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Preparation and characterization of epoxy/slag composite for cement slurry applications

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ABSTRACT: In this study, epoxy/slag composites (ESCs) were prepared by using diglycidyl ether of bisphenol-A epoxy resin, water-quenched granulated blast furnace slag as filler, phenolic aldehyde amine as hardener, and titanate as coupling agent. The properties of ESC, including chemical structure, thermal stability, wetting properties, and morphological structure, were investigated by using Fourier transform infrared spectroscopy, thermogravimetric analysis, a contact angle meter, scanning electron microscopy, and energy dispersive spectrometry. The results show that ESCs possess excellent thermal stability, hydrophilicity, and good compatibility with cement slurry compared to pure epoxy. In addition, the applications of ESC in a cement slurry were also investigated. It was found that the fluidity, free water, fluid loss, and content of $Ca(OH)_2$ decreased, while the compressive strength increased with the incorporation of slag into the epoxy matrix. These features were attributed to the pozzolanic reaction of slag by consumption of $Ca(OH)_2$ to form calcium silicate hydrate (C-S-H) gel which contributed more to the compressive strength of set cement. Finally, lightweight cement containing ESCs exhibited high strength without affecting the density of the light cement slurry under curing pressure and at high mixing rate compared with lightweight cement made of floating beads. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 43359.

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INTRODUCTION

The use of cement slurry with conventional density (1.7–1.9 g/cm^3) in completing highly depleted reservoirs and weak formations of oil frequently results in challenges of lost circulation and damage to the permeability of formations due to the higher hydrostatic pressure they exert on the well bore.[1] Therefore, a lightweight cement slurry is required for cementation in such wells to reduce the hydrostatic pressure of the fluid column.

Historically, three types of lightweight cements have been used to reduce the hydrostatic pressure on weak formations and to cement lost-circulation zones: water-extender cements, foam cements, and hollow-microsphere cements. They each have distinct advantages and also limitations when compared to one another. For example, water-extender cements are limited in density to nearly 1.38 g/cm^3, and the slurry is not stable.[2] Advantages of foam cements include good ductility and little fluid loss and gas migration, especially for excellent compressive strength development at low slurry density. However, the disadvantage of foamed cement is the need for specialized cementing equipment for both field applications and laboratory testing, which increases construction costs.[3] The idea of mixing floating beads or hollow microspheres with the cement was verified to reduce the cement density to as low as 0.90 and 1.02 g/cm^3 while yielding the highest compressive strength and the lowest permeability of any lightweight cement slurry.[4] Unfortunately, common floating beads are so sensitive to pressure that an increasing portion of these microspheres crush as the pressure increases during slurry displacement, which has an adverse effect on the density, rheology, pumpability, and stability of the cement slurry.[5] Currently, the use of hollow microspheres leads to less crushing. Nevertheless, it is high in price, which limits the potential field applications.[6] Accordingly, it becomes necessary to develop a novel lightweight additive characterized by light density, pressure insensitivity, good compatibility, and low cost for lightweight cement slurry applications.

Nowadays, the concept of forming composites using polymers and inorganic materials has received a significant amount of attention, especially for epoxy composites, due to the advantages of light weight, good mechanical properties, high modulus, outstanding thermal stability, good chemical stability, and so on.[7–9] Owing to the light weight and good mechanical properties, we consider using epoxy composites as lightweight additives for a cement slurry. However, as a kind of cementing

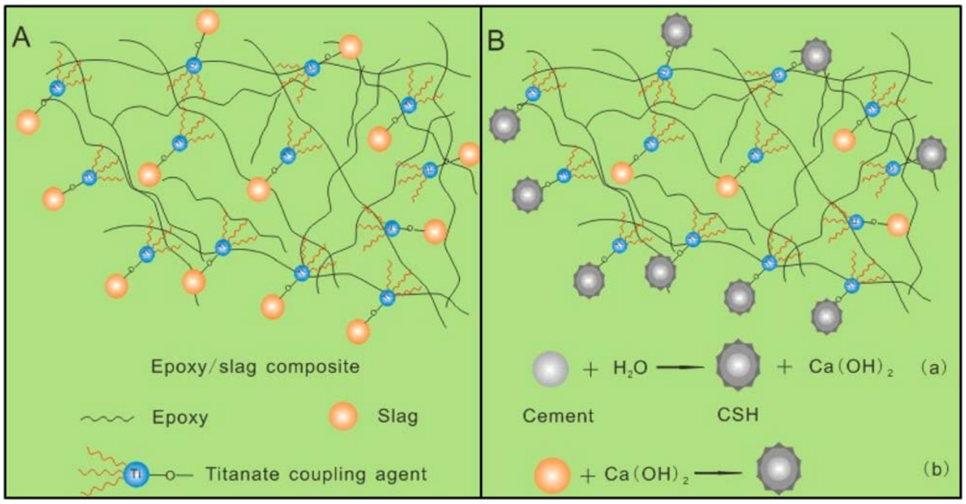

Figure 1. Schematic diagram of (A) epoxy/slag composite; (B) and epoxy/slag composite in cement slurry. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

additive, we anticipate that if the epoxy composites could react with the cement or cement hydrates, the addition of epoxy composites to the cement slurry would contribute to the development of lightweight cement slurry. Because these specific features in the development of epoxy composites are based on the addition of various fillers into pure epoxy, fillers are a key concern in preparing specific epoxy composites for applications in cement.[10]

Water-quenched granulated blast furnace slag is a byproduct of the metallurgical industry, being used as a kind of common supplementary cementitious material due to its potential hydraulicity.[11,12] When added to Portland cement, the activity of slag is stimulated by calcium hydroxide (CH) and could react with CH to form calcium silicate hydrate (C-S-H) gel. This reaction will contribute to an improvement in compressive strength.[13,14]

In this paper, a novel lightweight material, an epoxy/slag composite, has been synthesized. The composite is composed of an epoxy matrix, a coupling agent, and water-quenched granulated blast furnace slag. It is then cured to form an epoxy/slag composite, as shown in Figure 1(A). The coupling agent acting as a bridge is used to connect the organic epoxy resin with the inorganic slag. It was hoped that the pozzolanic characteristics of the slag as filler would increase the mechanical behavior.

To the best of our knowledge, no work was conducted previously to investigate epoxy/slag composites and their applications in cement slurry as a lightweight material. Therefore, the objectives of the present study are as follows. (1) Epoxy/slag composites were prepared. (2) The chemical structure, thermal stability, wetting property, fracture morphology, and chemical compositions of epoxy/slag composites were investigated. (3) The performance of cement slurry and set cement using the epoxy/slag composites as lightweight agents was studied.

EXPERIMENTAL

Materials

Diglycidyl ether of bisphenol-A epoxy resin was obtained from Nan Tong Xing Chen Synthetic Material Co., Nantong, China, in which an epoxy equivalent of 210–140 g/mol was used. The phenolic aldehyde amine hardener (HD) with an amine value of approximately 500 mg KOH/g was produced by Guang Zhou Hai Qi Trading Co., Guangzhou, China. The titanate coupling agent (TCP) was supplied by An Hui Tai Chang Chemical Co., Anhui, China, in which two different groups existed. One is a hydrophilic group that could react with inorganic fillers, such as alkoxy with short chains. The other is a hydrophobic group that could wrap or crosslink with the epoxy resin to improve the compatibility of inorganic fillers and epoxy resin, such as alkyl long chains. Therefore, TCP could bridge the inorganic fillers with the organic resin perfectly. Class G Portland cement (G-PC) was obtained from Si Chuan Jia Hua Enterprise Co., Leshan, China. Water-quenched granulated blast furnace slag was supplied by Nanjing Boyang Chemical Co., Nanjing, China. Micro silica powder (MS, <40 μm, silicon dioxide content greater than 95%) was purchased from Cheng Du Jia Mao Chemical Co., Chengdu, China. The specific minerals and content of the slag used in the research were CaO (30–42%), SiO_2 (35–38%), Al_2O_3 (10–18%), MgO (5–14%), and SO_3 (<4%). Floating beads (FB) including minerals (58% SiO_2, 33% Al_2O_3, 4% Fe_2O_3, 2.4% MgO, 2.6% CaO) were provided by Xinyang Jinhualan Mining Co., Xinyang, China. The density of FB used in this work is 0.5–0.7 g/cm^3. The dispersant SXY was obtained from Cheng Du Chuan Feng Chemical Engineering Co., Chengdu, China. SXY is a kind of sulfonated aldehyde-ketone condensation polymer compound. The fluid loss agent SWJ-4 was supplied by Shandong Wald Oilfield Technology Co., Jinan, China.

Methods

Preparation of Epoxy/Slag Composites. The preparation of epoxy/slag composites was as follows. First, 100 g of epoxy resin and 1.5 g of TCP were mixed and heated at 80°C with an electric shear mixer at 1000 rpm until a homogenous and watery liquid was obtained. After adding the desired amount of slag (0, 15, 30, 45 wt %) in the next step, the mixture was vigorously stirred at 80°C for 30 min to obtain the viscous and opaque liquid. Then, 15 g of HD was added to the mixture, followed by hand stirring for 10 min, and the well-mixed epoxy resin was then slowly poured into the homemade mold and cured at 95°C for 30 min. If desired, the powdery sample was obtained from cooling, demolding, crushing, and sieving (100 mesh) for the following analysis and tests.

Preparation of Cement Slurry. The cement slurry was prepared according to the following procedure.[15] Each sample was dry-blended, then blended with water for 15 s at 4000 rpm in a standard American Petroleum Institute (API) blender (Beijing, China) and then mixed at 12,000 rpm for 35 s.

Characterization

Fourier Transform Infrared Spectroscopy. The Fourier transform infrared spectroscopy (FTIR) spectrum was obtained with a Perkin Elmer RX-1 spectrophotometer (Beijing Reili Analytical Instrument, Beijing, China) in the optical range of 400–4000 cm^{-1}; its resolution was 4 cm^{-1} and the scanning number was 32. The KBr pellets were prepared with the samples.

SEM and EDS Analysis. The morphology of the fractured surfaces of the neat cured epoxy matrix (ESC-0) and the epoxy/slag composites was investigated with a Quanta 450 environmental scanning electron microscope (FEI, Hillsboro, U.S.A). The samples were attached to a glass slide using carbon tape and then sputtered with gold to reduce charging effects. In addition, energy dispersive spectrometry (EDS) testing was performed on the surface of the samples to investigate the chemical composition.

Thermal Analysis. Thermogravimetric analysis (TGA) was carried out using a Netzsch STA 449F3 (Netzsch, Selb, Germany). The ESC samples of 10.13 mg each in an alumina crucible were used for the TGA test with a heating rate of 5°C/min at atmospheric pressure, and the temperature range was from 40 to 800°C.

Contact Angle Measurements. To analyze the surface wettability, contact-angle measurements were performed on ESC-0 and the epoxy/slag composites (ESC-15, ESC-30, ESC-45) with a contact angle meter (HARKE-SPCA, Beijing, China). The samples were cut to 1 cm^2 size and then placed on the sample platform. Distilled water (0.8 μL) was dropped onto the surface of the sample with a microsyringe. This process must be very carefully done to ensure the water drop is fixed on the sample surface. A video camera captures the images of the drop when it is static and measures the contact angles automatically. To minimize errors, the average value of the contact angles of the drop were calculated approximately 30 s after the deposition from at least three parallel experiments.

Evaluation of Cement Slurry and Set Cement. The evaluations of the cement slurry and set cement were performed according to standard GB/T 19139-2012 issued by the China National Standardization Management Committee.

The prepared cement slurry was homogenized by using an OWC-2250B atmospheric consistometer (Shenyang Petroleum Instrument Research Institute, Shenyang, China) at 70°C for 20 min. The routine cement tests were performed as follows.

The fluidity was characterized by measuring the diameters of the cement pastes using a cone (60 mm height, 36 mm top diameter, 60 mm bottom diameter). The maximum diameter of the spread sample and the maximum width perpendicular to the diameter were measured. The average of these two values was defined as the fluidity.[16] Before the test, the slurry was stirred for 30 s.

The free-water test was designed to measure water separation using a 250-ml graduated cylinder. The duration of the test was 2 h at 20°C according the API 10B-2 procedure.

The fluid-loss test for cement slurry was performed using a high-temperature, high-pressure filtration press (Qingdao Tongchun Petroleum Instrument Co., Qingdao, China) after the slurry was homogenized using an atmospheric consistometer at 70°C for 20 min. The test continued for 30 min at 70°C and 6.9 MPa using nitrogen as the pressurizing gas.

To determine the compressive strength, the set cement cubes were removed from the molds and placed in a hydraulic press, where increasing force was exerted on each cube until failure, in compliance with API specifications for oil well cement testing.

All of these results were reported as an average of three parallel experiments.

X-ray Diffraction. The X-ray diffraction (XRD) patterns of the set cement samples containing ESC-15, ESC-30, and ESC-45 were recorded at 2θ angles of 5° to 70° in 15 min using an X Pert PRO MPD X-ray diffractometer (PANalytical, Almelo, Netherlands).

The preparation of the set cement samples for XRD analysis was as follows. First, cement slurries containing ESC-15, ESC-30, and ESC-45 were prepared according to the formulation 600 g G-PC + 30% ESC + 1% SXY + 1.5% SWJ-4 with W/C = 0.6 (the dosage of mixtures is based on the weight percentage of G-PC) as described in the section "Preparation of Cement Slurry." The blank cement containing all the mixtures except for the ESC samples was prepared as a control. Then the cement slurry was poured into the standard cube molds (5 cm × 5 cm × 5 cm) and cured at 90°C in the thermostatic water bath for 2 days. Afterwards, the set cement was broken, and the small pieces in the central part of the specimens were selected to soak in anhydrous ethanol to inhibit hydration for 2 h, and then they were ground and dried overnight at 90°C for XRD analysis.

TGA and Differential Scanning Calorimetry (DSC). The thermal analysis of the set cement samples without ESC and containing ESC-15, ESC-30, and ESC-45 was investigated with a TG/DSC simultaneous thermal analyzer (STA449F3 purchased from NETZSCH). The measurement was conducted under dynamic air atmosphere in the temperature range 40–1000°C, maintaining a heating rate of 10°C/min.

Details of the preparation procedure of the set cement samples for TG/DSC analysis were the same as for the XRD analysis in the previous section.

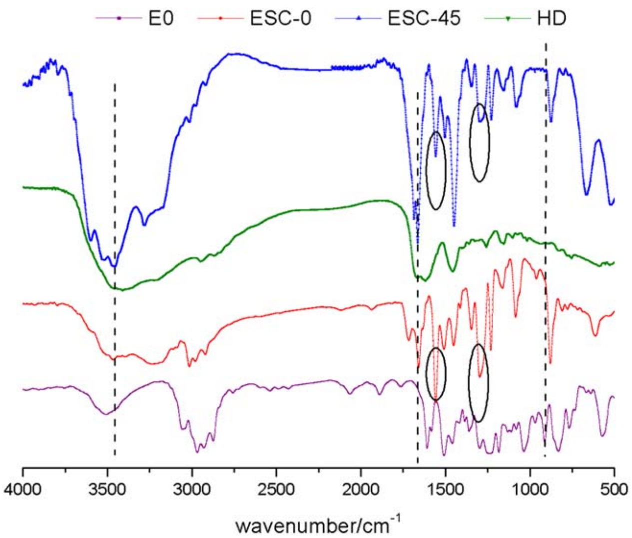

Figure 2. IR spectra of E0, ESC-0, HD, and ESC-45. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

RESULTS AND DISCUSSION

FTIR Analysis

Figure 2 exhibits the IR spectra of neat epoxy matrix (E0), cured epoxy (ESC-0), hardeners (HD), and epoxy/slag composite (ESC-45). The absorption peak at 915 cm^{-1} definitely refers to the characteristic peak of the epoxy group in E0 (see the spectra of E0). However, it disappeared after curing, while a strong and wide absorption band can be found around 3500 cm^{-1}, which is attributed to the OH in ESC-0 and ESC-45, which indicated the ring-opening reaction of the epoxy group to form OH in the process of curing. In addition, the strength of the bending vibration absorption peak of NH at 1600 cm^{-1} in ESC-0 and ESC-45 was weaker than that in HD. A reasonable explanation for this is the reaction of reactive hydrogen and epoxy groups and the primary amine group is reduced, which led to the reduction in strength of the absorption peak of NH. All of these characteristic peaks confirmed the successful preparation of ESC.

Thermal Analysis

Figure 3 shows the TGA results of slag and ESC with different loadings of slag (ESC-0, ESC-15, ESC-30, ESC-45). It can be seen that the weight loss of slag is less than 4% in the whole temperature range (50–800°C), which indicates that slag has excellent thermal stability because it contains mostly metal oxides that are not easily thermally evaporated. From 50°C to about 365°C, there is a minor weight-loss gap between the ESC samples with different loadings of slag. However, the degradation decompositions of ESC samples are shifted to higher temperature with the addition of slag at above 50% weight loss. This could be attributed to the protecting effect of slag against oxygen attack, which would provide a more resistant surface and network for the epoxy matrix.[17] The char yield value of the slag specimen at 800°C under nitrogen was 12.1%, 20.1%, and 27.5% in ESC-15, ESC-30, and ESC-45, respectively, while the ESC-0 specimen was 0.48% under the same test conditions. This was due to the slag migration to the char surface and the formation of a slag-protecting layer in the char surface, resulting in prevention of weight loss of the ESC samples with higher slag.[18]

SEM and EDS Analysis

Typical SEM observations of the fracture surfaces of ESC-0 and ESC-45 are shown in Figure 4. From Figure 4(a) it can be seen that the fracture character of the ESC-0 specimens has a river pattern, and the cracks are smooth. The direction of the fracture concentrates, which indicates that the crack resistance is low, and there is almost no plastic deformation. The fracture behavior is typical of brittle fracture.[19] In Figure 4(b), the fracture surface of ESC-45 is more dense, and the fracture orientation tends to be scattered. In addition, the fracture appears as an island-like structure, which is conducive to resisting and absorbing impact energy, showing the characteristics of ductile fracture. In addition, EDS was conducted on the surface of ESC-0 and ESC-45 (Figure 5). In ESC-0, the sample is mainly composed of oxygen, nitrogen, and carbon, and the surface texture is dense, whereas for ESC-45, a small amount of aluminum, magnesium, silicon, calcium, and iron can be found in the specimen, and these elements are exactly the ingredients of a water-quenched granulated blast furnace slag. This is why the surface of ESC-45 is more rough than that of ESC-0, and it is also the best proof of successful preparation of the epoxy/slag composite.

Wetting Properties

The contact angle of the ESC samples with different amounts of slag, namely condensed ESC-0, ESC-15, ESC-30, and ESC-45, were measured separately. As reported in Table I, it was found that ESC-0 exhibited the highest values of contact angle, and the contact angle decreased gradually with the addition of slag. This suggests that adding the slag filler imparts a hydrophilic effect to the surface of the pure epoxy, which further leads to a good compatibility with the cement slurry.

Performance Testing of Cement Slurry and Set Cement

Routine cement tests for fluidity, free water, fluid loss, and compressive strength were performed. The formulation of the cement slurry was as follows: 600 g G-PC + 30% ESC + 1%

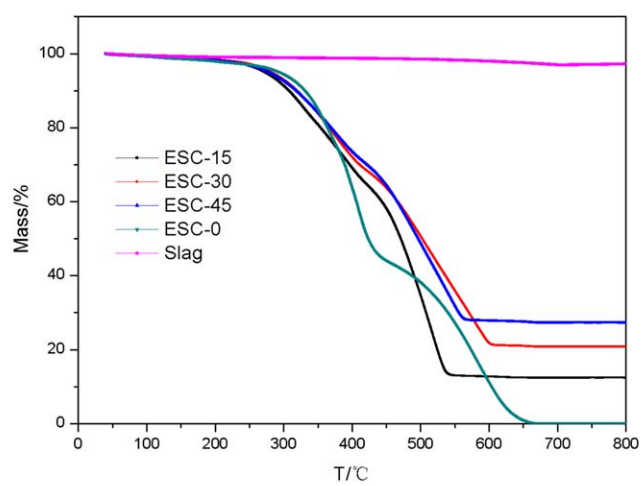

Figure 3. TGA curves of slag, ESC-0, ESC-15, ESC-30, and ESC-45. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

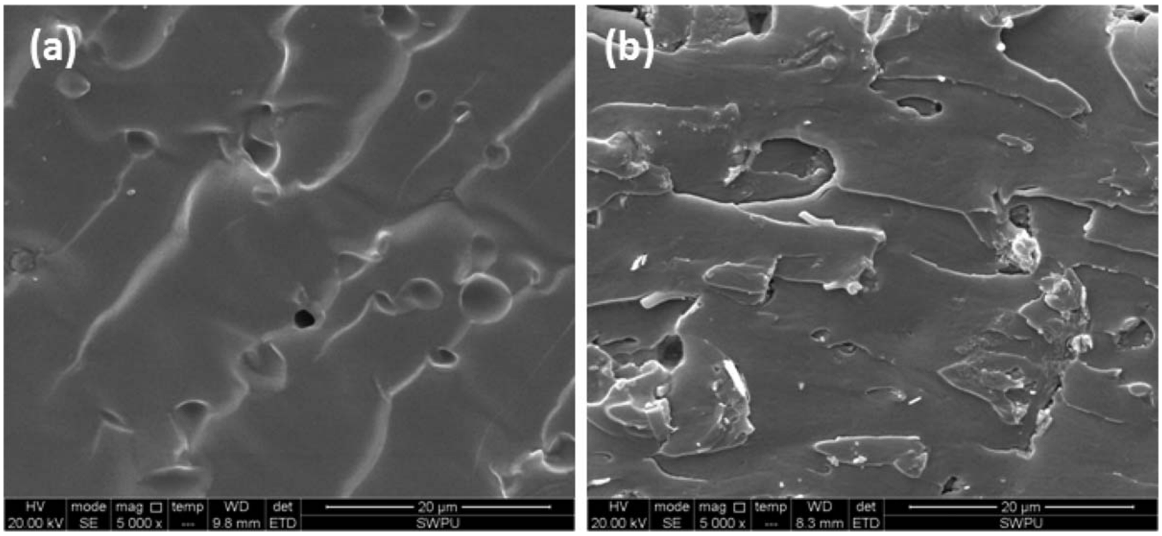

Figure 4. SEM of the specimens: (a) fracture surface of ESC-0, (b) fracture surface of ESC-45.

SXY + 1.5% SWJ-4; W/C = 0.6. The dosage of the mixtures is based on the weight percentage of G-PC. The properties of the cement slurry and the set cement containing unfilled and filled epoxy/slag composites are given in Table II.

It was found that the cement slurry containing ESC-0 showed the lowest compressive strength and the highest fluid loss and free water compared to cement slurry with slag incorporated in the epoxy matrix (ESC-15, ESC-30, ESC-45). The probable reasons were as follows. ESC-0 showed lower density and higher hydrophobicity than cement. On the one hand, it might be floating on the cement slurry, which would result in more free water and an unstable slurry due to the differences in density and wetting properties. On the other hand, the addition of

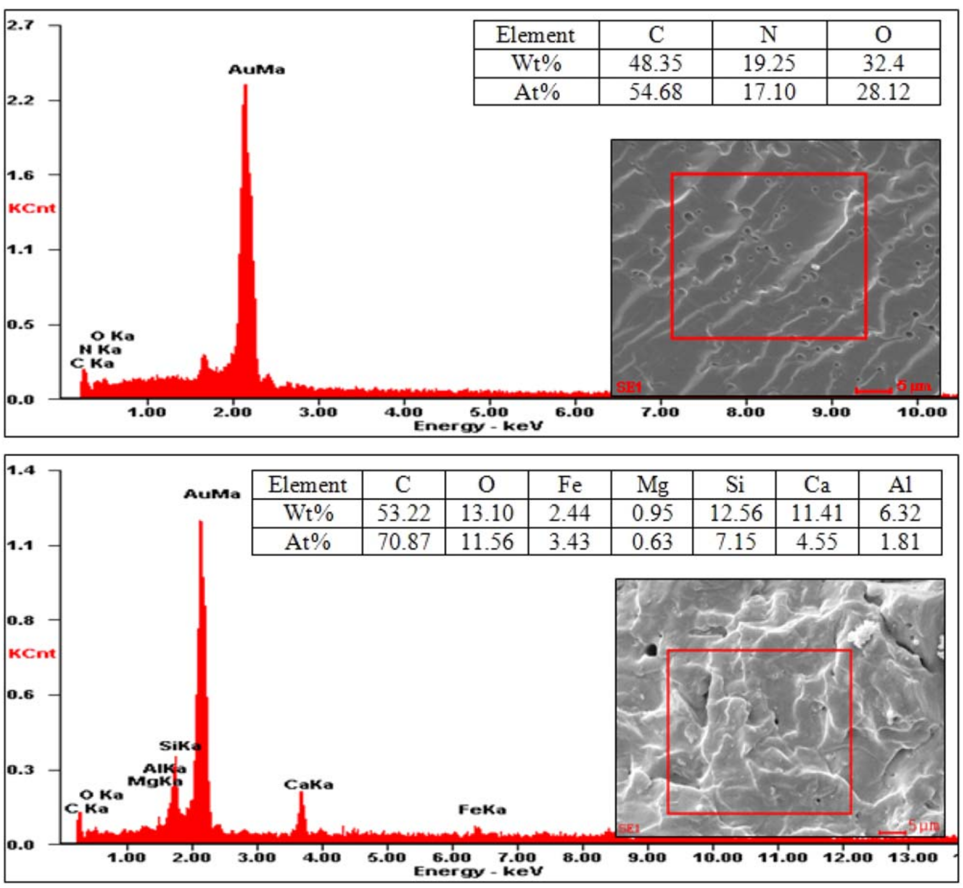

Figure 5. EDS analysis of (top) ESC-0 and (bottom) ESC-45. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table I. Surface Properties of the Specimens

Samples	ESC-0	ESC-15	ESC-30	ESC-45
θ (°)	85.8 ± 1.8	49.6 ± 2.3	38.2 ± 2.0	26.9 ± 2.1

ESC-0 might accelerate the cement particle sedimentation, which would result in excess free water and more fluid loss. In contrast, slag is a kind of hydraulic cementing material: it can not only bind free water but also take part in the hydration reaction to reduce free water. Therefore, it could be inferred that ESC-0 had poor compatibility with the cement slurry.

More importantly, the results showed that the compressive strength tended to increase with the addition of slag. Particularly in the case of the addition of slag from 30% to 45%, the compressive strength increased up to 27.1 MPa. This is somewhat expected and was in consonance with earlier work reported by Marie.[20] A plausible explanation for this phenomenon could be as follows. When the epoxy/slag composite was mixed in the cement slurry [Figure 1(b)], the slag, which has a feature of hydraulicity, can react with the calcium hydroxide (produced by cement hydration) to form C-S-H gel, which contributes to the mechanical behavior considerably.

XRD Analysis

The hydration products of set cement containing ESC-15, ESC-30, and ESC-45 were analyzed by XRD. Similar hydration products such as CH and C-S-H were observed in all the samples, as shown in Figure 6. However, with increasing slag, the content of C-S-H was on the increase, especially in the ESC-30 samples. Conversely, the intensity of the CH peaks reduced gradually with the addition of slag in the composites, which may be explained by the pozzolanic reaction of slag,[21] as in Equation (1):

$$\text{pozzolan} + \text{CH} + \text{H} \rightarrow \text{C} - \text{S} - \text{H} \quad (1)$$

When Portland cement comes into contact with water, its major components (C_3S and C_2S) react with water to form CH and C-S-H gel, and herein CH will be the alkaline exciting agent to slag. CH will react with the Ca^{2+}, AlO_4^{5-}, Al^{3+}, and SiO_4^{2-} contained in slag and generate new hydrates, such as C-S-H gel, which contributes more to the compressive strength of set cement.[22] This is somewhat expected and in consonance with our earlier work on compressive strength.

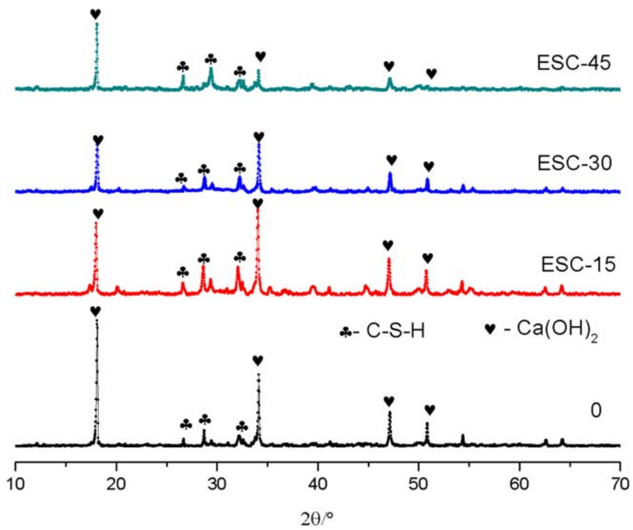

Figure 6. XRD spectra of blank cement and set cement containing ESC-15, ESC-30, and ESC-45. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TGA and DSC Analysis

Figure 7 shows the DSC and TGA curves of set cement containing ESC-15, ESC-30, and ESC-45 cured at 90°C for 2 days, and the blank set cement was used for comparison. It can be seen that the TGA and DSC curves of the set cement containing epoxy/slag composites are similar to each other, while they are different from those of blank set cement. From Figure 7(a), a reduction of weight of all the samples between 100 and 300°C was attributed to the evaporation of the physically absorbed water from the C-S-H gel and aluminosilicate gel.[23] In addition, there exists a small reduction of weight in all samples around 720°C, which conforms to the small endothermic peaks in the DSC curves and may be ascribed to the decarboxylation of calcite.[24]

As shown in Figure 7(b), the DSC curves exhibited big differences between blank set cement and set cement containing epoxy/slag composites from 300 to 500°C. In the blank set cement, an endothermic peak at approximately 467°C was due to the decomposition of CH. In the set cement containing epoxy/slag composite samples, a peak around 450–500°C for the presence of CH was not identified. This seems to be in conflict with the results of the XRD analysis. It is worth noting that a clear exothermic peak appears between the temperatures of 310 to 500°C

Table II. Routine Performances of Cement Slurry and Set Cement

Sample	Fluidity (cm)	Free water (%)	Fluid loss (ml)	Compressive strength (MPa) (90°C) 1 day	Compressive strength (MPa) (90°C) 2 days
0	24 ± 0.1	2	103 ± 3	25.2 ± 0.4	36.6 ± 0.3
ESC-0	23 ± 0.1	1	105 ± 3	11.5 ± 0.5	15.3 ± 0.4
ESC-15	22 ± 0.1	0	92 ± 4	14.3 ± 0.3	20.2 ± 0.5
ESC-30	22 ± 0.1	0	91 ± 2	15.8 ± 0.5	22.0 ± 0.6
ESC-45	22 ± 0.1	0	88 ± 3	18.4 ± 0.3	27.1 ± 0.4

Table III. Formulation of Cement Slurry with Different Densities

					Lightweight agent		
Sample	G-C (g)	SXY (%)	SWI-4 (%)	MS (%)	Type	Content (%)	Water (g)
1	100	1.8	2.5	35	ESC-15	40	185
2	100	1.8	2.5	35	ESC-30	40	185
3	100	1.8	2.5	35	ESC-45	40	185
4	100	1.8	2.5	35	FB	40	185

in all the samples except for the blank set cement specimens. Correspondingly, a large exotherm accompanied the significant weight loss of ESC-containing set cement around 310 to 500°C. A plausible explanation for this phenomenon could be the decomposition of epoxy, which covered the endothermic peak of CH. What's more, the exothermic peak intensity decreased with the addition of slag into epoxy and corresponded to the weight loss of about 22.3%, 20.2%, and 18.4% for set cement containing ESC-15, ESC-30, and ESC-45, respectively. This indicates that the increase of slag in epoxy influenced the reaction between the epoxy/slag composites and CH, thus influencing the complex thermal behavior of ESC-containing set cement samples.[25]

Application of ESC in Light Cement Slurry Density

As mentioned above, floating beads are prone to being crushed under the higher bottom hole pressure and higher mixing rate during premixing, which caused an increase in density of the cement slurry. We evaluated the ability of ESC to tolerate bottom hole pressure, high shearing and to enhance the compressive strength of set cement. The formulation of light cement slurry is listed in Table III.

Table IV summarizes the density and compressive strength of the cement containing various lightweight materials. As expected, the density of lightweight cement slurry made with ESC remained constant regardless of mixing rate and curing pressure, while for the floating beads (FB) lightweight cement slurry, the density under 4000 rpm, 8000 rpm, and 12,000 rpm exhibited an increase of approximately 1.54%, 3.08%, and 4.62%, respectively. This trend is probably attributed to the higher stirring rate leading to more crushed floating beads. On the one hand, more water from the cement slurry penetrated the broken floating beads, which resulted in great changes in density of the cement slurry. On the other hand, the reduction of available floating bead content could also lead to an increase in density of the cement slurry. To further verify the floating beads were broken under high stirring rate, we tested the set cement containing FB and ESC-45 by SEM, noting that all of the set cement was obtained by curing the cement slurry at 90°C for 48 h. As can be seen in Figure 8, obviously, cracks and holes were found on the surface of the floating beads [Figure 8(a)], while set cement containing ESC-45 had a compact structure, and we can hardly see the ESC-45 particles, which might be wrapped by the hydration products of cement [Figure 8(b)].

As discussed, the density of the floating bead lightweight cement slurry increased from 1.35 g/cm^3 to 1.46 g/cm^3 and 1.55 g/cm^3 at the pressures of 10 MPa and 20 MPa, respectively, which creates risks for cementing jobs.[26] To overcome these drawbacks, here ESC lightweight cement slurry was cured at pressures of 20 MPa, 40 MPa, and 60 MPa for 30 min, and then the density was determined. By comparing with the FB lightweight cement slurry, there is no obvious change for the density of ESC lightweight cement slurry; a slight increase of approximately 0.76% can be observed at the pressure of 60 MPa (sample 1). However, these variations are too tiny to affect a cementing job. In case of FB lightweight cement slurry, the density exhibited sharp increase with the increase of aging pressure, and a significant density growth value is 0.08 g/cm^3, a

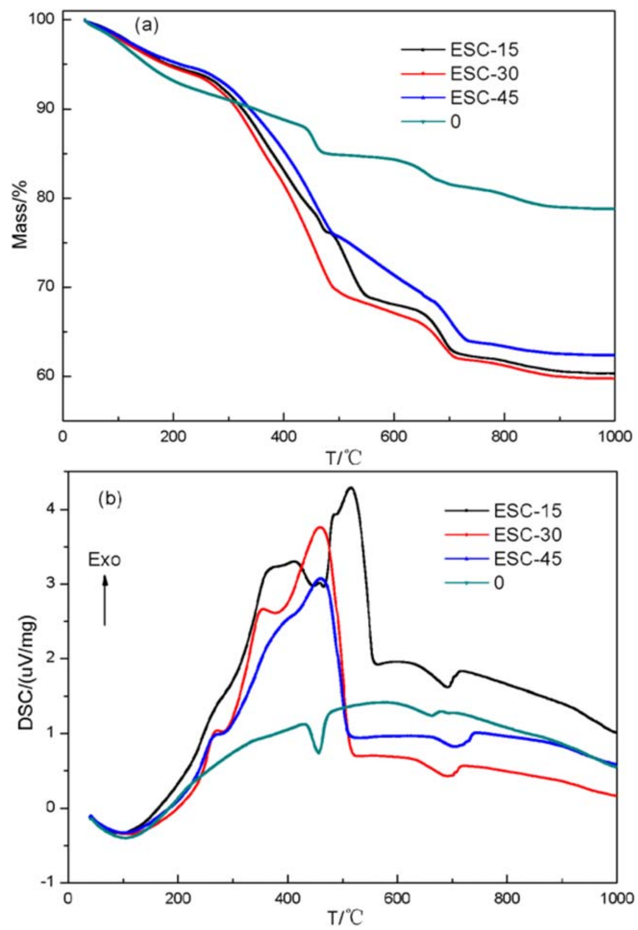

Figure 7. (a) TGA and (b) DSC curves of blank set cement and set cement containing ESC-15, ESC-30, and ESC-45. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

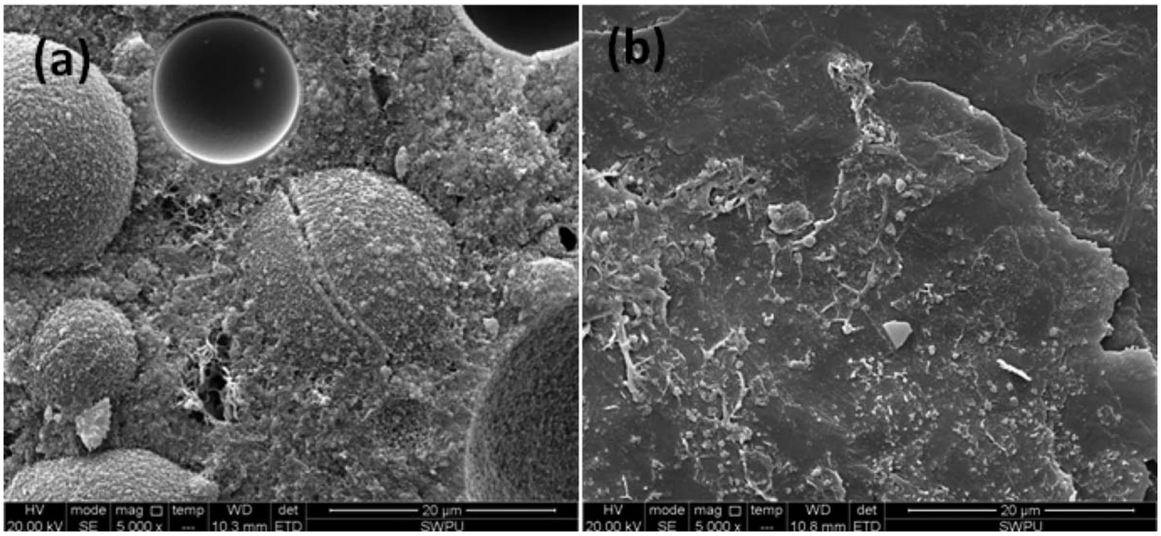

Figure 8. SEM micrographs of (a) FB set cement samples; (b) epoxy/slag composite set cement samples.

Table IV. Properties of Lightweight Cement

Samples	API standard density (g/cm^3)	Density (g/cm^3) Curing pressure (MPa) 20	40	60	Mixing rate (rpm) 4000	8000	12,000	Compressive strength (MPa)
1	1.30	1.30	1.30	1.31	1.30	1.30	1.30	10.4 ± 0.3
2	1.30	1.30	1.30	1.30	1.30	1.30	1.30	15.2 ± 0.5
3	1.30	1.30	1.30	1.30	1.30	1.30	1.30	12.6 ± 0.4
4	1.30	1.31	1.33	1.38	1.32	1.34	1.36	9.6 ± 0.3

significant density growth value is 0.08 g/cm^3. The big differences in density owing to pressure can easily cause lost circulation and even collapse accidents during well-cementing operations.

Here, in the compressive strength, a maximum strength of 15.2 MPa was obtained for the lightweight cement made with ESC-30. This value corresponded to an improvement in strength of 4.8 MPa above the ESC-15 and 5.6 MPa greater than the FB set cement specimens. It should also be noted that the incorporation of epoxy/slag composites with the addition of slag at 45% resulted in a reduction in strength. Therefore, the addition of slag of 30% should be used. In this way, a lightweight cement slurry having a slurry density of 1.3 g/cm^3, after curing at 90°C, and a compressive strength of >10 MPa at 48 h can be produced by incorporating the epoxy/slag composites. In conclusion, it could be inferred that this epoxy/slag composite is a promising alternative to floating beads as a lightweight agent for cement slurry applications.

CONCLUSIONS

Epoxy/slag composites were synthesized and characterized to investigate their chemical structures, thermal stabilities, wetting properties, morphological structure, and chemical composition. It has been shown that ESC exhibits outstanding thermal stability and hydrophilicity compared to ESC-0. In respect to the application of ESC in cement slurry, it can be found that the addition of slag into epoxy decreases the free water and fluid loss, which indicated that ESC has good compatibility with cement slurry. The improvement of compressive strength and the reduction of CH in set cement by the increase of slag into epoxy were due to the pozzolanic reaction of slag to form C-S-H gel by consumption of CH. Cement made with ESC possessed high compressive strength and a constant density of lightweight cement slurry regardless of curing pressure and mixing rate, in comparison to FB cement specimens, which indicated that ESC is a promising alternative to floating beads as a lightweight admixture for applications in lightweight cement slurries for oil and gas well cementing.

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