Carbon fibre reinforced cement-based composites as smart floor heating materials

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1. Introduction

Screed is an indispensable part of the modern interior design for height adjustment and has to withstand different static and dynamic loads. Common floor screeds are cement-based composites consisting of Portland cement, sand and aggregates, and in most cases they are fabricated as a free floating structure. Heating elements are placed in floor heating screeds between the screed plate and the underlying insulation. To be able to withstand shrinkage and temperature stresses, fibre reinforcement is often added to the composite mixture. If fibre reinforcement would be included as admixture of electrically conductive fibres, in-situ electrical heating of the screed would be feasible without installing heating elements below the floor plate, assuming that the admixed conductive fibres can provide good conductivity of the composite on the one hand and provide high flexural strength on the other hand. Previous research on carbon fibre reinforced mortar and concrete has shown that adding 0.5 to 3.0 percent by volume of chopped carbon fibres can increase strength [1–5] and decrease electrical resistance significantly [6–9]. Thus, electrical conductivity increases by several orders of magnitude upon adding a few volume percent of carbon fibres into the concrete or mortar [7–9]. Further studies have shown that it is possible to use carbon fibre reinforced concrete to deice roads [10] or to heat buildings [11,12] by applying a low voltage generated electrical current (e.g. up to 12 V). Electrical conductivity is provided by carbon fibre contact bridges granting high electrical conductivity over distances exceeding the length of single fibres [8], in comparison to concrete without carbon fibre reinforcement. It has been shown, that carbon fibre admixture does not influence thermal conductivity, which would have negative effects on thermal insulation of floor constructions [13].

The aim of this work is to investigate the electrical properties of a chopped carbon fibre reinforced cement-based composite in order to determine the optimal carbon fibre aspect ratio and volume content, and to report on influences of the electrical resistance heating on mechanical properties of the composite. Furthermore different embedded electrode materials are tested in order to report on electrode corrosion during electrical resistance heating. Since electrical heating is more expensive than conventional heating with gas or fuel oil, developing smart heating systems for saving energy is essential to reduce costs. This concept is implemented, for instance, in heating only selected areas of the floor heating screed in order to save energy for those areas where heating is typically not required (e.g. areas where pieces of furniture like cabinets or shelves are placed). Accordingly, a

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prototypic modular floor heating plate is presented, which allows individual heating of different areas by using a grid of embedded electrodes.

2. Material and methods

2.1. Materials and specimen preparation

The properties of the carbon fibres used in the studies and the material suppliers are summarized in Table 1. Portland cement (Type I 52.5 R) was used throughout with sand (d_{50} = 0.2 mm), silica fume (Elkem Microsil) and water reducing agent (BASF Glenium ACE 430). The composition of the cement mixture is shown in Table 2. The admixture of silica fume (d_{50} = 0.4 μm) and finely ground cement (d_{50} = 8 μm) is added to improve the fibre dispersion in the cement paste [14]. Water cement ratio was 0.35 (including water reducing agent).

Mixing all solid components, apart from carbon fibres, was carried out in a solid state. Water and water reducing agent were added and mixed in a rotary mixer at 400 RPM until a homogeneous mixture was achieved. Finally, the carbon fibres were added and the mixture was stirred again at 50 RPM until the fibres became uniformly dispersed. A batch size was 40 g (three specimens) for conductivity measurements, 80 g (six specimens) for 3-point-bending tests and 200 g for the plate heating test. The cement mixture was poured into Teflon molds with dimensions of 60 × 13 × 6 mm for each specimen for conductivity and flexural strength measurements and 100 × 100 × 8 mm for plate heating tests. Subsequent to preparation, the specimens were stored at 100% humidity for 24 h, then transferred for 6 days into a water bath and then kept another 3 weeks at 60% humidity prior to testing or further treatment.

2.2. Conductivity and flexural strength measurement

After 4 weeks of curing time, both ends of the 60 × 13 × 6 mm specimens were sand-polished and electrically contacted by applying silver lacquer. To reduce border effects, 0.5 mm of the specimen surface area was removed by sand polishing. Subsequently the resistance between the most distant specimen ends was measured. For low impedance measurement, a M-4650 CR multimeter and for high impedance measurement, a Metriso 5000 ohmmeter were used. After measuring the overall dimension of the specimen at an accuracy of ±0.01 mm, the specific resistivity ρ can be calculated according to Equation (1):

$$\rho = R * \frac{A}{l} \tag{1}$$

where R is the electrical resistance, A is the cross-sectional area (13 mm width × 6 mm height) and l is the length of the object (60 mm). The specific resistance is determined for at least three specimens, whereupon the average and the standard deviation are calculated. The reciprocal value of the specific resistivity is defined as the electrical conductivity σ of a material. It can be calculated by the Equation (2):

$$\sigma = \frac{1}{\rho} \tag{2}$$

Flexural strength measurements were carried out with a total number of six specimens for each testing series. The testing machine is a Zwick/Roell Zwicki-Line Z0.5 with a 5 kN load cell being attached. The experimental setup is illustrated in Fig. 1. By measuring the maximum force F, the flexural strength fs can be calculated by the following Equation (3):

$$fs = \frac{3}{2} * \frac{F * l}{b * h^2} \tag{3}$$

in which l represents the distance between the supports (50 mm), b is the specimen's width (13 mm) and h is the specimen's height (6 mm). The specimen dimensions are determined employing a caliper gauge prior to the measurement at an accuracy of ±0.01 mm.

For electrical heating purposes and for corrosion experiments, an Elektro-Automatik EA-4036 laboratory power supply was used in a 50 Hz AC mode. Infrared images were recorded by a FLIR i50 thermal imaging system.

Table 1
Properties of carbon fibres used in the studies.

Fibre	Diameter [μm]	Density [g/cm^3]	Tensile strength [MPa]	Young's modulus [GPa]	Fibre length [mm]	Supplier
HT C261	7	1.76	3950	230	3	Toho Tenax
HT C261	7	1.76	3950	230	6	Toho Tenax
CF-1 mm	7	1.7–2.0	3500	230	1	Procotex

Table 2
Composition of the cement mixture.

Material	Weight percent	Supplier
Cement Portland Type I	42.5	Schwenk Zement KG
Sand	34.5	–
Silica fume	8.0	Elkem AS
Water	14.0	–
Water reducing agent	1.0	BASF

3. Results and discussion

In conductive fibre reinforced cement-based composites, the properties and structure of non-conductive components, like sand or cement, do not affect the composites' conductivity [7]. The electrical conductivity of the composites depends merely on the volume fraction of carbon fibres, if uniform (in length and diameter) carbon fibres are being used [7]. Compared to concrete (having conductivity between 10^{-5} and 10^{-8} Ω^{-1} cm^{-1}, depending on the moisture content), carbon fibres possess by several orders of magnitude higher electrical conductivity (10^2 Ω^{-1} cm^{-1}) [15]. In the following sections the influence of fibre aspect ratio (defined as the ratio of fibre length to diameter) and volumetric fraction is studied in order to determine the optimal fibre dimensions and volume fraction for developing an in-situ heatable carbon fibre reinforced cement-based composite.

3.1. Fibre aspect ratio

It was shown that the fibre aspect ratio influences electrical conductivity of carbon fibre reinforced cement-based composites by using 1–15 mm long fibres [6]. Since shorter fibres (= lower aspect ratio) provide better workability of the cement paste it is reasonable to determine the optimal fibre length and subsequent

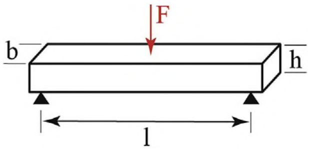

Fig. 1. Sketch of 3-point bending test for flexural strength measurement.

the optimal fibre aspect ratio (Table 3). The fibre diameter was 7 μm in all experiments, thus a low fibre aspect ratio of 140 was achieved for 1 mm fibres, a medium aspect ratio of 430 for 3 mm and a high aspect ratio of 860 for 6 mm long fibres. As it can be seen in Table 3, there are large differences in conductivity between the composites containing fibres having a low aspect ratio and the composites containing fibres having medium and high aspect ratios. The specimens containing low aspect ratio fibres exhibit electrical conductivity by 5 orders of magnitude lower than those containing medium and high aspect ratio fibres, at all tested fibre volume fractions. This can be ascribed to the fact that too short fibres (1 mm in length) cannot form a continuous fibre crosslinking network at fibre volume fractions between 1 and 2 vol. %. The conductivity increases significantly for specimens containing 3 to 4 vol. % of low aspect ratio fibres (10^{-2} Ω^{-1} cm^{-1}), but high conductivity values which are comparable to specimens containing medium or high aspect ratio fibres (10^{-1} Ω^{-1} cm^{-1}), have not been reached. Hence, the fibre aspect ratio seems to be an important factor as long as the ratio falls below about 430; however, employing fibres with an aspect ratio greater than 430 does not lead to a further enhancement of electrical conductivity. Only for low fibre contents (about 1 vol. %), a better conductivity is detected for fibres having a high aspect ratio of 860. If the fibre volume content is raised up to 4 percent, the specimens containing high aspect ratio fibres provide slightly lower conductivity compared to the specimens containing medium aspect ratio fibres. This effect might be explained by the poor dispersion of long fibres (6 mm in length) at higher fibre volume fractions.

3.2. *Fibre volume content*

Depending on fibre volume fractions, the conductivity of fibre reinforced cement-based composites exhibits characteristic features of percolation phenomena. Conductivity increases by several orders of magnitude when the concentration of carbon fibres reaches a critical value (threshold). The conductivity increases only slightly with an increase of fibre content in the post-threshold region. This phenomenon can be described by percolation theory dealing with the effects of random variation in the number and quality of fibre contact bridges and has been studied before [8,9].

In the pre-threshold region (Fig. 2) at very low fibre volume fractions, the fibres are distributed evenly in the non-conductive cement-based matrix and almost no contacts between single fibres do exist. At higher fibre concentrations, agglomerates and clusters are formed by cross-linking of single fibres. Reaching the threshold concentration of fibre volume fraction, individual clusters are cross-linked and the conductivity increases by several orders of magnitude with moderately growing fibre content. When all clusters are cross-linked, the post-threshold region is reached [7].

The fibre aspect ratio was determined to be optimal for 400 and above (3 mm in length and 7 μm in diameter). The optimal fibre volume content for heating purposes has to be determined empirically, since a good conductivity is essential for low voltage heating systems. The best fibre content/conductivity ratio can be expected in the post-threshold region. In Fig. 2, the composite's conductivity is plotted against fibre volume content (in logarithmic scale). Between 0 and 0.1 vol. % fibre content, a pre-threshold region can be detected, in which conductivity rises only slightly. Between 0.1 and 1.0 vol. % fibre content, the threshold region is found showing a progressively increasing conductivity, ranging from 10^{-6} Ω^{-1} cm^{-1} to 10^{-2} Ω^{-1} cm^{-1}. At more than 1.0 vol.-% fibre content, a post-threshold region can be seen, in which the composite's conductivity shows only a marginal dependency on the fibre content, ranging from 3×10^{-2} Ω^{-1} cm^{-1} to 2×10^{-1} Ω^{-1} cm^{-1} by admixing 1.0 up to 4.0 vol. % carbon fibres. Thus, the minimum content of carbon fibres, required to achieve a sufficiently high composite conductivity, is about 1.0 vol.-% of fibre content. Since workability deteriorates significantly if the fibre content exceeds 2.0 vol. %, adjusting the carbon fibre content between 1.0 and 2.0 vol. % in the composite seems to provide a practical compromise for ensuring good electrical conductivity and workability.

Table 3
Electrical conductivity versus fibre aspect ratio and fibre volume fraction.

Fibre volume fraction [vol.-%]	Conductivity (aspect ratio = 140) [Ω^{-1} cm^{-1}]	Conductivity (aspect ratio = 430) [Ω^{-1} cm^{-1}]	Conductivity (aspect ratio = 860) [Ω^{-1} cm^{-1}]
1.0	$(2.0 \pm 0.2) \times 10^{-7}$	$(2.8 \pm 0.2) \times 10^{-2}$	$(3.8 \pm 0.1) \times 10^{-2}$
2.0	$(3.8 \pm 0.5) \times 10^{-4}$	$(8.6 \pm 0.2) \times 10^{-2}$	$(8.4 \pm 0.3) \times 10^{-2}$
3.0	$(1.0 \pm 0.4) \times 10^{-2}$	$(1.6 \pm 0.2) \times 10^{-1}$	$(1.5 \pm 0.4) \times 10^{-1}$
4.0	$(2.8 \pm 1.2) \times 10^{-2}$	$(1.9 \pm 0.2) \times 10^{-1}$	$(1.6 \pm 0.2) \times 10^{-1}$

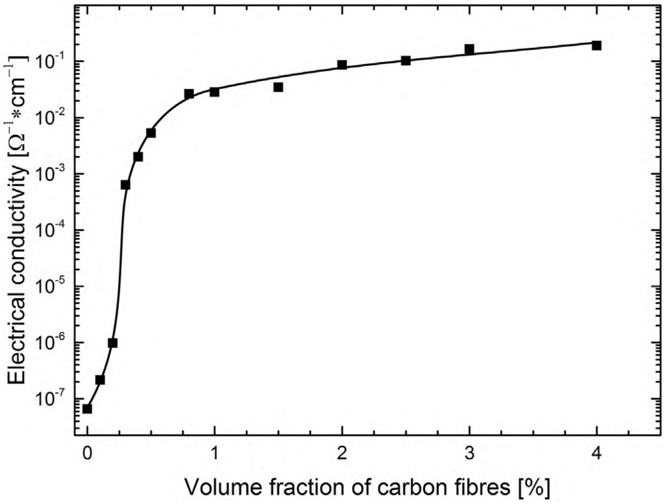

Fig. 2. Composite conductivity versus fibre volume fraction in logarithmic scale. Threshold region reaches from 0.1 to 1.0 vol. % and post-threshold region starting at 1.0 vol. % carbon fibre volume content.

3.3. *Electrode material*

Due to high pH matrix values of about 12.5 and high ion activity, cement-based materials are capable of corroding metals by forming metal oxides, hydroxides and carbonates. Additionally, applied electric currents can cause electrolysis of moisture producing oxygen at the electrode surface, which can amplify corrosion effects. Most research has been devoted to steel corrosion in concrete since steel is the most common reinforcement material [16–18]. In order to investigate which electrode material provides long-lasting and reliable conductivity in cement-based composites, copper, stainless steel, lead, graphite and silver electrodes were tested. The electrodes were embedded at each end of the specimen molds before the cement mixture was poured into the molds with 2 vol. % carbon fibre content (3 mm long fibres with 7 μm in diameter). For each electrode material, three specimens were manufactured and all specimens were cured as described in 2.2. After 4 weeks resistivity was measured. After that, 12 V AC voltage was applied to all specimens for 24 h with continuous electrical heating and resistivity was measured again. The results are summarized in Table 4.

Copper, stainless steel, graphite and silver electrodes provide good conductivity after 28 days of curing at resistances between

Table 4
Specimen resistance of different electrode materials.

Electrode material	Resistivity of specimens after 4 weeks curing (= initial resistance before heating) [Ω]	Resistivity of specimens after applying 12 V alternating voltage for 24 h [Ω]
Copper	$(5.7 \pm 2.5) \times 10^2$	$(1.1 \pm 0.5) \times 10^3$
Stainless steel	$(6.4 \pm 1.7) \times 10^2$	$(7.6 \pm 1.2) \times 10^5$
Lead	$(9.2 \pm 0.6) \times 10^4$	$(1.9 \pm 1.3) \times 10^6$
Graphite	$(1.5 \pm 0.4) \times 10^2$	$(1.9 \pm 0.5) \times 10^2$
Silver	$(3.0 \pm 0.9) \times 10^2$	$(1.4 \pm 0.3) \times 10^2$

$(1.5 \pm 0.4) \times 10^2$ and $(6.4 \pm 1.7) \times 10^2$ Ω. Graphite electrodes perform the best, whereas stainless steel electrodes show the lowest performance. Employing lead electrodes shows an increase by 2 orders of magnitudes $(9.2 \pm 0.6) \times 10^4$ Ω compared to the other electrode materials. Upon applying 12 V AC voltage for 24 h, copper, stainless steel and lead electrodes exhibit significantly enhanced resistances, which presumably is due to electrolysis and related corrosion effects reaching a maximum of up to $(1.9 \pm 1.3) \times 10^6$ Ω for lead electrodes. Both graphite and silver electrodes provide good conductivity; however, graphite electrodes show slightly higher resistances $(1.9 \pm 0.5) \times 10^2$ Ω after applying a 12 V AC voltage in contrast to silver electrodes which exhibit lower resistances $(1.4 \pm 0.3) \times 10^2$ Ω upon applying a 12 V AC voltage. Among tested electrodes, graphite and silver electrodes were found to provide the best corrosion resistance in cement-based composites, even if electrical voltages up to 12 V are being applied.

3.4. *Smart heating concept*

For presenting a smart heating concept for the presented composite material, a prototype cement plate having dimensions of 100 × 100 × 8 mm was fabricated containing 2 vol. % of carbon fibres (3 mm long carbon fibres with 7 μm in diameter). Smart heating concept implies that designated areas of the fabricated plate will be contacted exclusively for heating purposes. To enable heating in selected areas of the plate, a grid consisting of ten graphite electrodes (1 mm in diameter) was prepared (Fig. 3). Five electrodes were embedded in the upper part (number 1a to 5a) and five in the lower part (number 1b to 5b) of the plate. The upper and lower side electrodes were twisted by 90° to each other. Depending on which part of the plate should be heated, electrodes on the upper and lower side of the plate were connected in different patterns (Figs. 3 and 4). The heating principle is simple: The electrical current will flow through the smart heating plate and since the graphite electrodes exhibit higher electrical conductivity (about $3 \times 10^2\ \Omega^{-1}\ \mathrm{cm}^{-1}$) than the presented carbon fibre reinforced composite material (about $8 \times 10^{-2}\ \Omega^{-1}\ \mathrm{cm}^{-1}$) heat is generated inside the heating plate. The distance between electrodes on the same side of the plate was 20 mm; the minimum distance between electrodes on different sides of the plate was about 6 mm since the plate thickness was 8 mm. In order to heat only the left or right part of the plate, all electrodes on the lower side of the plate (1a to 5a) were connected to the AC power supply. In addition to that, for left side heating, electrode 1b, and for right side heating, electrode 5b were connected to the power supply. If electrodes 3a and 1b to 5b were connected to the power supply, it was possible to heat the middle part of the plate. To heat the whole plate surface, all electrodes need to be connected to the power supply. Fig. 4 shows that all cases discussed here could be realized with the prototype of a smart heating plate. A specific temperature in selected areas of the plate can be adjusted as well as uniform heating of the whole plate. In order to adjust the temperature of the plate surface, different AC voltages were applied to the graphite electrodes and the surface temperature was measured after 20 min of heating. For an AC voltage of 3 V a surface temperature of 34 C was achieved, if the voltage was raised to 6 V a surface temperature of 53 C was reached and by applying 12 V the surface temperature was pushed to 103 C.

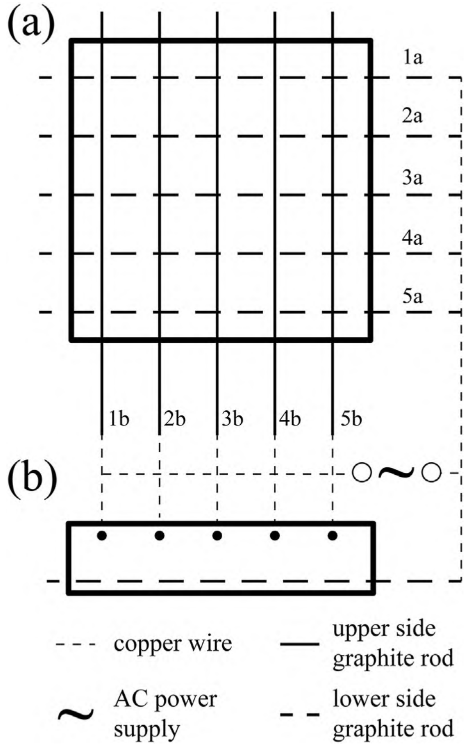

Fig. 3. Sketch of heatable plate and embedded graphite electrode grid for smart heating (a) top view (b) side view.

The experimental results show that specific areas of the smart heating plate can be addressed and a low AC voltage (3–12 V) is suitable to heat the plate surface to a designated temperature depending on the applied AC voltage. Thus the concept of a carbon fibre reinforced cement-based composite can be stated as suitable for heating floors and walls, since a surface temperature of 35 °C would be sufficient for practical application (e.g. heating of rooms). However it has to be proven as to whether or not the long-term mechanical strength of the material will not decrease upon continued and repeated electrical heating.

3.5. *Flexural strength before and after electrical resistance heating*

Electrical heating tests were carried out on specimens cured for 28 days. For the tests, the ends of the specimens were polished and covered with silver lacquer. All mechanical tests were performed 56 days after the specimen preparation. The flexural strength was determined according to Equation (3) via 3-point bending test. According to 3.1 and 3.2, 3 mm long and 7 μm thick fibres were used at a fibre volume content of 2 vol. %. The electrical conductivity of the specimens was $(8.6 \pm 0.2) \times 10^{-2}\ \Omega^{-1}\ \mathrm{cm}^{-1}$ at room temperature (Table 3). To investigate negative influences of resistive heating on the mechanical strength of the composite, six specimens of each batch were heated up to 60 °C in a desiccator for 1, 2, 3, or 4 weeks. Appling 8 V AC voltage was sufficient to keep the specimens at the designated temperature. When heated to 60 °C the conductivity of the specimens rised slightly to $(1.0 \pm 0.1) \times 10^{-1}\ \Omega^{-1}\ \mathrm{cm}^{-1}$ caused by the semiconductor characteristics of carbon fibres, since these

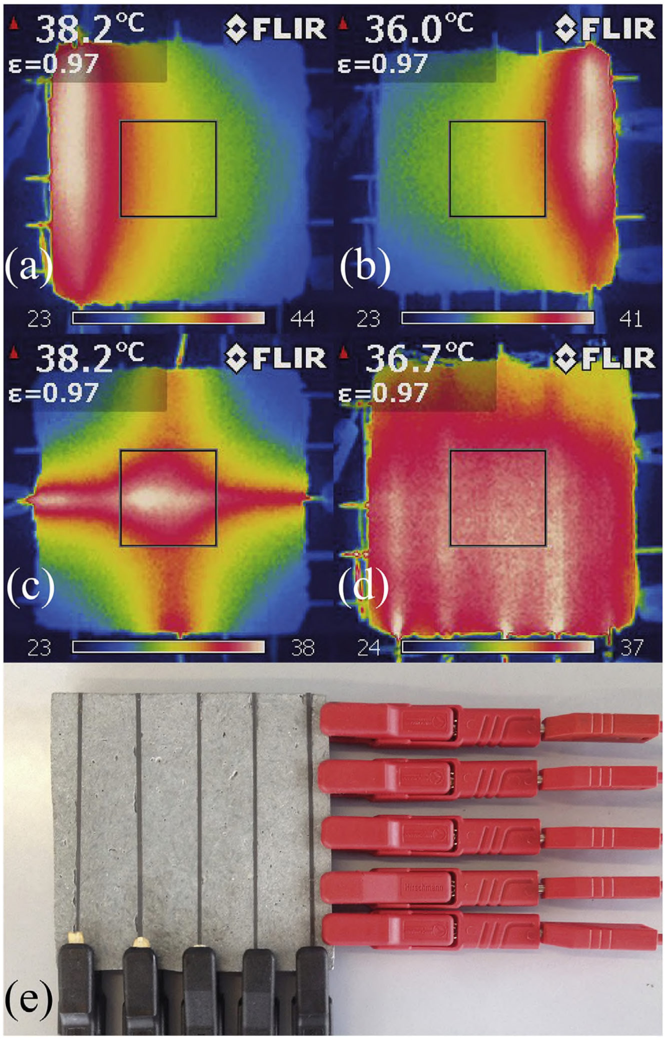

Fig. 4. Infrared images of smart plate heating (plate dimensions: 100 × 100 × 8 mm). (a) Electrodes 1a to 5a and 1b connected. (b) Electrodes 1a to 5a and 5b connected. (c) Electrodes 3a and 1b to 5b connected. (d) All electrodes connected. (e) Photograph of heating plate with all electrodes connected to power supply.

Table 5
Results of 3-point bending test.

Sample	Number of specimens	Flexural strength [MPa]
Reference, not heated	6	28.9 ± 3.6
1 week at 60 °C	6	32.3 ± 1.6
2 weeks at 60 °C	6	33.5 ± 3.2
3 weeks at 60 °C	6	34.4 ± 3.4
4 weeks at 60 °C	6	33.4 ± 1.7

materials exhibit higher electrical conductivity at elevated temperatures. The results of measuring flexural strengths are shown in Table 5 for each batch. By increasing the duration of resistive heating from 1 to 4 weeks at 60 °C, the flexural strength increases from 28.9 ± 3.6 MPa (not heated) to about 33.4 ± 1.7 MPa (heated for 4 weeks). The increase in flexural strength can be explained due to conversion of portlandite $Ca(OH)_2$ and silica fume into CSH phases at elevated temperatures [19]. Specimens heated for 3 weeks show slightly higher flexural strength when compared to specimens heated for 4 weeks, which might be caused by statistical variances since only 6 specimens were tested.

4. Conclusion

By screening matrix properties, we could empirically show that carbon fibre reinforced cement-based screed exhibits the best conductivity (and thus the best performance as floor heating material) if carbon fibres having an aspect ratio of about 400 (7 μm in diameter and 3 mm in length) are being used. Based on percolation theory, the fibre volume content suitable for electrical resistance heating is found to be 1 to 2 vol. % of carbon fibres. A permanent heating of carbon fibre reinforced composite at 60 °C for 4 weeks causes no measurable loss of strength, although additional long-term experiments should be performed to verify the long-term stability. All performed tests prove that a cement-based screed, suitable and applicable for in-situ electrical heating, can be fabricated by admixing 2 vol. % of carbon fibres into the composite and using graphite or silver electrodes for contacting purposes. Furthermore, it is shown that embedding a grid of electrodes into fibre reinforced cement plate opens up the possibility to heat selected areas of the plate up to 100 °C by applying an AC voltage of 12 V. Further studies on smart heating plates are needed to demonstrate that this concept can be scaled up and thus is suitable for similar plates exceeding dimensions of several meters.

Although electrical energy is high priced for heating purposes compared to wood, oil or gas, higher energy costs of the presented heating system can be compensated by a higher operating efficiency, since only desired areas can be heated. Additionally, the heated areas can always be changed just by reconnecting the grid of electrodes in the floor plate (e.g. when furniture is rearranged) which makes the system highly flexible. The simplicity of only admixing carbon fibres to a cement mixture and the absence of additional heating elements provide low system pricing. Thus carbon fibre reinforced cement-based composites can be stated as a suitable concept for floor and wall heating purposes.

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