Behavior of Portland Cement Pastes Admixed With Nano-Iron Oxide at Elevated Temperature

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Abstract- Nanotechnology helps in producing materials with prospective properties, for each field of science (physics, chemistry, bio-science as well as construction materials). Nano-materials cover the nanoscale of hydration products of cement. The aim of this work is to investigate the influence of nano-iron oxide (NF) on the fire resistance of OPC pastes. NF was synthesized by thermal decomposition of basic ferric acetate fired at 275, 600 and 800°C. The crystal size of the prepared NFs was determined by TEM technique which was found to be 14.6, 16.98 and 18.68 nm, respectively. OPC admixed with 1 wt. % NF prepared at 275°C shows higher fire resistance compared with those admixed with 2 or 3 wt. %, It gives the higher bulk density, compressive strength and lower porosity up to 450°C than the blank OPC. As the firing temperature of NF increases the fire resistance diminishes.

Keywords- Nano ferric oxide; Fire resistance; Bulk density; Total porosity; Compressive strength

I. INTRODUCTION:-

Nanotechnology has changed our vision, expectations and abilities to control the material world. The developments in nano-science can also have a great impact on the field of construction materials and other fields in science (physics, chemistry, bioscience, etc.) [1]. Also, Nano-technology is one of the most active research areas with both novel science and useful applications that has gradually established in the last two decades. Nanoparticles belong to be prospective materials in the field of civil engineering which have a high-surface area to providing high chemical reactivity [2]. They act as nucleation centers, contributing to the development of the hydration of PC. However, due to the high surface area very strong reactive properties can be obtained.

Concrete is a construction material most used in the world. Several types of concrete have emerged in recent years than ordinary concrete such, the high performance concretes, self-compacting concrete and eco-concrete. Nano materials cover the nano-scale of hydration products of cement; the use of nano-particles increases the strength and durability of cementitious composites. Concrete materials are likely exposed to high temperatures during fire [3-8]. Strength, modulus of elasticity and volume stability of concrete are significantly reduced during these exposures. This may result in undesirable structural failures [5-12]. Therefore, the properties of concrete after a fire are of still importance for determining the load carrying capacity and for resisting fire-damaged constructions [13].

High temperature causes dramatic physicochemical changes, resulting in deterioration of concrete [14, 15]. The dehydration or the release of chemically bound water from the calcium silicate hydrate (CSH) becomes significant above about 110°C [16]. The dehydration of C-S-H and thermal expansion of the aggregate increase internal stresses and from 300°C micro-cracks are induced through the material [13]. Calcium hydroxide CH, which is one of the most important compounds in cement paste, dissociates at around 530°C giving the shrinkage of concrete [6, 7, and 17]. The fire is generally extinguished by water and CaO turns into Ca(OH)₂ causing cracking and crumbling of concrete [17]. Therefore, the effects of high temperatures are generally visible in the form of surface cracking and spalling [5, 9, 11, 18-20]. The alterations produced by high temperatures are more evident when the temperature surpasses 500°C. Most changes experienced by concrete at this temperature are irreversible [3]. CSH gel, which is the strength giving compound of cement paste, decomposes further above 600°C. At 800°C, concrete is usually crumbled and above 1150°C, feldspar melts and the other minerals of the cement paste turn into a glass phase [13]. As a result, severe micro-structural changes are induced and concrete loses its strength and durability.

After the emergence of nanotechnology, nano materials such as nano silica, nano alumina, nano clay and nano titania which are used to improve the properties of cementitious composites are introduced to concrete [21, 22]. In general, nano materials can enhance the mechanical properties and durability of concrete in different fashions. Nano materials act as nuclei so that hydration products may diffuse and envelope then a more compact matrix with smaller crystals of calcium hydroxide is formed [23, 24]. Moreover, nano size particles can act as filler to fill up voids and lead to the densification of the interfacial transition zone [25, 26]. Amorphous nano particles such as nano silica not only physically modify the hydration but also act as pozzolans and react with the available calcium hydroxide (CH) as well as produce the secondary calcium silicate hydrate (C SH) which further densifies the cement matrix [25,27,28]. Also, it was shown that nano silica accelerated the dissolution of C3S and rendered a more rapid formation of CSH due to a highly reactive and large surface area of its particles [29].
So far, only a few studies have tried the incorporation of nano ferric oxide (NF) in cementitious composites. Yazdi et al. [30], showed an increase in both compressive and tensile strengths with the addition of 1 and 3 wt., % NF, whilst 5 wt., % NF decreased the strengths. Nazari et al. [31, 32], studied the workability, setting times and flexural strength of concretes modified with NF. The results showed a reduction in the workability with NF content. While, the flexural strength increased with the addition of NF.

Arioz [33] studied the effect of elevated temperatures on the physico-mechanical properties of concrete mixtures produced by different water/cement ratios and different types of aggregates. The results showed that weight of the concrete specimens reduced significantly with temperature. The relative strength of concrete decreased with temperature, and the effects of high temperature on the relative strength of concrete was more pronounced for concrete mixtures produced by river gravel aggregate. El-Didamony et al., [34] studied the fire resistance of basalt, limestone and micro-silica fume aggregate. It was found that the replacement of basalt by silica fume in the presence of limestone does not reduce the fire resistance of hardened cement pastes up to 600°C. This is attributed to the pozzolanic activity of homra which consumed portlandite forming additional CSH gel [35]. The effect of substitution of micro-silica – silica fume (SF) – and fly ash (FA) on the behavior of composite cement pastes exposed to elevated temperatures was studied [37]. It was concluded that the composite cement pastes made from 80, 10 and 10 wt.% of OPC, SF and FA, respectively have good fire resistance up to 450°C.

Objective of the Research - The present work aims to prepare nano-iron oxide (NF) by thermal decomposition of basic ferric acetate as a precursor at different temperatures. The effect of incorporation of NF to Portland cement pastes on the fire resistance up to 800°C was studied.

II. MATERIALS

The materials used in this investigation are Ordinary Portland cement (OPC) provided from Suez cement Company, Suez. Egypt, and basic ferric acetate [Fe(CH$_3$COO)$_2$].OH as chemical grade. The chemical and mineralogical composition of OPC is given in Table (1).

III. METHODS AND EXPERIMENTS

The NF was prepared by thermal decomposition of basic ferric acetate which heated in a muffle furnace at 275, 600 and 800°C with heating rate 5°C/min for one hour soaking time at each temperature. Mixes were prepared by substitution of OPC with 1, 2 and 3 wt., % NF prepared at 275°C. The dry constituents of each mix were mechanically mixed for one hour in a porcelain ball mill using four balls to attain complete homogeneity then kept in air tight containers.

A. Preparation of cement pastes

The water of consistency, and the initial as well as final setting times were determined according to ASTM methods using Vicat apparatus (ASTM: C191) [38]. The mixing of cement powder was carried out with the required water of consistency. The admixed cement was placed on non-absorbent surface and a crater was formed in the center. The required amount of water was poured into the crater by the aid of a trowel. The mixing operation was then completed by continuous vigorous mixing for three minutes with gauging trowel. At the end of mixing, the pastes were directly poured in 2cm cubic stainless steel moulds into two approximately equal layers. Each layer was compacted and pressed until homogenous specimen with soaking time of one hour, then left in the furnace over-night and kept in desiccator for further investigations.

B. The effect of fire on the cement pastes

To show the effect of fire on the cement pastes, the cubes were cured for 28 days under water then heat treated up to 800°C. The rate of heating was 5°C/min up to the desired temperature as 105, 250, 450, 600 and 800°C with soaking time of one hour, then left in the furnace overnight and kept in desiccator for further investigations.

C. Determination of weight loss

The weight loss (WL) of the heat treated samples was determined in relation with the original weight of samples cured for 28 days.

D. Determination of bulk density and total porosity

The bulk density and total porosity of thermally treated cement pastes were determined after immersing the pastes overnight under kerosene [39].

E. Determination of compressive strength

The compressive strength of cement pastes was determined according ASTM Designation: (C150-2007) [40].
IV. RESULTS AND DISCUSSIONS

A. Thermal Decomposition of Basic Ferric Acetate

A.1 Mineralogical composition of NF

Nano-sized iron oxides were prepared by the thermal decomposition of basic ferric acetate in a muffle furnace at heating rate of 5°C/min for one hour soaking time at 275, 600 and 800°C. The gradient increment of temperature was intended to identify the formed phases and the crystal size. The products of basic ferric acetate fired at different temperatures were examined by XRD analyzer as shown in Fig. (1). Sample fired at 275°C shows the formation of magnetite as the main phase in addition to hematite. The presence of magnetite in the fired product at 275°C is mainly due to the insufficient atmospheric air to oxidize the magnetite Fe₃O₄ to hematite Fe₂O₃. It is clear that as the firing temperature increases, the hematite increases (at 600°C) on the expense of Fe₂O₃. Magnetite is nearly converted to hematite with small amount magnetite (Fe₃O₄) at 800°C. When ferric acetate is fired in an open atmosphere (tubular furnace) the hematite Fe₂O₃ can be completely formed at 275°C [41].

The thermal behavior of basic ferric acetate during heating under nitrogen atmosphere at rate of 2°C/min was carried out by using thermal analyzer. Figs. (2, 3) show the differential thermal analysis (DTA) and thermal gravimetric (TG) curves of ferric acetate heated up to 800°C. Figure (2) illustrates the presence of four endothermic peaks at 75-125, 125-150, 150-300 and 700-800°C. The first endothermic peak is due to the removal of moisture whereas the second and third endothermic peaks are attributed to desorption of acetone and carbon dioxide, respectively as shown in the following equation [42]:

\[
2\text{Fe(CH₃COO)}₂\text{OH} \rightarrow \text{Fe}_₂\text{O}_₃ + 3\text{CH}_₃\text{COCH}_₃ + 3\text{CO}_₂
\]

The fourth endothermic peak may be attributed to the transformation of γ-Fe₂O₃ to α-Fe₂O₃ [43].

Hematite (α-Fe₂O₃) is the only thermally stable polymorph prepared by oxidative thermal treatment of iron salts [44]. Magnetite is one of the most thermally unstable iron oxides. It turns first into maghemite (γ-Fe₂O₃, brown) around 200°C, and then to hematite around 400°C. Therefore, hematite is a common contamination of any iron oxide polymorph prepared under heat [45].

Figure (3) illustrates the (TG) analysis of the basic ferric acetate up to 800°C. It gives TG loss 23.89%, up to 275°C. This mainly due to the loss associated with desorption of acetone and carbon dioxide from ferric acetate with the formation of hematite [41]. At 800°C, the total loss is 32.08%. The observed difference between the TG loss at 275 and 800°C is due to the formation of magnetite Fe₃O₄ which is accompanied by release of oxygen.

A.2. Microstructure and Morphology of NF

The SEM image of the fired basic ferric acetate [Fe(CH₃COO)₂OH] at 275, 800°C are shown in Figs. (4&5). Sample fired at 275°C shows dark aggregate of magnetite with some white crystals of hematite. On the other hand, sample fired at 800°C shows the appearance of hematite as white clusters [44, 45]. Also, it is observed that the Fe₂O₃ crystals have spherical disturbed shapes. The dark aggregate of sample fired at 800°C may be attributed to some cleavage or pores in the sample.

A.3. Crystal Size of NF

The crystal size of NF prepared at 275, 600 and 800°C is examined by the aid of TEM as shown in Fig. (6 a, b, c). From the figure, it can be seen that the crystal size of the samples fired at 275, 600 and 800°C is 14.6, 16.98 and 18.6 nm, respectively.

B. Fire Resistance of Cement Pastes

Fire is one of the hazards that attack building constructions. The damage of buildings is due to their continuous exposure to high temperatures. The chemical processes stimulated by elevated temperature in the particular phases of cement paste have a significant influence on the thermal deformation, cracking, spalling and compressive strength losses [5, 9, 11, 18-20].

Literature reports provide evidence that high temperatures induced loss of water; either free, adsorbed or chemically combined water, affect the microstructure of cement paste, capillary and total porosity increase as well as the nano-porosity associated with CSH gel collapses [46].

The effect of fire on the mechanical properties of cement pastes admixed with nano-iron oxide (NF) fired at 275°C can be studied by the determination of the weight loss, apparent porosity, bulk density, and compressive strength of hardened cement pastes treated up to 800°C.

B.1. Effect of NF content on fire resistance

B.1.1. Weight loss

The effect of fire on the cement pastes containing 0, 1, 2 and 3 wt.%, % NF heat treated up to 800°C is plotted in Figure (7). It is clear that the weight loss increases with the increase of temperature, which is due to the evaporation of free as well as combined water and the decomposition of hydration products.

As the temperature increases, the weight loss of all cement pastes also increases. Portland cement pastes give lower values than those containing 2 and 3 wt.%, % NF up to 450°C. This may be due to the low mixing water which tends to decrease the weight loss. At 600°C the weight loss of OPC is higher than all cement pastes containing 1, 2 and 3 wt.%, % NF. Generally, cement pastes incorporated with 1 wt.%, % NF show the lowest weight losses up to 800°C due to its lower mixing water in relation with 2 and 3 wt.%, %. The weight loss of cement pastes increases with the NF content up to 800°C. The decrease of the weight loss of
admixed cement pastes at higher temperature may be attributed to the effect of NF to enhance the formation of hydration products which precipitated in the open pores with low free water. Therefore, the neat OPC pastes give higher weight losses.

B.1.2. Bulk density

The bulk density of thermally treated OPC pastes admixed with nano-iron oxide is graphically represented up to 800°C in Figure 8. The bulk density of all cement pastes decreases from 105 up to 250°C, due to the removal of free and/or adsorbed water as well as combined water from CSH and CASH. This leads to the formation of micro-cracks from the decomposition of hydration products forming a sort of opening the pore system of cement pastes [48]. From 250°C up to 800°C the bulk density increases for all cement pastes. This is attributed to the volume shrinkage leading to a dense structure, as well as the reaction of aluminosilicate material with the decomposed CaO from Ca(OH)₂ forming ceramic products.

Bulk density of cement pastes admixed with NF is higher than OPC pastes at all firing temperatures. This is due to that NF particles act as a foreign nucleation site, which could accelerate CSH gel formation as a result of increased crystalline Ca(OH)₂ amount, and consequently leads to more compact and dense structure [9], 1 wt., % NF gives higher bulk density than that containing 2 and 3 wt., % NF.

B.1.3. Total porosity

The values of total porosity of cement pastes admixed with 0, 1, 2 and 3 wt., % NF are graphically plotted as a function of heat treatment and NF content in Fig. (9). Total porosity decreases for all cement pastes up to 250°C. This is mainly due to the additional hydration of products unhydrated cement grains as a result of steam effect or the so-called internal autoclaving effect (self-autoclaving) which leads to increase the rate of formation of hydration products that precipitated in the open pores. On the other hand, the increase of NF content in the cement pastes gives higher of total porosity due to the decrease of Portland cement portion which is the main source of hydration and also the increase of water of consistency. As the temperature increases up to 800°C, some of water of CSH and CASH in addition to water from the dehydroxylation of Ca(OH)₂ evaporate. This leads to increase the total porosity of cement pastes.

Generally the total porosity of cement pastes incorporated with NF is lower than that of the neat OPC paste due to the filler effect of the NF. It is clear also that 1wt. % gives the lower porosity than that containing 2 and 3 wt., % NF.

Nazari and Riahi [48] studied the effect of nano-Fe₂O₃ on the water absorption of admixed cement pastes. It was found that Fe₂O₃ nanoparticles up to 2 wt., % produce concrete with improved resistance to water permeability or decrease the porosity.

B.1.4. Compressive strength

The compressive strength of cement pastes admixed with NF heated up to 800°C is plotted in Fig. (10). The figure shows that the compressive strength of cement pastes increases up to 250°C as a result of enhancing the hydration of cement pastes [49] as well as due to self-autoclaving which increases the hydration products that precipitated in the open pores. Therefore, the compressive strength of cement pastes increases for all cement pastes. At 450°C the compressive strength of admixed cement pastes is higher than that of OPC at 105°C and nearly the same values at 250°C. As the temperature increases up to 800 °C, the compressive strength of cement pastes decreases due to the decomposition of the cementitious materials CSH, as well as Ca(OH)₂ forming micro-cracks [47]. All cement pastes containing NF give higher strength values at all heating temperatures than those of OPC pastes. Also, 1 wt., % NF shows the higher values than those admixed with 2 and 3 wt., % NF due to the decrease of water of consistency. The addition of NF as a foreign nucleation site could accelerate CSH gel formation, as a result of increased crystalline Ca(OH)₂ content and hence the strength. The sharp drop of compressive strength of all cement pastes is mainly attributed to the decomposition of Ca(OH)₂ leading to the formation of CaO with microcracks, which has no binding properties and fast hydration. The mechanism to refine the pore structure by nano-particles explained as follows. Assuming that the nano-particles are uniformly dispersed in the cement and each particle is contained in a cubic model, the distance between the nano-particles can be determined. After the hydration starts, hydrates spread and envelop the nano-particles. If the content of nano-particle sand and the distance between them are appropriate, the crystallization will be controlled in a suitable condition limiting the growth of Ca(OH)₂ crystal by nano-particles. In addition, nano-particles in cement paste can also promote the hydration of cement due to their high activity. This gives more homogeneous and compact cement matrix [50].

An earlier study concerned with the preparation of Cu-Zn nano ferrite particles and admixed with cement to show the effect of heat treatment at 200 and 400°C. Cement pastes were tested at normal temperature, as well as 200 and 400°C after 2 hours exposure at elevated temperature. The results showed that the optimum content of nano-ferrite was 1 wt., % to produce maximum increase of compressive strength [51]. Ginebara et al., [52] reported that particle size can greatly affect the rate of cement hydration which indicates that a reduction in particle size can lead to a more rapid setting and hardening of cement due to stronger electrostatic attractive forces and a greater specific surface area.

Scanning electron microscopic studies about the microstructure of cement mortar admixed with NF showed that the nanoparticles fill the pores and reduce the large crystals of Ca(OH)₂ as well as the hydration products are dense and compact. The increase of Fe₂O₃ nanoparticles up to 5wt% reduces the mechanical properties.
C. Effect of firing temperature of NF on the fire resistance

The effect of firing temperatures up to 800°C on Portland cement pastes admixed with 1 wt. % of NF prepared at 275, 600 and 800°C was studied. In agreement with the above results, the addition of 1 wt. % of NF gave the optimum results. Therefore, 1 wt. % NF was chosen for this factor.

C.1. Weight loss

Weight losses of Portland cement pastes admixed with 1 wt. % of NF prepared at 275, 600 and 800°C as a function of heat treatment temperatures are illustrated in Fig.(11). As the firing temperature of NF increases the weight loss also increases. This may be due to the increase of crystal size and the mixing water as well as decrease of surface area of NF. Sobolev et al., [53] concluded that the surface area of nano-particles treated at 400°C increased. On the other hand, the surface area of nano-particles decreased in upon heating at 800°C.

C.2. Total Porosity

The total porosity of OPC pastes admixed with 1% NF prepared at 275, 600 and 800°C is illustrated in Fig. (12). The total porosity of cement pastes increases with firing temperature of NF due to the increase of crystal size of iron oxide (nano-particles). Generally, the total porosity of all cement pastes decreased with firing temperature up to 450°C, then increased due to de-hydroxylation of Ca(OH)₂ leading to the formation of some micro-cracks ( pores).

The total porosity of cement pastes increases with firing temperature of NF. It can be concluded that the NF prepared at 275°C is the suitable firing temperature. This may be due to the decrease of crystal size of NF as well as high surface area. The rate of hydration increases with the increase of surface area of NF.

C.3. Bulk density

The bulk density of OPC pastes as well as admixed with 1% NF prepared at 275, 600 and 800°C with heat treatment temperature is graphically plotted in Fig. (13). The bulk density of cement pastes decreases up to 250°C and then increases up to 800°C. It is clear that the bulk density increases with the decrease of firing temperature of NF. Also, OPC paste gives lower bulk density than that of cement pastes admixed with NF. This may be due to the decrease of crystal size of NF which accelerates the hydration with firing temperature.

Fe₂O₃ nanoparticles can improve the filler effect and also the high pozzolanic action of fine particles increases substantially the quantity of CFH gel. If this phenomenon joints with low W/C ratio, it can improve the microstructure in the interfacial transition zones and thus the CFH content gel decreases the water permeability or porosity which increases the density [47].

C.4. Compressive strength

The compressive strength of Portland cement pastes admixed with 1% NF as a function of firing temperatures is plotted in Fig. (14). The compressive strength decreases with the firing temperature of NF. This may be due to the increase of crystal size of NF (which is 14.6, 16.98 and 18.6 nm at 275, 600 and 800°C, respectively). Also, neat OPC pastes give the lower strength at all firing temperatures. As the firing temperature increases up to 450°C the compressive strength enhances for all cement pastes due to the acceleration of hydration of cement pastes due to self-autoclaving which tends to increase the rate of hydration of cement pastes. Therefore, more CSH gel which is the source of strength increases. At 800°C, the strength drops for all pastes due to the dehydroxylation of Ca(OH)₂ giving CaO with micro cracks as well as pores, in addition to the recrystallization of the amorphous CSH into ß-C₂S and free lime which is easily hydrated to Ca(OH)₂ with low strength.

V. SUMMARY AND CONCLUSIONS

The following conclusions can be derived from the above findings:

1- Thermally heat treatment of basic ferric acetate fired at 275, 600 and 800°C gives magnetite as main phase with hematite at 275°C. At 600°C, the hematite increases on the expense of magnetite whereas that fired at 800°C shows nearly hematite.

2- The crystal size of the fired nano-feric oxide increases with firing temperature, i.e. it is 14.60, 16.98 and 18.6 nm at 275, 600 and 800°C, respectively.

3- The firing temperature of basic ferric acetate has an effect on the fire resistance of OPC pastes admixed with NF. Nano-particles of NF prepared at 275°C gives higher heat resistance than that fired at 600 and 800°C.

4- OPC admixed with 1 wt. % NF prepared at 275°C gives the optimum content to achieve fire resistance than 2 and 3 wt. % NF. It shows higher compressive strength as well as bulk density and lower total porosity.

5- The bulk density and total porosity of cement pastes decrease with heat treatment up to nearly 250°C then increase, whereas the compressive strength increases up to 450°C then decreases with heat treatment up to 600°C.

VI. REFERENCES


M.S. Akman, "Building damages and repair principles", Turkish Chamber of Civil Engineers 2000: Istanbul, Turkey.


**Table (1): The chemical composition of OPC, (mass %)**

<table>
<thead>
<tr>
<th>Oxide</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>Fe$_2$O$_3$</th>
<th>CaO</th>
<th>MgO</th>
<th>SO$_2$</th>
<th>K$_2$O</th>
<th>Na$_2$O</th>
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**Table (2): The phase composition and surface area of OPC**

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<th>C$_3$S</th>
<th>C$_2$S</th>
<th>C$_3$A</th>
<th>C$_4$AF</th>
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<td>10.53</td>
<td>6.88</td>
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![Fig. 1: X-ray diffraction (XRD) of iron oxide samples prepared from basic ferric acetate by thermal decomposition at different temperatures.](image-url)
Fig. 2: DTA-thermogram of ferric acetate

Fig. 3: TG thermogram of ferric acetate
Fig. 4: SEM of Nano-iron oxide prepared at 275°C

Fig. 5: SEM of Nano-iron oxide prepared at 800°C
Fig. 6 (a): TEM of iron oxide sample prepared at 275°C

Fig. 6 (b): TEM of iron oxide sample prepared at 600°C
Fig. 6 (c): TEM of iron oxide sample prepared at 800°C

Fig. 7: Weight loss of OPC pastes admixed with 1, 2 and 3 wt., % NF prepared at 275°C with heat treatment temperature
Fig. 8: Bulk density of OPC admixed with 1, 2 and 3 wt.% NF prepared at 275°C as a function of heat treated.

Fig. 9: Total porosity of OPC pastes admixed with NF prepared at 275°C as a function of heat treatment temperature.
Fig. 10: Compressive strength of OPC admixed with NF (prepared at 275°C) as a function of NF content and heat treatment

Fig. 11: Weight loss% of OPC admixed with 1% NF prepared at different temperatures as a function of heat treatment temperatures
Fig. 12: Total porosity of OPC admixed with 1% NF prepared at different temperatures as a function of heat treatment up to 600°C.

Fig. 13: Bulk density of OPC admixed with 1% NF prepared at different temperatures as a function of heat treatment up to 800°C.
Fig. 14: Compressive strength of OPC admixed with 1% NF prepared at different temperatures as a function of heat treatment.