one of the reasons for lower value reduced porosity has been suggested. As it can be seen from the SEM micrographs presented in Tables 3-2 to 3-4, it is doubtful if this is always the case. It can be seen that samples from Tau and Jelsa display a lot of (at least apparent) open porosity. The question of interest is then: if the possible mineral grain boundaries forming the porosity are also accessible to water and can in this way affect the water absorption of the filler particles and thus the rheological properties of matrix and concrete?

To answer this question let's first look at the different measured densities in Table 3-1. As expected the SSD density is lower than the values of the specific weight measured by the helium pycnometry. This is coherent since the water held in the filler particle pores has at least two times lower density than the solid particles even if it is in the surface adsorbed state. However, the SSD can't be directly compared to the values obtained by the gas pycnometer. In order to correctly recalculate the SSD density value into the corresponding apparent (dry) density one should have an idea of the open pore structure of the samples. Two in principle different cases can be discussed.

If a pore model as presented in Figure 3-5 is used, we then assume that the filler open pore sizes (mineral grain boundaries, cracks etc.) are high enough, in order to have most of the absorbed water in a bulk state, i.e. having a density of approx. 1 g/cm³ at 20 °C.

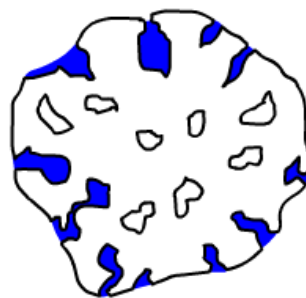


Fig. 3-5: SSD condition of a filler particle – water in open pores mostly in a bulk state (Pavement Interactive 2007)

The apparent (dry) density can then be calculated from the SSD density and water absorption measurement results by using the following equation:

$$\rho_{\text{dry}} = \frac{\rho_{\text{SSD}} - \rho_{\text{SSD}} \cdot \text{WA} / 100}{1 - \text{WA} / 100} \quad (3-3)$$

where

ρ_{dry}, ρ_{SSD} = apparent (dry) and SSD density of the particles, g/cm³;

WA – water absorption of the particles, %.

On the other hand, water in the pores of the fine particles can be in the surface adsorbed state in case if the pore sizes are very small. Then a model like in Figure 3-6 must be used.

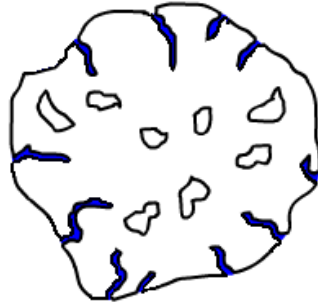


Fig. 3-6: SSD condition of a filler particle – water in open pores mostly in a surface adsorbed state (modified from Pavement Interactive 2007)

Then, in order to calculate the apparent (dry) density from the SSD density and water absorption measurements, we must make some assumptions regarding the density of the water in the surface adsorbed state. Starting from the bulk liquid water in the adsorbed state may vary in any number of ways. Two models are usually mentioned: solid and dimensional fluid. It must be noted that solid model does not mean that the water molecules are rigidly fixed into a well ordered lattice but only that there is more “order” than in normal liquid water (Martin 1962). No data of adsorbed water density values is available for the mineral fillers used in this study. However, some limited previous research has been carried out for clay minerals. Martin (1962) has used the data by DeWit and Arens (1950) to calculate the density of the surface adsorbed water for montmorillonite, kaolinite and illite. The results of the calculation are presented in Table 3-5. As it can be seen from the results, the density of the adsorbed water varies considerably with the clay mineral type and water content, i.e. relative humidity. Some average value from all the three mineral types at RH=75 % is then 1.14 g/cm³.

If we then assume the mineral filler pore size model as in Figure 3-6 and the density of the adsorbed water in the order of 1.14 g/cm³ the apparent (dry) density can be calculated according to the following equation:

$$\rho_{dry} = \frac{\rho_{SSD} - \rho_{SSD} \cdot WA/100}{1 - (WA/100)/\rho_{AW}} \quad (3-4)$$

where

ρ_{dry}, ρ_{SSD} = apparent (dry) and SSD density of the particles, g/cm³;
 WA = water absorption of the particles, %;
 ρ_{AW} = adsorbed water density = 1.14 g/cm³.

Table 3-5: Adsorbed water density on clay (Martin 1962)

Mineral	RH	Water content	Density of adsorbed water
	[%]	[mg/g]	[g/cm ³]
Montmorillonite	25	116	1.41
	50	166	1.37
	75	284	1.32
	100	460	1.16
	-	2440	1.02
	-	3010	1.02
Kaolinite	25	4	1.68
	50	8	1.12
	75	9	1.03
	100	65	0.99
Illite	25	30	1.36
	50	49	1.17
	75	69	1.08
	100	188	1.04
	-	1038	1

Results of the apparent (dry) density calculations according to Equations 3-1 and 3-2 are presented in Table 3-6.

Table 3-6: Results of apparent (dry) density calculations of the fillers

No	Quarry/ type	Specific weight of fillers	Saturated surface-dried (SSD) density	Water absorption	Apparent (dry) density*	Apparent (dry) density**	Tested fraction
		AccuPyc 1330 helium pycnometer	NS-EN 1097-6:2002				
		[g/cm ³]	[g/cm ³]	[%]	[g/cm ³]	[g/cm ³]	[mm]
1	Årdal (natural)	2.710	2.670	0.3	2.683	2.681	0/8
2	Årdal (crushed/ unwashed)	2.720	2.670	0.3	2.683	2.681	0/8
3	Årdal (crushed/ washed)	2.730	2.670	0.3	2.683	2.681	0/8
4	Tau	2.790	2.750	0.3	2.765	2.762	0/2
5	Jelsa	2.810	2.750	0.3	2.765	2.762	0/2
6	Hokksund	2.860	2.860	0.2	2.871	2.869	0/2
7	Brevik (limestone)	2.740	-	-	-	-	-

* Assuming that the water is in bulk liquid state (density 1 g/cm³).

** Assuming that the water is in adsorbed state (density 1.14 g/cm³).

As it can be seen from Table 3-6 the differences between the calculated apparent (dry) densities depending on the pore model used is in the order of 2-3 kg/m³. However, we must also remember the rather rough assumptions used to determine the density of the adsorbed water. In order to further justify the use of one or another pore model, let's calculate the total porosity of the samples (from the water absorption values) and divide the porosity by the inner specific surface of the same sample. The results of the division would give use an average thickness of the absorbed water layer, assuming that the pore system of the mineral particles consists of monosized slots.

Specific surface measurement results by the BET method are two to fifteen times higher than then corresponding results obtained by laser diffraction or SediGraph results (Table 3-1). This is because opposite to the other specific surface determination methods used BET is the only method that measures the “true surface area” of the fillers, i.e. both the internal and the external surface area of a material (see chapters 2.2.1 to 2.2.4). Thus, in order to obtain the inner surface area of the pore system, we would need to subtract the specific surface area determined by the SediGraph or laser diffraction from the BET “true surface are”. Since laser diffraction and SediGraph values are of approximately the same magnitude (see Table 3-1) the latter were chosen, since there are fewer variables when measurement based on the Stoke's law are performed. Thus this method is believed to be somewhat more accurate for the case. The results obtained by the Blaine method were not considered since it is believed to be more indirect than laser diffraction and SediGraph methods. Results of the average water thickness calculations in the monosized slot pores of the filler particles are presented in the Table 3-7. Bulk water density, i.e. 1 g/cm³ has been used for the calculations.

As it can be seen from the data presented in Table 3-7 the average thickness of water in the pores of the filler particles is probably very small, i.e. from 6 to 45 Å (23 on average) or only 0.6 to 4.5 nm. The mean van der Waals diameter of water (H₂O) molecule has been suggested to be in the order of 2.82 Å (Franks 2000). This means that on average less than 5 water molecule layers will be adsorbed on the surfaces of the monosized slit pores. Thus, water must be in the adsorbed state with the density higher than 1 g/cm³. It must be noted here that the van der Waals diameter of the nitrogen (N₂) molecule has been reported to be slightly larger – in the order 3.14 Å (Murphy 2010). In addition water molecule also has larger affinity forces (attraction) due to its two-polar covalent bonds (Figure 3-7). Thus the nitrogen molecules, that were used to determine the BET specific surface, might not be able to access pores of the same fineness as the water molecules. No data on specific surface area determination of mineral fillers – both with nitrogen and water vapour is available. However, due to the differences mentioned above BET surface area of the hydrated cement paste found from nitrogen isotherms are always smaller than those obtained by water isotherms (Pane and Hansen 2004). Some authors have thus suggested

(Esping 2008) that using water (moisture) in order to determine the specific surface of mineral fillers is probably closer to real conditions in concrete production than using nitrogen gas. In the light of the discussions regarding the pore structure of the fillers, using water vapour molecules instead of nitrogen would most likely mean higher BET specific surface values. Thus the average water layer thickness in Table 3-7 would be even lower, and would still approve that water in the pores of the mineral fillers is mainly held in the surface adsorbed state.

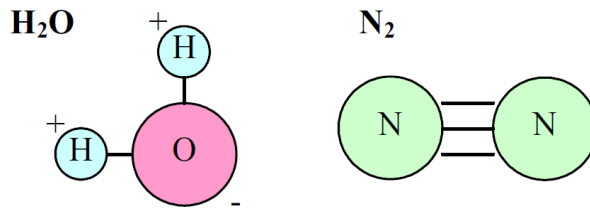


Fig. 3-7: The polar water (H₂O) molecule versus the non-polar nitrogen (N₂) (Esping 2008)

Table 3-7: Results of the average water layer thickness calculations if a monosized slot pore system is assumed

No	Quarry/ type	Specific weight of fillers	Saturated surface-dried (SSD) density	Water abs.	Volume of water in pores	Tested fraction	Average water layer thickness in monosized slots	Specific surface area			
		AccuPyc 1330 helium pycnometer	NS-EN 1097-6:2002					BET	Micrometrics SediGraph 5100*	Ratio of BET/ SediGraph	Inner surface
		[g/cm ³]	[g/cm ³]	[%]	[10 ⁶ · m ³ /kg]	[mm]		[Å]	[m ² /kg]	[1]	[m ² /kg]
1	Årdal (natural)	2.710	2.670	0.3	2.99	0/8	17	2140	357	6	1783
2	Årdal (crushed/ unwashed)	2.720	2.670	0.3	2.99	0/8	24	1600	364	4	1236
3	Årdal (crushed/ washed)	2.730	2.670	0.3	2.99	0/8	45	870	209	4	661
4	Tau	2.790	2.750	0.3	2.99	0/2	20	1750	283	6	1467
5	Jelsa	2.810	2.750	0.3	2.99	0/2	25	1520	302	5	1218
6	Hokksund	2.860	2.860	0.2	1.99	0/2	6	3760	421	9	3339
7	Brevik (limestone)	2.740	-	-	-	-	-	1170	584	2	586

* The surface area of the aggregate was calculated according to Erdem, Khayat and Yahia (2009) using sieve curves and assuming that particles are spherical in shape.

3.5. Specific surface of the fillers

It is clear from data in Table 3-1 that specific surface measurement results by the BET method are two to fifteen times higher than then corresponding results obtained by laser diffraction or SediGraph results (Table 3-1). As explained in the previous chapter, the theoretical explanation of this is because, opposite to the other specific

surface determination methods used, BET is the only method that measures the “true surface area” of the fillers, i.e. both the internal and the external surface area of a material (see chapters 2.2.1 to 2.2.4). Following this assumption the inner surface of the finis has been calculated (see chapter 3.4 and Table 3-7).

According to Table 3-7 the limestone filler has the lower inner surface while the Hokksund filler has the highest. If we not look at the SEM micrographs of all the fillers in Tables 3-2 to 3-4, we would probably agree that limestone filler has a smooth surface (no visible mineral grain boundaries, cracks, etc.), while Tau and Jelsa have rather rough, spongy surfaces with visible porosity. It must also be noted, that in contrast to all the other fillers, limestone fines are obtained from a sedimentary deposit while the others come from igneous or metamorphic rocks (see Table 2-2). However, when looking at the Hokksund filler particles there are no indications of a comparably very high inner surface. The same observation is true for the natural filler from Årdal (see Table 3-2) which has the smoothest surface while on the other hand – it has the second largest inner surface (see Table 3-7).

So how do we explain the difference between the observations and the measured values? One of the explanations might lie in the combination of measurement techniques used and mineralogical composition, at least for the Hokksund filler. The material from Hokksund is known to incorporate considerable amount of very flaky mica particles when compared to the other samples (Figure 2-3). While this does not cause any errors when using the BET method, it would affect both the laser diffraction and SediGraph results. Meaning that a very flaky particle would be registered as much coarser one and thus reduce the total specific surface area of a sample. Fines, incorporating a lot of flaky (such as mica) particles, would in the result seem to have higher inner surface that is defined as the difference between BET and laser diffraction or SediGraph test results (Table 3-7).

3.6. Precision of the apparent (dry) density test methods

As it can be seen from data presented in Table 3-6 the specific weight of the mineral fines determined by the gas pycnometer and the apparent (dry) density calculated from the SSD density measurements are different. The values obtained by helium pycnometry indicate consistently higher values (in the order of 29 – 49 kg/m³).

There are several sources of errors on sample-volume estimation in gas pycnometry (Tamari 2004). In particular, one of them is non-ideal-gas behaviour (Tamari 2004). To this respect helium has been recommended as the best gas to be used (Schumb and Rittner 1943, Franklin 1949, Biolders et. al. 1990, Huang et. al. 1995 and Marinder 1996). It has been demonstrated (Neimark and Ravikovitch 1997, Talu and Myers 2001) that helium can reasonably be considered as an ideal and non-adsorbing gas at

room temperature (300 K) and low pressure (<0.5 MPa). Even though, the actual volume of samples with large specific surface (such as mineral fillers) can be significantly overestimated. Because of their finite size, the gas molecules can't cover the surface of the sample perfectly (Ayrat et. al. 1992, Talu and Myers 2001). Due to this, the actual sample volume tends to be overestimated by gas pycnometry – as well as by any other pycnometric method (such as liquid water pycnometry). It has been estimated that an optimally designed gas pycnometer can determine the volume of solid particles with relative standard uncertainty $\leq 0.2\%$ (Tamari 2001). In case of average particle density of 2700 kg/m^3 this would mean an error of $\pm 5.4 \text{ kg/m}^3$. That is much less than the differences between gas and liquid water pycnometry results observed in the Table 3-6.

On the other hand, the precision of the much discussed (see chapter 2.2.5) SSD density measurements according to EN 1097-6 is lower. According to the Annex E of the EN 1097-6 (2002) the repeatability of the fine aggregate SSD density measurements with water pycnometry has been reported in the order of $8\text{-}17 \text{ kg/m}^3$ while the reproducibility is in the order of $12\text{-}16 \text{ kg/m}^3$. Those results are based on cross testing experiments carried out by 19 laboratories in 1998. Of course, in case of this method it is not possible to strictly identify the sources of all the errors since some of them are of a subjective kind. What this means is that it is not possible to determine a relative standard uncertainty value as for the gas pycnometer.

If we now compare the values of gas and water pycnometry reported in Table 3-6 we see that the differences between them are still higher than the precision (see above) of the used test methods. However, this difference can't be explained by different molecule sizes of water and helium (as for specific surface obtained by water vapour and nitrogen, see chapter 3.4). Helium (He) is a monoatomic gas with van der Waals diameter of the molecule being 2.11 \AA (Bondi 1964). This is less than 2.82 \AA that has been reported for water (Franks 2000). Thus, theoretically smaller molecules should give higher volume of the porous mineral filler particles and by that also lower density values of helium pycnoemetry, which is not the case. It must of course be noted that the molecule structure of water (H_2O) and helium (He) is very different – the same as when comparing water and nitrogen molecules (Figure 3-7).

4. Conclusions/ final remarks

The PSD of the tested mineral fillers should not have a noticeable effect on the water absorption and SSD density measurements of the corresponding sand aggregate determined according to EN 1097-6. This is because more than 99% of the filler particles are larger than those that would tend to bridge together due to capillary condensation.

The inner porosity of the tested fillers is extremely fine. If a monosized slit is used in order to model the pore structure, on average less than 5 water molecule layers will be adsorbed on the surfaces of the monosized slit pores. Thus, water in the filler particle pore system must be in the surface adsorbed state with the density higher than 1 g/cm³.

Due to the very low total porosity (in volume %) of the samples, variations in the density of the absorbed water (from 1 to 1.14 g/cm³) could only affect the density measurement results to an extent that is much lower than the precision of the test methods used. Thus, this phenomenon can't be measured by the methods described in EN 1097-6 (2002).

As it would usually be assumed, mineral particle apparent (dry) density measurements in a gas pycnometer is not necessary much more accurate than by the liquid pycnometry methods described in EN 1097-6 (2002). It has been estimated that an optimally designed gas pycnometer can determine the volume of solid particles with relative standard uncertainty $\leq 0.2\%$ (Tamari 2001). In case of average particle density of 2700 kg/m³ this would mean an error of ± 5.4 kg/m³. According to the Annex E of the EN 1097-6 (2002) the repeatability of the fine aggregate SSD density measurements with water pycnometry has been reported (based on results from 19 laboratories) in the order of 8-17 kg/m³ while the reproducibility is in the order of 12-16 kg/m³. Of course, in case of this method it is not possible to strictly identify the sources of all the errors since some of them are of a subjective kind. What this means

is that it is not possible to determine a relative standard uncertainty value as for the gas pycnometer.

Gas and water pycnometry density measurements gave differences that are higher than the precision of the used test methods. Since no particular reasons for that could be found, it can be suggested to use some simple method to check if the measured water absorption values of the fillers are correct. The fillers should be dried at temperature of 105 °C and then placed over a water bed in a container to reach 100% RH. As proven, no capillary effects should take place during this process. When the equilibrium is met, the filler should be dried again at 105 °C and the water absorption values calculated. Those values can then be compared to the ones obtained by EN 1097-6 (2002).

If we assume that the total water absorption of the tested fillers is at the same low level as for the corresponding coarse aggregate, we should probably focus much more on the determination of the “true outer surface of the filler”. This is since if say a 100 kg/m³ of filler is used in a concrete mix, a water absorption variation by 0.1% then means a variation of 0.1 kg/m³ in the water content, which is negligible from a practical point of view. We also shouldn't worry in how fine pore system of the filler is this water distributed. While on the other hand, the “true outer surface” of the filler will have direct impact on the rheological properties of such a concrete mix. None of the used widely available specific surface determination methods seems to correctly determine the outer specific surface area. It seems that specific surface measurements could be more useful when comparing fines from the same source, i.e. with equal mineralogy and shape. Due to this, other filler characterization methods must be used. In particular, wet packing experiments should be carried out and the possibilities of relating such characteristics to the rheological parameters of the corresponding fresh concrete and matrix must be investigated.

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Appendix A – Example Calculation for Surface Area of Filler

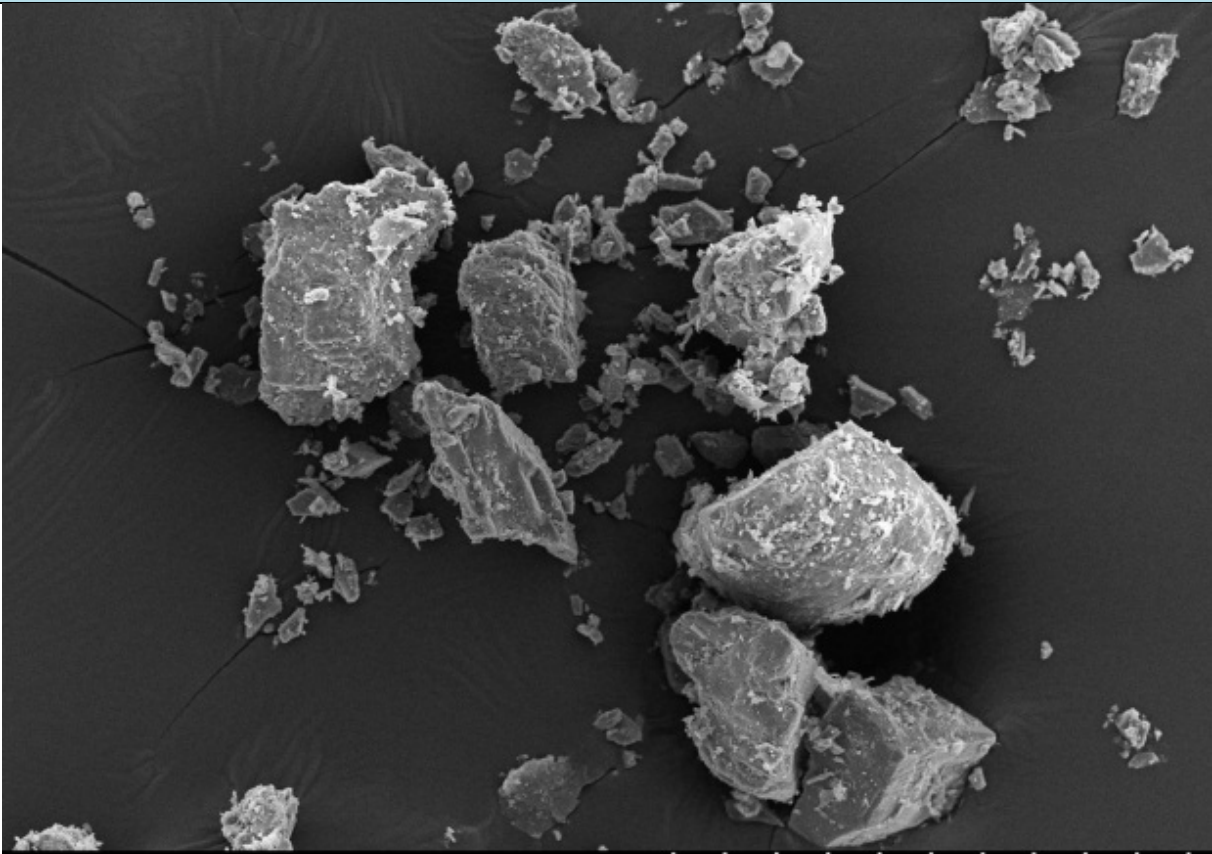
In the following (Table A-1) a numerical example of specific surface calculation (using LS Particle Size Analyzer results as an input) for one of the fillers (Limestone) is given.

Table A-1: Example calculation for specific surface area of limestone filler

A Measuring method	B Sieve aperture size, mm	C Cumulative % passing	D Percent passing among two sieves	E Amount of 1 kg of sand passing among two sieves, kg	F Volume passing among two sieves, m ³	G Average diameter D _i , m	H Volume of one particle, m ³	I No. of particles	J Surface area of one particle, m ²	K Specific surface area m ² /kg
				1 x D/100	Particle density, kg/m ³					
					2740					
					E/ρ					
mean size	πD _i ³ /6	F/H	πD _i ²	I x J						
LS Particle Size Analyzer	0.125	100.00	6.2	0.0620	2.26E-05	1.13E-04	7.46E-13	3.04E+07	3.98E-08	1.21
	0.100	93.80	3.6	0.0360	1.31E-05	9.00E-05	3.82E-13	3.44E+07	2.54E-08	0.88
	0.080	90.20	6.4	0.0640	2.34E-05	7.00E-05	1.80E-13	1.30E+08	1.54E-08	2.00
	0.060	83.80	11.6	0.1160	4.23E-05	5.00E-05	6.54E-14	6.47E+08	7.85E-09	5.08
	0.040	72.20	20.1	0.2010	7.34E-05	3.00E-05	1.41E-14	5.19E+09	2.83E-09	14.67
	0.020	52.10	17.2	0.1720	6.28E-05	1.50E-05	1.77E-15	3.55E+10	7.07E-10	25.11
	0.010	34.90	12.8	0.1280	4.67E-05	7.50E-06	2.21E-16	2.11E+11	1.77E-10	37.37
	0.005	22.10	17.2	0.1715	6.26E-05	3.00E-06	1.41E-17	4.43E+12	2.83E-11	125.18
	0.001	4.95	3.0	0.0300	1.09E-05	7.50E-07	2.21E-19	4.96E+13	1.77E-12	87.59
	0.001	1.95	2.0	0.0195	7.12E-06	2.50E-07	8.18E-21	8.70E+14	1.96E-13	170.80
		Σ=	100.0	1.0					Σ=	469.90

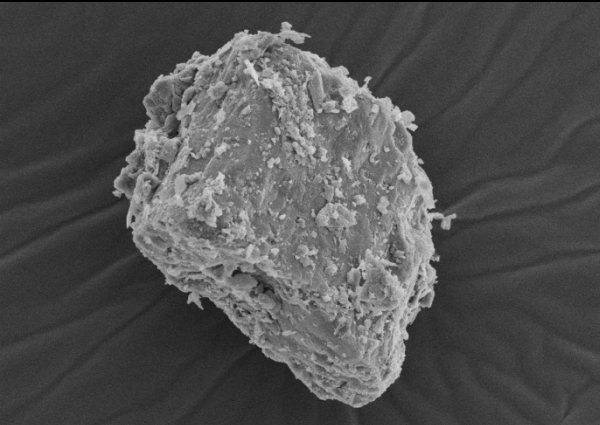
**Appendix B – Compilation of the SEM micrographs of
all the fillers and STD FA cement**

Årdal natural filler



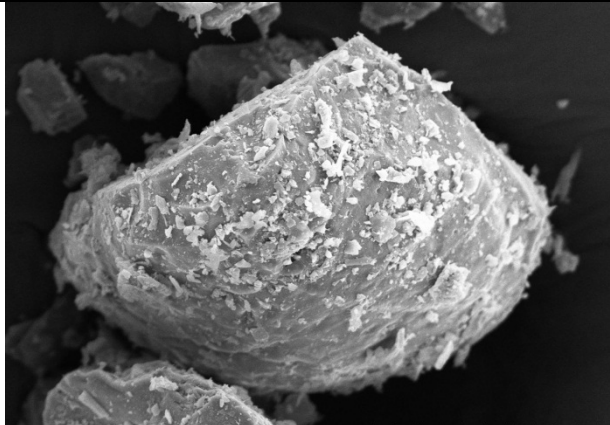
15.0kV 11.4mm x270 SE 28-09-11

200um



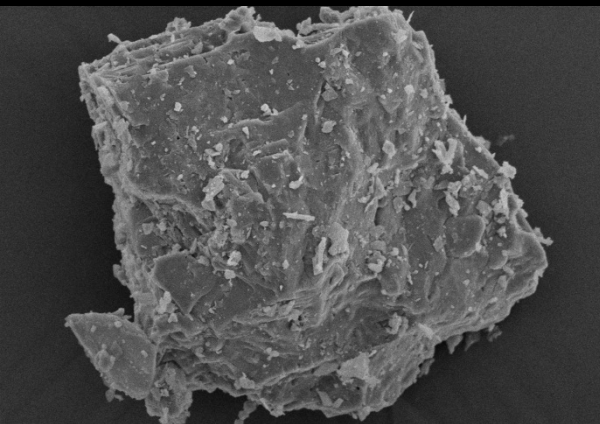
10.0kV 11.4mm x1.50k SE 28-09-11

30.0um



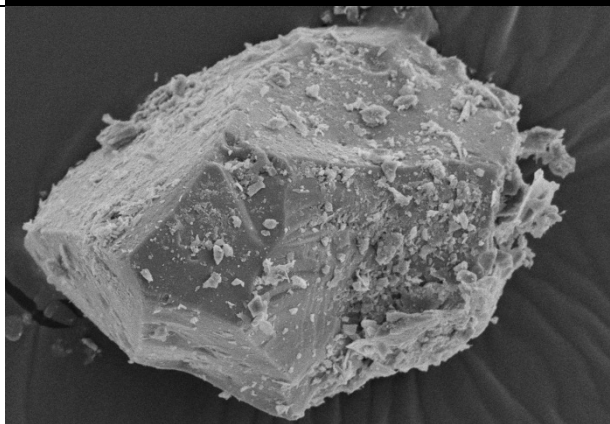
15.0kV 11.4mm x950 SE 28-09-11

50.0um



10.0kV 11.3mm x1.50k SE 28-09-11

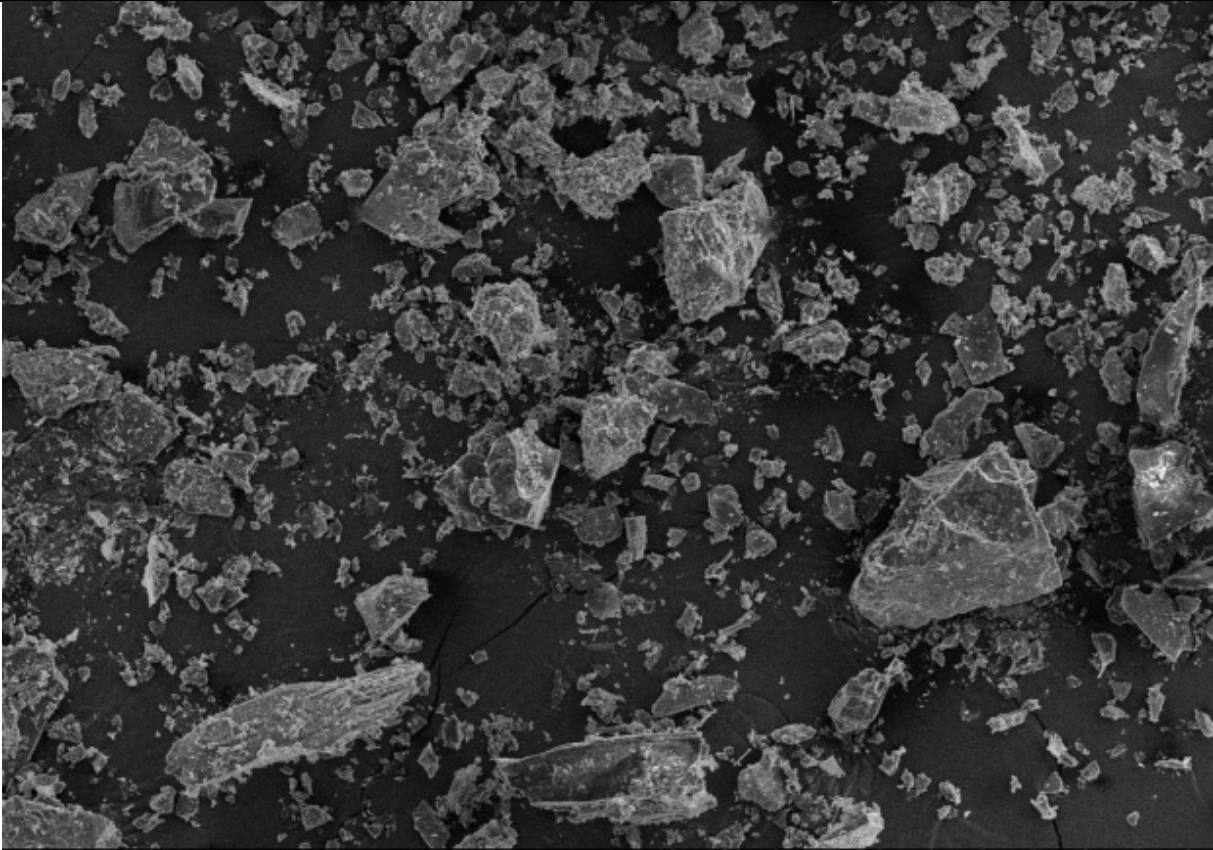
30.0um



10.0kV 11.3mm x1.40k SE 28-09-11

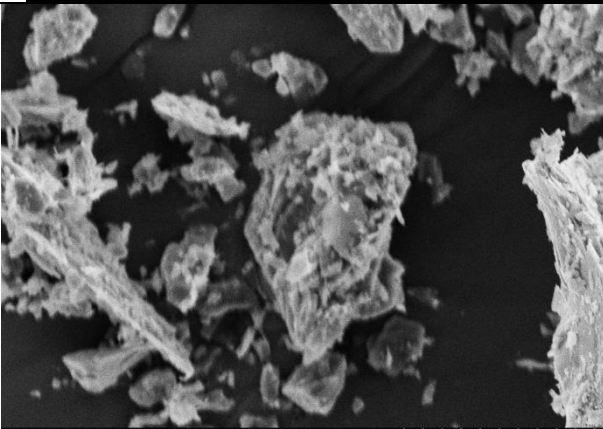
40.0um

Årdal crushed/ unwashed filler



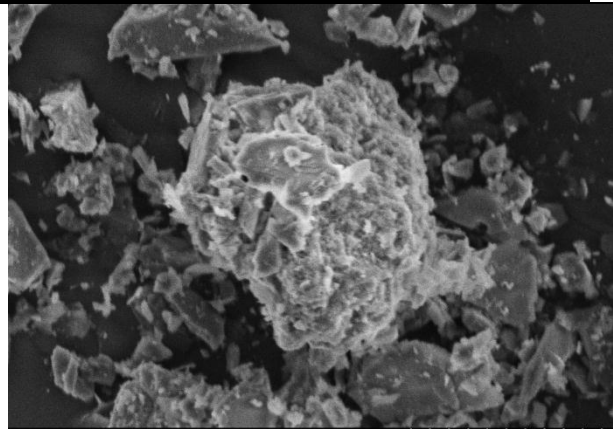
10.0kV 11.8mm x270 SE 30-09-11

200um



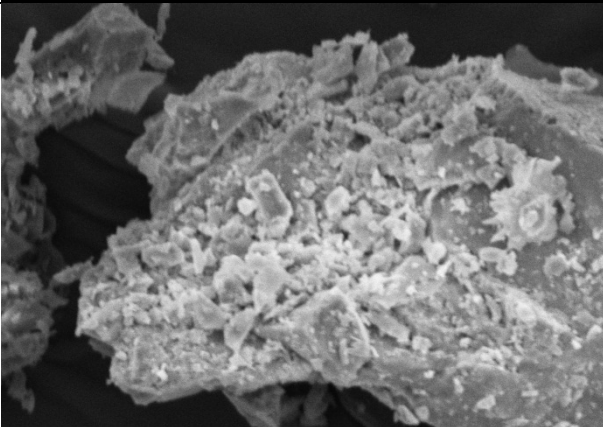
10.0kV 11.7mm x2.00k SE 30-09-11

20.0um



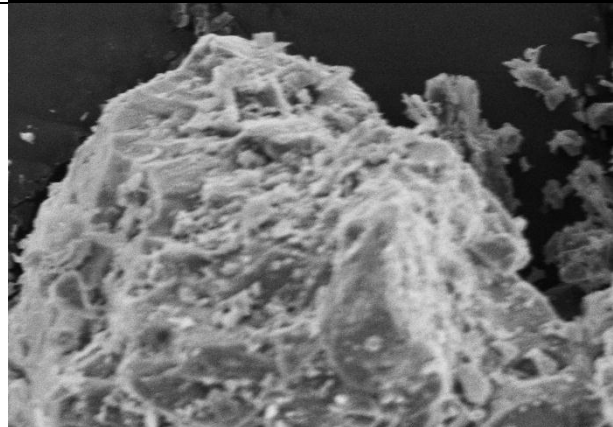
10.0kV 11.8mm x2.00k SE 30-09-11

20.0um



10.0kV 11.7mm x2.10k SE 30-09-11

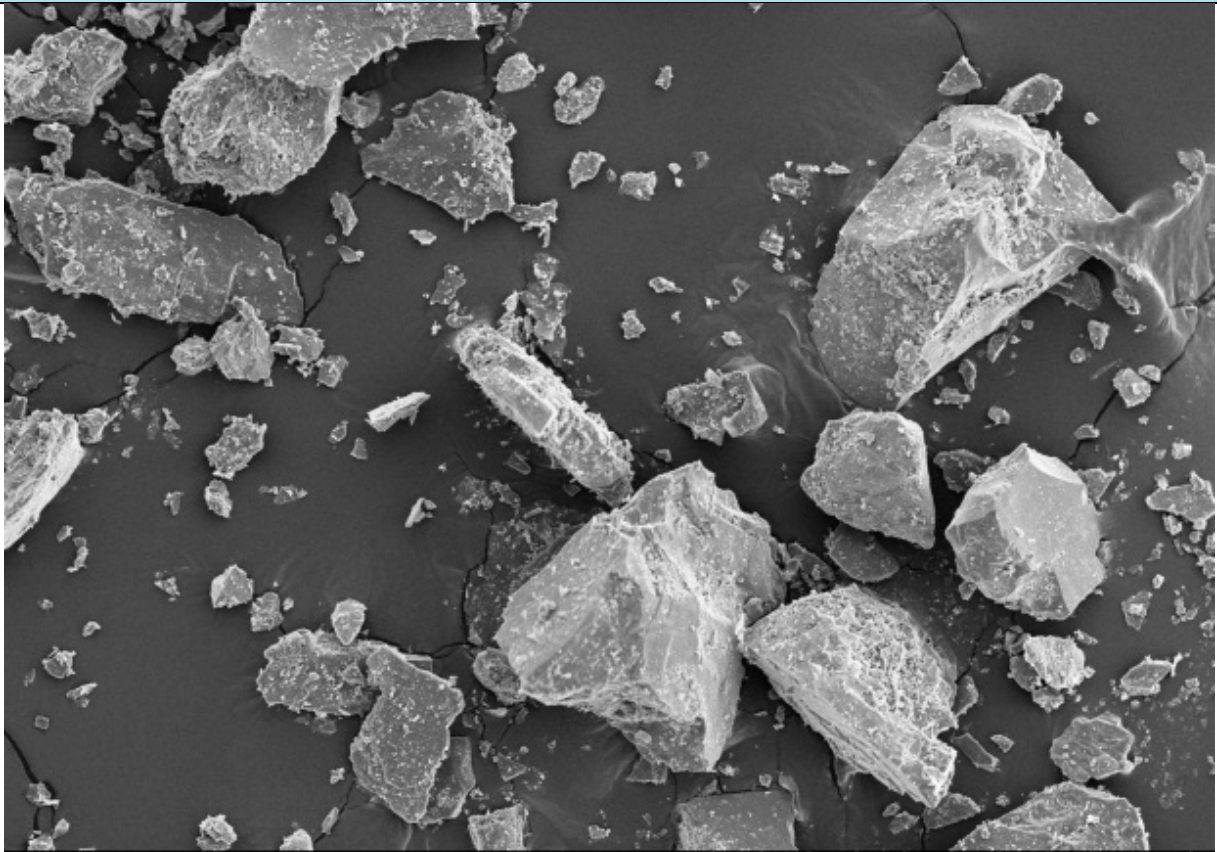
20.0um



10.0kV 11.9mm x2.00k SE 30-09-11

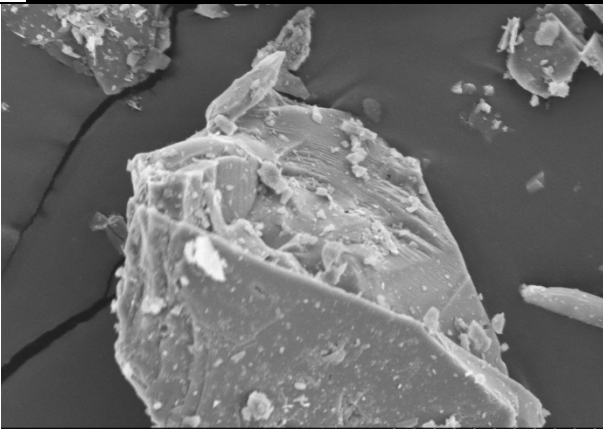
20.0um

Årdal crushed/ washed filler



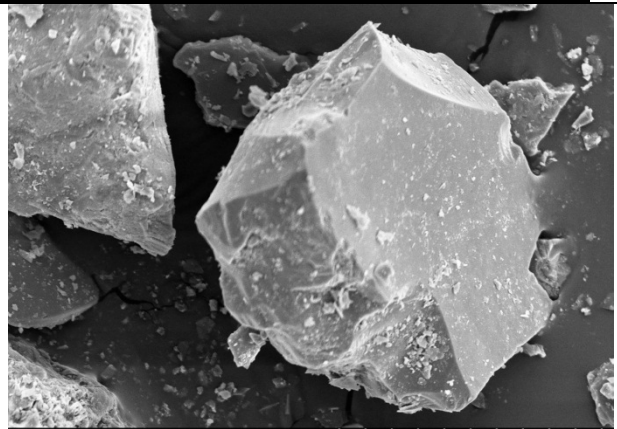
10.0kV 11.2mm x250 SE 30-09-11

200um



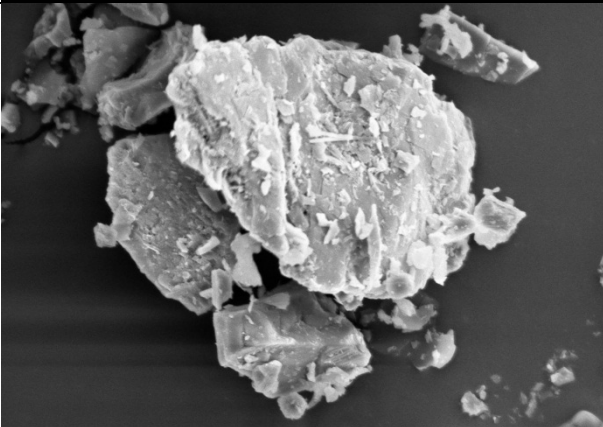
10.0kV 11.2mm x2.10k SE 30-09-11

20.0um



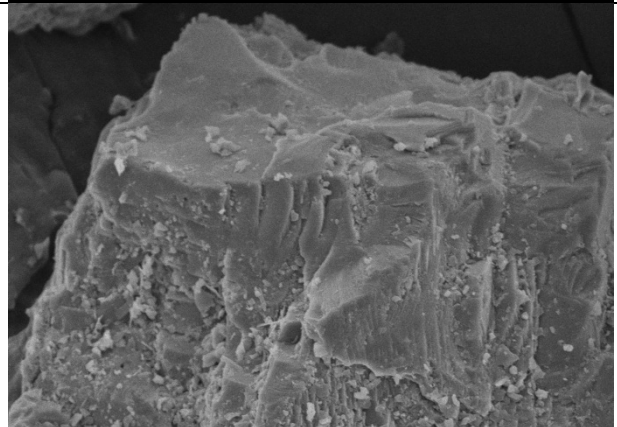
10.0kV 11.2mm x1.10k SE 30-09-11

50.0um



10.0kV 11.2mm x2.50k SE 30-09-11

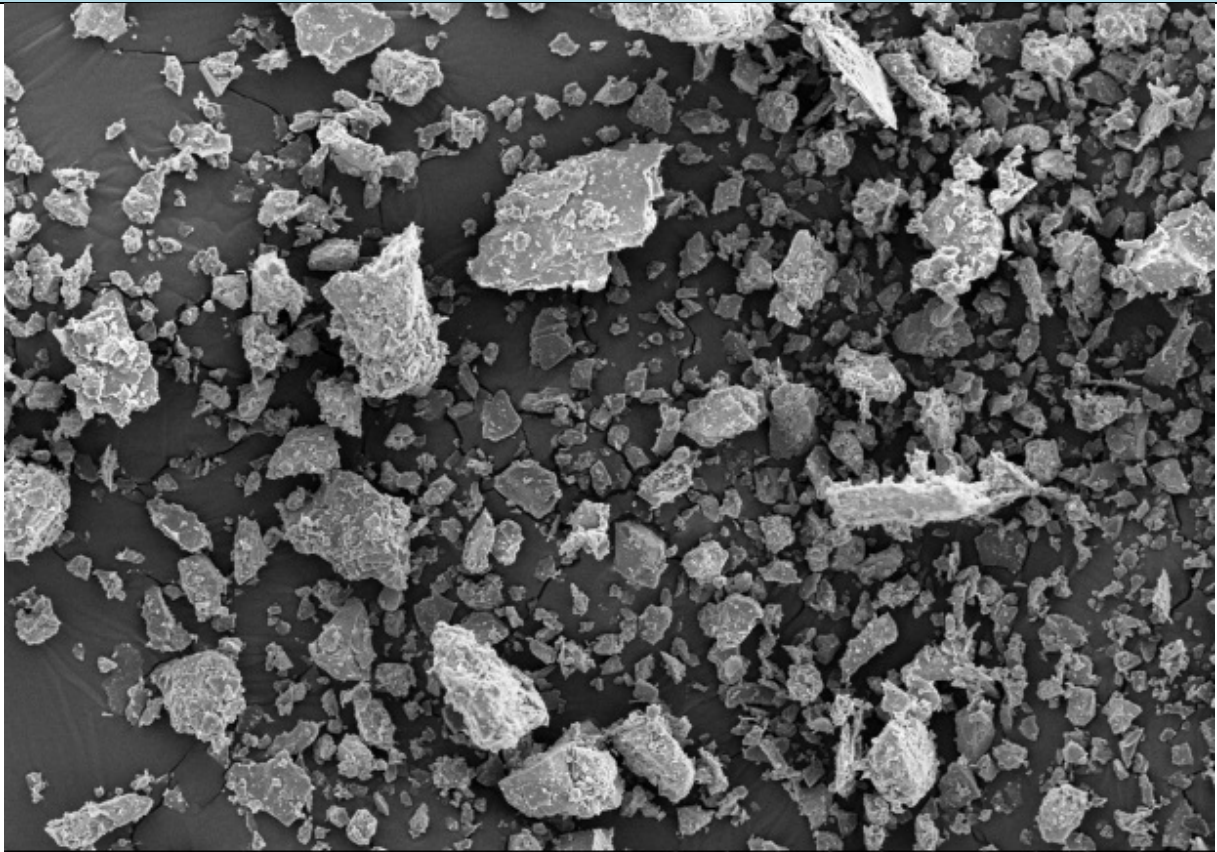
20.0um



10.0kV 11.1mm x2.10k SE 30-09-11

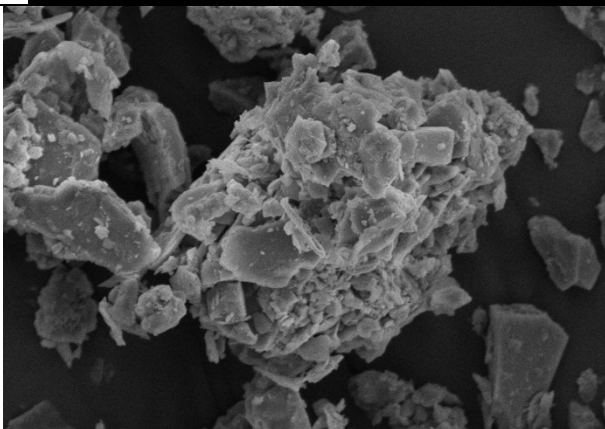
20.0um

Tau filler



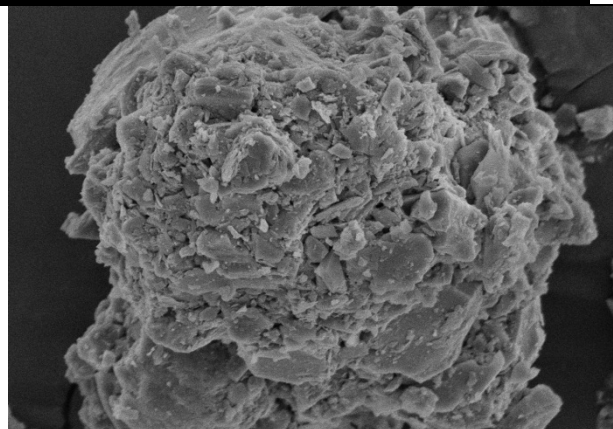
10.0kV 11.1mm x270 SE 30-09-11

200um



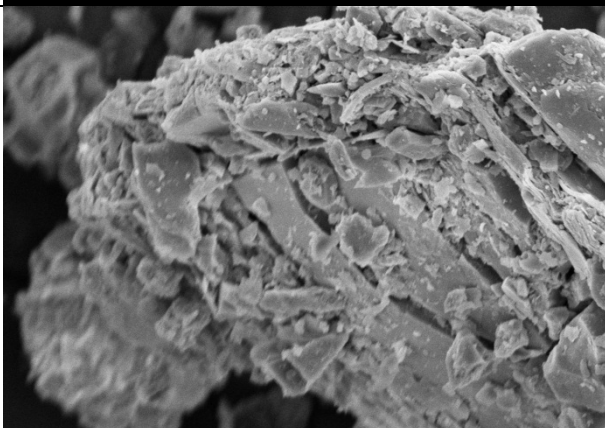
10.0kV 11.2mm x2.30k SE 28-09-11

20.0um



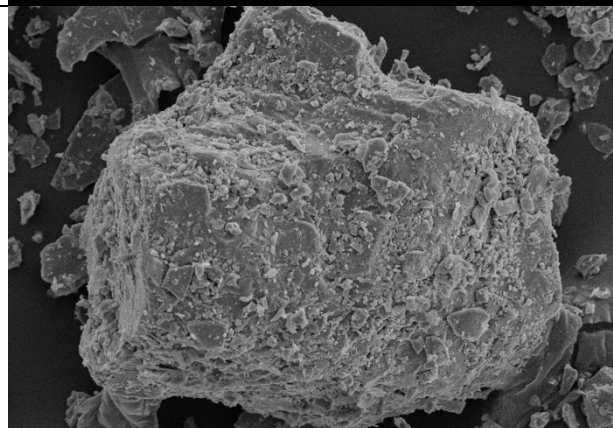
10.0kV 11.1mm x2.10k SE 28-09-11

20.0um



10.0kV 11.0mm x2.10k SE 30-09-11

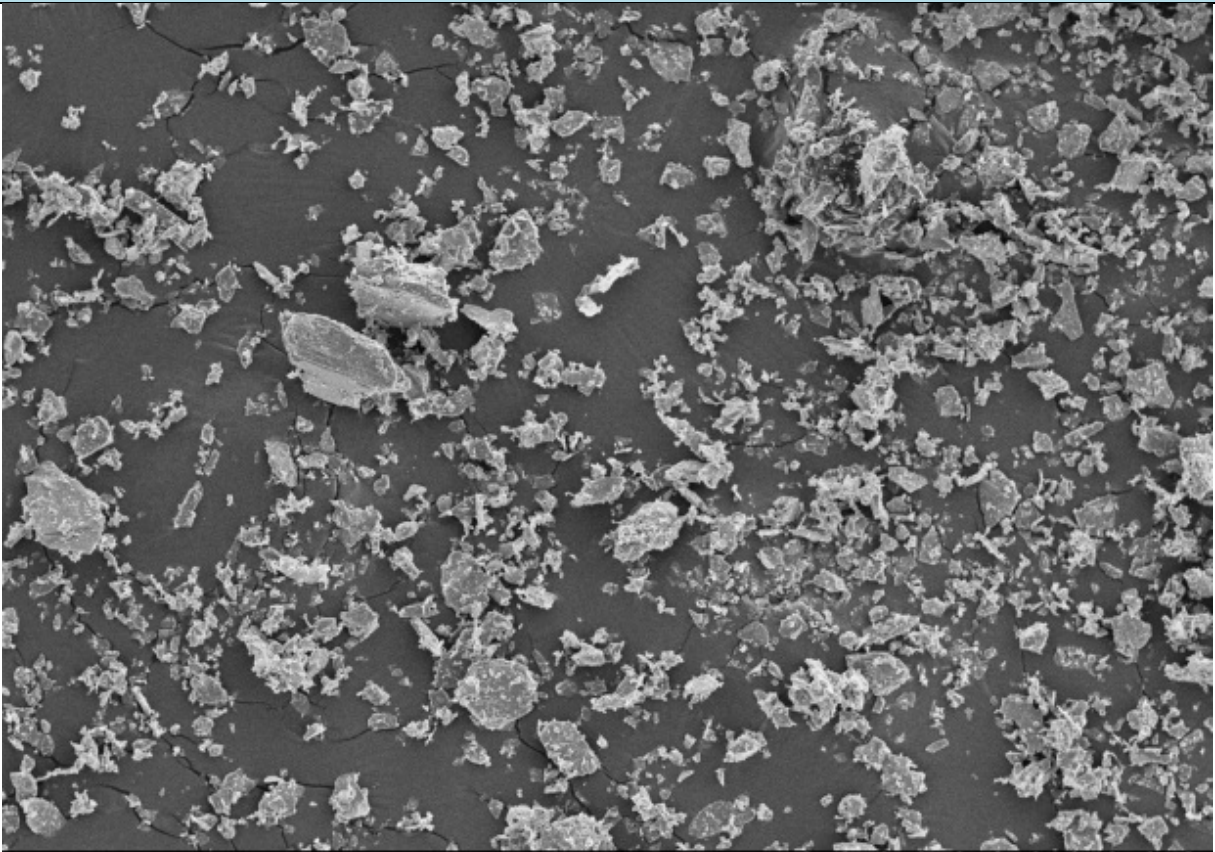
20.0um



10.0kV 11.1mm x800 SE 28-09-11

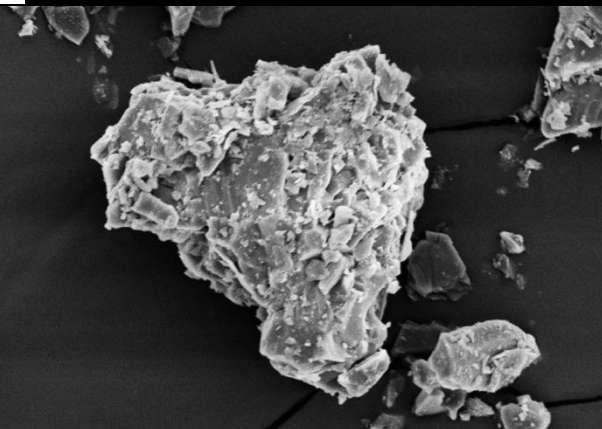
50.0um

Jelsa filler



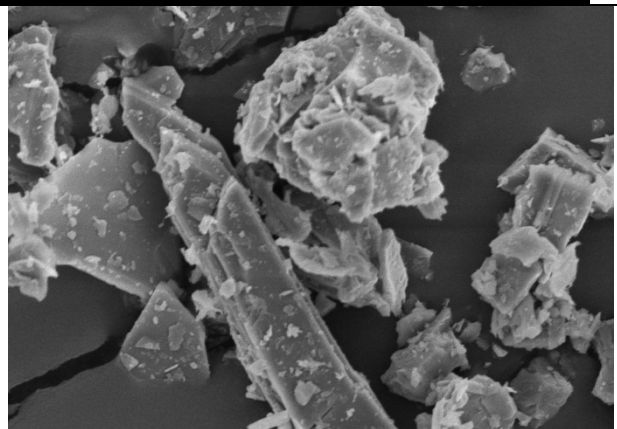
10.0kV 11.1mm x200 SE 30-09-11

200um



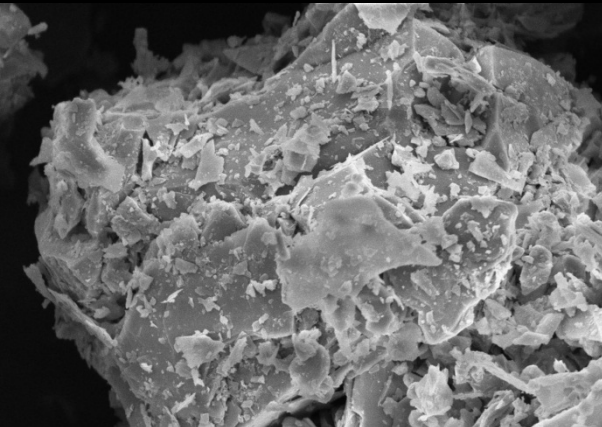
10.0kV 11.2mm x2.00k SE 30-09-11

20.0um



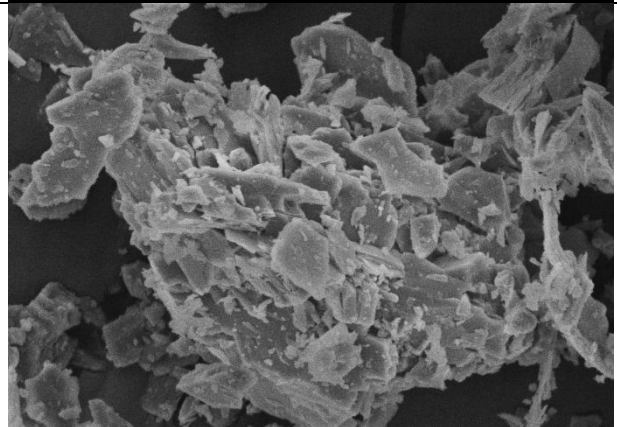
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20.0um



10.0kV 11.2mm x2.10k SE 30-09-11

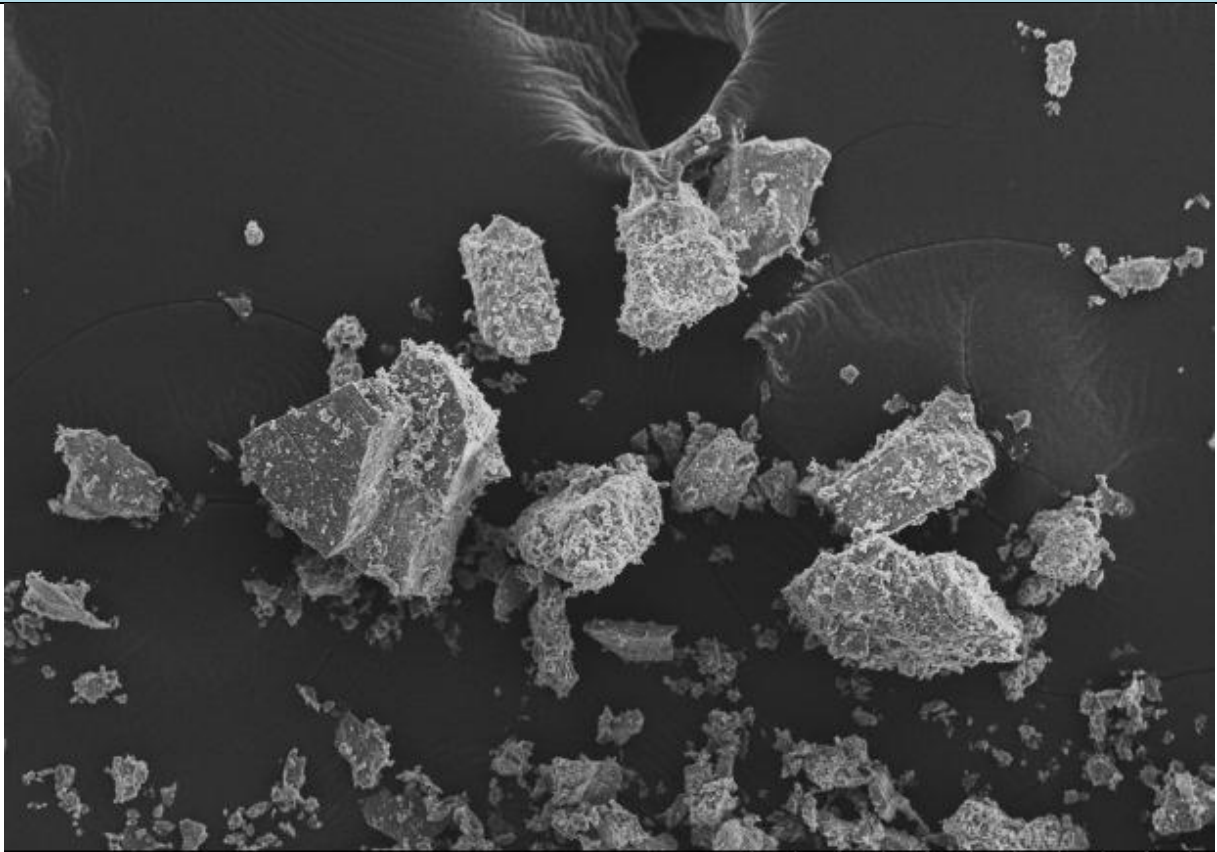
20.0um



10.0kV 11.2mm x2.50k SE 30-09-11

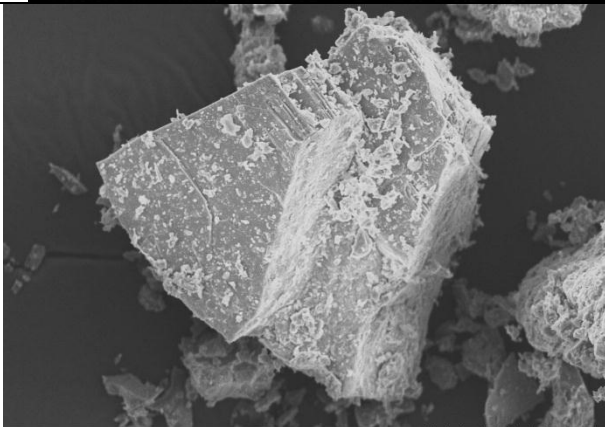
20.0um

Hokksund filler



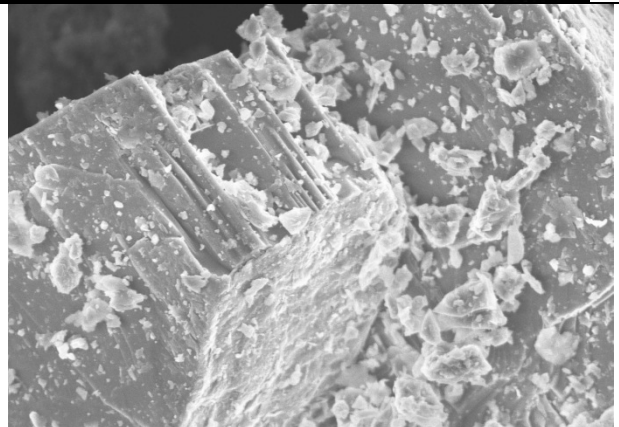
10.0kV 11.6mm x230 SE 30-09-11

200um



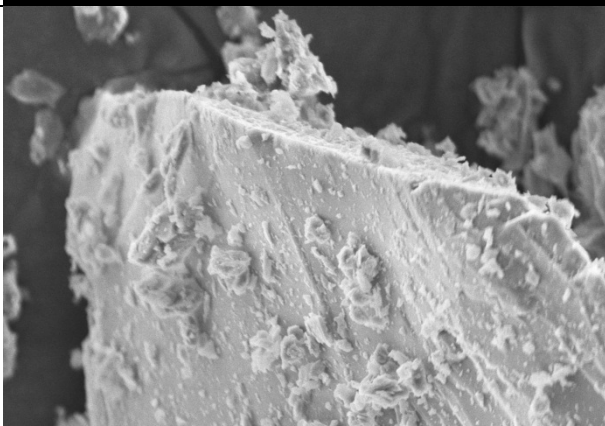
10.0kV 11.6mm x700 SE 30-09-11

50.0um



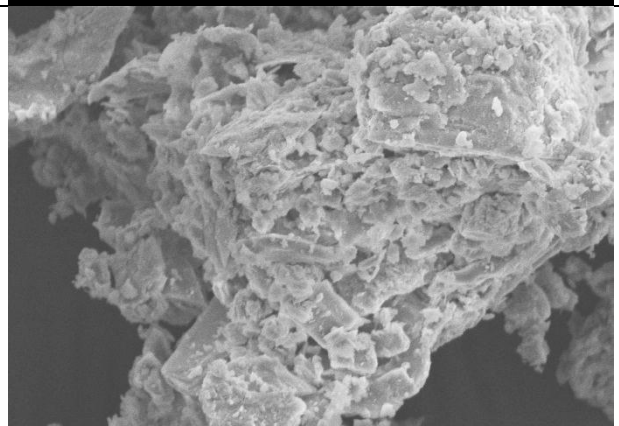
10.0kV 11.6mm x2.10k SE 30-09-11

20.0um



10.0kV 11.7mm x1.90k SE 30-09-11

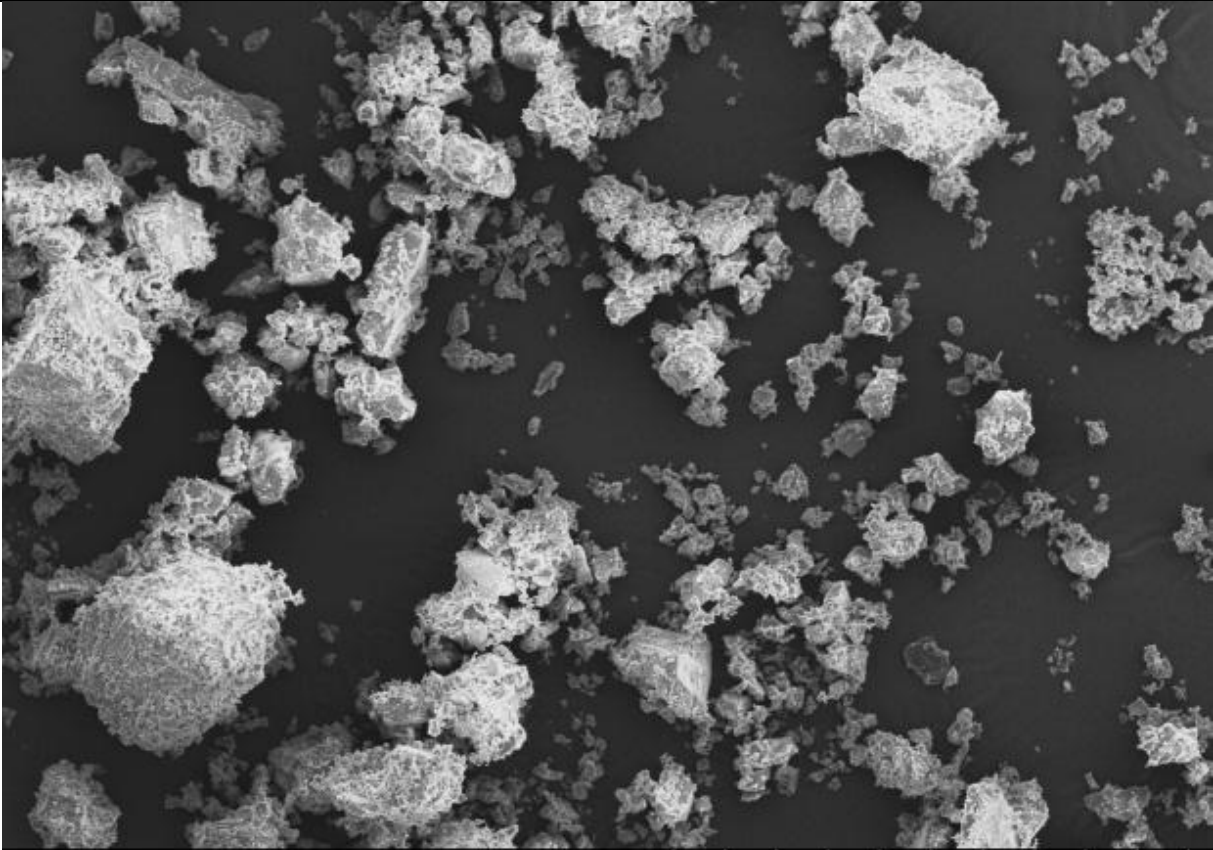
30.0um



10.0kV 11.7mm x2.00k SE 30-09-11

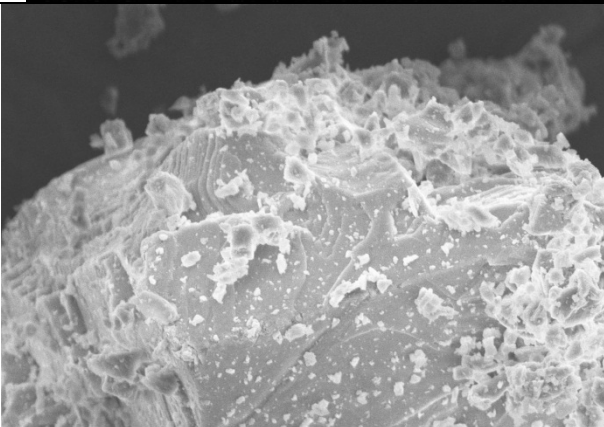
20.0um

Limestone filler



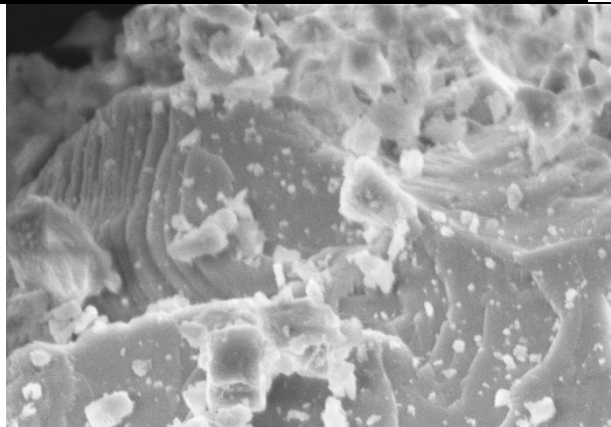
10.0kV 11.6mm x270 SE 30-09-11

200um



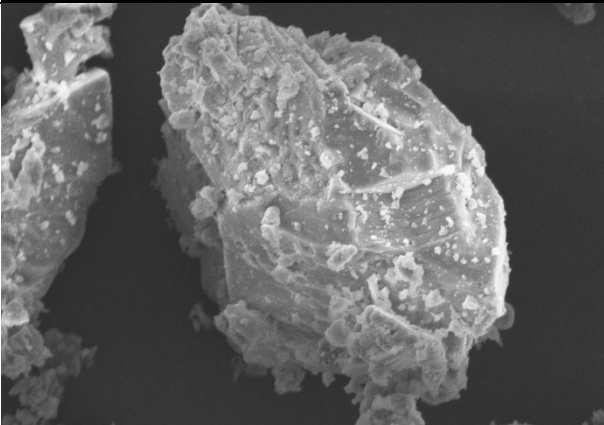
10.0kV 11.7mm x2.00k SE 30-09-11

20.0um



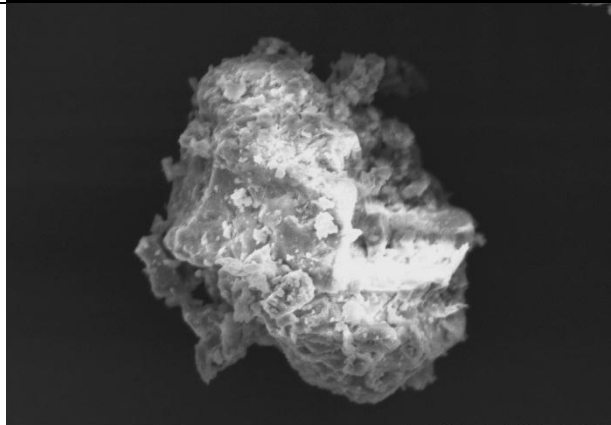
10.0kV 11.7mm x5.00k SE 30-09-11

10.0um



10.0kV 11.6mm x2.10k SE 30-09-11

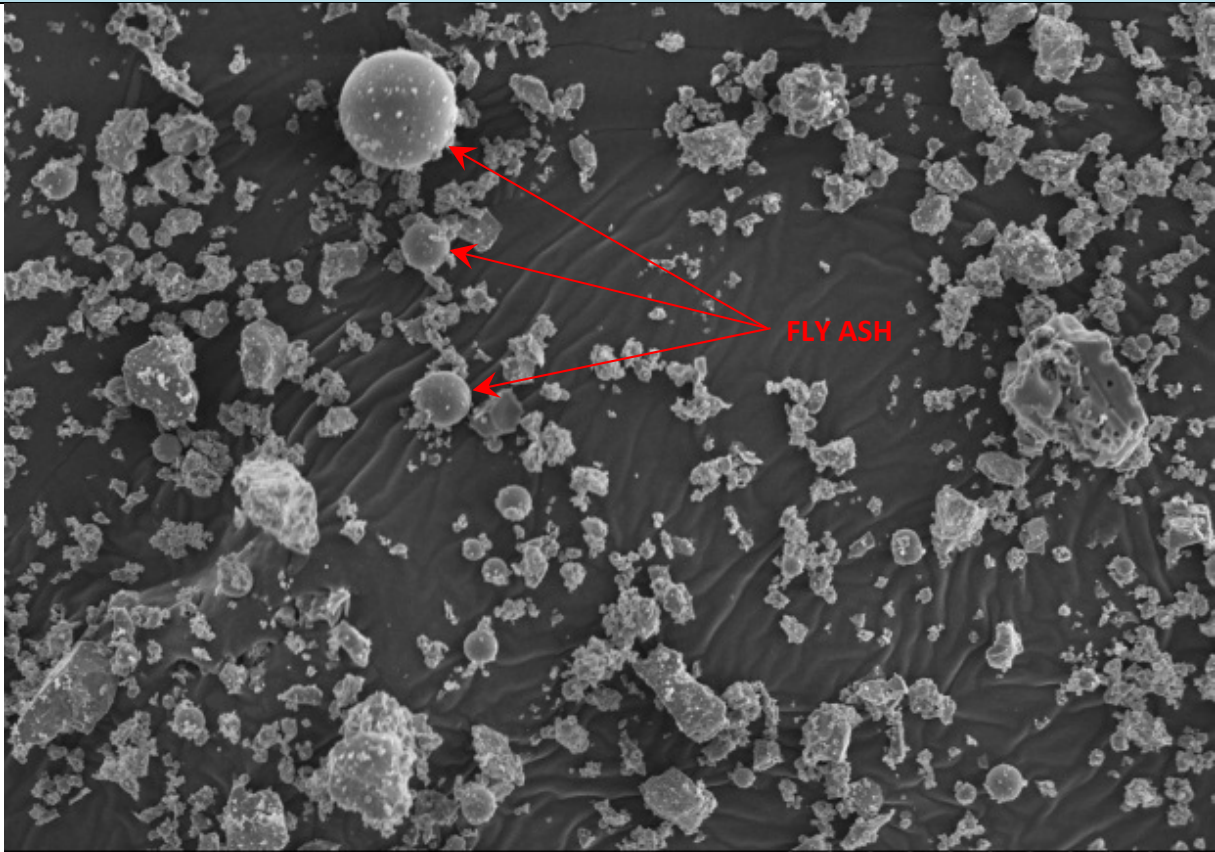
20.0um



10.0kV 11.6mm x2.00k SE 30-09-11

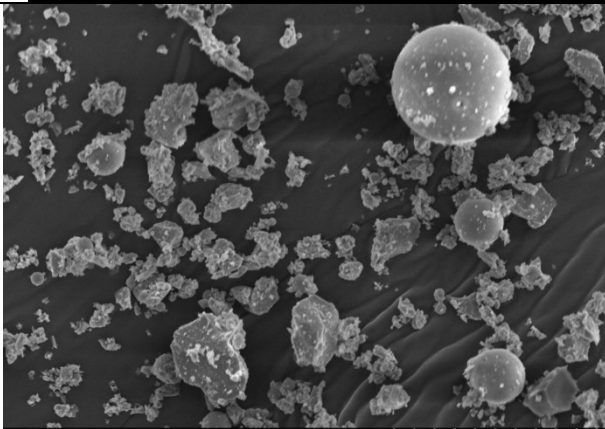
20.0um

STD FA cement



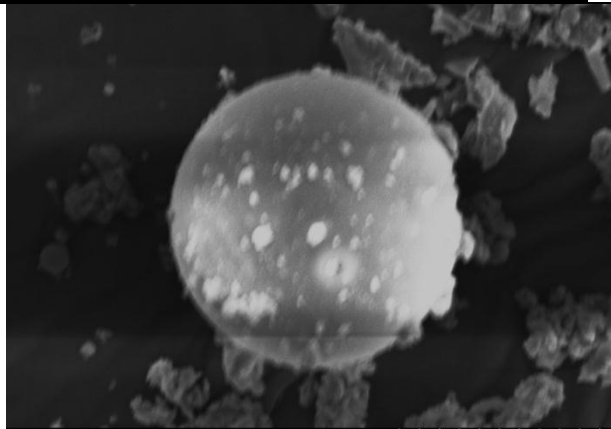
10.0kV 11.6mm x420 SE 30-09-11

100um



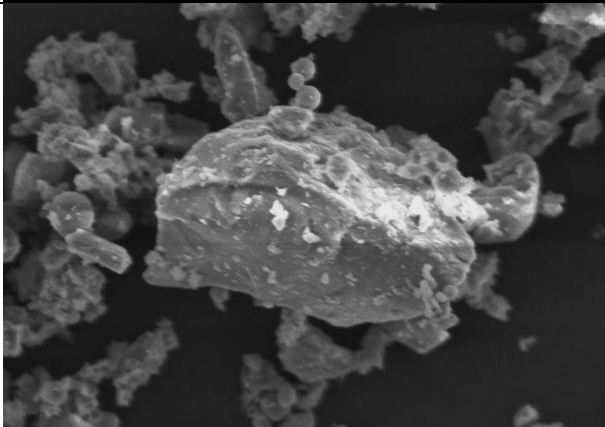
10.0kV 11.6mm x850 SE 30-09-11

50.0um



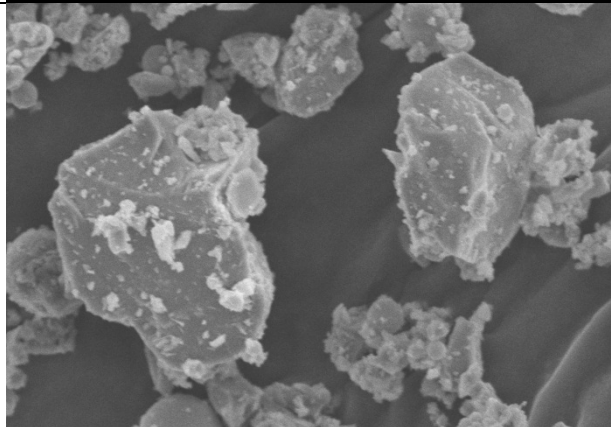
10.0kV 11.5mm x2.10k SE 30-09-11

20.0um



10.0kV 11.5mm x2.10k SE 30-09-11

20.0um



10.0kV 11.5mm x2.50k SE 30-09-11

20.0um

SINTEF Building and Infrastructure is the third largest building research institute in Europe. Our objective is to promote environmentally friendly, cost-effective products and solutions within the built environment. SINTEF Building and Infrastructure is Norway's leading provider of research-based knowledge to the construction sector. Through our activity in research and development, we have established a unique platform for disseminating knowledge throughout a large part of the construction industry.

COIN – Concrete Innovation Center is a Center for Research based Innovation (CRI) initiated by the Research Council of Norway. The vision of COIN is creation of more attractive concrete buildings and constructions. The primary goal is to fulfill this vision by bringing the development a major leap forward by long-term research in close alliances with the industry regarding advanced materials, efficient construction techniques and new design concepts combined with more environmentally friendly material production.

