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ORIGINAL ARTICLE

Vacuum mixing technology to improve the mechanical properties of ultra-high performance concrete

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Abstract Ultra-high performance concrete is an important evolution in concrete technology, enabled by the combination of a good particle packing density, a suitable mixing procedure and compatible binders and admixtures. In the last decades a lot of research has been performed to explore the boundaries of this new type of concrete. Mixers equipped with a vacuum pump able to lower the mixing pressure from 1,013 to 50 mbar are an interesting way to improve the performance by lowering the air content. Profound research is necessary, because little is known about this technique of air content reduction. The influence of a reduced air content on the mechanical properties of ultra-high performance concrete is tested at The Magnel Laboratory for Concrete Research. This paper reports the results of the compressive strength, the splitting and bending tensile strength and the modulus of elasticity. All the mechanical properties after 28 days curing are improved by reducing the air content in the ultra-high performance concrete. An increase in compressive strength between 7 and 22 % is measured. The bending tensile strength increases maximum with 17 % and the splitting tensile strength gains 3–22 % in performance. Furthermore, the modulus of elasticity improves with 3–8 %. In conclusion, the air content can be controlled and a higher performance can be achieved by vacuum mixing technology. Finally, it is shown that the vacuum technology is not as effective in a 75 l capacity vacuum mixer as it is for a smaller vacuum mixer with a capacity of 5 l.

Keywords Vacuum mixing · UHPC · Air content · Mechanical properties

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1 Introduction

During the last decades, the interest in ultra-high performance concrete (UHPC) has grown significantly. Researchers all over the world have tried to explore the boundaries of UHPC and made useful contributions to improve this concrete. Currently, UHPC is used in a wide range of applications. Tests are done to protect important facilities as nuclear plants, high rise buildings and power plants from aircraft impacts, by the use of UHPC panels [1]. Furthermore, this type of concrete has an important aesthetic advantage [2]. UHPC also plays an important role in making offshore structures more cost-efficient [3]. The high mass and damping coefficient make UHPC ideal for the production of machine beds [4]. The advantage of this concrete is two folded, on the

one hand it is often made self-compacting, a useful characteristic to fill up complex formworks with a high reinforcement ratio. On the other hand the hardened concrete has a very good performance. This is seen in the improved mechanical properties [5] and the longer lifetime [6]. The higher durability [7, 8] makes UHPC also ideal for constructions in harsh environments. A comprehensive explanation on the mix design of UHPC can be found in literature [9–11].

In order to make UHPC a good mixing procedure should be applied. Parameters as mixing time, mixing speed, temperature, mixing sequence and the way of addition should be monitored carefully in order to obtain the desired properties of the concrete [12]. Also the amount of entrapped air plays an important role in the quality control of the mix and its level of performance. Some examples on the influence of entrapped air can be found in literature e.g. fluctuations in air content can change the compressive strength [9] and the workability [9, 13, 14] of the mixture. Furthermore, the accumulation of air bubbles under fibers or rebars can have a detrimental effect on the bond strength between the reinforcement and the surrounding concrete [15, 16]. Finally, water demand tests used to determine the wet packing density of powders, often neglect the amount of entrapped air [17]. This leads to an overestimation of the packing density and the properties calculated from it [18]. Thus, reducing the amount of entrapped air, will presumably give rise to a better estimation of the wet packing density of powders.

Due to these unfavorable effects research and industry has shown interest in methods to reduce the amount of air bubbles. One possibility is mixing the concrete under a reduced air pressure. Some earlier work from the National Bureau of Standards in Washington D.C., reported an increase of the flexural strength of vibrated mortar by decreasing the maximum void size. In their investigation a vacuum chamber reduced the pressure from 1,014 to 800 mbar [19]. Another research team at Vicksburg, Mississippi, equipped a drum mixer with a vacuum pump [20]. During mixing the pressure was reduced from 1,014 to 630 mbar. From their study they concluded that vacuum mixing had no beneficial effect on vibrated concrete. More recent work, performed at the center of building materials in Munich [21], obtained an increase in compressive strength from 175 to 250 MPa at 28 days for one UHPC mixture. In this case an intensive vacuum mixer was used [22]. The pressure in the mixing pan was lowered from 1,014 to 100 mbar, consequently the air content dropped from 8 to 1 %. Recently the authors confirmed the increased mechanical behavior for one reference HPC with different types of cement. A maximum increase from 110 to 125 MPa was obtained at 7 days. A decrease in air content from 3 to 0.5 % was reported by lowering the pressure from 1,013 to 50 mbar in the mixing pan [23].

Due to the strength increase associated with the air content reduction, it is interesting to investigate the possibility of replacing a heat curing by vacuum mixing. At this moment, a large amount of the prefab manufacturers are equipped with a steam curing chamber in order to demold the concrete earlier. Although these chambers are often limited to a temperature of 50 °C a notable increase can be expected on the mechanical properties of UHPC. Unfortunately the equipment and energy demand are expensive. Therefore, it would be interesting if this equipment can be replaced by a vacuum mixer.

2 Research significance

In literature limited data is available concerning the effect of vacuum mixing on the hardened properties of UHPC. According to the authors this data is restricted in two ways. First, the influence on the mechanical performance mainly focuses on the compressive strength [21, 23]. Therefore this paper tested the vacuum technology on five different UHPC mixtures which were selected out of literature and reproduced with Belgian materials. Besides the compressive strength, the effect of vacuum mixing on the splitting and bending tensile strength and Young's modulus is also examined. To have a more fundamental insight on the data, an attempt is made to link the results of the compressive strength to the solid concentration [18] and the effect on the tensile strength to the maximum void size [19]. Secondly, literature mostly gives results obtained with a 5 l capacity mixer [21, 23]. The data obtained with a larger vacuum mixer (75 l) indicate an increase of the compressive strength which is less pronounced compared with a small vacuum mixer (5 l) [23]. Consequently, one of the reservations of the concrete industry concerns the feasibility of this technology on an industrial scale. For this, the same

Table 1 Chemical composition of cement and silica fume 1 (M%)

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3
CEM I 52.5 N HSR LA	20.90	3.64	5.19	63.68	0.77	0.17	0.62	3.03
CEM I 42.5 R HSR LA	21.35	3.58	4.09	63.25	1.77	0.17	0.50	2.64
Silica fume 1	94.73	0.36	0.71	0.20	0.39	0.20	0.90	0.27

Table 2 Mix proportion of six different UHPC mixtures used in this work

kg/m^3	MIX 1	MIX 2	MIX 3	MIX 4	MIX 5	MIX 6
CEM I 52.5 N HSR LA	721	632	–	868	840	–
CEM I 42.5 R HSR LA			926			
Silica fume 1	226	198	231	217	–	–
Silica fume 2	–	–	–	–	336	–
Quartz sand 0/0.5 mm	992	434	1,018	–	339	–
Quartz sand 0/0.4 mm	–	–	–	912	–	–
Porphyry 2/4 mm	–	–	–	–	785	–
Basalt 0/4 mm	–	870	–	–	–	–
Quartz flour M400	180	158	–	–	84	–
Quartz flour M800	–	–	–	217	–	–
Superplasticizer 1[a]	28	27	42	44	–	–
Superplasticizer 2[a]	–	–	–	–	24	–
Water[b]	157	133	174	155	0	70
Ducorit® D4[c]	–	–	–	–	–	930
W/B	0.185	0.180	0.150	0.170	0.180	–
C/A	0.73	0.49	0.91	0.95	0.75	–
Origin	A	A	D	B	C	E

A University of Kassel [11], *B* University of Michigan [10], *C* Belgian Building Research Institute [8], *D* Scientific Division Bouygues [25], *E* premix [3], *C/A* cement to aggregate mass ratio, *W/B* water-to-binder ratio

[a] Suspension

[b] Water compensated for water present in superplasticizer and silica fume if applicable

[c] Composition not given by the private company

mixture is made in a 5 and 75 l capacity mixer. It is checked how long the vacuum time has to be prolonged for the 75 l capacity mixer, to obtain the same reduction in air content as in the 5 l capacity mixer.

3 Materials

In this project two superplasticizers (SP) were used. Both of them were a polycarboxylate ether. SP1 and SP2 had respectively a solid content by mass of 35 and 40 %. Most of the mixtures contained a densified silica fume (SF1) with 95.6 % SiO_2, a N_2-BET specific surface of 17.765 m^2/g, a d_{50} of 0.29 μm and a density of 2,232 kg/m^3. The particle size distribution is determined in a Zetasizer Nano ZS [24]. A sonification time of 5 min is applied in order to get a good dispersion. SF2 is a slurry with a solid content of 50 %. The quartz sand 0/0.5 and 0/0.4 had respectively a d_{50} of 342.0 and 187.0 μm. The filler M400 and M800 had a d_{50} of respectively 10.3 and 1.7 μm. The chemical composition of the cements and the densified silica fume is given in Table 1. CEM I 52.5 N HSR LA had a d_{50} of 10.46 μm, a Blaine fineness of 4,322 cm^2/g and a density of 3,077 kg/m^3. CEM I 42.5R HSR LA had a d_{50} of 18.59 μm, a Blaine fineness of 3,490 cm^2/g and a density of 3,170 kg/m^3.

The composition of the five UHPC mixtures used in this project as well as their origin can be found in

Fig. 1 A 5 l intensive vacuum mixer with inclined mixing pan. *A* The pin-agitator, *B* the vacuum pump, *C* mixing pan and outer protection ring

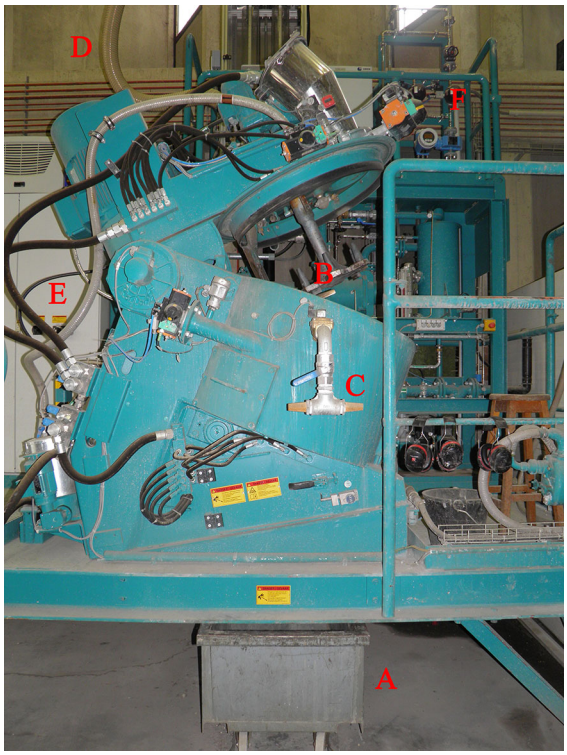

Fig. 2 A 75 l intensive vacuum mixer with inclined mixing pan. *A* discharge bucket, *B* pin-agitator, *C* filter to increase the pressure stepwise, *D* connection pipe to the dedusting machine, *E* connection pipe to the vacuum pump, *F* automatic water balancing system

Table 2. MIX 1-3-4 are reactive powder concrete [25] where the coarsest material is a fine quartz sand. MIX 2 and 5 are ultra-high performance concrete where a coarser sand as basalt 2/4 or porphyry 2/4 is used. The sixth mixture listed in Table 2 is a premix Ducorit® D4 from which the exact composition is unknown.

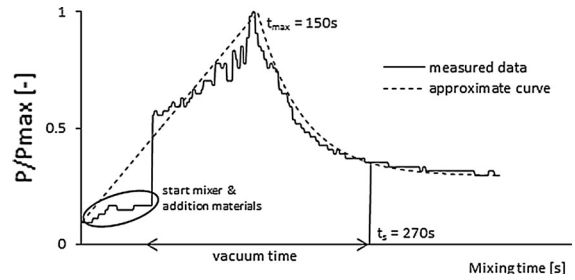

Fig. 3 Normalized power curve of the mixing process with an agitator speed of 6 m/s. t_{max} is the time to reach the maximum power and t_s the stabilisation time of MIX 3. The approximated curve is calculated in a similar way as Mazanec and Schiessl [28]

4 Mixing procedure

A 5 l capacity mixer, Fig. 1, is used to determine the effect of intensive vacuum mixing on the compressive strength, the bending and tensile strength and the Young's modulus. A 75 l capacity mixer is used to check if the mixing volume influences the ability of an intensive vacuum mixer to reduce the air content, Fig. 2. A pin-type agitator is chosen for its effectiveness to produce UHPC mixtures [26]. To eliminate the influence of mixing energy, the same circumferential speed at the extremity of the mixing blade is used in both mixers.

The mixing procedure is determined on the 75 l capacity mixer for MIX 3. In a first step, cement, silica fume and sand are weighed in a mobile scale and introduced in the mixer simultaneously while it is rotating. The dust produced during this discharge is removed by a dedusting machine. The dry powders are mixed during 15 s. In the next 20 s the water is automatically added at a mixing speed of 1.6 m/s and the superplasticizer is manually added to the mixture. This is followed by an intensive mixing period. The duration is determined based on the powercurve [27], for which the agitator speed is kept constant at 6 m/s, Fig. 3. The stabilisation time is considered to be reached when the curve has a gradient of −0.0006. Based on Fig. 3, the authors chose a hybrid mixing procedure. This consists of an intensive phase for

150 s at a speed of 6 m/s until the maximal power is reached and a slow phase for 120 s at a speed of 1.6 m/s until stabilisation. The speed and the procedure are adopted from literature [28] and gave a good workability for MIX 3. All the other batches are produced according to this protocol despite of the difference in composition or mixing volume. In case of the 5 l capacity mixer, the water is also added manually just before the addition of the superplasticizer. Furthermore the mixer started to rotate when all dry materials were inside.

In case of vacuum a reduction from 1,013 to 50 mbar is established at the moment of the intensive phase until the end of the mixing procedure. The final pressure is reached after approximately 30 s in both mixers. In total, a lowered air pressure is present in the mixing pan during 270 s. In case of the 75 l capacity mixer an automatic filter incrementally increased the air pressure back to 1,013 mbar, Fig. 2. At the end of the procedure the fresh concrete is discharged in a bucket. Next an automated cleaning system removes all the rest inside the mixer. For the 5 l capacity mixer the pressurization, the discharge and the cleaning is done manually. If the vacuum time is longer than 270 s the slow mixing phase is prolonged until the desired vacuum time is reached.

5 Sample preparation and methods

5.1 Fresh air content

The air content of fresh UHPC is determined according to the pressure gauge method described in EN 12350-7. In this test a known volume of air at an established higher pressure is allowed to equilibrate with the known volume of concrete in a sealed container. The drop in pressure, measured in the high-pressure air chamber, can be related to the amount of air within the concrete based on Boyle's law [29]. The test is preformed within 5 min from the completion of mixing. For each batch one test is done. As the concrete is self-compacting no vibration energy is applied. In this project only dense aggregates are used, consequently no air in the interconnected porosity within the aggregate particles will be compressed. For UHPC the fresh air content ranged between 0 and 5 vol%, in this range a gradation of 0.1 vol% could be read. Furthermore, the test has a reproducibility limit of 1.3 %. For the 75 l capacity mixer a container with a volume of 8 l has been used. In case of the 5 l capacity mixer a 1 l container has been used.

5.2 Hardened air content

The air cavities of the hardened concrete are determined by a Rapidair 457 air void analyzer according to ASTM C457-linear traverse method. Two types of samples are tested in this work. For the characterization of the different mixtures slices of 100 × 100 × 10 mm are cut from the middle of cubes with sides 100 mm. To test the influence of the maximum void size or the total air content in the fracture surface on the bending tensile strength, slices of 40 × 40 × 10 mm are made. The tested surface corresponds to a ±5 mm deep strip across the fracture surface and closest to the tension surface. The preparation of the surface comprises three phases. First, one side is polished to provide a perfectly planar and smooth surface. Next, a binary image is obtained by coloring the surface black with a marker in one direction and filling the voids with a dry white powder ($BaSO_4$) having an average diameter of 2 μm. Holes present in aggregates are painted black with a fine tipped marker pen in a final step. The measurement is performed on an area of 25 × 25 and 50 × 50 mm for samples with sides 40 and 100 mm, respectively. This area is subdivided in 10 traverse lines to cover a total length of 2,413 and 12,500 mm respectively. The measuring range is 10 μm to 3.5 mm. The threshold value is kept constant and based on the experience of the operator. Furthermore, the paste content is deduced from the known volumes of components added to the mixer. The result of every sample is an average of 4 measurements. The sample is turned 90 °C between individual readings. A good reproducibility limit between 0.2 and 0.62 % is found in literature [30].

5.3 Sample preparation for hardened concrete properties

The compressive strength is determined on cubes with sides 100 mm according to NBN EN 12390-3. The top layers of these cubes are not cut off to remove possible weak zones. The specimens are stored in a climate room for 48 h at a relative humidity of 90 ± 5 % and a temperature of 20 ± 2 °C before they are demolded.

Until the age of testing they are put back in the same climate room. After 28 days the tests are performed on a press of type MFL BP 600 V. The tensile bending strength is done according to NBN-EN 196-1 (2005), the prisms are stored for 2 days in the climate room before demolding. Thereafter, they are kept under water until the age of testing. After 28 days they are tested in a three point bending machine of walter + bai at a speed of 50 ± 10 N/s. The splitting tensile strength is determined following NBN EN 12390-6 on cylinders with a diameter and a height of 50 mm. The same curing and testing age is used as for the prisms. The Young's modulus is investigated conform NBN B 15-203 (1990) on cylinders with a diameter of 100 mm and a height of 200 mm. The same curing and press is used as for the cubes. At 21 days the cylinders are grinded to obtain two smooth surfaces. The tests are scheduled at an age of 28 days.

5.4 Heat treatment

A heat curing is often performed in order to obtain the full potential of the mixture by accelerating the hydration process. For this the specimens are positioned 2 cm above a bath that is held at a constant temperature of 90 °C in a sealed container for 2 days. The effect of it is checked for the compressive strength, the splitting tensile strength and the Young's modulus. This data is then compared with the strength increase due to an air content reduction to check if the latter is able to replace the heat curing.

6 Results and discussion

6.1 Influence of vacuum mixing on the air content

6.1.1 Introduction

This section summarizes the influence of vacuum mixing on the hardened and fresh air content. The correlation between both types of air content is also checked. This is interesting as the fresh air content is often measured and used as an acceptance criterion for the frost durability of the concrete on site. However other parameters such as the spacing factor are known to be much more related to the frost durability [31]. Therefor a lot of work is done to correlate the hardened air content determined with the microscope and the fresh air content determined with a pressure meter. A good overview is given by Roberts [29]. For low air contents, as is the case in this paper, generally a good correlation is reported in literature [29, 31].

Furthermore, each test is used to explain a specific property of UHPC. It is logic to use the fresh air content for effects dealing with workability and rheology [13, 14]. The hardened air content is useful in discussions concerning mechanical properties [9, 10] and durability [31].

According to the authors, a more profound distinction for the hardened air content can be made. One should determine this parameter in such a way that it is representative for the examined concrete property. Air void analyzes are useful when the property is determined mainly by one surface. Therefore, this technique can give a representative air content for the splitting and bending tensile strength. For these tests an analysis should be performed on the failure surface [19, 32, 33]. Computed tomography (CT) gives the air content of an entire volume. It is thus a good tool to determine a hardened air content representative for the compressive strength and the Young's modulus. Unfortunately, CT-scans are rather expensive compared with an air void analysis and the specimens need to be very small in order to get a good resolution [34]. Therefore, large amount of CT-scans should be performed to have a representative air content for the compressive strength determined on a cube with size 100 mm. This is the reason why the authors did not use the technique to explain the increase in compressive strength and Young's modulus. Instead the evolution of the solid concentration [17] is used.

6.1.2 Fresh and hardened air content

Table 3 gives a summary of the fresh and hardened air content. The fresh air content is tested for five mixtures. The hardened air content only for MIX 1-2-5-6. The initial fresh air content (NV) ranged between 4.7 and 3.2 %, the hardened air content between 4.9 and 2.5 %.

Since the measurements of the air content have been performed by different operators, it is necessary to evaluate the reproducibility of the data. The standard deviation (σ) of the fresh air content is situated between 0.1 and 0.5 % which is well under the reported value of 1.3 % in Sect. 5.1. The standard deviation of the hardened air content ranges from 0.2

Table 3 Effect of vacuum mixing on the hardened and fresh air content

	MIX 1		MIX 2		MIX 4		MIX 5		MIX 6	
	NV	*V*	NV	*V*	NV	*V*	NV	*V*	NV	*V*
A_{fresh} (%)	3.9	0.7	3.2	1.5	4.7	1.1	3.9	1.4	4.0	1.7
σ (%)	0.3	0.2	0.5	0.4	0.3	0.2	0.1	0.1	0.4	0.5
#Specimens (−)	12	20	6	11	4	8	2	4	4	9
Δ_{fresh} (%)	3.1		1.7		3.6		2.5		2.3	
A_{hardened} (%) (size ≤3.5 mm)	4.9	1.6	2.5	1.3	–	–	3.1	1.0	3.8	1.8
σ (%)	0.5	0.5	0.2	0.2	–	–	0.0	0.0	0.3	0.4
#Specimens (*)	5	6	3	3	–	–	1	1	3	5
Δ_{hardened} (%)	3.3		1.2		–		2.2		1.9	
A_{hardened} (%) (size ≤300 μm)	2.4	1.0	0.8	0.2	–	–	1.2	0.8	0.8	0.2
α (μm^{-1})	23.67	31.90	17.19	9.71	–	–	15.93	19.63	29.11	48.71
L (μm)	332	366	523	1257	–	–	524	699	255	213
μ_{MB} (Pa s)	30.3	31.7	36.2	41.3	–	–	–	–	–	–

NV non-vacuum, *V* vacuum, *A* air content, α specific surface, *L* spacing factor, *MB* modified Bingham [35]

to 0.5 % which is within the values reported in Sect. 5.2.

Next, it is observed that MIX 1 has a large amount of small air voids compared with the other mixtures, Fig. 4 and Table 3. It also contains a larger amount of small air voids (<300 μm) in comparison with vibrated concrete without air entraining agent [36]. In some cases MIX 1 has even a similar amount of small air voids (<300 μm) when it is compared with vibrated concrete with air entraining agent [36]. Consequently, the question rises how accurate the pressure gauge method can capture these fine air bubbles [29, 31] and give an accurate value of the fresh air content in case of MIX 1. Due to this uncertainty the fresh air content is not taken into account in the next discussion.

In general, vacuum mixing reduces the total fresh and hardened air content of all mixtures, Table 3. In case of the hardened air content the largest value under atmospheric pressure and the largest reduction by vacuum mixing is obtained for MIX 1. The first observation can be explained by the use of very fine quartz sand 0/0.5. This fine sand has a high specific surface area compared with basalt 2/4 or porphyry 2/4. Consequently, the air bubbles are more difficult to escape the concrete mass under the normal gravity force (1,013 mbar) [18]. It is the author's opinion, this effect outweighs the difference in viscosity as shown in Table 3 between MIX 1 and MIX 2 [37, 38]. The second observation can be understood by the suction force accompanied with a pressure drop from 1,013 to 50 mbar in the mixing pan. This force probably enables the air bubbles to overcome the viscosity and contact forces in all the mixtures, leading to a similar hardened air content after vacuum mixing for MIX 1-2-5-6 taking in account the standard deviation. To validate this explanation a more profound examination of the bubble movement in a viscous liquid is necessary. However, this would lead us too far from the aim of the paper. Therefore, the authors refer to literature which elucidates the complexity of this problem. Dimensionless parameters as the Reynolds number, Ëotvös number and the Morton number control the movement of the bubble [37, 38]. Furthermore, the sand particles provide extra contact forces and the mixing process changes the gas–liquid system significantly.

Generally the slope of the cumulative air void distribution of non-vacuum mixtures, Fig. 4, is more reduced by vacuum mixing for larger void sizes than for smaller void sizes. An exception is MIX 2 where an increase of the slope for the larger void sizes is noticed after vacuum mixing. This explains the evolution of the specific pore surface (α) in Table 3. By removing more larger air voids the proportion of the smaller voids becomes higher leading to a higher specific pore

Table 4 Effect of vacuum mixing on hardened properties of UHPC

MPa	MIX 1		MIX 2		MIX 4		MIX 5		MIX 6	
	NV	V	NV	V	NV	V	NV	V	NV	V
$f_{c,cub,28d}$	144	162	141	151	129	157	132	152	187	215
σ	4	6	8	7	8	8	4	5	3	15
#Specimens (−)	5	14	6	14	4	9	6	11	7	8
Δ_{NV} (%)	–	12	–	7	–	22	–	15	–	15
$f_{ct,splitting,28d}$	13.9	15.7	13.5	13.8			13.7	16.6	17.9	20.2
σ	1.7	1.9	2.1	1.2			0.9	2.7	1.5	1.5
#Specimens (−)	13	13	13	13			4	5	5	5
Δ_{NV} (%)	–	13	–	3			–	22	–	12
$f_{ct,bending,28d}$	21.4	24.9	19.6	21.7			19.1	20.1	25.8	25.7
σ	1.6	1.7	1.9	1.0			2.2	1.5	1.9	1.9
#Specimens (−)	5	10	5	5			5	5	5	5
Δ_{NV} (%)	–	17	–	11			–	5	–	–1
E-modulus	48,845	52,315	49,842	53,659			–		68,248	70,246
σ	317	6	721	702					2,609	1,039
#Specimens (−)	2	2	3	3					3	3
Δ_{NV} (%)	–	7	–	8					–	3

Δ_{NV} percentage strength increase relative to non-vacuum

surface. For MIX 2 more larger air voids are captured during vacuum mixing and the smaller air voids are reduced. This leads to an increase of the specific pore surface. In general the spacing factor (L) is increased by an air content reduction.

Finally a good correlation is obtained between the fresh and hardened air content for the UHPC mixtures, Fig. 5. The largest deviation from the unity line is found for MIX 1. A possible reason is the high amount of small air bubbles leading to an underestimation of the fresh air content. Roberts postulates this underestimation is unlikely to exceed 1 % air content [29]. For MIX 1 this 1 % of fresh air content would be enough to equal the fresh and hardened air content.

6.2 Vacuum mixing and its impact on the mechanical properties of UHPC

Until now the influence of vacuum mixing on the amount of entrapped air was examined. In this section the effect of an improved microstructure on the mechanical properties will be highlighted. The influence of vacuum mixing on the compressive strength, the splitting and bending tensile strength and the Young's modulus is investigated. An overview of the data is given in Table 4.

6.2.1 Compressive strength

Figure 6 gives the results of the compressive strength at an age of 28 days. A comparison is made between specimens mixed under different pressures (1,013 and 50 mbar).

All five mixtures show an increase of the compressive strength when a lowered pressure is applied in the mixing pan. An average strength gain is obtained between 7 and 22 %. The mixture with a low air content reduction, MIX 2, had a lower strength increase, Tables 3, 4. MIX 4, with the largest fresh air content reduction, profits the most of the vacuum technology. Furthermore, the standard deviation of the strength tests does not decrease by lowering the amounts of air voids. Other parameters than the variation in air content affect the deviation of the compressive strength.

It is remarkable that MIX 6 has a greater strength increase than MIX 1, although the air content reduction is smaller for MIX 6, Table 3. Possibly the obtained strength level plays an important role in the effect of vacuum mixing. MIX 6 attains a mean strength of 187 MPa under atmospheric conditions due to presence of the bauxite [40]. At this level air bubbles will be more critical as for the other mixtures,

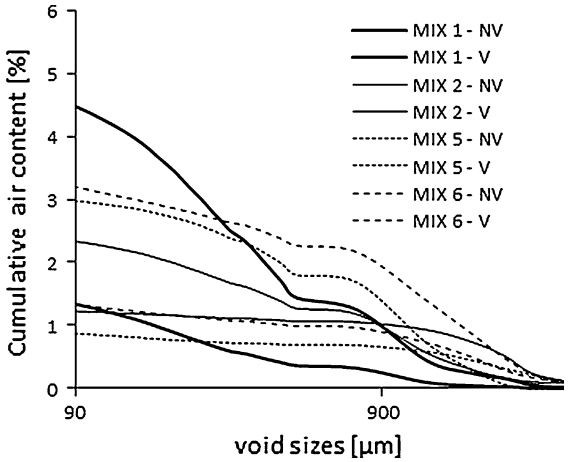

Fig. 4 Cumulative air void distribution curves of MIX 1-2-5-6. The void size is limited to 90 µm according to the definition of entrapped air by Mindess et al. [39]

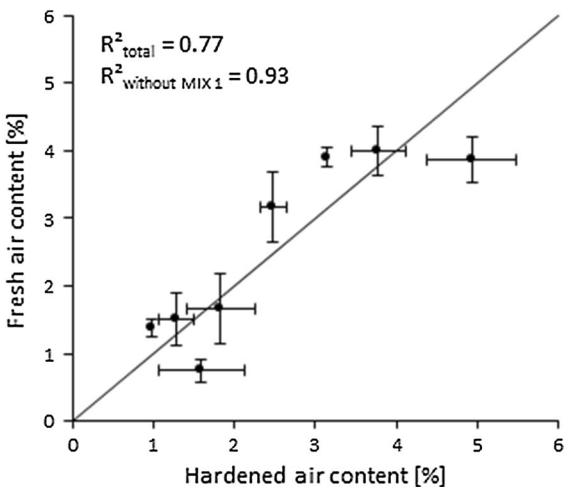

Fig. 5 Comparison between the air content determined with a pressure meter in the fresh stage with that determined with a Rapidair 457 air void analyzer in the hardened stage

even if the initial air content is slightly lower. This can be proven by the law of Feret [41], namely:

$$f_{\mathrm{ccub}} = K_1 \times \left(\frac{1}{1 + \frac{V_{\mathrm{w}} + V_{\mathrm{a}}}{V_{\mathrm{c}}}} \right)^2, \quad (1)$$

where K_1 is a factor depending on the strength of the cement at the age of testing (28 days), V_{w} is the effective water volume, V_{c} is the absolute volume of cement and V_{a} is the volume of air in the concrete. In case of MIX 1, 2 and 5, the mix proportioning of Table 1 together with the information of Table 3 enables to calculate K_1. In case of MIX 6 the authors assumed a *W/B* ratio of 0.12. This is necessary due to the unknown cement content of the mixture. Besides this, the absence of secondary cementitious materials such as silica fume is assumed. Together with the information in Tables 1 and 3, the reader can also determine the K_1 value of this mixture.

In conclusion, Table 5 confirms the importance of the strength level of the specimens made under atmospheric pressure. The law of Feret predicts an increase (Δ_{Feret}) of 19 MPa for MIX 1 and 28 MPa for MIX 6. The latter clearly benefits more from the vacuum technology. The air content reduction explains the increase in the compressive strength for all mixtures except for MIX 5. For this mixture only one air void analysis is preformed, Table 3. This could give rise to the higher discrepancy between $f_{\mathrm{exp,50\ mbar}}$ and $f_{\mathrm{Feret,50\ mbar}}$. Another explanation may be found in the type of silica fume. Different as for the other mixtures, a silica fume in slurry form is used. Therefore, deviations can be suspected between two batches depending on how well the slurry is dispersed during storage. As no continuous dispersion was applied during storage more silica fume particles will be found at the bottom part of the container. Consequently, the first batch at 1,013 mbar will contain less silica fume particles and have a lower compressive strength than the second batch at 50 mbar. In this case not only the air content is changing but also the amount of silica fume particles.

In case of MIX 1 and MIX 2 extra cubes are prepared to compare the effect of an air content reduction with a heat curing, Fig. 7. Clearly, vacuum mixing cannot fully replace the effect of a heat treatment on the compressive strength. However, most of the time other parameters are also considered in the selection of the procedure and the curing. For example the higher the strength level, the higher the standard deviation, Table 4. Also the cost of both techniques must be weighed against each other.

To conclude this section, it is interesting to put the results of the compressive strength at 28 days of MIX 1-2-4-5 from Table 4 (69 cubes) in function of their solid concentration ϕ. This is calculated with Eq. (2) [17]:

$$\phi = \frac{M/V}{\rho_{\mathrm{w}} \times u_{\mathrm{w}} + \rho_{\mathrm{a}} \times R_{\mathrm{a}} + \rho_{\mathrm{f}} \times R_{\mathrm{f}} + \rho_{\mathrm{cm}} \times R_{\mathrm{cm}}}, \quad (2)$$

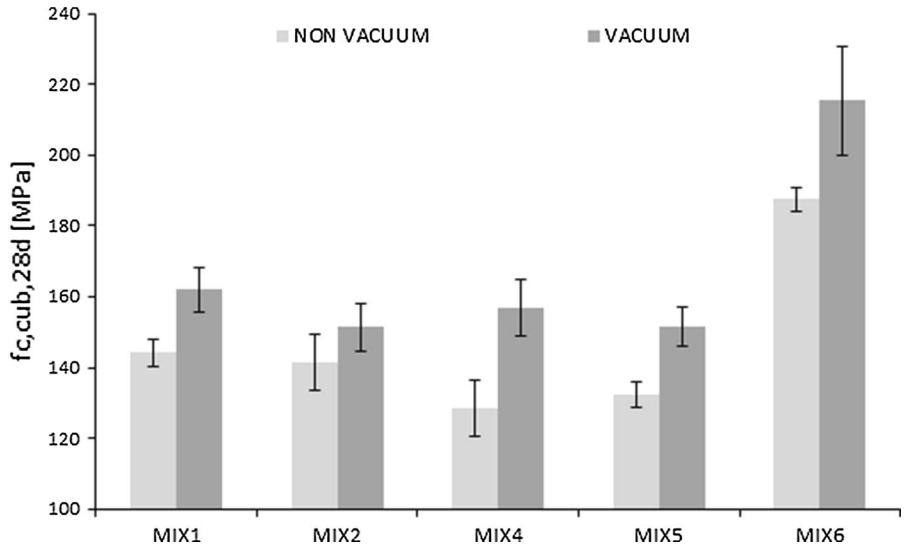

Fig. 6 Effect of mixing pressure on the compressive strength of UHPC

Table 5 Prediction of the strength increase of an air content reduction by the law of Feret

	$A_{1013\ mbar}$ (%)	$A_{50\ mbar}$ (%)	$f_{exp,1013\ mbar}$ (MPa)	$f_{exp,50\ mbar}$ (MPa)	K_1 (MPa)	$f_{Feret,1013\ mbar}$ (MPa)	$f_{Feret,50\ mbar}$ (MPa)	Δ_{Feret} (MPa)
MIX 1	4.9	1.6	144	162	401	144	163	19
MIX 2	2.5	1.3	141	151	359	141	149	8
MIX 5	3.1	1.0	132	152	329	132	141	9
MIX 6	3.8	1.8	187	215	461	187	215	28

A hardened air content, f_{exp} experimental compressive strength at 28 days, f_{Feret} theoretical compressive strength at 28 days, Δ_{Feret} strength increase according to the law of Feret

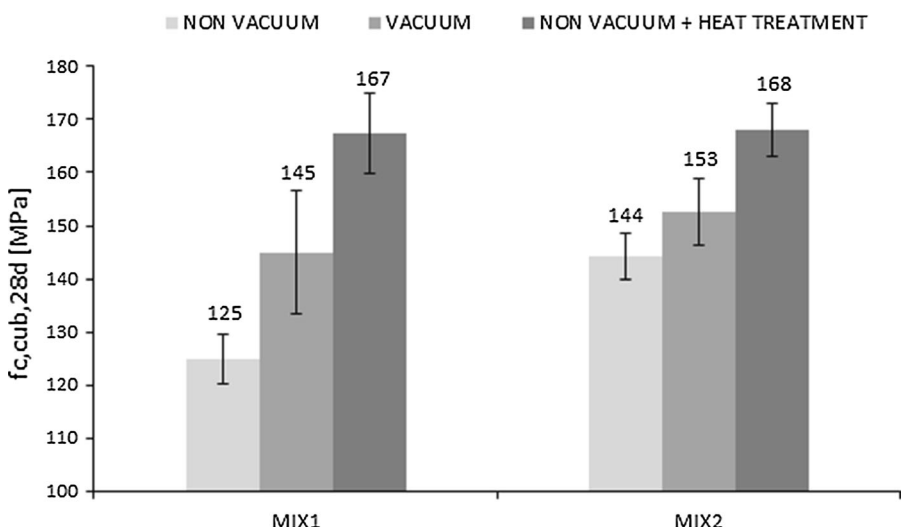

Fig. 7 Comparison between the effect of an air content reduction and a heat curing on the compressive strength of MIX 1 and MIX 2 at 28 days

where M is the mass of the specimen, measured before the mechanical test at 28 days, V is the volume of the specimen, ρ_w is the density of the water, ρ_a, ρ_f, ρ_{cm} are the respective solid densities of the aggregates, the fillers and the cementitious materials, u_w is the water to solid volume ratio of the mixture and R_a, R_f, R_{cm} are

the respective volumetric ratios of the aggregates, the fillers and the cementitious materials to the solid particle content.

By reducing the air voids inside the concrete specimen, the solid concentration is increased and more material is available to withstand the compressive force. This can be seen in Fig. 8 by comparing the colored points with the empty points of an UHPC mixture. The results of MIX 2 slightly deviate from the general trend. This can be seen by comparing the coefficient of determination of the linear fit with and without the points of MIX 2. Obviously, other parameters than the solid concentration alone influence the difference in compressive strength of cubes made with a different composition. Hydration of the binders and the quality of the transition zone also play a major role. This is the reason why no heat treated cubes are included in Fig. 8. In Table 6 the solid concentration of heat treated cubes mixed at 50 mbar of MIX 1 and MIX 2 are included. The mixtures are prepared with the same materials as the cubes in Table 4. It is clear that the solid concentration is not able to explain the increase in strength by a heat treatment but only by the air content reduction. This is a fairly obvious observation, because the former technique accelerates the hydration of the binders.

6.2.2 Splitting tensile strength

An average strength increase of the splitting tensile strength between 3 % (MIX 2) and 22 % (MIX 5) is obtained with vacuum mixing at 28 days, Fig. 9. For MIX 1 and MIX 2, some specimens are also subjected to a heat treatment. In case of MIX 1 a strength increase is obtained from 14 to 16 MPa. This result is similar as the effect of an air content reduction, but lower than the results of Graybeal [42], who obtained an increase from 19 to 24 MPa for his reactive powder concrete. The larger air bubbles after vacuum mixing in MIX 2 probably nullify the effect of the heat curing, Fig. 4. For MIX 2 no strength increase by heat curing or by an air content reduction is registered.

Table 6 Effect of mixing pressure and curing regime on the solid concentration of UHPC

ϕ (−)	MIX 1	MIX 2	MIX 4	MIX 5
Non vacuum	0.798	0.815	0.793	0.796
Vacuum	0.824	0.838	0.824	0.812
Vacuum + heat treatment	0.821	0.836	–	–

6.2.3 Bending tensile strength

The influence of a reduced air pressure on the bending tensile strength is illustrated in Fig. 10. Especially for MIX 1 a clear increase of the strength is determined, namely 17 %. For the other mixtures a less important increase is observed. Furthermore, the obtained values are remarkably higher than some results reported in literature for UHPC [43].

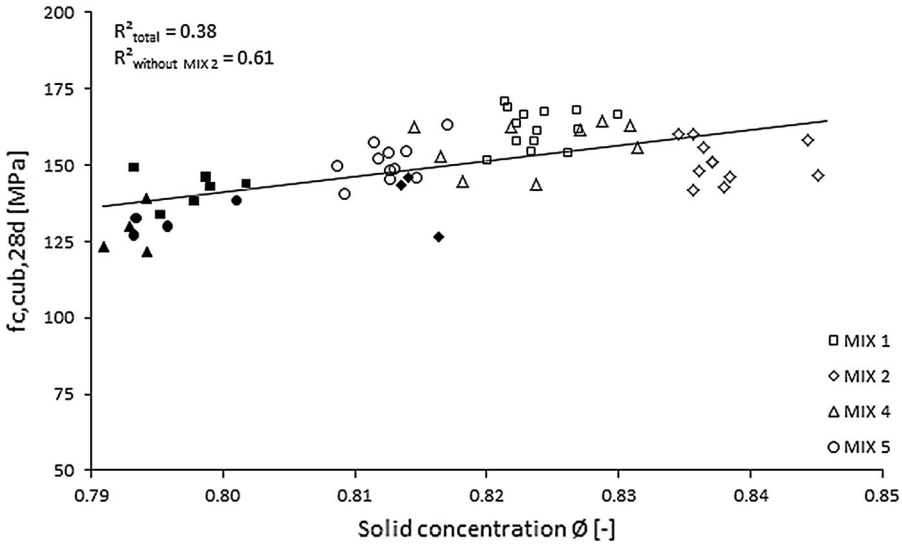

Fig. 8 Compressive strength of MIX 1-2-4-5 in function of the solid concentration (*colored points* correspond with non-vacuum and *empty points* with vacuum)

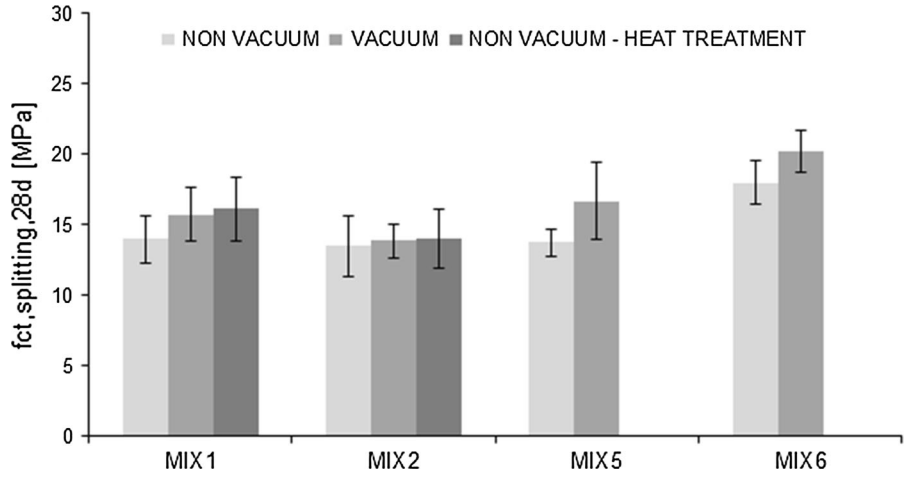

Fig. 9 Splitting tensile strength for MIX 1-2-5-6 at 28 days under different mixing pressures and curing regimes

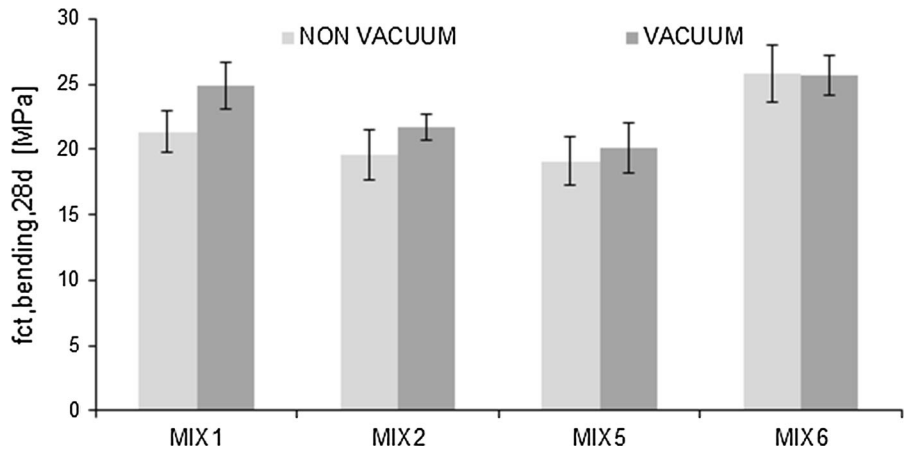

Fig. 10 Bending tensile strength of MIX 1-2-5-6 at 28 days under different mixing pressures

6.2.4 *Differences between splitting and bending tensile strength*

Figures 9, 10 and Table 4 highlight some differences in the increase of the splitting and bending tensile strength after vacuum mixing. For example, the bending tensile strength of MIX 1 and MIX 2 increases more by an air content reduction than the splitting tensile strength. However, the reader should remember that the obtained effect of vacuum mixing is not necessarily the same for the bending and splitting specimens. In order to investigate this, the authors first check if the total air content or rather the maximum void size of the failure surface gives the best correlation with the bending tensile strength. Therefore, one half of the prisms is held after the determination of the bending tensile strength of MIX 1-2-5 at 28 days and is prepared for air void analyzes. The maximum void size in the failure surface is determined under a stereoscope on the same samples. In total three samples are prepared per mixture and per mixing pressure.

In contrast to literature [32, 33] the best correlation is found between the total air content in the failure surface and the bending tensile strength, Fig. 12. A possible reason why the maximum void size gave a lower correlation, Fig. 11, can be related to the mixing procedure. Apparently, the procedure is not able to decrease the maximum void size under 1 mm, which is necessary to influence the bending tensile strength in a significant way, as mentioned by Birchall et al. [32]. Another difference with literature is the fact that the capillary pores are not taken into account in the total

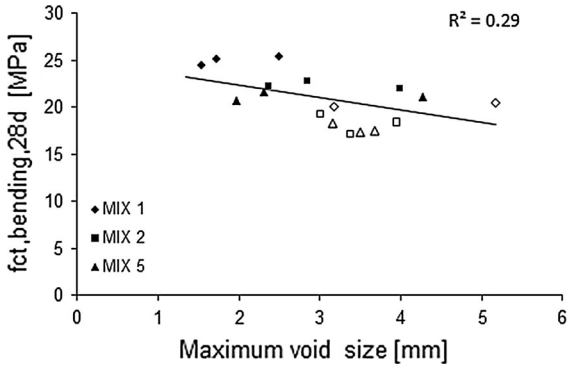

Fig. 11 Bending tensile strength of MIX 1-2-5 at 28 days in function of the maximum void size in the failure surface (*colored points* correspond with vacuum and *empty points* with non-vacuum)

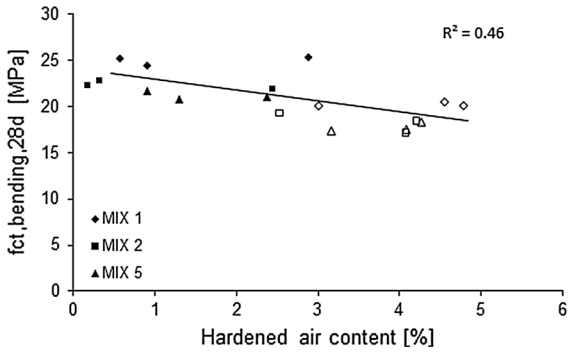

Fig. 12 Bending tensile strength of MIX 1-2-5 at 28 days in function of the total air content in the failure surface (*colored points* corresponds with vacuum and *empty points* with non-vacuum)

air content of Fig. 12. Hence, a better correlation is found between the bending tensile strength and the total amount of air voids instead of the sum of air voids and capillary pores. Furthermore, the coefficient of determination in Fig. 12 is rather low, which indicate that the air content is not the only parameter controlling the bending tensile strength. Factors relating to the cement-aggregate bond and the influence of unhydrated cement particles also control this property [19].

Another observation from Table 4 and Figs. 9, and 10 is the different increase of the splitting and bending tensile strength by an air content reduction. To explain this, the total air content in the failure surface of the splitting specimens of MIX 1 and MIX 2 is also determined with the Rapidair 457. Three samples are

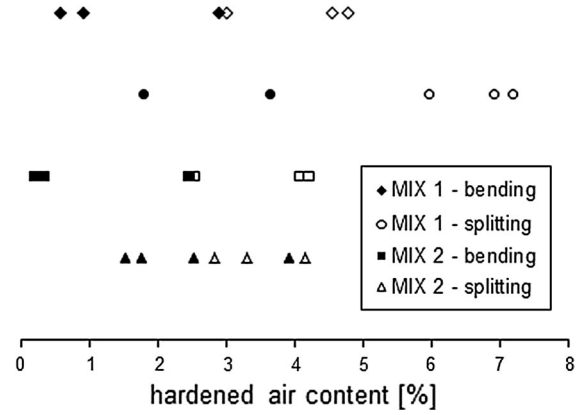

Fig. 13 Total air void content in the failure surface of the splitting and bending specimens of MIX 1 and MIX 2 (*colored points* corresponds with vacuum and *empty points* with non-vacuum)

prepared per mixture and per mixing pressure. The individual measuring points are presented horizontally for both the splitting and bending specimens in Fig. 13.

A student test is conducted to check whether the difference between the average air content in the failure surface of the bending and splitting specimens made under almost vacuum conditions, is significant. For MIX 1 there is not a significant difference ($t = -1.165$, $p = 0.364$). In contrast the difference for MIX 2 is significant ($t = -6.987$, $p = 0.02$). Despite a lower significance level of MIX 1, the principal reason for a lower strength increase of the splitting specimens compared with the bending specimens is due to the a lower air content reduction during vacuum mixing. A similar methodology can be handled to explain the differences between MIX 5 and MIX 6 with respect to the tensile tests.

6.3 Influence of an air content reduction on the deformability of UHPC

Figure 14 gives the result of the Young's modulus at 28 days as a function of the compressive strength determined on the same cylinders. An average increase of 7 % is obtained for MIX 1 and MIX 2 by the air content reduction. Only an increase of 3 % is registered for MIX 6, Table 4. An additional heat treatment on cylinders of MIX 1 and MIX 2 mixed under almost vacuum conditions, did not improve the

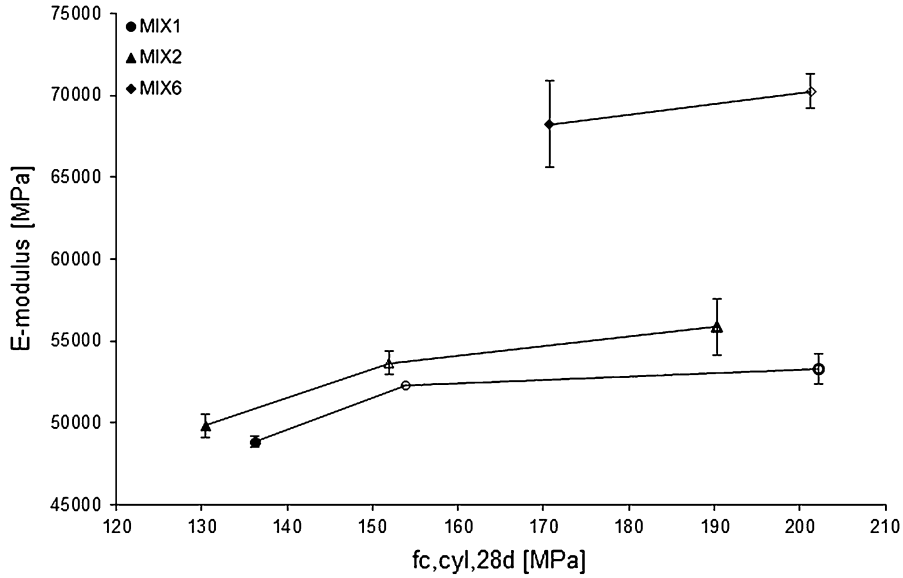

Fig. 14 Young's modulus of MIX 1-2-6 for different mixing pressures and curing regimes in function of the compressive strength (*colored* non vacuum, *empty* vacuum, *tick marker line* vacuum + heat treatment)

Young's modulus as much as the air content reduction itself. In contrary, the heat curing leads to a larger standard deviation of the Young's modulus. Apparently the removal of air bubbles makes the UHPC less elastic. A similarity can be drawn with the fresh concrete properties of UHPC where a lowered air content seems to decrease the workability of UHPC [44]. Finally, the high values of the modulus of MIX 6 are due to the bauxite used in these mixtures.

6.4 Impact of the mix volume on the effectiveness of vacuum mixing

Sections 6.1 to 6.3 examine the impact of a reduced air pressure in a 5 l intensive vacuum mixer with inclined drum, Fig. 1. For this mix volume an improvement of the microstructure is determined by air void analyzes, which give an increase in mechanical performance. The compressive strength, the bending and splitting tensile strength and the modulus of elasticity increase by the air content reduction. In practice, larger mix volumes are commonly used. The question can be raised, if vacuum mixing has the same effectiveness on these volumes. To get some insights MIX 3 is made in an intensive vacuum mixer with a capacity of 5 and 75 l, Figs. 1, 2. The vacuum time is varied from 70 to 540 s. For the 75 l mixer the time is even prolonged to 1,200 s. Three cubes are casted for air void analyzes and three for the compressive strength at 28 days, in case of the 75 l mixer. Due to the limited volume available in the 5 l mixer only one cube is casted for air void analyzes and two cubes for the compressive strength at 28 days. Figure 15 gives the decreasing hardened and fresh air content in function of the vacuum time. For the fresh air content a reference point at atmospheric pressure (1,013 mbar) is also measured.

For the 5 l mixer a reduction is established after a vacuum time of 70 s, based on the fresh air content. This effect is confirmed in literature where a reduction after 25 s of vacuum mixing is reported [21]. The hardened air content follows the same trend, except the values are slightly higher. The reason for this has been explained in Sect. 6.1. In case of the 75 l mixer the fresh air content stays constant up to 120 s and then starts to decrease until a vacuum time of 540 s. Extending this time up to 1,200 s does not improve the fresh air content in a clear way. Independent of the vacuum time the air content of MIX 3 made in the 75 l mixer is always higher than the content of the same mix made in the 5 l mixer. At this stage it seems the vacuum technology is less effective for large concrete volumes. Further research is necessary to validate the effect for mixers with a capacity larger than 75 l.

The impact of a longer vacuum time in the 75 l mixer is clearly observed for the compressive strength, Fig. 16. A maximum value of 128 MPa is only reached after 540 s vacuum mixing. Under this condition an

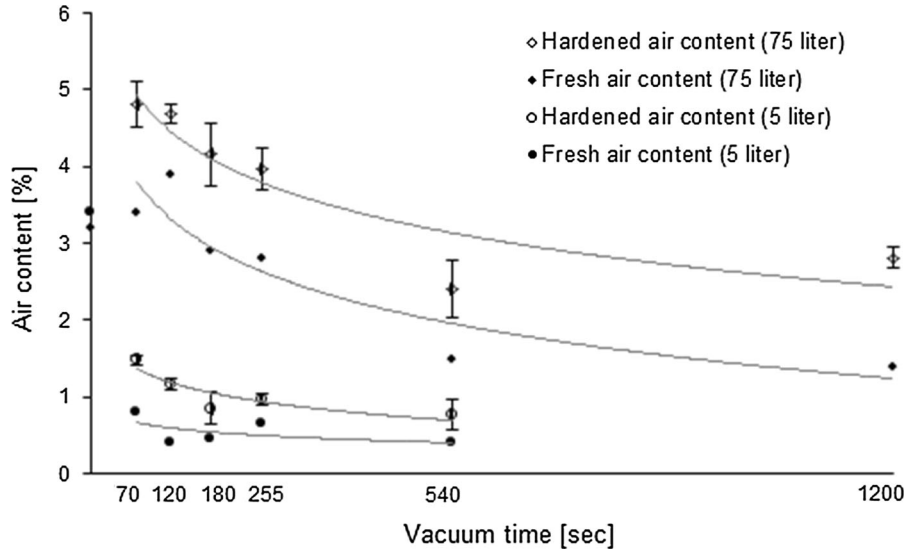

Fig. 15 Comparison of the fresh and hardened air content reduction between a 5 l capacity mixer and a 75 l capacity mixer

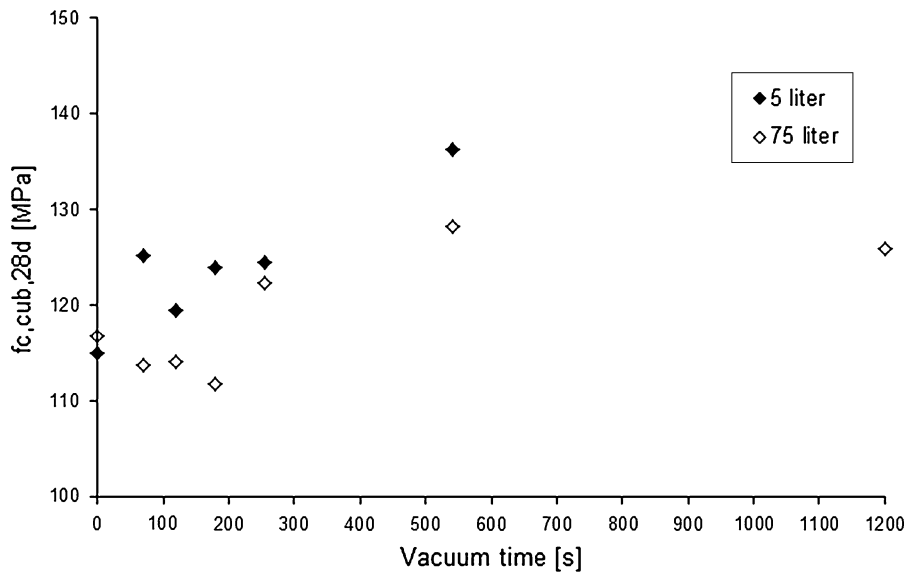

Fig. 16 Compressive strength at 28 days in function of the vacuum time for a 5 l capacity mixer and a 75 l capacity mixer

average strength gain of 10 % is obtained. This is different for the 5 l mixer where an immediate increase is noticed. Furthermore, a second jump is observed after 540 s of vacuum mixing, although the air content did not change significantly, Fig. 15. The prolonged mixing time possibly reduced the amount of capillary pores which were not measured in Fig. 15. Besides, the results related to the 5 l capacity mixer were only an average of two tests. Thus more tests should be done in order to confirm this trend. In conclusion, the mixing time should be doubled if an improved mechanical performance by vacuum mixing is wanted in a large concrete mixer (75 l).

7 Conclusions

In this paper, the effect of an air content reduction in UHPC is examined. By connecting an intensive mixer with inclined drum to a vacuum pump ultra-high

performance mixtures were made at 50 mbar. Five different mix designs are tested. First the effect on the microstructure is examined by air void analyzes. Next the influence on the mechanical properties is tested. The impact on the deformability is demonstrated by measurements of the modulus of elasticity. At the end, the possibility to use this new technology in large volume concrete mixers is examined. From the results, the following conclusions can be made:

- Vacuum mixing reduces as well the fresh as the hardened air content. Generally the effect of an air content reduction is more pronounced for the larger air voids than for the smaller air voids. As a consequence the specific pore surface and the spacing factor increase.
- Decreasing the air content improves the mechanical performance of ultra-high performance concrete. For the compressive strength an average gain is obtained between 7 and 22 %. In case of the bending tensile strength a maximum increase of 17 % is determined. As for the splitting tensile strength the increase is situated between 3 and 22 %.
- In this project, a reasonable correlation between the compressive strength and the solid concentration is found. As for the bending tensile strength the best correlation is found with the total air content in the failure surface. Furthermore the influence of the aggregate-cement bond is also acknowledged to influence the data.
- A reduced air content leads to a stiffer ultra-high performance concrete. It is determined that the modulus of elasticity increases with maximum 8 %.
- Especially for the compressive strength the vacuum technology cannot fully replace a heat treatment. In case of the splitting tensile strength and the Young's modulus the difference in increase between both techniques becomes smaller.
- At this stage, the vacuum technology is not as effective in large volume concrete mixers as it is for small volume mixers. Further research is necessary to improve the technology so that it can be applied in practice. Nevertheless, a longer mixing time will be necessary for larger concrete volumes. Consequently, a consideration has to be made between the gain in strength and the additional mixing time. Depending on the outcome vacuum mixing can be taken in consideration.

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