Measuring water demand or packing density of micro powders – comparison of methods.

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Introduction

The particle packing models and optimisation techniques nowadays used in the design of concrete have resulted in new mixture compositions with very high compressive strengths. These new methods and theories can also be used to investigate the effects of cement replacing materials and fine fillers. This way the particle packing theories would not serve an increase of strength, but a reduction of the cement content, thus creating a more ecological concrete. However, the existing particle packing models do not yet include the particle packing of fine particles (< 100 micrometer) in a sound way. One of the reasons for this might be caused by the difficulty measuring the maximum packing density of fine powders.

With particle packing measurements of particles > 1 mm gravitational force and shear forces are dominant. However, with fine particles, the inter-particle forces become increasingly important. These inter-particle forces can cause, for instance, agglomeration of particles, thus lowering the packing density. Since the inter-particle forces depend on the conditions (dry, wet) of the packing structure, also packing density is influenced by this. Therefore it is important that the maximum packing density of the particles is measured under the same conditions as under which the particles would be used in concrete and in the model.

The maximum packing density of dry particles, can be determined according to NEN-EN 1097-3 for loose bulk density. The method can be extended to determine the maximum packing density at a certain compaction level, by applying external loads such as vibration or top-weight. To determine the maximum packing density of wet particles no single method is generally accepted and therefore different countries use their own test methods to determine packing density and/or water demand of fine particles. In this paper a comparison between a number of these techniques is made to evaluate them on number of tests, accuracy, repeatability, reproducibility and suitability to use for cementitious materials.

Methods

The methods to determine packing density and/or water demand of fine particles evaluated in this paper are:

- Water demand France [de Larrard, 1999]

- Water demand Germany [Puntke, 2002]
- Water demand - mixing energy [Marquardt, 2001]
- Proctor test [NEN-EN 13286-2]
- Centrifugal consolidation [Miller, 1996]
- Water demand – Japan – [Okamura, 1995]
- Rheology – Krieger and Dougherty [Mansoutre, 1999]

A description of each method is presented in the next subsections. Most methods determine the minimum amount of water necessary to fill the voids between particles in a packing. In this basic principle maximum packing density is achieved when all voids are filled with water, but no excess amount of water is available to surround the particles. Some of the methods determine this minimum water demand directly by mixing a paste with a very low water powder ratio (water, powder superplasticizer) and then slowly adding water until the point were all voids are filled is reached. Other methods calculate the minimum water demand from a relation found by mixing and testing pastes with a water powder ratio higher than the water demand. Figure 1.

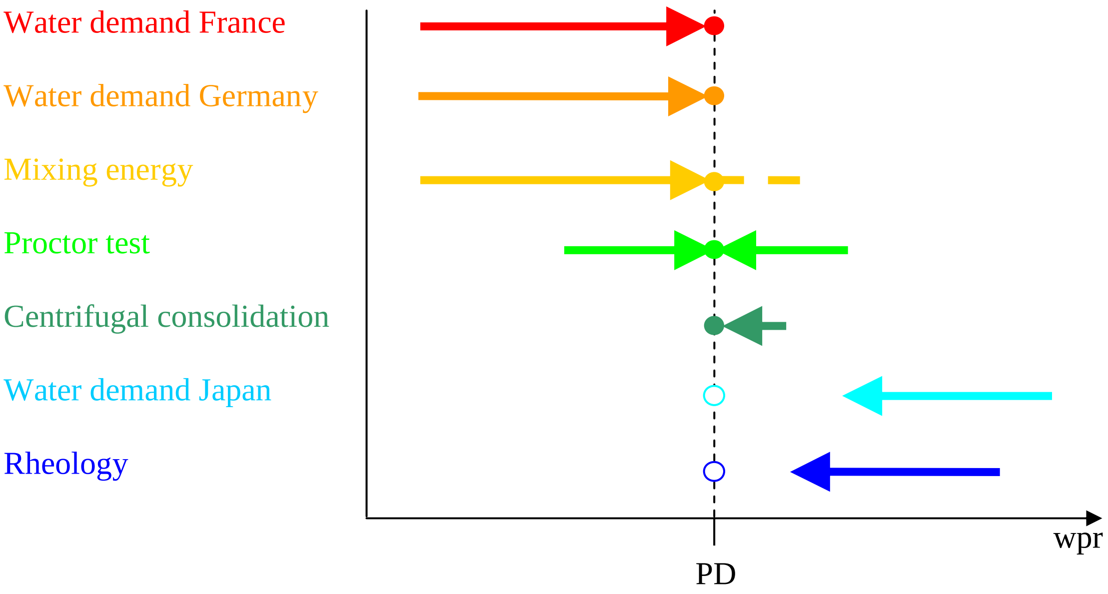

Figure 1 Direct (●) and indirect (○) determination of the minimum water demand depending on the water powder ratio of the paste(s) during the test.

Methods to determine packing density and/or water demand of fine particles not evaluated in this paper:

- Vicat test [Hunger]
- (Gas) pressure filtration [Mansoutre]
- ….

Water demand France

This method aims at finding the minimum water dosages to produce a thick paste. A slightly lower amount of water should give a humid powder [Larrard]. Since the water demand measurement is influences by the type and amount of superplasticizer, it should be dosed as a percentage of the powder dosage. A mass of 350 gram of powder is mixed with water according to the next procedure: The amount of water at minimum water demand is estimated (Formula 1) and mixed with the superplasticizer. First the water (including SP) is added to the mixing bowl, then the powder. The paste is mixed for 1 minute at low speed, then rested/ scraped and subsequently mixed for one minute at high speed. During the mixing at high speed an extra amount of water is added using a pipette to adjust the workability of the paste. The test is repeated with a slightly lower amount of water than in the first test. The packing density of the powder is calculated by formula 1, as average of two tests, in which the amount of water in the pipette was lower than 5 grams.

$$PD = \frac{1000}{1000 + \rho \frac{M_w}{M_p}} \qquad (1)$$

PD = packing density
M_w = mass of the water
M_p = mass of the powder
ρ = density of the powder in kg/m3

The difficulty of the method is recognizing the transition from a humid powder to a thick homogeneous paste, especially when the humid powder forms a sticky non-homogeneous ‘paste’.

Water demand Germany

The method is based on the idea that a fine, low-cohesion particle packing without a load, then and only then can be compacted to a powder specific value, when the water content is sufficient to fill all the voids in that packing. With humid but not yet saturated particle packings of fine powders, the surface tension (capillary forces) will block the water from surrounding the particles. At the saturation point the capillary forces will disappear and the particles can easily be packed to the characteristic highest packing density. Not the compaction energy is important, but the compactability. The transition from ‘not yet compactable’ to ‘compactable’ can occur by adding just 0.1 grams of water to a sample containing 100 gram of powder. An excess amount of water will also lead to possible compaction, but it will result in a lower packing density or possible bleeding. For this reason it is very important to approach the saturation point by carefully adding water according to the next procedure: Place 50 grams of powder in a plastic or metal container with a flat bottom. Water is added slowly by making use of a siphon/pipette while the humid powder is mixed with a steel blade or rod. The saturation point is reached when after repeatedly tapping against the container the powder surface levels off and starts to shine. The test should be repeated at least two times with a slightly lower amount of water. The final water demand is calculated according to formula 1 from the smallest amount of water of three tests.

A disadvantage of the test is that the method can only confidently be used when the the existence of air voids in the 'saturated paste' can be ruled out.

Water demand - mixing energy

When water is added to a powder it condenses on the particles to form capillary bridges (pedular bonds) localized at the particle contacts. In this way, agglomerates of particles are formed. The strength of the pendular bond increases with the liquid-vapor surface energy and depends inversely on the square of the particle diameter. At less than total saturation, the strength of the agglomerates increases with the amount of liquid and the surface energy of the liquid. The absence of internal liquid-vapor surfaces at 100 % saturation causes the strength to suddenly decrease at this point. [German]

The method described by [Marquardt] is based on the idea that the differences in internal pendular bond strength can be measured by measuring the mixing energy according to the following procedure: A powder volume of about 200 cm^3 is mixed in a mortar mixer (DIN EN 196, Teil 1), with a constant water supply of 1.5 ml/s during the entire mixing time, at a mixing speed of 140 rounds/minute. During mixing, the voltage, electricity consumption and the phase shift between the voltage and the electricity consumption of the mixer are registered to determine power use. The water demand of the mix is recorded as the water to powder ratio at which maximum power use is measured.

Proctortest

The proctortest is normally used to determine maximum mixture density of unbound and hydraulically bound mixtures used in road construction and civil engineering work; however, it can also be used on fine powders. In that case a powder is mixed thoroughly with a certain amount of water. The moist mixture is placed in a mould (diameter 100 mm, height 120 mm) in three layers, such that after compaction the sample is higher than the mould body. After placing each layer it is compacted by applying 25 blows of a 2.5 kg rammer dropped from a height of 305 mm above the mixture in such way that the blows are uniformly distributed over the surface of the sample. The extension of the mould is removed and the surface of the compacted mixture is carefully leveled off. After determining the mass of the sample (moist mixture) by weighing, the water content w is determined by drying according to EN 1097-5.

The compacted dry density of the mixture is calculated for each compacted sample by formula 2.

$$\rho_d = \frac{100\rho}{100+w} \tag{2}$$

ρ_d = dry density [$\mathrm{Mg/m^3}$]
ρ = bulk density of the sample after proctor compaction [$\mathrm{Mg/m^3}$]
w = water content of the mixture [%]

The dry densities obtained from at least five determinations with different water content are plotted against the corresponding water contents. A curve of best fit is drawn to the plotted points to identify the position of the maximum on this curve. The dry density at the maximum of the curve is considered to correspond to the maximum achievable packing density of the moist mixture.
Unfortunately, because of the necessary drying of the powder after testing to determine the water content, this method is not suitable do determine the packing density of cement very accurately.

Centrifugal consolidation

The particle packing density of a powder can be determined by centrifugal consolidation according to the following procedure: A paste, with a known composition, is mixed in a three-litre Hobart mixer. First, the dry powders are mixed for ten seconds after which the water and superplasticizer are added. The paste is mixed for 1 minute at low speed, then rested/ scraped for one minute and subsequently mixed for another minute at low speed. The paste is poured into 90 mm long test-tubes with an internal diameter of 22 mm. By determining the mass of the paste in the test-tube, the amounts of powder and water in the test-tube at the beginning of the test are known. The test-tube is then centrifuged for ten minutes at 4000 rounds per minute in a Dumee Jouan E82N Centrifuge with an internal diameter of ± 300 mm. By centrifuging the test-tubes, the particles in the paste are compacted and less amount of water is necessary to fill the voids in between the compacted particle matrix. Therefore, the total sample will possess an excess amount of water, which will occur as a water layer on top of the (compacted) paste. This water layer can be removed with a pipette, after centrifuging. By determining the amount of removed water, the amount of water and particles in the compacted sample are known and thus the packing density of the powder can be calculated at the applied compaction energy.

Water demand Japan

This method is based on the idea that the water demand of a mixture can be determined indirectly from a linear relationship between the relative flow area R_p, formula 3, and the water by powder ratio by volume V_w / V_p [Okamura 1995].

$$R_p = \frac{D^2 - D_0^2}{D_0^2} \tag{3}$$

D = the average spread diameter in a slump flow test

D_0 = the base diameter of the cone in a slump flow test.

When R_p would be zero, $D = D_0$ and no flow is initiated. This state is considered to be achieved when the amount of water in the paste is just sufficient to adsorb on the particle surfaces and fill all the voids in the particle system (saturation point). This saturation point which corresponds to a certain $V_\mathrm{w}/V_\mathrm{p}$ is called the retained water ratio β_p (or water demand). Since it is not possible to perform a slump flow test on mixtures with a water powder ratio close to the saturation point a number of mixtures with higher water powder ratios are tested and β_p is calculated from the linear relation between $V_\mathrm{w}/V_\mathrm{p}$ and R_p as the interception point at R_p is zero.

For this method, measurements were performed according to the following procedure:
A paste, with a known composition, is mixed in a three-litre Hobart mixer. First, the dry powders are mixed for ten seconds after which the water and superplasticizer are added. The paste is mixed for 1 minute at low speed, then rested/ scraped for one minute and subsequently mixed for another minute at low speed.The slump flow was determined by a mini cone test (upper/lower diameter 20/37 mm and height 57 mm) on a flow table (Tonindustrie) with a 300 mm diameter glass plate. The slump flow is taken as the average spread diameter, calculated in four directions.

Rheology – Krieger and Dougherty

In this method maximum packing density (ϕ_M) is determined indirectly by fitting the results of viscosity measurements of pastes to the Krieger-Dougherty equation (4)

$$\eta_r = \eta / \eta_c = \left(1 - \frac{\phi_s}{\phi_M}\right)^{-[\eta]\phi_M} \tag{4}$$

In which η_r is the relative viscosity, η is the apparent shear viscosity of the cement paste, η_c is the apparent viscosity of the liquid phase, ϕ_s is the volume fraction of the solids, $[\eta]$ is the intrinsic viscosity of the particles, and ϕ_M is the maximum packing volume fraction of the cement particles. η_c is assumed to be the viscosity of water at 20 °C, 0.001 Pa·s.

For this method, measurements were performed according to the following procedure:
A paste, with a known composition, is mixed in a three-litre Hobart mixer. First, the dry powders are mixed for ten seconds after which the water and superplasticizer are added. The paste is mixed for 1 minute at low speed, then rested/ scraped for one minute and subsequently mixed for another minute at low speed. A coaxial cylinder viscometer, PAAR Physica MC1, is used to determine the apparent viscosity of a paste. To avoid

slippage, a sandblasted cylinder with a diameter of 25 mm (standardized geometry: Z3) is used. The measurement is started 5 minutes after the beginning of the mixing procedure. The applied measuring sequence was adopted from [Weerdt 2007] and is shown in Figure 2. The solid contents and their apparent viscosities at a shear rate of 30 s^{-1} were fitted to the Krieger-Dougherty equation.

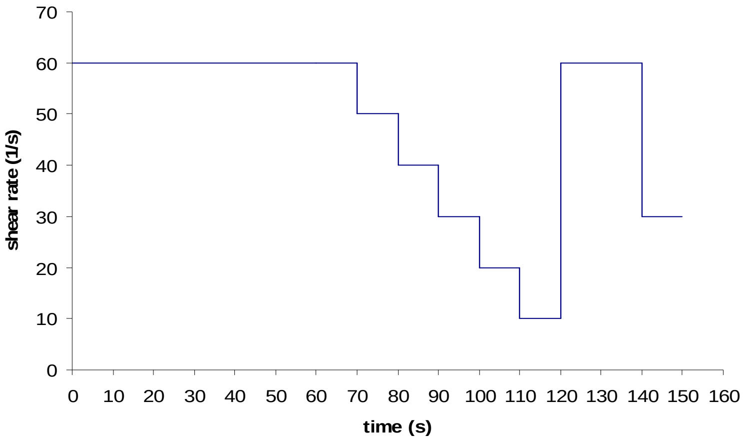

Figure 2 Measuring sequence, viscometer PAAR Physica MC1.

Results

In the section all measurements are presented for each method described in the previous section. When the measurement procedure differed from the standard procedure as described in the previous section, changes are reported. Furthermore, some comments on the estimated precision are presented for each method.

Water demand France

Changes to the method:

- Mixing procedure: Mixing of dry material + water and superplasticizer for 1 minute at low speed, 1 minute resting, ± 1 minute mixing at low speed while adding the last ± 5 gram of water.
- Amount of powder

Table 1 Results from determining the water demand by the French method.

CEM I 42.5 N [g]	Water [g]	Glenium 51 [g]	WCR [-]	Packing density [-]
1500	266.68	18	0.186	0.631
1500	268.39	18	0.187	0.630
1500	269.3	18	0.187	0.629
1500	268.93	18	0.187	0.629
1500	267.52	18	0.186	0.630
300	60.99	3.6	0.211	0.601
Technician 2				
1500	304.54	18	0.211	0.601

Estimated accuracy one mixing method, one technician: ± 0.002.
Measured water demand depends on the technician and on the mixing procedure (in this case: amount of material mixed).

Water demand Germany

Changes to the method:

- Mixing procedure – hand mixing is replaced by mixing in a Hobart mixer for homogeneity of the mixture.
- Amount of powder – in relation to bowl size, same amount as other methods.

Table 2 Results from determining the water demand by the German method.

Material	Powder [g]	Water [g]	Glenium 51 [g]	Hobart mixer	WCR [-]	Packing density [-]
CEM I 42.5 N	416.4	114.3		No	0.274	0.536
CEM I 42.5 N	1500	263.55	18	Yes	0.184	0.634
CEM I 42.5 N	1500	236.51	18	Yes	0.165	0.657
Quartz powder M10	1500	438.45	18	Yes	0.300	0.557

Estimated accuracy one mixing method, one technician: ± 0.02.
Measured water demand depends on the technician, the mixing procedure and the amount of tapping.

Water demand - mixing energy

Changes to the method:

- Amount of powder - in accordance to other methods
- Adding the water / mixing procedure: First 264 grams of water and the superplasticizer (according to the French method) are added. After one minute of mixing and one minute of resting the remaining water is added in drops.

Table 3 Results from determining the water demand by measuring mixing energy.

Material	Powder [g]	Water [g]	Glenium 51 [g]	WCR [-]	Packing density [-]
CEM I 42.5 N	1500	268.93	18	0.187	0.629
CEM I 42.5 N	1500	267.51	18	0.186	0.630
CEM I 42.5 N	300	74.84	3.6	0.257	0.552
Quartz powder M10	1500	412.34	18	0.283	0.572

Estimated accuracy one mixing method, one technician: ± 0.001
Measured water demand depends on the amount of material mixed (300 gram powder is assumed to be below the capacity of the mixer).

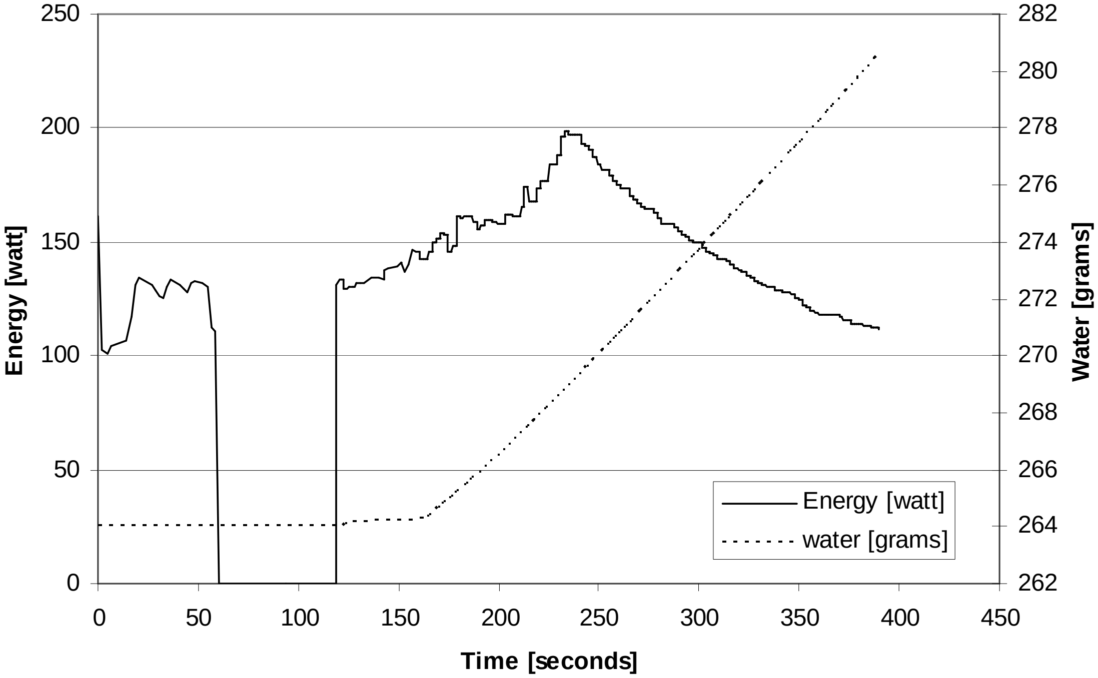

Figure 3 Results from 'mixing energy' test 1.

Proctortest

Table 4 Amount of sand and water in the Proctor mould after the test and the corresponding water cement ratio and packing density.

	Sand 0.125-0.25 [g]	Water [g]	WCR [-]	Packing density [-]	Maximum packing density [-]
Series 1	1400	309	0.221	0.576	0.587
	1433	302	0.210	0.587	
Series 2	1406	246	0.175	0.580	0.585
	1414	281	0.199	0.584	
	1424	287	0.202	0.585	
	1379	291	0.211	0.584	
	1400	310	0.221	0.577	

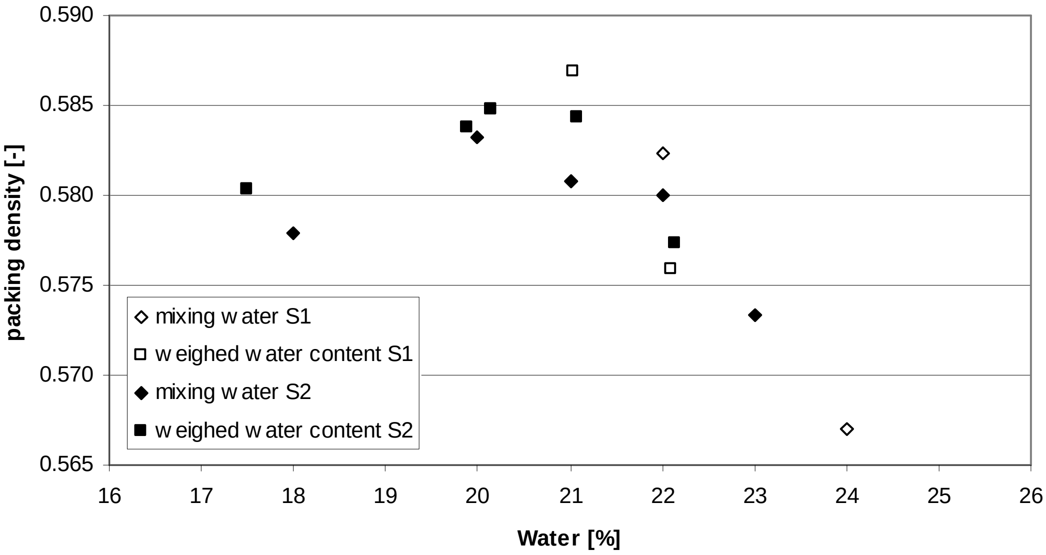

Figure 4 Amount of water in relation to packing density for series 1 (S1) and 2 (S2).

Estimated accuracy for one series, when drying the materials after the test: ± 0.002
When this method would be used on powders which react with water, such as cement, the accuracy is estimated to be ± 0.004, without drying the materials (Packing density is calculated from the initial water content of the mix).

Centrifugal consolidation

In all mixtures Glenium 51 was added as 1.2% by mass of the cement content.

Table 5 Results from determining the water demand by centrifugal consolidation.

Material	Powder [g]	WPR [-]	Packing density [-]
CEM I 32.5 R	1500	0.24	0.604
CEM I 32.5 R	1500	0.24	0.610
CEM I 32.5 R	1500	0.27	0.586
CEM I 32.5 R	1500	0.27	0.581
CEM I 32.5 R	1500	0.27	0.584
CEM I 32.5 R	1500	0.3	0.573
CEM I 32.5 R	1500	0.35	0.544
CEM I 32.5 R	1500	0.35	0.546
CEM I 42.5 N	1500	0.24	0.605
CEM I 42.5 N	1500	0.26	0.591
CEM I 42.5 N	300	0.27	0.586
CEM I 42.5 N	1500	0.28	0.582
CEM I 52.5 R	1500	0.24	0.599
32.5 R / 52.5 R	750 / 750	0.24	0.604
Quartz powder M10	1500	0.30	0.576
Quartz powder M10	1500	0.34	0.555
Quartz powder M10	1500	0.36	0.551
Sand 0.125-0.25	-	-	0.596

Estimated accuracy for one measurement: ± 0.004
Measured packing density depends on the water cement ratio of the mixture.

Water demand Japan

In all mixtures Glenium 51 was added as 1.2% by mass of the cement content.

Table 6 Results from determining the water demand of CEM I 32.5 R by the Japanese method.

CEM I 32.5 R [g]	Water powder ratio [-]	Flow value [mm]	Rp [-]	Packing density [-]	Estimated precision [-]
1500	0.24	177	21.8	0.685	± 0.15
1500	0.25	180	22.5		
1500	0.27	169	19.9		
1500	0.29	177	21.8		
1500	0.30	161	18.0		
1500	0.32	180	22.6		
1500	0.34	180	22.6		
1500	0.35	186	24.2		

Table 7 Results from determining the water demand of CEM I 52.5 R by the Japanese method.

CEM I 52.5 R [g]	Water powder ratio [-]	Flow value [mm]	Rp [-]	Packing density [-]	Estimated precision [-]
1500	0.24	170	20.1	0.726	± 0.03
1500	0.26	187	24.3		
1500	0.28	201	28.5		
1500	0.3	207	30.2		
1500	0.32	219	34.0		
1500	0.34	224	35.4		

Table 8 Results from determining the water demand of a mixture of 50% CEM I 32.5 R and 50% CEM I 52.5 R by the Japanese method.

CEM I 32.5 R [g]	CEM I 52.5 R [g]	WPR [-]	Rp [-]	Packing density [-]	Estimated precision [-]
750	750	0.24	21.0	0.713	± 0.05
750	750	0.26	28.1		
750	750	0.28	29.8		
750	750	0.3	30.3		
750	750	0.32	32.0		
750	750	0.34	39.8		

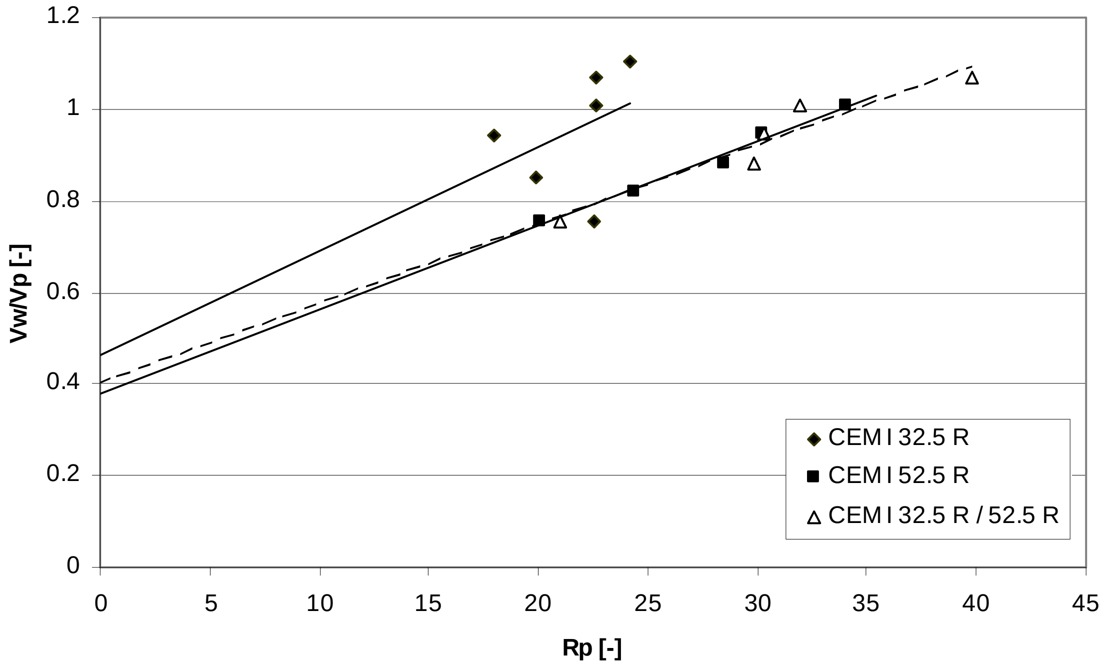

Figure 5 Determination of Vw/Vp at Rp=0 from the measured Rp values.

Rheology – Krieger and Dougherty

All mixtures were composed with 18 grams of Glenium 51.
The presented estimated precision is the possible error when fitting the results to the Krieger-Dougherty function.

Table 9 Results from determining the packing density of CEM I 32.5 R by rheology measurements according to Krieger and Dougherty.

CEM I 32.5 R [g]	Water powder ratio [-]	Apparent viscosity [Pa.s]	Packing density [-]	Estimated precision [-]
1500	0.24	1.217	0.63	± 0.03
1500	0.30	0.647		
1500	0.32	0.403		
1500	0.34	0.295		
1500	0.35	0.182	segregation	

Table 10 Results from determining the packing density of CEM I 52.5 R by rheology measurements according to Krieger and Dougherty.

CEM I 52.5 R [g]	Water powder ratio [-]	Apparent viscosity [Pa.s]	Packing density [-]	Estimated precision [-]
1500	0.26	0.806	1.3	± 0.7
1500	0.28	0.674		
1500	0.30	0.408		
1500	0.32	0.379		

Table 11 Results from determining the packing density of a mixture of 50% CEM I 32.5 R and 50% CEM I 52.5 R by rheology measurements according to Krieger and Dougherty.

CEM I 32.5 R [g]	CEM I 52.5 R [g]	WPR [-]	Apparent viscosity [Pa.s]	Packing density [-]	Estimated precision [-]
750	750	0.26	0.747	1.01	± 0.14
750	750	0.28	0.493		
750	750	0.30	0.427		
750	750	0.32	0.309		
750	750	0.34	0.221		

The tests presented in Table 12 are performed according to the measuring sequence in Figure 6:

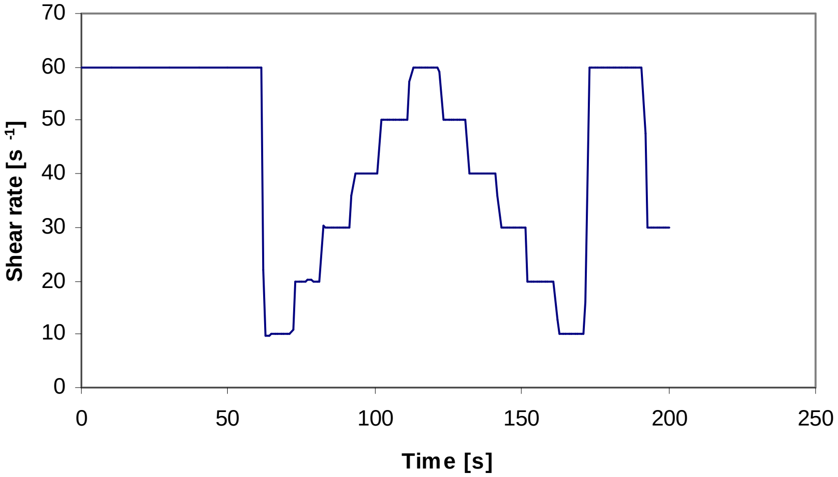

Figure 6 Alternative measuring sequence.

Table 12 Results from determining the packing density of CEM I 42.5 N by rheology measurements according to Krieger and Dougherty.

CEM I 42.5 N [g]	Water powder ratio [-]	Apparent viscosit [Pa.s]	Packing density [-]	Estimated precision [-]
1500	0.24	0.765	1.11	± 0.5?
1500	0.26	0.557		
1500	0.28	0.493		
1500	0.30	0.315		
1500	0.32	0.181		

The following seven tests were performed with a parallel plate rheometer (Paar physica). Glenium 51 was added as 1.2% by mass of the cement content.

Table 13 Results from determining the packing density of CEM I 42.5 N by rheology measurements (parallel plate rheometer) according to Krieger and Dougherty.

CEM I 42.5 N [g]	Water powder ratio [-]	Viscosity [Pa.s]	Packing density [-]	Estimated precision [-]
567.00	0.219	1.26	0.687	± 0.03?
562.28	0.224	1.10		
557.55	0.228	0.937		
552.83	0.233	0.735		
548.10	0.238	0.596		
538.65	0.247	0.467		
519.75	0.268	0.162	segregation	

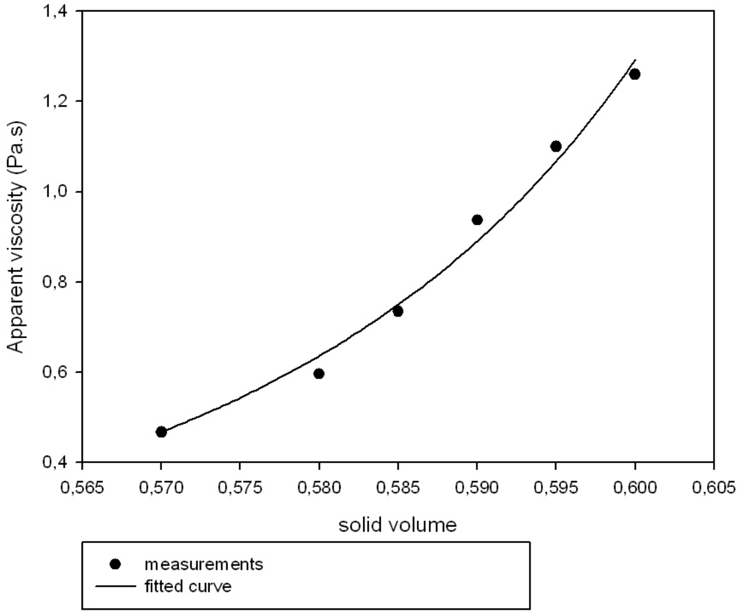

Figure 7 Fitting measurements by Krieger Dougherty equation for a shear rate 30 s^{-1}.

Discussion

Table 14 Comparison of the packing density measured by different methods and the estimated measuring accuracy.

CEM I 42.5 N	Packing density [-]	Accuracy [-]
Water demand France	0.630	± 0.002
Water demand Germany	0.64	± 0.02
Water demand - mixing energy	0.630	± 0.001
Centrifugal consolidation (wcr=0.24)	0.605	± 0.004
Water demand – Japan	0.69	± 0.15
Rheology – Krieger and Dougherty	0.63	± 0.03
	0.69	± 0.03
Sand	Packing density [-]	Accuracy [-]
Centrifugal consolidation	0.596	± 0.002
Proctor test	0.585	± 0.002

The Japanese method to determine water demand has the highest inaccuracy, Table 14. This is caused by the extrapolation towards much lower water powder ratios than the ones that can be used during the measurements. Low water powder ratios can not be used during the measurement, because of the large variation in slump flow of these measurements.

The other methods all give a reasonably accurate prediction of the water demand; however, they do not all comply with each other. Water demand measurements according to the French method and determining the mixing energy are in good agreement, predicting a packing density of 0.630 for CEM I 42.5 N. Centrifugal consolidation shows a lower packing density, which can be explained in terms of effective compaction energy. With a higher initial water powder ratio, the result of the packing density measurement is lower. In other words, the high amount of water in mixtures with a high water powder ratio is not completely pushed out of the sample during the 10 minutes of the centrifugal consolidation test. Analysis according to Figure 8 shows that the maximum possible packing density would be 0.634 if the test could be performed with a correspondingly low water powder ratio of 0.183. This method then is also in compliance with the French method and the determination of mixing energy.

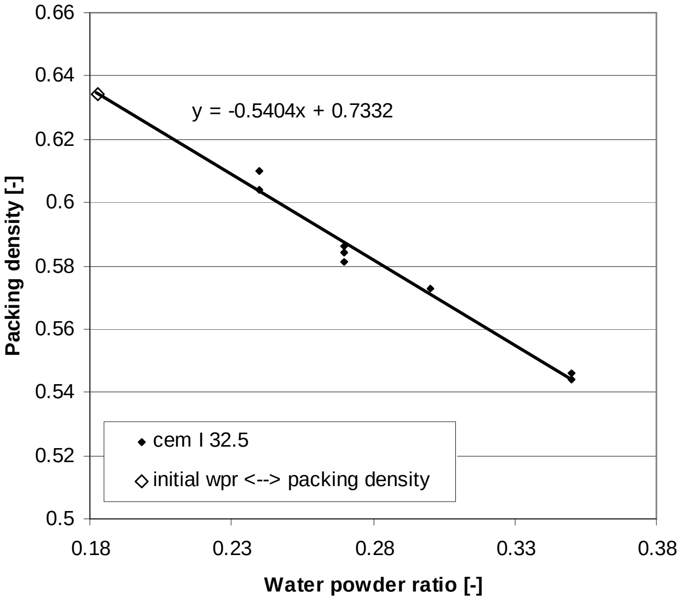

Figure 8 Extrapolation of centrifugal consolidation results to predict the minimum water content corresponding to the maximum packing density.

Determining packing density by rheology measurements and fitting to the Krieger Dougherty equation was not as accurate as expected from literature. Since the method is not based on the same physical relations as the other methods (The equation is an extrapolation of a fluid with a low amount of particles to a fluid containing crowding particles, instead of the concept where the fluid is only filling the voids in between a particle skeleton), this method will not be taken into account any more.

The final two methods, the Proctor test and water demand Germany, seem to be less accurate because of the same physical problem. In the Proctor test a curve is found, which shows the highest packing density at the ‘optimal’ water content. However, the state of total saturation is not achieved. At the highest measured packing density the voids of the powder skeleton are not completely filled with water and some air voids are left. For a ternary system containing powder, water and air, maximum packing density depends on the amount of compaction energy. Since the amount of compaction energy in the Proctor test is exactly prescribed, the test can be used to measure a comparative packing density (at a certain compaction level) for different types of powders; however, it can not be used to determine the maximum packing density. The German water demand test as described by Puntke suffers from the same problem. At the ‘maximum packing density’ and its corresponding amount of water, still some air voids can be present in the mixture (with fine powders, mixing becomes harder and achieving a homogeneous mixture without air voids becomes difficult). Because of the air voids present in the mixture, the differences in the amount of the applied compaction energy will result in differences in the reached packing density. Since the volume of the paste at the highest packing density is not taken into account in this method, the predicted amount of water is too low (extra water should be added to fill the air voids) and the packing density is too high.

Conclusions

In Figure 9 all test methods are compared to each other and for each method the water cement ratio corresponding to the measured packing density is presented. The only method which is certainly not able to predict the maximum packing density of a powder is the Proctor test. From its results it can be concluded that mixtures containing powder, water and voids are not suitable for predicting the maximum packing density. For this reason it is believed that the water demand test from Germany [Puntke] is also not able to predict the maximum packing density accurately. Though the existence of air voids in the water demand test from France and the mixing energy test can not completely ruled out, the results from these tests seem very good and also comply with the centrifugal consolidation test. The centrifugal consolidation test was believed to be a direct test; however, it proved not to be able to measure the maximum packing density. In order to determine the maximum packing density of a powder with this test at least three measurements and an indirect analysis of the packing density are necessary. With enough measurements the method is quite accurate and complies with the water demand from France and the mixing energy test.

Water demand testing from Japan and determining the water demand by rheology measurements (Krieger-Dougherty equation) are inaccurate methods and not suitable to make a precise estimation of the maximum packing density of a powder.

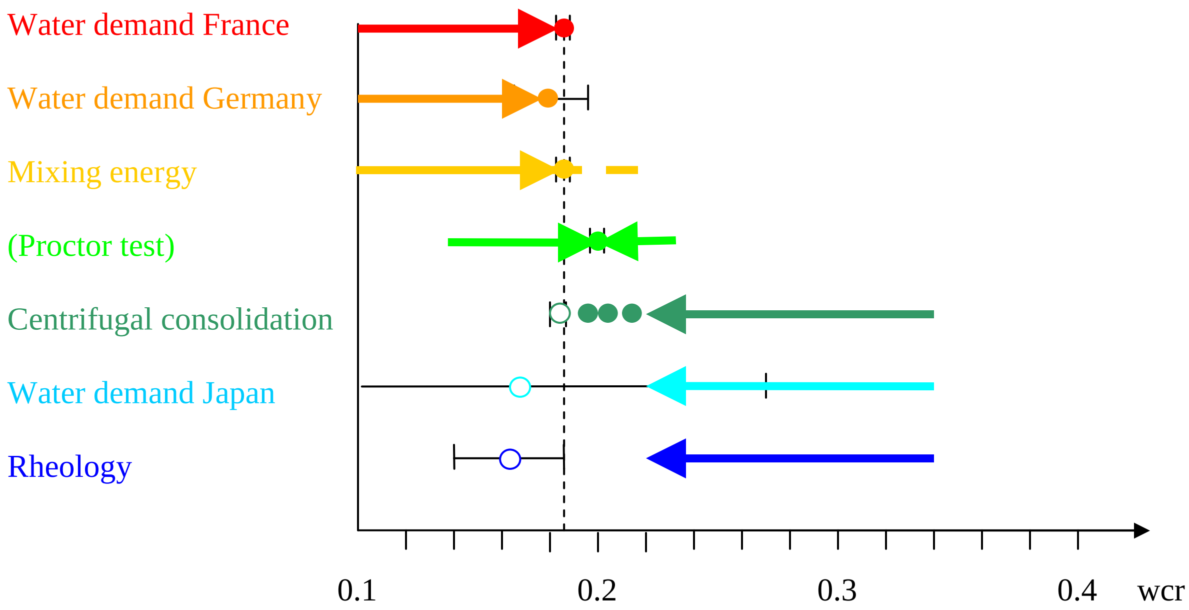

Figure 9 Schematic comparison of test methods to determine the water demand of cement.

To determine the packing density of powders, two methods can be recommended: Determining water demand by mixing energy and centrifugal consolidation. The mixing energy method is preferred above the French method, because it does not depend on the technician performing the test.
For both methods it is very important that mixing will result in a homogeneous paste. This is because no air voids or clumps should be present in the mixing energy method and for the centrifugal consolidation it is important to know exactly how much water and powder is put in the container. For both methods it should be taken into account that there could be differences in compaction level. With the centrifugal consolidation test, the compaction level depends on the water powder ratio. By doing several tests with various water powder ratios the maximum packing density at ‘infinite’ compaction energy can be estimated. Also the water demand test by determining mixing energy might result in different compaction levels for different powders, for instance, because of totally different mixing behavior of powders. While adding water, some mixtures transform from dry sandy mixtures, via a state with paste ‘balls’, to a paste, while other mixtures do not have this intermediate stage and transform directly in a ‘clump’.
An advantage of the centrifuge test is that the mixture can directly be used for other tests such as viscosity measurements or strength measurements. An advantage of determining water demand by measuring mixing energy is that the test method is fast and accurate.

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