



Saw dust ash substitution for Portland cement pastes-Part II: Chemical resistance against sulfate attack

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General Note

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ABSTRACT

The chemical resistance or durability of Portland cement pastes (PS0) incorporating Saw dust ash (SDA) against the attack of 4 % MgSO_4 solution up to 12 months were investigated. The results showed that the water of consistency was increased, while setting times were decreased. The compressive strength slightly improved during the early ages of immersion only up to three months with the blank batch (PS0), but up to six months with other cement batches (PS1-PS6). Thereafter, certain deleterious or detrimental reactions started till forming high levels of ettringite and/or gypsum. These new phases are responsible for the decrease of compressive strength. Both combined water and total sulfate contents increased with time of immersion up to 12 months, but decreased with SDA content if compared with those of the blank (PS0). The blank cement pastes (PS0) recorded the highest values of total sulfate content, i.e. it is very sensitive for SO_4^{2-} ions. At the end of the 12th month of immersion, the SO_4^{2-} ions only precipitated on the external and lateral surfaces of the hardened cement pastes, and some horizontal minor cracks and little deformation could be seen on the edges of specimens with samples containing SDA, whilst large swelling was noted with cement

pastes of the blank (PS0).

Keywords: OPC, SDA, pozzolanic reactivity, sulfate attack, strength, ettringite, gypsum.

1. INTRODUCTION

1.1. Scope of the problem

The use of nanomaterials as nanoalumina, nanosilica and generally nanoparticles could significantly increase their mechanical properties and durability owing to the reduction in the total pore structure of cement-based materials [1]. Nanosilica, which is one of the earliest nanomaterials applied in cement-based composites, had a more obvious and evident effect on the performance improvements compared to other nanoparticles. This is due to that the nanosilica had a high pozzolanic reactivity with $\text{Ca}(\text{OH})_2$ in cement composites to create more hydration products or CSH and/or CAH- gels). Thereby, it is making the composites more compacted [2-4]. Generally, there are two types of sulfate attack can occur. The first is often accompanied by the formation of gypsum in the cement pastes with low C_3A immersed in high sulfate concentrations which is the first factor for the formation of ettringite. The second is characterized by the formation of ettringite ($\text{C}_3\text{A} \cdot 3 \text{CaSO}_4 \cdot 32 \text{H}_2\text{O}$), in the cement pastes with high C_3A immersed in low sulfate concentrations which is the first step of deterioration. The transition between the two types of sulfate attack was gradual, where both ettringite and gypsum could simultaneously occur. The formation of ettringite always leads to expansion, while gypsum usually leads to the loss of cohesion and softening, but no more to a marginal degree of expansion. The loss of cohesion is more directly due to the incorporation of sulfate ions into the CSH and / or CAH-gels [5-8].

The influence of these aggressive media on the behavior of building materials is a topic of significant interest for durability of cement structures [7-12]. This problem could be overcome or at least reduced by the use of mineral admixtures in cement to improve the strength development by the modification of the microstructure of the cement. The use of these cements is well accepted due to the resulting savings through the conservation of energy and cement as well as the production of low-lime products with good mechanical properties and durability [10-14]. The most common types of mineral admixtures are granulated blast furnace slag (GbfS), fly ashes (Fa) and silica fume (SF) which are coming from the various sectors of industry are pozzolanic materials [15-20]. When the pozzolanic materials dispersed into the cement paste, they generate a large number of nucleation sites for the precipitation of the produced hydration products. Hence, this mechanism makes the paste more homogeneous and dense as for the distribution of the finer pores due to the pozzolanic reactions between the amorphous silica and $\text{Ca}(\text{OH})_2$ formed during the normal hydration process of cement. This positively affects the resistance of concrete [6,7]. So, it is not surprising that some advances in cement and concrete technology have occurred as a result of three driving forces: the speed of construction, durability of concrete and environmental friendliness of industrial materials [19-23]. The sulfate attack has been reported to cause a damage of concrete for over a century. The media containing sulfate ions lead to the aggressive attack of cement pastes, mortars and concrete. Some constituents of the cement can enter into some deleterious reactions with these ions [5-8]. At least, six types of reactions could be described by sulfate attack. The most common of which often used to explain the defined sulfate attack are ettringite and gypsum formation.

The addition of supplemental cementitious materials such as Fa, GbfS and SF could improve the mechanical properties and durability of cement-based materials [24-26]. In the present study, the saw dust ash blended cement pastes were conducted to sulfate attack to evaluate its effect on the resistance of cement pastes containing it.

1.2. Objectives of the study

In Part I, the physical and mechanical properties of Portland cement pastes incorporated saw dust ash (SDA) were investigated [27]. were selected and focused on the effect of MgSO_4 solution on their hydration. So, the main objective of the current study is to evaluate and focused on the influence of 4 % MgSO_4 solution on the durability of Portland cement composites containing the waste of saw dust ash up to 12 months.

2. EXPERIMENTAL

2.1. Raw materials

The raw materials used in the present study are Ordinary Portland cement (OPC, Type I- CEM I 42.5R) and saw dust ash (SDA). The OPC sample was supplied from Sakkara cement factory, Giza, Egypt, and its commercial name is known as "Asmant El-Momtaz", while the saw dust sample (SD) was received from a local wood plant, Egypt. Sawdust is an industrial by-product coming from

cutting, grinding, drilling, sanding, or otherwise pulverizing wood using a saw or any other similar tool (Fig. 1). Saw or wood dust is tiny or very fine particles of wood that are formed from sawing or sanding wood using a saw in cutting. It represents a hazard especially in terms of its flammability. The sample of saw dust was sieved using 63 μm sieve. The Blaine surface area of the OPC and sawdust were 3400 and 4150 cm^2/g , respectively. The chemical analysis of the starting raw materials is shown in Table 1. The mineralogical composition of the OPC sample is given in Table 2.



Fig. 1- The saw or wood dust (SD)

Table 1 Chemical analysis of the Raw Materials using XRF method %

Materials Oxides	OPC	SD	SDA
L.O.I	2.64	0.84	0.84
SiO ₂	20.12	72.37	66.17
Al ₂ O ₃	5.25	5.75	4.35
Fe ₂ O ₃	1.29	2.36	2.36
CaO	63.13	10.06	10.06
MgO	1.53	4.61	4.41
MnO	0.36	1.96	2.10
Na ₂ O	0.55	0.22	0.08
K ₂ O	0.30	0.18	0.12
P ₂ O ₅	0.19	0.31	0.46
SO ₃	2.54	0.52	0.30
Blaine surface area, cm^2/g	3400	4150	4720

Table 2 Mineralogical composition of the OPC sample, mass %.

Phase Material	C ₃ S	β -C ₂ S	C ₃ A	C ₄ AF
	46.81	28.43	5.90	12.56

2.2. Preparation and methods

At first, the sample of saw dust was processed and washed separately with running water for few minutes and also it washed with distilled water. It then was well dried in the open air under direct sun for two days. It then burned at 600°C for 4 hours, and screened to pass through 200 mesh sieve (63 μm) to obtain what is known as "Saw Dust Ash or SDA". The batches of cement composites are given in Table 3.

Table 3 Cement batch composition, density, g/cm^3 and fineness, cm^2/g of all cement batches

Materials Batches	OPC, Wt. %	SDA, Wt. %	Fineness, cm^2/g	Density, g/cm^3
PS0	100	---	3350	2.3205
PS1	95	5	3764	2.1043
PS2	90	10	4100	1.9705
PS3	85	15	4445	1.9546

PS4	80	20	4670	1.9231
PS5	75	25	4825	1.8796
PS6	70	30	4975	1.8571

The physical and mechanical properties of Portland cement mixed with saw dust ash (SDA) were investigated before in Part I [27]. In the present Part II, the resistance of the Portland cement pastes blended with SDA against 4% MgSO_4 was investigated and compared with that of the blank (OPC). The blending process was mechanically made in a porcelain ball mill containing three balls for one hour to assure the complete homogeneity of all batches. Before casting of cement cubes, all moulds were oiled with a thin film of motor engine oil, to facilitate the release of the cubes from the moulds during the de-moulding. During casting of cement pastes, a certain percentage of Na-lignosulphonate admixture was added to all cement mixtures during mixing to avoid the agglomeration of the nanoparticles of cement powder. Sodium lignosulfonate admixture (SLA) was applied due to its higher activity than other conventional ones (Fig. 2).

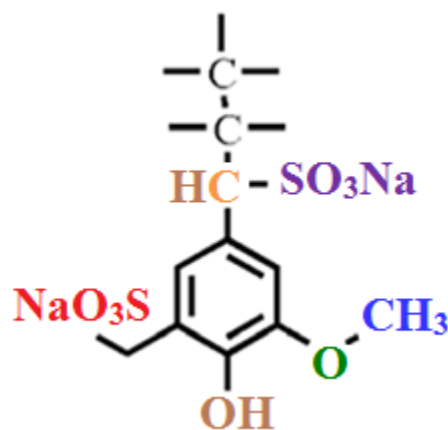


Figure 2-The chemical structure of sodium lignosulphonate.

Each already prepared oil mould was filled with the premixed cement composite and rammed 10 minutes to remove all air bubbles tapped within the mixture. The moulds were filled to the top surface and smoothed with a flat stainless steel trowel or spatula to obtain a flat and smooth surface [28].

The standard water of consistency as well as setting time of the prepared cement pastes were directly determined by Vicat Apparatus [29,30] as follows:-

$$\text{WC, \%} = A / C \times 100 \quad (1)$$

The cement pastes were mixed using the predetermined water of consistency, then molded into one inch cubic stainless steel moulds ($2.5 \times 2.5 \times 2.5 \text{ cm}^3$), vibrated manually for five minutes, and then on a mechanical vibrator for another five minutes. After casting of all cement cubes, they were covered with a black wet sheet for the first 24 hours to prevent moisture loss. The moulds were kept inside a humidity cabinet for 24 hours at $23 \pm 1^\circ \text{C}$ and 100 % R.H., de-molded in the next day and soon cured under water up to 28 days. The measured values at 28-days for combined water, compressive strength and sulfate content for each cement mix were considered as zero readings. The compressive strength [31] was carried out and the loading was applied perpendicular to the direction of the upper surface of the cubes as follows:-

$$\text{CS} = L (\text{KN}) / \text{Sa} (\text{cm}^2) \text{ KN/m}^2 \times 102 (\text{Kg/cm}^2) / 10.2 (\text{MPa}) \quad (2)$$

Where, L is the load taken, Sa is the surface area. The rest of samples were then immersed in a freshly prepared 4% MgSO_4 for 12 months. The sulfate solutions were renewed every week so as to keep its concentrations nearly constant.

After carrying out the compressive strength, about 10 g of the broken samples was dried at 105°C for one hour, and then were placed in a solution mixture of 1:1 methanol: acetone to stop the hydration at any interval, and then kept in an airtight bottle [32,33].

The combined water content (CWn) of the hydrated samples was determined on the basis of ignition loss at 1000 °C for 30 minutes [34-36] as follows:-

$$\text{CWn, \%} = \frac{W1 - W2}{W2} \times 100 \quad (3)$$

Where, CWn, W1 and W2 are combined water content, weight of sample before and after ignition, respectively.

The total sulfate content [5-7] was determined by placing one gram of the dried sample (W1) in a beaker containing 300 ml freshly prepared half saturated lime water. The content was stirred for one hour using a magnetic stirrer and then filtered through a sintered glass funnel G. 4. The residue was washed several times with lime water and then with distilled water. Add 5 ml conc. HCl acid (Sp. gr. 1.18) and drop wise 10 ml BaCl₂ (100 g/l) to the boiling filtrate. The mixture was boiled gently for 5 minutes, let to cool gradually, and then filter. Wash several times with hot distilled water till free from chloride. Thus, the total sulfate content precipitated. Ignite the residue up to 850 °C for 30 minutes, cool in a desiccator and weigh (W2). The total sulfate content was calculated from the following relation:

$$\text{SO}_3\% = \frac{M2 \times 34.3}{M1} \times 100 \quad (4)$$

Where, M1 is the weight of the original sample, M2 is the ignited weight of the residue and 34.3 is the molecular ratio of SO₃ to BaSO₄ (0.343) multiplied by 100.

3. RESULTS AND DISCUSSION

3.1. Water of consistency

The water of consistency and setting times (initial and final) of the various cement pastes with and without saw dust ash (PS0-PS6) are shown in Fig. 3. The water of consistency of the OPC pastes (PS0) was found to be 29 %. This ratio tends to increase step by step with the increase of SDA content. This is mainly attributed to the gradual increase of the specific surface area or fineness of all batches (PS0-PS6). This is certainly responsible for the high water demand during mixing [7,9]. Despite of Na-lignosulfonate admixture, the water of consistency increased due to the high atmospheric temperature during testing.

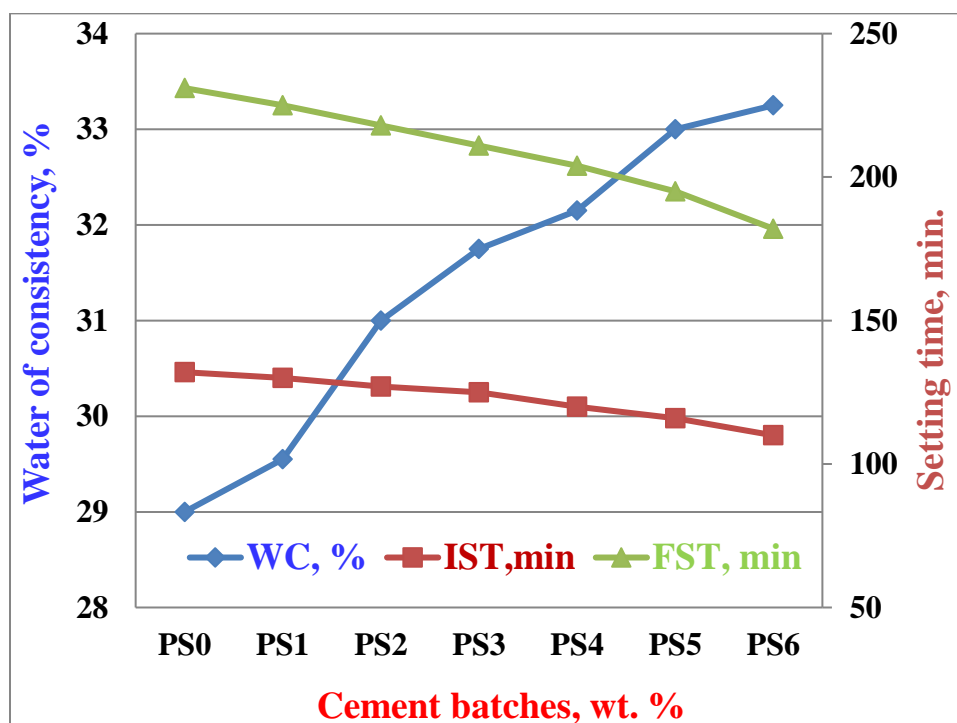


Fig. 3- Water of consistency and setting times of the various cement pastes containing saw dust ash.

On the other hand, the initial and final setting times are also shortened compared with those of the blank pastes (PS0). The reduction of setting times is often due to the higher atmospheric temperature during testing. Though the decrease of the cement portion, which is responsible for the setting and hardening of the cement pastes, the setting times decreased. This is because the OPC has a faster setting and hardening properties than the SDA. Furthermore, this may be attributed to the complex pozzolanic reactions of the SDA constituents with the emitted $\text{Ca}(\text{OH})_2$ resulting from the hydration of C_3S , C_3A and C_4AF of Portland cement forming some additional hydration products. Therefore, the setting is declined or accelerated [5,7,9,32]. Despite of Na-lignosulfonate admixture, the setting times also decreased due to the high atmospheric temperature during testing.

3.2. Compressive strength

The compressive strength results of the hardened cement pastes with and without saw dust ash (PS0-PS6) immersed in 4 % sulfate solution (MgSO_4) are represented as a function of immersing time up to 12 months in Fig. 4. The compressive strength of the hardened cement pastes of the OPC (PS0) increased slightly up to 3 months and then slightly decreased up to 6 months, but sharply decreased up to 12 months of immersion. This means that the 28-compressive strength of the pure OPC pastes (PS0) can withstand for only up to three months in 4 % MgSO_4 (M_2). This is mainly due to that at early stages of immersion, the total porosity of the hardened cement pastes are nearly the maximum. Therefore the rate of diffusion of sulfate ions could be partially eliminated and only form a white coat on the lateral surfaces of the specimens which hinders the further penetration of the sulfate ions through the cement surfaces [19]. So, a slight increase in the compressive strength due to the normal hydration process and the slight activation of sulfate ions [9,15,19].

The decrease of compressive strength is mainly attributed to the aggressive attack of sulfate ions so that the cement pastes could not resist the diffusion of SO_4^{2-} ions inside the cement pastes more than three months. This gave a big chance to occur some detrimental reactions ended by the formation of a large quantity of ettringite and/or gypsum. The latter phases often cause expansion followed by internal minor cracks and the disintegration of the formed hydrates, which are the centers of binding or cementing properties. These are the main factors of a strength loss [2-4,16,19].

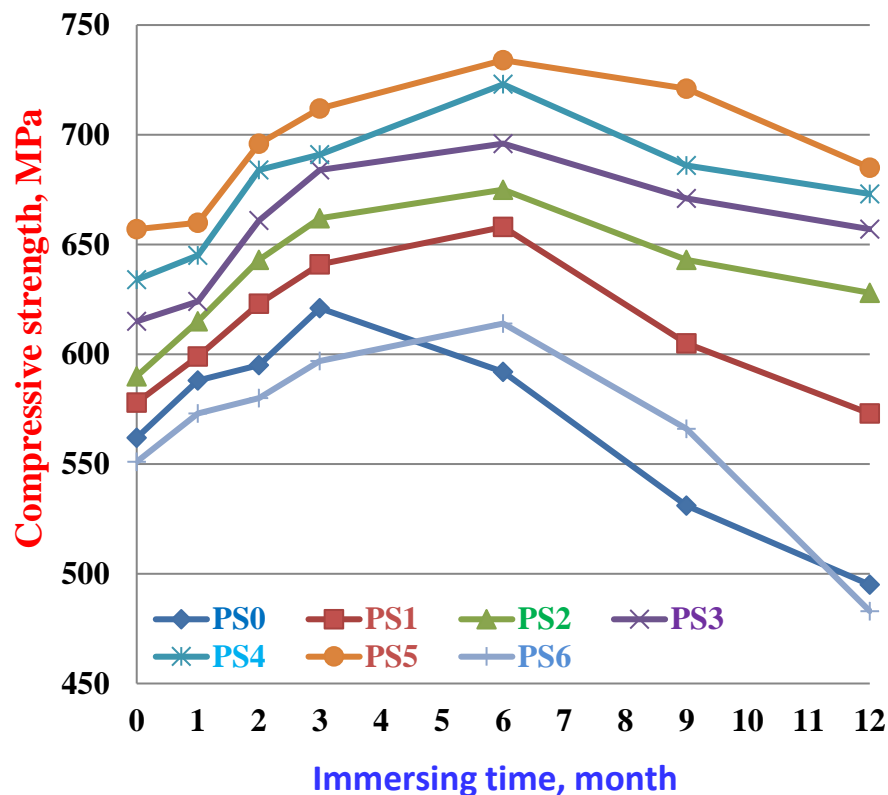


Fig. 4- Compressive strength of the various cement pastes containing saw dust ash immersed in sulfate solution up to 12 months.

The compressive strength of the hardened cement pastes containing SDA (PS1-PS6) immersed in 4 % MgSO_4 solution increased with immersing time up to 6 months, and then decreased onward up to 12 months. The increase of compressive strength is

essentially attributed to the deficiency of the OPC phases, particularly C_3A , which are very sensitive to sulfate ions and are easily degraded by these ions and was compensated with the pozzolanic mineral admixtures (SDA) [37]. This is because the silica nanoparticles from SDA particles are difficult to disintegrate easily by the SO_4^{2-} ions due to its high amorphous glassy structure. Moreover, the slight activation of the OPC and SDA, in addition to the good dispersion and the good compaction of these pozzolanic materials due to its filling action, and the relatively lower amount of free lime due to its consumption by the pozzolanic materials to form CSH made the rate of sulfate ions penetration into the hardened cement pastes was the minimum because it reduces the permeability [38]. Adámek and Juránková [39] concluded a similar result that FA could be created soft parts of fresh cement pastes which improve its plasticity, infill gaps among ingredients, compress the contact zone among the different grains and the matured cement. Also, the hydraulic features of FA especially contribute to the mechanical strength enlargement. Also, the decrease of permeability to liquids seemed to improve the durability. As a result, the compressive strength tends to increase [37,40,41]. The same trend was displayed with all cement pastes incorporated SDA but exhibiting various values of compressive strength.

The decrease of compressive strength may be due to the release of water from the cement pastes into the solution as a result of the detrimental reactions. This usually accompanied with a consequent increase in the porosity and permeability. This tends to speed up the action effect of sulfate ions. Therefore, a strength loss would be expected [4, 9, 32]. $MgSO_4$ was always ionized to produce $Mg(OH)_2$. This is basic, and so the SO_4^{2-} ions are more effective particularly in older immersing times, which always dissociated into or through the matrix causing a more pore structure. This led to an increase in both porosity and permeability followed by a more strength loss [15, 42, 43]. Also, the SDA often hydrated inside the pastes by reacting with $Ca(OH)_2$. This seemed to decrease the pH value of the pastes, and therefore the cement pastes are accordingly attacked leading to a decrease in the compressive strength [9,15]. Consequently, the PS6 is the most and strongest negatively effective with SO_4^{2-} ions, while the PS1-PS5 are the least effective on the resistance and durability of the cement pastes. This was agreed perfectly with that suggested by many investigators [2, 25-28].

Though the different sulfate ions attacked the cement pastes aggressively after the 9th month of immersion, no visual cracks or any signs of deterioration on the lateral surfaces or edges could be seen by the naked eyes. Figure 5 illustrates that at the end of the 12th month of immersion, the SO_4^{2-} ions only precipitated and/or adsorbed on the external surfaces of the hardened cement pastes, and some horizontal minor cracks and little deformation could be seen on the edges of specimens, where large cracks and swelling were noted with cement pastes of the blank (PS0). Consequently, it could be concluded that PS5 has the best degree of resistance, while PS0 and PS6 recorded the least.



Fig. 5-The OPC cement pastes incorporated SDA (PS5) and immersed in 4 % $MgSO_4$ up to 12 months.

3.3. Combined water content

The combined water contents of the various cement pastes with and without saw dust ash (PS0-PS6) immersed in 4 % $MgSO_4$ solution up to 12 months are plotted as a function of immersing time in Fig. 6. Generally, the combined water contents of the various cement pastes (PS0-PS6) slightly increased with immersing time up to 12 months. This is mainly due to the continual formation of hydration products, particularly CSH and/or CAH gel, ettringite and gypsum and may be others [7]. The increase of combined water contents during the first 3-6 months of immersion does not only due to normal hydration of the OPC, but also due to the pozzolanic reactivity of the SDA with the release of $Ca(OH)_2$ from the hydration of the cement. In case of the blank batch (PS0), the continuous increase of combined water contents from 6 up to 12 months is due to the formation of more and more ettringite and/or gypsum phases resulting from the deleterious reactions with SO_4^{2-} ions with the different ingredients [5,7,18]. However, the combined water contents of the various cement pastes are lower than those of the pure OPC pastes at all immersing ages (Fig. 5). This is mainly attributed to the decrease of the main hydrating or cementitious material of the OPC by 5-30 mass %,

which is responsible for the hydration. In addition, the OPC phases are very sensitive to SO_4^{2-} ions, and therefore it reacted with them more higher than other materials. Also, it is noticed that all cement pastes displayed the same trend in MgSO_4 , but with slight differences. The cement composite containing 30 mass% SDA (PS6) recorded the lowest values of combined water contents. This may be due to the higher ratio of SDA that is leading to the formation of lower amounts of hydration products comparing with other blended cement pastes. Furthermore, the SO_4^{2-} ions have no effect on the SDA. So, the amount of formed hydration products is lower [2-4, 19, 20].

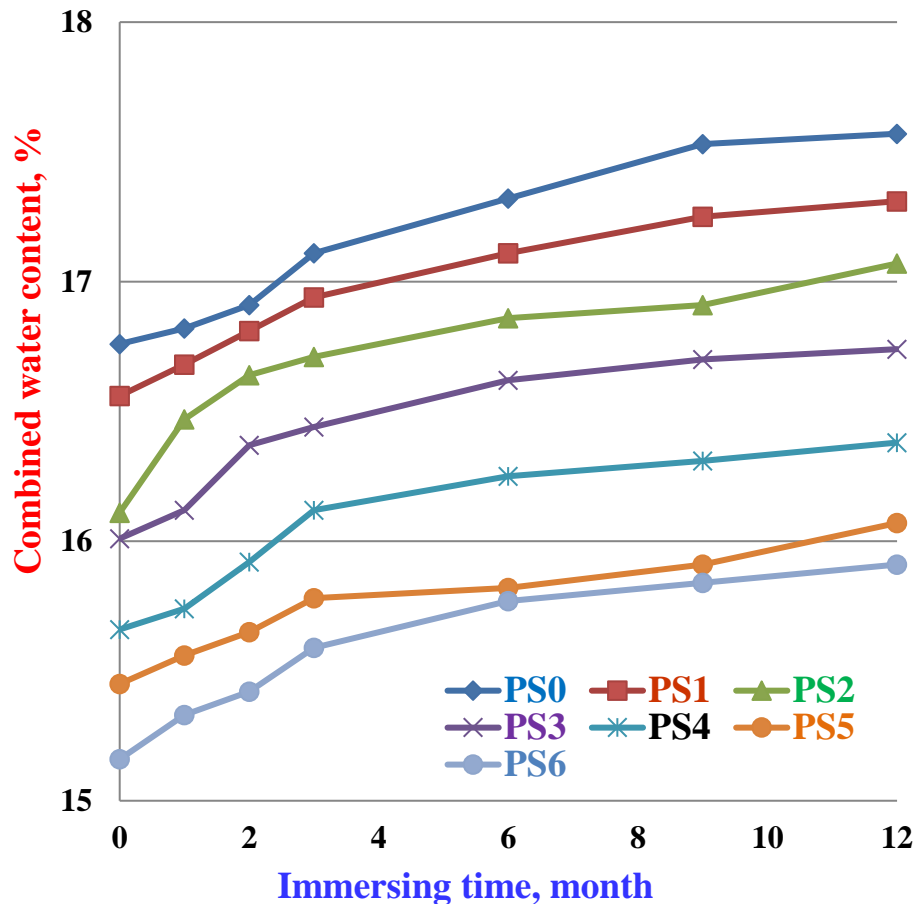


Fig. 6- Combined water contents of the various cement pastes containing saw dust ash immersed in 4 % sulfate solution up to 12 months.

3.3. Total sulfate content

The total sulfate contents of the various cement pastes with and without SDA (PS0-PS6) immersed in 4 % sulfate solution up to 12 months are graphically plotted as a function of immersing time in Fig. 7. The total sulfate contents increased with the increase of immersing time up to 12 months. This is due to the migration of SO_4^{2-} ions into the cement pastes, which in turn reacted with aluminate, ferrite and $\text{Ca}(\text{OH})_2$ to form calcium sulfoaluminate hydrates (ettringite) and/or monosulfate hydrate [7,16,19]. The cement composite containing 30 wt. % SDA (PS5) exhibited the lower values of total sulfate than those of other blended cement pastes nearly at all immersing times, whereas that of the blank (PS0) recorded the higher values. It is clear that the values of total sulfate contents of all cement blends are much lower than those of the blank (PS0) due to the formation of a dense white coat of SO_4^{2-} ions on the lateral surfaces of immersed samples. This coat hinders the penetration and diffusion of SO_4^{2-} ions into the cement pastes. This in turn slows down the action effect of the SO_4^{2-} ions [5,7], in addition to other factors as the good dispersion and good compaction of samples during moulding. It is also clear that the values of total sulfate contents of PS1-PS6 are close to each other and are much lower than those of the blank (PS0), i.e. the pure OPC pastes (PS0) are so sensitive to SO_4^{2-} ions that they are highly affected with these ions [4,5,7,16].

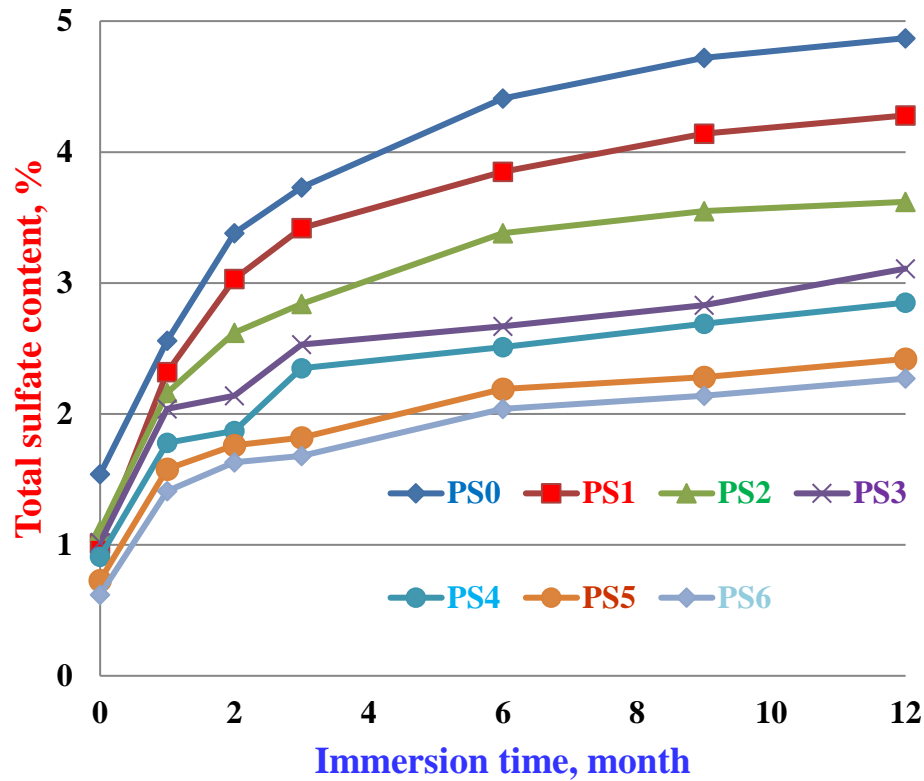
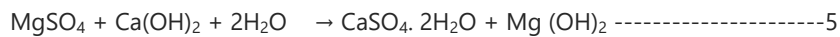


Fig. 7- Total sulfate contents of the various cement pastes containing saw dust ash immersed in 4 % sulfate solution up to 12 months.

4. DISCUSSION

During the early ages of immersion in MgSO_4 solution, it directly reacted with the released $\text{Ca}(\text{OH})_2$ from the normal hydration to produce gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and brucite, $\text{Mg}(\text{OH})_2$ or magnesium hydroxide as follows:



The formed $\text{Mg}(\text{OH})_2$ is insoluble and always decreases the pH-value of the hydrates. So, the lower pH-value de-stabilizes either CSH or ettringite. Moreover, there is no additional ettringite is formed because the MgSO_4 ions further react with CSH to form additional gypsum, $\text{Mg}(\text{OH})_2$ and SiO_2 -gel [44,45] as follows:



The latter phase (SiO_2 -gel) has less or no cementing properties than CSH. The CSH further hydrates to liberate $\text{Ca}(\text{OH})_2$ to re-increase the pH-value and establish its equilibrium. But, the liberated lime instead of re-increasing the pH-value, it reacts with MgSO_4 ions to form additional $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ and $\text{Mg}(\text{OH})_2$, and so on. Consequently, the $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ and $\text{Mg}(\text{OH})_2$ contents increased gradually, whereas the CSH lost its cementing properties leading to further deleterious action. Furthermore, the $\text{Mg}(\text{OH})_2$ reacted with the SiO_2 -gel to form MSH as follows:



The resulting product (MSH) is non-cementitious and when it is formed will cause softening or dusting of the cement pastes. Hence, the damaging effect is mainly due to the formation of high amounts of gypsum and Sodium hydroxide, and finally MSH [46-48].

5. CONCLUSION

The hardened cement pastes containing SDA (PS1-PS5) showed the higher resistance against 4 % SO_4^{2-} ions up to 6 months when compared with those of the corresponding pure OPC pastes (PS0). The water of consistency of cement batches was increased with SDA content, while setting times were decreased. The compressive strength of the hardened cement pastes of the (PS1-PS5) are in a good agreement with each other, where they can withstand for six months in 4 % MgSO_4 , whereas those of the OPC pastes could not withstand more than three months in the sulfate media. The most and strongest effect of SO_4^{2-} ions, started to occur after six months. The combined water as well as total sulfate contents increased with immersing time up to 12 months, but decreased with SDA content. The same trend was displayed by all cement batches. Consequently, the degree of damage was in the descending order: PS5 > PS4 > PS3 > PS2 > PS1 > PS0 > PS6. The cement batch (PS5) containing 25 wt. % SDA achieved the best resistance against sulfate solution, whereas that of PS6 exhibited the lowest. As it is well seen from the results, the OPC/ SDA cement composites are more resistant to sulfate solution than the pure OPC cement.

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Conflicts of Interest: The author declares no conflict of interest.

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