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Effect of Some Prepared Superplasticizers (Cyclohexanone Based) on Compressive Strength and Physico-chemical Properties of Oil Well Cement Pastes

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TWO different superplasticizers particularly Cyclohexanone formaldehyde sulfanilate (CFS) and Cyclohexanone glyoxylic sulfanilate (CGS) were prepared; also, their effect on mechanical and physico-chemical properties of oil well cement was assessed. The chemical structures were affirmed by FTIR technique. The designed chemical compounds were predestined as superplasticizers for cement pastes. The pastes made by superplasticizers (CFS or CGS) addition to cement by the ratios of 0, 0.25, 0.50, 0.75, and l.00 as mass % of cement. The consistence water, setting time, chemically combined water content (Wn), the hydration rate and compressive strength of the admixed hardened pastes were predestined at various time periods. The phase composition was intended by DSC and XRD techniques. The results revealed that as the admixture dose rate increases the demand cement paste water of consistency decreases. Also, as the admixture addition rate increases the chemically combined water content decreases, so the rate of hydration decreases; meanwhile compressive strength magnitudes increase in accounts for the low water/cement (initial porosity) of the sample.

The factors affecting the cementing process of oil wells are the temperature, pressure, minerals, impurities and the water/cement ratio [(1,2)]. These factors jointly or individually having a fastening effect on the initial setting time of cement which negatively affects the workability and makes it impossible to pump as a result of the coming down of the cement deep into the ground and this process may take several hours. So begin the use of chemical mixtures [(3)], such as superplasticizers, which work to increase fluidity and to facilitate the process of pumping cement slurries to the depths of wells easily with no changing in the cement properties [(4)]. Strong repulsion happens because the superplasticizer and the cement particle's charge by a similar charge [(5)]. Given the large molecular weight of superplasticizers, the plasticizers are also working as pore fillers between the particles of cement which reduces the water/cement ratio and therefore gives a high magnitude compressive strength [(6-9)]. The synthesis processing parameter of water soluble sulfonated cyclohexanone formaldehyde (SCF) superplasticizer has influence on the inherent viscosity and the dispersing

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ability [10]. The SCF resin was regarded as an aliphatic high polymer containing block hydrophilic and hydrophobic structures in the molecular chain, which show little surface activity. So we need to increase the hydrophobic and/or hydrophilic groups by entering a sulfanilic acid in the reaction as a copolymer synthesis.

Experimental

Materials

Cement

A freshly produced sample of class G moderate sulfate resistant cement supplied by Schlumberger Company, Egypt was used. Its chemical composition was found to be: CaO, 60.4%; SiO_2, 20.2%; Al_2O_3, 2.2%; Fe_2O_3, 2.7%; MgO; 6.0%; SO_3, 3.0%; total alkali expressed as Na_2O, 0.75%; insoluble residue, 0.75% and loss on ignition, 3%. The specific surface area as determined by the Blaine air-permeability method was found to be 3800 cm^2/g.

Synthesis of superplasticizers

Cyclohexanone formaldehyde sulfanilic acid sodium salt condensate

Sulfanilic acid 86.5 g (0.5 mol), 98 g of cyclohexanone (1.00 mol) and 300 ml water were placed into three necks flask with a stirrer and 20% aq. NaOH was placed to adjust pH 9. Thereafter, the mixture was stirred at 60-65 °C, and 168 g of 37% aq. HCHO was dropwise added into the reactor. After 3.5 hr, the mixture calmed down to ambient temperature and the pH adjusted to 11.0 with 20 wt % aq. NaOH. 1 hr later, the unreacted cyclohexanone and formaldehyde were separated by distillation.

Cyclohexanone glyoxylic sulfanilic acid sodium salt condensate

122.1 g (1.649 mol) of aq. glyoxylic acid (50%) and 270 g of water was added to a one L reaction flask with a three neck. Reflux condenser, stirrer and dropping funnel. 123.4 g of 50% aq. NaOH is added with stirring and the pH was adupted to 4.0. Adjust the temperature to 50 °C and 98 g of cyclohexanone (1.00 mol) was added with continued stirring for 75 min till the mixture has turned to a transparent solution. The pH ascent during this periode to 5.9. After cooling, 88 g (0.509 mol) of sulfanilic acid and 48g of 20% aq. NaOH are added simultaneously, which causes the pH to drop to 5.2. Stirr the vessel content at 90 °C till obtaining a viscosity of 5.52 cSt (20 wt. % solution at 20 °C). Adjust the pH to 10.0 by adding 39.2 g of 50% aq. NaOH.

Methods of measurement

FT-IR analysis

The synthetic routes of modern superplasticizer were trappable by FT-IR spectroscopy. The FT-IR analysis was done in Egyptian Petroleum Research Institute using ATI Mattsonm Infinity Series™, Bench top 961 controlled by Win First ™ V2.01 Software.

Water of consistency and setting time

The standered water of consistency, initial setting and final setting times were decided using a Vicat apparatus [11,12].

Moulding and curing

The various cement pastes made by mixing the dry cement with the required water of consistency including various doses of superplasticizer using the W/C

ratios as presented in Fig. 3 (a, b). The operation was completed by continuous and vigorous mixing for about three minutes. After complete mixing the pastes were molded into cubic specimens by using stainless steel (2.54 x 2.54 x 2.54 cm^3) cubic moulds. Compacting by vibration was carried out, and the leveling and smoothness of the top surface of the pastes was made by a thin edged towel [13]. Immediately after moulding, the specimens with their moulds were cured at about 100 % relative humidity at ambient temperature for the first day to attain the final setting of the specimens. The hardened pastes were then extracted from the moulds and cured under tap water for various time intervals of 3, 7, 28 and 90 days.

Determination of compressive strength

The compressive strength trials were achieved on the hardened pastes at every time interim using three cubic specimens at every hydrated time and the rate value was determined as kg/cm^2. This test was fulfill using a Ton-industric machine (West Germany) for 60 tons as maximum load.

Stopping of hydration

The hydration stopping operation was achieved on the crushed cubic specimens after determination of the compressive strength according to the method reported [14].

Determination of chemically combined water content (Wn, %)

Two representative samples of the desiccated specimens, exactly about 1g each, were weighted in crucibles from porcelain and ignited for one hour at 1000˚C in an amenable muffle furnace, desiccated to cool and then weighted. The chemically combined water content was calculated using the next equation:

$$Wn\ (\%) = [(W_1 - W_2) / W_2] \times 100 - L$$

W_1: is the dryish sample weight before ignition (g) and

W_2: is the sample ignited weight (g)

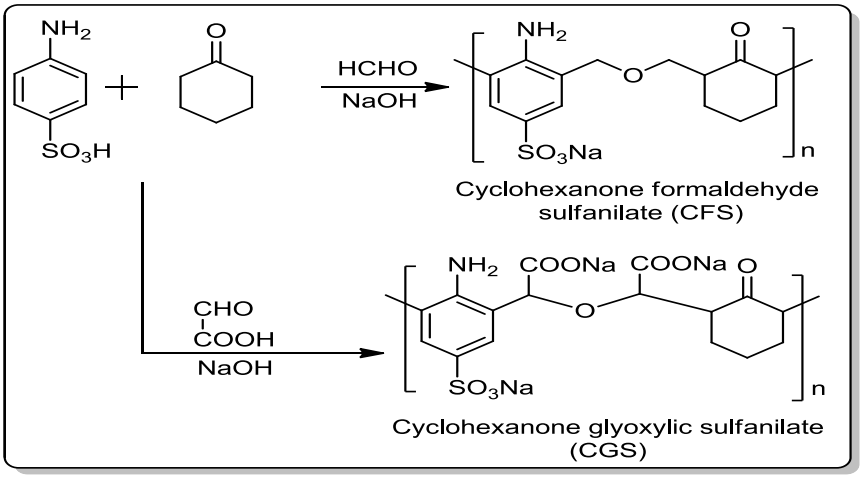
L : is ignition loss

Results and Discussion

IR spectrophotometric analysis

The synthesized superplasticizers particularly cyclohexanone formaldehyde sulfanilate (CFS) and cyclohexanone glyoxylic sulfanilate (CGS) have common groups according to preparation methodology. The general process is the reactivity of formaldehyde or glyoxylic acid which react with active methylene ($-CH_2-$) of cyclohexanone to make methylol group formation. As the reaction progresses, the condensation takes place among methylol ($-CH_2OH$) groups leading to the ether linkage formation ($-CH_2OCH_2-$) of CFS or CGS products. Finally, the methylol group on the terminal part reacts with sulfanilic acid to produce the CFS or CGS product (Scheme 1).

IR spectra are represented in Fig. 1,2 referring to the hydrogen bonding intermolecular in the solid materials as the result of the coiling form of the polymer molecules. In all synthesized superplasticizers spectra, the characteristic absorption of the ether linkage (C-O-C) formed due to condensation of methylol groups is a strong band in the 1035-1178 cm^{-1}. S=O stretching frequency range is 1354 cm^{-1}, see also Table 1.



Scheme 1. Synthetic route of CFS and CGS superplasticizer.

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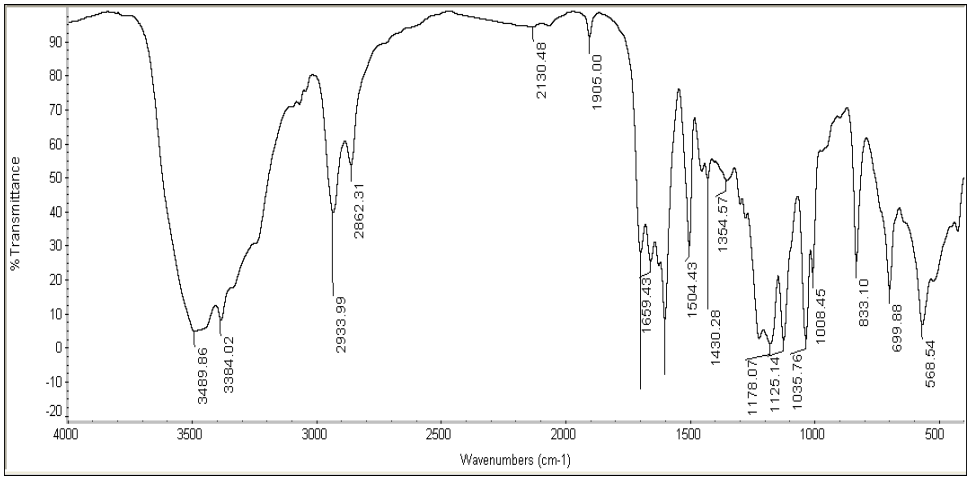

Fig. 1. FT-IR spectrum of CFS,

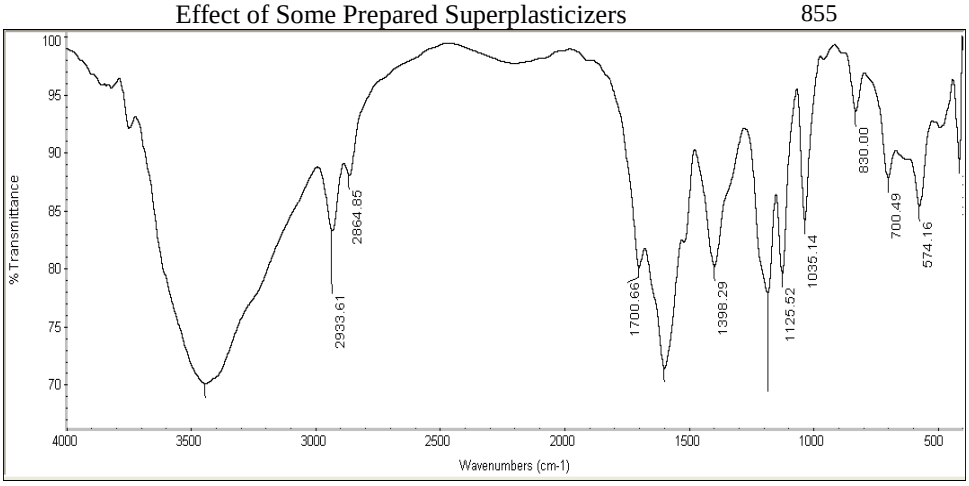

Fig. 2. FT-IR spectrum of CGS.

TABLE 1. IR Characteristic bands of the synthesized superplasticizers.

Compounds. **Band (cm^{-1})**	**CFS**	**CGS**
NH_2 stretching	3384	3444
C-O-C stretching	1035-1178	1035-1184
C=O stretching	1699	1700
C=C stretching	1504- 1602	1598
S=O stretching	1354	1398
C-H stretching aliphatic	2862-2933	2933-2864

Water of consistency and setting time

Cement reacts with water and CaOH is liberated during the Portland cement hydrated. Calcium hydroxide reacts with the colloidal acid hydrates to form hydrated calcium aluminates, silicates and hydrogarnets[15,16]. The reactions of tricalcium aluminates solid-solution phases predominate at the morning hydration. The initial setting time predominates from calcium silicate phases reactions [17]. Immediately water is in contact with cement, several chemical reactions take place which lead to cement pastes setting and hardening.

The pastes made by addition of the admixture, cyclohexanone formaldehyde sulfanilate (CFS) or cyclohexanone glyoxylic sulfanilate (CGS) to cement by the ratios of 0.0, 0.25, 0.50, 0.75, and l.00 mass % of cement, respectively. The water of consistency, setting times of the different cement pastes made with various ratios of admixtures are presented in Fig. 3 (a , b). From Fig. 3 (a , b), as the dose of admixture increases the consistence water tends to decrease. The reduction of mixing water at 0.25% admixture is 13% and 15% for CFS and CGS, respectively. But the reduction of mixing water at 1.00 % admixture is 25% for the two admixtures. Therefore, the consistence water decreases with increasing admixture dosage for both CFS and CGS.

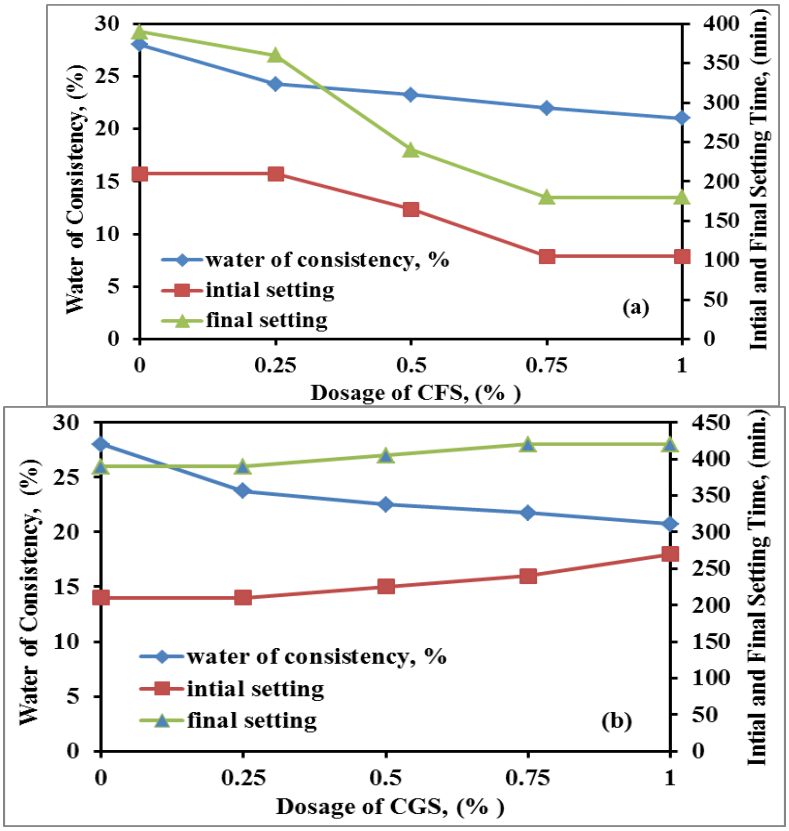

Fig. 3 (a, b). Water of consistency and setting time of admixed OWC pastes with CFS and CGS superplasticizers.

Evidently, the cement pastes setting times admixed by CFS are accelerated as a result to the reduction of consistence water with increasing dose of admixture (Fig.3a); It can be concluded that the prepared CFS superplasticizer is water reducer and accelerating admixture.

Figure 3b exhibits that the cement pastes setting time, admixed by CGS is retarded by the addition of this admixture. As the dose of admixture increases, the setting time increases. Therefore, it can be concluded that the CGS acts as water reducer and retarder.

The operation mechanism depends on the superplasticizer adsorption upon the cement particle surface; this could decrease flocculation. The prepared compounds increase the zeta-potential of cement molecules which carry a surface charge of the same sign (negative charge); therefore, the cement particles dispersion due to repel each other. Basically, dispersion mechanisms are found: electrostatic repulsion and steric hindrance. The relatively high negative zeta-potentials obtained when superplasticizers with a sulfonic group, were suggesting that the cement particle dispersion mechanism is fundamentally striped by the repulsion of electrostatic

between negatively charged particles. This proves that the mechanism by which cement particles are dispersed is not attributed to electrostatic repulsion. Cement particles dispersion in this case must be due to steric effects as reported by Aiad [9].

Chemically combined water content (Wn, %)

The chemically combined water contents (Wn, %) of the different cement pastes admixed with different dosages of CFS or CGS (0.25, 0.5, 0.75 and 1.0% by cement weight), in addition, the control cement pastes are graphically represented in Fig. 4 and 5, respectively.

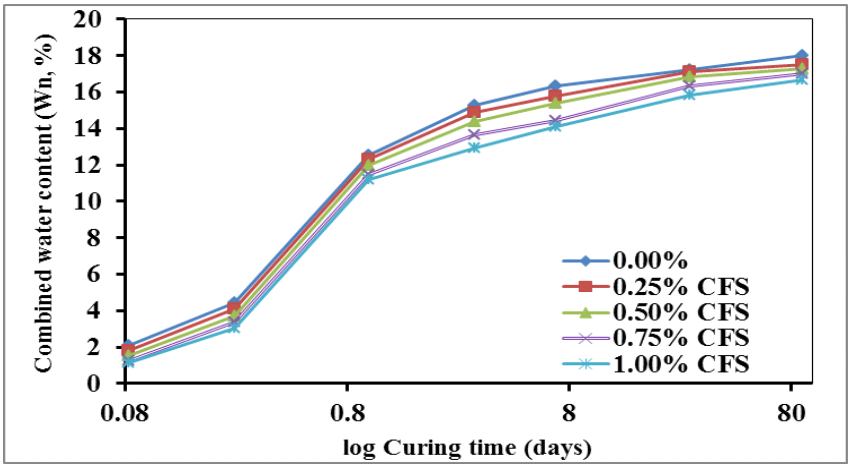

Fig. 4. Chemically combined water contents of cement pastes admixed with different dosages of CFS at various ages of hydration.

During the admixed cement pastes hydrated, two stages of hydration could be distinguished; the first stage is established on the dosage of admixture, while the second stage is established on the initially formed products of hydration. The hydration rate of the control is higher than that of cement pastes admixed with CFS or CGS. As the time interval increases from 2hr to 90 days, Wn increases of 2%, 1.77%, 1.45%, 1.26% and 1.19% to 17.99%, 17.15%, 17.05%, 16.85% and 16.54%, corresponding to 0, 0.25, 0.5, 0.75 and 1.00% dosages by cement weight but Wn% decreases as a result to increasing the CFS dosages which is observed in Fig. 4.

Wn% of the cement pastes admixed with cyclohexanone glyoxylic sulfanilate (CGS) additive are observed in Fig. 5.

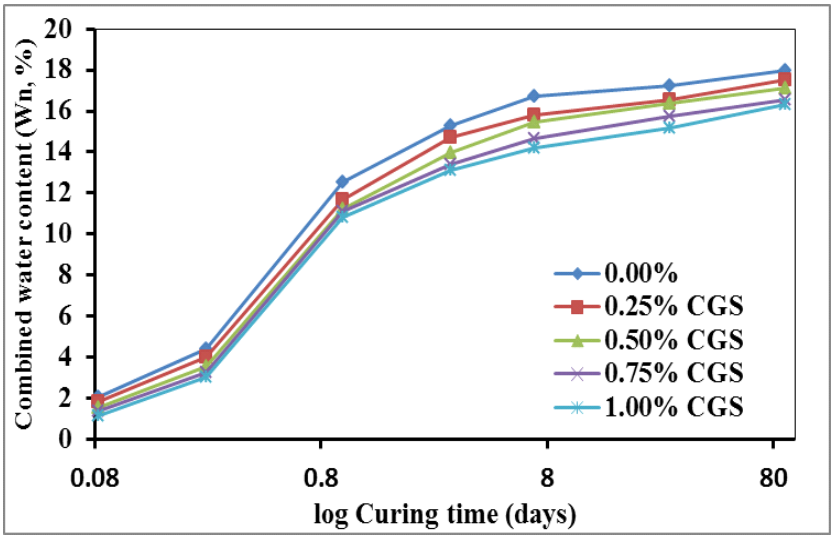

Fig. 5. Chemically combined water contents of cement pastes admixed with different dosages of CGS at various ages of hydration.

Degree of hydration (α)

The extent to which the cement has hydrated was expected by the hydration degree α, which is realized as the weight fraction of original cement which has become completely hydrated ($0 \leq \alpha \leq 1$) as shown in Equation (1).

$$\alpha = \frac{W_n}{W_\infty} \quad \ldots\ldots\ldots\ldots (1)$$

where: W_n is the combined water content (%) at a presented curing time.

W_∞ is the whole combined water content after full hydration ≈ 0.23

The factor of 0.23 in Equation (1) represents the non-evaporable water content per gram of cement in the mixture for complete hydration.

It is an predestined value calculated by the Bogue composition of cement [18]. Tables 2 and 3 show the values of the hydration degree of the hardened pastes made with CFS and CGS respectively, at the various ages of hydration. It is evident that the hydration degree is building on the initial W/C ratio. Thus, all of the values of the hydration degree of hardened pastes which mixed with admixtures are lower than those of the control samples. In addition, as the dosage of admixtures increases the hydration degree decreases.

Gel/Space ratio

The gel/space ratio represents the cement volume ratio hydrates to the volume available to accumulate these hydrates; the latter volume consists of the hydration products volume plus the residual pores volume (capillary pores). The use of gel/space ratio results in a reduction in the number of variables as it represents a

combined effect of the hydration degree and the initial porosity as controlled by the initial W/C ratio. The gel/space ratio is given by the following equation:

$$X = \frac{0.646\ \alpha}{0.319\ \alpha + W_o / C} \quad \text{..... (2)}$$

where: α is the fraction of the hydrated cement (degree of hydration).

C is the cement weight.

W_o is the mixing water volume.

W_o/C is the initial water/cement ratio (by weight).

TABLE 2. The degree of hydration (α) of the hardened cement pastes made by different dosages of CFS at various ages of hydration.

Curing Time	Degree of Hydration (α)				
	Doses				
	0%	0.25 %	0.50%	0.75 %	1.00 %
2 hrs.	0.091	0.079	0.066	0.054	0.050
6 hrs.	0.193	0.178	0.162	0.147	0.133
1 day	0.545	0.536	0.521	0.498	0.486
3 days	0.664	0.647	0.625	0.593	0.563
7 days	0.711	0.685	0.669	0.628	0.613
28 days	0.748	0.744	0.732	0.711	0.689
90 days	0.782	0.762	0.750	0.740	0.723

TABLE 3. The degree of hydration (α) of the hardened cement pastes made by different dosages of CGS at various ages of hydration.

Curing Time	Degree of Hydration (α)				
	Doses				
	0%	0.25 %	0.50 %	0.75 %	1.00 %
2 hrs.	0.091	0.080	0.068	0.060	0.050
6 hrs.	0.193	0.175	0.155	0.143	0.132
1 day	0.545	0.509	0.489	0.483	0.472
3 days	0.664	0.640	0.608	0.582	0.570
7 days	0.711	0.687	0.673	0.636	0.617

28 days	0.748	0.720	0.713	0.684	0.661
90 days	0.782	0.762	0.744	0.720	0.708

The gel/space ratio values for the hardened pastes which made with different dosages of admixtures in addition to the control paste (free of admixture) at different curing time intervals are graphically represented in Fig. 6 and 7.

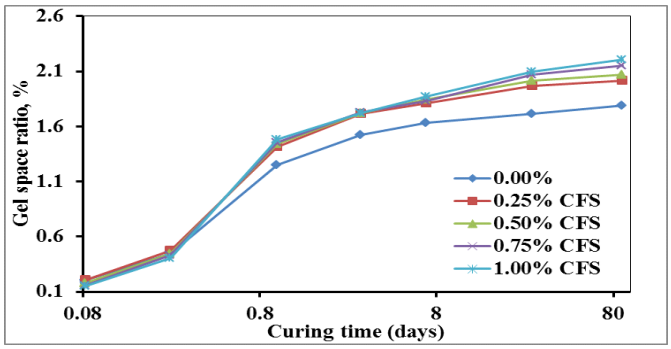

Fig. 6. Variation of gel/space ratio values of the various hardened cement pastes made by different dosages of CFS with age of hydration.

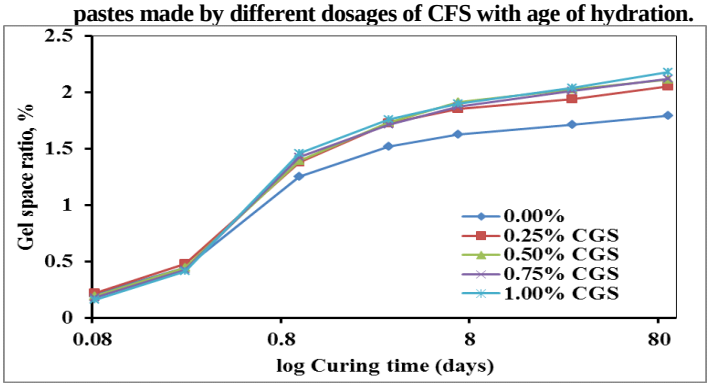

Fig. 7. Gel/Space ratio values of the various hardened cement pastes made by different dosages of CGS at various ages of hydration.

Figure 6 and 7 indicate that the curves of the cement pastes having admixtures are shifted to higher values of gel / space ratio wih higher values of compressive strength at each time of hydration. In other words, as the gel / space ratio increases the compressive strength increases [19]. This can be explained by the fact that as the curing time increases, the rate of hydration reaction increases and leads to increase the hydration products (gel products), which can fill the limited pores existing in the pastes leading to higher compressive strength values.

Compressive strength

The compressive strength values of the hardened cement pastes made with different dosages of CFS or CGS admixtures are graphically represented in Fig. 8

and 9, respectively. A sharply increasing in the compressive strength values showed during the morning ages of hydration up to 3 days, recognized by a gradual increase up to 28 days; then at later curing time of hydration (90 days) a very slight increase was observed. The previous results are attributed to the Portland cement hydration and formation of hydration products, mainly as calcium silicate hydrates (C-S-H) having good hydraulic characteristics and work as powerful binding centers between the cement grains. Evidently, the chemical structure and the dosages of the admixtures are essential factors affecting the improvement of mechanical characteristics of the hardened paste of cement.

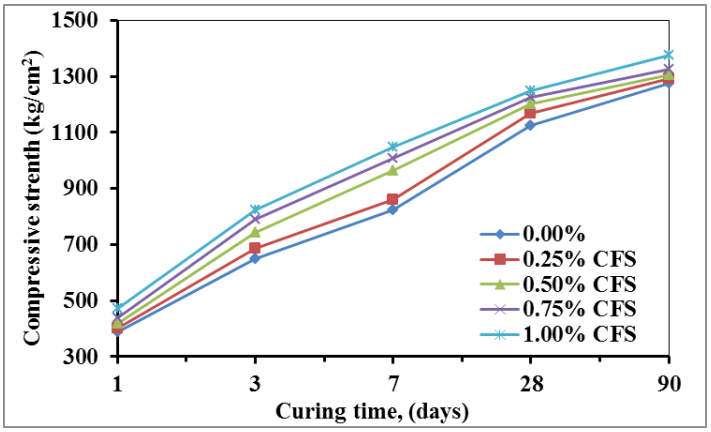

Fig. 8. Variations of compressive strength of the various cement pastes having different dosages of CFS with age of hydration.

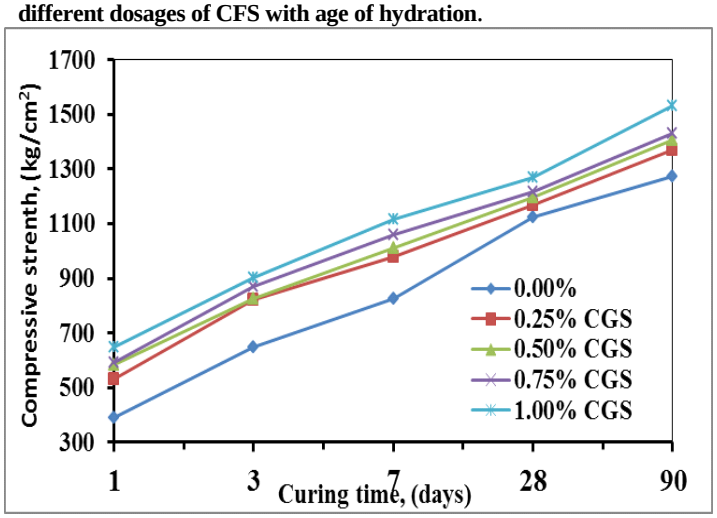

Fig. 9. Variations of compressive strength of the various cement pastes having different dosages of CGS with age of hydration.

For all the admixed cement pastes, as the dosage increases the compressive strength increases. Evidently, there are some inter-related parameters; namely, W/C

ratio, admixture dosage and hydration degree that affect the fresh and hardened cement pastes main hydration characteristics. By comparing the admixture dosages effect on the combined water content and compressive strength, it is evident that as the dosage of these admixtures increases, the combined water content decreases and the strength improvement enhances.

In other words, this result is fundamentally refered to the more dense structure of the cured admixed cement pastes affected by the strong water reduction caused by each admixture addition [20]. The stronger the water reduction caused by any admixture, the lower the initial porosity and both compressive strength and gel/space ratio increasing values of the cured admixed cement paste. The strength results seem to be dependable on the nature and the physical state of the hydration products formed within the pore system of the admixed cement pastes.

Phase composition of the formed hydrates of the admixed hardened OWC pastes

The composition of phase of the formed hydrates obtianed for some selected admixed hardened OWC pastes was identified using X-ray diffraction (XRD) analysis and differential scanning calorimetry (DSC).

X-ray diffraction (XRD) analysis

The X-ray diffractograms obtained for the hardened OWC pastes admixed with 0.25 and 0.75% of CFS and CGS admixtures after 7 and 90 days of hydration are displayed in Fig. 10 and 11, respectively. Figures 10 and 11 presented that the distinguished phases identified at various ages of hydration are for both, (i) unhydrated phases; alite (C_3S) has been identified by its characteristic basal reflections. (ii) hydrated phases; calcium hydroxide ($Ca(OH)_2$) formation, as a result of tricalcium silicates hydration (C_3S) could be distinguished. Calcium silicate hydrates, C-S-H (I) and (II), could also be distinguished, as main hydration products.

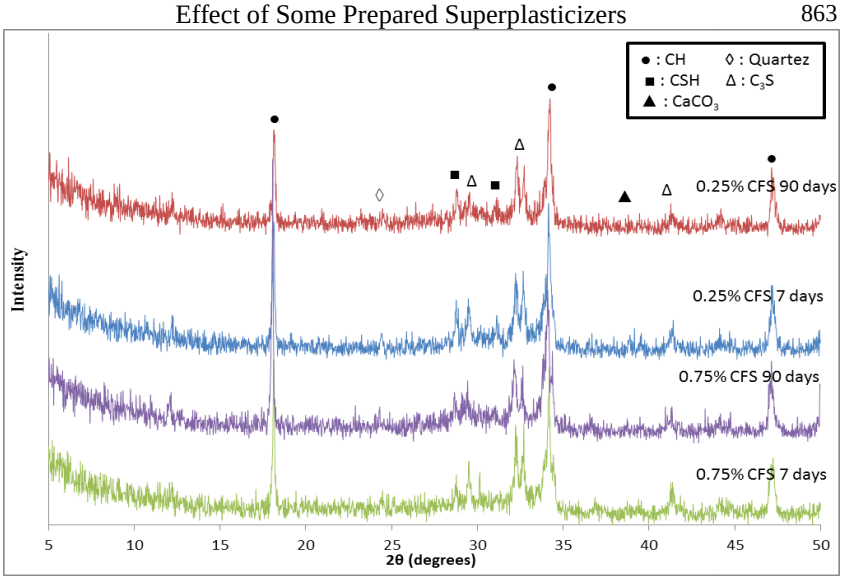

Fig. 10. XRD patterns of the admixed hardened paste by 0.25% and 0.75% CFS polymer after different ages of hydration.

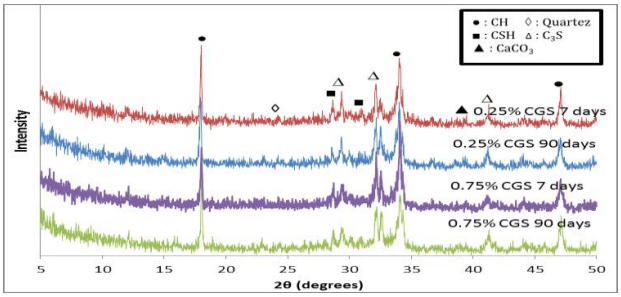

Fig. 11. XRD patterns of the admixed hardened paste by 0.25% and 0.75% CGS polymer after different ages of hydration.

In conclusion, Fig. 10 and 11 show that the peaks intensities characterizing C_3S decrease with increasing hydration age; meanwhile, the peaks intensities characterizing the hydration products, namely calcium silicate hydrates (CSH) and (CH), increase with increasing hydration age up to a final hydration age (90 days).

Differential scanning calorimetry (DSC)

The thermograms of the cured OWC pastes admixed with 0.25% and 1.00% of CFS and CGS which hydrated for 3 days and 28 days are presented in Fig. 12 and 13, respectively. The thermograms indicate three endothermic peaks at 90-105, 490 – 510 and 718 – 750°C. The first endotherm existing at 90 – 105°C is mainly because of the removal for free water and the dehydration of the amorphous part of (CSH) [20]**.** The second endotherm existing at 490-510 °C, showed the majority of weight loss, is fundamentally regarding to portlandite (CH) decomposition [22]. The intensity of this endotherm increases with increasing hydration age for 3 and 28

days of hydration respectively; this is referred to the amount increasing of liberated free lime. Finally, the third endotherm existing at 718 – 750°C is because of the CaCO3 decomposition.

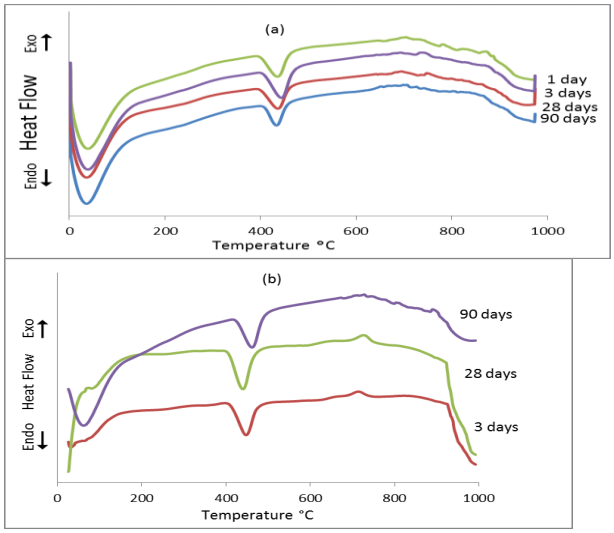

Fig. 12. DSC patterns of the admixed hardened OWC paste with:
a) 0.25% CFS after 3 and 28 days of hydration.
b) 1.00% CFS after 3 and 28 days of hydration.

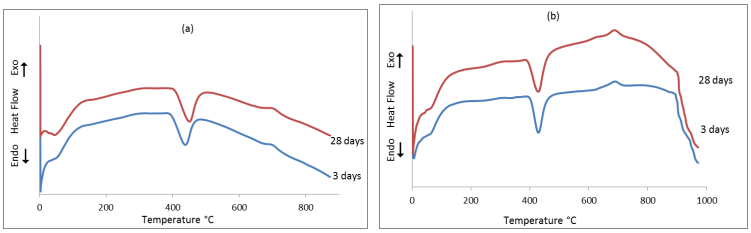

Fig. 13. DSC patterns of the admixed hardened OWC paste with:
(a) 0.25% CGS after 3 and 28 days of hydration.
(b) 1.00% CGS after 3 and 28 days of hydration.

Conclusion

From the obtained results, the following conclusions can be drawn:

1- CFS or CGS superplasticizers have significant reduction in the water of consistency reached to 24%. CFS accelerates the initial setting time because of the reduction of mixing water. As well as, the final setting time is accelerated by the admixture dosages increase. As the dose of CGS increases the initial and final setting times increase. Meanwhile, as the admixture dosage increases the compressive strength increase for all cured cement pastes investigated.

2- By comparing the admixture dosages effect on the combined water content and the compressive strength, it is evident that as the admixtures dosages increases, the combined water content decreases and the strength improvement enhances.
3- The results of phase composition of the formed hydrates for some selected samples of the admixed OWC pastes obtained by X-ray diffraction (XRD) analysis are in agreement with those obtained by differential scanning calorimetry (DSC).
4- CFS act as accelerating agent due to the low charge density on the polymer molecule which makes the repulsion force low.
5- CGS act as retarders agent due to the highly charge density on the polymer molecule which makes the repulsion force high. That is leads to highly dispersion effect of cement particles which leads to viscosity decreasing and increasing the fluidity, so the workability become easier.

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تأثير بعض ملدنات السيكلوهكسانون المحضرة على الخواص الفيزيقوكيميائية والميكانيكية لعجائن أسمنت آبار البترول

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تهدف هذه الدراسة إلى تحضير اثنين من الملدنات المختلفة وهي السيكلوهكسانون سلفانيلات الفورمالديهايد (CFS) و السيكلوهكسانون سلفانيلات الجليوكسيليك (CGS) ومن ثم دراسة تأثير هذه الملدنات علي الخواص الفيزيقوكيميائية والميكانيكية لأسمنت أبار البترول. تم إثبات التركيب الكيميائي عن طريق تقنية (FT-IR).وكذا تم دراسة تأثير المركبات المحضرة كخلطات كيميائية لمعاجين الإسمنت وكانت الإضافة من CFS أوCGS كالتالي 0، 0.25، 0.50، 0.75 و1.00 ٪ من كتلة الاسمنت. تم تحديد نسبة الماء القياسية وكان لكل من المركبين تأثير علي انخفاض ماء الخلط القياسي ليصل إلي 24.1٪ عند إضافة 1٪ من إحدي المركبين. كما تم دراسة زمن الشك الإبتدائي والنهائي وكان لكل من المركبين تأثير مغاير علي زمن الشك الإبتدائي أو النهائي. تم دراسة الماء المتحد كيميائيا وقوة تحمل الضغط علي عجائن الأسمنت المتصلبة في مختلف الأوقات الفاصلة من يوم واحد إلي 90 يوما, تم اثبات التركيب الصنفي لنواتج التأدرت للعجائن الصلبة بواسطة DSC وXRD . وأوضحت النتائج أنه كلما زادت جرعة الملدنات المحضرة انخفضت نسبة مياه الخلط المطلوبة لعجائن الأسمنت ومن ثم انخفاض كمية الماء المتحد كيميائيا وزيادة قيمة قوة التحمل للضغط الهيدروليكي نظرا لانخفاض نسبة المسامية الأولية للعينات.