

**Characterization of Construction Materials**  
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**Lecture-27**

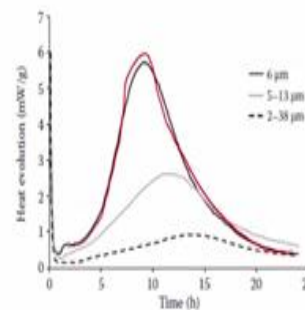
**Surface Area Measurement: Sampling and particle size distribution- Part 1**

Hello everyone, in this module we will discuss surface area measurements, which are commonly performed on construction materials, for an example, cement and SCMs - fly ash, slag, silica fume so, that will be the topic for this module.

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### Why Surface Area Measurement?

- Reactivity of a powder is influenced by its surface area (or fineness).
- The particle size and specific surface area of a cement powder have a direct influence on its hydration kinetics.
- These parameters have a direct impact on the rheological and mechanical properties of concrete and on the interaction between the cement and chemical admixtures such as superplasticizers.



M Palacios et al., 2016

So, why do we need to care about surface area measurements? The reactivity of a powder is influenced by its surface area or fineness. The particle size and specific surface area of cement powder have a direct influence on its hydration kinetics. So, as you see in this plot, there are three different types of, in this case, Tricalcium silicate ( $C_3S$ ) (Alite) powder of different particle sizes. So, in one case it is around 6  $\mu\text{m}$ , then it is 5 - 13  $\mu\text{m}$ , and 2 - 38  $\mu\text{m}$ . So, basically, we are looking at the effect of the fineness. 6  $\mu\text{m}$  is the finest one. So you see it is affecting the hydration kinetics. As you go towards the higher sizes, there is not as much as acceleration as you see in the case of the finest powder, in terms of particle size.

So, it tells you that the size and surface area have direct influence on hydration kinetics. If it has direct influence, then it will also impact your rheological and mechanical properties.

Rheological properties like flow, for an example, and mechanical properties, because this hydration kinetics will have a role on that. So, these are for different sizes and the size range is around that as given in graph.

These parameters influence the interaction between cement and chemical admixtures, such as superplasticizers, we use these admixtures to modify the early-age properties in some cases, like rheological properties. So, that interaction also is influenced by the size and surface area. So now it is clear that we need to measure the surface area of a powder, for an example, in this case, we are talking about cement powder.

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## Measurements

- Particle size distribution
  - Size and size distribution influence the handling, storage and domain of use of a powder

- Specific surface area
  - Total surface area normalized by the amount (expressed as  $\frac{\text{cm}^2/\text{g}}$  or  $\frac{\text{m}^2/\text{kg}}$ )

by 20g SSA =  $\frac{SA}{mass}$

So, what are the typical measurements which we do? All of you are familiar with these two aspects. One is particle size distribution, we want to get an estimate of particle size distribution, that is, what is the size range of particles in powder? Basically, the size and size distribution influences the handling, storage and domain of use of a powder, because that will affect your hydration kinetics, in case of cement as we have already seen.

Surface area is nothing but total surface area, we are talking about the total surface area of powder. Specific surface area is a normalized surface area. So, usually it is normalized by the amount. If you have two different amounts of powder, so in one case you have 10 grams of powder in other case you have 20 grams of powder, so your total surface area of 10 gram will be lower than 20 gram surface area. But we are talking about specific surface area, where we divide

the surface area by amount (mass). So we are talking about per gram, one gram of powder, how much surface it has. It is used to compare two different powders or materials. So, we are talking about one gram of material A has surface area of this, one gram of material B have surface area of this. So, now you understand why we are doing this normalization, so that we can use it for comparison. It is typically expressed as centimeter square per gram ( $\text{cm}^2/\text{g}$ ) or meter square per kg ( $\text{m}^2/\text{kg}$ ), which means that one gram of a particular material has so much square centimeters of area, .

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## Particle Size Distribution



So, if you look at the particle size distribution, we are talking about the sizes. It can vary; you will have a difference in the size, shapes. As you can see, we can have very irregular size, as you see in the case of GGBFS - Ground granulated blast furnace slag, which does not look like spherical, it has very irregular shape. You see in silica fume, you have some spherical shapes, also you have some agglomeration. And in fly ash also you have spherical primary particles, you have irregular primary particles which look irregular and not spherical. Then you have agglomerates and you have aggregates.

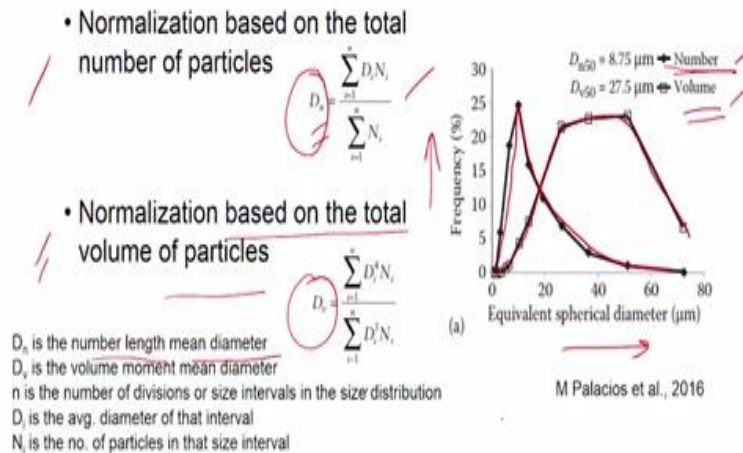
So you may have a lot of particles together, some are bonded chemically, some are not bonded chemically, there are some physical bonds. That is the difference. So in aggregates, you have some chemical bonding and in agglomerates you have only physical bonds, like van der Waals bonds, so the attraction is not very strong, that is the only difference.

Now if we have to define particle size, considering you have irregular size, how do you do it? That is the challenge, we cannot measure since it is very irregular. So we define, Equivalent Spherical Diameter – ESD for non-spherical particles. For sphere, it is easy to define diameter, as the diameter will be characteristic of a sphere. But if you have non-spherical particles, we define Equivalent Spherical Diameter.

For an example, just to understand what it means, imagine you have a cube of 1 cm size, so you have a volume of 1 cc. Now if you find the diameter of a sphere which has equal volume (in this case volume = 1 cc), that is the meaning of equivalent spherical diameter, this is on the volume basis. You can do it on surface area basis also, so, what you do is suppose the radius of sphere is R, volume of sphere ( $\frac{4}{3} \cdot \pi R^3$ ) has to be equal to 1. So, then you can calculate R, and that you can use to calculate diameter, which is equal to 2R. So, you can say that the sphere of this size will have equal volume as cube. So, that is a convenient way of expressing the diameter. So, this is on the volume basis, anyway we will come back to it, but I am just telling you, you will encounter this terminology, Equivalent Spherical Diameter.

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## Average Particle Size



So, we can do this normalization based on total number of particles also. So, let us see, commonly used normalization which we use for average particle size. So, one is based on number of particles. So, you define  $D_n$ , which is the number length mean diameter.

$$D_n = \frac{\sum_{i=1}^n D_i N_i}{\sum_{i=1}^n N_i}$$

where  $D_i$  is average diameter in that interval.

So, you have now, we are talking about size intervals. You can divide your whole range in multiple size intervals and you know that, what the average diameter in that interval is. And you also know what  $N_i$  is ( $N_i$  is the number of particles in that size interval). So these are two different ways of normalizing things. So,  $D_n$  is normalization based on the total number of particles.

And you can also do normalization based on volume. So, again the formula will be different, you can use this term ( $D_v$ ).

$$D_v = \frac{\sum_{i=1}^n D_i^4 N_i}{\sum_{i=1}^n D_i^3 N_i}$$

So these are two common normalization we do - normalization based on total number of particles and normalization based on total volume of particles. Both are different, it is very important when you look at PSD, what is the metric. So, it is illustrated here in this graph, what do you see? We are plotting distribution curve, on Y-axis we have frequency, X-axis we have ESD. So, when you plot on number basis, you get this distribution, and when you plot on volume basis you get another distribution. So the distributions are different, which is why it is very important to pay attention to the basis, lot of papers will not even mention what kind of basis they have used. But there is a difference, when you do number of particles versus volume of particles, with the same data.

So we will come back to it but these are commonly used normalization as far as PSD is concerned, but you have to know what normalization you are using. If just frequency and equivalent surface diameter is given, it is very hard to tell what is it based on and both will have different implications.

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## Aggregation: Agglomeration Factor

- Many powders have aggregates (chemical bonds between particles) or agglomerates (physical bonds such as van der Waals forces between particles)

• Agglomeration factor,  $F_{AG} = \frac{D_{V50}}{D_{BET}}$ ,  $D_{BET} = \frac{6}{SSA \cdot \rho}$

Median volume diameter ( $\mu\text{m}$ )

Average diameter ( $\mu\text{m}$ ) from specific surface area

So, many powders have aggregates; aggregation is always an issue. So, you will have aggregates where we are talking about the chemical bonds between the particles. And also it may have agglomerates. In agglomerates, we are talking about physical bonds like weak bonds, van der Waals bonds. And we can measure the agglomeration by defining this agglomeration factor. So, it can be defined as ratio of mean volume diameter ( $D_{V50}$ ) to the average diameter ( $\mu\text{m}$ ) from specific surface area ( $D_{BET}$ ), that is:

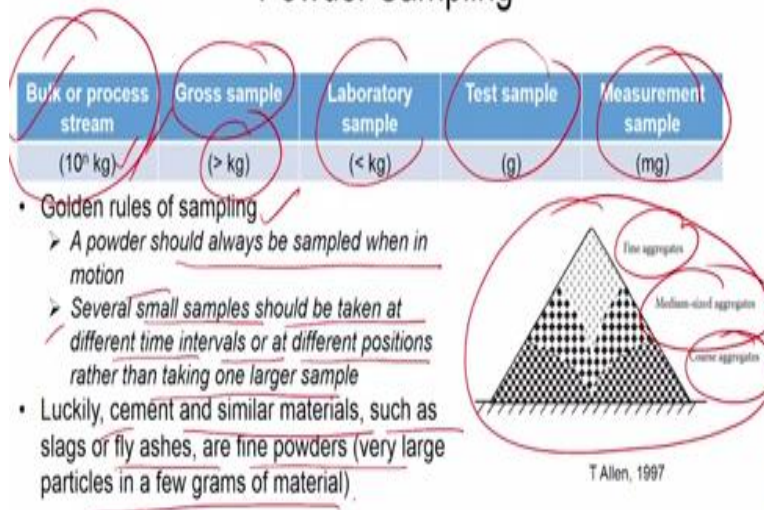
$$F_{AG} = \frac{D_{V50}}{D_{BET}}$$

So, if you have a high agglomeration factor that means you have a higher extent of agglomeration.  $D_{BET}$  is average diameter from specific surface area where BET is a theory we use to determine the surface area, so once you know the surface area (SSA) and the density ( $\rho$ ) we can calculate the diameter.

$$D_{BET} = \frac{6}{SSA \cdot \rho}$$

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# Powder Sampling



So before you do particle size distribution, you have to have a representative sample, which is very important. So, you are interested in, suppose in determining particle size distribution of a bulk or some process stream, we are talking about significant amount and you are interested in knowing the particle size distribution in that. It could be intense, that is the idea. But you cannot carry out particle size distribution on that scale. So you have to bring it down.

So, you always take sample out of it, first your sample will be in really significant amount, then it could be in kilograms, you have taken sample out of it, (gross sample). Then from gross sample you will have laboratory sample. You will take some sample where you will do some tests. So, it could be in less than kilograms; you are reducing the size, but at the same time it has to be representative. Then with laboratory sample, you will have to do multiple tests, different tests. So, you need sample for different types. Finally, your test sample will be in grams and each test sample may have multiple measurements, finally you are talking about in milligrams.

So, during this sequence we have to make sure the sample is representative as we want to know the particle size distribution in the bulk or process stream. And finally the measurement is done in milligrams, for an example, this particle size distribution, or any other technique use, so, sample size is very small. So, these things you have to keep in mind. So, for that we have to follow some rules of sampling. These are called golden rules of sampling.

- A powder should always be sampled when in motion.

- Several small samples should be taken at different time intervals or at different positions, rather than taking one large sample. So, these are two rules of sampling to avoid the errors.

You see here what happens when you just have a heap of a powder (Figure on right in slide). If you just leave it, what happens is the coarse aggregate will tend to come to the bottom. So, you already have segregation here. So that segregation will induce errors.

There are two types of errors we are talking about. One is because of the material heterogeneity, which we cannot avoid, that is, if the material itself is heterogeneous, it will lead to some errors. But the second type of error, we are talking about is because of segregation, which can be prevented. For that, you have to follow proper sampling process. So, luckily cement and similar materials such as slags, fly ashes, are very fine powders. So, very large number of particles is there in very few grams of materials. So, if you take a few gram of material, it can give you a good statistical average. So, that is the benefit. But anyway, it is good to follow these sampling rules.

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## Representative Sample

- Minimum sample weight can be calculated using:

$$W_m = 0.5 \left( \frac{\rho_p}{\sigma_i^2} \right) \left( \frac{1}{w_1} - 2 \right) \left( \frac{D_1^3 - D_2^3}{2} \right) \times 10^3$$

where  $W_m$  is the minimum weight (in grams),  $\sigma_i^2$  is the variance of the tolerated sample error,  $\rho_p$  is the powder density (in g/cm<sup>3</sup>),  $w_1$  is the mass fraction of the largest size class sampled,  $D_1$  is the maximum diameter of the largest size class samples (in cms) and  $D_2$  is the minimum diameter of the largest-size class sampled (in cms).

- For a typical cement, this is close to 1 g for a 5% tolerated error and about 300 mg for a 10% tolerated error.

M Palacios et al., 2016

Also, how do we define a representative sample? How do you know, how much of sample you need to take? So, this is the formula you can use. The minimum sample weight ( $W_m$ ) can be calculated using this formula,

$$W_m = 0.5 \left( \frac{\rho_p}{\sigma_i^2} \right) \left( \frac{1}{w_1} - 2 \right) \left( \frac{D_1^3 - D_2^3}{2} \right) * 10^3$$



where  $W_m$  is the minimum weight required (in grams).

$\sigma_i^2$  is the variance of the tolerated sample error

$\rho$  is the powder density

$w_1$  is the mass fraction of largest size class sampled.

$D_1$  is the maximum diameter of the largest size class sampled (cm)

$D_2$  is the minimum diameter of the largest-size class sampled (cm).

So, once you know this, you can find what should be the minimum sample weight for a given variance of the tolerated sample error. For an example, for a typical cement, this is close to 1 gram for 5% tolerated error. If you want 5% tolerated error, so you know  $\sigma$ , you know the  $\rho$ , and you know all these quantities based on your particle size distribution. So, you need 1 gram for 5% tolerated error. If you decide to take only 300 milligrams, then your tolerated error will go up to 10%. You see, that's a simple example. So, it will depend on the material you are working with, you cannot just assume that you will use the same amount for different materials. It depends on these factors, we are not going in detail, but this can serve as a guide, whenever you do sampling.

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## Sieving

- Simple and widely used method of classifying powders according to their physical size alone
- Covers a wide particle size range, from  $\sim 37 \mu\text{m}$  to 125 mm using woven wire sieves
- Key variables: particle shape, presence of very fine particles, initial sieve loading, time and method of agitation, and cohesiveness of the powder (in dry sieving only)
- Due to size limitations at the lower end, sieving, by itself, is not a suitable method for characterizing the complete PSD of cement powders
- Better suited for analyzing the large size fraction contained in cement powders, i.e., larger than  $50 \mu\text{m}$
- Care must be taken while cleaning and handling sieves with apertures below  $250 \mu\text{m}$  for accuracy

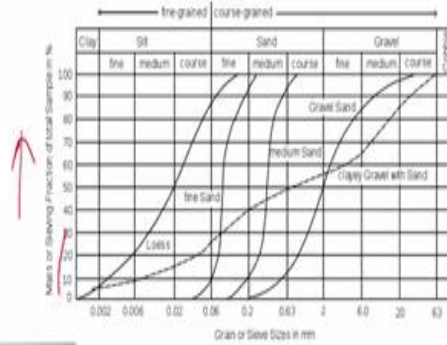
C Ferraris et al., 2002

So how do we determine particle size distribution? Especially for large sizes, for an example, sand and coarse aggregate, which we use in concrete we do sieving. Sieving is a commonly used technique.

- So it is a simple and widely used method for classifying powders according to their physical size alone. We do not care about chemical makeup, just physical, how they, what the shape and size is.
- It covers a wide particle size range from 37  $\mu\text{m}$  up to 125 millimeters. We use woven wire sieves for sieving.
- So, when you are talking about sieving, what is important? Key variables are particle shape, presence of very fine particles, initial sieve loading (how much you are putting), time and method of agitation - how are you performing your sieving? Is it manual, is it mechanical?, cohesiveness of the powder. These are the key variables, which can affect your sieving results.
- So, due to the size limitation at the lower end, we see that we cannot go below, for an example 30  $\mu\text{m}$ , which becomes really difficult to see. So, sieving by itself is not a suitable method for characterizing the complete PSD of cement powders. So, we can use it for coarse materials, but for cement, where your particle size ranges from less than micron to hundreds of micron, it may not be a suitable method.
- So, better suited for analyzing large size fraction contained in cement powders, like, which are larger than 50  $\mu\text{m}$ , for an example.
- But also care must be taken while cleaning and handling sieves with apertures below 250  $\mu\text{m}$  for accuracy. What happens when you do sieving it clogs your sieves. So, if you do not clean your sieves, then your results will get affected, very important. You have to pay attention especially as you are going down the size.

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# Sieving Apparatus and Analysis



[https://en.wikipedia.org/wiki/Sieve\\_analysis](https://en.wikipedia.org/wiki/Sieve_analysis)

Everyone must have seen this. This is a typical sieving apparatus, the mechanical one. So you do not have to manually shake, you can mechanically shake it. So, you have different sizes, which are stacked on top of each other.

So this is a typical particle size distribution. On the Y-axis, mass or sieving fraction of total sample in % is plotted and on the X-axis we have sieve sizes (in mm) for different types of materials. Starting from gravel, sand, silt, clay, it can tell you which one is finer and what the range is.