

CAN VACUUM MIXING REPLACE HEAT CURING IN UHPFRC?

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Abstract

In order to obtain the full capacity of cementitious materials incorporated in ultra-high performance fibre-reinforced concrete (UHPFRC) a heat curing can be applied. This method represents a high cost for the manufacturer. Therefore other techniques that increase the mechanical performance are interesting. Vacuum mixing is a new technology that can improve the compressive strength of such mixtures. Research is necessary to check whether it can replace the expensive heat treatment. We compared both techniques for several mixtures. This paper reports the effect of vacuum mixing on the compressive strength and splitting tensile strength of UHPFRC. First the effect of vacuum mixing on the compressive strength of ultra-high performance concrete was tested and compared with heat cured specimens. Next, two mixtures were chosen to incorporate different types of fibres. The effect of vacuum mixing on the splitting tensile strength was investigated. In conclusion, vacuum mixing improves the mechanical performance. Nevertheless, this new technique is not able to fully replace a heat treatment, based on the compressive strength. Further research should be done to examine the effect of vacuum mixing on the post-cracking behaviour of UHPFRC.

Résumé

Pour tirer tout le potentiel des constituants cimentaires des bétons fibrés à ultra-hautes performances (BFUP), un traitement thermique est appliqué. Ce procédé représente un coût élevé pour le fabricant. D'autres techniques d'augmentation de la performance mécanique sont donc recherchées. Le malaxage sous vide est une technique nouvelle susceptible d'augmenter la résistance en compression de ces bétons. Des études sont nécessaires pour savoir s'il peut remplacer le traitement thermique onéreux. Les deux techniques ont été comparées pour quelques compositions. L'article détaille les effets du malaxage sous vide sur la résistance en compression et en traction par fendage des BFUP. L'effet du malaxage sous vide sur la résistance a d'abord été testé et comparé au traitement thermique. Puis différents types de fibres ont été ajoutés à deux mélanges. L'effet du malaxage sous vide a alors été étudié sur la résistance en traction par fendage. En conclusion, le malaxage sous vide augmente les performances mécaniques. Néanmoins il ne peut totalement remplacer le traitement thermique vis-à-vis de la résistance en compression. Des recherches sont à poursuivre pour étudier l'effet de ce procédé sur le comportement post-fissuration des BFUP.

1. INTRODUCTION

Ultra-high performance fibre-reinforced concrete, UHPFRC, is characterized by a large amount of cementitious materials. Often 70 % to 80 % (by mass) of the binder is cement. The rest is typically silica fume, which enhances the packing density and mechanical performance¹. Furthermore UHPFRC has a very low water-to-cement ratio in order to obtain a high compressive strength. Thus, such mixtures have a high cement content and a low amount of water. This led in the earlier UHPFRC compositions to a fraction of non-hydrated cement, only contributing to the packing of the mixture². To reduce the high cost, some of the cement is replaced by filler leading to a same or better packing density³. Beside this, a heat curing is often performed, in order to obtain the full potential of the mixture by accelerating the hydration process. After demoulding the specimens are stored in a special chamber and subjected to steam of 90°C for two or more days⁴.

A large amount of the prefab manufacturers are already equipped with a steam curing chamber, in order to demould 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 UHPFRC. For in-situ concrete, this treatment is unpractical, furthermore the equipment and energy demand are expensive. Therefore other techniques that can increase the compressive strength of UHPFRC are interesting to investigate. A vacuum mixer is such a technique. By lowering the air pressure in the mixing pan, an air content reduction is established. The improved packing density leads to a better mechanical performance¹. Besides this, the technique is combined with an intensive mixing process, reducing the mixing time of UHPFRC⁵. A larger initial cost compared to a planetary mixer has to be made, but a more efficient manufacturing of the concrete is possible. This paper investigates the potential to replace a heat treatment by an air content reduction. Several ultra-high performance fibre-reinforced concrete mixtures are made under atmospheric and vacuum pressure, the effect on the hardened air content is used to explain an increase of the compressive strength. A part of the specimens made under atmospheric pressure are subjected to a heat treatment for comparison with the effect of the vacuum technology. Furthermore, the impact of a lowered air pressure in the mixing pan on the splitting tensile strength is examined for two mixtures. For each of them, three different combinations of fibres are tested.

2. EXPERIMENTAL WORK

2.1 Materials and mix proportion

Four UHPFRC mixtures were composed to examine the influence of a reduced air pressure on the compressive strength. The mix proportioning can be found in Table 1 as well as the origin of the different designs. The foreign compositions differ slightly from the reference work, because materials were used, that could be provided by local Belgian companies. The superplasticizer (SP) used in this project is a polycarboxylate ether, with a solid content by mass of 35%. Most of the mixtures contained a densified silica fume (SF) with 95.6% SiO₂, a N₂-BET specific surface of 17.765 m²/g and a d₅₀ of 0.315 µm. The quartz sand 0/0.5 has a d₅₀ of 342.0 µm. The Flour M400, M500 and D6 had a d₅₀ of respectively 10.31 µm, 3.41 µm and 58.52 µm. The last flour is dedusted, in order to remove the particles smaller than 9.83 µm which are respirable and can possibly harm the lungs.

The chemical composition of the cement is given in Table 2. CEM I 52.5N HSR LA had a d₅₀ of 10.46 µm and a Blaine fineness of 4322 cm²/g.

Table 1: Mix proportioning UHPFRC (kg/kg cement)

	MIX1	MIX2	MIX3	MIX4
CEM I 52.5 N HSR LA	1.000	1.000	1.000	1.000
Silica Fume	0.314	0.314	0.250	0.230
Quartz Sand 0/0.5 mm	1.375	0.687	1.100	1.100
Basalt 2/4 mm	/	1.376	/	/
Quartz Flour M400	0.250	0.250	/	/
Quartz Flour M500	/	/	/	0.117
Quartz Flour D6	/	/	/	0.273
Superplasticizer	0.032	0.043	0.046	0.053
Water	0.218	0.236	0.120	0.175
Fibres	0.262	0.305	/	/
Origin	A	A	B	C

A=University of Kassel³, B=Scientific Division Bouygues², C=Magnel Laboratory for Concrete Research

Table 2: Chemical composition of the cement (%)

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	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

2.2 Mixing procedure

A 5 liter intensive mixer with inclined mixing pan was used for all mixtures (Figure 1).

First the dry materials were mixed for 15 s, before the water and superplasticizer were added. The mixing time or rather, the stabilisation time (t_s) was determined based on a power curve⁶. The best workability was obtained with a hybrid mixing procedure. An intensive mixing phase (rotor speed of 6 m/s) was kept until the maximum in power consumption was reached (150 s). At this point an optimal



Figure 1: Intensive mixer connected to a vacuum pump.

dispersion of the components occurred. Next, a slow mixing phase (rotor speed of 1.6 m/s) of 120 s was applied to reach an optimal workability. In case of vacuum, a reduction from 1013 mbar to 50 mbar was established at the moment of the intensive phase. During 270 s a lowered air pressure was present in the mixing pan. In case of fibres, they were added after the intensive phase. The vacuum was first released, if necessary, and the amount of fibres were added while the mixer was in rest. Next a slow mixing phase of 120 s dispersed the fibres properly.

3. RESULTS AND DISCUSSION

First the effect of vacuum mixing on ultra-high performance concrete is examined, it is checked whether this technique is able to replace a heat treatment. Therefore, the authors validated if the same strength is obtained after 7 days independent of the technique that is used. The compressive strength on 28 days was also determined. The air content reduction was examined by help of air void analyses on a section of 100 mm on 100 mm by a Rapidair 457 air void analyzer according to the ASTM C457-linear traverse method. This technique gives a first indication of the usefulness of the vacuum technology on ultra-high performance concrete (UHPC).

In conclusion, Figure 2 gives a reduction of the hardened air content for both mix 1 and mix 2. Hereby, the air content decreases more in case of mix 1. The large amount of entrapped air in mix 1 at 1013 mbar, is due to the lack of a coarser aggregate compared with mix 2¹. Furthermore, the standard deviation is reduced in the case of mix 1, leading to a more reliable measurement. The obtained air content reduction in Figure 2, should have a significant influence on the mechanical properties of the ultra-high performance concrete⁷. For this reason, two series of cubes with side 100 mm were made. The first series were made under atmospheric pressure (1013 mbar) and the second under almost vacuum conditions (50 mbar). A third series was also produced at 1013 mbar and got a heat treatment of 48h after demoulding. For this, the cubes were positioned 2 cm above a bath that was held at a constant temperature of 90°C in a sealed container. All the series were demoulded at the age of 2 days and were stored in a climate chamber with relative humidity of 90% and 20°C \pm 2°C until the moment of testing. At the age of 7 and 28 days, the compressive strength was determined. The results are given in Figure 3 and Figure 4.

Figure 3 shows a large increase of the compressive strength when the cubes are subjected to a heat treatment. The formation of hydration products is accelerated by the contact with steam at 90°C during 48h. Mix 2 with basalt as coarse aggregate, gains less benefit of this treatment. A possible reason can be found in the cement spacing factor (CSF)⁸.

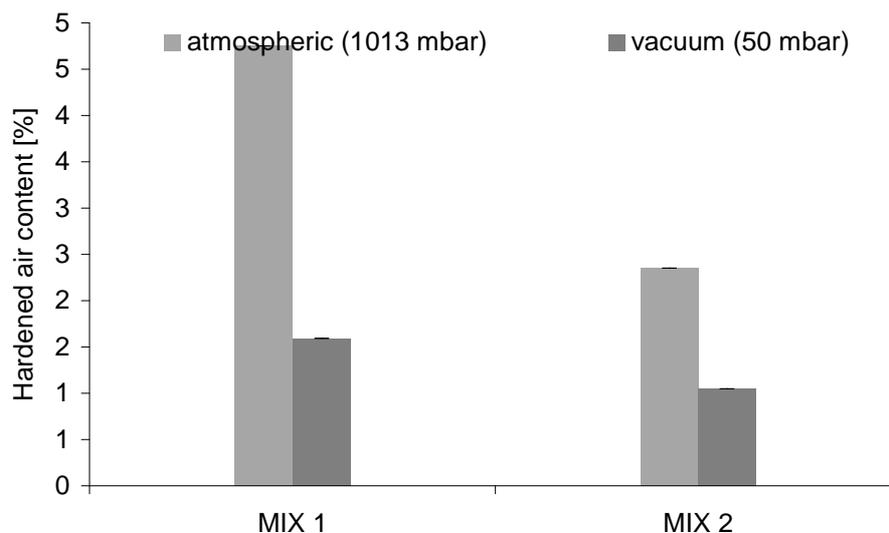


Figure 2: Hardened air content reduction after vacuum mixing for mix 1 and mix 2.

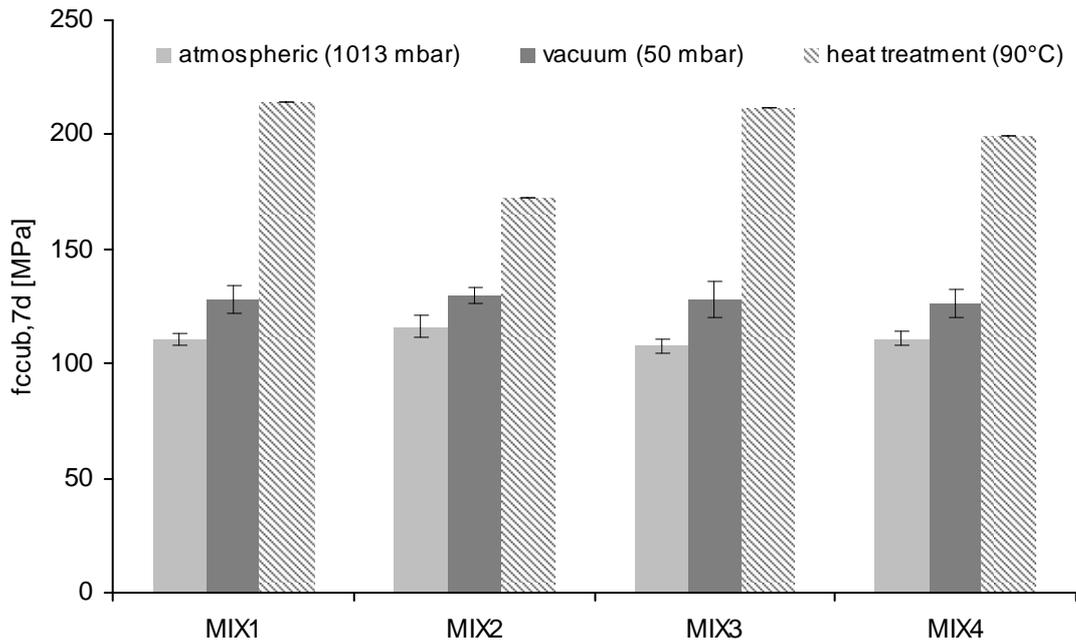


Figure 3: Compressive strength at 7 days of cubes made under three different conditions.

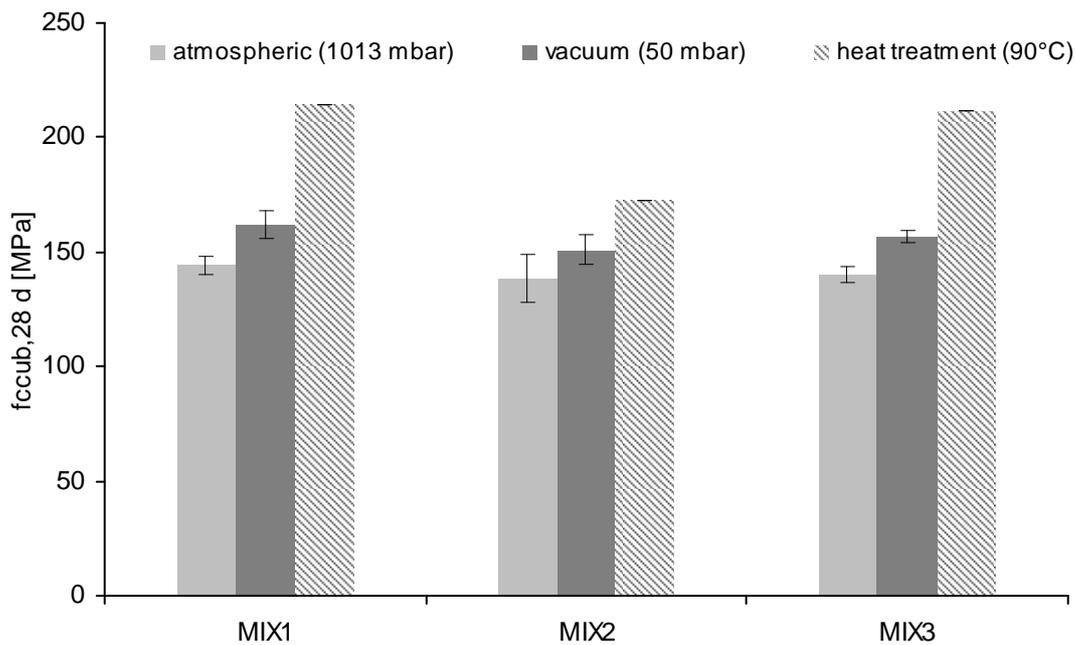


Figure 4: Compressive strength at 28 days of cubes made under three different conditions.

$$CSF = \frac{\varphi_{cem}}{\varphi_{cem}^*} \cdot \frac{\varphi_{mix}}{\alpha_t} \quad (1)$$

Where φ_{cem} is the volume in the mixture which is occupied by the cement, φ_{cem}^* is the maximum volume that cement may occupy given the presence of the other particles, φ_{mix} is the partial volume of all the particles of a mixture in an unit volume, α_t is the calculated

packing density of the mixture with the compaction-interaction packing model (CIPM)⁸. The factor $\varphi_{\text{mix}}/\alpha_t$ reduces when the amount of excess water in the mixture increases. For comparison, mix 1 and mix 2 are chosen.

Table 3: Amount of binder and water (l/m³)

	MIX1	MIX2
CEM I 52.5 N HSR LA	234.3	202.0
Silica Fume	101.3	87.1
Water	175.1	163.6
W/B	0.185	0.200

Table 3 gives the volumes of binder and water for a unit volume. From Table 3, it can be seen that the volume of cement and silica fume is lower in mix 2 than mix 1. In contrary the amount of water is slightly lower for mix 2. If the packing density (α_t and φ_{cem}^*) of both mixtures is more or less the same³. The marked reduction in cement volume will

lead to a decreased CSF of mix 2, eq.(1). Namely, with a lower cement volume and a rather constant $\varphi_{\text{mix}}/\alpha_t$ and φ_{cem}^* factor, the distance between the cement particles is statistically greater. In that case, during the hydration process, the hydration products of the cement particles need to bridge a larger distance, eventually leading to a lower strength⁸. This larger cement spacing factor can be one of the reasons why the end result of the heat treatment is less for mix 2. It also indicates that the long term strength of this mixture, without heat treatment, will be lower than the other mixtures. Further research is necessary to determine the CSF exactly by taking in account the packing density of the mixtures.

The lowered air pressure in the mixing pan also improves the compressive strength in an important way (cf. Fig. 3). By reducing the air content and thus the amount of weak spots in the concrete, a larger resistance against the compressive force is obtained. In case of UHPC with a coarse aggregate, mix 2, an increase of the compressive strength at 7 days from 115 MPa to 130 MPa was registered. For UHPC with only sand as aggregate an increase was noted from 107 MPa to 128 MPa. This gain in strength is much lower than the increase obtained by a heat treatment at 7 days. As a consequence, the vacuum technology cannot fully replace a heat treatment in the prefab industry of UHPC. Still, the technique can be useful to increase the performance of this concrete. Therefor only a change in mixing principle⁹ is necessary and no special treatments are required. The potential of the technique is also seen on the compressive strength at 28 days, Figure 4. For mix 1 a strength gain from 144 MPa to 162 MPa was registered and for mix 2 the strength increased from 139 MPa to 151 MPa. Thus the authors were not able to produce UHPC¹⁰ under atmospheric pressure, but needed the vacuum technology. In Figure 3 and Figure 4 the same test results for the cubes subjected to a heat treatment, are used. This is justified, because the treatment allows the concrete to hydrate to a much higher degree. As a consequence the compressive strength at 28 days will not differ a lot from the compressive strength at 7 days.

The addition of fibres in concrete can reduce the packing density and thereby the performance of the concrete. De Larrard noted an important decrease of the packing density for dry concrete mixtures with a coarse aggregate, when the proportion of fibres was systematically raised. No perturbing of the packing density was noted for concretes with only sand as aggregate¹. Based on these results, it is questionable if vacuum mixing still improves the packing density and is not counteracted by the addition of fibres. First the influence of vacuum mixing on the compressive strength of UHPFRC is tested. It is checked if the technology still leads to a clear improvement. For this, mix 1 and mix 2, Table 1, are used with fibre combination A, Table 4. For all the combinations the volume of fibres to the total

volume of the mixture, was held constant at 2.36% for mix 1 and 2.45% for mix 2. If a combination of two fibres is used, each fibre had a partial volume of 50%. The data in Table 4 are taken from the technical sheets provided by the manufacturer of the fibres.

Figure 5 confirms the previous results, namely an air content reduction by vacuum mixing increases the compressive strength of UHPFRC in a similar way as UHPC. In this project three cubes were cast for each mix and pressure, and tested at the age of 28 days.

An improvement from 141 MPa to 170 MPa is registered for mix 1. In case of mix 2 the strength rised from 159 MPa to 170 MPa. In general, the addition of fibres leads to a higher compressive strength as well for cubes made under atmospheric and vacuum conditions. This is in agreement with Rossi¹¹, the short fibres will stitch the micro-cracks formed during the test and limit their propagation. Apparently, the improvement is more pronounced for mix 2 than for mix 1. The latter even showed a small decrease in compressive strength when fibres are added and made under atmospheric pressure. One explanation is the use of short fibres, Nr. 1 in Table 4. The fibres with a length of 6 mm are possibly short enough to fit in the packing of the particles of mix 2, without disturbing its natural packing¹. In case of mix 1, the short fibres fit the interstices to a lesser extent than in mix 2, leading to a smaller increase of the compressive strength.

Table 4: Properties of fibres used in this study

Nr.	Type of fibre	form	d_f [mm]	l_f [mm]	l_f/d_f	Tensile strength [MPa]
1	OL 6/.16	Smooth	0.16	6	37.5	2600
2	OL 13/.16	Smooth	0.16	13	81.3	2600
3	RL45/30BN	Hooked	0.62	30	48.4	1050
4	RC80/30CP	Hooked	0.38	30	79.0	3070
Fibre Combinations	A	B	C			
	1+3	2	1+4			

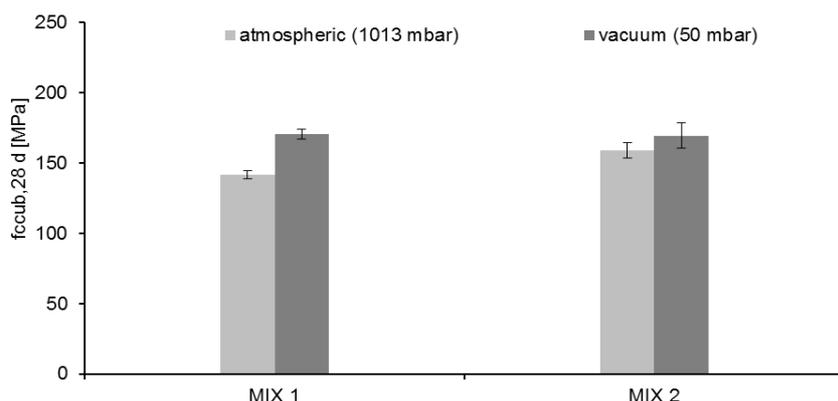


Figure 5: Effect of vacuum mixing on the compressive strength of two UHPFRC mixtures.

Next, the influence of vacuum mixing on the splitting tensile strength is investigated. Although more suitable tests are available to determine the tensile strength of UHPFRC, a comparative investigation on the influence of vacuum mixing is still possible. For each mix and pressure, three cubes were cast and tested at the age of 28 days according to NBN EN

12390-6. In this stage of the research, the authors were only interested in the maximum tensile strength. Further work can prove the usefulness of this technique on the post-cracking behaviour of UHPFRC. Figure 6, gives the influence of an air content reduction on the splitting tensile strength of mix 1 and mix 2 with fibre combination A. For both an increase is registered, in case of mix 1 the tensile strength improved from 19.0 MPa to 20.9 MPa. For mix 2 a larger increase was noted, namely from 16.3 MPa to 19.7 MPa. In conclusion, vacuum mixing can improve the performance of UHPFRC in an important way and this without any special treatment after casting. By evacuating and controlling the entrapped air, a higher tensile strength can be achieved.

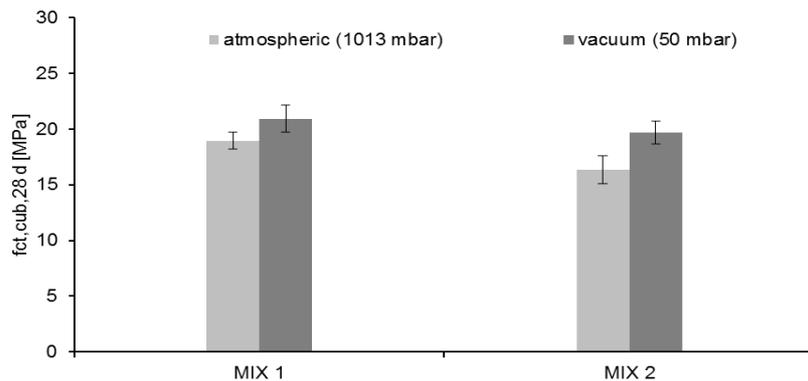


Figure 6: Effect of vacuum mixing on the splitting tensile strength of two UHPFRC mixtures.

Finally, the influence of different fibre combinations, Table 4, on the splitting tensile strength is tested. For each mix and combination three cubes were made under a reduced air pressure. Again, the authors were only interested in the maximum tensile strength. Combination A is chosen as a reference where the short fibres possibly can fulfil two functions. On the one hand they could fit in the packing of the particles and thereby increasing the packing density. On the other hand they can help to bridge the micro-cracks during the test. In contrary, the long fibres are more useful to reduce the brittleness of UHPC. Combination B is chosen to fulfil both the function of long and short fibres, this because of its intermediate fibre length. The higher tensile strength of the long fibres in combination C, makes them interesting regarding the brittleness and post-cracking behaviour of the UHPC. Furthermore the smaller diameter will lead to more flexible fibres, disturbing the natural packing of mix 1 and mix 2 possibly in a lesser extent.

For mix 2, Figure 7 shows the highest splitting tensile strength for the combinations with long and short fibres. On the one hand, these short fibres will be able to bridge micro-cracks during the test, which leads to an overall improvement of the splitting tensile strength¹¹. On the other hand, similar as for the compressive strength, the packing density of mix 2 could be enhanced by the addition of short fibres. These fibres are possibly short enough to fit in the packing of the particles. In contrary, the fibres of combination B, are probably too long to fit perfectly in the interstices and will perturb the natural packing¹. As a consequence, a lower splitting tensile strength is found for combination B. Furthermore, no influence was found between the two types of long fibres in the case of mix 2. In conclusion, the greatest improvement of the maximum splitting tensile strength was found, if the short fibres were able not only to stitch the micro-cracks during the test¹¹, but also could enhance the packing

density of the mixture. The long fibres did not have an important influence on the maximum. Nevertheless, they are necessary to reduce the brittleness and improve the post-cracking behaviour. For mix 1, the interstices are filled in a lesser extent, by the short fibres. Apparently, the highest splitting tensile strength is obtained for a combination that perturbs the natural packing of mixture the least and still stitch the micro-cracks during the test. For mix 1, combination B led to the best performance. Thus, in order to produce a suitable UHPFRC, an optimization from nano-scale to macro-scale of both the matrix and fibre content is necessary.

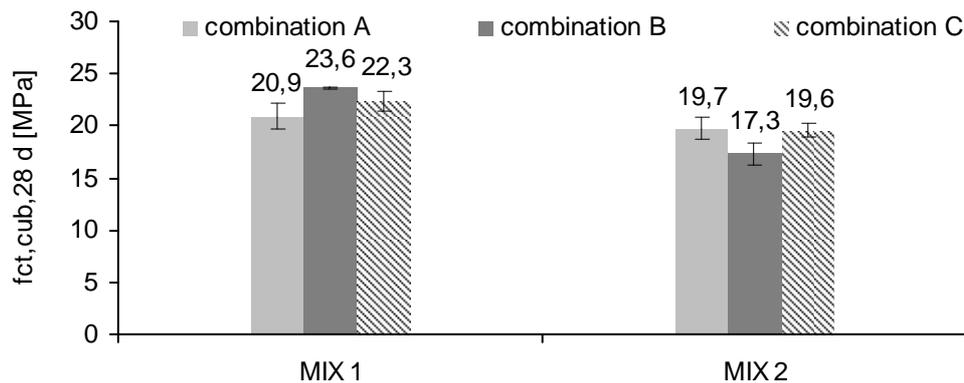


Figure 7: Comparison of different fibre combinations on the splitting tensile strength of UHPFRC made under a reduced air pressure.

4. CONCLUSIONS

In this research project the effect of vacuum mixing on the compressive strength of UHPFRC is investigated. First the influence of a reduced air pressure is examined on the microstructure by means of air void analyses. Then, a comparison between the influence of vacuum mixing and a heat treatment on the compressive strength of different UHPC mixtures is discussed. Next, it is checked whether the improved compressive strength by a reduced air pressure, is also obtained when fibres are added. At the end, the increase in the splitting tensile strength by an air content reduction is investigated. From the results, the following conclusions can be made:

- The hardened air content of ultra-high performance concrete can be reduced by vacuum mixing. The reduction was more pronounced for mixtures with only sand as aggregate.
- The reduction of the hardened air content leads to an increase of the compressive strength of the ultra-high performance concrete. Nevertheless, the improvement is not large enough to fully replace a heat treatment.
- Vacuum mixing also improves the compressive strength when fibres are added. If larger interstices are available between the coarse aggregate, the addition of short fibres itself can also increase the compressive strength in an important way.
- A reduction of the air pressure increases the splitting tensile strength in a clear way. Thus the vacuum mixing leads to a higher performance of the UHPFRC.
- A good fibre combination can enhance the splitting tensile strength. On the one hand the addition of short fibres can stitch the micro-cracks and on the other hand it is important

they do not disturb the natural packing of the UHPFRC. In order to confirm the latter, the influence of different fibre combinations on the packing density of the dry mixture could be examined.

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REFERENCES

- [1] De Larrard, F., 'concrete mixture proportioning: a scientific approach', 1st edition, London; E& FN Spon, 1999.
- [2] Richard, P. and Cheyrezy, M., 'Composition of Reactive Powder Concrete' *Cement and Concrete Research*, **25**(7) (1995) 1501-1511.
- [3] Schmidt, M. and Gelsenhanslüke, C., 'Optimierung der Zusammensetzung des Feinstkorns von Ultra-Hochleistungs- und von Selbstverdichtendem Beton, UHPC – 10 Jahre Forschung und Entwicklung', *Heft 7 Structural Materials and Engineering Series*, **7** (2007) 144-164.
- [4] Heinz, D., Urbonas, L. and Gerlicher, T., 'Effect of Heat Treatment Method on the Properties of UHPC', Proceedings of Hipermat 2012 3rd International Symposium on UHPC and Nanotechnology for High Performance Construction Materials', Germany, Kassel, 2012, 283-290.
- [5] Mazanec, O., Lowke, D. and Schiessl, P., 'Mixing of high performance concrete: effect of concrete composition and mixing intensity on mixing time' *Materials and Structures*, **43**(3) (2010) 357-365.
- [6] Chopin, D., De Larrard, F., et al., 'Why do HPC and SCC require a longer mixing time?', *Cement and Concrete Research*, **14** (2004) 2237-2243.
- [7] Aligizaki, K.K., 'Pore Structure of Cement-Based Materials', 1st edition, London, Taylor & Francis, 2006.
- [8] Fennis, S.A.A.M, 'Design of ecological concrete by particle packing optimization', (2011) 1-256 (Doctoral thesis).
- [9] Dils, J., De Schutter, G. and Boel, V., 'Influence of mixing procedure and mixer type on fresh and hardened properties of concrete: a review' *Materials and Structures*, **45**(11) (2012) 1673-1683.
- [10] Graybeal, B., 'Technote: Ultra-high Performance Concrete' *FWA Publication* No: FHWA-HRT-11-038 (2011).
- [11] Rossi, P., 'High Performance Multimodal Fibre Reinforced Cement Composites (HPMFRCC): The LCPC Experience' *ACI-Materials Journal*, **94** (6) (1997) 478-483.