A New Mix Design Method for UHPC based on Stepwise Optimization of Particle Packing Density

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Abstract:

This paper presents a new approach which is based on stepwise optimization of particle packing density for ultra-high performance concrete (UHPC) mix design. At first the method of superplasticizer-water solution demand measurement was used to find out the compatibility of the commercial raw materials of cements, silica fumes, quartz powders and superplasticizers regarding packing density and strength. As a result, the most suitable components and the optimum cementitious pastes were determined. Subsequently the optimum cementitious pastes with a fixed volume were combined with the blended aggregate which was formed by the combination of different grain aggregates of quartz sands or (and) crushed basalts. Based on the determination of the highest self-flowability and the lowest plastic viscosity for concretes the optimum mix proportions for UHPCs were recognized corresponding to the optimum blended aggregate. Four different self-compacting UHPC series with the different maximum grain sizes of 1 mm, 2.5 mm, 4 mm, 8 mm and compressive strength higher than 190 MPa were developed. In comparison to the reference mixtures the developed optimum UHPCs were much more effective in term of materials saving and performance improving.

Keywords: ultra-high performance concrete (UHPC), self-compacting concrete, particle packing density, concrete mix design, concrete rheology

1. Introduction

Mix design is one of the most important tasks of UHPC production. It focuses on optimizing the properties of concrete in fresh and hardened states. The particle packing density optimization for the granular ingredients of UHPC has been accepted as the key concept for mix design regarding improvement in workability, strength and durability (Schmidt and Fehling, 2005). Fibres can be added into UHPC for improving the ductility and energy absorption capacity of concrete, the term ultra-high performance fibre reinforced concrete (UHPFRC) is then used.

In reality the modification of the existing UHPC recipes of the available literature by trial and error is very popular for UHPC mix design. The success of this practice is limited due to different sources for the input materials. There are many challenges starting a UHPC design using regional commercial materials.

The main questions answered in this research were, with regard to the improvement of the packing density and strength, how the proper materials can be selected for UHPC production, how the optimum cementitious paste can be determined, how the optimum aggregate can be found out to obtain the optimum concrete considering the combination of the optimum paste and the optimum aggregate. The authors developed a systematic experimentation that is based on the stepwise optimization of the particle packing density (SwOPPD).

Without any special treatment, four different self-compacting UHPC series with different maximum grain sizes of 1 mm, 2.5 mm, 4 mm, 8 mm and compressive strength higher than 190 MPa, that were the required properties of concretes in this study, were developed.

There were many input materials at the beginning of this study. The characteristics of the used materials including cements, silica fumes, quartz powders and quartz sand are described in Table 1. In addition, three different polycarboxylate ether based superplasticizers (SP1, 2, 3) in liquid form (solid contents of 30 wt.%, densities of 1.05 g/cm^3) were used. Two crushed basalts with particle size of 4 – 8 mm (CrB1) and 2 - 4 mm (CrB2) (densities of 2.95 g/cm^3) were used as coarse aggregates

Table 1: Characteristics of used cements, silica fumes, quartz powders and fine aggregates.

Cement (particle size in μm)						
	Type		**Blaine fineness (cm^2/g)**	**D_{50}**	**Density (g/cm^3)**	**28 days Comp. Strength (MPa)**
Cem.No.1	CEM I 52.5 N – no C_3A		4500	7.7	3.1	61
Cem.No.2	CEM I 42.5 N – no C_3A		3800	10	3.1	56
Cem.No.3	CEM I 42.5 R – no C_3A		4300	10	3.1	57
Cem.No.4	CEM I 52.5 N – no C_3A		4300	9.5	3.1	61
Undensified silica fume						
	SiO_2 (%)	**C_{free} (%)**	**Loss on Ignition (%)**	**pH**	**Grain size without agglomeration (μm)**	**Density (g/cm^3)**
SF1	98	< 1	< 1	6 - 7.5	< 1	2.2
SF2	97	< 1	< 1	7 - 8	< 1	2.2
Quartz powder (particle size in μm)						
	SiO_2 (%)	**D_{10}**	**D_{50}**	**D_{90}**	**D_{99}**	**Density (g/cm^3)**
QP1	99	3.99	33.78	112.41	196.59	2.63
QP2	99	1.72	22.84	69.65	154.96	2.63
QP3	99	1.02	13.13	41.87	83.65	2.63
QP4	99	0.65	3.40	10.64	18.46	2.63
Quartz sand (particle size in μm)						
	SiO_2 (%)	**D_{10}**	**D_{50}**	**D_{90}**	**D_{99}**	**Density (g/cm^3)**
QS1	99	1323.54	1696.19	2093.64	2533.40	2.63
QS2	99	515.78	683.12	901.49	1081.73	2.63
QS3	99	103.32	148.53	204.50	283.8	2.63

2. Background

The available literature for UHPC mix design based on particle packing density optimization is not sufficient (de Larrard and Sedran, 2002; Geisenhanslüke, 2008; Wille et al, 2011; Yu et al., 2014). It should be noted that the compressible packing model of De Larrard (2002) still uses the packing of monosized classes to predict the packing of the concrete made up of different polydisperse grain groups with different wettability and adhesion. Also, in the modified Andreasen and Andersen particle packing model, which was used by Yu et al. (2014), the materials characteristics like shape, packing density, wettability and adhesion are not taken into account.

In this study, based on the idea that the void space between larger grains must be filled by smaller and smaller grains in order to improve the packing density of the granular mixture of concrete, the authors propose a rule of materials combination as follows:

- At the beginning of the mix design a collection of *n* different polydisperse grain groups are available. The group *(i+1)* is finer than the group (*i)*. Further it is assumed that the maximum grain size of the expected mixture is the same as the maximum grain size of the group*(i)*. The combination procedure is: Step 1, group *(i)* and the next finer group, group *(i+1)*, are combined. If the compatibility of the groups *(i)* and *(i+1)* is sufficient, the smaller particles will fill up the void between larger particles and this leads to a higher packing density of the

new grain group, corresponding to the optimum proportion of the group *(i+1)* to the group *(i)*. If not, the groups *(i)* and *(i+2)* will be combined and so on, until a new group composition with a higher packing density is found. As a result, the optimum proportion of the group *(i+k)* to the group *(i)* is determined. Then, step 2 can start. In step 2, the group *(i)U(i+k)* is combined with the next finer group, group *(i+k+1)*, and the process like in step 1 is repeated. The combination process is completed when the group with the smallest grain size, group *(n)*, is used.

- The wettability and adhesion of powders and aggregates are different, therefore, at first the rule is applied for powders system to find out the optimum cementitious paste, then the rule is used for aggregates system to determine the optimum blended aggregate corresponding to the optimum of the whole UHPC with a fixed volume of the optimum paste.

3. Testing Methods

3.1. Determination of the particle packing density of cementitious materials

For determining the packing density of powder in wet condition the method of water demand measurement, developed by Marquardt (2002), is an accepted method (Fennis-Huijben, 2008). In this method powder is mixed while water is added continuously. The power consumption of the mixing process increases with the amount of liquid and its surface energy. At 100% saturation, a thin water film surrounds each particle, the voids between the particles are filled with the liquid and the liquid pressure as high as the air pressure. With further adding of liquid the power consumption suddenly drops. The powder's packing density (β) is then calculated by Eq. (1), where V_P is the powder volume and V_L is the total volume of added liquid until reaching the maximum power consumption.

$$\beta = \frac{V_p}{V_L + V_P} \qquad (1)$$

However, in own experiments the authors found out that the real packing density of powders as well as the compatibility of powders and superplasticizers were only determined correctly when a superplasticizer-water solution was used as liquid. Therefore, superplasticizer-water solution demand measurement based on determining mixing energy was used for investigating the powder's packing density.

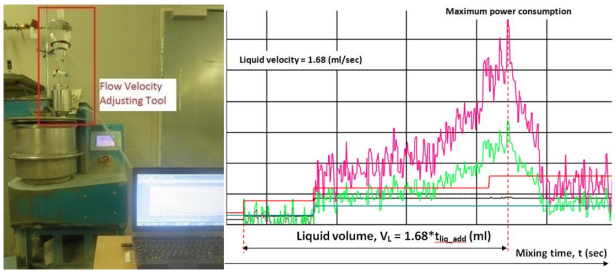

Figure 1: The equipment for the superplasticizer-water solution demand test (left), and the development of power consumption during addition of the solution (right).

In own experiments an Eirich intensive mixer was used, Fig. 1 (left). In case of the investigation of single powder a material amount of 5000 g was used. For the blended powder including cement the cement amount was 4000 g constantly. The solution with a superplasticizer-water ratio of 10 (wt.%) was added to the powder with a constant velocity of 1.68 ml/second. The test procedure was as follows: The mixing pan and mixing tool were controlled to rotate in

reverse order. The dry powder was mixed for 60 seconds at a mixing tool speed of 84 rpm. Subsequently the solution was added to the powder at a mixing tool speed of 150 rpm for 120 seconds. Then, the speed of the mixing tool was increased up to 450 rpm. The solution was added to the powder until a decrease of the power consumption was observed. Afterwards the duration of the solution adding (t_{liq_add}) from the beginning to the maximum power consumption was calculated. As a result, the value V_L in Eq.(1) is 1.68*t_{liq_add} (ml), as illustrated in Fig.1 (right).

3.2. Assessment of the particle packing density of concrete mixture

When the proportion of cementitious material, the water-cement ratio, the superplasticizer-cement ratio and the paste volume are constant, the combination of different aggregates will change the particle packing density of the whole concrete. In this context the ideal combination of aggregates can be determined by the best workability of UHPC in the spread-flow test. Furthermore, based on the study of Ferraris and de Larrard (1998) the plastic viscosity can be used to assess the packing density. Here the best combined aggregate can be recognized by the lowest plastic viscosity of the concrete in rheological tests. Both ideas were applied in this study to obtain the optimum particle packing density of the whole concrete.

3.2.1. The spread-flow test

Five minutes after mixing (deaeration purpose) the fresh concrete was placed in a mould (the Haegermann cone) on a dry glass plate, see Fig. 2. Then the cone was lifted straight upwards to allow a free flow of concrete without jolting. Two minutes after the cone lifting two concrete diameters perpendicular to each other were determined and the mean value was reported.

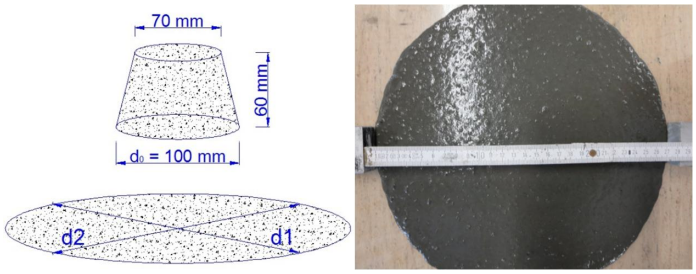

Figure 2: The spread-flow test to evaluate the flowability of fresh concrete using Haegermann cone.

3.2.2. The plastic viscosity assessment using a Rheometer

The Viskomat NT and Viskomat XL, produced by Schleibinger Geräte, were used to assess the plastic viscosity of UHPC. In the test for cementitious paste, the Viskomat NT accelerated the rotational speed from 0 to 80 rpm within 150 seconds. For the test of UHPCs with aggregates smaller than 5 mm the rotational speed increases from 0 to 15 rpm within 150 seconds. The Viskomat XL was used for the test of UHPC with a maximum grain size of 8 mm. It accelerated the rotational speed from 0 to 10 rpm within 150 seconds. Using the Bingham model the relation between torques T (N.mm) and rotational speeds N (rpm) can be described with Eq. (2).

$$T = g + h * N \qquad (2)$$

3.3. Specimen preparation and compression test

For each UHPC mixture, six cubes specimens of 100x100x100 mm^3 were produced to determine the compressive strength. Additional six cubes 100 mm and three cylinders with 100 mm diameter and 200 mm height were tested for the compressive strength and the modulus of elasticity of each optimum mixture. All specimens were cast without compaction. After casting, the specimens were covered with plastic sheet and stored at room temperature for 24 hours. Then the specimens were demoulded and cured in water at 20°C for 6 days. Subsequently they were stored at ambient laboratory conditions for additional 21 days. The compression test was carried out at a concrete age of 28 days. Before testing, both loading faces of all specimens were ground by a grinding machine. The loading rate was 0.6 MPa/s according to DIN EN 12390-3. The mean values were reported.

4. Results and Discussion

4.1. Determination of the water – cement ratio

According to the studies of Powers (1947) and of Mills (1966) the relation between water-cement ratio (W/C) and compressive strength (f_c) of mortar was calculated. Table 2 shows the results. Based on this calculation the water-cement ratio of 0.25 was chosen for further investigations, because it is not only low enough for an expected concrete strength of 180 MPa due to the strength improvement by silica fume pozzolanic reaction but also high enough to ensure a good workability of concrete.

Table 2: The relation between W/C and strength of mortar without supplementary cementitious materials.

W/C	0.29	0.28	0.27	0.26	**0.25**	0.24	0.23	0.22	0.21
Comp. Strength (MPa)	135	141	146	152	**158**	164	170	177	185

4.2. Materials selection

The compatibilities of the cements and the superplasticizers were recognized exactly by the improving packing density in the tests of superplasticizer-water solution demand. Cem.No.1 had the highest packing density of 63.7% when SP1 was used, see Fig. 3a. In addition, the combination of Cem.No.1 and SP1 also enabled the highest compressive strength, as shown in Fig. 3b. For these reasons, Cem.No.1 and SP1 were selected for further investigations. The silica fume SF2 was selected for further investigation due to a lower SP1-Water solution demand and a higher pozzolanic activity of the silica fume SF2 in comparison to SF1, as described in Fig. 3c.

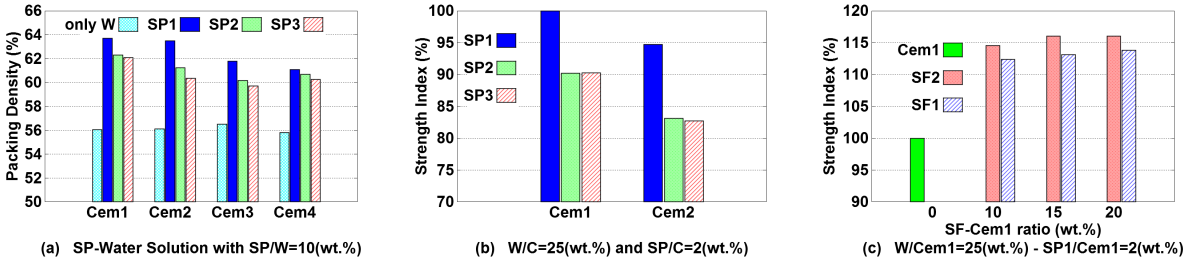

Figure 3: **(a)**: The influence of SP on the packing density of the cements using a solution with SP/W of 10 (wt.%); **(b)**: The influence of three superplasticizers on the 28 days compressive strength of Cem.No.1 & 2.; **(c)**: Influence of types and content of silica fumes on the 28 days strength of silica fumes – Cem.No.1 binders.

4.3. Mixture-proportioning of cementitious paste

The results of the combination of the quartz powders and Cem.No.1 are described in Fig. 4a. There was no improvement of the packing density due to the combination of QP4 and Cem.No.1. Thus, QP4 was not used for further investigation. The silica fume SF2 was then combined with the three optimum mixtures of Cem.No.1 and QP1, 2, 3. The increasing of SF2 improved the packing density of the cementitious material significantly. The higher the SF2 content in cementitious paste was, the thinner the water layer surrounding the particles got. Therefore, the decrease of the flowability of the cementitious pastes was observed in the experiments. For instance, Fig. 4b describes these phenomena for the cementitious powder of (QP3 + Cem No.1 + SF2). The NIMBUS program of multi-objective optimization (Miettinen et al., 2000) was applied to determine the SF2 content in the cementitious materials (QP+Cem.No.1+SF2) in such a way that the concretes have a high packing density cementitious material with a high volume ratio of water to powder. As a results, three optimum cementitious materials were found out. Based on these optimum cementitious materials, Viskomat NT was used to measure the viscosity of the pastes with the W/C of 0.25, the optimum SP1 content for each optimum cementitious material was specified, as shown in Fig. 4c. The optimum cementitious pastes were: Paste 1 (QP1:Cem.No.1:SF2:W:SP1 = 0.4:1:0.11:0.25:0.035), Paste 2 (QP2:Cem.No.1:SF2:W:SP1 = 0.35:1:0.115:0.25:0.030) and Paste 3 (QP3:Cem.No.1:SF2:W:SP1 = 0.3:1:0.12:0.25:0.030).

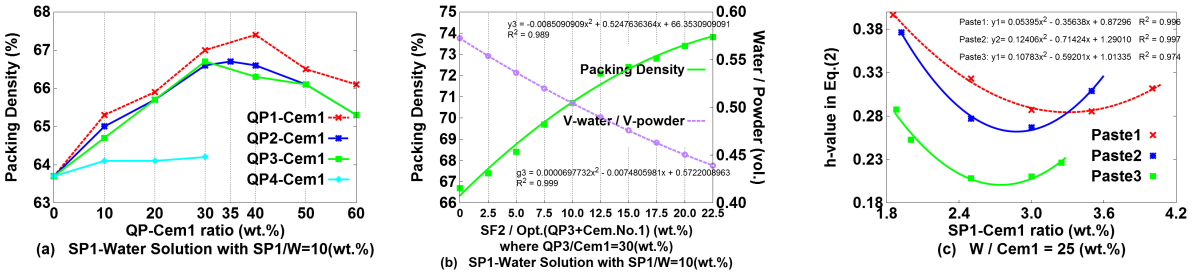

Figure 4: **(a)**: Determination of the optimum packing density mixtures of the quartz powders and Cem.No.1; **(b)**: The increase of the packing density of cementitious materials of (QP3+Cem.No.1.+SF2) (solid curves) and the decrease of the volume ratio of water to powder in concrete with W/C of 0.25 (dashed curves) by the increasing of SF2; **(c):** Influence of SP1 content on the plastic viscosity of the cementitious pastes with the W/C = 0.25.

4.4. UHPC with a maximum grain size of 1 mm

The amount of 720 kg Cem.No.1, which corresponds to 564.1, 551.2, 539.1 litres/m^3 of the Paste 1, 2, 3 respectively, was selected to ensure the self-flowing properties of concrete. QS2 and QS3 were combined. Regarding the compressive strength and viscosity the UHPC with a combination of QS2 (80%) and QS3 (20%) and Paste 3 is the best mixture, as shown in Fig. 5c.

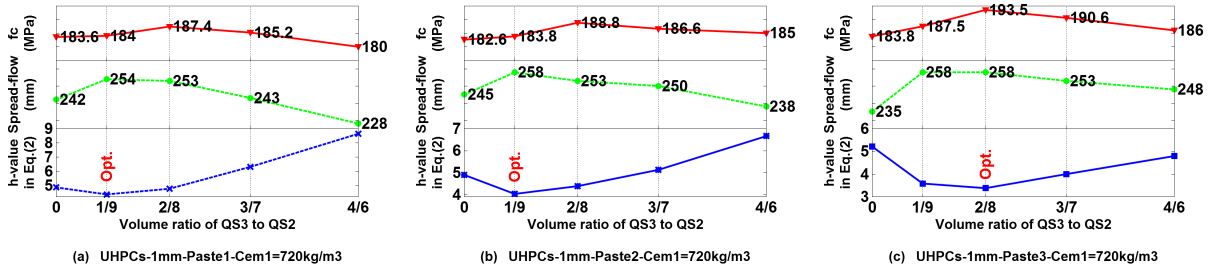

Figure 5: Relations between the plastic viscosity, self-flowability, compressive strength of UHPCs_1mm with different combinations of QS3 and QS2 - (a): Paste 1, (b): Paste 2, (c): Paste 3.

4.5. UHPC with a maximum grain size of 2.5 mm, 4 mm, 8 mm

For the investigations of UHPCs with a maximum grain size of 2.5 and 4mm Paste 3 with 720 kg/m³ of Cem.No.1 (or 539.1 litres of Paste3) was used. The cement content for UHPCs with a maximum grain size of 8 mm was 640 kg/m^3 (or 479.2 litres of Paste 3). Fig. 6 presents the processes of the SwOPPD for UHPC_8mm with the aggregate system of 4 components. It was clear to perceive that the number and the total surface area of the smaller aggregate particles increase significantly. However, the plastic viscosity, the self-flowability and the compressive strength of the concretes were improved notably. This can only be attributed to the increasing of the particle packing density leading to less void space between particles and a simultaneous remarkable increase of the paste layer thickness. The mix-proportion and the properties of the developed optimum UHPCs in this study and the reference mixtures, published by Fröhlich and Schmidt (2014), are summarised in Table 3. In comparison to the reference mixtures, the optimum UHPCs were much more effective in term of materials saving and performance improving (0-13% lower cement, 35-50% lower silica fume, 0-55% lower quartz powder, 25-35% lower superplasticizer, 2-14% higher comp. strength).

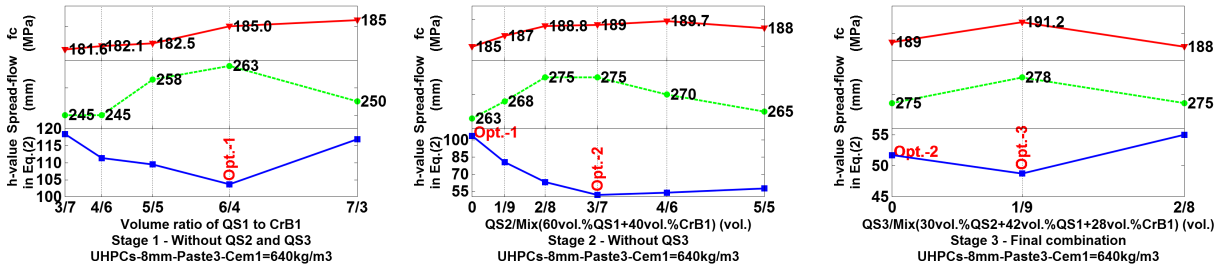

Figure 6: The process of the SwOPPD for UHPCs_8mm-Paste 3-CrB1 & QS1&2&3 (optimum volume ratio QS3/QS2/QS1/CrB1 = 10/27/37.8/25.2).

Table 3: Mix proportion and properties of the developed UHPCs in comparison to the reference mixtures.

		Mix-proportion (wt. ratio for paste and vol. ratio for aggregate)	Spread-flow [mm]	28d Comp. Strength [MPa]	28d - E [GPa]
Optimum mixtures	Paste	+ QP3/Cem.No.1/SF2/W/SP1=0.3/**1**/0.12/0.25/0.030			
	UHPC 1mm	+ Cem= 720 kg and QS2/QS3 = 80/20	258 (800*)	193.5 (183.3**)	53.4
	UHPC 2.5mm	+ Cem= 720 kg and QS1/QS2/QS3 = 54/36/10	283 (875*)	196.5 (189.8**)	54.9
	UHPC 4mm	+ Cem= 720 kg and CrB2/QS2/QS3 = 24/56/20	283 (840*)	197.4 (188.1**)	53.8
	UHPC 8mm	+ Cem= 640 kg and CrB1/QS1/QS2/QS3 = 25.2/37.8/27/10	278 (805*)	191.2 (185.6**)	57.0
Reference mixtures	M2Q	+ Cem= 832 kg and 2.5 vol.% steel fibre 9/0.15mm + QP/Cem/SF/W/SP=0.25/**1**/0.16/0.22/0.035 + QS 0.125-0.5mm = 100%	--	172.2	--
	B5Q	+ Cem= 650 kg and 2.5 vol.% steel fibre 9/0.15mm + QP/Cem/SF/W/SP=0.7/**1**/0.27/0.24/0.046 + QS 0.125/0.5mm / Basalt 2-8mm = 40/60	(800*)	187.4	--

(*) the slump-flow test results according to DIN EN 12350-8; (**) cylinder specimens

5. Conclusions

A systematic and highly effective approach for UHPC mix design was presented. The advantage of the proposed approach was convincingly verified by the success of the comprehensive investigations, in which four self-compacting UHPCs with different maximum grain sizes of 1 mm, 2.5 mm, 4 mm, 8 mm and compressive strength higher than 190 MPa at 28days age, were developed.

The output from the proposed approach is reliably the optimum particle packing density concrete mixture when considering the effect of materials characteristics and materials compatibility on improvement in the performance of concrete.

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