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Microstructure and strength of iron-filing Portland cement paste and mortar

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Abstract. This study investigated the role played by iron-filing in the strength and microstructural characteristic of high ordinary Portland cement (OPC) based paste and mortar at substitution levels that ranged from 0 to 15 wt.%. It was found that, iron-filing mainly comprised Fe_2O_3, reduced the workability, influence carbonation, early strength development and impacted the nature of the product formed especially if prepared within the w/c ratio of 0.25 and 0.35–0.4 for paste and mortar, respectively. Moreover, iron-filing also induced and enhanced belite hydration, and influenced the crack-bridging effect within the microstructural matrix, while substitution level less than 5 % affected not the early strength. Besides, iron-filing could also influence the formation of calcium ferrosilicate hydrate as revealed in the microstructural analysis or elemental analysis from energy-dispersive X-ray spectrometer. The 28-day strength of 114 MPa and 61 MPa were achievable in paste and mortar, respectively for iron-filing replacement level within 5–10 %, even substitution up to 15 % could perform better than the sample prepared with OPC only. The study promotes waste valorization and the use of iron-filing in mass-concrete production.

1. Introduction

Iron filings (IF) are mostly byproduct obtained from metal cutting, grinding, filing, or milling of finished iron products especially in workshops and foundry. Their history largely tracks the development of iron, and it is another waste generated by steel production other than blast furnace slag. They are fine powder of small pieces of iron, and often used in science demonstrations to show the direction of a magnetic field [1]. Consumption of IF through contamination by grinding or milling could be dangerous to human health due to the tendency to cause chronic diseases like hemochromatosis, and siderosis [2]. There has been an attempt to re-design food grinding machines to reduce contamination due to IF by [2] in order to emphasize its danger on public health. However, among other benefits of iron-filing is their use in the removal of chloride contamination of groundwater [3] even though this fact still remains controversial among other pro-environmental researchers.

There have been several attempts towards utilizing IF and other ore related materials replacement for fine aggregates (sand) or paste in concrete production such as silicomanganese fume [4], volcanic pumice[5], coal gangue [6] and copper mine tailing [7]. For instance, Adeyanju and Manohar [8] had reported that using enough quantity of IF and steel fibers could enhance thermal and mechanical properties of energy storage plant due to increase in its capacity in comparison with the plain concrete. Alserai et al. [9] also asserted that an increase in IF composition up to 1 % of the mixture could increase the compressive, tensile and flexural strength of geopolymer concrete when used with recycled aggregates. Alzaed [1] also reported that the contribution of iron-filings to concrete strengths could be up to 10 % of the mixture beyond which the insignificant contribution could be observed. Their findings were also correlated with the results obtained by other researchers [10].

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Moreover, Kumar et al. [11] replaced sand with iron-ore tailing and there was no significant change in the strength of the resultant beam due to sand replacement. Similarly, Familusi et al. [12] reported that quarry-dust can be used in binary with iron-filing to produce concrete of comparable strength with conventional concrete strength such that the waste combinations were 37.5 and 50 % of the mixture. The increase in strength has been reported to be from pore blockage such that 2 % of iron powder inclusion could reduce the total porosity by 50 % [13]. Olutoge et al. [14] also reported that the optimum sand replacement for IF was 20 % with 13.5 % gain in the recordable compressive strength.

Further, Ghannam et al. [15] reported the optimum performance of iron-filing in terms of compressive and flexural strengths for sand replacement to be 20 % whereas Noori and Ibrahim [16] asserted that 12 % sand replacement by IF gave the optimum in terms of mechanical strengths characteristics. In the contrary, Vasudevan [17] reported decreasing slump value with the addition of 5 % iron-filing in replacement for sand while the maximum compressive strength was only 3.2 % greater than the control. The difference in the percentage reported could be due to difference in the water/c ratio, sizes of the IF and contaminations such as carbon content or other elements used in the alloy. However, just a few authors presented the oxide composition of the IF used in their studies.

Meanwhile, none of these studies captures the microstructural interaction of IF within the binder matrix, likewise, its performance in paste strength, and bond characteristics within the mixture. These points are very important and serve as the aims of this present study with a view to establishing the actual roles being played by IF in OPC paste and mortar production through sand partial replacements, respectively. It is expected that this study will also provide more information towards understanding the IF interaction within the microstructure and to provide an empirical fact towards the promotion of green environments, and its excessive utilization in mass concrete structures such as walk ways and blinding to foundation of reinforced concrete structures among other applications.

2. Materials and Methods

2.1. Materials

2.1.1. Ordinary Portland cement.

The cement used in the study satisfies the requirement of ASTMC 150 [18] and the oxide compositions determined by x-ray florescence technique is as shown in Table 1 with particle size distribution as shown in Figure 1.

Table 1. Oxides composition of Ordinary Portland and iron-filing Oxides.

Oxides	OPC, %	Iron filing (IF), %
SiO_2	19.01	1.38
Al_2O_3	4.68	0.61
Fe_2O_3	3.20	96.5
CaO	66.89	0.02
MgO	0.81	–
Na_2O	0.09	–
TiO_2	0.22	0.03
K_2O	1.17	0.13
P_2O_5	0.08	0.05
SO_3	3.66	0.23
MnO	0.19	0.5
Cl	–	0.05
Cr_2O_3	–	0.11
ZnO	–	0.07
SnO	–	0.08
SrO_2	–	0.02
CuO	–	0.22
LOI	2.48	–
$SiO_2 + Al_2O_3 + Fe_2O_3$	26.89	98.49

2.1.2. Iron-filing powder

It is collected at mechanical lathe machine workshop at Senaiya Hafr Al Batin in the Eastern Province of Saudi Arabia with the fineness modulus of 2.89. The particle size distribution of the material is as shown in Figure 2 while the oxide composition (Table 1) reflects the dominance of iron oxide.

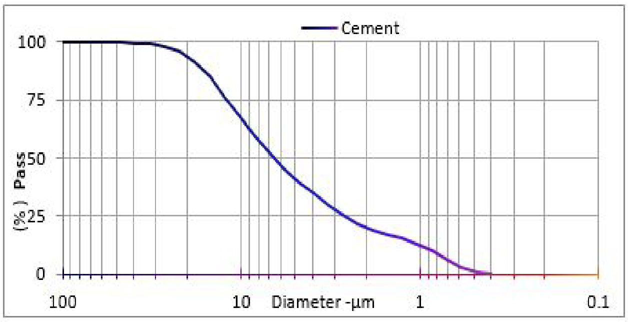

Figure 1. Particle size distribution of the ordinary Portland cement.

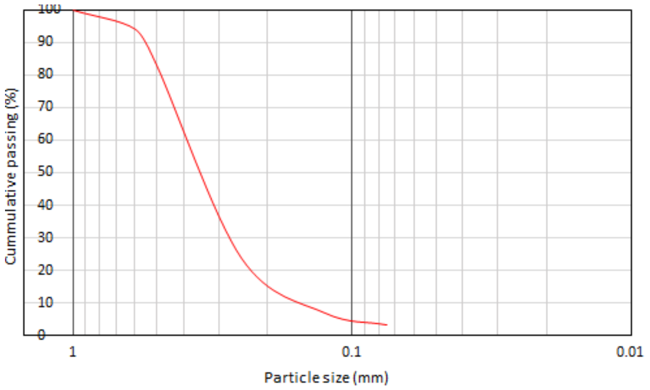

Figure 2. Particle size distribution curve of iron-filing powder.

2.1.3. Fine aggregate

It comprised dune sand with fineness modulus of 1.85, specific gravity in saturated dry condition was 2.62 while its absorption capacity was 0.65 %. The physical properties of OPC, iron-filing (IF) powder and fine aggregates are as shown in Table 2.

Table 2. Physical properties of fine aggregates and iron filing.

Physical Properties	Sand	Iron Filing	cement
Bulk Density	1699	1932	3100
Specific gravity	2.67	6.85	3.41
Fineness modulus	3.3	2.89	–
Average diameter, D_{50} (μm)	–	250	6.69

2.2. Research methodology

The research methodology involved development of experimental design for paste and mortar at w/c ratios of 0.25 and 0.35, respectively. The proportion of IF varied from 0–15 % in both cases while the compressive strengths were determined at 7, 14 and 28 days. The microstructural analysis of the paste was determined to understand the micrograph of the sample matrix while Fourier transform infra-red spectroscopy (FTIR) was used to determine the impact of iron-filing (IF) on the bond characteristics of the binders.

2.2.1. Microstructural and Fourier infrared analysis

The JEOL scanning electron microscopy coupled with energy dispersive spectroscopy (SEM + EDS) model 5800 LV, was used to test the morphology of the 28 days sample paste obtained from compressive strength sample by first coating it in gold film [19, 20]. The pulverized paste was used for the Fourier transform infra-red (FTIR) spectroscopy bond characterization within the sample matrix by using Perking Elmer 880 spectrometer that is based on the attenuated total reflection (ATR) technique.

2.2.2. Mix proportion

Mix design was conducted such that the percentage composition of the IF was used in partial replacement for ordinary Portland cement (OPC) in paste (w/b = 0.25) and sand in mortar (w/b = 0.35) in varied quantities as 0, 5, 10 and 15 % as shown in Table 3 and 4. The superplasticizers used (Glenum®) was 0.63 wt.% in paste and 1 wt.% in mortar, respectively to achieve the required consistency. The Sand/OPC ratio was maintained at approximately 2.5.

Table 3. Paste constituent materials in kg/m³.

Sample ID	OPC (kg/m³)	Iron filing (kg/m³)	Water (kg/m³)	SP (kg/m³)	Unit weight (kg/m³)
$M_{0.35}F_0$	1872	0	477	12	2361
$M_{0.35}F_5$	1819	95	477	12	2403
$M_{0.35}F_{10}$	1730	193	477	12	2412
$M_{0.35}F_{15}$	1630	289	477	12	2409

Table 4. Mortar Material composition.

Sample ID	OPC (kg/m³)	Sand (kg/m³)	Water (kg/m³)	SP (kg/m³)	Iron filing (kg/m³)	Unit weight (kg/m³)
$M_{0.35}F_0$	625	1569	221	6.3	0	2421.0
$\mathbf{M_{0.35}F_5}$	**625**	**1519**	**221**	**6.3**	**80**	**2450.8**
$M_{0.35}F_{10}$	625	1449	221	6.3	162	2463.2
$M_{0.35}F_{15}$	625	1410	221	6.3	245	2507.2

2.2.3. Mixing of the specimen

OPC was first placed in Hobart planetary mixer after which the iron-filing (IF) powder was added, and then mixed for 3 mins. Water (70 %) together with superplasticizer was added and then further mixed for 2 mins. Sand was then added and the whole mixture was mixed continuously for additional 3 mins before adding the remaining water (30 %) to ensure homogeneous mixture. The paste and mortar were then cast into 50×50×50 mm mould in 3 layers on the table vibrator to vibrate for 15 secs. The samples were demoulded after 12 hrs and then lowered into curing tank at the room temperature of 25 $\pm$ 2 °C.

2.2.4. Workability

The workability of the specimen was determined by flow table in accordance with ASTM C 1437 [21]. The slump value was expressed as a percentage:

$$\text{Slump}(\%) = \frac{\text{measured flow} - 100}{100} \times 100.$$

2.2.5. Compressive strength

Compressive strength of the specimen prepared in triplicate were tested using universal testing machine at the loading rate of 2.4 kN/s. The compressive strength test was conducted upon removing the samples from curing tank and drained for 6 hrs. The samples were tested after 7, 14 and 28 days.

3. Results and Discussions

3.1. The physical characteristics of iron filing

Iron-filing (IF) has a bulk density lower than ordinary Portland cement (OPC) and greater than sand particles (Table 2). Its specific gravity is almost twice that of OPC. Therefore, its inclusion increases the density of the resultant paste or mortar as applicable. Therefore, concrete produced with iron-filing based paste or mortar can be used for providing adequate thrust for underground buried pipes. The average size D_{50} is around 250 μm which could be categorized into fine aggregates by virtue of its being less than 4.75 mm (No. 4) in accordance with ASTM C33 [22]. Figure 3 shows the increment of resultant mortar density as the quantity of iron-filing increases. The initial density of OPC mortar for w/c ratio of 0.4 increases by 3.5 %, 7.4 % and 10.34 % for increment level of IF by 0, 5, 10 and 15 %, respectively. These values slightly reduced when the w/c ratio reduced to 0.35. The decrease observed could be due to self-desiccation phenomenon that led to autogenous shrinkage in the sample of lower w/c ratio [23–25].

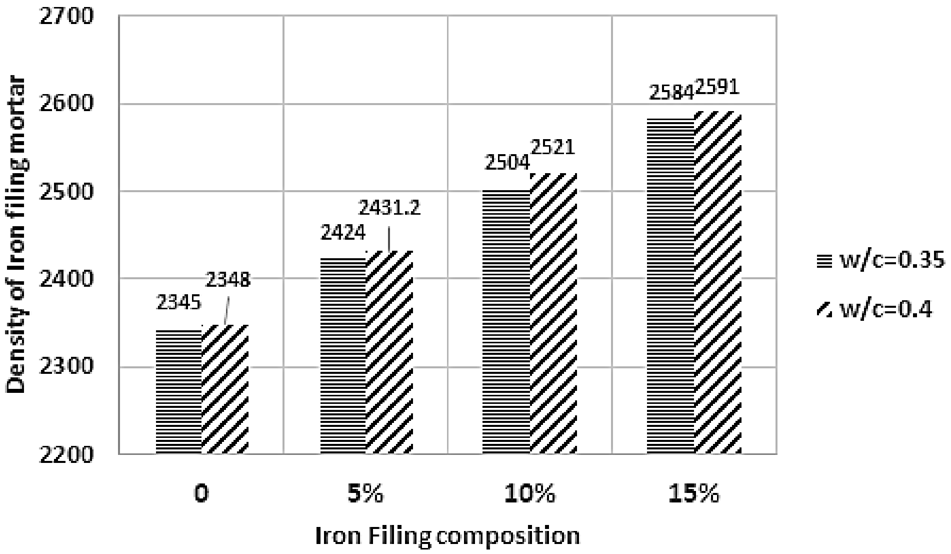

Figure 3. Density of mortars at different iron-filing substitution levels.

3.2. Workability of the samples

From Figure 4, the consistency of the iron-filing (IF) mortar decreases with lower water-cement ratio. There is improvement in the workability as the w/c ratio increases from 0.35 to 0.4 due to decrease in the slope of the curve as shown in Figure 4. This suggests that inclusion of IF could increase the consistency of the mixture especially when the mixture is workable. The workability of the control mortar (OPC only) decreased by 63.6 % upon reducing the w/c ratio from 0.4 to 0.35. This further decreased to 68.6 %, 94.4 % and 97.6 % upon replacing sand with IF in the percentages of 5 %, 10 %, and 15 %. Therefore, with adequate consistency (fluidity) of the mixture, inclusion of IF could be used successfully to improve the workability of the mortar.

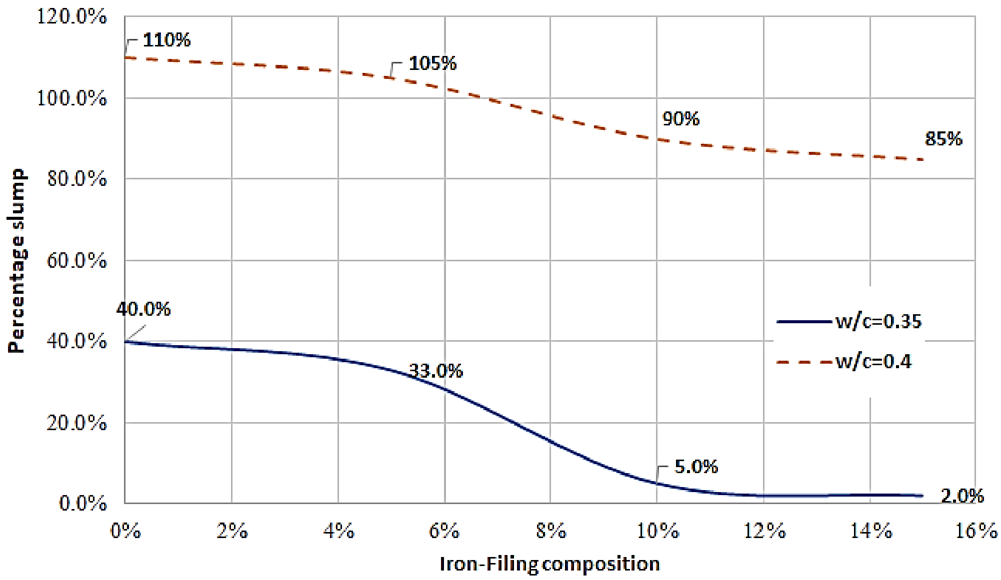

Figure 4. Slump values at different iron-filing substitution level.

3.3. Impacts of iron-filing on the compressive strength of paste and mortar

From Figure 5, addition of 5 % of iron-filing has the highest 7-day compressive strength in comparison with other mixtures. The compressive strengths compared to the control (0 %) at 7, 14 and 28 days for 5 % replacement were 1.7 %, 2.1 % and 2.94 %, respectively. As replacement increases to 10 %, the early (7-day) strength decreases by 14 % due to an increase in the interfacial transition zones (ITZ) between the IF particles and the paste matrix. The rate of strength development at 14 days noticed to be 37.5 % at 10 % replacement became 4.2 and 6.1 % in 5 % and 15 % substitutions, respectively. As the hydration process continues, belite (C_2S) hydration was significantly aided by the presence of IF thereby leading to denser C-S-H in comparison with IF-free sample. The possibility of the reaction shown in Equation (1) indicates that IF at optimum quantity could influence hydration reaction to form calcium ferrosilicate hydrate (CAFSH) or a complex mineral of modified CSH configuration. However, this requires further investigation to determine the extent of possibility and stoichiometry of this reaction.

$$C_2S + H + F \rightarrow C(F)SH + CH. \tag{1}$$

The maximum 14- and 28-day strengths at 10 % level substitution were 12.2 and 13.8 %, respectively of the control while that of 15 % cement replacement performed less as the percentage reduced to 7.3 %. Contribution of iron-filing to strength development could be due to micro-reinforcement, microstructural stability, pore-filling or crack-bridging effects within the microstructure as shown in SEM (Figure 6). The micrograph shows a localized crack-bridging effect in IF paste in comparison with the interconnected cracks of OPC (control) paste.

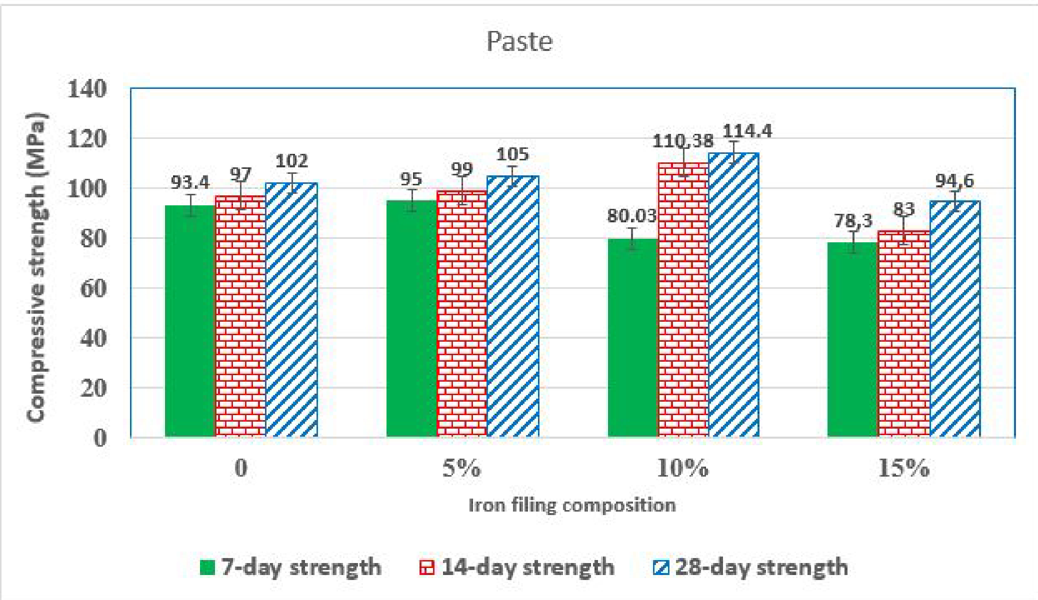

Figure 5. Compressive strength of ironfiling-OPC pastes.

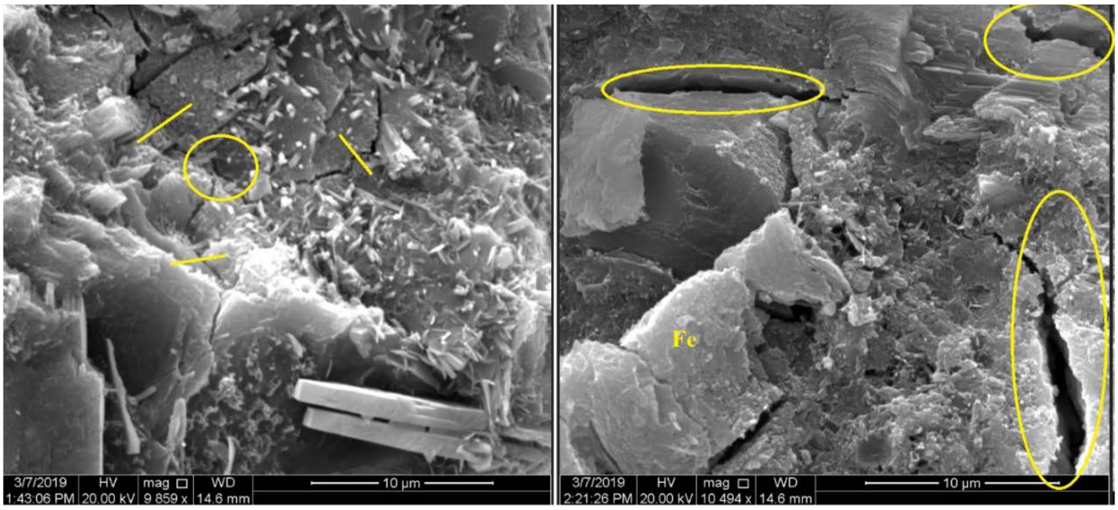

Figure 6. Micrographs of OPC paste (left) and OPC-Iron filing paste (right).

Be that as it may, the introduction of iron-filing (IF) into mortar increases the heterogeneity of the mixture (mortar) compare to the paste sample. There was a formation of two ITZs which exist in-between the paste/iron-filing and sand/paste. Besides, the frictional interaction between IF and sand particle could also improve the compressive strength compared to IF-free sample. More ITZ paste/iron filing in 10 % sand replacement could be responsible for the slowing down the hydration of tricalcium silicate (alite-C_3A) and tricalcium silicate (C_3S). For instance, the lower 14-day strength and the subsequent higher strength gain between 14–28 day in 10 % sand replacement compared to that of 5 % and 15 % substitution as shown in Figure 7. This could indicate that IF at its optimum percentage hinders the rate of strength development at early hydration process. The resultant strength improved due to frictional resistant between sand particle, paste, and IF. Therefore, IF/Paste ratios play a significant role in determining the maximum achievable strength. The maximum strength of 61 MPa can be achieved with OPC replacement of 5–10 % as also observed in the paste samples.

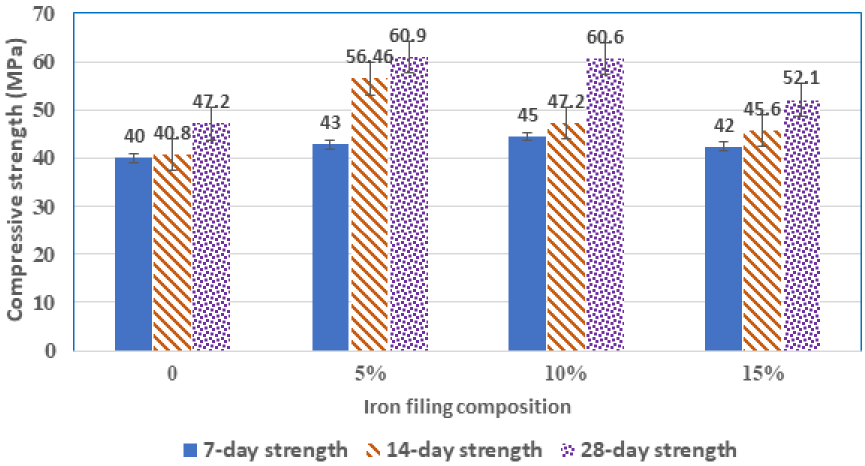

Figure 7. Compressive strength of OPC-iron filling mortar.

In addition, 15 % sand substitution level also performed better than OPC control mortar. There was 28.4 % higher strength gain between 14 and 28 days in mortar sample with 10 % IF ($M_{0.35}F_{10}$) when compared to 7.9 % in the 5 % replacement ($M_{0.35}F_5$).

3.4. Effect of iron-filing on the carbonation of the product

From Figure 8, inclusion of iron-filing has no significant effects on the asymmetric stretching of Si-O-T as the frequencies of vibrations remain 958 cm^{-1}. However, the asymmetric stretching of C-O (CO_3^{2-}) shown by a deeper trough at the wavenumber 1418 cm^{-1} is more apparent in IF sample compared to the OPC only sample [20, 26, 27]. Therefore, there is a possibility for the formation of $Fe(CO_3)_2$ that could oxidize to form $Fe(CO_3)_3$ due to the following two-stage reactions of the interaction of Fe with portlandite that formed during the primary hydration reaction.

$$C_3S + H_2O \rightarrow CSH + Ca(OH)_2; \qquad (2)$$

$$Ca(OH)_2 + Fe + H_2O \rightarrow Fe(OH)_2 + CaO + H_2; \qquad (3)$$

$$Fe(OH)_2 + CO_2 \rightarrow Fe(CO_3)_2. \qquad (4)$$

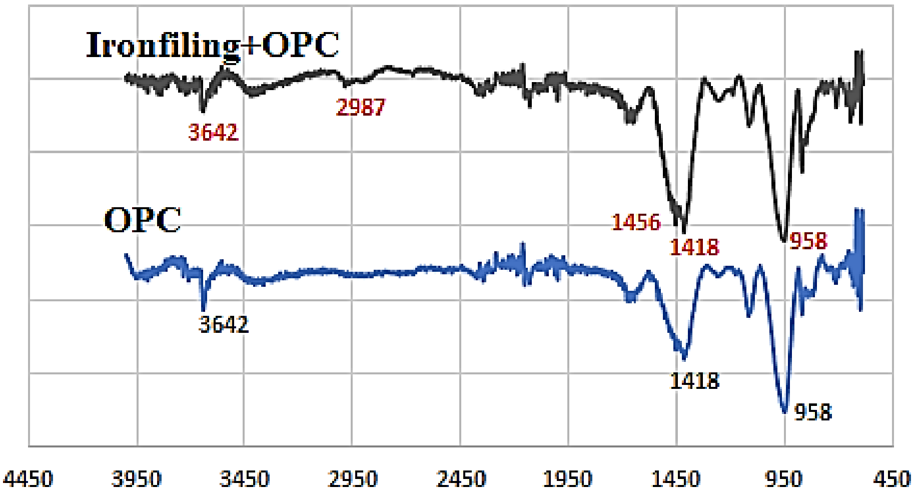

Figure 8. FTIR of the iron filing-OPC and OPC pastes.

Similarly, the vibration of water molecule is observed at 2987 cm^{-1} which indicates the asymmetric stretching or hydrogen bonding within H-O-H molecules [20, 28, 29]. This is possible because of the proliferation of hydroxyl ion in the proximity to one another as shown in the Equations 2 and 3 thereby leading to hydrogen bonding.

Figure 9 shows the presence of CSH (regions 3 and 4) and Fe-based compound (C-F-S-H) or $FeCO_3$ as shown in the Equations 1 and 4. The IF-free sample predominantly contains Ca, Si and O that depict the dominance of CSH in the two distinctive regions (3 and 4). The sample containing IF is heterogeneous in nature when compared C-(A)-S-H region 1 to C-A/F-S-H region 2 within the micrograph as shown in the EDS result (Figure 9).

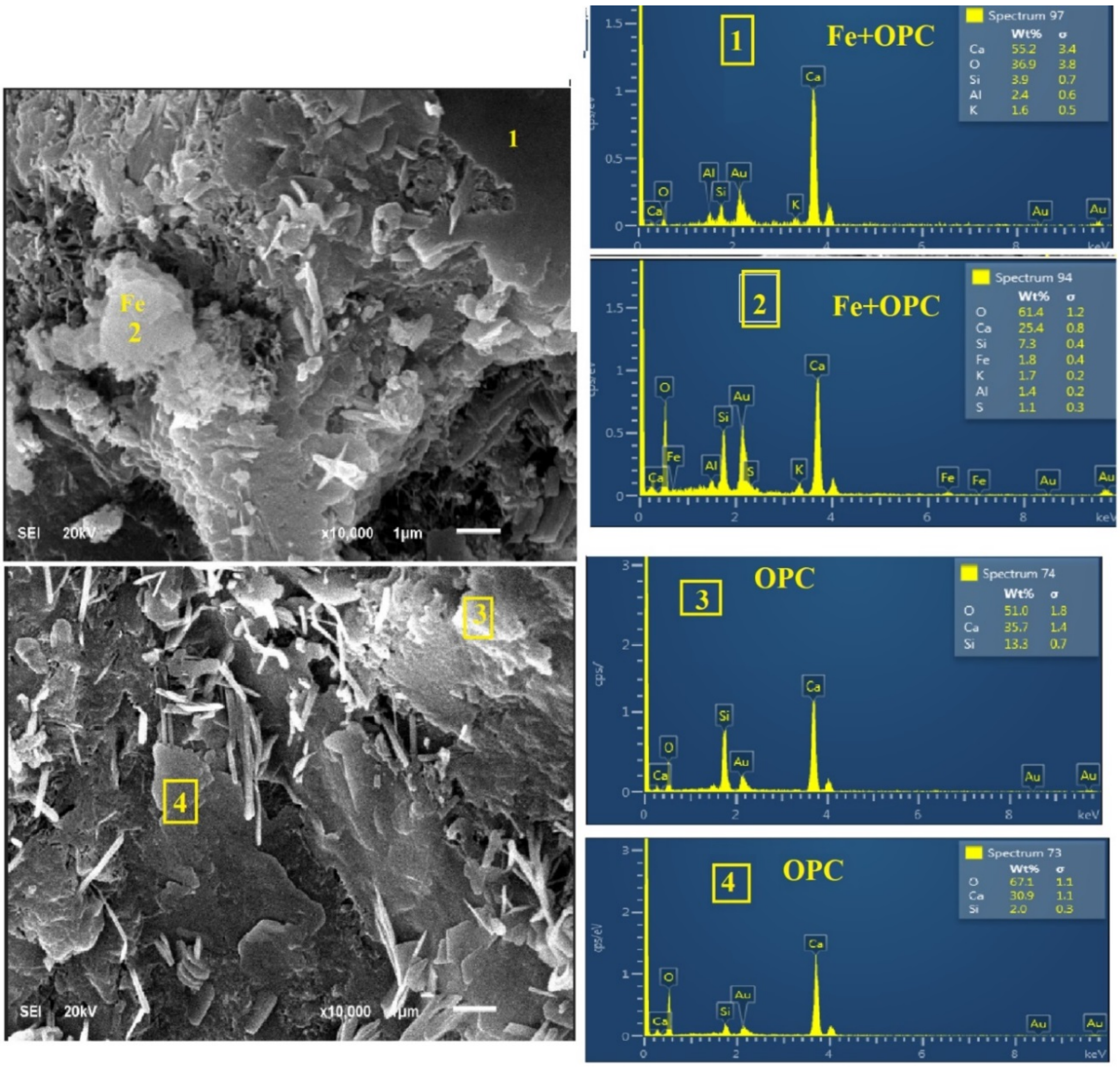

Figure 9. Micrographs of Iron-filing+OPC (top) and OPC paste only (bottom).

4. Conclusions

Iron-filing (IF) powder was used in partial substitution for cement and sand in ordinary Portland cement based paste and mortar with a view to understanding its roles in terms of workability, strength, bonds and microstructural characteristics of the samples. The following are the conclusions:

- At lower water cement ratio up to 0.35, iron-filing decreases the workability of the mixture of mortar. It could however enhance the consistency of the mixture at higher water-cement ratio due to IF low water absorptive tendency.

- Iron-filing substitution level in moderate quantity in mortar could increase the compressive strength of paste and mortar specimens considerably. However, excessive substitution could have a debilitating effect on workability and strength performance.

- Substituting sand with iron-filing at very low quantity has no significant effect on the early hydration of OPC.

- Iron-filing influenced the formation calcium ferrosilicate hydrate intertwined with the calcium silicate hydrate within the microstructures.

- From the C-O bond vibration as noted in FTIR, iron-filing could influence the carbonation of the paste and mortar due to the propensity of the formation of iron carbonate.

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