

Effect of Newly Prepared Admixture on The Rheology and Early Hydration Characteristics of The Oil Well Cement Pastes.

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Abstract

Cement additives are typically used to modify the behavior of oil-well cement (OWC) and control its fluidity to resist the higher temperatures and pressures during the drilling process of the well. In this study, synthesis and characterization of a new aliphatic organic compound namely; acetone formaldehyde sulfonate (AFS) condensate are presented. The prepared compound (AFS) was characterized using FT-IR and by elemental analysis. The effect of different additions of this admixture on the physicochemical and mechanical characteristics of OWC pastes was investigated. The results obtained revealed that, addition of AFS to OWC pastes causes a notable improvement in both of the rheological and mechanical properties of the various hardened pastes. XRD and DTGA results obtained for the neat and admixed OWC pastes, indicate that the main hydration products formed are nearly amorphous calcium silicate hydrates (mainly as CSH-I and CSH-II), calcium sulphoaluminate hydrates (ettringite and monosulphate hydrates) and portlandite (CH).

Keywords: Oil well cement; Admixtures; Setting times; Rheological properties; Compressive strength.

1. Introduction

Among all operation being performed during oil or gas well drilling, oil-well cementing can certainly be known as one of the most critical operations in petroleum and gas industry. Therefore, it becomes one of the most interesting area of research for the last years. The main objectives of well cementing are to restrict movement of fluids between formations at different levels, support vertical and radial loads applied to casing which is placed in the drilled borehole, protect the casing from corrosion and sealing of abnormal pressure formations (1 - 3). Thus the main purpose of a primary cementing job is to provide effective zonal isolation for the life of the well so that oil and gas can be produced safely and economically (4). Oil well cementing is less tolerant to errors than conventional cementing works. If the cement does not provide a good seal, gas or liquid fluids can migrate to the surface and lead to work accidents or environmental problems. So, fully understanding of interfacial phenomena behind oil well cement procedures has scientific, economic and environmental importance (5). In cementing operation, Proper slurry design is critical to the success of a cementing job (6). So that appropriate cement slurry design for well cementing is a function of different parameters such as; the well bore geometry, casing hardware, formation integrity, drilling mud characteristics, presence of spacers, mixing

and pumping conditions. Therefore the rheological behavior, setting and thickening times of the cement slurries must be optimized with appropriate retarders and accelerators to achieve an effective wellbore cementing operation (7). The American Petroleum Institute Specification for materials and testing for well cements (API Specification 10A, 2002) has established nine special classes of cements (Class A – Class J). They are arranged according to the depths, to which they are placed, and the pressures and temperatures to which they are exposed (8). OWCs are classified into three grades based upon their C₃A (tricalcium aluminate) content: Ordinary (O), Moderate Sulphate Resistant (MSR), and High Sulphate Resistant (HSR). Each class is suitable for a certain range of well depth, temperature, pressure, and sulphate environments. Class A, Class G and Class H are the three most commonly used oil well cements (9). During the manufacture of Class G well cement, No additives other than calcium sulfate or water, or both, are interground or blended with the clinker. This product is intended for use as basic well cement. Available in moderate sulfate resistant (MSR) and high sulfate resistant (HSR) grades (10).

Over the last few decades, a wide variety of cement admixtures are currently available to enhance oil well cement slurry properties, to achieve successful placement and rapid compressive strength development for

adequate zonal isolation during the lifetime of the well (11). Early age and hardened properties of cement based systems are highly depended on the type and dosage of chemical admixtures used. The proper selection of chemical admixtures is mainly based on a trial and error procedure using tests such as the Marsh cone flow, mini slump test, and other rheological tests (7). The performance of chemical admixtures is strongly influenced by the chemical and physical properties of the cement. Most of the commercial chemical admixtures have been used with ordinary Portland cement and for general purpose use. Hence, the technical data sheets provided by the manufacturers are not generally applicable for oil well cementing. The interactions of OWC with different types of admixtures and the associated cement admixture compatibility at high temperature/high pressure are still largely unexplored (7). In the field of cement products, recent advances are related to the use of admixtures specially those of organic materials like polymers added in small quantities to modify and to enhance the properties of cement products (12). Thus, the protective characteristics of oil well cements may be controlled by the addition of polymeric additives. The use of specific polymeric nets has been highly effective in preventing destruction and increasing the internal cohesion of oil well cement packs (13, 14). Various studies have been published that deal with the effect of addition of admixtures on the physicochemical and rheological properties of cement pastes (15 - 24). The effect of molecular weight of sulphonated acetone-formaldehyde condensate (SAF) on the adsorption and dispersion properties in cementitious system was studied. Results showed that the molecular weight is the key factor for the dispersion property of SAF where that the SAF fraction with a higher molecular weight has larger adsorption amount, higher absolute value of zeta potential, and thicker adsorbed film, which induces better dispersibility of the corresponding cement paste. An adsorption model of SAF on cement surface is presented. The SAF fraction with a higher molecular weight is more likely to twist and be adsorbed on cement particles through "loop and tail" adsorption. On the contrary, SAF fraction with a lower molecular weight tends to be adsorbed on cement particles through "flat train" adsorption (25).

Finally, using several additives in cement can cause undesirable interactions (26). Although, very good recipes have been developed, problems of compatibility between

cement and additives have arisen (27). Therefore, intensive research in this area is a must (26, 27).

The aim of this study is to prepare a new type of aliphatic organic admixture and to investigate its effect on the physicochemical and mechanical properties of oil well cement pastes.

2. Experimental

Materials

The cement used in this investigation was oil well cement (OWC) which is moderate sulphate-resistant (MSR) Class G according to American Petroleum Institute (API) with a specific gravity of 3.15 g/cm³. This cement was supplied from (Dyckerhoff AG, Wiesbaden, Germany). Its chemical and mineral compositions are given in Table (1).

A new type of aliphatic organic admixtures namely acetone formaldehyde sulfonate (AFS) was synthesized.

The starting materials used for preparation of AFS are: acetone (100% purity), formalin solution (37 %), sodium metabisulfite (Na₂S₂O₅) and 10N NaOH solution.

Preparation of acetone formaldehyde sulfonate

The AFS resin was prepared from formaldehyde, acetone and sodium metabisulfite through a three-step reaction including sulfonation, low-temperature condensation and high-temperature condensation. The threesteps of preparation are as follows:

(i). Sulfonation

95 g (0.5 mole) sodium metabisulfite was dissolved in water in a jacketed reactor flask equipped with a baffle stirrer and a reflex condenser at 50 °C. The temperature of solution was maintained at 50 °C before the solution was clear. As soon as the solution became clear, the temperature was decreased to 40 °C and, then, 29.04 g (0.5 mole) acetone was added. Then the reaction was carried out at 40 °C for 1 hour.

(ii). Low-temperature condensation

100 ml (1mole) formaldehyde aqueous solution of 37% concentration was fed into the reactor through a dropping funnel. During the dropping process, the temperature of the reaction is automatically increased. In the process, the temperature was controlled at 60–65 °C. The reaction was continued at 60–65 °C for an additional 1 h when the feeding of formaldehyde was complete.

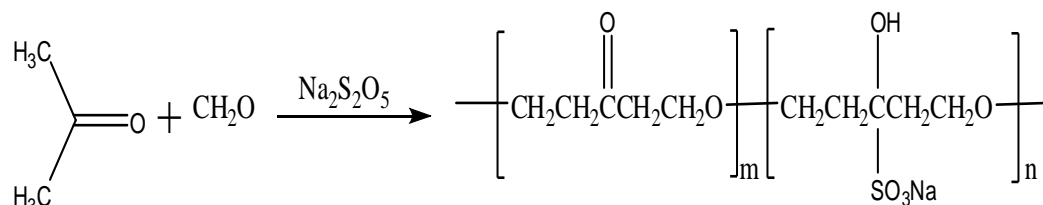
(iii). High-temperature condensation

The temperature was increased to 85 °C and the reaction was continued for 4 h. After cooling, the resin was prepared according to

above procedure which had a solid content of about 30% and the final pH of the red-brown solution was 13.

Table1. Chemical and mineral compositions of oil well cement class G (MSR).

Item	Wt (%)
Sulfur trioxide SO ₃	3.0
Magnesium oxide MgO	6.0
Loss on ignition L.O.I	3.0
Insoluble residue	0.75
Total alkali expressed as Na ₂ O	0.75
Mineral composition according to Bogue's calculation, %	
Tricalcium silicate (C ₃ S)	58-48
Di -calcium silicate (C ₂ S)	-
Tricalcium aluminate (C ₃ A)	0.8

Reaction equation of AFS formation:

Acetone formaldehyde sulfonate

Preparation of admixed OWC pastes

The hardened cement pastes were prepared from OWC using the water/cement (W/C) ratios of standard water of consistency with various AFS additions of 0.25%, 0.5%, 0.75% and 1.0% by weight of cement. The values of standard water of consistency and setting times for each paste are given in Table (2). The pastes were molded in 1 inch cubic molds, cured at 100% relative humidity up to 24 hrs, then cured under tap water for of 3, 7 and 28 days. For hydration kinetics, the applied time intervals are 0.5, 1, 2, 6 hrs and 1, 3, 7 and 28 days.

Techniques**Characterization and experimental testing of the aliphatic organic admixture FTIR spectra analysis**

FTIR spectra were obtained with a Perkin Elmer spectrometer (Model Paragon 500) using 15 scans summation and equipped with a universal ATR, with range 450–4000cm⁻¹ and resolution 4 cm⁻¹ was used to identify the molecular structure of polymers. The analysis was carried out on thin films obtained by grinding the polymer with potassium bromide (KBr).

Microanalysis of carbon, hydrogen, nitrogen, oxygen and sulfur

The elemental analysis of carbon, hydrogen, nitrogen, oxygen and sulfur were carried out using Perkin-Elmer 2408CHN analyzer at the micro analytical center, Cairo University, Giza, Egypt.

Characterization of the hardened cement pastes

At each time interval, three cubes of each hardened cement paste were subjected to compressive strength test and the average value was recorded. This was accomplished using a Tonindustrie machine (West Germany) for maximum load of 60 tons. The resulting crushed specimens of the hardened cement pastes were ground and the hydration reaction was stopped (28). The samples were then dried at 90 °C for 3 hrs in CO₂-free atmosphere.

Kinetics of hydration were studied by the determination of chemically combined water and free lime contents at different ages of hydration using the ground dried samples.

The chemically combined water content, (W_n, %), was determined from ignition loss at 1000 °C for 1 h. Duplicate measurements were carried out for each sample and the mean value was recorded.

$W_n, \% = [(W_o - W_i) / W_i] \times 100$; where, W_o = dried sample mass and W_i = ignited sample mass.

The free lime content, $CaO(\%)$, was determined by using the glycerol/ethanol extraction method and the mean value of the two independent determinations was recorded (28).

Phase composition of the formed hydrates was investigated for some selected hydrated samples using X-ray diffraction analysis (XRD) and differential thermogravimetric analysis (DTGA).

Rheological measurements of cement pastes

The performance of aliphatic organic admixtures in cement was tested by measuring the paste flow (mini-slump) test. For the determination of the paste flow, a "mini-slump" test according to DIN EN 1015 was utilized and carried out as follows: As indicated by its name, this method consists of carrying out a slump test on a small amount of cement paste. In a typical experiment, 300 g of cement were added within 1 min to the mixing water, then rested for 1 min without stirring and were again stirred for 2 min manually using a spoon. After the stirring, the cement paste was immediately poured into a Vicat cone (height 40 mm, top diameter 70 mm, bottom diameter 80mm) placed on a glass plate and the cone was vertically removed. The resulting spread of the paste was measured twice, the second measurement being in a 90° angle to the first and averaged to give the spread value.

3. Results and Discussion

Molecular structure identification of AFS admixture infrared spectroscopy

FTIR spectroscopy is a powerful tool for studying the physicochemical and conformational properties of polymers. The spectra of the conventional and the innovative polymers are almost identical and the peaks revealed could be attributed to the characteristic functional groups of the polymer such as the characteristic band observed from OH stretching at 3454 cm^{-1} . The other bands show: $C=O$ at 1715 cm^{-1} , 2939 cm^{-1} for stretching vibration of C-H groups. The broad band at 1197 cm^{-1} is related to stretching vibration of $S=O$ and $C-S$ of $R\text{SO}_3^-$ group. The main band characterizing the condensation process is the ether linkage, which appear at (1047 cm^{-1}) Fig. (1).

Elemental analysis

The results of elemental analysis of (AFS) are given in Table (3). The data of elemental analysis of polymer is in agreement with the molecular structure of the polymer.

Physicochemical characteristics of hardened pastes compressive strength

The results of compressive strength obtained for hardened neat OWC paste (Mix Ao) showed a relatively fast rate of increasing during the early stages of hydration up to 7 days; followed by a slightly slow rate of increasing compressive strength up to 28 days of hydration, Fig. (2). The previous results is attributed to the hydration of OWC leading to the formation and later accumulation of hydration products, namely as calcium silicate hydrates (CSH-I, CSH-II), which act as a good binding centers between the remaining unhydrated parts of cement grains. Also the slower rate of increase in the values of compressive strength during the later stage of hydration (from 7-28 days) is due to the fact that the initially formed hydration products shield the remaining unhydrated parts of cement grains leading to a slower rate of hydration during this period (29). However, the OWC hardened pastes admixed with 0.25%, 0.5%, 0.75% and 1.0% of AFS (Mixes AI-AIV) showed nearly the same trend as in case of blank (Mix Ao), but with a notable lower values of compressive strength especially during the early ages of hydration (up to 7 days), Fig.(2). After 28 days of hydration all the admixed pastes showed a comparable compressive strength values and/or higher values as compared to blank (Mix Ao). It is clear also from the results of Fig. (2) that, as the amount of AFS increases; the decrease in the values of compressive strength increases up to 7 days and the reverse occur after 28 days of hydration. The previous results can be attributed to the retardation effect of the AFS admixture only during the early ages of hydration.

Hydration Kinetics

The kinetics of hydration was studied by the determination of chemically combined water and free lime contents at each age of hydration.

The results of combined water content ($W_n, \%$) obtained for the different hardened OWC pastes (Mixes Ao - AIV) are shown in Fig. (3). Evidently, the W_n - values obtained for all tested specimens indicate a gradual continuous increase up to the final hydration ages studied (28 days). This result is attributed to the progress of the hydration process. The relatively high W_n - value during the initial stage of hydration (up to 24 hours) is attributed to the fast rate of direct interaction between water and cement grains. Also as shown in Fig. (3), the hardened pastes made of Mixes (AI - AIV) showed a notable lower values of chemically combined water as compared to those of the blank Mix (Ao) at all the investigated hydration stages; this result is attributed to the decrease in the values of the standard water of consistency from 0.264 in the neat OWC (Mix

Ao) to 0.257, 0.255, 0.2548 and 0.2534 by addition of 0.25, 0.50, 0.75 and 1 (wt. %) of AFS admixture, respectively, (Mixes AI - AIV).

The results of free lime contents (CaO, %) for the different hardened neat and admixed OWC pastes (Mixes Ao - AIV) indicate a gradual continuous increase with increasing hydration age up to 28 days, Fig. (4). This increase is attributed to the continuous liberation of free caoh as a result of progressive hydration reaction. As shown in

Fig.(4) all the admixed OWC hardened pastes (Mix AI - AIV) showed lower values of free lime content as compared to those of the neat OWC (Mix Ao) during all hydration ages studied; it is also noted that as the percentage addition of AFS admixture increases, the decreasing extent in the free lime content increases. The previous results can be attributed to the decrease in the values of standard water of consistency exerted by addition of AFS admixture to OWC.

Table2. Standard water of consistency and setting times of the neat OWC and OWC-AFS admixed pastes.

Mixes	Mix proportion (Wt., %)		W/C (%)	Initial setting time (min)	Final setting time (min)
	OWC	AFS			
Ao	100	-	0.2640	150	200
AI	100	0.25	0.2570	270	345
AII	100	0.50	0.2550	260	330
AIII	100	0.75	0.2548	195	250
AIV	100	1.00	0.2534	180	240

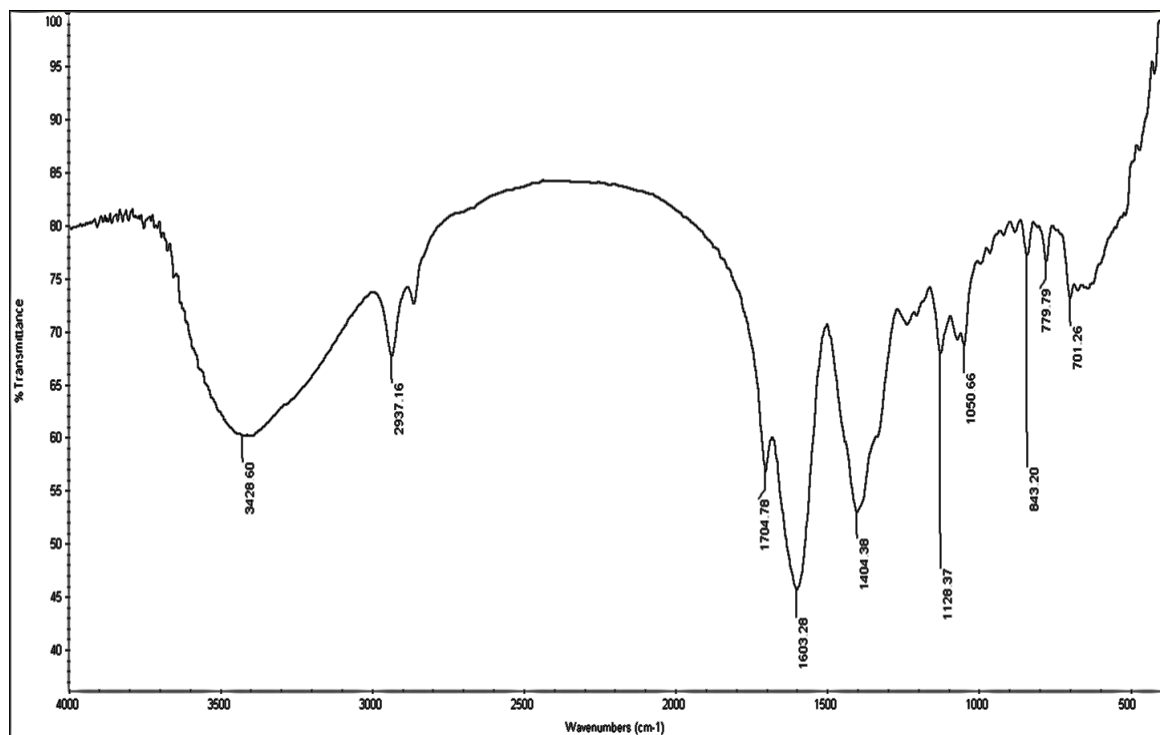


Fig 1. FT-IR (ATR) spectrum of AFS

Phase Composition

X-ray diffraction (XRD) analysis

X-ray diffractograms obtained for the hardened neat OWC paste (Mix Ao) as well as OWC admixed with 0.25% of AFS (Mix AI) after 6 hrs., 3 and 28 days are shown in Figs. (5) and (6), respectively. As shown in Fig. (5), the main hydration products identified from hydration of neat OWC are microcrystalline calcium silicate

hydrates (CSH), calcium aluminosilicate hydrates (CASH) and portlandite (CH); the peak characteristics for unhydrated parts of C_3S grains could also be distinguished. The peaks characteristic for minor amounts of calcium carbonate are also appeared with low intensities. The results of Fig.(5) indicate that the intensities of the peaks characterizing C_3S (the main constituent of anhydrous oil well cement)

decrease with increasing age of hydration; meanwhile, the intensities of the peaks characterizing the hydration products, namely calcium silicate hydrates (CSH) increase slightly with increasing age of hydration up to 28 days. The slight increase in the intensity of the peak characteristic for CSH hydrates is attributed to the amorphous and illcrystalline nature of these hydrates. However, the intensities of the peaks characteristic for the free calcium hydroxide (CH) showed a notable increase with increasing age of hydration up to 28 days. These results can be explained in terms of increase in the amount and/or degree crystallinity of the formed CH as a result of progressive hydration of OWC. Addition of 0.25 of AFS to OWC (Mixes AI) indicate the same diffraction patterns and nearly the same trend of variation of the peaks intensities with increasing the age of hydration as those of neat OWC paste (Mix Ao), Fig. (6). This reveals that addition of AFS to OWC does not alter the formed hydration products; it may only affect their degree of crystallinity.

Differential thermogravimetric analysis (DTGA)

The results of differential thermogravimetric analysis obtained for the hardened neat OWC paste (Mix Ao) as well as OWC admixed by 0.25 % AFS (Mix AI), after 6 hrs, 3 and 28 days are shown in Figs. (7) and (8), respectively. The DTGA thermograms of the hardened OWC cement paste made of blank (Mix Ao) indicate the presence of three endothermic peaks located at temperature range 100 - 150, 450 - 520 and 650 - 750 °C, Fig. (7). The first endotherm located at 100 - 150 °C is due to the dehydration of calcium silicate hydrates (CSH) and calcium sulphoaluminate (ettringite and monosulphate) hydrates (30). The values of percentage weight losses of this endotherm are 1.12 %, 6.06 % and 7.23 % after 6 hrs, 3 and 28 days of hydration, respectively. The notable increase in these values is explained in terms of progress of hydration reaction and increase in the amount of the formed hydration products (mainly as CSH and calcium sulphoaluminate hydrates). These results are in agreement with the results obtained from compressive strength test. The main endothermic peak observed in the temperature range 450 - 520

°C, with maximum at 480 °C, is due to the thermal decomposition of $\text{Ca}(\text{OH})_2$ (31). The values of percentage weight losses of this endotherm are 0.55 %, 3.93 % and 4.69 % after 6 hrs, 3 and 28 days, respectively. The observed increase in the percentage weight loss with increasing age of hydration is explained in terms of increase in the amount of the formed CH as a result of the progress of hydration of OWC. Furthermore, it is evident that a part of hydrated product was carbonated during handling of the cement pastes as demonstrated by endotherm at 650 - 750 °C (30, 32). The values of weight loss of this endotherm are accounted for the different degrees of carbonation of the hydrated specimens. Addition of 0.25 of AFS (wt.%) to OWC (Mixes AI) indicates the same endothermic peaks, as well as the same trend of increasing values of weight loss with increasing the age of hydration as those of the neat OWC paste. The obtained results of weight loss in both CSH and CH obtained for hydrated specimens of Mix AI showed slightly lower values and/or comparable values relative to those of Mix Ao during the early ages of hydration (6 hrs and 3 days); mean while, slight higher values are obtained for admixed OWC pastes than those of the neat OWC paste after 28 days of hydration, Fig. (8). These results are confirmed with the results of compressive strength test which can be explained in terms of the retardation effect of AFS on OWC hydration during the early ages of hydration (6 hrs to 3 days). Finally, the values of weight loss due to decomposition of CaCO_3 are accounted for the different degree of carbonation of the hydrated specimens.

Rheological measurements

The effect of additions of AFS on the rheological properties of OWC pastes was investigated by measuring the mini-slump of the OWC cement paste. The obtained results showed that, the mini-slump values obtained for OWC paste admixed with 0.25, 0.50, 0.75 and 1.00 % of AFS (Mixes AI - AIV) are higher than those of the neat OWC (Mix Ao), and as the amounts of AFS admixture increase the values of mini-slump increase. This result is mainly attributed to the notable increase in the degree of fluidity of OWC pastes by addition of AFS admixture.

Table 3. Elemental analysis of the synthesized organic admixture.

Organic admixture	C %		H %		O %		S %	
	Theor.	Found	Theor.	Found	Theor.	Found	Theor.	Found
AFS	39.47	39.45	5.63	5.61	36.81	36.80	10.54	10.52

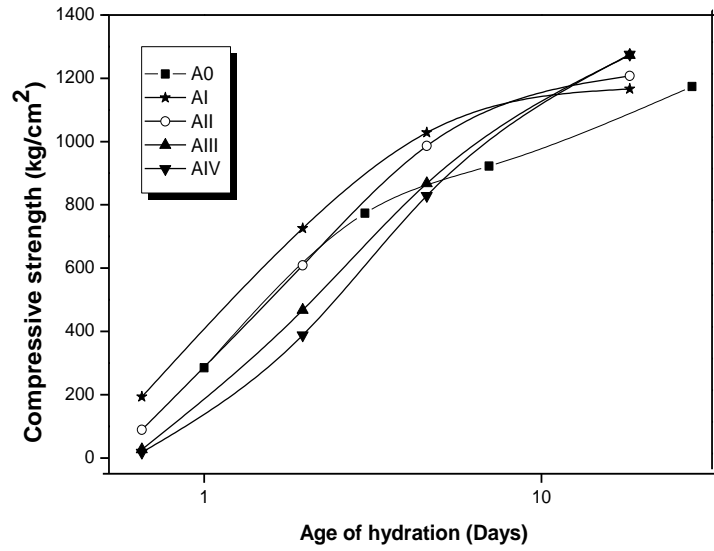


Fig 2. Compressive strength values for hardened OWC pastes, Mixes (Ao-AIV).

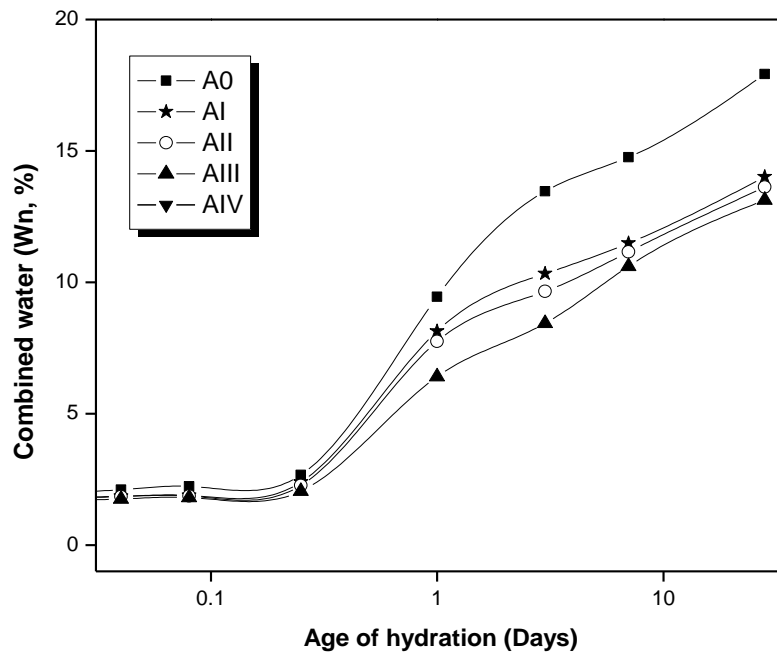


Fig 3. Chemically combined water contents for hardened OWC pastes, Mixes (Ao-AIV).

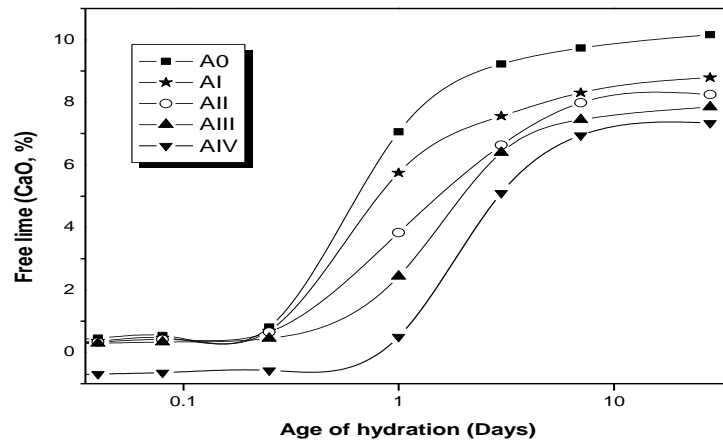


Fig 4. Free lime content for hardened OWC pastes, Mixes (Ao-AIV).

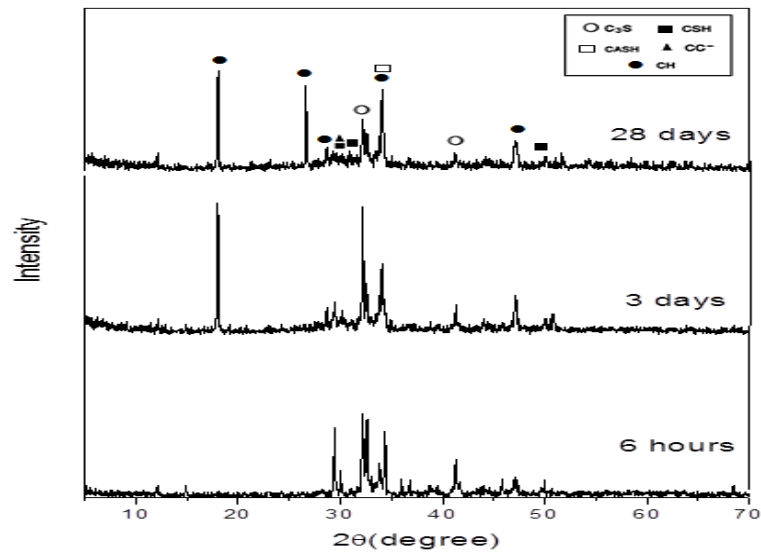


Fig 5. XRD patterns of hardened neat OWC pastes, (Mix Ao) at different ages of hydration.

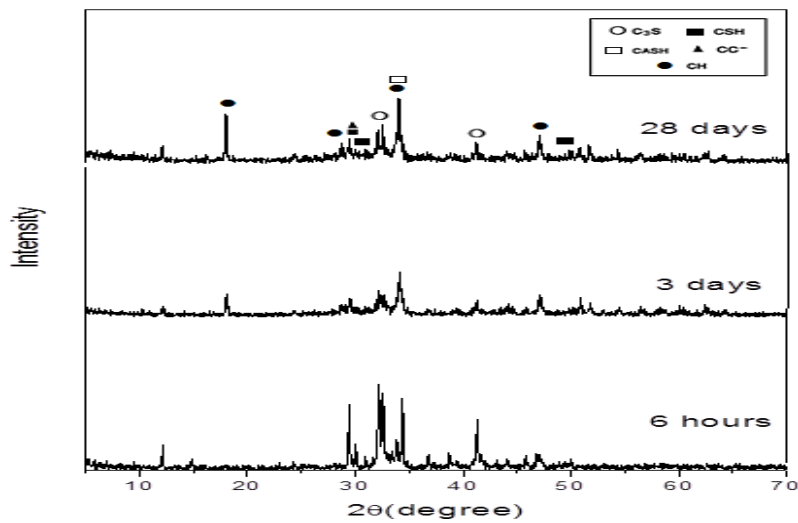


Fig 6. XRD patterns of hardened OWC pastes admixed with 0.25% of AFS (Mix AI) at different ages of hydration.

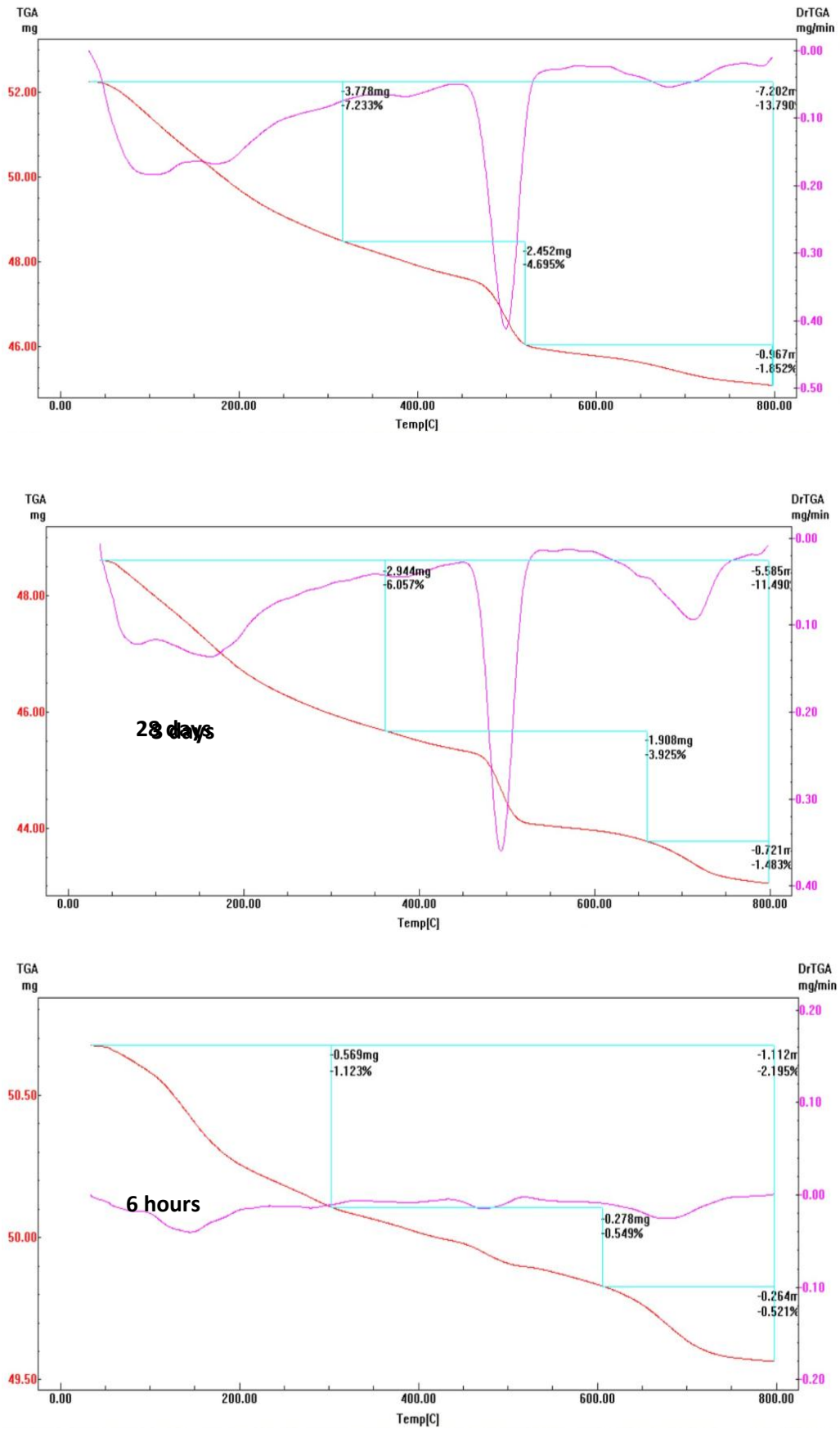


Fig 7. DTGA thermograms of hardened neat OWC pastes (Mix Ao) at different ages of hydration.

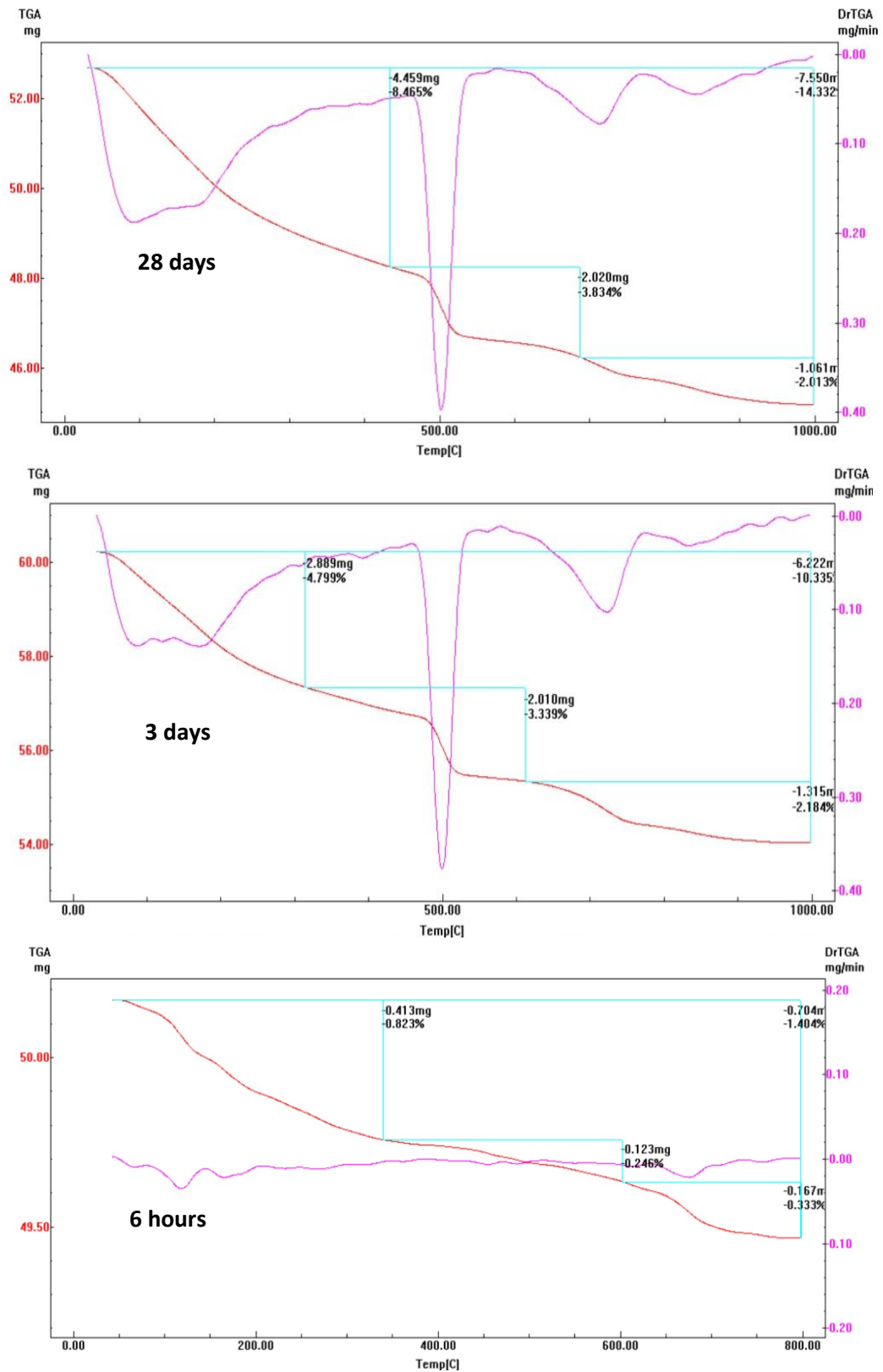


Fig 8. DTGA thermograms of hardened OWC pastes admixed with 0.25% AFS (Mix AI) at different ages of hydration.

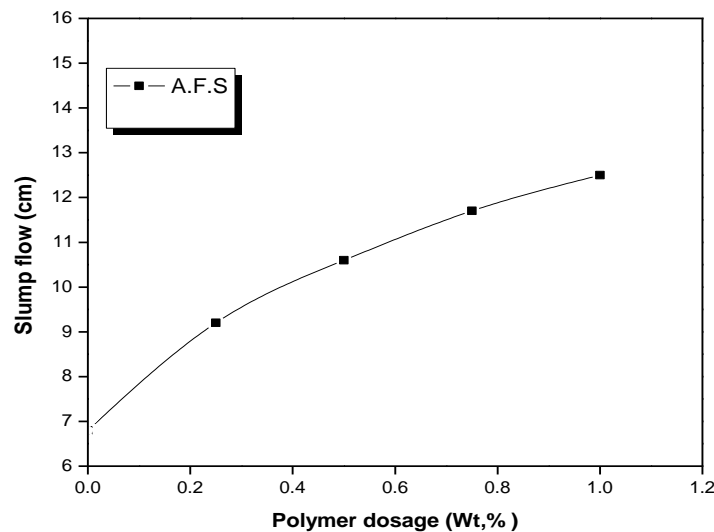


Fig 9. Mini- slump values of OWC pastes admixed with different amounts of AFS.

4. Conclusions

On the basis of the results obtained in this investigation, the following conclusions could be derived:

- 1- The values of compressive strength for hardened pastes made of OWC admixed by different amounts of AFS (Mixes AI- AIV) showed a notable lower values especially during the early ages of hydration (up to 7 days), and showed a comparable and/or higher values after 28 days of hydration as compared to the control paste (Mix Ao).
- 2- The values of combined water content obtained for all AFS admixed OWC hardened pastes are lower than those of the neat OWC paste.
- 3- The free lime contents (CaO, %) obtained for all the AFS admixed OWC hardened pastes are lower than those of neat OWC paste.
- 4- Rheological measurements indicated that the mini-slump values of cement pastes admixed with different doses of AFS increase with increasing AFS doses as a result of increasing fluidity of admixed cement pastes.
- 5- DTGA thermograms and XRD diffractograms obtained for the tested pastes indicate the formation of: nearly amorphous calcium silicate hydrates (mainly as CSH-I and CSH-II), calcium sulphoaluminate hydrates (ettringite and monosulphate hydrates), calcium hydroxide and calcium carbonate.

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