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The Effect of Waterborne Epoxy Resin Emulsion on the Physical Properties of Oil Well Cement

(Kesan Emulsi Resin Epoksi Bawaan Air ke atas Sifat Fizikal Simen Telaga Minyak)

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ABSTRACT

The main objective of this work was to investigate the influence of waterborne epoxy resin emulsion (WER) on the physical properties of oil well cement slurries. Cement slurries containing 5%, 10% and 15% of WER bwoc were compared with WER-free slurries. The rheological behavior was carried out according to API standard. Uniaxial compressive strength and shear bond strength of cement stone were evaluated at the ages of 24, 48 and 72 h. The experimental results illustrate that the addition of WER does not alter the rheological behavior. The addition of WER has increased the shear bond strength almost 52% at 24 h of aging for 10% WER bwoc when compared with unmodified slurry. The enhancement on shear bond strength was attributed to the mechanical anchoring and resin film forming at the interface.

Keywords: Mechanical properties; oil well cement; rheological properties; waterborne epoxy resin emulsion

ABSTRAK

Objektif utama penyelidikan ini adalah untuk mengkaji pengaruh emulsi resin epoksi bawaan air (WER) ke atas sifat fizikal sluri simen telaga minyak. Sluri simen yang mengandungi 5%, 10% dan 15% WER bwoc dibandingkan dengan sluri tanpa WER. Tingkah laku reologi telah dijalankan mengikut piawai API. Kekuatan mampatan unipaksi dan kekuatan ikatan ricih batu simen telah dinilai pada umur 24, 48 dan 72 jam. Keputusan kajian menunjukkan bahawa penambahan WER tidak mengubah tingkah laku reologi. Penambahan WER telah meningkatkan kekuatan ikatan ricih hampir 52% pada umur 24 jam untuk 10% WER bwoc apabila dibandingkan dengan sluri tidak diubah suai. Peningkatan pada kekuatan ikatan ricih adalah disebabkan oleh yang tambatan mekanik dan resin filem yang membentuk di antara muka.

Kata kunci: Emulsi resin epoksi bawaan air; sifat mekanik; sifat reologi; simen telaga minyak

INTRODUCTION

Cement materials are widely used in the oil and gas industry for cementing casing strings in the well. In cementing an oil-gas well, the cement slurry was pumped down into the casing string to the bottom of the well and then flows up through the annulus between the casing and the borehole wall where it sets. The cement sheath fulfills several important functions (Erik & Dominique 2006). It fastens the pipes in place. It seals the pipe, isolating and protecting water sources and productive formations. It can provide a counter-balancing force to force from high-pressure zones. It prevents loss of oil and gas into 'thief' zones. Most of all, the major goal of the cementing is to provide a complete and permanent zonal isolation of the well. This permanent isolation is expected not only during the oil production but also after the plugging of the wells (de Paula et al. 2014; Saout et al. 2006). The ability of improving bond strength at first interface and secondary interface are crucial to the long zonal isolation of the well. However, many of today's wells are still at risk due to interfaces bond failure or have sustained annular pressure, despite modern advances in well construction processes and materials (Cavanagh et al. 2007). The search for modified materials for cementing of oil well has increased worldwide. It is desirable that these modified materials can improve the physical properties and microstructure of cement (Choolaei et al. 2012; de Paula et al. 2014).

Waterborne epoxy resin emulsions have recently attracted tremendous scientific interest due to its remarkable and useful physical and chemical properties: High bonding strength. The polarities of hydroxyl and ether caused electromagnetism or chemical adsorption between the epoxy resin and its nearby surfaces. Moreover, the reaction exists on adjacent surface with metals containing active hydrogen to form chemical bond. Thus, the adhesive force is very strong; easy to work and operate. Either at room temperature or heated, it can be solidified. Besides, it is possible to be solidified rapidly under water or on moist surface; and excellent corrosion-resisting performance. Because the solidified epoxy resin molecules are connected high densely, the fatty hydroxyls contained in the molecules have no reaction with alkali (Chen et al. 2003). These characteristics can be used in cement composites to improve the interface bond strength between both pipe walls and the rock formation. Waterborne epoxy resin emulsions are now commonly used in numerous industrial applications.

Previous researchers reported that waterborne epoxy resin emulsions were found to improve strength properties of Portland cement. The obtained results have shown that the waterborne epoxy resin emulsions could provide chemical bonds between the ether and epoxy-based groups of the molecular structure of epoxy resin and the calcium silicate hydrate phase (CSH) of the cement matrix, which lead to the formation of special bridge bond force to meliorate the organization structure of the hardened cement (Chen et al. 2002; Liu 2000; Yu 1995). Recently, epoxy cement mortar has been widely used as a type of polymer cement mortar because of its high bond strength, toughness, water resistance, chemical resistance and good durability (Benzarti et al. 2006; Chen 2002; El-Hawary & Jaleel 2010; Ohama 1997).

However, the physical properties of oil well cement slurries containing waterborne epoxy resin emulsion have never been investigated to ensure its workability, which directly influences the preparation and performance of the cement sheath in the annular space, especially concerning the resulting interfaces shear-bond strength and the flow behavior. Therefore, the work described in this paper was initiated to study the effects of waterborne epoxy resin emulsions as admixture mixed with oil well cement on the physical properties of the oil well cement-WRE composites.

EXPERIMENTAL DETAILS

MATERIALS

The Class G oil well cement (GOC), with medium particle size 19.63 μm (Laser diffraction particle size analyzer LMS-24, Japan), was obtained from the Sichuan Jiahua Cement Company, China. The particle size distribution of the cement used in the present study is shown in Figure 1. The oxide composition of this cement (GOC) measured by XRF is presented in Table 1. Oil well cements are usually based Portland cement compositions, are particularly rich in silicate phase. According to American Petroleum Institute standards, tricalcium aluminate content of class G cement must be lower than 3%. Four principal mineral phase compounds of GOC used in this work-tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A) and tetracalcium alumino ferrite (C_4AF)-are 52.8%, 24.2%, 1.7% and 14.4%, respectively, as calculated by Bogue equations (Lota et al. 1998).

The waterborne epoxy resin emulsion (WER) used in this work were supplied by the Zhejiang Anbang New Material Development Limited Company, China. The solidifying agent (SA) is an alkaline matter which was made at own laboratory and was added by 15% of epoxy resin weight. The base properties of the resin and hardener are shown in Table 2.

CEMENT SLURRY PREPARATION

Slurries were prepared as specified by the American Petroleum Institute (API) (2002) to ensure a constant specific mass of 1.9 g/cm^3 and a water-cement ratio of 0.44. The sulfonated acetone formaldehyde poly-condensate was employed to improve the rheological property of the fresh cement slurry. The dispersion/cement ratio was 0.5% (bwoc). The fluid loss controller/cement ratio was 3% (bwoc), which can reduce water loss of cement slurry. The

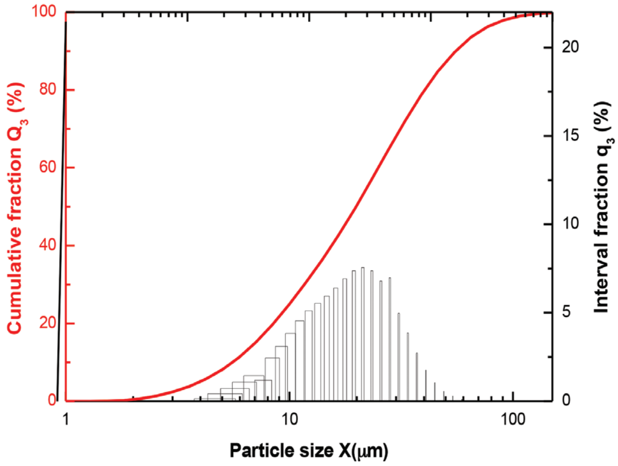


FIGURE 1. Particle size distribution of the Class G oil-well cement

TABLE 1. Oxide composition of the Class G cement (GOC) measured by XRF

Materials	Oxide content/ wt.%							
	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O+K_2O	LOI
GOC	64.05	22.32	3.63	4.73	1.30	2.48	0.46	0.81

TABLE 2. Physical characterization of waterborne epoxy resin emulsion (WER) and solidifying agent (SA)

Materials	Apparent description	Solid content /%	pH value (25°C)	Epoxy value /eq/100 g	Amine value /mgKOH/g	Apparent viscosity /Pa.s
WER	Milk white liquid	61	8	0.20	/	3
SA	Shallow brown liquid	48	12	/	280	12

retarder/cement ratio was 0.2% (bwoc), which was chosen because of it slows the hydration of cement. The defoamer/cement ratio was 0.05% (bwoc), which can remove foam from mixing. The rations of WERs were equal to 5%, 10% and 15% of the cement weight. Four different slurries were mixed and submitted to lab for experiments. Table 3 shows the composition of each experiment and the corresponding name which will be referred to in this paper. According to API standard, the fluids (water, filtrate reducer, retarder and de-foamer) were firstly mixed for 15 s at 6000 rpm, then the dry ingredients were added under the same 6000 rpm and it was mixed for 30 s at 14000 rpm. After mixing, the cement slurry was poured into atmospheric pressure thickening instrument at 90°C for 20 min in order to homogenize.

CEMENT SLURRY RHEOLOGICAL CHARACTERIZATION

The Fann Rotational Viscometer was used in this study with a muff heater to keep the temperature constant and atmospheric pressure (API 2002). The cement slurry obtained after stirring in an atmospheric consistometer at 90°C was poured into the annular space between two concentric cylinders of the FANN viscometer. The viscosity readings were taken at rotations of 300 rpm (θ_{300}), 200 rpm (θ_{200}), 100 rpm (θ_{100}), 6 rpm (θ_6) and 3 rpm (θ_3). It was generally accepted that cement slurry can be typified by the yield power law model (Guillot & Denis 1988). The yield-power-law model relates shear stress and shear rate by (1)

$$\tau = \tau_y + K\gamma^n, \tag{1}$$

where τ is the shear stress; t_y is the yield point; n is the flow behavior index; and K is the consistency index. According to yield-power law model, the rheological parameters, including K, n and t_y were calculated from 300, 100 and 3 rpm readings using (2),

$$\begin{aligned} &\tau_y = 0.511\theta_3 \quad \text{(Pa)} \\ &n = 3.322\lg[\theta_{300} - \theta_3)/(\theta_{100} - \theta_3)] \qquad (2) \\ &K = 0.511[\theta_{300} - \theta_3)/511\text{n} \quad (\text{Pa}\cdot\text{s}^n), \end{aligned}$$

where θ_{300} is the 300 rpm dial reading; θ_{100} is the 100 rpm dial reading; and θ_3 is the 3 rpm dial reading.

CEMENT STONE MECHANICAL PROPERTIES

The mechanical properties studied include uniaxial compressive strength and shear bond strength in this study. Before starting the test, the cement slurry was degassed and homogenized in an atmospheric consistometer (Model 1250 from Ametek Chandler Engineering, Broken Arrow, OK). Then, the cement slurry was poured into moulds (Figure 2(a) and 2(b)) and cured in a water bath at 90°C and atmospheric pressure for 24, 48 and 72 h. For uniaxial compressive strength test, the 50.8*50.8*50.8 mm cube specimen was prepared. For shear bond strength test, the cement slurry is initially placed inside a coaxial cylindrical mold with a height (h* d_o) of 50.8*50.8 mm and internal core or steel column diameter (d) of 25.4 mm (simulating first interface or secondary interface). Figure 3 represents the working mechanism of the shear bond strength test system. The mechanical properties test was carried out on 3000 kN compressive testing machine (Figure 2(c)) and the tests were performed at a constant loading rate of 71.7±7.2 kN/min at room temperature. In order to help minimize statistical error, the strength results were the average of four specimens for each condition.

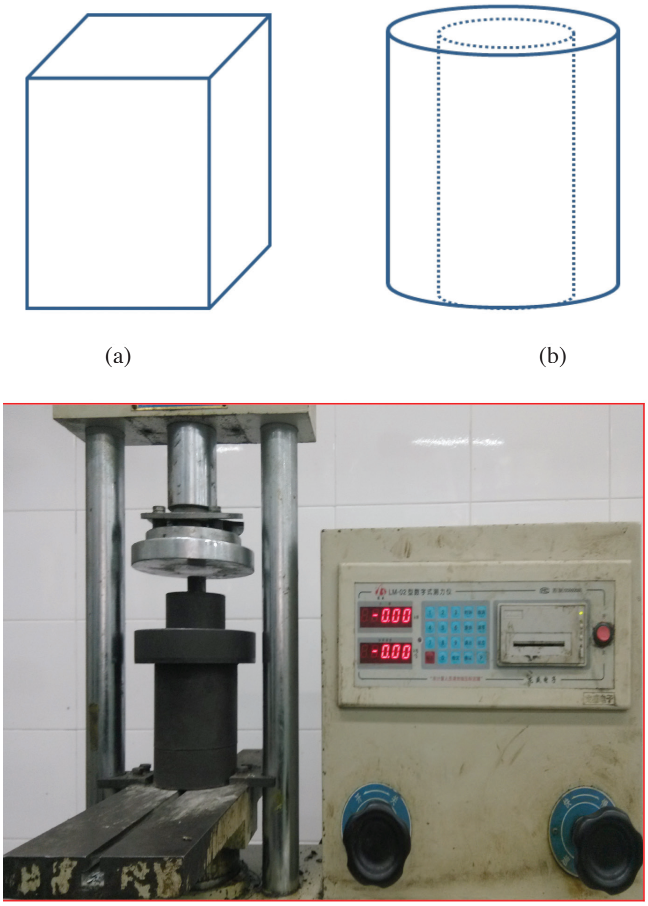
Based on the test data, the uniaxial compressive strength and shear bond strength can be calculated using (3) and (4), respectively.

$$P_1 = F/50.8^2, \tag{3}$$

TABLE 3. Composition of the cement slurries in this study

Sample abbreviation	Sample composition
S05	Cement+water+0.5%A[a]+3%B[b]+0.2%C[c]+0.01%D[d]
S05W05	Cement+water+5%WER+0.5%A+3%B+0.2%C+0.05%D
S05W10	Cement+water+10%WER+0.5%A+3%B+0.2%C+0.05%D
S05W15	Cement+water+15%WER+0.5%A+3%B+0.2%C+0.01%D

[a] A_Cement friction reducer: Sulfonated acetone-formaldehyde poly-condensate, [b] B_Fluid loss controller: AMPS-based polymer, [c] C_Retarder: AMPS-based polymer, [d] D_Antifoam: Polyethylene glycol glyceryl ether



(a)

(b)

(c)

FIGURE 2. Used for testing the mechanical properties (a) schematic of uniaxial compressive strength specimen, (b) schematic of shear bond strength specimen and (c) mechanical properties tester

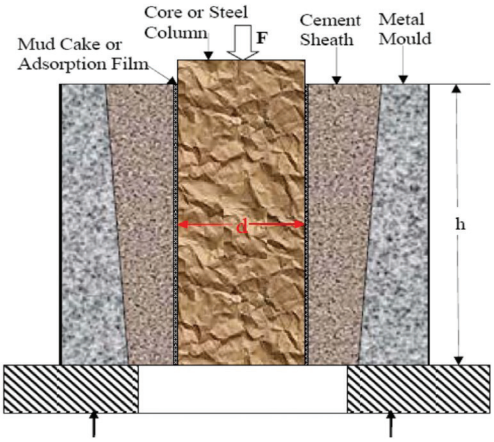


FIGURE 3. Schematic of shear bond strength testing system

$$P_2 = F/\pi dh, \tag{4}$$

where P_1 *and* P_2 are the uniaxial compressive strength and shear bond strength (MPa), respectively; F is the maximum load (N); h is the height of mould (mm); and d is the diameter of core or steel column (mm).

Results and Discussion

RHEOLOGICAL CHARACTERIZATION OF WER-MODIFIED CEMENT SLURRY

The basic rheological properties are associated with the performance of cement slurry. Water-cement suspension is a multi phase complex system. Often, the yield power law (Herschel-Bulkley) model best fits the rheological properties of cement slurries. The yield-power-law model of flow differs most notably from a Newtonian fluid by the presence of a yield stress. A yield-power-law fluid will not flow until the applied shear stress exceeds a minimum value that is known as the yield point (Caenn et al. 2011). Table 4 summarizes the rheological properties evaluated directly or indirectly by the viscosity readings as well as the units of measurement. The properties present in Table 4 are yield point (τ_y), flow behavior index (n) and consistency index (K). These parameters are responsible for the load loss by friction, with a fundamental influence on cementing displacement. The analysis of these results indicates that no significant change in the rheological behavior of the fresh cement slurries was observed after the addition of WER by the use of a lignosulfonate based dispersant. All

TABLE 4. Rheological parameters for the designed cement slurries

	Dial readings			
	S05	S05W05	S05W10	S05W15
Rotation per minute (rpm)				
300	190	176	168	156
200	153	142	131	125
100	115	106	101	94
6	28	25	23	21
3	23	21	20	18
n	0.8602	0.8668	0.8696	0.8606
K (Pa·s^n)	0.3994	0.3558	0.3337	0.3291
τ_y (Pa)	11.7530	10.7310	10.2200	9.1980

the experimented slurries showed a low k value, which are preferred for oil well cementing and provide good pump ability and easy annulus penetration. The WER-modified slurry presented lower τ_y values than the corresponding slurries prepared without WER addition. These results are in agreement with previous study because the cement slurry happened during thixotropic and hydration (irreversible) process at the same time as soon as the cement and water were mixed together (Jarny et al. 2005).

MECHANICAL PROPERTIES OF WER- MODIFIED CEMENT SAMPLES

The mechanical properties of the set cement sample are one of the most important characteristics of oil-gas well cementing, because it is related to the preserve well structure and the interface sealing properties of cement sheath and helps to extend service lifetime. In specific cases, it could be a significant support to resist again cement failure caused by wellbore stress.

UNIAXIAL COMPRESSIVE STRENGTH (MPA)

The uniaxial compressive strength of WER-free and WER-modified slurries is shown in Figure 4. The values are the average compressive strength of four specimens. As expected the compressive strength of WER-modified cement sample increased with the increase of the cure time and decreased with the increase of the WER percentage in all mixes and under all cure conditions. It can be seen that the compressive strength of the sample S05 is higher than that of the other samples under the same cure time and the compressive strength of the sample S05W15 is the lowest. As shown in Figure 4, the test results of all specimens show a decrease in compressive strength with an increase of the WER percentages under the same cure time, the biggest strength drop ratio is 67.5%, occurring for sample S05W15 curing for 12 h and the smallest strength drop ratio is 43.3%, occurring for sample S05W15 curing for 72 h. The longer the cure time, the higher the compressive strength of the WER modified samples. This may be attributed to the fact that the long cured time is helpful for the cement hydration and the formation of the polymer film in cement samples. Compared with the sample S05, the sample S05W15 contains a certain amount of WER. This indicates that a certain amount of polymer may have a negative effect on the compressive strength. The early age compressive strength of WER-modified samples is lower than that of unmodified sample. This could be attributed to the reason that WER can be adsorbed on the un-hydrated particles and the hydrating compounds, as a result, the early hydration of cement is inhibited in the presence of WER. On the other hand, the polymer occupies some volume in the set cement sample, which contributes little to the compressive strength compared with the calcium silicate hydrate gel. Similar conclusions were drawn by Wang using styrene-butadiene rubber emulsion modified cement mortars (Wang et al. 2005). Compressive strength of the set cement sample was used as a way to determine the degree of hydration reaction. As can be seen clear in the results, the addition of WER in oil-gas well cement slurry can influence the degree of hydration reactions and a higher WER addition cannot simply achieve a higher mechanical performance.

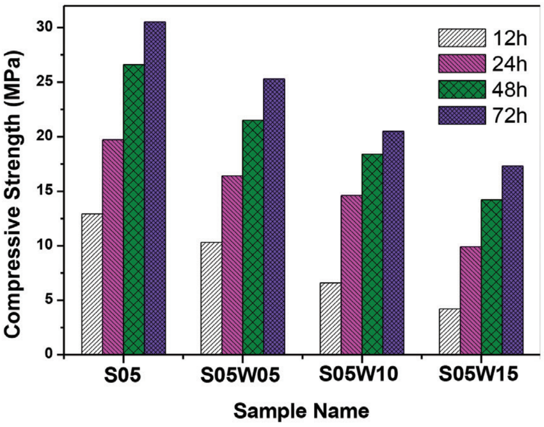
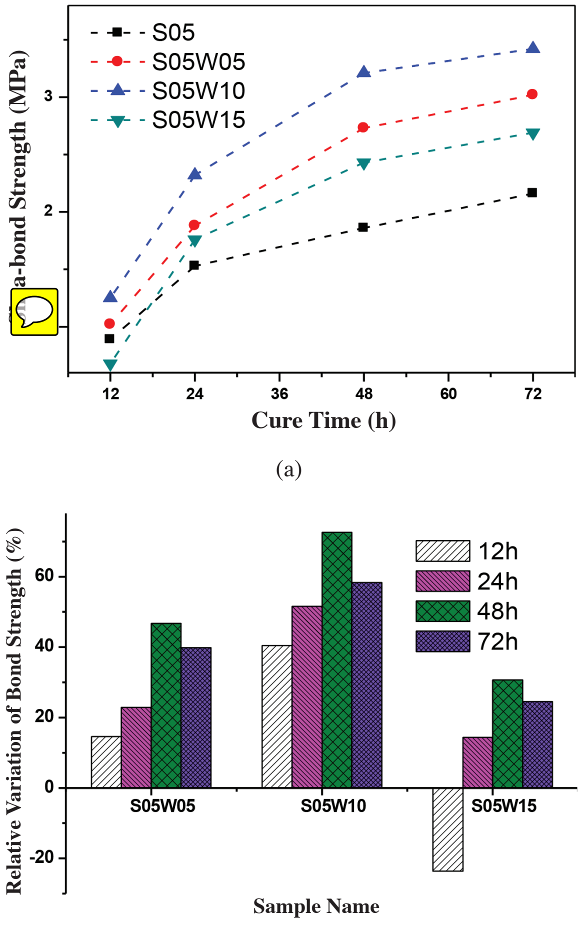


FIGURE 4. Compressive strength of WER-free and WER-modified slurries with different WER/cement mass ratios

SHEAR BOND STRENGTH (MPA)

The shear bond strength at the simulated first interface (at the interface between casing and cement) and secondary interface (at the interface between cement and formation) are shown in Figures 5(a) and 6(a), respectively. It was obvious from the current study that the addition of WER enhanced the shear bond strength for samples S05W05 and S05W10 when compared to the reference mix (S05). WER-modified slurry can form a continuous resin film, which has excellent toughness and binding performance. Sample S05W15 has also presented an improvement of shear bond strength after 24 h curing. Figures 5(b) and 6(b) indicate the variation of shear bond strength due to sample modification with different WER.



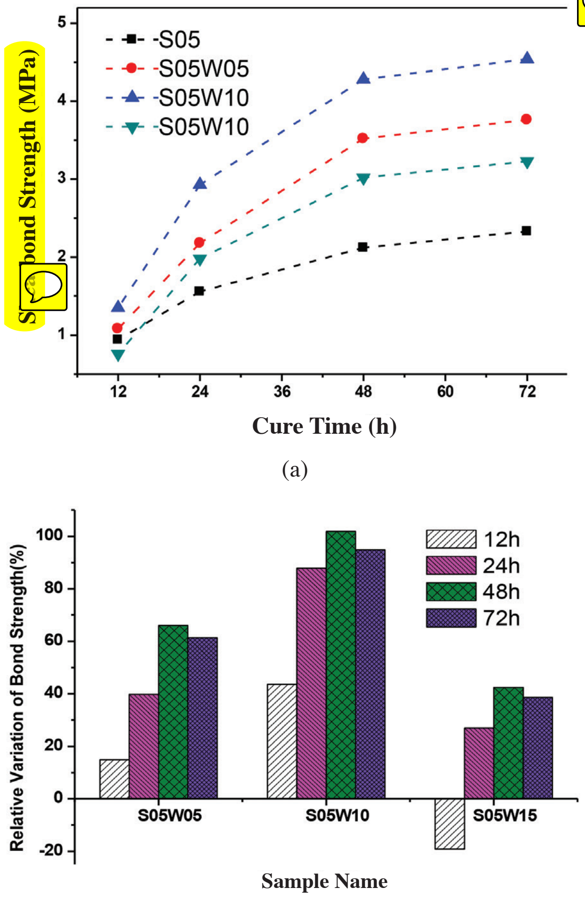
(a)

(b)

FIGURE 5. (a) Shear-bond strength of WER-modified slurries with different cure time, (b) variation of shear bond strength due to WER modification at the interface between cement/steel column

The obtained results showed that the shear bond strength was increased due to the increase in cure time from 12 to 72 h in all samples. This may be attributed to the long cure time which was helpful for the cement hydration and the formation of the polymer film in mortars. For example in S05W05, an increase of 14.6, 22.9, 46.8 and 39.8% were noticed after 12, 24 48 and 72 h curing when compared to S05 at first interface, 14.9, 39.7, 66.0 and 61.4% at secondary interface, respectively. However, for example in S05W15, an increase of -23.6, 14.4, 30.7 and 24.5% were noticed after 12, 24, 48 and 72 h curing when compared to S05 at first interface, -19.2, 26.9, 42.5 and 38.6% at secondary interface, respectively. The sample of S05W15, prepared using high addition amount of WER, however, the shear bond strength rises slightly. This could also be attributed to the reason that WER can be adsorbed on the un-hydrated particles and the hydrating compound, as a result, the early hydration of cement is inhibited in the presence of WER. The above results suggested that the shear bond strength of the WER-modified samples can be improved markedly at WER/cement below 10%. The higher the WER/cement ratio, the better are the shear bond strength for the WER/cement ratios smaller than 10%. When WER/cement ratio is higher than 15%, the effect on the shear bond strength is slight.

Compared with Figures 5 and 6, it was found that the shear bond strengths at secondary interface are remarkably higher than that at first interface no matter what the curing time in general. In order to properly address the difference, we have summarized in a schematic model as shown



(a)

(b)

FIGURE 6. (a) Shear-bond strength of WER-modified slurries with cure time and (b) variation of shear bond strength due to WER modification at the interface between cement/core

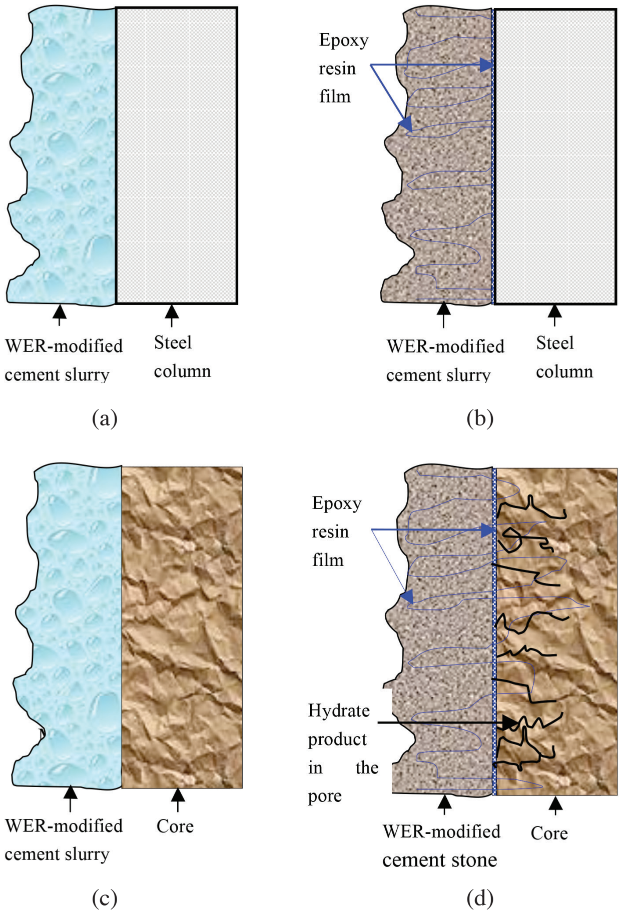


FIGURE 7. Schematic representation of types of interaction interfaces in different conditions (a) Before set, WER-modified cement/steel column, (b) after set, WER-modified cement/column, (c) before set, WER-modified cement/core and (d) after set, WER-modified cement/core

in Figure 7. Such representation is endorsed by widely reported analogous models in the literature (Isenburg & Vanderhoff 1974; Sakai & Sugita 1995). The different interface characteristics can be observed. The filter liquid of cement slurry permeates into the core pores, cement hydrate products and resin can precipitate inside the pore of core and promote a mechanical anchoring at the interface between cement and the core when compared the interface between cement and steel column. In summary, mechanical anchoring and resin film forming are significantly increase the shear bond strength at cement/core interface, which will be of benefit to the long zonal isolation of the oil well (Jenni et al. 2003; Mansur et al. 2009).

CONCLUSION

The work presented here showed that WER addition does not alter the rheological behavior of the oil well cement slurries and has a crucial influence on mechanical properties. The specimens with WER addition show a slightly decrease in uniaxial compressive strength with an increase of the WER percentages when compared with unmodified specimens, primarily due to the fact that WER can be adsorbed on the un-hydrated particles and the hydrating compounds, which inhibits the hydration of cement. On the other hand, the addition of WER was highly beneficial in improving the shear bond strength due to the mechanical anchoring and resin film forming at the interface. WER has great potential for developing new oil well cement modified materials.

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