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Microstructure Study for Effect of Local Admixture in Concrete Durability

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Abstract: Concrete permeability plays the main role in concrete durability because of the entry of harmful substances such as chlorides, sulfates, carbon dioxide (CO2), oxygen (O2). Concrete permeability is affected by factors such as size and proportion of aggregate, type, and quantity of cement, water-cement ratio, type and dose of admixture used, which help concrete have a high life. This research aims to investigate the effect of local admixture prepared to improve concrete properties, especially reducing the permeability of concrete using its proven effectiveness in salt attack. This research studies the effect of this admixture on concrete microstructure for several concrete specimens. This chemical admixture, which contains calcium oxide (CaO), chloride (Cl), and others, helps obtain a good performance of concrete. Adding calcium oxide (CaO) plays an important role in improving the physicochemical properties of concrete. An increase in the compressive strength was observed for all specimens. Incorporating additives without CaO to concrete has been shown to decrease its strength. In the present study, concrete showed higher compressive strength than other concrete, which has not contain this admixture which may be explained by the larger quantity of CaO in the cement.

Keywords: concrete, microstructure, admixtures, durability, scanning electron microscope.

局部外加劑對混凝土耐久性影響的微觀結構研究

摘要：由於氯化物、硫酸鹽、二氧化碳、氧氣等有害物質的進入，混凝土的滲透性對混凝土的耐久性起著主要作用。混凝土的滲透性受骨料的大小和比例、水泥的種類和數量、水灰比、外加劑的種類和劑量等因素的影響，有助於混凝土具有較高的壽命。本研究旨在調查為改善混凝土性能而製備的局部外加劑的效果，尤其是利用其在鹽侵蝕中已證明的有效性來降低混凝土的滲透性。本研究研究了這種外加劑對幾個混凝土試樣的混凝土微觀結構的影響。這種含有氧化鈣、氯化物等的化學外加劑有助於獲得良好的混凝土性能。添加氧化鈣對改善混凝土的理化性能具有重要作用。觀察到所有試樣的抗壓強度增加。已證明在混凝土中加入不含氧化鈣的添加劑會降低其強度。在本研究中，混凝土顯示出比其他混凝土更高的抗壓強度，而其他混凝土沒有包含這種外加劑，這可能是由於水泥中的氧化鈣含量較大。

关键词：混凝土、微觀結構、外加劑、耐久性、掃描電子顯微鏡。

1. Introduction

Concrete is the main element of many infrastructures worldwide, so it needs to have the highest quality, strength, and durability. To get it is necessary to take a Microstructural study on concrete. Microstructure study on concrete is a unique technique to find out the features of concrete and know the material of its components. There are other materials than water, aggregates, or cement. Admixtures are one of them. Admixtures of concrete are used to improve the properties of concrete. It increases workability or reduced water content and improves resistance to weather and chemical attacks. During winter, calcium chloride ($CaCl_2$) can accelerate the strength development in mass concrete. Alternatively, permeability-reducing admixture has to maintain the amount of water inside the concrete. So, it is possible to modify the properties of concrete to get workability, water reduction, dispersion and air-entrainment, impermeability, and durability factors.

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Scientists always strive to improve and develop the properties of concrete for its ability to be used in various fields. One of these methods is adding chemical admixtures to concrete to obtain long-life and economical concrete. Chemical admixtures having CaO on their components play a major role during the interaction of water and cement (Hydration). In [1], the authors noticed that higher temperatures increase cement hydration. Calcium oxide cement can be used effectively to seal concrete pores and retain mixture components. CaO improves the setting time of cement, from about 100 to 380 minutes in their case, which is beneficial for cement application. In [2], the author said that C3S is consists of C_2S plus an additional mole of CaO. During the hydration of C_3S, the extra CaO must also effectively hydrate, releasing heat (1135.5 kJ/kg CaO) More specifically, if the hydration products of C_3S and C_2S are the same, the heat of hydration of C_3S should equal the sum of the heats of reaction for C_3S from C_2S and CaO, the heat of hydration of C_2S, and heat of hydration of CaO.

$C_2S + CaO > C_3S + 13.4$ kJ/mole

$C_2S + 2H_2O > CaO.SiO_2.H_2O + Ca(OH)_2 + 45$ kJ/mole

$CaO + H_2O > Ca(OH)_2 + 63.7$ kJ/mole

Therefore, theoretically, the total heat of hydration for C_3S is (13.4 + 45 + 63.7) = 122.1 kJ/mole or 534.6kJ/kg C_3S, which agrees with the experimentally obtained values, which incidentally range from 504 to 530 kJ/kg C_3S [2].

1.1. Microstructure of Concrete

In [3], the authors mentioned that concrete under a microscope shows its microstructure, consisting of three components. These components are cement paste, pore structure, and interfacial transition zone (ITZ) between the cement paste and aggregates. As for aggregates, it represents the largest proportion of the volume of concrete, as it occupies about 60-70% of the total volume of the concrete mass. Moreover, aggregate grains are held together by the cement paste that covers the surface area of the aggregate. As for the cement paste, it represents 30-40% of the concrete volume.

The purpose of the cement paste is the cohesion between the grains of aggregate and cohesion between concrete and rebar. It acts as filler as it fills the spaces between the grains of aggregate and facilitates the mixing and pouring process of concrete. As for the voids and Air trapped where It improves operability, they represent 3-7% of the volume of concrete. In some cases, high-compacted concrete can be obtained by adding other materials to the mixture as admixture materials such as floors and walls of water tanks and swimming pools, all shown in Fig. 1.

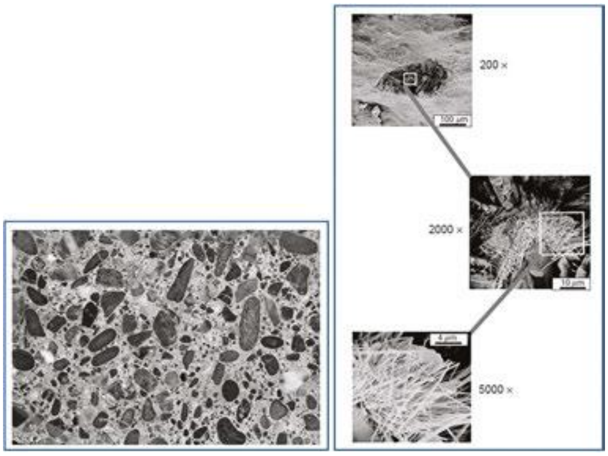


Fig. 1 Left: polished section of concrete specimen. Right: microstructure of a hydrated cement paste

The concrete must improve these components. It leads to enhance mechanical strength and durability of concrete. Cement consists of silica (SiO_2), its symbol (S), alumina (Al_2O_3), its symbol (A), lime (CaO), its symbol (C), and iron oxide (Fe_2O_3) its symbol (F). The main compounds of ordinary Portland cement are tri-calcium silicate (C_3S), di-calcium silicate (C_2S), tri-calcium aluminate (C3A), and tetra-calcium aluminoferrite (C_4AF), as shown in Fig. 2).

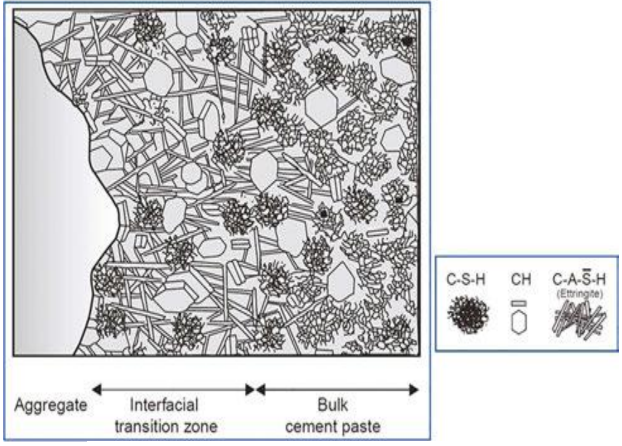


Fig. 2 A Schematic diagram of ITZ in concrete

Many experiments and research have been done to investigate the influence of different chemical admixtures and mineral admixtures on concrete properties when added to the concrete mixture in different ratios in concrete. Improve workability, durability, permeability setting time, and resist the concrete from Sulfate salts.

[4] discussed the effect of SO2 on the permeability of water in concrete. It was found that SiO2 can improve the microstructure of concrete and water permeability resistant capacity. They were also able to conclude that the mechanism can be reduced to the fact that the ability of concrete to resist water permeability is affected by the presence of a hexagonal $Ca(OH)_2$ crystal, which is located in the intermediate transfer zone (ITZ) between the aggregate and the binder paste matrix. Nano-SiO2 has a very high activity due to its

galactic surface area. Nano-SiO_2 reacts with $Ca(OH)_2$ crystals rapidly, producing a C–S–H gel. The size and quantity of the calcium hydroxide crystals are reduced. The C–S-H gel fills the voids to improve density from the intermediate transition zone (ITZ) and the binding paste matrix. The study [4] found that 70% of hydration products are C–S–H gel. The average diameter of the C–S–H gel is approximately 10 nm. The nano-SiO2 particles can fill the voids of the C–S–H gel structure, making the binding paste matrix denser. A nano- SiO_2 particle can act as a nucleus to tightly bond with C–H gel particles in the C–S–H gel structure. Thus, the integration and stability of the hydration product structure are improved, and long-term mechanical properties and durability of concrete are expected to be increased.

In [5], the authors discuss the effect of superplasticizer and molarity of (NaOH) solution in concrete. It works on self-compacting geopolymer concrete's workability, microstructure, and compressive strength (SCGC).

SCGC is an improved way of executing concrete, and it does not require compaction made by the complete elimination of ordinary PC content. SCGC was activated by combinations of sodium hydroxide (NaOH) and sodium silicate solutions (Na_2SiO_3) and by incorporating superplasticizer for self-compact ability.

Also, [6] studied and obtained that when the weather becomes lower than 5°C, some special measures should be taken to prevent a decrease in the rate of hydration and fresh concrete from freezing.

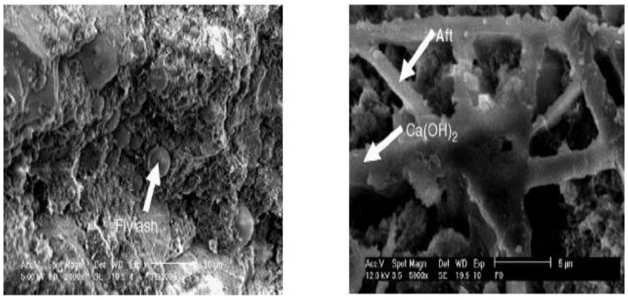
Also, they investigated the behavior of fresh and hardened concrete, which contained calcium nitrate at different curing with low temperatures less than freezing temperature of the water, and compared the results with both control samples. For this reason, calcium nitrate is used at the level of 6% by weight of cement dosage in mixes. Calcium nitrate increased the compressive strength of concrete (between 48–964, 50–721, 29–393 and 24–183%, for − 5°C, − 10°C, − 15°C and − 20°C, respectively, when compared to mixes without antifreeze admixtures. They showed that using calcium nitrate as an antifreeze admixture in concrete technology in cold weather concreting without additional precautions is possible.

[7] showed the effect of mineral admixtures on concrete. Mineral admixtures are materials added to concrete to get specific engineering properties. It was added admixture for economic and environmental benefits. Unlike chemical admixtures, they are used in relatively large amounts to replace cement and/or fine aggregate in concrete. Recently, many industrial waste by-products as admixtures such as fly ash, silica fume, red mud, and slag were added.

There are different types of mineral admixture:

1. Fly ash
2. Silica fume
3. Pozzolan
4. Ground granulated blast furnace slag
5. Cutting stone and Tiles waste

[8] reviewed that Calcium hydroxide (CH_n) is the only hydration product with well-defined stoichiometry and crystal structure and normally forms as massive, relatively pure crystals with a euhedral hexagonal habit. The calcium silicate hydrate in cement paste is a gel that shows no long-range crystallinity. Its composition which uncertain (and possibly variable) with a C/S ratio of about 1.7 to 2.0; consequently, it is usually written as C-S-H. The short-range order of this phase is probably related to the layer structure of the crystalline calcium silicate hydrates-1.4 nm tobermorite (C5S6H9) and jennite (C9S6H11). This phase appears to adopt a wide range of morphologists; some based on thin sheets, which may give fibrillar or honeycomb structures at early ages, others with a more compact structure forming at later ages.). In addition to possible substitution of aluminum by iron, hydroxide, carbonate, or chloride ions may replace the sulfate. These phases have hexagonal plate morphology. Fig. 3 shows the microstructure for normal concrete and the effect of admixture in concrete.



Microstructure of normal concrete at curing age of 28 days. Microstructure of normal concrete at curing age of 180 days.

Fig. 3 Microstructure for normal concrete (NC)

[9] used tire rubber as aggregate, and cement replacements with different percentages mechanical tests included compressive, tensile strength, and modulus of elasticity was performed. The durability tests included permeability and water absorption. The results showed that no major changes in concrete characteristics would occur with up to 5% replacement in each set. However, with a further increase in replacement ratios, considerable performance reduction was observed.

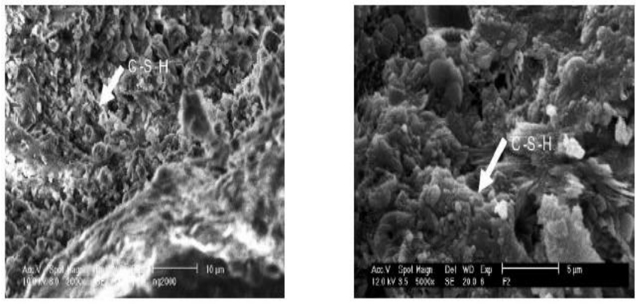
In [10], the authors mixed tiles, glass, wood, and clay bricks to replace 10% of the weight of aggregate and measured Various properties of the samples such as density, compressive strength, tensile strength, water absorption, corrosion resistance, and toughness and found the compressibility resistance of the samples after 28 days ranged between 71.5% and 82.3% of the sample base mass.

In [11], the authors modified the properties of concrete using mineral admixture which used ground granulated blast furnace slag GGBS. (GGBS) with the average particle size of 4–6 μM was used as a replacement mineral admixture (10%) to ordinary Portland cement (PC) while calcium nitrate Ca $(NO_3)_2$was used as a chemical admixture at (2%) amount of all cementitious material, in the preparation of concrete. They found that Ultrafine GGBS decreases the value of workability and water absorption and increases the compressive strength of concrete by 18% and the percentage of bond strength of the steel bar is 45%. Adding calcium nitrate further decreased water absorption of the concrete but improved the workability, compressive strength to 32%, and bond strength to 131%; on the other side, corrosion currents in GGBS and nitrate-modified concrete decreased. All these results show that ultrafine GGBS and calcium nitrate $Ca(NO_3)_2$ as admixtures improve the mechanical properties of concrete and reduce the corrosion of rebar. They obtained that shown Fig. 4:

The best improvement of mechanical and chemical properties of concrete has been obtained from condensed silica fume (CSF);

Modifying the microstructure of cement paste enables obtaining great properties of concrete for particular application conditions.

Resistance of aggressive was the highest in OPC mortar and lowered in condensed silica fume (CSF) and Fly Ash (FA).



Microstructure of nano-SiO_2 concrete at curing age of 28 days.

Microstructure of nano-SiO_2 concrete at curing age of 180 days.

Fig. 4 Microstructure for concrete with the admixture

[12 studied the performance of recycled aggregate concrete, which is prepared with mineral admixture by-products, such as palm oil fuel ash (POFA), rice husk ash (RHA), and palm oil clinker powder (POCP). Their all were alternatives for SCMs. It was tested the effect of hydrochloric acid (HCl) and magnesium sulfate (MgSO4) on concrete. It was measured the change in mass compressive strength. Also, in microstructural analysis, adding RHA, POFA, and POCP to concrete reduced its deterioration up to 30%. Loss in compressive strength decreased when the specimens were exposed to HCl solution. In Fig. 5, the case of the MgSO4 attack showed less propagation of micro-cracks caused by expansive ettringite. The results proved that RHA is more effective than POFA and POCP.

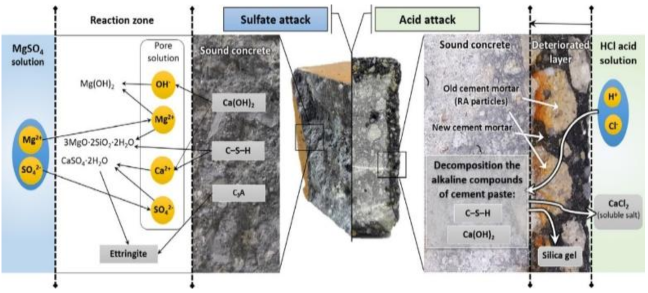


Fig. 5 Scanning electron microscopy image of specimens of concrete

In [13], the authors noticed that concrete composite structures could be divided into three phases. The first stage is called cement paste, the second stage is coarse aggregate, and the third stage is an (ITZ) between the aggregate and cement paste. After scanning electron microscopy, it was observed, as shown in Fig. 6, that the microstructure of M-0.4 % GF after heating at 22, 200, 400, and 600℃ effect may be attributed to PPF had been melted at 170C, which can result in interconnected micro cracks. These micro-cracks stresses were generated by the expansion of the polypropylene (10 %) during the melting.

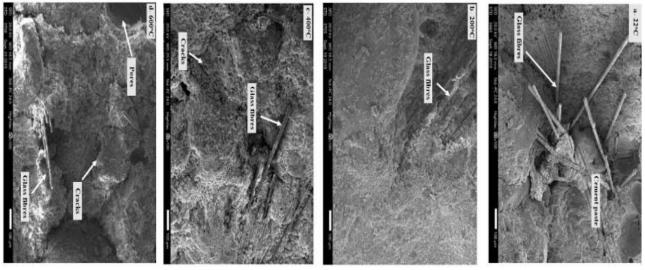


Fig. 6 SEM micrograph of M-0.4% and effects of the elevated temperatures on ITZ between GF and cement paste

There is good bonding between fibers GF in LWC and cement paste at heating from 22℃ to 400 C.LWC's vulnerability to cracks, and the fiber was exposed paste interface at 600℃. Fig. 7 shows that at 22℃, the aggregate has a porous interior structure. At 200℃, the cement pastes of both mixes shrank, and micro-cracks started to appear and weakened ITZ, as shown in Fig. 7. At 600℃, it was found that shrinkages and micro-cracks increased, which was increasing with increased temperatures. As shown in Fig. 7 at 400℃, it was noticed that the calcium hydroxide (CH) was separated into lime and water. At 200℃, it converted to small particles hydrates, and at 400℃, it transformed into CSH gel. These cracks spread after a rising temperature of more than 400℃.

In [13], the authors noticed, as shown in Fig. 8 from the SEM image, that after 7 days, it is observed that some particles with spherical shapes appeared less homogeneous with structure. It happened because some particles did not fully react at an early age ported as mentioned in the literature [14, 15].

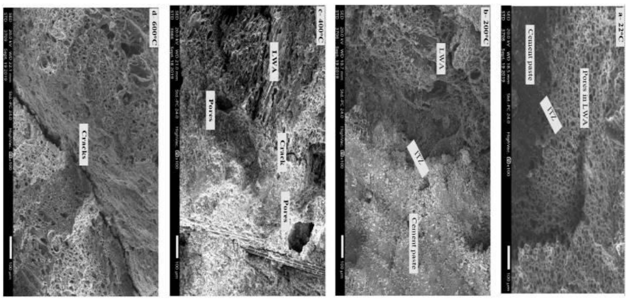


Fig. 7 SEM micrograph for effects of the elevated temperatures on ITZ between aggregates and cement

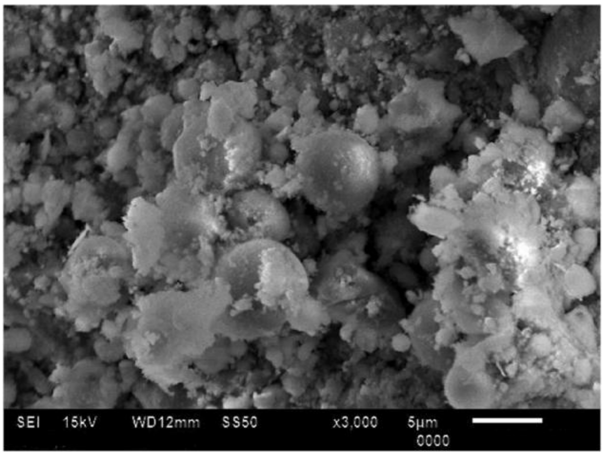


Fig. 8 SEM image of 7.5LMSF2 at 7 days

Fig. 9 shows mechanical properties achieved at 28 days; it appeared homogeneous and compact. High resistance compressive strength was achieved on this sample. It noticed that there are all particles of fly ash were reacted. This can be explained by the unreacted particles observed at the age of 7 days in contact with free silica and calcium from silica fume, and lime causes the formation of NASH and CASH. It was very beneficial that use lime and silica fume as additives in the fly ash-based geopolymer. It was noticed that compressive strength was high at 7 and 28 days for 10LMSF3, 7.5LMSF1, 7.5LMSF2, and 7.5LMSF3 [16].

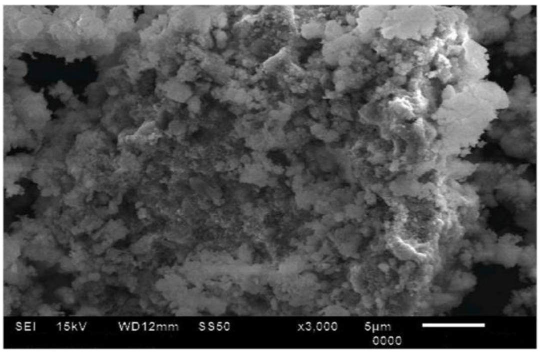


Fig. 9 SEM image of 7.5LMSF2 at 28 days

[17] mentioned that many fine particles resulting from the crushing processes were the reason for the lower pore volume of reused recycled concrete aggregates (RRAC) compared with recycled aggregate concrete (RAC). The number of finer particles in RRCA is more than the number of particles in recycled concrete aggregate (RCA), as shown in Fig. 10.

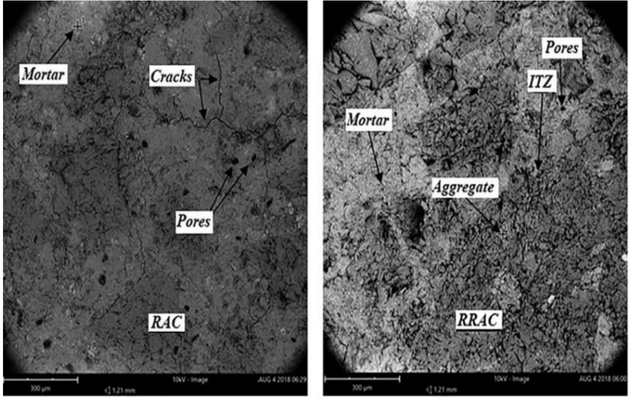


Fig. 10 SEM images of RAC and RRAC

2. Materials and Methods

2.1. Samples Preparation

Two groups of microstructure concrete specimens were prepared. The first group was using an organic admixture (type: BM 2010) fabricated in the laboratory and consisted of wastes from petroleum industries and silica fume and Wastes of coke factory and pure water [18]. The second group was the control sample (without admixture). Each group consisted of 60 concrete cubes with a dimension of 15x15x15 cm.

2.2. Mixing Admixture

It was added admixture doses with different percentages, and as shown in Fig. 11, there is a reduction in the value of Fc from 390.5 kg/cm^2 to 353 kg/cm^2 with increasing of admixture dose percentage from 0 to 0.5%. Then it was obtained that increasing in Fc of concrete regularly until optimum dose percentage (Admixture dose percentage = 0.9%, Fc = 477 Kg/cm^2). After that, a big downturn resulted from increasing admixture dose until admixture dose percentage 1.4%, which got Fc = 273 kg/cm^2.

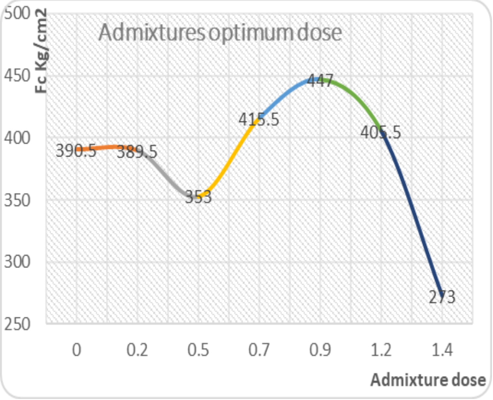


Fig. 11 Admixture optimum doses

2.3. Durability Test

All groups of cubes were tested under axial static compression load. 120 concrete cubes subjected to 25 cycles of durability test After 28 days hardening in drink water. Each cycle takes 4 days, as shown in Table 1.

Table 1 Cycle of durability test

NaCl 3% + Na2So4 6% concentration in salt solution	**1st day**
At 100℃	2nd day
At lab. temperature	3rd day
In drinking water	4th day

2.4. Materials

2.4.1. Cement

Portland cement, called Assiut cement, was used. Physical properties, chemical properties, and compressive strength were checked. The comparison was made with ESS No. 2421-1/2005 and ESS No. 4756-1/2009. The results shown in Tables 2, 3, and 4 were obtained.

Table 2 Physical properties of cement

Test		**Result**	**ESS No. 2421-1/2005**
Cement expansion (Le Chatelier)		1 mm	< 10 mm
Specific gravity		3.15	-
Setting time	Initial	165 minute	≥ 45 minutes

Table 3 Chemical properties of cement

Test	**Result**	**ESS No. 4756 – 1 /2009**
Loss on ignition	1.0 -2.0%	≤ 5%
Insoluble residue	0.3 % - 0.6%	≤ 5%
% of sulphate ions content (SO_3)	2.5 % - 3.0%	≤ 3.5%
% of chloride ions content (Cl)	0.02 – 0.04	≤ 0.1%

Table 4 Cement compressive strength

Test		**Result**	**ESS**
Compressive strength	2 days	20.7 N/mm^2	-
	28 days	42.7 N/mm^2	42.5 N/mm^2

2.4.2. Aggregate

Local natural gravel as coarse aggregate and local natural sand were used as fine aggregate. It was tested the physical, mechanical, and chemical properties of used Aggregated. It was compared with ESS and obtained results as shown in Table 5.

Table 5 physical, mechanical, and chemical properties of used aggregated

Property	**Gravel**	**ESS**	**Sand**	**ESS**
Volume weight (t/m^3)	1.75	-	1.73	-
Specific gravity	2.6	-	2.63	-
F. M.	6.29	-	2.51	-
% of clay & Fine	0.4	1.0%	1.9	≤ 3%
Crushing value %	13	≤ 30	---	-
Cl	0.01	≤ 0.04	0.04	≤ 0.06
So_4	0.013	≤ 0.4%	0.26	≤ 0.04
PH	7.1	-	7.4	-

Also, it was analyzed sieves for gravel and sand. The results were obtained as shown in Fig. 11 and 12.

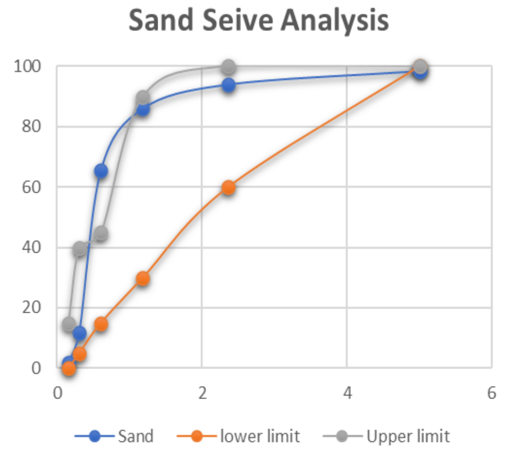


Fig. 12 Results of sieve sand analysis

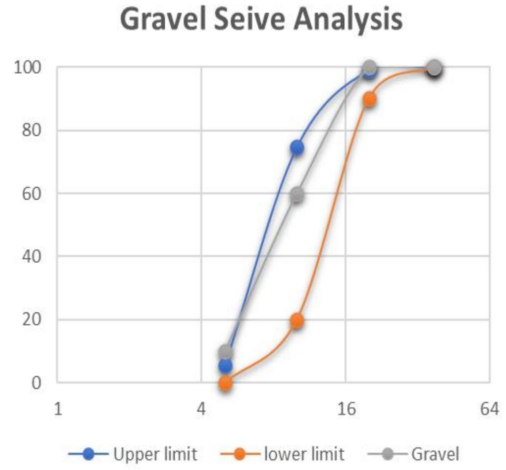


Fig. 13 Results of sieve gravel analysis

2.4.3. Concrete Mix

Compressive strength of cubic concrete mixes was used to produce normal strength 250 kg/cm^2. Table 6 shows the component of the concrete mixes.

Table 6 Details and properties of concrete mixes at optimum dose

Mix No.	**Cement (Kg/m^3)**	**Sand (Kg/m^3)**	**Coarse Agg. (Kg/m^3)**	**Additives**		**Water ($Liter/m^3$)**	**Slump cm**	**Fc kg/cm^2**
				Dose Kg/m^3	**Type**			
C1	400	650	1120	0	Control	220	7	255
C2	400	665	1130	3.6	BM 2010	207	7	332

2.4.4. Test Procedure

A testing machine tested all cubes under axial static compression load (Control 2000 KN). Each specimen was loaded axially and gradually, keeping the rate of loading constantly. The concrete specimens were tested under static axial compression loading after 28 & 128 days of hardening in freshwater, divided into 120 cubes into two groups. The first group of the concrete cubes (60 cubes) was hardened in freshwater. The retained cubes (60 cubes) were subjected to a durability test of 25 cycles after 28 days of hardening in freshwater. Each cycle consists of 4 days.

2.4.5. Physical and Microstructure Properties

The effect of Admixture (BM2010) on the porosities of normal strength (NSC) was investigated. Examining the spatial distribution and volume of the internal pores of the concrete is a key factor in understanding and modeling the transport phenomena influencing concrete's durability [19]. Hence, the microstructure of concrete has a remarkable influence on the other properties of concrete [20]. The influence of admixture (BM2010) on the concrete microstructure could be investigated by scanning electron microscope (SEM), which visually presented the porosities, microcracks, and the possible interactions between the aggregates and the mortar [21].

3. Discussion

The effect of admixture and its components on improving concrete microstructure is shown in Fig. 14 by reducing bores reducing water content. This local admixture improved resistance to weather, chemical attacks, dispersion, air-entrainment, and impermeability and durability factors. Chemical analysis of local admixture was shown in Table 7.

Table 7 Components of local admixture

Parameter	Description
Chloride	0.0132 % (wt./vol)
Sulfate	0.1416 % (wt./vol)
Calcium oxide	0.1624 % (wt./vol)
Aluminum oxide	0.0560 % (wt./vol)
Bulk Density at 24 °c	1.065
PH	6.8
% of solids	15.88

Using CaO was a major reason for the improvement of concrete, as it has better strength and less permeability, which keeps water inside the concrete mixture for a longer period. Closing the pores works to increase the resistance of the concrete to acids and sulfates that harm the components of the concrete and the reinforcing steel reinforced with it. Also, the volumetric reduction is about 10% of set cement.

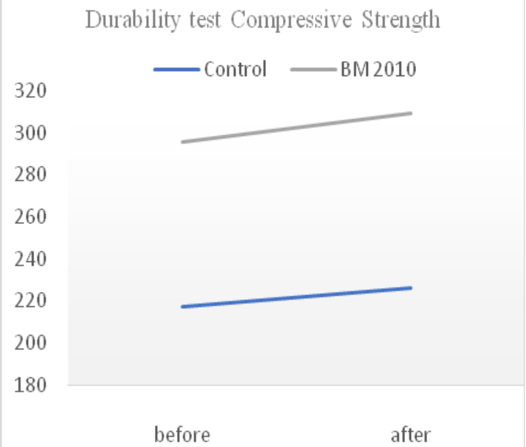
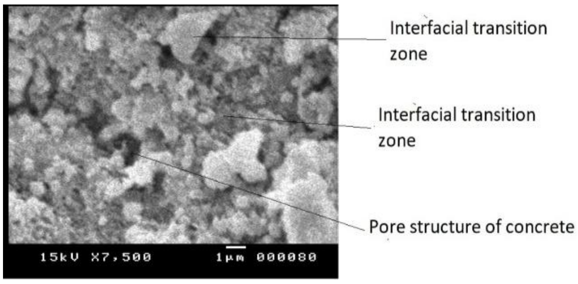


Fig. 14A Microstructure of concrete without the admixture



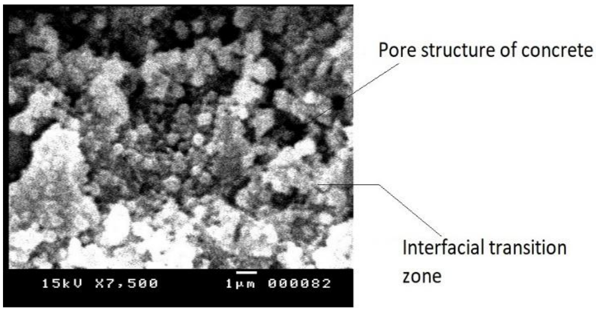


Fig. 14B Microstructure of concrete without the admixture

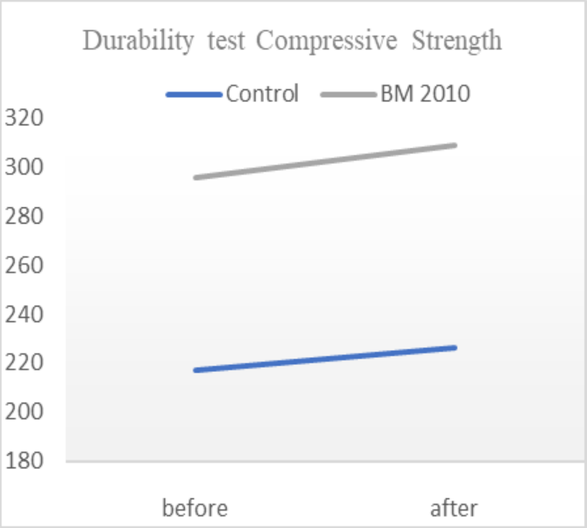


Fig. 15A FCU of cubes after the durability test

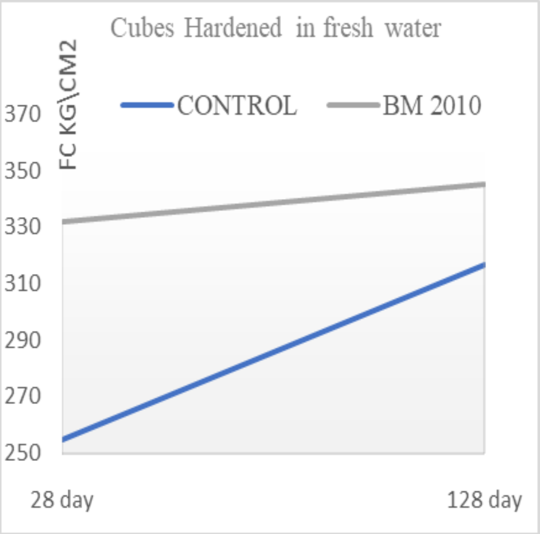


Fig. 15B FCU of cubes which harden in freshwater

Using Al_2O_3 works for early fixation and early strength. The aluminum hydroxide combines with calcium hydroxide and chlorine to form the chloro-aluminate complex. The formation of chloro-aluminate reacts with reinforced concrete immobilization of chloride ions penetrating through the concrete to the reinforcement. The formation of chloro-aluminates could lower the risk of corrosion on reinforced concrete.

The use of a locally manufactured admixture has greatly impacted the microstructure of concrete samples. The admixture served to fill the intersections inside the concrete and thus reduce the porosity, as evident from Fig. 14. This helps the concrete retain water for a longer period and thus gives greater compressive strength and more and more durability. Due to the effect of admixture in concrete in durability as shown in Fig. 15, it helped increase compressive strength, and it is shown from the curves that were found compressive strength of control cubic 217 kg/m2 and increased within 28 days to 296 kg/m^2. There is increasing in the durability of 30.2%, while compressive strength of BM2010 cubic 226 kg/m^2 increased within 28 days to 309 kg/m^2. There is increasing in the durability of 36.7%. There is a big effect of admixture to cubes that compressive strength for control in 28 days 255 Kg/m^2 and 128 days 316.6 Kg/m^2.

On the other hand, compressive strength for BM 2010 in 28 days 332 Kg/m^2 and 128 days 345.25 Kg/m^2, as shown in Fig. 15, is due to decreased pores in the concrete. When looking at the microstructure of BM2010, it is found that admixture fills these pores. The presence of an integrated mixture that is not subject to any cause of loss or attack from harmful substances is the most important factor in obtaining high-strength concrete.

4. Conclusion

Admixture BM2010 increases durability 36.7%, and increases harden in freshwater 24% for control and 5% for BM 2010, where fill the concrete pores and prevent losing water and decreasing permeability. Volumetric reduction of about 10% of set cement, where concrete resists harmful substances such as acids and sulfates, protects concrete components and reinforcing steel bars. It helped to reduce the risk of corrosion on reinforced concrete. Also, reduce the porosity obtained high strength and resist more and more compressive stresses. It gives the long life of the concrete and bears different ambient conditions.

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