

The Behavior of Lauryl glucoside as cement paste admixture

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Abstract:

The use of air entraining agents have been accepted practice in cement technology for many years. A large number of uniform and stable micro bubbles is intentionally entrained to improve the durability and reduce the potential for damage from freezing and thawing, but these bubbles are inversely affected the hardened cement paste compressive strength. The Physiochemical and Mechanical Properties of cement pastes containing synthetic nonionic surfactant Lauryl glucoside (LG) as air-entraining agents are investigated. Different cement pastes with and without LG are prepared for five interval times (3, 7, 28, 90 and 360 days). The influence of the different surfactant concentrations on the air content of the pastes and accordingly the bulk density, compressive strength and microstructure of the hardened cement specimens is discussed. The optimal level of factors to achieve the desired goals are determined. The results show that LG causes a uniform distribution of small air voids in the cement paste, beside its ability to accelerate the hydration reaction, which may greatly improve the compressive strength of the pastes.

Keywords: air-entraining admixtures, density, compressive strength, XRD, Lauryl glucoside, nonionic surface- tent.

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I. Introduction

It is well known that the pore structure of hardened cement pastes strongly influence their physical properties. The presence of an air-entraining agent (AEA) not only improves workability, but also enhances its freeze-thaw resistance (Lea's, 2004). Du and Folliard (2005) noted that there is a minimum dosage of AEA required to entrain air in the concrete. Rosen (2004) reported that the formed air bubbles in cement pastes are unstable and have limited lifetimes. Therefore, surfactants as AEAs are used in many cases to entrain air bubbles and stabilize them in the fresh cement pastes.

Whiting and Nagi, (1998) reported that an increase in air content leads to a reduction in the compressive and the flexural strengths. It is found that an increase in air content by a percentage point leads to an average reduction of 2 to 6 percent in the compressive strength and 2 to 4 percent in the flexural strength. Qaraman et al (2016) and Algurnon (2013) stated that a number of factors influences entrained air. Examples of such as factors include the duration

of mixing and the nature and concentration of the surfactants used as air-entraining agents.

Carmel et al. (2003) concluded that the processes of air entrainment and air void stabilization in cement pastes depend on the surface activity of the surfactant available in solution. Lauryl glucoside (figure 1) is a natural sugar based surfactant. This surfactant as example of nonionic surfactants have hydrophobic /hydrophilic balance wherein there is nither a negative nor a positive charge in either parts of the molecule, thus giving it the nonionic terminology. These surfactants have the advantage that they are not affected by water hardness or by pH changes as the anionic surfactants. They are considered medium to low foaming agents and especially advantageous when a very low air- entraining agent is required.

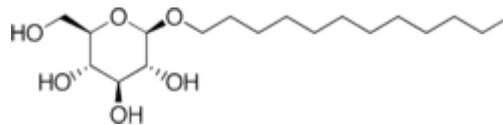


Figure 1: Lauryl glucoside (LG)

The objective of this work is to study the effect of nature and concentration of the nonionic surfactant LG on the air content and accordingly on the bulk density and the compressive strength of the cement specimens. In addition, the microstructure of the hardened cement samples has to be examined using X-ray diffraction and SEM techniques.

2. Experimental:

2. a. Materials:

Portland cement of mark CEM I 52.5N obtained from El-Arish cement factory. Its chemical composition is given in (Table (1)).

Lauryl glucoside, a nonionic surfactant is supplied from Merck and used as such.

Table (1): Chemical composition of the used ordinary Portland cement (OPC).

| Oxide (%) | OPC |
|--------------------------------|-------|
| SiO ₂ | 22.12 |
| Al ₂ O ₃ | 5.56 |
| Fe ₂ O ₃ | 3.69 |
| CaO | 62.87 |
| Na ₂ O | 0.26 |
| K ₂ O | 0.11 |
| Cl | 0.02 |
| MgO | 2.36 |
| SO ₃ | 0.91 |
| Free CaO | 0.92 |
| Ignition Loss | 1.22 |

2. b. Techniques and Instrumentation:

2. b. 1 Paste preparation:

The cement specimens are prepared by dissolving different concentrations of LG (W/C) is 0.43 as recommended (Carmel et al., 2003). The percentages of the surface- tent in 650 g H₂O then adding to 1500 g cement. The water to cement ratio surfactants used range from 0.005 to 0.090 % by weight of cement.

The mixing are carried out under continuous and vigorous stirring for about three minutes (Carmel et al., 2003). After complete mixing the resulted pastes is poured into (12 ×4 ×4 cm³) moulds. The moulds are kept at about 100% relative humidity at room temperature for one day. The hardened cement pastes are then removed from the moulds after they attained the final setting and cured under water for the rest of the hydration ages (up to 360 days).

2. b. 2 Compressive Strength:

Three specimens of each mix at different hydration times (3, 7, 28, 90 and 360 days) are used for examination the compressive strength of the pastes. The mean value of the three specimens at each hydration age is considered as the determined compressive strength. The strength test machine used is of point load taster (20063 cemasco S/N-Controls) type, Milano-Italy.

2. b. 3 Stopping of hydration:

This is performed after doing the compressive strength test by taking about 10 g of the crushed hardened pastes and putting into a beaker containing 100 ml of acetone/ethyl alcohol (1:1 by volume) to stop the hydration process. The mixture is stirred for 0.5 hr. The residue is filtered off, washed with ethanol and dried at 50° C for about 24 hrs. The dried samples are then stored in a desiccator for the following physico-chemical analysis.

2. b. 4 Determination of the bulk density of the cement pastes:

The bulk density is determined by measuring the weight of the sample in air and under water. The density is then calculated as mentioned in (ASTM Standard C 138-08 – 2008).

2. b. 5 Determination of the air entrained in the cement pastes:

The percent of air entrained in pastes was determined by the difference in weight of a hardened cement paste in absence and in presence of surfactants by the equation:

$$\text{Percent of air entrained} = \frac{A - B}{A} \times 100 \%$$

Where: A: The weight of hardened cement paste in absence of surfactant.
B: The weight of hardened cement paste in presence of surfactant.

2. b. 6 X-Ray Diffraction analysis (XRD):

XRD patterns of the samples are recorded by using a Philips X Pert MPD diffractometer using copper target with nickel filter under working conditions of 40-kilo volts and 20 milliamperes.

2. b. 7 Scanning Electron Microscopic (SEM) measurements:

The morphology and microstructure of the dried hydrated samples are studied using JEOL JXA 840 an electron Probe micro analyzer SEM. The specimens are coated with a thin film of gold, under vacuum evaporator with cathode rays then analyzed.

3. Results and Discussion

3. a. Effect of the presence of surfactant on the air content and the bulk density of cement pastes.

Air content is a controlling factor, which affects many physical and mechanical properties of cement pastes (e.g., bulk density, compressive strength, workability, etc.).

On adding an air entraining surfactant to cement pastes, its molecules are inserted between adjacent molecules at the water surface; the mutual attraction between the separated water molecules is reduced. Lowering the surface tension stabilizes the bubbles against mechanical deformation and rupture, making it easier for bubbles to be formed.

The values of the air content entrained in Portland cement pastes hydrated for 90 days in presence of different percentages of LG are determined. It should be mentioned that the content of air entrained in cement pastes show a slight change at the different hydration ages (From 3 up to 90 days). However, the air content exhibits the best value at 90 days of hydration. The results in Table (2) show a gradual increase in the air content with increasing surfactant concentration and reach a maximum value at a concentration of 0.09 %. However, the maximum air content is 11.81% on using the LG surfactant.

Table (2): effect of LG concentration on the air content and the density of the hardened cement paste at 90 days.

| Surf. conc. (Wt %) | 0.0 | 0.005 | 0.025 | 0.035 | 0.06 | 0.09 |
|------------------------------|------|-------|-------|-------|-------|-------|
| % of air entrained | 0.00 | 3.01 | 6.05 | 9.21 | 10.87 | 11.81 |
| ρ (g/ cm ³) | 2.00 | 1.94 | 1.91 | 1.82 | 1.79 | 1.77 |

The density of cement pastes is directly affected by the air content; they are inversely proportional to each other. The results show that the density decreases with increasing surfactant concentration, and reaches its minimum value at 0.09% wt of the surfactant. It is found that, the density decreases by about 0.3g/ cm³ when the air content is increased by about 14%. (Qaraman, 2016).

3. b. Effect of surfactants on the compressive strength of the hardened cement pastes.

Compressive strength of hardened cement is an important criterion specified in the design of lightweight cement pastes and should be considered equally important as density (Al-gurnon, 2013). The properties of these pastes are time-dependent, therefore, any test method on the cement pastes should be performed at a certain hydration age.

In this work, the determined values of the compressive strength of Portland cement pastes

in absence and in presence of the surfactant LG at different concentrations and different hydration ages are given in (figure (2)).

Generally, all mixes show an increase in the values of the compressive strength with increasing hydration time. This increase is believed to be due to progress of the cement hydration with time. In nearly most cases, the compressive strength increases on increasing surfactant concentration until reaches maximum at 0.025 % then decreases. The LG specimens show the highest compressive strength values (131,128,123, 115 N/mm²) at the concentrations (0.025, 0.035, 0.06 and 0.09 %, respectively). The optimal L G concentration is thus considered 0.025%.

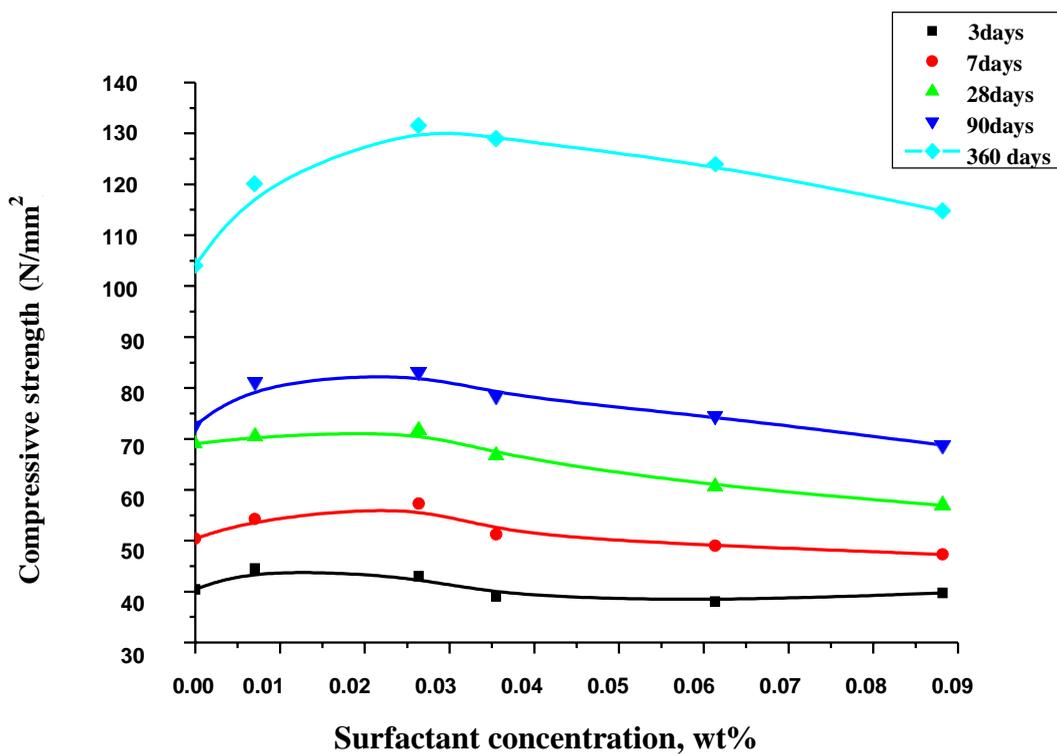


Figure (2): The compressive strength of hardened cement pastes containing LG at 3, 7, 28, 90 and 360 days.

3. c. Effect of surfactants on the phase composition of the hardened cement pastes.

The rates of change in physical and chemical properties of Portland cement pastes depend upon the rates of hydration of the components in cement. Both C3S (alite phase) and β-C2S (belite phase) react with water to form calcium silicate hydrate (CSH) and calcium hydroxide (CH). The CSH provides most of the strength developed by Portland cement. C3S hydration occurs more rapidly than C2S hydration. Therefore, C3S provides most of the early age strength while C2S contributes mostly to the later age strength (Gartner et al., 2002).

The phase composition of the various hardened Portland cement pastes in absence and presence of the surfactant LG is examined at the two hydration ages 7 days as an early hydration age and 360 days as a late age of hydration using 0.025 and 0.09% surfactant concentrations applying the X-ray diffraction technique. The reactants (C3S and C2S) and products (CSH and CH) of cement hydration, (both for the control mix and on using LG) are shown in figure (3). It can be observed that, the intensity of the characteristic peaks of the reactants such as alite and belite phases decreases with increasing the hydration age.

At 7 days, it is found that the intensity of the peaks characteristic of calcium silicate hydrate (CSH) increases on adding either 0.025 or 0.09 % L G surfactant compared with the blank specimens. This indicates that L G accelerates the hydration of the cement pastes in early ages.

After one year, the peaks characteristic of β -C2S (belite phase) disappeared completely and converted to the hydration products, calcium silicate hydrate (CSH) and calcium hydroxide (CH). The intensity of the CSH peak increases in the samples containing of 0.025 % L G, while in case of 0.09% concentration both CSH and CH peaks remain the same as in the blank.

Hence, it can be concluded that both 0.025 and 0.09 concentrations of L G accelerate the hydration reaction in the early age but after a year only the low dosage 0.025% accelerates the hydration reaction.

The ability of LG to accelerate the hydration reaction and accordingly improving the compressive strength may be refer to its ability to form a hydrogen bonds with the CSH network.

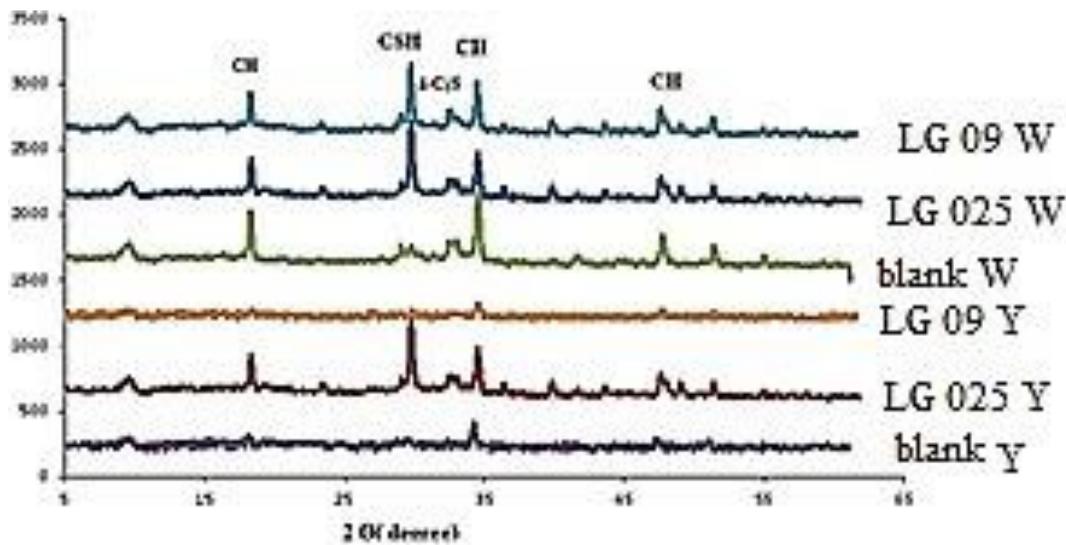


Figure (3): X-ray diffraction patterns of the control hardened cement paste and the hardened cement pastes with 0.025 and 0.09 % L G at 7 days (W) and 360 days (Y).

3. f. Effect of surfactants on the microstructure and morphology of the hardened cement pastes.

The scanning electron microscope (SEM) is a powerful tool for imaging and chemical analysis in cement research. With a high resolution and a large depth of focus, it enables a detailed study of surface topography of the rough surfaces of e.g. the formed calcium silicate hydrate (CSH) and calcium hydroxide (CH).

The micrographs of the hardened cement pastes in absence and presence of the surfactants after 7 days are shown in Figure4 (a &b). Figure (4-a) illustrates the hydration products formed in absence of surfactants. Calcium hydroxide appears as hexagonal plates and the ettringite needles exist beside the fibrous CSH phase. Addition of L G (figure 4-b) shows an increase in the amount of calcium hydroxide, which has a layered structure, appears beside CSH crystals.

(Figure 5) (a & b) shows the micrographs of the hardened Portland cement pastes in absence of surfactants and in presence of 0.025% L G after 360 days hydration. It can be noticed that the hydration products have more compact structure composed of calcium silicate hydrates, which explains the improvement in the strength after year for all mixes.

On other hand, the structure of cement paste with addition of L G produces consistent bubble structure with a uniform small air voids system (76.92 μm) it seems to be more compact. These findings may indicate that L G uniform the air void system beside its ability to accelerate the hydration reaction. This may help improving the compressive strength of the L G, these results are great confirm previous findings that non ionic surfactants uniform the air void system beside its ability to accelerate the hydration reaction (Qaraman ,2016).

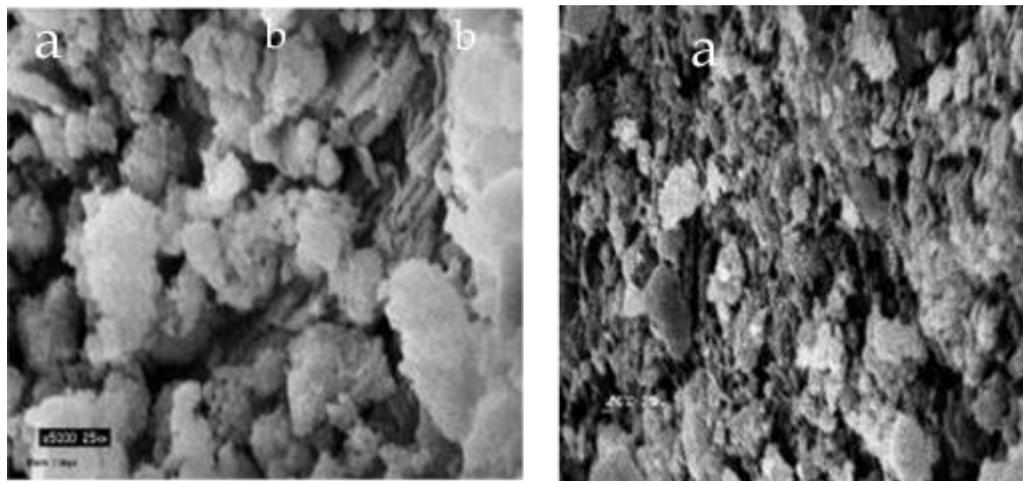


Figure (4): SEM of hardened cement pastes after 7 days hydration (X = 5000).
a) Without surfactant b) With 0.025% L G

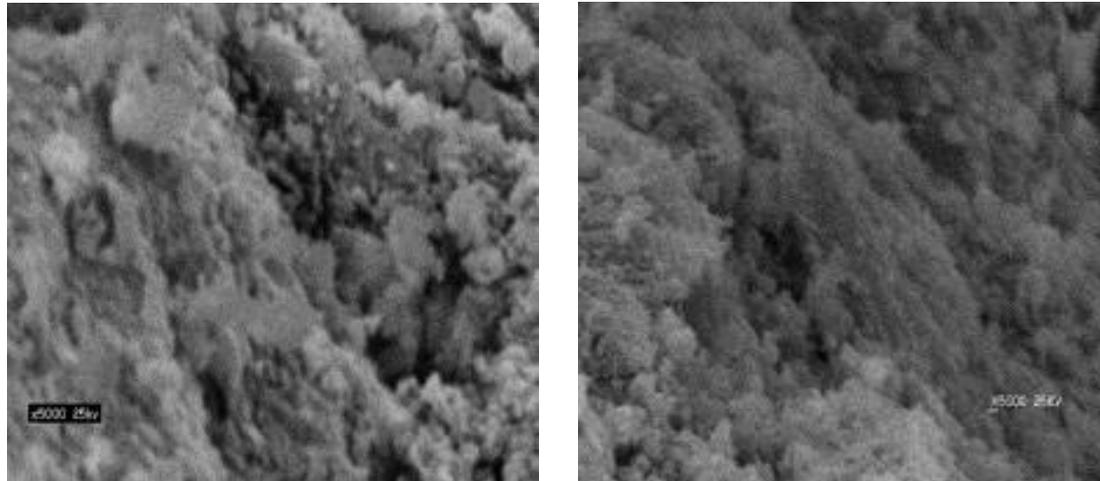


Figure (5): SEM of hardened cement pastes after 360 days hydration (X = 5000).
a) Without surfactant b) With 0.02% L G.

Conclusion and Future Work:

According to the obtained results, we can conclude that:-

1. L G improves the compressive strength indicating due its ability to accelerate the hydration reaction.
2. The best compressive strength of LG specimens is attained at LG concentration of 0.025 % by weight of cement.
3. SEM shows that LG causes a uniform distribution of small air voids, which may greatly improve the compressive strength of the pastes.

In our future work, we may concentrate an in depth study of other surfactant types on the durability and compressive of concrete and mortar. In general, the subject is quite interesting and the results are valuable to using a new surfactants as air-entraining agents to improve light-weight hardened pastes without significantly losing in compressive strength.

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