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Thermal behavior of polypropylene fiber-reinforced concrete at elevated temperatures

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Abstract The influence of polypropylene (PP) fibers on thermal behavior of concrete at elevated temperatures was studied by simultaneous thermal analysis. Two kinds of polypropylene fibers differing in form and size were applied as fillers. Special emphasis was given on the thermal behavior of PP fiber-reinforced concrete subjected to heat treatment, i.e., at 200 and 300 °C. Thermal events of concrete samples with and without fibers were characterized. In comparison to concrete sample without fibers, the results showed that the presence of polypropylene fibers affected the thermal stability of concrete samples. It was found that the influence of the kind and the amount (1.8 vs. 3.0 kg m^{-3}) of PP fiber on the thermal stability of concrete samples was not significantly pronounced.

Keywords Polypropylene fibers · Reinforced · Concrete · Degradation · Simultaneous thermal analysis · Thermogravimetry · Spalling · Elevated temperatures

Introduction

Spalling is a phenomenon related to the ejection of concrete during fire [1–4]. It is reported that especially high-performance concrete displays higher propensity for heat-induced concrete spalling when exposed to severe heating or fire [5]. The occurrence of explosive spalling in concrete leads to deterioration in the load resistance of structures, drastically reduces their lifetime, and in some cases can lead to their collapse [6]. Up to now, the economically and technologically most worthwhile method to prevent explosive spalling is the addition of polypropylene fibers. It is noteworthy that the method is also recommended by Eurocode 2 [7]. The use of fiber-reinforced concrete in tunnel linings has increased in the two last decades, especially with regard to precast tunnel segmental linings and in underground structures [8].

Though the effectiveness of the fibers could be shown empirically, the mechanisms preventing explosive spalling are still debatable [1]. The results show that due to the thermal decomposition of the polypropylene fibers, microchannels are created and simultaneously connected due to a netlike microcrack formation. This enables the relief of internal stresses (mechanical effect) and the formation of a permeable transport system for the escaping water vapor (permeation effect).

The risk of fire spalling of concrete has been mainly studied, and some important results have been observed: the high risk of instability of high-performance concretes and the positive role of polypropylene fibers on the fire behavior of concrete were highlighted. Indeed, the addition of a few kilograms of fibers in fresh concrete can reduce or even eliminate the risk of fire spalling of structures [2].

Many studies presented the results on the influence of various fibers on the mechanical and/or physio-chemical properties of concrete at room and elevated temperatures [4–16].

The effect of different fibers (polypropylene and steel fibers) on the flexural behavior of the concrete exposed to normal and elevated temperatures (280 °C) was reported by Choumanidis et al. [10]. The residual flexural strength and the heat-resistant properties of the concrete exposed to

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high temperatures using different fiber cocktail loadings including steel, polymer or cellulose fibers were also studied [11].

The residual mechanical properties and spalling resistance of strain-hardening cementitious composite (SHCC) with hybrid PVA and steel fibers were investigated by Liu et al. [14]. Compressive strength tests were conducted on SHCC specimens at 30 °C, and exposed to 200, 400, 600 and 800 °C. The residual compressive strength of SHCC with hybrid fibers showed an increase after 200 °C heating. Beyond 200 °C, the residual compressive strength reduced with temperature increase.

Effects of the fiber type, dosage and length on the explosive spalling of ultrahigh-strength concrete under rapid heating and rapid cooling were experimentally investigated [17]. It was reported that the polypropylene (PP) fiber dosage affected the spalling resistance of concrete. The spalling resistance was poor when the dosage was lower than 0.20%. Meanwhile, the spalling resistance was best for a PP fiber dosage of 0.20% or more, in which case the compressive strength residual rate exceeded 70% and the mass loss rate was within 8%. The PP fiber length affected the spalling resistance of the concrete specimen. Specimens with PP fiber lengths of 6 and 9 mm underwent appreciable explosive spalling, while specimens with PP fiber length of 12 and 19 mm had appreciable spalling resistance at high temperature. Improvement of the spalling resistance of ultrahigh-strength concrete by PP fiber was explained by the formation of tubular channels from the external to internal via the melting of the PP fiber. It was explained that, additionally, there was an increase in capillary porosity due to the dehydration of hydration products. Thus, the combination of these behaviors results in the release of internal vapor pressure and prevents specimen spalling.

The effect of using steel fibers in addition to polypropylene fibers to reduce explosive spalling of concrete was investigated [18]. The combination of PP and steel fiber (hybrid) exhibited the best performance for spalling reduction. It was believed that this reduction could be attributed to the steel fiber resisting the initiation and expansion of cracks in the concrete matrix, while the melting action of the polypropylene fiber created microchannels in the concrete mass which alleviated vapor tension.

Starting from the identification of the permeability as the parameter with the greatest influence on spalling, the results of permeability tests on normal-strength in situ concrete without and with PP fibers (1.5 kg m^{-3}) were presented [19]. The values for the permeability, which are obtained for concrete pre-heated to different temperature levels, are related to the pore structure, accessible by mercury-intrusion porosimetry (MIP) tests.

The results from an experimental study on the optimum amount of polypropylene fibers to be used in lightweight high-strength concrete to prevent spalling when exposed to hydrocarbon fire, taking into consideration the characteristics of the lightweight aggregate, the water-to-cement ratio (W/C) of the mixtures, and the length and thickness of the fibers were presented [20].

The effects of various fibers, i.e., polypropylene (PP), jute, and water-soluble polyvinyl alcohol (WSPVA) fibers, on high-temperature spalling in high-performance concrete (HPC) was studied [21]. The vapor pressures of the water inside the HPC specimens reinforced with the three types of fibers were compared with the saturated vapor pressure of water in the specimens. It was reported that the jute, WSPVA, and PP concrete specimens did not spall explosively. The maximum vapor pressures of water within the jute, WSPVA, and PP specimens were 2.5, 1.5, and 1.0 MPa, respectively.

While the knowledge and experience with fiber-reinforced concrete behavior under ambient temperature are well known, the behavior under elevated temperature has to be deeply investigated. Most studies focus on observing the behavior of fiber-reinforced concrete under ambient and elevated temperature with the aim of understanding the thermo-physical mechanisms leading to spalling, thus studying the factors which influence its occurrence, and to determine the mechanical properties of the material. Therefore, fundamental knowledge of the effect of elevated temperatures on the thermal properties of polypropylene fiber-reinforced concrete, particularly the thermal stability behavior of PP reinforced composites, is scarce.

The present work focuses on studies of the polypropylene fiber-reinforced concrete by the simultaneous thermal analysis (STA) method. Special emphasis is given on the thermal behavior of PP fiber-reinforced concrete when exposed to elevated temperatures.

The aim of the work was to investigate the influence of polypropylene fibers on the thermal behavior of the concrete subjected to elevated temperatures, i.e., 200 and 300 °C. Different kinds and amounts of polypropylene fibers were used. The polypropylene fibers studied differed in their form (monofilament vs. fibrillated, bundled) and size. The tests were carried out on the polypropylene fiber-reinforced concrete and unmodified concrete (control specimens).

Experimental

Materials and methods

Portland cement CEMI I 42.5 R (Lafarge) was used in this work. The cement was tested in accordance with EN

197-1:2012P. According to the test results, the chosen type of cement is appropriate for the mixes. To eliminate the influence of coarse aggregate, the fine aggregate was used, i.e., river sand (fraction 0/2 mm). Silica fume SILIMIC, 90% composed of SiO_2, is most commonly used as an active additive to concrete and building materials silica (Huta Łaziska). Specific surface: 15–35 $m^2\ g^{-1}$. The superplasticizer (polycarboxylate-based Chrysofluid Optima 185) was used to adjust the workability of the concrete mixes. The superplasticizer density was 1.05 kg dm^3 with pH 5, 5 ± 1.

Two types of polypropylene fibers were used: monofilament Ignis, Schomburg Retheimer (abbreviation: I), and fibrillated Fibrofor, Brugg Contec AG (abbreviation: F).

The images of the PP fibers are given in Figs. 1 and 2. Table 1 specifies the properties of the PP fibers according to the producers' information.

Preparation of concrete samples

Concrete samples were prepared in accordance with the mix design shown in Table 2.

The materials were mixed in a laboratory mixer in the following order: cement, silica fume, and sand. These materials were dry mixed until they were homogenous. Then, superplasticizer and water were added and mixed for about 3 min. Finally, the polypropylene fibers were added and mixed. The polypropylene fibers were added according to the following ratios: 0, 1.8 and 3 kg m^{-3}. The finished batches were poured into molds and consolidated using a vibrating table. After 24 h, the specimens were held under water for 27 days. Then, the specimens were maintained in the climate chamber for 60 days at a temperature of 20 °C and a relative humidity of 99%. The specimens in the form of "eight" were cast for further studies. The sample size conformed to Polish standard PN-B-4500 used for the studies of physical and mechanical properties of cement mortars. The tensile properties of concrete samples with and without PP fibers were determined (unpublished data). The exposure of polypropylene fiber-reinforced concrete to elevated temperature decreased its tensile strength. However, the tensile strength of PP-reinforced concrete samples was higher in comparison with the tensile strength of samples without fibers for each heat treatment temperature,

Fig. 1 Polypropylene fibers "F"

Fig. 2 Polypropylene fibers "I"

Table 1 Polypropylene fiber characteristics (according to producer's data)

Property	Fiber	
	Ignis/"I"	Fibrofor High Grade 190/"F"
Color	Transparent	Beige
Form	Monofilament	Fibrillated, bundled
Length/mm	12	19
Diameter/μm	18	80
Bulk density/g cm^{-3}	0.91	0.91
Tensile strength	min 28 cN tex^{-1}[a]	~400 N mm^{-2}
Softening point/°C	≈165	≈150

[a] cN tex^{-1}—tensile strength unit for fiber, where tex is a unit for the linear mass density of fibers

Table 2 Composition of the studied concrete samples

Components	Abbreviation				
	0F	1.8F	3.0F	1.8I	3.0I
Cement/kg m^{-3}	846	846	846	846	846
Silica/kg m^{-3}	84.6	84.6	84.6	84.6	84.6
Sand/kg m^{-3}	1249	1249	1249	1249	1249
Plasticizer/% cement mass	2	2	2	2	2
Water/dm^3	215	215	215	215	215
Polypropylene fibers/kg m^{-3}	0	1.8	3.0	1.8	3.0

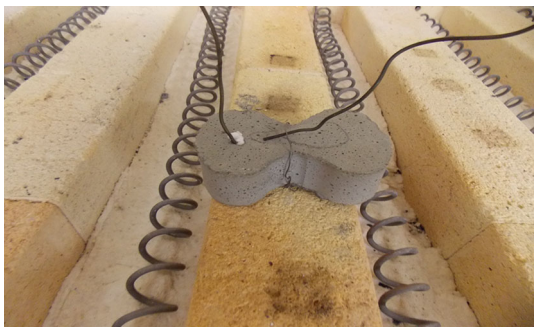

Fig. 3 Arrangement of concrete sample in the furnace

i.e., 100, 200, 300, 400, 500 and 600 °C. Seven specimens were used for measurement of tensile strength of concrete sample after heat treatment at a chosen temperature. Example of an image of a concrete sample is given in Fig. 3.

Heat treatment of samples

Concrete samples with and without polypropylene fibers were subjected to high temperatures, i.e., 200 and 300 °C. They were placed in the furnace and heated until a given temperature was reached. The heat treatment process followed the standard fire exposure curve according to ISO 834. The temperature inside and outside the samples was monitored by thermocouples. Three thermocouples were applied: thermocouple TR (temperature control) measuring the temperature inside the furnace; and two thermocouples: T1 on the surface of the concrete sample and T2 in the core of the concrete sample (halfway through the height).

The image of the concrete sample and arrangement of thermocouples in the furnace are given in Fig. 3. An example of the heating curve is presented in Fig. 4.

For comparison, one series of concrete samples was not subjected to heat treatment.

The abbreviation used for the sample:

0F/Temperature—amount of fibers and kind of fiber/ treatment temperature

For example:

0F/20C—concrete sample without fibers; no heat treatment
1.8I/200C—concrete sample containing 1.8 kg m^{-3} polypropylene fibers (Ignis), subjected to temperature 200 °C.

STA analysis

The thermal analysis was carried out using a simultaneous TGA/DSC unit, model SDT Q600 from TA Instruments. Samples were heated from 30 to 1000 °C at a constant rate of 10 °C min^{-1} in the air atmosphere (air flow: 100 L min^{-1}). For STA measurements, 14 samples were taken from tensile strength concrete samples. The sample mass was about 30 mg.

Results and discussion

STA analysis of concrete samples without polypropylene fibers

The degradation of concrete samples due to exposure to elevated temperatures was investigated by STA. Figures 5 and 6 show examples of the TG/DTG/DTA profiles of concrete sample untreated and subjected to a temperature of 300 °C, respectively. The effects corresponding to

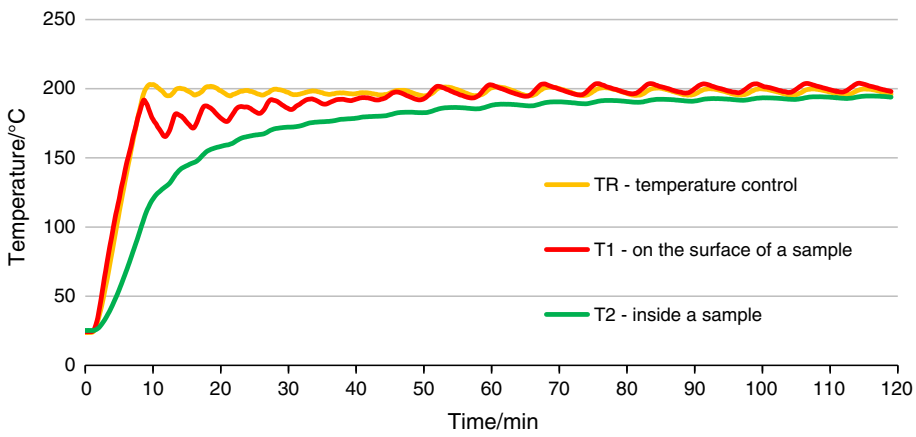

Fig. 4 Example of heat treatment of concrete sample at 200 °C

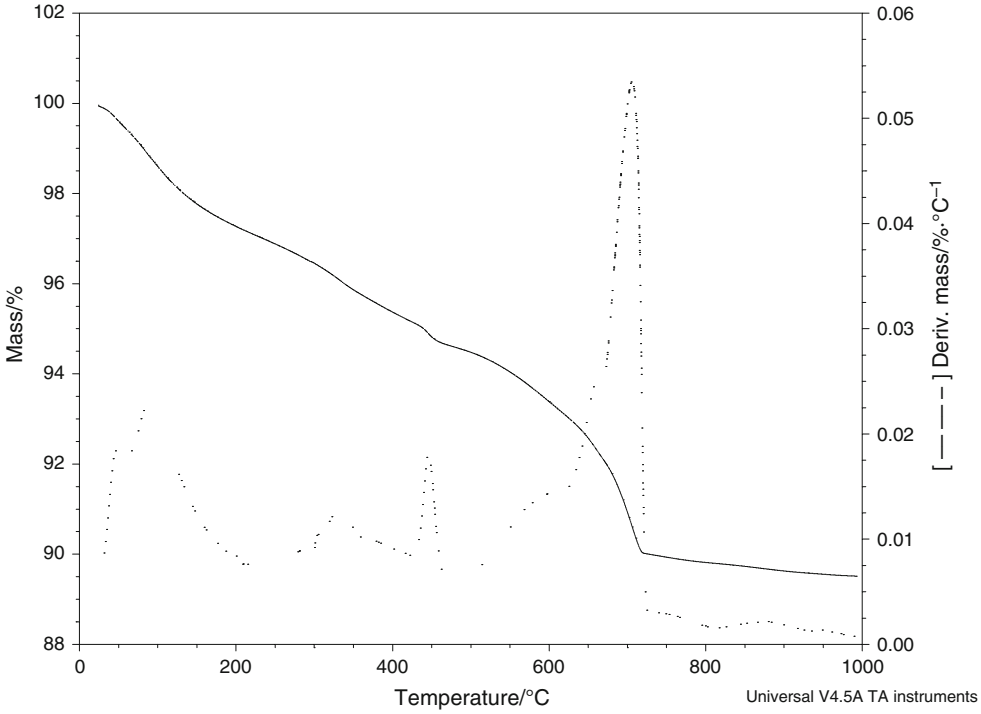

Fig. 5 TG/DTG curves of concrete sample 0F/20C

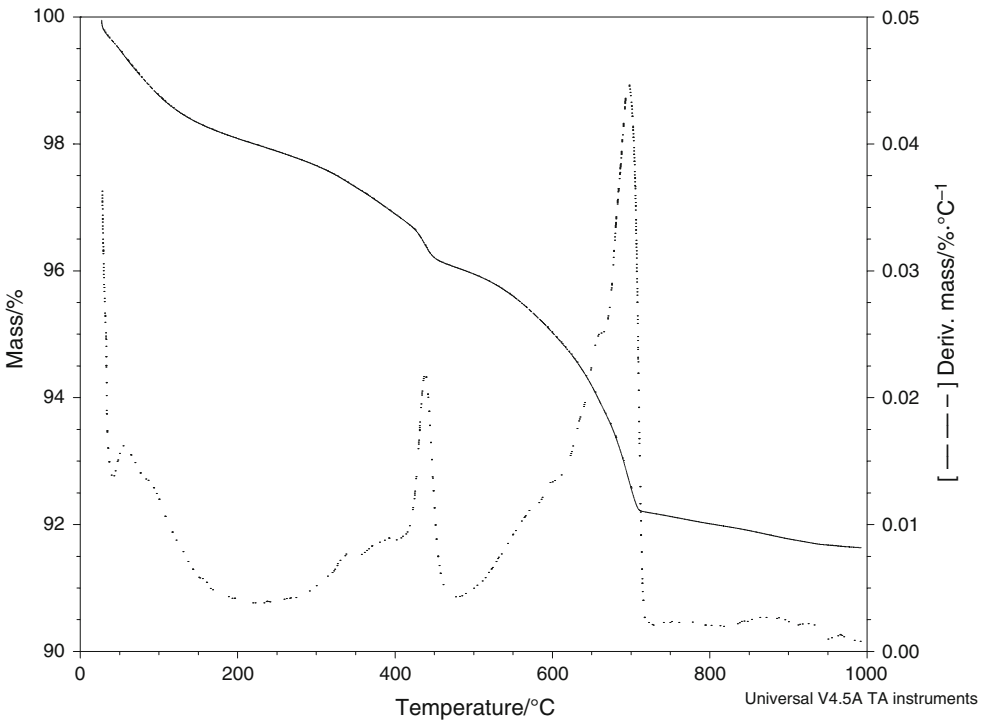

Fig. 6 TG/DTG curves of concrete sample 0F/300C

dehydration and degradation of products of hydration and carbonation are seen. The degradation process can be divided into three significant stages based on the DTG profile. The results are shown in Table 3.

The first step between 35 and 200 °C is related to the drying (capillary pore residual water) and/or with the dehydration of ettringite [3, 21–25]. It is usually associated with overlapping peaks that are likely to take place which includes capillary pore water, interlayer water and adsorbed water [22]. At higher temperatures, dehydroxylation of calcium hydroxide $Ca(OH)_2$ can be observed, which occurs between 370 and 470 °C [21–24].

Table 3 DTG peak temperatures of concrete samples without PP fibers

Concrete sample	Peak temperature Tp_1/ °C	Peak temperature Tp_2/ °C	Peak temperature Tp_3/ °C
0/20C	85.0	445.6	706.0
0/200C	48.8	441.5	699.9
0/300C	56.3	438.1	696.6

The third mass loss step at about 700 °C can be attributed to the decomposition of calcium carbonate $CaCO_3$ and other carbonates present in the initial cement composition due to the loss of CO_2 [23–25].

The following reactions occur [3]:

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \text{ (carbonation)},$$

$$CaCO_3 \rightarrow CaO + CO_2 \text{ (decarbonation)}.$$

With increase in the temperature of heat treatment, a shift of peak temperature of the corresponding stages of degradation is observed toward lower temperature.

The thermal effects observed on DTA curves can be identified as follows (cf. Fig. 7):

- the endothermic effect with mass loss, at peak temperature between 55 and 110 °C, corresponding to the liberation of physically bound water from pores and dehydration of the CSH phase;
- the exothermic effect with peak temperature about 333 °C probably associated with tobermorite transition;
- the endothermic effect of dehydroxylation of $Ca(OH)_2$—portlandite at peak temperature about 446 °C linked with mass loss;
- the endothermic effect at peak temperature 573 °C probably associated with quartz transition;
- the endothermic effect at peak temperature 710 °C associated with decarbonation of calcium carbonate.

The DTA peak temperatures of heated concrete samples corresponding to the endothermic effects of dehydroxylation of $Ca(OH)_2$ and decarbonation of calcium carbonate are slightly shifted toward lower temperature in comparison with untreated concrete samples (see Fig. 7). Moreover, with increase in heat treatment temperature, the peak temperature shifts toward a lower temperature. Thus, the DTA peak temperature of untreated concrete sample corresponding to dehydroxylation of portlandite (449.9 °C) is shifted to 446.3 and 443.2 °C for concrete samples heated to 200 and 300 °C, respectively. Similarly, the DTA peak temperature of untreated concrete sample corresponding to decarbonation of calcium carbonate (710.5 °C) is shifted to 704.8 and 698.7 °C for concrete samples heated to 200 and 300 °C, respectively. It is observed that the endothermic effect at 573 °C is not affected by heat treatment. The peak is attributed to quartz transformation from α rhombohedral shape to β hexagonal shape [21].

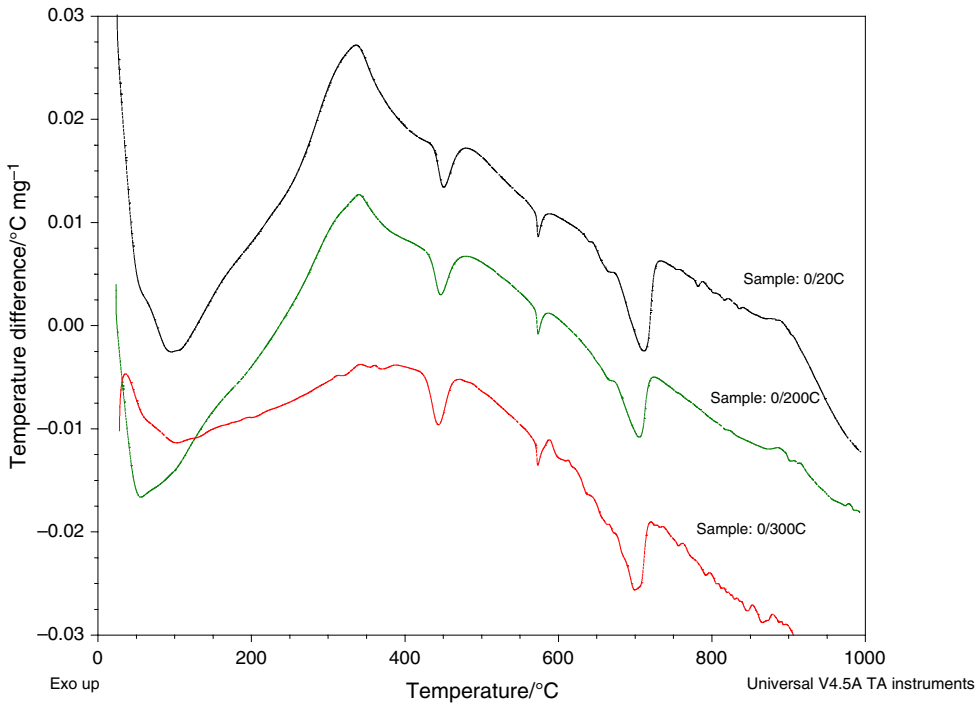

Fig. 7 DTA curves of concrete sample without fibers subjected to elevated temperatures

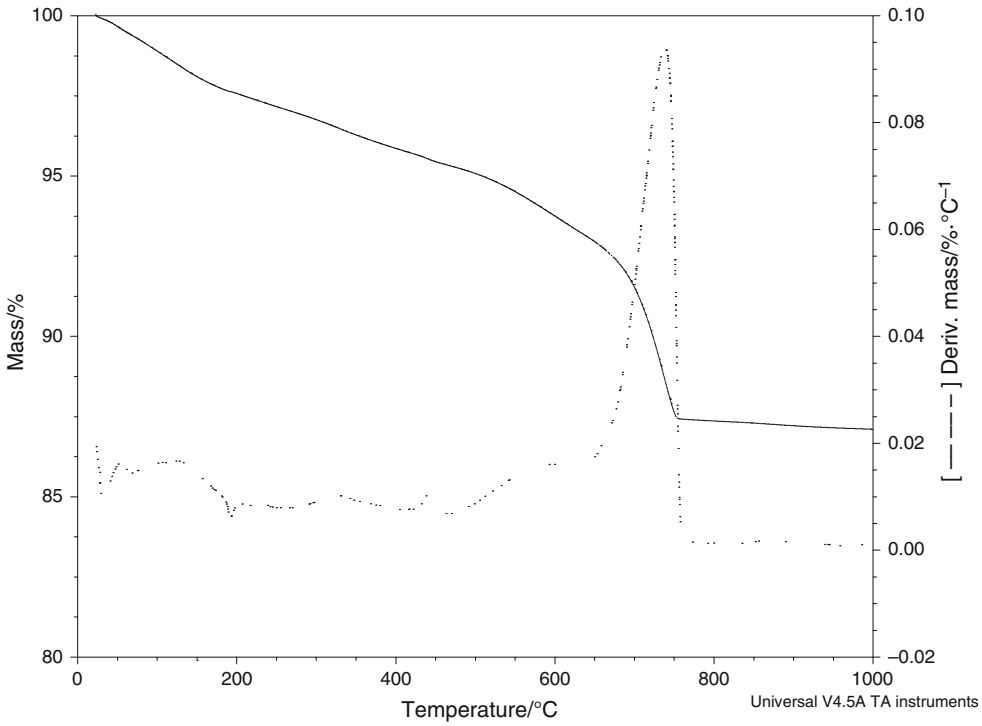

Fig. 8 TG/DTG curves of concrete sample 1.8F/20C

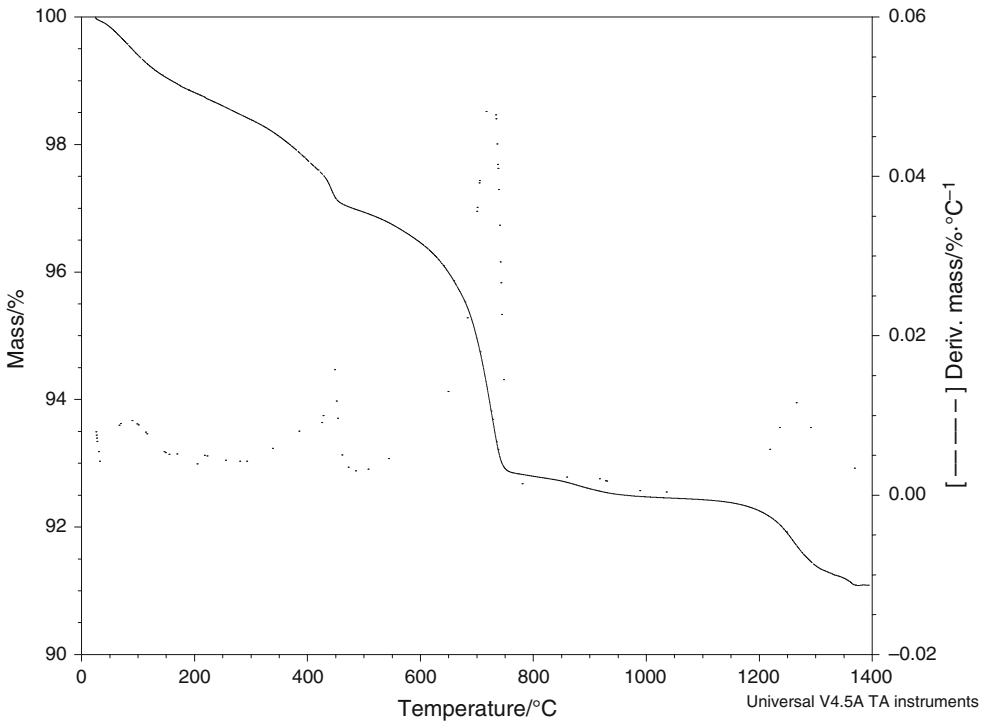

Fig. 9 TG/DTG curves of concrete sample 1.8F/300C

STA analysis of polypropylene fiber-reinforced concrete samples

Examples of the TG/DTG curves for polypropylene fiber-reinforced concrete samples are given in Figs. 8 and 9, respectively, for unheated concrete sample and concrete sample after heat treatment at 300 °C. The TG/DTG curves of polypropylene fiber-reinforced concrete samples are similar to the ones without fibers. Three main degradation stages are observed corresponding to water loss at

Table 4 DTG peak temperatures of PP fiber-reinforced concrete samples

Concrete sample	Peak temperature Tp_1/ °C	Peak temperature Tp_2/ °C	Peak temperature Tp_3/ °C
1.8/F/20C	51.5	440.0	738.1
1.8/F/200C	94.5	440.9	719.3
1.8/F/300C	85.7	441.7	725.9
1.8/I/20C	82.1	433.8	697.8
1.8/I/200C	89.5	337.9	699.2
3.0/F/20C	90.2	439.5	712.8
3.0/F/200C	88.8	440.3	718.6
3.0/F/300C	70.8	440.4	718.3
3.0/I/20C	93.6	441.8	715.5
3.0/I/200C	80.1	441.9	715.1
3.0/I/300C	74.9	439.6	721.7

30–100 °C, dehydroxylation of portlandite at 400–500 °C and decarbonation of calcium carbonate at 700–800 °C, respectively (cf. 8 and 9). The DTG peak temperatures of PP fiber-reinforced concrete samples are summarized in Table 4. The influence of the kind and amount of PP fibers on the DTG curves of concrete samples is not evident. Similarly, it seems that the heat treatment does not influence the DTG curves pattern significantly.

The DTA curves of polypropylene fiber-reinforced concrete samples show a similar pattern to the concrete sample without fibers (cf. Figs. 7 and 10). The main thermal effects observed are related to water evaporation and C–S–H dehydration (DTA peak temperature range: 55–105 °C), $Ca(OH)_2$ decomposition (DTA peak temperature range: 442–450 °C) and $CaCO_3$ decomposition (DTA peak temperature range: 703–745 °C). It was reported that polypropylene fibers melted, vaporized and burned at 170, 341, and 447 °C, respectively [19]. However, these endothermic effects related to polypropylene fibers overlapped with DTA peaks corresponding to concrete behavior. Melting of polypropylene fibers is important from the point of view of theories explaining the efficiency of polypropylene fibers in reducing the risk of concrete fire spalling. Melting of fibers allows 'draining' water out of the first centimeters of concrete, resulting in highly increased permeability of the first centimeters of exposed concrete [4]. Pore pressure measurements performed on heated concrete specimens showed that the presence of fibers led to a large decrease in the extent of the pressure fields that build up in the porous network during heating [21]. It was reported that the permeability measurements carried out after various heat treatments and for various fiber dosages showed the striking effect of fibers from 200 °C up, that is, very close to their melting temperature [21].

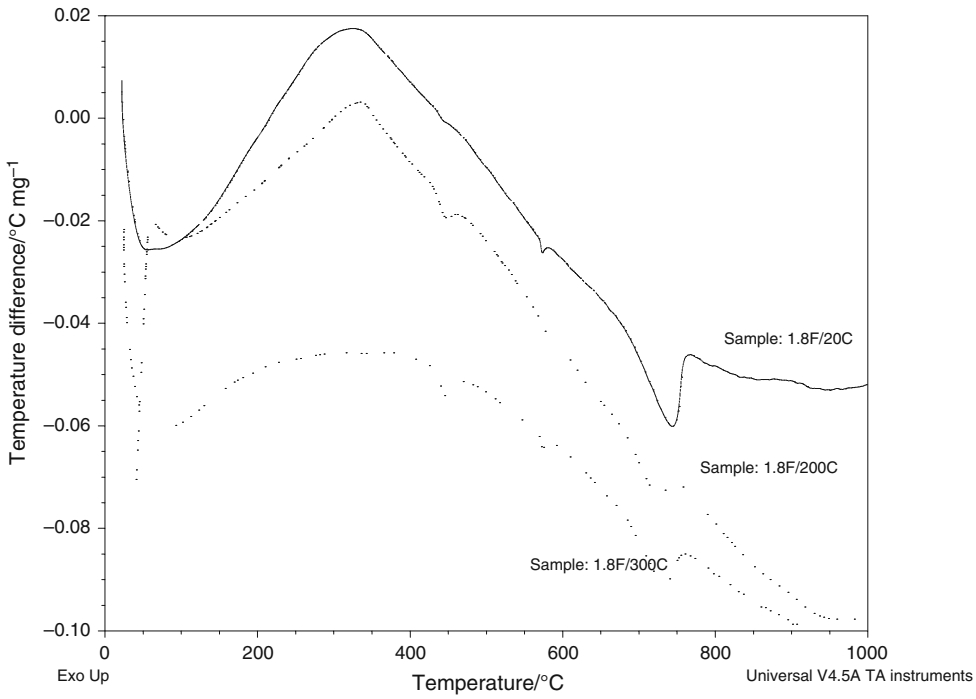

Fig. 10 DTA curves of concrete sample containing polypropylene fibers (1.8 kg m^{-3}, Fibrofor) subjected to higher temperatures

Table 5 Results of thermogravimetric analysis of polypropylene fiber-reinforced concrete samples

Sample	Mass loss W100C/%	Mass loss W100–450C/%	Mass loss W450–520C/%	Mass loss W600–900C/%	Residue W1000C/%
0/20C	1.3	3.8	0.5	3.8	89.6
0/200C	1.3	3.5	0.3	2.8	91.3
0/300C	1.2	2.6	0.4	3.3	91.7
1.8/F/20C	1.1	3.4	0.6	6.5	87.1
1.8/F/200C	0.8	3.5	0.4	3.2	91.5
1.8/F/300C	0.6	2.2	0.3	3.9	92.5
1.8/I/20C	1.1	2.3	0.5	2.1	93.1
1.8/I/200C	0.5	2.1	0.5	2.3	93.3
3.0/F/20C	1.3	3.6	0.3	2.8	91.4
3.0/F/200C	0.9	3.2	0.2	2.7	92.5
3.0/F/300C	0.5	1.9	0.3	2.9	93.8
3.0/I/20C	1.8	4.6	0.3	3.0	89.8
3.0/I/200C	0.9	3.2	0.3	2.9	92.2
3.0/I/300C	0.7	2.2	0.3	3.5	92.7

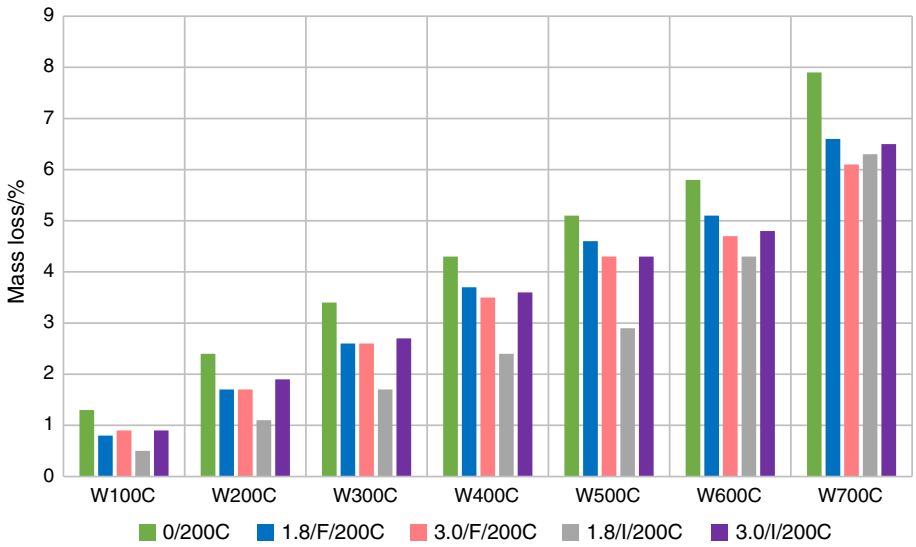

Fig. 11 Mass losses up to given temperatures for concrete samples after heat treatment at 200 °C

It was found that endothermic event above 700 °C attributed to decarbonation of $CaCO_3$ is shifted to higher temperature for polypropylene fiber-reinforced concrete samples in comparison with unreinforced concrete sample.

The results of thermogravimetric analysis are given in Table 5. Mass loss associated with water evaporation ranges between 0.5 and 1.8%. The endothermic effect corresponding to CSH dehydration accompanies a mass loss of 1.9–4.6%. Mass loss due to the C–S–H dehydration between 100 and 450 °C is smaller for concrete samples heated to 200 and 300 °C, in comparison with unheated concrete sample regardless of the amount and kind of polypropylene fibers. The mass loss in the temperature range 450–520 °C corresponding to $Ca(OH)_2$ decomposition is similar, indicating that the concrete samples had the same amount of portlandite. Similar results were reported by Ozawa and Morimoto [21]. The mass loss corresponding to carbonate decomposition is in the range 2.1–6.5%. The total mass loss up to temperature 1000 °C reaches 7–13%. The smallest mass loss is observed for concrete sample reinforced with polypropylene fiber "F" in the amount of 3.0 kg m^{-3} heated at 300 °C.

Mass losses up to a given temperature for polypropylene fiber-reinforced concrete samples after heat treatment at 200 and 300 °C are given in Figs. 11 and 12, respectively. It was found that the mass losses for polypropylene fiber-reinforced concrete samples were smaller than for corresponding concrete samples without fibers. The behavior is similar regardless of the temperature of heat treatment, i.e., for concrete samples heated to 200 and 300 °C mass losses

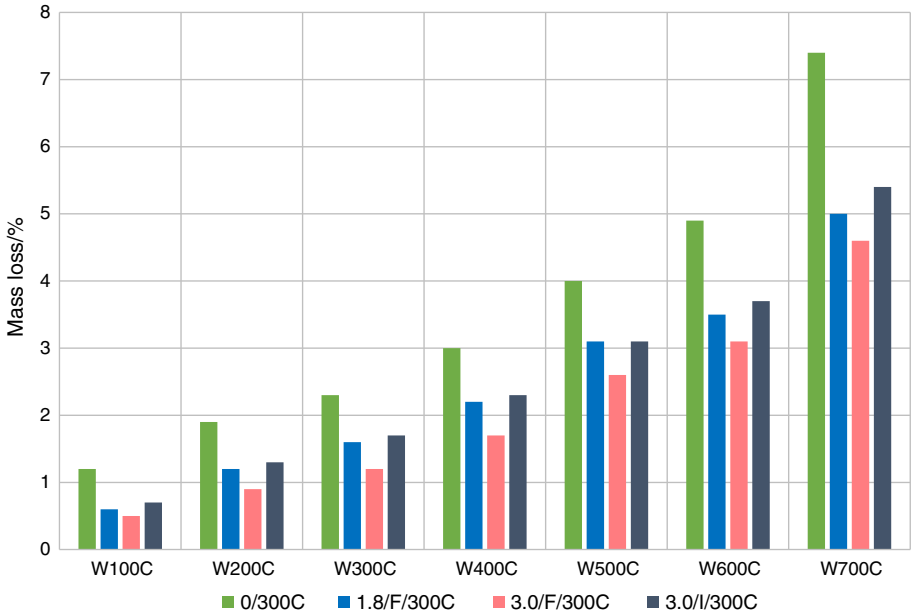

Fig. 12 Mass losses up to given temperatures for concrete samples after heat treatment at 300 °C

were smaller for concrete samples with polypropylene samples in comparison with concrete ones without fibers. It seems to indicate that the presence of polypropylene fibers influences the thermal stability of concrete samples. However, the influence of the kind, i.e., the form (monofilament vs. fibrillated, bundled) and the size of the polypropylene fibers is not significantly pronounced. Similarly, the amount of polypropylene fibers (1.8 vs. 3.0 kg m^{-3}) seems to not affect significantly the thermal stability of concrete samples.

Conclusions

The thermal behavior of the polypropylene fiber-reinforced concrete samples subjected to a high temperature, i.e., 200 and 300 °C, was studied by simultaneous thermal analysis.

Thermal events were characterized and compared to concrete samples without polypropylene fibers. It was observed that DTG and DTA curves of polypropylene fiber-reinforced concrete samples and concrete samples without fibers showed a similar pattern. Interestingly, it was found that the endothermic event above 700 °C attributed to decarbonation of $CaCO_3$ was shifted to higher temperature for concrete samples containing polypropylene fibers, in comparison to unreinforced concrete sample.

In comparison to concrete sample without fibers, the results obtained showed that the presence of polypropylene fibers affected the thermal stability of concrete samples. It seems based on the results that reinforcement with PP fibers increased the thermal stability of concrete samples exposed to elevated temperatures such as 200 and 300 °C. It may be explained by the combination of dehydration of hydration products and melting of PP fibers after heat treatment which affects the residual permeability of heated concrete samples and leads to changes in the formed structure.

Moreover, based on the results within the scope of the work, the influence of the kind (monofilament vs. fibrillated, bundled), the size (12 and 19 mm length), as well as the amount (1.8 vs. 3.0 kg m^{-3}) on thermal behavior and thermal stability is not significantly pronounced. It was reported elsewhere [5] that relatively short (3 mm long) PP fibers exhibited a higher propensity for spalling than practically identical mixes (equivalent PP fiber dose) with longer fibers (6 or 12 mm long). Thus, the results need further studies involving a wider range of parameters.

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