Cavitation Treatment of Nano and Micro Filler and Its Effect on the Properties of UHPC

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The effect of cavitation treatment of micro and nano fillers in relation to ultra-high performance concrete (UHPC) was investigated in this paper. Specially designed and patented apparatus was applied to create turbulent flow and employ hydrodynamic cavitation process as a tool for silica fume micro and nano particle agglomerate disaggregation and surface activation. Silica fume/water slurry at the ratio 1 : 2 was subjected to the impact of the hydrodynamic cavitation process.

UHPC mixes with optimized particle packing were designed in order to evaluate effect of specially treated silica fume on the mechanical properties of the UHPC. Due to the cavitation treatment the agglomerates disaggregated into nano and micro particles and uniform suspension in water was obtained and incorporated into the UHPC mix.

Keywords: UHPC, microsilica dispersion, cavitation

1 Introduction

Pozzolanic additives are used in the concrete production to enhance mechanical properties and durability of the material. Silica fume, metakaolin, rice husk ash and nanosilica are some of the additives that can be beneficial and improve performance of the concrete. As pozzolanic additive particle sizes normally are less than those of cement (average silica fume particle size is around 0.15 µm), pozzolans in concrete can have dual effect – to improve physical particle packing and to react chemically with calcium hydroxide forming calcium silicate-hydrates [1].

However, sometimes desired effect is not reached due to the fact that smaller particles have increased surface energy and during the storage time aggregate more easily to secondary particles, having sizes on the order of hundreds of micrometres. It is not possible to break such agglomerates in conventional mixing process due to the low shear forces. As agglomerates can be bigger than cement particles, packing of the mix is not improved and chemical reaction is hindered due to the reduced active surface area of pozzolan. Moreover undispersed silica fume agglomerates can react as alkali–silica reactive aggregates in concrete [2] (See Figure 1).

Cavitation treatment of pozzolanic materials before concrete mixing is proposed in this paper in order to disaggregate agglomerated particles and therefore reach better particle dispersion in concrete mixture. A novel hydrodynamic dispersion method for fine particles was developed and patented in Germany (patent number 20 2007 914 913.1), Latvia (patent number 13592), Russia (patent number 2297876) and EU (patent application number P-11-67).

Hydrodynamic cavitation phenomena occur if the fast flowing liquid (e.g. water) encounters obstacles in its way. In some areas of disturbed flow continuity of water is broken and rapid pressure drop below the

Figure 1: Silica fume agglomerate causing alkali-silica reaction [2].
water vapour pressure is encountered. Vapour cavities in the water are created. Source of cavitation nuclei is gas that is dissolved in the water. If the liquid pressure exterior to a bubble will be lower than the saturated vapour pressure, the nucleus will be activated and the radius of the bubble will increase. The increase of the liquid pressure results in the cavitation bubble collapse. Cavitation bubbles collapse violently generating either the micro-jets or shock pressure waves featured by high velocities, pressures and temperatures [3]. The temperature inside the cavitation bubble during collapse can reach 5075 K, but such extreme temperature exists for the very short time [4; 5]. Impacts of shock pressure waves and micro-jets are the source of the noise and the material damage. The impulse pressure might reach the pressure of approximately 1 GPa and impact velocity of 1000 m/s [6].

Cavitation is specifically avoided in the design of machines such as turbines or propellers because of possible damage risk. However, in this study it proved to be beneficial as a tool for silica fume agglomerate disjoining.

2 Materials and methods

Traditionally there are two types of UHPC mixtures - coarse grained with maximum aggregate size 4-8 mm and fine grained with maximum aggregate size 1-2 mm. To evaluate effect of silica fume cavitation treatment, fine-grained UHPC mixture was designed. Materials used in this study were the following:

- cement CEM I 52,5 R UHPC;
- silica fume;
- nanosilica;
- quartz sand 0 – 0.3 mm;
- quartz sand 0.3 – 0.8 mm;
- quartz sand 0 – 1 mm;
- polycarboxylate based superplasticizer;
- steel fibers Ø=0.6mm L= 13 mm;
- water.

The chemical composition of cementitious materials is presented in Table 1.

<table>
<thead>
<tr>
<th></th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>CaO</th>
<th>SO₃</th>
<th>MgO</th>
<th>Na₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I 52.5R</td>
<td>25%</td>
<td>2.1%</td>
<td>3%</td>
<td>69%</td>
<td>2.3%</td>
<td>0.7%</td>
<td>0.2%</td>
</tr>
<tr>
<td>Silica fume</td>
<td>97.5%</td>
<td>0.4%</td>
<td>0.1%</td>
<td>0.2%</td>
<td>0.1%</td>
<td>0.1%</td>
<td>0.1%</td>
</tr>
<tr>
<td>Nanosilica</td>
<td>99.9%</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
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</table>

Specially designed and patented apparatus was applied to create turbulent flow and employ hydrodynamic cavitation process as a tool for silica fume micro and nano particle agglomerate disaggregation and surface activation (Figures 2, 3) [7].
The setup consists of four main components:

1) Cavitation chamber where high speed flow is created and cavitation phenomena occurs
2) The reservoir, where materials can be added and collected after treatment.
3) The electrical engine.
4) Frequency converter to adjust engine speed and choose most effective working regime.

The slurry was produced by adding silica fume and water in proportions 1:10 and 1:2. The volume of slurry was 3 liters and it was processed for 10 minutes. Device speed was 6000 rpm.

The concrete mixing procedure was organised in a following way: all dry materials were mixed for 1 minute in the high speed paddle mixer. Then silica fume slurry and 70% of total water amount were added. After 2 minutes of mixing rest of water and superplasticizer was added and mixing was continued for 5 minutes.

Mix consistency was determined by cylinder flow test. Cylinder with the internal diameter of 50 mm and height of 100 mm was filled with UHPC concrete mix and lifted up. The diameter of the flow was measured after 1 minute.

Specimens with dimensions 100x100x100 mm were cast and stored in climatic chamber (t=20°C, RH=90%) At the age of 1 day specimens were demoulded and stored in water bath (t=20°C). Specimens were tested according to standard [7] at the age of 7 days. The second testing was performed at the age of 12 days. Accelerated curing conditions (t=90°C) were applied for last 48 hours. Five concrete mixtures were produced. All mixtures had cement content of 950 kg/m³. Two mixtures with silica fume content of 20 kg/m³ were produced. One of them was reference mix (REF-1) without cavitation treatment and for second (CAV-1) cavitation treatment was applied. Three other mixtures had silica fume content of 100 kg/m³. First was reference mix (REF-2) without treatment, second (CAV-2) was treated by cavitation and third (CAVNS-2) additionally to cavitation treatment had nanosilica addition 1% of cement mass. Prepared concrete mix compositions and cone flow values are summarized in the Table 2.
## Table 2: UHPC mix compositions and cone flow values.

<table>
<thead>
<tr>
<th>Materials</th>
<th>REF1</th>
<th>CAV-1</th>
<th>REF-2</th>
<th>CAV-2</th>
<th>CAVNS-2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland cement CEM I 52.5</td>
<td>950</td>
<td>950</td>
<td>950</td>
<td>950</td>
<td>950</td>
</tr>
<tr>
<td>R</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quartz sand 0.3/0.8 mm</td>
<td>470</td>
<td>470</td>
<td>470</td>
<td>470</td>
<td>470</td>
</tr>
<tr>
<td>Quartz sand 0/0.5 mm</td>
<td>200</td>
<td>200</td>
<td>200</td>
<td>200</td>
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<tr>
<td>Quartz filler 0/0.3 mm</td>
<td>400</td>
<td>400</td>
<td>340</td>
<td>340</td>
<td>340</td>
</tr>
<tr>
<td>Silica fume 971U</td>
<td>20</td>
<td>20</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Nanosilica Elkem</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>9.5</td>
</tr>
<tr>
<td>Superplasticizer</td>
<td>25</td>
<td>25</td>
<td>25</td>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>Steel fibre 13 mm/0.16 mm</td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>Water</td>
<td>200</td>
<td>200</td>
<td>200</td>
<td>200</td>
<td>200</td>
</tr>
<tr>
<td>Cavitation:</td>
<td>-</td>
<td>10 min</td>
<td>-</td>
<td>10 min</td>
<td>10 min</td>
</tr>
<tr>
<td>Cone flow, mm</td>
<td>226</td>
<td>260</td>
<td>260</td>
<td>250</td>
<td>255</td>
</tr>
</tbody>
</table>

Concrete density, kg/m$^3$: 2285 2285 2365 2365 2374.5

### 3 Results and discussion

The effect of cavitation treatment first can be evaluated visually. For untreated silica fume suspension larger agglomerates can be noticed even without microscope, while for processed suspension without microscope no visible particles are found. If untreated suspension (manually mixed) and cavitation treated suspension (silica fume/water ratio 1 : 10) is left undisturbed in the clear vessel, segregation can be observed for untreated suspension after 15 minutes, while processed suspension does not show any segregation (see Figure 4a). After two hours both suspensions are segregated (Figure 4b). And for untreated suspension large agglomerates are visible (Figure 4c).

![Figure 4: a) Suspension segregation after 15 minutes. Left – untreated, right - cavitation treated. b) Suspension segregation after 2 hours. Left – untreated, right - cavitation treated. c) Agglomerates in untreated slurry after 2 hours.](image)

Consistency was measured by means of cylinder flow test (see Figure 5). Results (see Table 2) have shown that cylinder flow increase from 226 to 260 mm after silica fume treatment for mixes with silica fume content of 20 kg/m$^3$. Reason could be better particle packing (that reduces necessary water amount and improves flowability of mixture. For mixes with silica fume...
fume content of 100 kg/m$^3$ after cavitation treatment cylinder flow decreased from 260 to 255 and 250 mm. This effect could be explained by significantly increased silica fume surface area after agglomerate disaggregation that absorbs extra water and reduces flowability.

Compressive strength was tested at the age of 7 days (normal curing) and 12 days (10 days normal curing and 48 hours accelerated curing at the temperature of 90ºC). For the mixtures with silica fume content 20 kg/m$^3$ results (see Figure 6) show compressive strength increase of 6% at the age of 7 days in normal curing regime and 4% increase at the age of 12 days in accelerated curing regime compared to those of reference mix.

Mixture with silica fume content 100 kg/m$^3$ show similar strength increase after silica fume cavitation treatment (see Figure 7). At the age of 7 days compressive strength increase is 4% and at the age of 12 days 5%. Mixture with the nanosilica addition shows best performance with the strength increase of 9% at the age of 7 and 12 days and reaches compressive strength of 164.5 MPa.
4 Conclusions

Cavitation treatment of silica fume with the aim of agglomerate disaggregating and surface activating proved to be simple, fast and effective way to improve UHPC properties. Average compressive strength increase for both – normal and accelerated curing was 5%. In case of nanosilica addition compressive strength increased for 9%. Also risk of alkali – silica reaction due to silica fume agglomerates was minimized. Silica fume suspension in water (ratio 1:10) after cavitation treatment has shown higher stability. Cylinder flow value for higher silica fume amount decreased due to the higher cavitation treated particle surface area that can absorb more water.

The cavitation treatment apparatus can be easily upscaled for the purposes of industrial use.

5 Acknowledgement

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References