

# Influence of mechanically treated carbon fibre additives on structure formation and properties of autoclaved aerated concrete

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## ABSTRACT

This work deals with investigations on effect of chemically and thermally resistant carbon fibre (CF) additives of various mechanical treatment on structure formation and properties of autoclaved aerated concrete (AAC). It was established that various methods of CF crushing, such as mixing with sand pulp, chopping, grounding, milling in dry way, milling in wet way cause different fineness of disintegrated CF particles and that along with increase in fineness, the crystallinity of AAC binding material is growing what leads to the following: increase in compressive strength by 6–22%; after exposure at temperature of 700 °C, decrease in thermal deformation by 5–20%; decrease in mass loss by 7–20%. Upon addition of mechanically not treated CF of 0.1%, the flexural strength increases by 29% versus that of AAC without CF additive. The effect of added mechanically treated CF on flexural strength is less than that of not treated CF. Due to increased CF fineness, the flexural strength increment drops from 21% to 4%. Basing on the results obtained, one can draw a conclusion that CF particles resulted in mechanical treatment have an activated surface and can serve as nuclei of crystallization during the hardening of AAC binding material, which contains a mechanically treated CF additive, thus causing increase in crystallinity of hardened binding material and, consequently, improved performance of AAC.

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## 1. Introduction

Fibre-reinforced composites are new materials based on carbon or other fibre types with exceptional physical and chemical properties to be profited by in production of advanced products [1].

It was found out that CF positively influences strength of non-autoclaved cement-based concretes (NACBC), as well as autoclaved aerated concretes (AAC).

Kim and Park [2] investigated the effect of various-shaped CF (C, hollow and round shape) on tensile and flexural strengths of lightweight NACBC and established that the shape of CF fibre exerts an effect on tensile and flexural strengths.

Xu and Chung [3] fixed that addition of short CF (length and diameter of 5 mm and 15 µm, accordingly) to cement paste containing silica fume and methylcellulose results in decrease of loss

tangent under flexure by up to 25% and in increase of storage modulus by up to 67%. These effects depend on CF surface treatment (by ozone, aqueous solution of potassium dichromate and sulphuric acid or silane).

Fu et al. [4,5] increased tensile strength, modulus and ductility [4] and improved strain-sensing ability [5] of carbon fibre reinforced cement paste by ozone treatment of CF before usage. The ozone treatment involved exposure to O<sub>3</sub> gas (0.3 vol.%, in air) for 5–10 min at 160 °C.

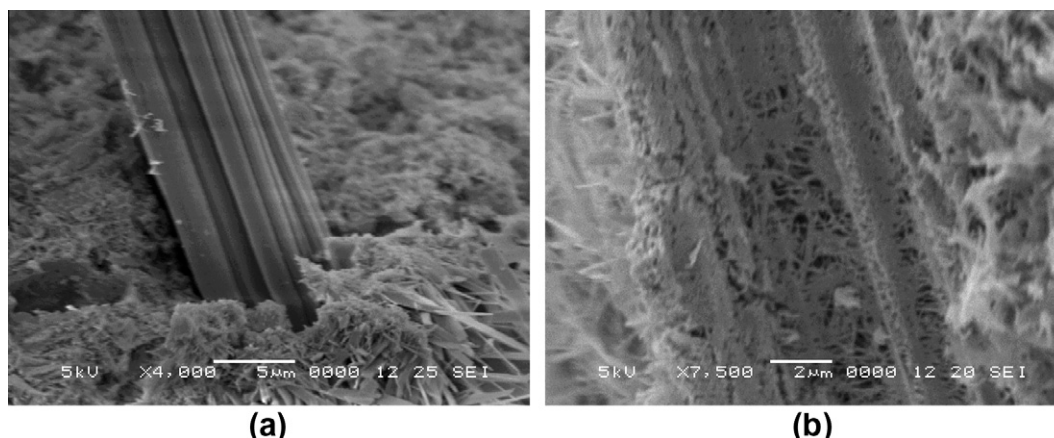
We established earlier [6] that the strength properties of AAC can be improved by CF additive. The non-hydrophilized CF additive of 0.3% increased the compressive strength of AAC (450 kg/cm<sup>3</sup>) by 42%, the flexural strength by 143%. The same amount of hydrophilized CF additive increased the compressive strength of AAC (450 kg/cm<sup>3</sup>) by 49%, the flexural strength by 166.7%. The investigations by scanning electron microscope (SEM) showed (Fig. 1) that upon exertion of destructive force on concrete, during fracture of AAC, CF filaments creep out of hardened AAC matrix (Fig. 1a). This explains why the not treated CF increases the flexural strength more than the compressive strength. The seal (“imprint”) is retained in the hardened matrix after the creeping out of filaments (Fig. 1b). The relief of seal (Fig. 1b) shows that the seal of filament echoes exactly that of CF surface visible in Fig. 1a. It signifies that before the

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**Fig. 1.** Fragments of microstructure of AAC fracture surface: (a) CF pulled out from hardened binding material during AAC fracture, (b) microstructure of binding material contacted during hardening with CF filament surface and opened when CF filament is pulled out during AAC fracture [6].

AAC hardening, the surface of filaments was entirely covered by forming mixture and thoroughly wetted by it. The microstructure of seal (Fig. 1b) shows that in the binder's layer, which contacts to CF filament surface, the crystallization of hydrosilicates occurs and that needle-like crystals of calcium hydrosilicate are formed during the AAC matrix hardening. One can suggest that some areas of CF filament surface act as centres of crystallization of hydrosilicates in the matrix contacting with fibre surface.

In the work [7] we showed that the replacement of milled sand of 1.0% in AAC by amorphous nanodispersive  $\text{SiO}_2$  (ANS) leads to better crystallinity of hardened binding material. The formed structure of better crystallinity caused an increase in AAC compressive and flexural strengths, as well as in thermal resistance. Basing on the results of work [7], a hypothesis was framed out to say that nanosized particles of ANS serve not only as a pozzolanic additive, but also as nuclei during the hardening of AAC and hereby stimulate a better crystallinity versus that without additive and contribute to improvement of AAC mechanical properties. In the work [7] we stressed the correlation of our results with those of investigations performed in the field of hardening of non-autoclaved cement based material [8,9], demonstrating that the targeted nanostructure formation is an effective way for enhancement of quality and that one of possible measures to increase the AAC quality is usage of nuclei of crystallization during AAC hardening.

In the work [10] Sanchez and Sobolev presented the recent progress in nanoengineering and nanomodification. They reviewed numerous works investigating how NACBC properties are influenced by used additives of nanosized particles, such as nanosilica ( $\text{nanoSiO}_2$ ), nanotitanium oxide ( $\text{nanoTiO}_2$ ), nanoiron ( $\text{nanoFe}_2\text{O}_3$ ), nanoaluminia ( $\text{nanoAl}_2\text{O}_3$ ), nanoclay. The mentioned authors stressed in their review [10] that the nanoparticles can act as nuclei for cement phases, promoting hydration and that one of positive actions of nanoparticles is improvement of mechanical characteristics of concrete. For example, there are cited articles where it was noticed that very small amounts of  $\text{nanoSiO}_2$  increase compressive and flexural strengths of concrete;  $\text{nanoTiO}_2$  and  $\text{nanoFe}_2\text{O}_3$  improve compressive and flexural strengths;  $\text{nanoAl}_2\text{O}_3$  significantly increases the modulus of elasticity.

Basing on the foregoing, an idea came to us to use CF particles of different mechanical treatment as nuclei in the process of AAC hardening.

The aim of this work is to investigate a usage of CF additives of various mechanical disintegration as nuclei in the AAC hardening and thus to extend our research into the field of nanotechnologies applicable in the production of AAC.

## 2. Materials and methods of testing

### 2.1. Materials

The forming mixtures of AAC were prepared using the raw materials described below. Milled lime CL 90-Q (according to standard [11]) with available CaO of 85.2% and reactivity expressed by time  $t_u$  of 3 min and temperature  $T_u$  of 61.3 °C. Portland cement CEM I 42.5 R (according to standard [12]) of the following mineral composition (in %):  $\text{C}_3\text{S}$  58.54;  $\text{C}_2\text{S}$  15.29;  $\text{C}_3\text{A}$  10.40;  $\text{C}_4\text{AF}$  10.17. Quartz sand (according to standard [13]) with fineness of 2766  $\text{cm}^2/\text{g}$ . The size distribution of milled quartz sand particles in sand is provided in Fig. 2. As a gas-generating agent, in AAC forming mixtures, aluminium paste DEG 4508/70 was used. Specific surface of 18,000  $\text{cm}^2/\text{g}$ , content of pure aluminium in the paste 70%. For better distribution of CF additives, the surfactant UFAPORE TCO was used in amount of 0.003% (counting from dry mass of solids).

### 2.2. Sample preparation

Quartz sand was milled by ball mill (capacity of 10 l, speed of 100 rpm, porcelain balls of 20.0 mm diameter). The size distribution of milled quartz sand (Fig. 2) was determined by laser particle size analyzer 1090 LD with measuring range for wet dispersion of 0.04–2500  $\mu\text{m}$  and accuracy of 3.0%. The sand sample was dispersed in distilled water for 1.0 min using both mechanical stirrer (speed of 400 rpm) and ultrasonic transducer (power of 60 W, frequency of 36 kHz).

CF additives in AAC samples were as follows: mechanically untreated (in sample series B); mixed with sand pulp (in sample series C); chopped by knife disintegrator (in sample series D); ground in laboratory mortar (in sample series E); dry milled (in sample series F); wet milled (in sample series G). The morphology of mechanically treated CF additives is presented in Fig. 3. The chemical composition of used raw materials is presented in Table 1.

The average filament length of untreated CF was about 50 mm, average diameter about 7.0  $\mu\text{m}$ . The surface morphology of mechanically untreated CF is provided in Fig. 3a. The CF mechanical treatment was performed as described below.

The mixing with sand pulp for 10 min was performed by hand mixer kMix HM 790 of maximal power 400 W and rotation speed 350 rpm in plastic vessel of 2 l. The surface morphology of CF particles obtained by such mechanical treatment is provided in Fig. 3b.

The chopping of CF was performed by knife cutting mill PULVERISETTE 15 with rotor speed 2800 rpm, the cutting material being tool steel. Chopped CF was sieved through sieve (mesh size 250  $\mu\text{m}$ ). The size of thus received CF particles, which deposited on crushed filament surface, are provided in Fig. 3c. The photos of CF show CF particles of various size dispersed on surface of CF filaments. The size of some particles makes few microns, while the majority is smaller than 0.5  $\mu\text{m}$ .

The grinding was performed by laboratory mortar of 0.5 l and lasted 10 min. The morphology of ground CF, as well as the size of thus generated CF particles is provided in Fig. 3d. While grinding CF in the laboratory mortar, only some CF segments remained. Ground CF consists mainly of fine particles sized from less than 100 nm to several micrometres. The photo shows that some particles adhere to surface of filament segments, while others form accumulations, which are distributed in mass of ground fibre.

The CF milling in dry or wet ways was performed by planetary micro mill PULVERISETTE 7 with two bowls of 80 ml and agate balls of 5.0 mm diameter and lasted for 120 min (main disc speed of 300 and 600 rpm, accordingly). For wet milling, distilled water was used. The morphology of CF milled in dry and

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