



Inherent relations between yield stress and stability of bubble petroleum coke water slurry

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Abstract

The stability of petroleum coke water slurry (PCWS) is currently a hot topic. The inherent relationship between yield stress and stability of bubble-PCWS was studied through orthogonal experiments and range analysis in this work. The results showed that the stability of bubble-PCWS was positively related to the yield stress and that the yield stress could be greatly impacted by the operation conditions during preparation of bubble-PCWS. The main factors affecting the yield stress of bubble-PCWS were solid concentration, aeration time and dosage of frother. However, the effects of aperture size of air distribution plates and type of frother on the yield stress were slight within the experimental range. The optimal conditions for the greatest yield stress were as follows: aeration time of 30 min, solid concentration of 65 wt%, frother dosage of 0.030 wt% of the air-dried pulverized petroleum coke, aperture size of air distribution plate of 2–5 μm and AOS frother. The yield stress and the pour rate of bubble-PCWS under this optimum operation condition could reach maxima of more than 0.4 Pa and 96%, respectively.

Keywords Petroleum coke · Petroleum coke water slurry (PCWS) · Stability · Yield stress · Orthogonal experiment

1 Introduction

Petroleum coke, an end product of the petroleum refining process, generally possesses high carbon content and shows good slurry ability for petroleum coke water slurry (PCWS) (Wang et al. 2012; He et al. 2011; Gao et al. 2010). PCWS usually has high calorific value and similar flow characteristics to oil, and may become a favorable substitute for fuel–oil (Gao et al. 2012, 2016). But the development and application of PCWS are restricted by its inferior stability (Wang et al. 2012; Zhan et al. 2010; Liu et al. 2013; Ma et al. 2013). A new solution, bubble-PCWS, which can make PCWS obtain both a high mass concentration and a good stability, has been presented (Gao et al. 2014). In the bubble-PCWS, bubble-particle complexes can be formed

because of adsorption interaction at the bubble–particle phase boundary (Yianatos et al. 2008; Polat and Chander 2000; Mathe et al. 1998; Contreras et al. 2013; Zheng et al. 2004). And the three-dimensional network structures based on the bubble-particle complexes can effectively make the bubble-PCWS show ideal stability (Gao et al. 2014). That is to say, the stability of the bubble-PCWS has a direct relationship with the stability of three-dimensional network structures of the slurry.

Yield stress is the minimum stress that the slurry needs to overcome to start flowing when sheared. When the shear stress applied is not big enough to overcome the yield stress, the slurry will not flow. The yield stress is directly related to the three-dimensional network structure of the slurry and can represent the strength of the network structure to some extent. Therefore, there must be some kind of internal relations between the yield stress and the stability of bubble-PCWS. In order to comprehensively examine this relationship between them, orthogonal experiments and range analysis have been done in this paper. Five factors (i.e., solid concentration, aeration time, aperture size of air distribution plate, dosage and type of frother) were considered while preparing bubble-PCWS. Orthogonality means that all lever values of each factor are

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symmetric and orthogonal to each other and that the levers of all factors are assembled uniformly to arrange operation conditions without omissions and repetitions (Liu 2006). The orthogonal experiments can not only obtain much more information, but also reduce the experimental workload.

2 Experimental

2.1 Materials

A petroleum coke from Jinshan Petrochemical Co. Ltd. was used in the experiments. The proximate and ultimate analysis results of the petroleum coke sample are shown in Table 1. The molecular formula of the petroleum coke can be drawn from the ultimate analysis results in Table 1, which turns out to be 'C₁₀₆H₅₁NSO₄'.

The dispersing agent used was sodium lignosulfonate at a concentration of 0.8 wt% of the air-dried pulverized petroleum coke, which could optimize the slurryability of PCWS (Gao et al. 2015). Two types of anionic surface active agents, sodium lauryl sulfate (K12) and α -olefin sulfonate (AOS), were used as frothers. Frothers with amphiphilic molecular structure are adsorbed and aligned at the liquid–vapor interface. This produces a reduction in the liquid–gas surface tension and system energy, which causes air to disperse in the slurry to form bubbles of smaller diameter (Zhao et al. 2012).

2.2 Equipment and methods

2.2.1 Preparation of bubble-PCWS

Figure 1 shows a flowchart of the process for preparing bubble-PCWS. The petroleum coke was ground in a ball mill to obtain pulverized samples, and particles below 100 mesh were selected by an electric sieve shaker. The petroleum coke particles, deionized water, dispersing agent and frother were mixed with an electric mixer at 1000 r/min for 10 min forming an initial bubble-PCWS. The initial bubble-PCWS was put into a container with an air distribution plate. Subsequently, compressed air (0.7 MPa, 1.5 m³/h) was blown into the container from the bottom of

the container through the air distribution plate. The samples of bubble-PCWS were obtained after a certain amount of compressed-air blow time. The compressed air was produced by an air compressor and was measured by a pressure gauge and a flow meter. In addition, a thermostatic bath set to 20 °C was used to avoid the effect of variation in compressed air temperature on the nature of the slurry.

2.2.2 Determination of granularity distribution

Granularity distribution of the selected petroleum coke powder was measured with a British Mastersizer (2000) laser particle size instrument, as shown in Fig. 2. And the average diameter of petroleum coke particles was about 27 μ m.

2.2.3 Determination of yield stress

The yield stress values were obtained by fitting the shear stress–shear rate curve using Herschel–Bulkley models. Both shear stress and shear rate were measured on a rotary viscometer (NXS-4C, Thermo, China). Three-parameter Herschel–Bulkley models are described as Eq. (1) (Ma et al. 2013).

$$\begin{cases} \dot{\gamma} = 0 & \tau \leq \tau_y \\ \tau = \tau_y + k\dot{\gamma}^n & \tau > \tau_y \end{cases} \quad (1)$$

where $\dot{\gamma}$ is the shear rate, s⁻¹; τ the shear stress, Pa; τ_y the yield stress (Pa); k the consistency coefficient, Pa sn; n the dimensionless flow characteristic exponent.

2.2.4 Determination of stability

The stability of bubble-PCWS was measured by an inversion method. The measurement started by keeping the slurry in a sealed container for 7 days. Then, the container was tilted for 30 s to make the slurry flow out freely and then, inverted vertically for 8 min. Finally, we weighed and calculated the mass of poured slurry, and used the pour rate and the ratio of poured mass to the total mass of slurry as indicators to evaluate slurry stability. A higher pour rate indicates a more stable slurry.

Table 1 Proximate and ultimate analysis results of the petroleum coke

| Proximate analysis, wt% | | | | $Q_{b, ad}$ | Ultimate analysis, wt% | | | | |
|-------------------------|----------|----------|-----------|---------------------|------------------------|----------|----------|-----------|----------|
| M_{ad} | A_{ad} | V_{ad} | FC_{ad} | kJ kg^{-1} | C_{ad} | H_{ad} | N_{ad} | St_{ad} | O_{ad} |
| 1.07 | 0.73 | 10.0 | 88.2 | 35,461 | 88.77 | 3.55 | 1.01 | 2.22 | 4.45 |

Subscript ad stands for air-dried basis; M, A, V and FC stand for moisture, ash, volatile and fixed carbon, respectively; $Q_{b,ad}$ stands for the high heating value in air-dried basis; C, H, N, St and O stands for carbon, hydrogen, nitrogen, total sulfur and oxygen, respectively. The oxygen data are obtained by calculation

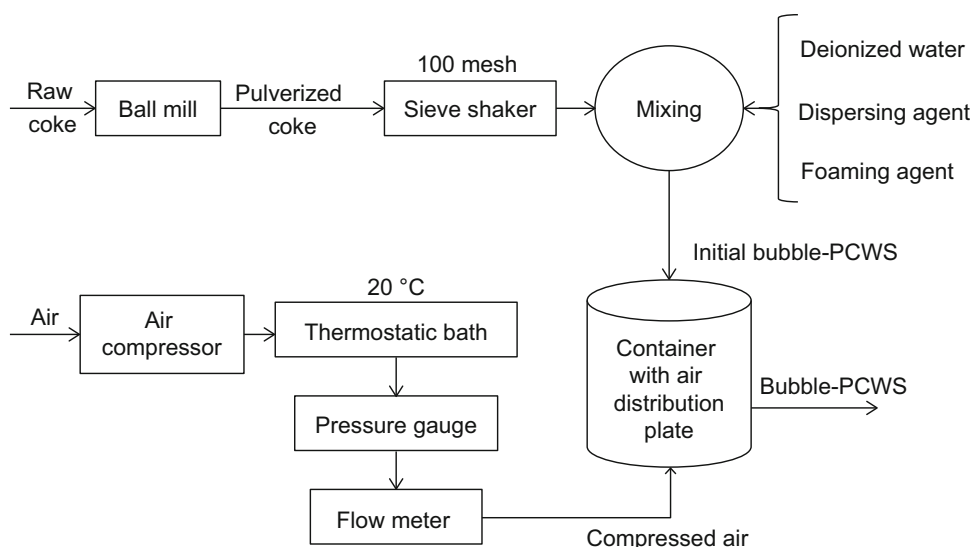


Fig. 1 Process flowchart for preparing bubble-PCWS

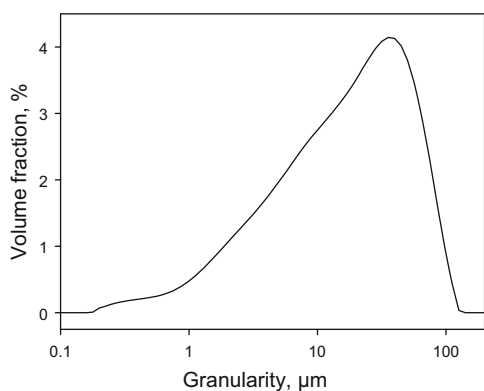


Fig. 2 Granularity distribution of petroleum coke powder

2.3 Choice of levers of the factors in orthogonal experiments

There were five factors (i.e., solid concentration, aeration time, aperture size of air distribution plate, dosage and type of frother) considered in the orthogonal experimental design, as shown in Table 2. The factor, type of frother, had two levers, and the other factors each possessed four

Table 2 Factors and levers of bubble-PCWS in orthogonal experimental design

| Factors | | | Lever 1 | Lever 2 | Lever 3 | Lever 4 |
|-------------|---------------------|------|---------|---------|---------|---------|
| Factor code | Factor name | Unit | | | | |
| A | Solid concentration | % | 65 | 66 | 67 | 68 |
| B | Aeration time | min | 0 | 20 | 10 | 30 |
| C | Aperture size | µm | 5–15 | 15–40 | 40–80 | 2–5 |
| D | Dosage of frother | % | 0 | 0.030 | 0.015 | 0.045 |
| E | Type of frother | – | K12 | AOS | – | – |

levers. In order to simplify the experimental process, the interaction between various factors was not considered.

According to the results of single factor analysis (Gao et al. 2014), the ranges of operational parameters were arranged as follows. Solid concentration of slurry was set as 65%–68% and the aeration time as 0–30 min. Four different air distribution plate apertures were used in experiments, and aperture ranges were 40–80 µm, 15–40 µm, 5–15 µm and 2–5 µm, respectively. Two types of frother, K12 and AOS, were used, respectively. The dosage of frother was at a concentration of 0–0.045 wt% of the air-dried pulverized petroleum coke.

3 Results and discussion

3.1 Design and results of orthogonal experiments

The orthogonal array (Liu 2006), $L_{16} (4^4 \times 2^3)$, was applied in this paper, and 16 operation conditions were chosen by assembling the levers of different factors, as shown in rank 2–6 in Table 3. Two repeated experiments were made on each operation condition. The orthogonal

experimental results, the yield stress and the pour rate, are shown in rank 7–10 in Table 3. It can be seen from Table 3 that the operation conditions while preparing bubble-PCWS greatly impacted the yield stress and the pour rate of slurry and that the pour rate was positively related to the yield stress. In fact, the yield stress is related to the stability of the space structure of bubble-PCWS. The more stable the space structure is, the greater the shear stress will be needed to overcome the high spatial structure resistance, and the bigger the yield stress shows. The bubble-PCWS with lower yield stress possesses lower initial viscosity and better liquidity, but its steric hindrance is weaker, and the particles are much easier to sink down and agglomerate during the storage, resulting in a lower pour rate and an inferior slurry stability.

3.2 Range analysis of orthogonal experimental results

Range analysis can be applied to the orthogonal experimental results to quantitatively analyze the integrated optimal levels of multiple factors and distinguish the primary and secondary influential factors on the yield stress. Range means the biggest difference between the experimental results at different levels of each factor. The bigger the range of one factor, the greater the effect of this factor on the yield stress (Liu 2006). For all levers of one factor, every lever of other factors appears equally because of orthogonality of the orthogonal array. Therefore, when one factor is studied, the other factors cannot be considered temporarily because their effects cancel each other out.

The number of operation conditions is marked as n , and the sequence number is i , $i = 1 \sim n$. The same experiments are repeatedly made by k times on every operation condition, and the sequence number of repeated experiments is j , $j = 1 \sim k$. A single experimental result is marked as y_{ij} . The lever amount of a factor is M , and the sequence number of a lever is r , $r = 1 \sim M$. The number of experiments on the same factor and same lever is L in total, and the virtual sequence number is q , $q = 1 \sim L$. The experimental result in lever r of a factor is marked as y_{rq} , and then, the sum for each factor in each level, $\sum(r)$, is presented as Eq. (2).

$$\sum(r) = \sum_{q=1}^L y_{rq} \tag{2}$$

The average experimental result for each factor in each level, k_r , can be described by Eq. (3).

$$k_r = \frac{\sum_{q=1}^L y_{rq}}{L} \tag{3}$$

The indicator k_r can accurately reflect the effective extent of a factor in lever r . The higher the value of k_r is, the more beneficial the lever r turns out to be to improve slurry yield stress. The optimal lever for the yield stress can be found by comparing k_r on each lever of one factor, and the range R can be calculated by Eq. (4).

$$R = \max k_r - \min k_r \tag{4}$$

The primary and secondary influential factors on the yield stress can be distinguished by comparing the range of

Table 3 Orthogonal experimental design and results of bubble-PCWS

| Serial number | Factor A | Factor B | Factor C | Factor D | Factor E | Yield stress, Pa | | Pour rate, % | |
|---------------|----------|----------|----------|----------|----------|------------------|----------|--------------|----------|
| | | | | | | 1st test | 2nd test | 1st test | 2nd test |
| 1 | 65 | 0 | 5–15 | 0.000 | K12 | 0.02 | 0.01 | 15.11 | 15.09 |
| 2 | 65 | 20 | 15–40 | 0.030 | K12 | 0.31 | 0.34 | 89.97 | 95.04 |
| 3 | 65 | 10 | 40–80 | 0.015 | AOS | 0.03 | 0.04 | 30.77 | 34.43 |
| 4 | 65 | 30 | 2–5 | 0.045 | AOS | 0.35 | 0.38 | 94.61 | 95.81 |
| 5 | 66 | 0 | 15–40 | 0.015 | AOS | 0.04 | 0.05 | 22.29 | 24.99 |
| 6 | 66 | 20 | 5–15 | 0.045 | AOS | 0.23 | 0.21 | 83.07 | 81.06 |
| 7 | 66 | 10 | 2–5 | 0.000 | K12 | 0.06 | 0.05 | 31.75 | 30.22 |
| 8 | 66 | 30 | 40–80 | 0.030 | K12 | 0.24 | 0.28 | 83.84 | 86.39 |
| 9 | 67 | 0 | 40–80 | 0.045 | K12 | 0.07 | 0.06 | 17.91 | 16.79 |
| 10 | 67 | 20 | 2–5 | 0.015 | K12 | 0.01 | 0.02 | 25.01 | 27.48 |
| 11 | 67 | 10 | 5–15 | 0.030 | AOS | 0.11 | 0.13 | 27.65 | 29.11 |
| 12 | 67 | 30 | 15–40 | 0.000 | AOS | 0.05 | 0.04 | 23.89 | 22.46 |
| 13 | 68 | 0 | 2–5 | 0.030 | AOS | 0.07 | 0.06 | 17.41 | 16.03 |
| 14 | 68 | 20 | 40–80 | 0.000 | AOS | 0.05 | 0.03 | 16.15 | 16.02 |
| 15 | 68 | 10 | 15–40 | 0.045 | K12 | 0.06 | 0.07 | 19.21 | 20.77 |
| 16 | 68 | 30 | 5–15 | 0.015 | K12 | 0.09 | 0.08 | 28.99 | 27.82 |

Table 4 Range analysis of yield stress of bubble-PCWS in orthogonal experiments

| Analysis indicators | Yield stress, Pa | | | | |
|---------------------------------------|-------------------|----------|----------|----------|----------|
| | Factor A | Factor B | Factor C | Factor D | Factor E |
| Sum for each factor in each level | | | | | |
| Σ1 | 1.48 | 0.38 | 0.88 | 0.31 | 1.77 |
| Σ2 | 1.16 | 1.20 | 0.96 | 1.54 | 1.87 |
| Σ3 | 0.49 | 0.55 | 0.80 | 0.36 | – |
| Σ4 | 0.51 | 1.51 | 1.00 | 1.43 | – |
| Average for each factor in each level | | | | | |
| k_1 | 0.19 | 0.05 | 0.11 | 0.04 | 0.11 |
| k_2 | 0.15 | 0.15 | 0.12 | 0.19 | 0.12 |
| k_3 | 0.06 | 0.07 | 0.10 | 0.05 | – |
| k_4 | 0.06 | 0.19 | 0.13 | 0.18 | – |
| Range R | 0.13 | 0.14 | 0.03 | 0.15 | 0.01 |
| Optimal lever | A1 | B4 | C4 | D2 | E2 |
| Order of influential factors | D > B > A > C > E | | | | |

each factor. The results of range analysis of yield stress of bubble-PCWS are shown in Table 4.

It can be seen from Table 4 that the bubble-PCWS could obtain the biggest yield stress when the operational parameters were set as follows, aeration time of 30 min, solid concentration of 65 wt%, frother dosage of 0.030 wt% of the air-dried pulverized petroleum coke, aperture size of air distribution plate of 2–5 μm and AOS frother. It can also be seen from the range values of the five factors in Table 4 that the main factors affecting the yield stress of bubble-PCWS were solid concentration, aeration time and frother dosage. However, the effects of aperture size of air distribution plate and type of frother on the yield stress were slight within the experimental range.

3.3 Verification of orthogonal experimental results

According to range analysis of orthogonal experimental results, the integrated optimal levels of multiple factors for the greatest yield stress were as follows, aeration time of 30 min, solid concentration of 65 wt%, frother dosage of 0.030 wt% of the air-dried pulverized petroleum coke, aperture size of air distribution plate of 2–5 μm and AOS frother. However, this optimum operation condition was not included in the original experimental plan. Therefore, confirmatory tests under this optimum operation condition were necessary, and results are shown in Table 5.

It is shown from Table 5 that the yield stress and the pour rate of bubble-PCWS under these optimum operation conditions could, respectively, reach more than 0.4 Pa and 96%, which were greater than all the results in the original

Table 5 Results of confirmatory tests on the optimal levels of multifactors

| Indicators | Under the optimum operation condition | | |
|------------------------------------|---------------------------------------|----------|---------|
| | 1st test | 2nd test | Average |
| Yield stress, Pa | 0.407 | 0.415 | 0.411 |
| Pour rate, % | 96.03 | 96.25 | 96.14 |
| Slurry density, g cm ⁻³ | 0.854 | 0.863 | 0.859 |
| Apparent viscosity, mPa s | 575.0 | 637.0 | 606.0 |

experimental plan. Hence, the combination condition with aeration time of 30 min, solid concentration of 65 wt%, frother dosage of 0.030 wt% of the air-dried pulverized petroleum coke, aperture size of air distribution plate of 2–5 μm and AOS frother was the integrated optimal levels of multiple factors for the greatest yield stress and the best stability within the experimental range. The apparent viscosity of bubble-PCWS is now around 600 mPa s, which is appropriate for industrial application of the slurry.

3.4 Effects of yield stress on bubble stability

The stability of bubble-PCWS depends on the stability of bubbles in the slurry. Ignoring bubble gravity, bubbles are generally impacted by the buoyancy impelling bubbles to float up and the stress from slurry restraining bubble movements. It has been pointed out (Asmatulu 2008; Oshitani et al. 2012; Galvin et al. 2001; Sikorskia et al. 2009) that the bubbles are stably suspended in the slurry when the buoyancy was less than the yield stress of the slurry, and on the contrary, the bubbles rose out of slurry

when the buoyancy was greater than the yield stress. On the basis of this theory, the precondition of stable suspension of bubbles in the slurry without rising had been obtained as Eq. (5).

$$\tau_y > \frac{\rho_s g \cdot (\pi D^3 / 6)}{\pi D^2 / 4} \quad (5)$$

where τ_y is the yield stress, Pa; ρ_s the slurry density, kg/m^3 ; g the acceleration of gravity, m/s^2 ; D the bubble diameter, m.

Hence, the bubble diameter range of stable suspension can be calculated based on the value of yield stress, shown as Eq. (6).

$$D < \frac{3\tau_y}{2\rho_s g} \quad (6)$$

It can be seen from Eq. (6) that the critical diameter of stable suspended bubbles is directly proportional to the yield stress. The greater the yield stress is, the bigger the critical diameter of bubbles becomes. In other words, the diameter range of bubbles that can stably suspend in slurry increases with increasing yield stress. Therefore, the greater the yield stress is, the more bubbles there are in the stable suspension system to prevent sedimentation of particles, leading to a better slurry stability. This is identical with the above experimental result that the slurry stability was positively related to the yield stress.

3.5 Effects of yield stress on particle stability

The main force causing sedimentation of particles in slurry system is gravity. Settling stress from gravity is determined by particle mass, action area of gravity and density difference between particle and dispersion medium (Cosgrove 2009). Only when this settling stress is less than the yield stress of slurry can particles stay in suspension in the slurry. That is to say, the precondition of a stable suspension of particles in the slurry without settling is that the yield stress is greater than the settling stress, shown as Eq. (7).

$$\tau_y > \frac{4 \times \Delta\rho g a}{3} \quad (7)$$

where $\Delta\rho$ the density difference between particle and dispersion medium, kg/m^3 ; a the particle radius, m.

In the above orthogonal experiments, the average radius of petroleum coke particle was $13.5 \mu\text{m}$, the true density of petroleum coke was 2140 kg/m^3 , and the density of the dispersion medium was 1000 kg/m^3 . Therefore, it can be calculated according to Eq. (6) that only when $\tau_y > 0.194 \text{ Pa}$ could the bubble-PCWS have the possibility for possessing good stability within the experimental range. It can be seen from Tables 3 and 5 that the bubble-PCWS

with yield stress being more than 0.194 Pa possessed better stability with pour rate being over 80%, while the bubble-PCWS with yield stress being less than 0.194 Pa possessed worse stability with pour rate being under 35%. Hence, the experimental results were in conformity with the above theory that the slurry could possess much better stability when the yield stress was greater than the settling stress.

4 Conclusions

- (1) Through orthogonal experiment design, it is clear that the operation conditions while preparing bubble-PCWS greatly impacted the yield stress and the pour rate of slurry and that the pour rate was positively related to the yield stress.
- (2) Through range analysis, the following conclusions can be drawn. The main factors affecting the yield stress of bubble-PCWS were solid concentration, aeration time and dosage of frother. However, the effects of aperture size of air distribution plate and type of frother on the yield stress were slight within the experimental range. The optimal levels of multiple factors for the greatest yield stress were as below, aeration time of 30 min, solid concentration of 65 wt%, frother dosage of 0.030 wt% of the air-dried pulverized petroleum coke, aperture size of air distribution plate of 2–5 μm and AOS frother type. And on this occasion, the bubble-PCWS could possess the best stability within the experimental range. The yield stress and the pour rate of bubble-PCWS under this optimum operation condition could reach more than 0.4 Pa and 96%, respectively.
- (3) The bigger the yield stress was, the better the slurry stability turned out to be, which could be interpreted from two aspects. On the one hand, the diameter range of bubbles that can steadily suspend in slurry increases with increasing yield stress, and there will be more bubbles in the stable suspension system to prevent sedimentation of particles. On the other hand, particles can steadily suspend in the slurry only when the yield stress is greater than the settling stress, which makes it possible that the slurry possesses good stability.

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