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Mortar Composites Based on Industrial Wastes

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Abstract The effect of granulated slag (GBFS), fly ash (FA) and silica fume (SF) substitution by 35 mass% on the physical and mechanical properties of Portland cement mortar was investigated. The results showed that the combined water, bulk density, flexural strength and compressive strength increased gradually with FA content and a further increase was noted when the mortar composite contained equal ratios from the three waste materials. So, it selected to be the optimum mortar composite. The FTIR analysis showed that the free lime content was consumed with FA and SF contents. The SEM images illustrated the formation of abundant fibrous CSH embedded in the matrix due to the hydration and pozzolanic reactions between different components.

Keywords: *mortar, composite, slag, fly ash, silica fume, density, strength, IR, SEM*

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1. Introduction

1.1. Scope of the Study

Since a long time up till now, the Ordinary Portland cement is the only construction material in cement and concrete industry. Its effectiveness on mechanical properties and durability has made it indispensable for the construction process. The high temperature (1400-1450°C) used for the production of Portland cement made it very costly, in addition to the emission of high amounts of CO_2↑ into the atmosphere, which is one of the mean greenhouse gases leading to air pollution so that it is stated that about 7 % of CO_2↑ of the total emission worldwide come from the production of Portland cement. Also, the production of each ton of Portland cement emits about one ton of CO_2↑. The use of industrial and biogenic wastes in concrete as supplementary cementing materials is the present vital issue to obtain a sustainable environmental solution, save energy and natural resources. Some of the commonly used supplementary pozzolanic and cementing materials are rice husk ash, silica fume, granulated blast furnace slag, fly ash and ash from timber --- etc [1-5]. These wastes can be found as natural materials, by-products or industrial wastes. Unfortunately, most of those wastes are dumped into environment without any commercial return. In consequence, environmental pollutions are increased day by day. So, due to growing environmental concern and the need to conserve energy and resources, utilization of industrial and biogenic wastes as supplementary cementing materials has become an integral part of concrete construction. During recent decades, a lot of researches have been conducted to use different types of agro-waste ashes such as rice husk ash [1,4,6], bagasse ash [7], palm oil fuel ash [8,9], industrial by products as granulated blast furnace slag, silica fume and fly ashes as cement replacement materials [10-16]. Their utilization not only improves properties and durability of concrete, but also makes it cost effective and environment-friendly [17-26].

On the other hand, the industrial wastes or byproducts are continuously increased day by day. The reutilization of these waste materials has gained great importance worldwide. The blast furnace slag cements or concretes produce low heat of hydration, its resistance to sea water and sulfate media is very high. Its production utilizes industrial wastes and requires less energy than that of the OPC. Moreover, it is associated with low CO_2↑ emission [27,28].

The silica fume (SF) is a pozzolanic waste material and can consume the alkali hydroxides in the pore solution of the cement to form non-expansive CSH, which has a low CaO/SiO_2 ratio (C/S). This will deplete the CH in the cement pastes and the low C/S ratio enables entrapment of alkalies. Both of which reduce the amount of OH- ions available to participate in the alkali silica reaction [29-33]. One of the greatest advantages of using SF in concrete results from its small size, where the addition of SF can widen the size distribution of the cement particles of concrete. This was allowing more efficient particle packing, densifying the interfacial transition zone and converting CH into CSH. These factors evidently increase the strength of concrete. However, the small particle size of SF makes it difficult to transport and distribute. So, the SF must inter-ground with cement before its use or a suitable superplasticizer must be added to avoid the accumulation of large clumps. This is due to the fact that the large agglomerates behave as reactive aggregates more than pozzolanic material [34,35]. However, the higher amounts of SF decreases the plastic viscosity of the fresh cement pastes or concretes, and as a sequence, it worsens its workability due to its high surface area [30,31,32].The fly ash is a secondary raw material that could be exploited for building purposes among others since a long time as in

cement, mortars and concrete. Fly ash is a waste material originating in heat and power plants with dust coal combustion. The mineralogical components of fly ash are exposed to high temperatures (1200-1700°C) for time periods during combustion and pass through oxidizing and reducing units. About 10-15 mass% of the solid residues (10-100 μm) in the form of ash could be settled at the bottom of the combustion chamber, which could be removed mechanically and collected in their electrostatic separators. The resulting fly ash contains more than 50 % glassy phase. During combustion, CO_2↑ and SO_2↑ gases are produced. The toxic SO_2↑ could be trapped in a de-sulphorizer for disposal [22,26,33]. The production of fly ash in Europe is 37.6 million tons yearly, of which about 18.2 % could be applied in cement and concrete industry, where the European Standards EN 197-1, 2000 allows maximum percentages of fly ash with 35 % in CEM II, 55 % in CEM IV and 50 % in CEM V [2,3]. However, the effective amounts of FA on the properties of fresh and hardened cement pastes or concretes is limited by 10-20 mass% [33,34,35].

1.2. Objectives of the Study

The artificial waste materials are not cementitious in nature, but they could be activated by the lime released from the hydration of C_3S and β-C_2S phases of the cement and react with it to form compounds possessing cementing properties [33,35,36]. The use of both mineral admixture as GBFS, SF, FA and/or chemical admixtures as accelerators, retarders, air-entrainments, water-reducers and superplasticizers have great attention because they play an important role to offset some of the undesirable properties of concrete. This will improve some of the physical and chemical properties of concretes. On this basis, the main objective of the present study is to show the influence of slag, fly ash and silica fume substitution by 35 wt. % on the physical and mechanical properties of Portland cement mortar.

2. Experimental

2.1. Raw Materials

The raw materials are Type I – Ordinary Portland cement (OPC), sand, granulated blast furnace slag (GbfS), silica fume (SF) and fly ash (Fa). The cement sample was obtained from Swiss cement company, Swiss, Egypt, the GbfS sample, activated by dried sodium silicate (Na_2SiO_3) was supplied by Helwan cement company, Helwan Egypt. The Fa sample was taken from the electrostatic precipitators of Trinec Power plant north of Moravia, Czech Republic, while the SF sample was imported from Norway. The Blaine surface area of the raw materials is 3400, 3100, 18x10^4 and 2750 cm^2/g, respectively. The chemical analysis of the raw materials is listed in Table 1. The mineralogical composition of the cement sample is C_3S, 59.4 %; β-C_2S, 14.13 %; C_3A, 8.67 % and C_4AF, 6.72 % as determined from Bogue Equations [37]. The XRD patterns of FA sample showed that it consisted mainly of a vitreous phase and composed of quartz and mullite phases as well as small amounts of hematite (Figure 1). Table 2 shows the data of calculated physical properties of raw materials, as the basicity coefficient, Kb = (CaO + MgO/ SiO_2 + Al_2O_3), hydration modulus, Hm = (CaO/ SiO_2 +Al_2O_3 + Fe_2O_3), silicate modulus, Sm = (SiO_2/Al_2O_3 +Fe_2O_3), aluminate modulus, Am = (Al_2O_3/Fe_2O_3), lime modulus, Lm (100 x CaO/2.8 SiO_2 + 1.1 Al_2O_3 + 0.7 Fe_2O_3) and specific gravity, g/cm^3 [5].

Table 1. Chemical composition of the raw materials, mass %

Materials Oxides	Cement	GBFS	FA	SF
L.O.I	2.44	0.33	1.81	0.93
SiO_2	20.53	38.04	52.56	94.38
Al_2O_3	4.68	5.76	26.85	1.11
Fe_2O_3	2.21	2.32	6.72	1.16
CaO	63.36	37.72	5.13	1.12
MgO	0.96	12.84	1.75	0.43
MnO	----	0.76	----	----
Na_2O	0.08	----	0.76	0.08
K_2O	1.16	----	3.84	0.51
SO_3	2.75	0.63	0.18	0.28
I.R.	1.73	1.60	0.40	----
Total	100 %	100 %	100 %	100 %

L.O.I: Loss on Ignition, I.R: Insoluble Residue

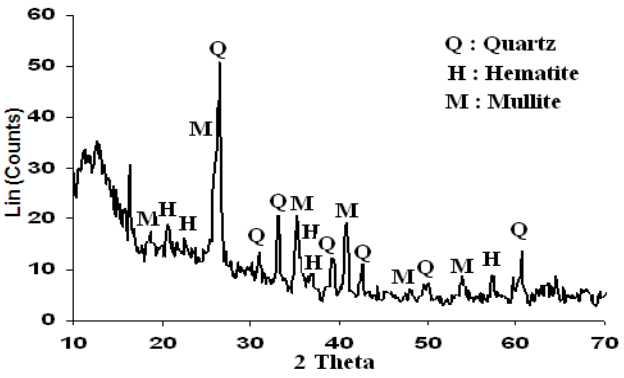

Figure 1. The XRD analysis of the used fly ash sample

Table 2. Physical properties of the raw materials, mass %

Property Mix	Kb	Hm	Sm	Am	Lm	Surface area, cm^2/g	Density, g/cm^3
CEM-I	2.45	2.23	2.6	2.57	97.06	3400	2.8957
GBFS	1.11	0.79	4.91	2.48	31.72	3800	2.4976
FA	0.09	0.06	1.57	4.00	2.83	4150	2.0381
SF	0.02	0.01	41.58	0.96	0.42	18x10^4	1.5928

2.2. Preparation and Methods

The Mortar (Mr) was prepared from CEM I and sand with the ratio 1:2, respectively. The reference mortar (Mr) was partially substituted by totally 35 mass% GBFS, FA and SF at the expense of sand, where 30 mass% variable ratios of GBFS, FA and a constant amount of SF by 5 mass% as shown in Table 3. The mortar composites were given the symbols M1, M2, M3, M4, M5 and M6, respectively. The blending process was mechanically done in a suitable mixer for an hour using five porcelain balls to assure the complete homogeneity of the prepared batches. The water/cement (w/c) ratio as well as setting times (initial and final) of the prepared mortar composites

(Mr-M6) were directly determined by Vicat apparatus [38,39]. The mortar pastes were mixed using the predetermined w/c ratios, then poured into 4 x 4 x 16 cm^3 stainless steel moulds for compressive and bending strengths, manually vibrated for three minutes and on a mechanical vibrator for 3 cycles to ensure the complete elimination of air bubbles and voids. The surfaces of moulds were smoothed by spatula and then kept in a humidity chamber at 100 % R.H and room temperature (17±1°C) for 24 hours.In the following day, it de-moulded and then cured at more than 95 % R.H up to 28 days. The physico-mechanical properties of the different cement mortars were measured at 1, 3, 7 and 28 days.

Table 3. Batch composition and Blaine area of mortar composites containing variable Proportions of GBFS, FA and S, mass%

Materials Mixes	Cement Mortar	GBFS	FA	SF	B. Area, cm^2/g
M0	100	-----	-----	-----	2880
M1	65	25	5	5	3160
M2	65	20	10	5	3290
M3	65	15	15	5	3350
M4	65	10	20	5	3470
M5	65	5	25	5	3520
M6	65	11.66	11.66	11.66	3385

The compressive strength [40] was carried out using a digital reading BAUTOFF PRÜF TONI TECHNIK testing machine–Model: TONINDUSTRIE PRÜFTECHNIK GMBH, D-1000 Berlin, Type 2560-249, 1997. The loading was applied perpendicular to the direction of the upper surface of the cubes. The compressive strength was calculated from the following relation:

$$Cs = L\,(KN) / Sa\left(cm^2\right) KN/m^2 \times 102\left(Kg/cm^2\right) / 10.2\,(MPa) \quad (1)$$

Where, Cs: Compressive strength (MPa), L is the load (KN), Sa is the surface area (cm^2). The flexural [41] was carried out using WAM-VEB THÜRIHGER INDUSTRIEWERK, testing machine - model RAUENSTEIN WPM, Berlin. The beam simply with three-point symmetrical loading system was applied to produce a constant loading on the mid-span area. The beam load was applied perpendicular to the axis of the sample (Figure 2). The values were calculated by the following equation:

$$Fs = 3/2\left(F \times S / W \times T^2\right) KN/cm^2 \times 102\left(Kg/cm^2\right) / 10.2\,(MPa) \quad (2)$$

Where, Fs is the bending strength (MPa), F is the loading force (KN), S is the span (cm), W and T are width and thickness of the sample (cm). To stop the hydration at any interval, about 10 g of the broken specimens after the determination of compressive and flexural strengths was dried at 105°C for 30 minutes. The combined water content of the hydrated samples pre-dried at 105°C for 24 hours was determined on the basis of ignition loss at 1000°C for 30 min. using BMT Venticell Oven, Brněnská Medicinská Tecknika a.s.

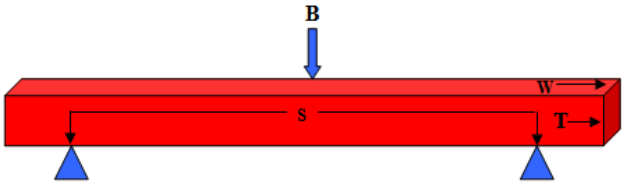

Figure 2. Schematic diagram of flexural (bending) strength system, B: beam, S: span, W: width and T: thickness

The phase composition of the hardened mortar pastes was investigated by infrared spectroscopy (FT-IR) and scanning electron microscopy (SEM. The FT-IR spectra was performed by FT-IR spectrometer in the range 4000-400 cm^{-1} and resolution 4 cm^{-1}, Nexus 670, Nicolet, USA. The SEM Microscopy, Mod. Jeol, ISM-T_{120}-Japan, was used. The fractured surfaces were coated with a thin layer of gold and scanned with a secondary electronic beam.

3. Results and Discussion

3.1. Physical Properties

The physical properties of the raw materials are listed in Table 2 and represented in Figure 1, while the relationship between the fineness of the raw materials and its density is plotted in Figure 3. It is clear that the OPC has the higher and nearly maximum hydration modulus and the SF has the higher silica modulus than those of other raw materials, while the raw materials have very near or close rates of other properties. Also, as the fineness of raw materials increased, the density decreased (Figure 4). This indicates that the three types of waste materials are siliceous and they have not any hydraulic properties in nature. The data shown in Table 4 indicate that the waste materials are conform the conditions of ASTM Standard-C618-01 to be used as mineral admixtures for cement pastes, mortars or even concretes.

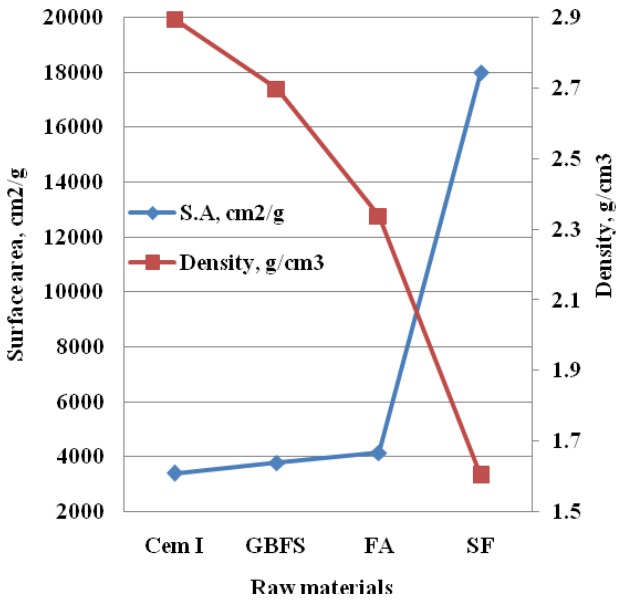

Figure 3. The relationship between the fineness and density of the starting raw materials

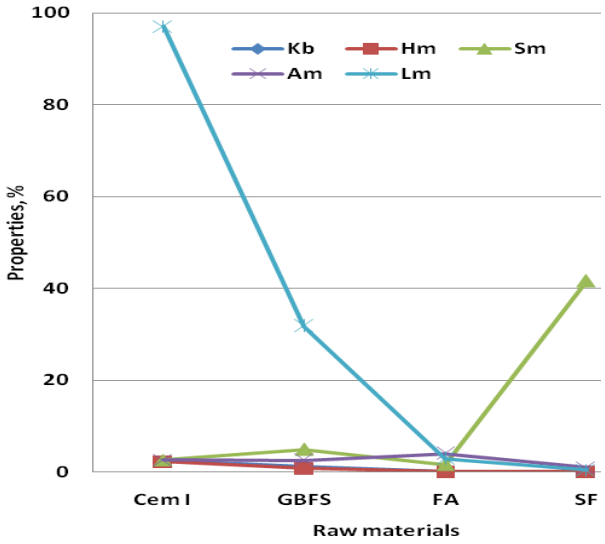

Figure 4. The relationship between the physical properties of the starting raw materials

Table 4. Conformance of the used waste materials to ASTM-C618-01

Materials Character	GBFS	SF	FA	Requirements Of control
(SiO_2+Al_2O_3+Fe_2O_3) Content, %	46.12	96.65	86.13	Min. 70 %
SO_3 content, %	0.63	0.28	0.18	Max. 4 %
L.O.I	0.33	0.93	1.81	Max. 10 %
Fineness, retained on 45 µm sieve, %	≈24	≈33	≈28	Max. 34 %

3.2. Water / Cement Ratio and Setting Time

The w/c ratio and setting times of the different mortar composites are plotted as a function of mortar composites in Figure 5. The w/c ratio as well as the setting times of the reference composite (Mr) was found to increase with FA content up to 25 mass% (M1-M5), and then decreased with mortar composite containing equal amounts of the three components (M6). However, the values of w/c ratio and setting times (initial & final) are still higher than those of the reference composite. This is mainly attributed to the higher surface area of these mortar composites comparing with that of the reference [42,43]. So, the incorporation of any of the three waste components increased both mixing water and setting times.

3.3. Combined Water Contents

The combined water contents of the different mortar composites are plotted as a function of mortar composites in Figure 6. Generally, the combined water content increased slightly with curing time up to 28 days. This is mainly due to the slight formation of hydration products, particularly during the early ages of hydration up to 7 days [44,45]. The little amount of hydration products is mainly due to the incorporation of sand in the reference mortar (Mr) which has no hydraulic properties, in addition to the siliceous nature of the additive components [43]. The combined water content of the reference mortar (Mr) tends to increase with FA content up to 25 mass% (M1-M5) and further increased when the mortar composite contains equal amounts of the three additive waste materials (M6) particularly at 28 days of hydration. The lower values of combined water contents of the reference mortar (Mr) and other mortar composites are essentially attributed to the reduction of the main hydrating phases by 35 mass%, in addition to that the sand has no hydraulic properties.

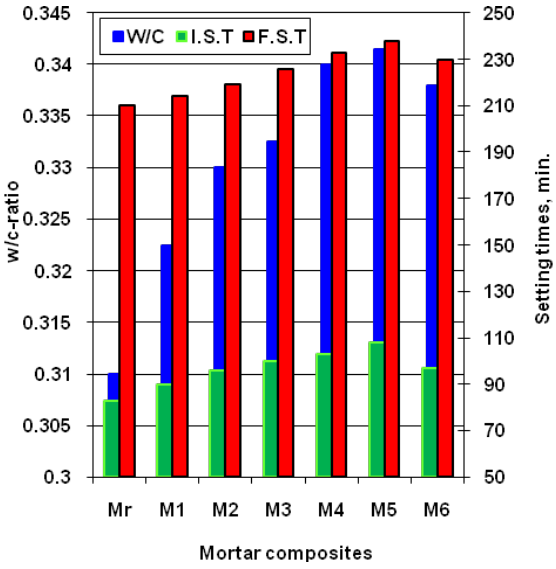

Figure 5. The W/C ratio and setting times of the various mortar composites

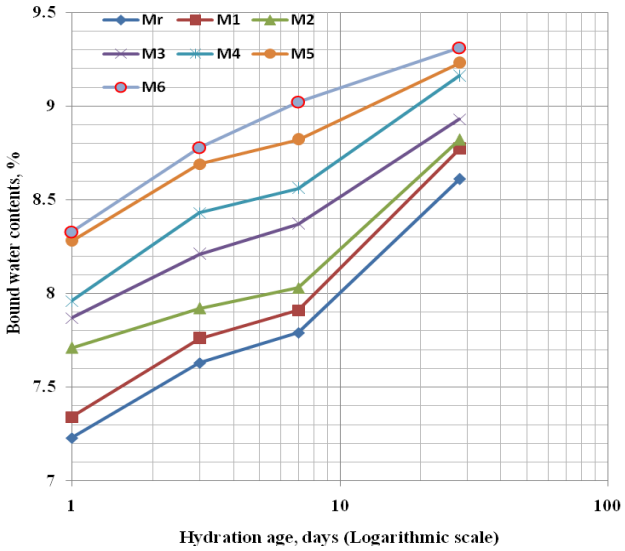

Figure 6. Boud water content of the various mortar composites hydrated up to 28 days

The increase of combined water contents of the different mortar composites is due to the pozzolanic reactions of FA and SF besides slag with a part of the released lime from the hydration of cement phases to form CSH, which in turn increase the combined water content. It is clear that the combined water contents of all mortar composites are higher than those of the reference at all curing ages. This is due to the presence of the hydrating and more pozzolanic materials than sand. The mortar composite containing equal amounts of the three waste materials (M6) achieved the highest values of combined water than those of the reference (Mr) and all other mortar composites (M1-M5) at all curing ages of hydration. This may be due to that the pozzolanic action is the maximum when the three components existed together with equal amounts [43,45,46].

3.4. Bulk Density

The bulk density of the different mortar composites are plotted as a function of mortar composites in Figure 7. The bulk density increased gradually with curing time up to 28 days due to the continual deposition of the formed hydration products in the pore volume of the hardened samples. The bulk density of the reference mortar (Mr) slightly increased when it was substituted with 35 mass% GBFS, FA and SF. This means that the bulk density of the different mortar composites (M1-M6) is slightly higher than those of the reference (Mr) at all curing ages of hydration. The later composite (M6) containing equal amounts of the three waste materials exhibited the higher values of bulk density compared with those of the other mortar composites (M1-M5).

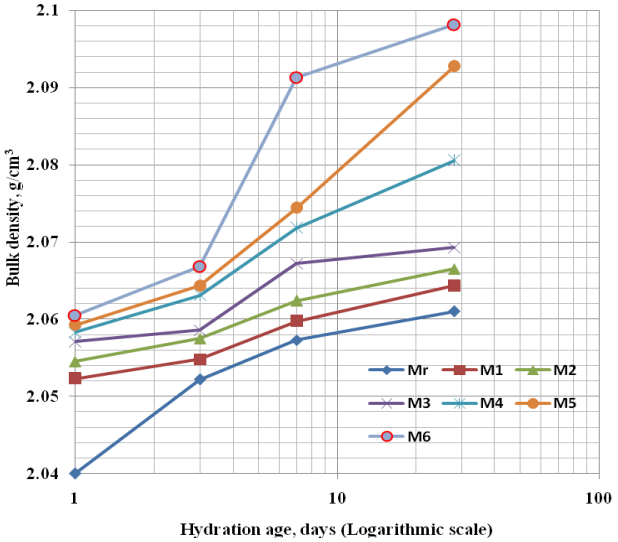

Figure 7. Bulk density of the various mortar composites hydrated up to 28 days

3.5. Compressive Strength

The compressive strength of the different mortar composites are plotted as a function of mortar composites in Figure 8. The compressive strength is generally increased with curing time up to 28 days. This is mainly due to the formation of hydration products which deposited into the pore structure of the hardened mortar samples. So, the total porosity decreased and the bulk density increased. This reflected positively on the mechanical strength (46). During the early stages of curing up to 7 days, the compressive strength of the reference mortar (Mr) increased slightly when replaced with 35 mass% GBFS, FA and SF (M1-M5) and largely increased during the later age of curing (28 days). At all curing ages, the compressive strength results displayed the same trend. This means that the compressive strength of the reference mortar (Mr) increased with FA content but decreased with slag content in presence of a constant ratio of SF [43,47,48]. This may be due to the variation of oxide composition of the mortar composites. This is attributed to that FA and SF enhances microstructure of the hardened cement pastes or concretes and the higher strengths are a consequence of the modification of microstructure as well as the lower permeability [47,48,49,50]. However, the mortar composite containing equal amounts of the three waste materials (M6) achieved the higher values of compressive strength compared with other composites. Consequently, the mortar composite (M6) could be selected to be the optimum mortar composite.

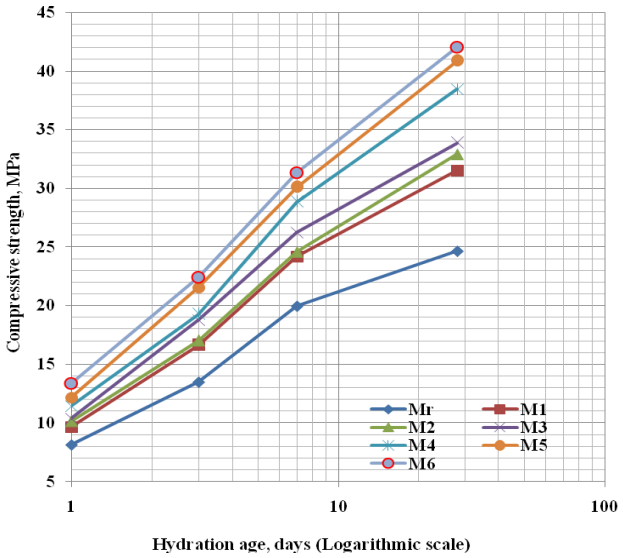

Figure 8. Compressive strength of the various mortar composites hydrated up to 28 days

3.6. Flexural Strength

The flexural strength of the different mortar composites are plotted as a function of mortar composites in Figure 9. The flexural strength displayed the same trend as compressive strength, but with little differences where the values of bending strength are lower than those of the corresponding compressive strength. The flexural strength of the different mortar composites (M1-M5) increased with FA content on the expense of slag in presence of SF. Also, the flexural strength values of the different mortar composites are higher than that of the reference mortar (Mr) at all curing ages. The mortar composite (M6) composed of equal amounts of GBFS, FA and SF recorded higher flexural strength results than those of the different composites at all curing ages.

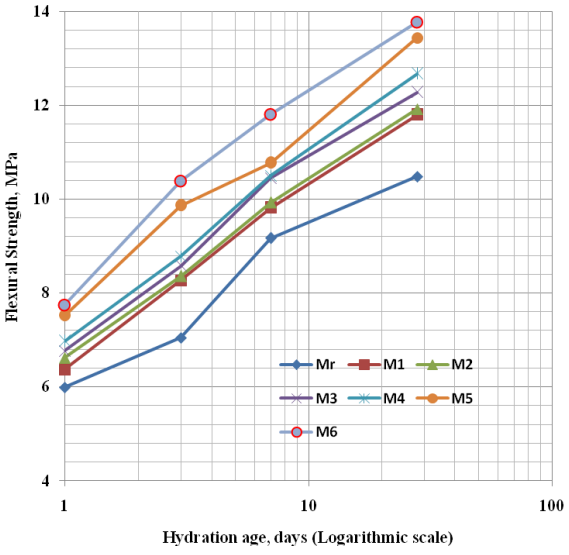

Figure 9. Flexural strength of the various mortar composites hydrated up to 28 days

3.7. The FT-IR Spectra

The FT-IR spectra of the reference mortar (Mr) as well as M2, M4 and M6 mortar composites, respectively are shown in Figure 10. The sharp absorption band at wave number 3644 cm^{-1} is related to the free OH^{-1} group coordinated to Ca^{2+} (free lime). The intensity of the 3644 cm^{-1} absorption band of the free CH of Mr decreased with FA content due to the continual consumption of free lime. The intensity of the broad absorption band at wave number 3434-3450 cm^{-1} which is due to the OH^{-1} group associated to H^{+} bond, i.e. related to the symmetrical stretching frequency of water, increased also with FA content due to the consumption of large amount of water molecules in the formation of hydration products. The two absorption bands at 1690-1684 and 1395-1380 cm^{-1} are related to the main silicate band involve Si-O stretching vibration bands of CSH and/or CAH. The intensity of the triplet absorption band at 1300-710 cm^{-1} characterizing CO_3^{2-} and/or SO_4^{2-} is irregular due to the rate of carbonation or sulphonation of CSH and /or CAH.

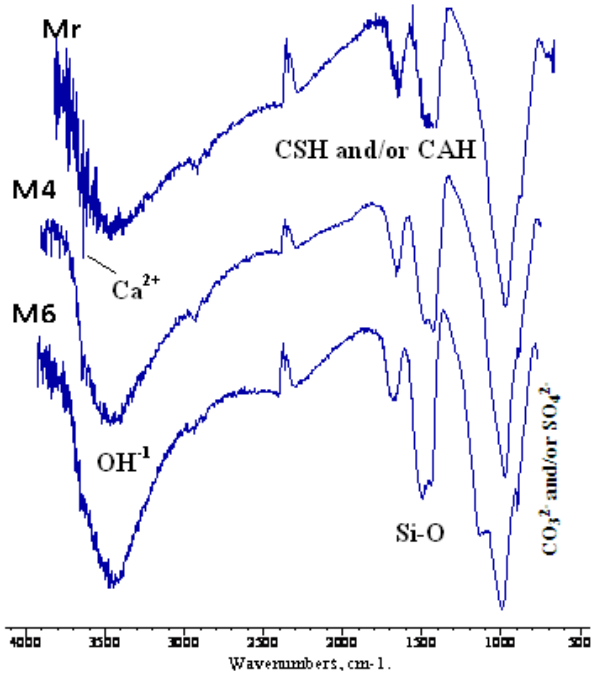

Figure 10. The FT-IR spectra of Mr, M4 and M6 mortar composites cured up to 28 days

3.8. Scanning Electron Microscopy

The SEM micrographs of Mr, M1, M3, M5 and M6 mortar composites are shown in Figure 11. The hardened cement pastes of Mr are very dense and exhibit a very low crystallinity, while those of M1 and M3 showed lower porosity and higher crystallinity than those of Mr. The hardened cement pastes of M5 and M6 showed that the formed phases in the matrix are not homogeneous due to the hydration and pozzolanic reactions between the different components. Moreover, M5 indicates the presence of abundant fibrous binder or CSH, while M6 showed that the porosity was further decreased due to the presence of high percentage of very fine fly ash and Silica fume.

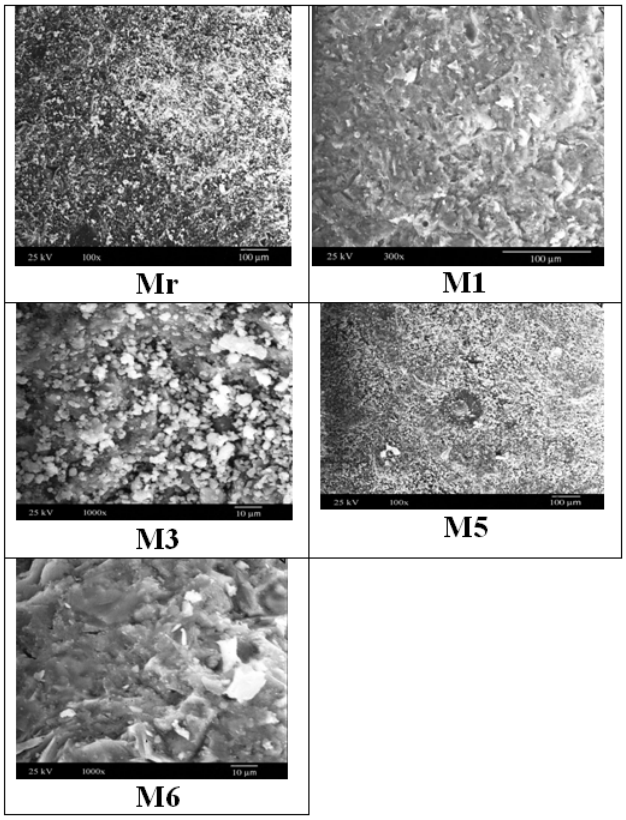

Figure 11. The SEM micrographs of the different mortar composites

4. Conclusions

Blending GBFS and FA with a constant ratio of SF in combination with cement mortar increase the W/C ratio and setting time of the fresh mortar as well as the chemically combined water content and bulk density of the hardened cement pastes. On the other side, the total porosity decreases. The compressive and flexural strengths were also improved and enhanced particularly at later curing ages. The combination of the three pozzolanic materials with equal amounts shows the most effective action on the properties of mortar composites compared with those of other combinations. The FT-IR spectra showed that the free lime content decreased with FA contents. Also, a larger amount of CSH and a lower amount of free lime could be detected by SEM micrographs.

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