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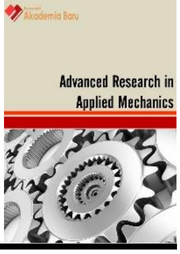

Evaluation of Superplasticized Mortars Containing High Volume Basic-Oxygen Slag Fine Aggregate Replacing Sand against Sea Water Attack

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ABSTRACT

The influence of superplasticizer addition on durability reinforcement of cement mortars made with basic-oxygen blast-furnace slag fine aggregate (BOF) as completely substitute for natural sand after being exposed to sea water solution for 6 M was evaluated by determining physico-mechanical characteristics in terms of water absorption and compressive strength in addition to microstructure analysis. In this procedure, all studied mixtures were doped with 1% and 2% SP. After the initial treatment of samples in tap water for 28 days, they were subjected to sea water solution for 6 M using water/binder ratios of 0.35 and 0.45. The new hydration phases and microstructure of hardened specimens were identified by X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. The results showed that superplasticized cement mortars with slag fine aggregate blended with 2% SP offered better properties and microstructure than their counterparts with natural sand against sea water attacks. Therefore they can be used in offshore structure applications.

Keywords:
Basic- oxygen blast-furnace slag, Cement mortar, Durability, Water Absorption, Compressive strength, SEM

1. Introduction

The development of concrete structures exposed to offshore environment is one of the most prominent issues facing the concrete industries in the twenty-first century. The use of such industrial by-products having desirable characteristics in concrete can result in saving of energy as well it is limiting the consumption of natural resources [1]. The iron and steel industry over the world produce huge amounts of slag as by-products. Of which, ground granulated blast furnace slag (GGBFS), basic-oxygen blast-furnace slag (BOF) and electric-arc-furnace slag (EAFS) [2]. Basic oxygen blast-furnace slag (BOF) is a final waste material in the basic oxygen furnace steel making process [3]. Some previous studies have been conducted to use BOF as fine aggregate substitute for sand. For example, Yung-Chin *et al.*, [4] used the BOF slag fine aggregate for making cement mortar. They concluded that the compressive and flexural strengths of specimens can reach 45.4 MPa and 7.1 MPa, respectively after different curing times. Bodor *et al.*, [5] prepared cement mortar specimens using the BOF slag replaced with 50% of natural sand aggregate.

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The mechanical and chemical properties of these mortars were enhanced at replacement level of 37.5%, by weight. In the same vein, Monkman *et al*., [6] indicated that cement mortars made of the fine aggregate of carbonated BOF slag can achieve high 28-day strengths compared to those containing natural sand. On the other hand, Tung-Hsuan *et al*., [7] indicated that the use of BOF slag fine aggregate in cementitious mortars significantly reduced the compressive strength and slightly increased the volume expansion. Durability is one of the most important characteristics to be considered in the design of reinforced concrete exposed to marine environment. Marangu *et al*., [8] pointed out that deterioration of concrete structures is generally caused by ingress of aggressive ions. Previous works indicated that various slags as by-products could replace natural sand in mortar and concrete structures against marine and aggressive environments.

Santamaría *et al*., [9] reported satisfactory results in terms of physical and mechanical properties as well dimensional stability of concrete mixtures containing electric steelmaking aggregate (ESMA) after being exposed to aggressive environments compared to reference ones. Authors confirmed the suitability of slag-concrete for use in offshore structures. Whilst, Polanco *et al*., [10] prepared concrete structures using two types of steelmaking slags, namely electric-arc furnace slag (EAFS) and ladle-furnace slag (LFS). Specimens were evaluated for durability and resistance to external agents in terms of freezing-and-thawing cycles, wetting-and-drying cycles, potential expansion in hot water, and climatic chamber aging. They concluded that mixtures made with EAFS in form of coarse and fine aggregates offered excellent performance in aggressive media; however, the inclusion of LSF fines in EAFS mixtures negatively affected on the physico-mechanical properties of tested samples.

Arribas *et al*., [11] studied the effect of chemical degradation such as sulphate attack, alkali–aggregate reaction and marine environment on concrete incorporating electric-arc furnace slag as a substitute for the conventional aggregate. The study emphasized the possibility of producing steel-reinforced concrete with slag aggregate. Humam and Siddique [12] studied the effects of replacement of sand- fine aggregate with high percentages of iron slag on compressive strength, split tensile strength, sulphate resistance and rapid chloride permeability test of cement mortar. The percentages of replacement were 0%, 10%, 20%, 30%, and 40% by weight of fine aggregate. Results indicated that inclusion of iron slag as partial replacement with fine aggregate enhances the properties of Mortar. Other studies have included the use of blast-furnace as a partial substitute of cement for enhancing concrete resistance against aggressive environment. Among them, Najimi *et al*. [13] conducted a series of tests including compressive strength, expansion measurements and microstructural analysis on concrete made by replacing OPC with copper slag at levels of 0%, 5%, 10% and 15% against sulphate solution attack. The results indicated that the inclusion of slag had a significant effect on concrete properties. With regard to the use of BOF slag as a substitute of natural aggregate for reinforcement the durability performance of mortar and concrete structures against sea water and aggressive environments, and after reviewing several previous researches, it was turned out to this day there has not been any research paper dealt with the sustainability of superplasticized cement mortar incorporating basic-oxygen blast-furnace slag as a replacement of natural sand versus sea water attacks. Accordingly, the prime goal of current study is to evaluate the durability performance of superplasticized cement mortar made with high volume basic-oxygen blast-furnace slag fine aggregate before and after being exposed to marine environment for 6 months, and then comparing with those made with natural sand. While designing some mixtures, BOF slag fine aggregate was completely substituted with sand. To enhance long-term sustainability, all mixtures were doped with 1% and 2% superplasticizer. The physico-mechanical characteristics including water absorption and compressive strength of hardened specimens were determined at different curing ages of 0 (28 days), 1, 3 and 6 months. The composition of new phases and microstructure analysis of studied samples were identified using XRD and SEM techniques.

2. Experimental Details

2.1 Materials and Mix Proportioning

The following section deals with the physico-mechanical properties of raw materials involved in the preparation of mortars. In the present paper, materials used include ordinary Portland cement-CEM I (42.5 N), in accordance with the requirements of ASTM C-150 [14], supplied by Suez Cement Plant. Two types of fine aggregates were used in the design of mixtures. The first was natural sand (S) with fineness modulus of 2.65 which was used in preparation of reference samples, while the other was basic-oxygen-blast-furnace slag aggregate (BOF), characterized by cubical shape and rough surface texture, provided from the Iron and Steel Plant, Helwan, Egypt. In the design of some mixtures, BOF slag was completely substituted by sand. Before mixing procedure, materials were carefully washed to get rid of the adhesive fine particles, and then oven dried at 100 ^oC for 24 h. Also, the BOF slag aggregate was passed through a 1 mm diameter sieve and retained on a 600 µm sieve. The physical features of slag including roughness and hardness nature make it more durable than sand. Table 1 outlines the chemical composition of raw materials as conducted by XRF technique. The quantified crystalline phases of BOF slag aggregate were investigated using XRD spectrum as given in Figure 1. Slag aggregate consists mostly of oxides which are similar to natural rocks and it also has alkaline properties such as OPC cement Kim *et al.* [15]. The physical properties of BOF slag in comparison with natural sand were illustrated in Table 2. As shown in Table, BOF slag meets the requirements of ECCS 203 [16] and ESS 1109 [17] specifications. A Sikament-R 2004- based superplasticizer-type G (SP) with specific gravity of 1.195 kg/l and solid content of 40 %, supplied from Sika Misr Company for Chemicals of Building Materials, Cairo, Egypt was used to promote sustainability of structures.

Table 1
Chemical composition of the raw materials, (wt., %)

Oxide content	OPC	S	BOF
SiO_2	21.26	94.84	8.70
Al_2O_3	4.49	2.12	0.78
Fe_2O_3	3.49	0.82	39.41
CaO	63.81	0.52	39.52
MgO	2.02	0.10	0.92
SO_3	3.11	0.11	0.24
L.O.I	1.57	0.22	0.11
Na_2O	0.14	0.27	-
K_2O	0.09	0.69	-
Cl^-	0.03	0.06	-
Total	99.97	99.98	89.68
Ins. Res.	0.53	-	-
Na_2O Eq.	0.20	-	-
L.S.F	0.92	-	-
C_3A	6.00	-	-
C_3S	54.15	-	-
C_2S	20.18	-	-

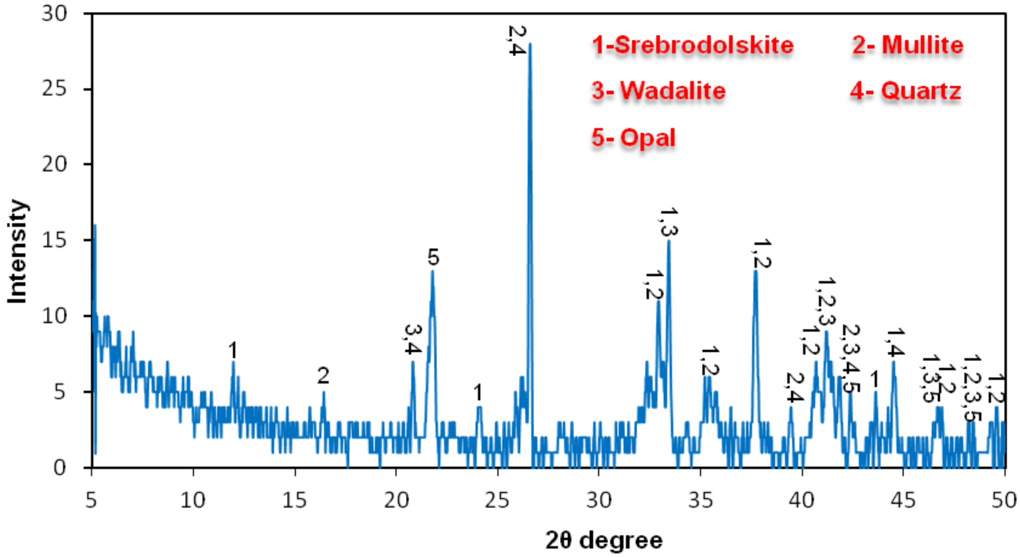

Fig. 1. XRD pattern of basic oxygen blast-furnace slag

Table 2
Physical and mechanical properties of fine aggregates

Properties	S	BOS	Acceptance and rejection limits
Specific gravity	2.52	2.9	-
Water absorption (%)	< 1	1.6	$\leq$ 2.5 according to ECCS 203 [16]
Moisture content (%)	0.63	0.3	-
Bulk density (t/m^3)	1.52	1.87	-
Impact coefficient (%)	-	14	$\leq$ 45 according to ESS 1109 [17]
Crushing coefficient (%)	-	18	$\leq$ 30 according to ECCS 203 [16]

Two classes of cement mortars with water/binder ratios of 0.35% and 0.45% were prepared. Reference superplasticized mortars blended with 1% and 2% SP of total weight of OPC was manufactured using sand as fine aggregate. Other superplasticized mixtures containg similar ratios of SP were made with BOF slag as fine aggregate. The mixture proportions of all samples were summarized in Table 3. They had given labels S0, S1, S2, BOF0, BOF1 and BOF2 for sand and slag mortars, respectively. In dry conditions, the ratio of cement to fine aggregate has been determined to be 1:3 for all mixtures. The raw materials were experimentally mixed together on dry using a bench-mounted mixer of 5 L capacity to ensure complete homogeneity. After that the required amount of mixing water was poured. The superplasticizer and remaining of mixing water was added. Continuous and vigorous mixing was conducted for 5 min. The fresh pastes were cast in three layers into 25x25x25 mm cubic steel molds, and then manually pressed until each layer being consolidated using a vibrating table. After compacting top layer by hand, the outer surface of paste was smoothed by the aid of thin edged trowel. Subsequently, the specimens were covered with wet burlap, and then kept in laboratory at room temperature of 25°C for 24 hrs. After demoulding, specimens were cured in tap water for 28 days (zero-time). Thereafter, samples were submerged in sea water for 6 months. In order to keep the solution concentration as constant as possible, the solution is changed every month. The chemical composition of solution is presented in Table 4. The influence of sea water attacks on the physico-mechanical properties of hardened mixtures was investigated by determination of water absorption and compressive strength at different curing times. Once the compression test was performed on the selected samples, the hydration reaction was stopped by immersing crushed species in 1:1 methanol/acetone solution for 24 hrs. After that specimens were exposed to drying at 70 °C for 1 h, and then they kept in an air-tight bottle for further investigation.

Table 3
Mixing proportions of all studied specimens

Mixture ID	Mix proportioning, (%)				
	OPC	S	BOF	SP	w/b
S0	25	75	-	-	0.45
S1	25	75	-	1	0.35
S2	25	75	-	2	0.35
BOF0	25	-	75	-	0.45
BOF1	25	-	75	1	0.35
BOF2	25	-	75	2	0.35

Table 4
The chemical composition of sea water solution

pH	SO_3	Cl^-	TDS	Ca^{2+}	Mg^{2+}	Na^+	K^+	Total hardness
7.41	3601	21584	42650	311	1458	11993	480	6756

Water absorption test was conducted in accordance with ASTM C140 [18]. Absorption ratio was calculated using equation below:

$$\text{Absorption (\%)} = [(W_2 - W_1)/ W_1] \times 100$$

where W_1 is weight of sample after drying at 105 °C, W_2 is the final weight of surface dry sample after immersion in water for 24 hrs.

2.2. Testing Procedure

At specified immersion times, the compression test was measured in accordance with ASTM C109 M [19] by exposing three cubes of each mixture. The chemical composition of raw materials was performed using XRF Spectrometer Philips PW1400. The samples were prepared for analysis using (Rubidium) Rb-kα radiation tube at 50 kV and 50 mA. The XRD procedure was investigated using a Philips PW 1050/70 Diffractometer. The data were identified according to the XRD software (pdf-2: database on CD-Release 2005). The microstructure of hardened samples was studied using scanning electron microscope, SEM Inspect S (FEI Company, Holland).

3. Results and Discussions

3.1. Water Absorption Measurements

To evaluate the pore characteristics, water absorption of the cement mortars has been determined. Figure 2 describes water absorption ratios of different mixtures blended with proportions of SP after being exposed to sea water for 6 M. Reference samples manufactured using natural sand showed a decrease in water absorption values with increasing the exposure times up to 3 M, particularly S0, S1 and S2. Whilst similar mixtures made with BOF slag termed as BOF0, BOF1 and BOF2 exhibited a decrease of up to 6 M. This trend is attributed to reduction of the pore volume as a result of the deposition of hydration products including C-S-H that is caused to a dense structure. The results indicated that the inclusion of SP to mix structure caused a decrease in water absorption depending on applicable ratio compared to plain mixtures. Superplasticized mortars with sand aggregate doped with 1% SP displayed ~ 16.24%, 34.65% and 36.81% at 0, 1 M and 3 M, respectively lower values than their counterparts free of SP. However, they offered a significant increase of 5.95% at 6 M. Reduction trend of water absorption was achieved upon completely replacement of BOF slag with S.

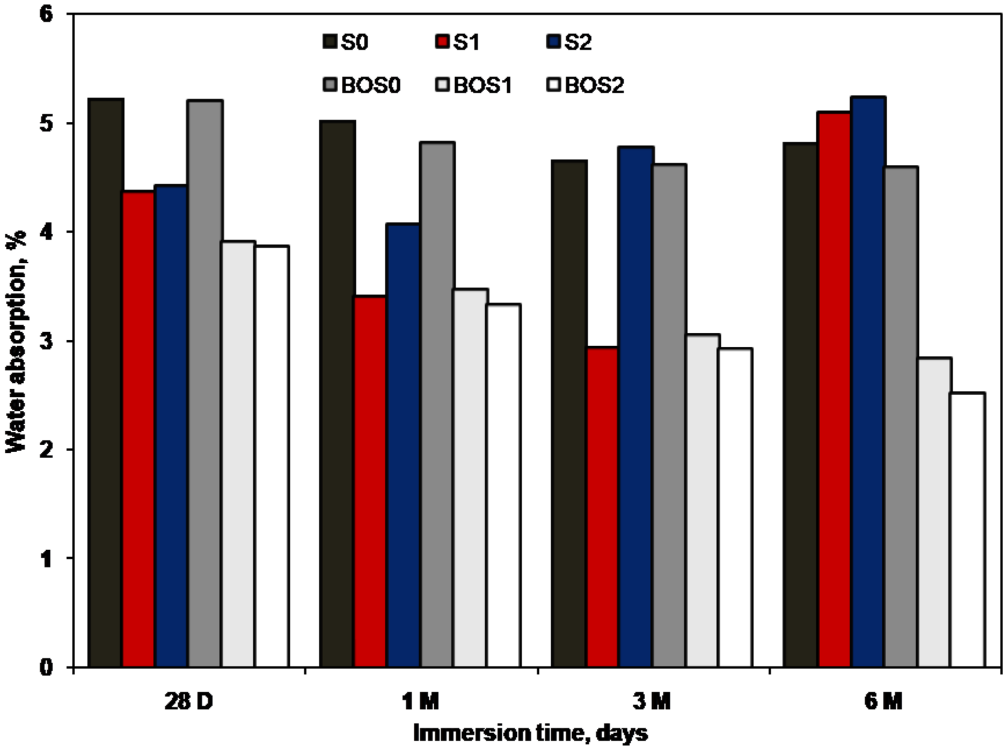

Fig. 2. Water absorption of cement mortars at different curing ages

By comparing the reference samples for all mixtures, it was found that specimens with BOF aggregate experienced lower values of 0.13%, 3.84%, 0.66% and 4.57% at 0, 1 M, 3 M and 6 M, respectively than those containing sand aggregate. Nadeem and Pofale [20] indicated that the reduction of water absorption could be attributed to good adhesion force between crystallized slag fine aggregate and cement paste as a result of rough surface of aggregate particles. The inclusion of SP ratio in mortar matrix with BOF slag had played a prominent role in water absorption reduction. Mixtures blended with 1% exhibited ~ 24.87%, 27.93%, 33.76% and 38.28%, respectively at all curing times compared to reference ones. However, they exhibited values of ~ 25.78%, 30.74%, 36.67% and 45.26%, respectively upon increasing dosage to 2%. Alsadey [21] pointed out that the decrease of water absorption for mixes blended with different ratios of SP is principally attributed the increase of entrapped water inside the cementitious matrix, which led to promotion of cement hydration.

3.2 Compressive Strength Development

The compression test is the most important procedure that can be used to assure the engineering quality in the application of building structures. The results of compressive strength of all mortars as a function of curing times up to 6 months are graphically represented in Figure 3. As shown, compressive strength of mixes made with natural sand increased with increasing the testing time up to 3 M, and then decreased at 6 M. In contrast, mixes containing BOF slag as a fine aggregate exhibited a remarkable increase with age up to 6 M. Reduction of w/b ratio from 0.45 to 0.35 resulted in an increase in compressive strength values. Accordingly, mix S1 doped with 1% SP showed a slight increase in an average of 1.33% at ages up to 3 M, followed by a decrease at later ages compared to the neat mixtures. With an increase of SP to 2%, mix S2 indicated higher strength values in an average of 1.26% up to 1 M, followed by a decrease of 0.6% at 3 and 6 M than corresponding reference mortars. Poon *et al.*, [22] showed that addition of extra amounts of SP may cause strong segregation for different components of cementitious matrix, which had a significant impact on decrease of strength. As it would be expected, superplasticized reference specimens containing high volume BOF slag significantly increased compressive strength by 1.85%, 1.52%, 1.65% and 1.95%, respectively at all different ages than those containing sand.

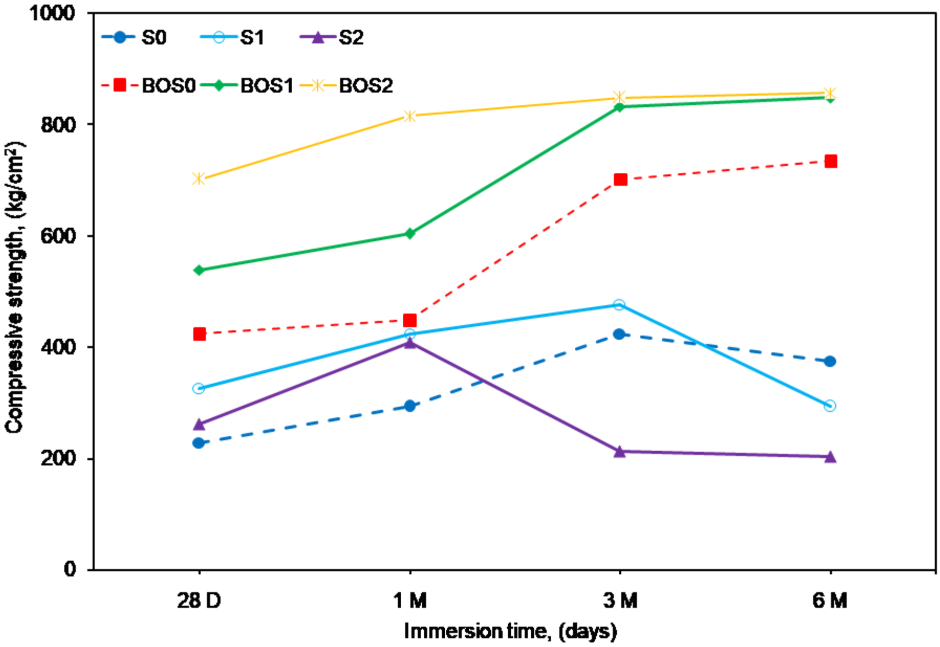

Fig. 3. Compressive strengths of cement mortars at different curing ages

Khanzadi and Behnood [23] attributed this tendency to the mechanical characteristics of slag aggregate. Moreover, the surface texture of aggregate is partly responsible for the increase of adhesion force between cement paste and aggregate in cementitious matrix due to the mechanical interlocking. By comparing the strength development of mixtures doped with ratios of SP at w/b of 0.35; it was observed that the mix BOF1 has achieved higher values of 1.65%, 1.42%, 1.74% and 2.90%, respectively than corresponding mixes with sand at all ages. On the other hand, BOF slag mixtures with 2% SP (BOF2) offered a gradual increase by 2.68%, 2%, 3.36% and 3.82% compared to their counterpart manufactured by sand. From the aforementioned results, it can be concluded that superplasticized slag mortars with 2% SP along with reduction of w/b to 0.35 has achieved the highest compressive strengths among all studied mortars, indicating their significant effect on long-term sustainability against sea water attacks.

3.3 Phase Composition by XRD

The results of X-ray diffraction analysis of cement mortars containing natural sand blended with 2% SP after being exposed to sea water solution for 6 M are given in Figure 4. The samples were compared with their counterparts free of SP as well those hydrated in tap water for 28 days. The main hydration phases clearly identified in patterns were portlandite [$Ca(OH)_2$] as well un-hydrated larnite (β-C_2S). Obviously, dominant peaks of quartz, microcline and albite as constituents associated with sand aggregate were also detected. The area and intensity of portlandite peaks located at 2θ of 17.97 and 34.13 decreased with the progress of curing times. Although hydration occurs with liberation of $Ca(OH)_2$, however, it is not inferred the existence of C-S-H phases in the patterns. This trend can be attributed to increase the intensity of quartz peaks or their appearance in amorphous structure. XRD pattern of cement mortar doped with 2% SP displayed similar phases as well similar behaviour as in case of control mixtures, suggesting that the inclusion of 2% SP does not alter the formed hydration products as explained earlier by Alsadey [21].

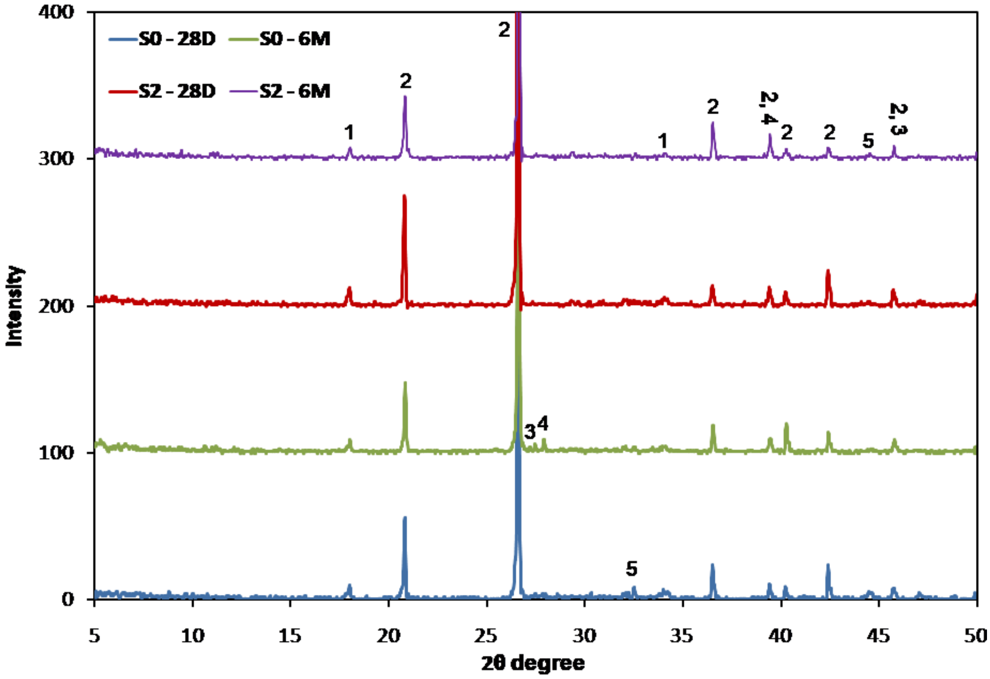

Fig. 4. XRD patterns of superplasticized cement mortars with sand aggregate cured for 28D and 6M; 1: Portlandite, 2: Quartz, 3: Microcline, 4: Albite, 5: Belite

On the other hand, Figure 5 shows the XRD patterns of superplasticized slag-cement mortars with 2% SP subjected to sea water solution for 6 M. After comparing them with plain mixtures, the patterns indicated the presence of $Ca(OH)_2$, C-S-H and β-C_2S cement hydration phases with minor peak detected for C_3S. In addition, crystalline phases of srebrodolskite, mullite, wadalite, quartz and opal were also appeared as main constituents of BOF slag aggregate. The results revealed that the area and intensity of peaks characterized for portlandite decrease as the age of curing increases. The reduction of $Ca(OH)_2$ intensity and presence of C-S-H phases indicated that free lime was consumed with time forming C-S-H gel that is filled some of open pores. Likewise, the inclusion of SP increased the entrapped water and promoted hydration of cement, which resulted in a further enhancement of durability against seawater attacks. At all curing times, the peak distinguished for β-C_2S is still present; pointing out the rate of hydration of C_2S is slower than C_3S. Furthermore, crystalline phases characteristic for BOF slag aggregate are still detected for all samples, which contribute to good mechanical stability.

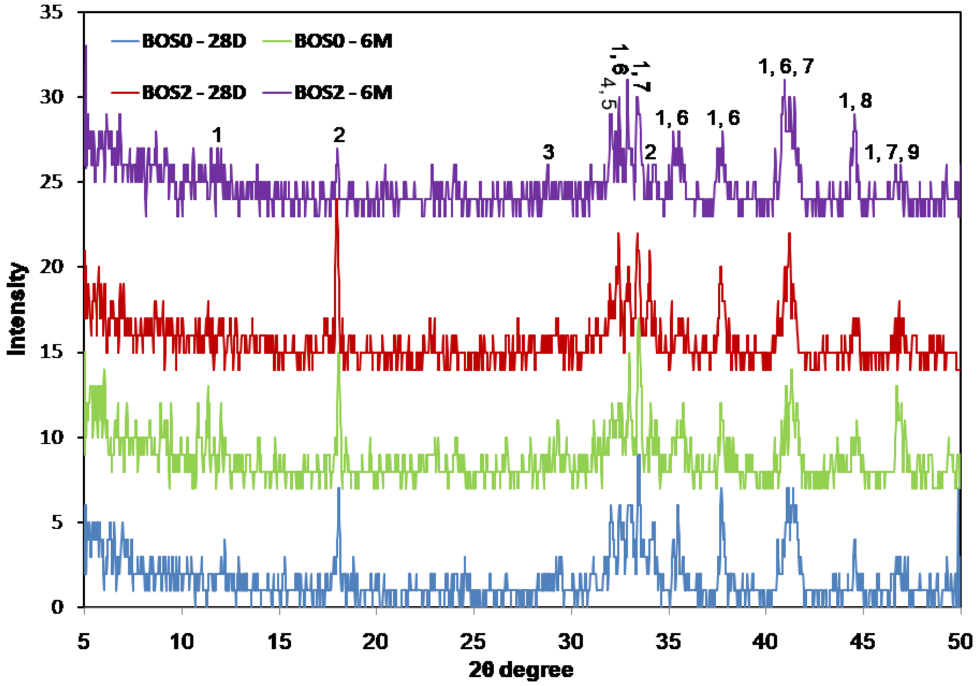

Fig. 5. XRD patterns of superplasticized cement mortars with slag aggregate cured for 28D and 6M; 1: Srebrodolskite, 2: Portlandite, 3: Calcium silicate hydrate, 4: Belite, 5: Alite, 6: Mullite, 7: Wadalite, 8: Quartz, 9: Opal

3.4 Microstructure Analysis by SEM

The SEM micrographs of superplasticized cement mortars made with slag and natural aggregates blended with 2% SP cured for 6 M are presented in Figure 6, and then compared with their control mixtures at 28 days. Regarding reference mixture S2 as shown in Fig. 6a, the SEM photograph exhibited dense closely packed microstructure that consisted of crystal-like C-S-H phases distributed symmetrically throughout mortar matrix. Evidently, the inclusion of 2% SP developed a stronger interfacial transition zone (ITZ) between aggregate and cementitious paste. In the same context, the SEM micrographs of sample S2 hydrated for 6 M (Fig. 6b), presented a relatively permeable microstructure by formation of microcracks spread over the microstructure, which resulted from the direct effect of sea water attacks. On the other hand, the SEM of superplasticized control slag mixture (BOF2) offered compacted and tough microstructure with lower porosity as well massive content of hydration products composed mainly of C-S-H from the hydration of OPC that filled most of the available pore spaces (Fig. 6c). Samples doped with 2% SP (Fig. 6d) showed the densest microstructure which formed mainly of C-S-H. By comparison, we find that the obtained results fall in line with compressive strengths reported in Fig.3. This enhancement related to the surface texture of slag aggregate responsible for increasing the adhesion force between binding material and aggregate particles in cementitious matrix, in addition SP accelerated hydration reaction by increasing the entrapped water.

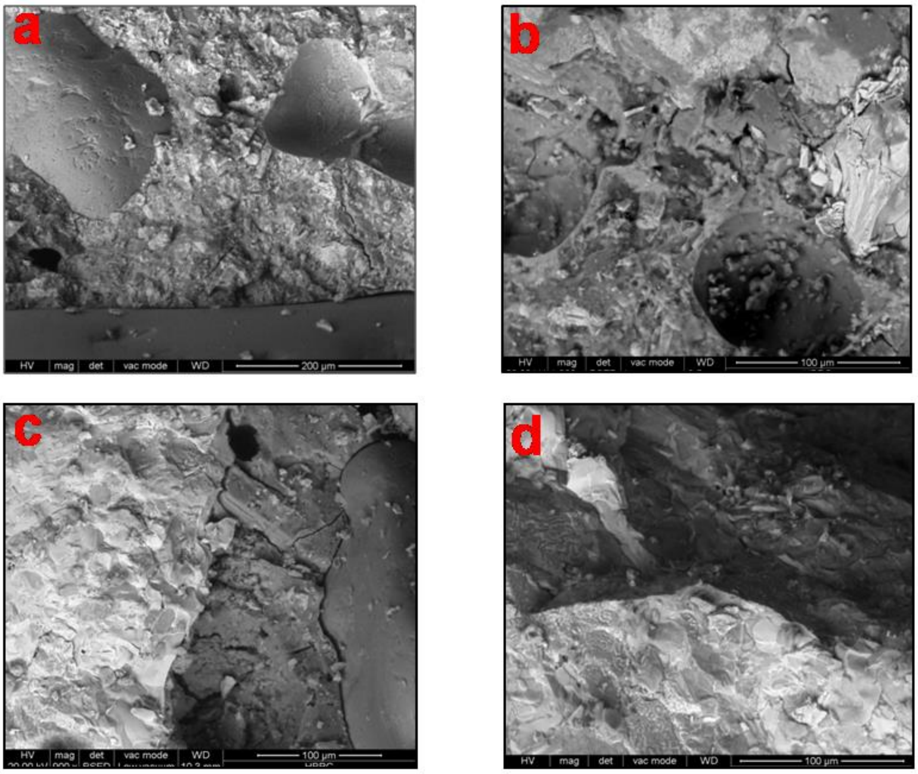

Fig. 6. SEM micrographs of superplasticized cement mortars containing sand and slag fine aggregates doped with 2% SP; a: S2 - 28D, b: S2 - 6M, c: BOF2 - 28D, d: BOF2 - 6M

4. Conclusions

This study presents an investigation for the durability performance of superplasticized cement mortars incorporating basic-oxygen blast-furnace slag fine aggregate as a substitute for natural sand after being exposed to sea water attacks for 6 M. The treated samples were compared with their reference counterparts hydrated for 28 days in tap water. After extensive discussion for physico-mechanical and microstructural properties, the following conclusions have been drawn.

1. Basic-oxygen blast-furnace slag aggregate exhibited favorable physical and mechanical properties in terms of specific gravity, moisture content, bulk density and crushing coefficient compared to natural sand.
2. The water absorption ratios of superplasticized sand-mortars doped with 2% SP decreased until age of 3 M, followed by an increase up to 6 M. While their counterparts made with completely replacement of BOF slag aggregate offered low values at all exposure times.

3. The inclusion of SP caused a positive effect on the compressive strength development for all studied specimens before and after exposure to sea water solution.

4. Regarding mechanical characteristics, superplasticized slag- mortars blended with 2% SP showed the highest compressive strengths among all mixtures at different curing times. This tendency can be attributed to the surface texture of slag aggregate responsible for increasing the adhesion force between the binding material and aggregate particles; moreover, addition of SP increased the entrapped water and then promotes the hydration reaction of OPC.

5. In terms of micrographs, the incorporation of 2% SP in cementitious mortar resulted in more durable microstructure, particularly for mixtures containing slag aggregate.

6. Ultimately, superplasticized cement mortars with BOF slag aggregate can be used as a substitute to their counterparts containing natural sand for offshore structure applications.

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