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## Experimental investigation on the influencing factors of preparing three-phase foam

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**Abstract:** A three-phase foam is considered one of the promising advanced materials for fighting fires. However, the preparation conditions, cost and effect are key factors for industrial applications. In this study, new three-phase foam systems with fly ash and a complex surfactant are proposed. Five types of surfactants alcohol polyoxyethylene ether sodium sulfate, coconut oil diethanolamine, sodium lauryl sulfate, polyacrylamide and polyether-modified silicone resin emulsion were selected as foaming agents. Through laboratory experiments, the effect on the expansion ratio and foam stability of the surfactant type/concentration, fly ash particle concentration/size and pH were investigated. The foaming condition was determined by numerical optimization. The results of this study may serve as a reference for understanding the preparation of a novel three-phase foam. It is hoped that this work could provide useful guidance for the preparation of efficient three-phase fire-extinguishing foam for the safe guarding of process safety in the field of chemical production, transportation, and storage suitable for drug delivery than  $Al_{12}P_{12}$  and  $Al_{12}N_{12}$  based on their recovery times.

**Keywords:** fighting fire foam; expansion ratio; half-life; surfactant; fly ash; pH.

### INTRODUCTION

Coal accounts for 60 % of the fuel used for power generation in China at present,<sup>1</sup> and for a long time into the future, the main body of China's energy consumption will remain coal.<sup>2</sup> However, a gradually expanding demand has brought more risks, such as coal field fire accidents,<sup>3</sup> this usually brings heavy losses and danger in the field of mining, transportation and storage. Therefore, effective and efficient fire-extinguishing material is particularly important in the process safety of firefighting and safeguarding. Foam mainly includes solidified

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foam, a type of dispersed gas-liquid/solid system and is an effective environmentally friendly and promising material in extinguishing fires, mineral floatation, and enhanced oil recovery.<sup>4,5</sup> Foam two-phase foam and three-phase foam, screening, cooling and insulating are the three main effects to extinguish fires.<sup>6</sup> Two-phase foam is a dispersion system composed of an aqueous surfactant solution and gas (air or nitrogen), which has an important role in fighting fires. Used properly, two-phase foam has previously effectively controlled mine fires.<sup>7</sup> However, its high specific surface area and surface energy lead to thermodynamic instability.<sup>8,9</sup> Moreover, the gravity and Laplace pressure in the aqueous foam cause foam drainage to coalesce and coarsen, which leads to foam decay.<sup>10</sup> The efficiency of the fire extinguishing foam is mainly determined by its stability and heat insulation performance. Therefore, stabilized two-phase foams have limited applications in large-scale coal and oil fields.<sup>11</sup>

Three-phase foam is a system for dispersing gas and solid particles in a liquid phase,<sup>12</sup> in which particles stabilize the foam by adsorbing onto the gas/water interface.<sup>13</sup> Compared with a traditional gas-liquid two-phase foam, the addition of solid particles will inevitably affect the precipitation rate of water in the liquid film and the stability of the foam. Researchers have tried to add a burning-resistant powder to two-phase foam to form three-phase foam that was composed of water, air (or N<sub>2</sub>) and solid.<sup>14,15</sup>

Fly ash (FA) is a massive waste residue produced as a by-product of coal combustion in power stations, out of nearly a billion tons produced in sample amount, not more than 25 % is utilized and the remaining is left untreated.<sup>16,17</sup> Using FA produced by coal-fired boilers as solid particles to form FA three-phase foam system could reduce the cost of the system. At the same time, it could reduce the pollution of solid waste by reducing FA discharge. Pre-ground FA particles are commonly used to stabilize the three-phase foam and enhance oil recovery.<sup>18</sup> Previous studies have reported that FA particles produced by ball-milling could significantly improve the stability and resistance factor of the foam system. Although FA particles can effectively improve foam stability, grinding causes a significant increase in cost and work load. A water-based foam can produce an efficient fire prevention performance because it has good cooling and a filling effect in the fire extinction stage. Therefore, a water-based and non-ground FA stabilized foam system could promote the development of fire extinguishing materials. Currently, three-phase foams stabilized by unground FA have been applied to try and prevent and control spontaneous combustion in coal.<sup>15,19,20</sup>

The stability of three-phase foam is determined by the synergistic effect between surfactant and particles.<sup>21–23</sup> The synergistic effect is associated with the physicochemical properties, concentrations of surfactants and solid particles.<sup>24</sup> Mixed surfactants show a stronger synergistic interaction compared with a single surfactant.<sup>25</sup> However, whether it helps to improve the properties of fire

fighting foam, and the influence of FA/surfactant on the stability and expansion ratio of three-phase foam still require clarification.<sup>26</sup>

In this study, because the used fly ash was negatively charged, cationic foam agents are unsuitable for the fly ash-based foam, anionic foamers, *i.e.*, fatty alcohol polyoxyethylene ether sodium sulfate (AES) and sodium lauryl sulfate (K12), and nonionic foamer, *i.e.*, polyacrylamide (PAM), coconut oil diethanolamine (6501), foam stabilizer: modified silicone resin polyether emulsion (FM-550), were first evaluated for the foaming agent system. Here air as gas phase and non-ground FA particles as solid phase were used to generate water-based three-phase foam. The formulation of foaming agent was optimized by orthogonal experiment. The effects of the FA particle size/content, pH on improving the expansion ratio and foam stability were investigated. As a result, the FA supported foam exhibited better stability and expansion ratio. The design in this work could be used to study the firefighting efficiency of different three-phase foams and serve as a prototype to develop better generators for both lab research and practical applications.

## EXPERIMENTAL

### *Materials and instrumentations*

All chemicals including AES, K12, 6501, PAM and FM-550 (active ingredient: 55 %) were industrial grade purchased from Lvsen Chemical Reagent Co., Ltd., China and used without further purification. Deionized water was used in all experiments. The FA used was obtained from Huainan Tianjiaan Power Co., Ltd., China. Air compressor (DET750-30 L, purchased from Shenba Compressor Manufacturing Co. Zhejiang, China): air pressure of 0.8 MPa, and air speed of 80 L min<sup>-1</sup>. The foam gun delivered foam with a high-pressure spray by an air compressor.

### *Preparation of foam*

The three-phase foam was prepared using a self-made designed system, which was mainly composed of an air compressor and a foam gun (shown in Fig. 1). First, a foam solution is prepared in a polyvinyl chloride (PVC) jar according to the ratio of ingredients, then FA particles were added as the solid phase, and fully stirred until a homogeneous slurry was obtained. The foam gun was provided with an air compressor to provide gas and pressure, then air was introduced through an air inlet on the tube and mixed with the slurry. The pressure then enables spray through the nozzle, whereby uniform and stable three-phase foams were jetted by the air through the outlet for fire extinguishing. After the foam was formed, the

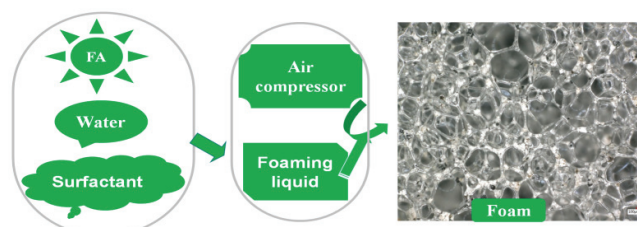


Fig. 1. Preparation process of the three-phase foam.

initial foam volume was immediately recorded, which was used to describe foaming capacity and stability. All the foam tests were performed at room temperature. A study of the properties of the foam in different foaming agent, different FA content/size and pH enabled the optimal parameters to be found.

#### Characterizations and calculation

The particle size distribution of FA sample was measured by a Laser Particle Sizer (Marvin Mastersizer 2000, UK). The FA was investigated by X-ray diffraction (XRD, Lab XRD, Shimadzu, Japan) test in the range of 10–60° with monochromatic CuK $\alpha$  radiation at 40 kV and 3 nm.

Foaming ability is one of the most fundamental properties of foam, which is expressed by the expansion ratio ( $F$ ) and was calculated based on Eq. (1) according to the current Chinese national standards (GB/T 1966–1996):

$$F = \frac{\rho v}{m_2 - m_1} \quad (1)$$

$\rho$ : the density of the foaming solution (assumed to be equal to the density of water, 1 kg dm<sup>-3</sup>);  $v$ : volume of foam collector, dm<sup>-3</sup>;  $m_1$ : the mass of the empty foam collector, kg;  $m_2$ : the mass of the foam collector was filled with foam, kg.

Every experiment was repeated 5 times, and three values of good precision were averaged as the measurement result. The relative change in foam volume as a function of time can be considered an indication of foam stability. Generally, foam stabilization performance is expressed by the defoaming time (decay half-life,  $t_{1/2}$ ),  $t_{1/2}$  for which the foam volume decreases by 50 %.

## RESULTS AND DISCUSSION

### Characterization of FA

FA is a highly heterogeneous material usually consisting of silicon dioxide and aluminum oxide as the major mineral components.<sup>27</sup> Laser particle analyzer is commonly used to measure the particle size distribution. As shown in Fig. 2a, the used FA particle size distribution range was 1–500  $\mu$ m, and most FA diameter of particles were about 300  $\mu$ m. The FA was also characterized by XRD, with Fig. 2b showing the spectra of the sample particles, the primary diffraction

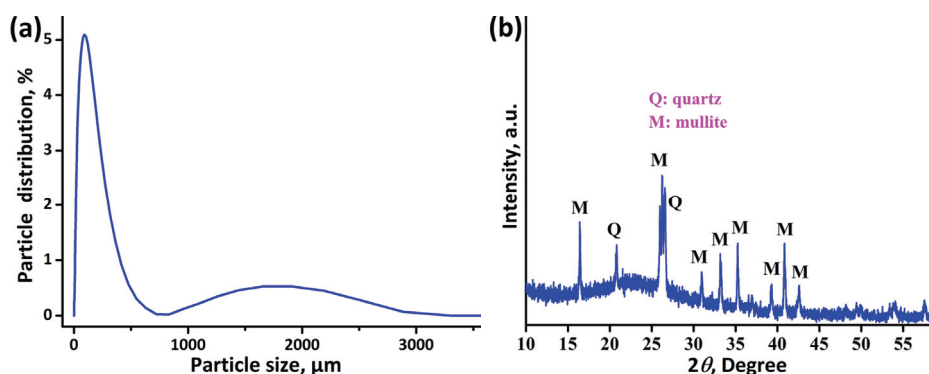


Fig. 2. Particle size distribution (a) and XRD analysis (b) of FA.

peaks at the  $2\theta$  values of the particles were located. These results indicate that the used FA contained mainly  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ , the major absorptive capacity of water was low and was relatively resistant to dissolution.

#### *Factors influencing of foam properties*

*Optimization of foaming agent.* The surfactant in the foam could be adsorbed at the air/liquid interface to form and stabilize the foam. The orthogonal test is a method used in multiple factors analysis, which is the method of partial factor design by selecting the representative points from the comprehensive test. To determine the optimal foaming condition of the generator, the foaming agents were optimized by means of orthogonal experiment and extremum difference analysis. A five-factors four-levels including AES, 6501, K12, PAM and FM-550 were designed and a total of 16 tests were performed to analyze their foam expansion ( $F$ ) and half-life ( $t_{1/2}$ ).<sup>28</sup> In the experiment, the basic system was 200 g of water. Factor level values are shown in Table I and the scheme of the orthogonal test are shown in Table II. Each group of experiments was repeated five times, and the average value of the three with good precision was recorded.

TABLE I. Factors and levels of foaming agent compounding system

Level	Content, g				
	AES	6501	K12	PAM	FM-550
1	0.0	0.0	0.0	0.0	0.0
2	3.0	1.0	3.0	0.1	1.0
3	4.5	2.0	4.5	0.15	1.5
4	6.0	3.0	6.0	0.2	2.0

TABLE II. Orthogonal experiment of foaming agent compound system

Number	Content, g					$F$	$t_{1/2}$ / min
	AES	6501	K12	PAM	FM-550		
1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
2	0.0	1.0	3.0	0.1	1.0	38.4	224
3	0.0	2.0	4.5	0.15	1.5	30.0	294
4	0.0	3.0	6.0	0.2	2.0	25.6	246
5	3.0	0.0	3.0	0.15	2.0	32.7	335
6	3.0	1.0	0.0	0.2	1.5	19.2	426
7	3.0	2.0	6.0	0.0	1.0	42.5	223
8	3.0	3.0	4.5	0.1	0.0	53.4	42
9	4.5	0.0	4.5	0.2	1.0	32.2	327
10	4.5	1.0	0.0	0.15	0.0	38.8	37
11	4.5	2.0	6.0	0.1	2.0	26.8	280
12	4.5	3.0	3.0	0.0	1.5	28.2	256
13	6.0	0.0	6.0	0.1	1.5	29.2	294
14	6.0	1.0	4.5	0.0	2.0	25.2	310
15	6.0	2.0	3.0	0.2	0.0	41.4	56
16	6.0	3.0	0.0	0.15	1.0	23.2	471

In order to systematically analyze the degree of influence of different foaming agents, the range analysis method was introduced according to the orthogonal test.

The results showed that the sequence of the influence of surfactant on the  $F$  and  $t_{1/2}$  were as follows: K12>AES>PAM>6501>FM-550 (Table III), FM-550 >PAM>AES>K12>6501 (Table IV). The best array was  $A_2B_3C_3D_2E_2$  and  $A_4B_4C_4D_3E_3$ , respectively. These results suggested that the surfactant plays the main role in the process of fire-retardant foam. Based on the range analysis, the best proportion of the foaming agent compounding system is AES:6501:K12: :PAM:FM-550 = 6:6:9:0.3:2 (mass ratio, 200 g water, which was recorded as the foaming agent in the follow-up experiments).

TABLE III. Analysis of foam expansion ratio range;  $K$  is the sum of the foaming multiples in parallel experiments,  $k$  is the average foaming multiples,  $A$  is the experimental results of AES,  $B$  is the experimental results of 6501,  $C$  the experimental results of K12,  $D$  the experimental results of PAM,  $E$  the experimental results of FM-550

Parameter	$F$				
	$A$ (AES)	$B$ (6501)	$C$ (K12)	$D$ (PAM)	$E$ (FM-550)
$K1$	94.0	94.1	81.2	95.9	133.6
$K2$	147.8	121.6	140.7	147.8	136.3
$K3$	126.0	140.7	140.8	124.7	106.6
$K4$	119.0	130.4	124.1	118.4	110.3
$k1$	23.5	23.5	20.3	24.0	33.4
$k2$	37.0	30.4	35.2	37.0	34.1
$k3$	31.5	35.2	35.2	31.2	26.6
$k4$	29.8	32.6	31.0	29.6	27.6
Range	13.5	11.7	14.9	13.0	8.5
Primary and secondary sequence order $C>A>D>B>E$					
Optimization	$A_2$	$B_3$	$C_3$	$D_2$	$E_2$

TABLE IV. Analysis of foam half-life range

Parameter	$t_{1/2} / \text{min}$				
	$A$ (AES)	$B$ (6501)	$C$ (K12)	$D$ (PAM)	$E$ (FM-550)
$K1$	764	956	934	789	135
$K2$	1026	997	871	840	1245
$K3$	900	853	973	1137	1270
$K4$	1131	1015	1043	1035	1171
$k1$	191	239	233	197	34
$k2$	256	249	218	210	311
$k3$	225	213	243	284	318
$k4$	282	254	261	264	293
Range	91	41	43	87	284
Primary and secondary sequence order $E>D>A>C>B$					
Optimization	$A_4$	$B_4$	$C_4$	$D_3$	$E_3$

### *Foaming agent and water ratio (foaming liquid)*

The best proportion of foaming agent were mixed with water in the mass ratio of 1:10, 1:15, 1:20, 1:25, 1:30 (recorded as the foaming liquid) to study the foaming capacity and foam stability. As shown in Fig. 3, with other condition remaining unchanged, the  $F$  and  $t_{1/2}$  of the foam first increase and then decreases with decreasing water, when 1:20 was added, both had the maximum at 34.2 and 7.5 h respectively, which depicts that the foaming agent and water ratio play other main roles for the foaming capacity and foam stabilization.

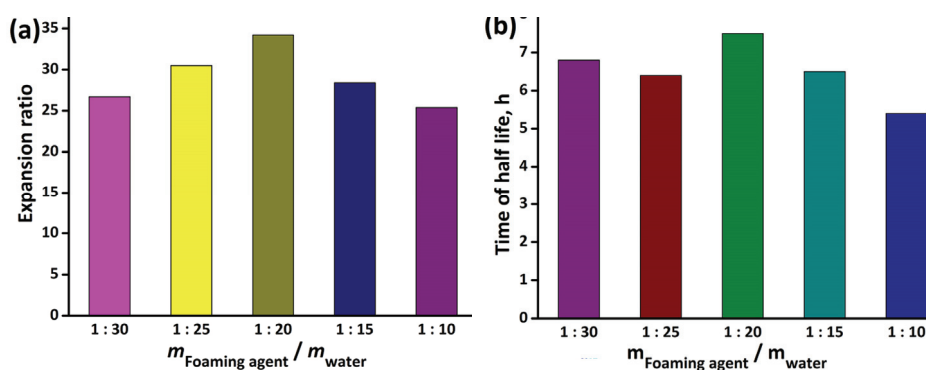


Fig. 3. Optimal ratio of foaming agent and water.

### *The ratio of foaming liquid/FA*

FA particles are added to the foaming liquid (1:20 ratio of foaming agent and water) to form a three-phase foam system. Comparing the  $F$  and  $t_{1/2}$ , the performance of three-phase foam is evaluated under different solid-liquid ratios. As observed in Fig. 4, with increasing FA mass fraction, the  $t_{1/2}$  values of the foam increased, but foam volume decreased continuously, thereby indicating that the FA can increase foam stability, but has an inhibitory effect on foam volume. The enhancement of foam stability of three-phase foam was attributed to the existence of FA particles. The attached particles decrease the pressure difference between the gas/liquid interface, slowing down the drainage process. Foam fire extinguishing mainly depends on covering the surface of burning objects to prevent them from contacting oxygen and continuing burning. Therefore,  $F$  has a greater impact on the foam extinguishing property than its  $t_{1/2}$  period. According to experiment results, when the solid to liquid ratio is lower than 1:3, the expansion ratio below 10, limits its practical application as a fire extinguisher. When choosing the proportion with the solid to liquid ratio of 1:4 for the fire-retardant foam, not only ensures comprehensive properties such as  $F$  and stability are the best, but the use of stabilizer can also be reduced at the same time, and the  $F$  meets the current requirements of coal mine fire-extinguishing materials. By focusing on its  $F$  and  $t_{1/2}$ , a solid-liquid ratio of 1:4 was determined. Based on

this, the optimized FA three-phase foam system has excellent stability with a foam  $t_{1/2}$  that can reach more than 28.7 h.

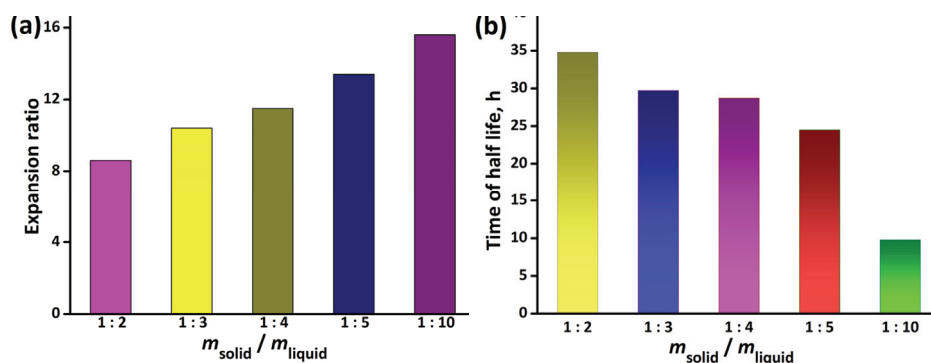


Fig. 4. Influence of solid-liquid ratio on the performance of three-phase foams.

#### FA particle size

FA particle is widely applied in the physical property test. To avoid particle size effect, specific graded particles were selected for physical parameter measurement experiments.<sup>29</sup> In lab research, there are some particle size distribution testing such as sieving, laser particle analyzer, *etc.* Sieving is the simplest powder particle size test method, the size of the lower and upper standard sieves as the lower and upper limits of the particle size. That is, broken FA samples can be separate by sieves of different sizes, and the FA particles after sieving were divided into different particle groups.: <38, 38–75, 75–106, 106–150 and 150–380  $\mu\text{m}$ . Fig. 5 shows that with increasing particle size, the foam  $t_{1/2}$  tends to stabilize and then decrease.

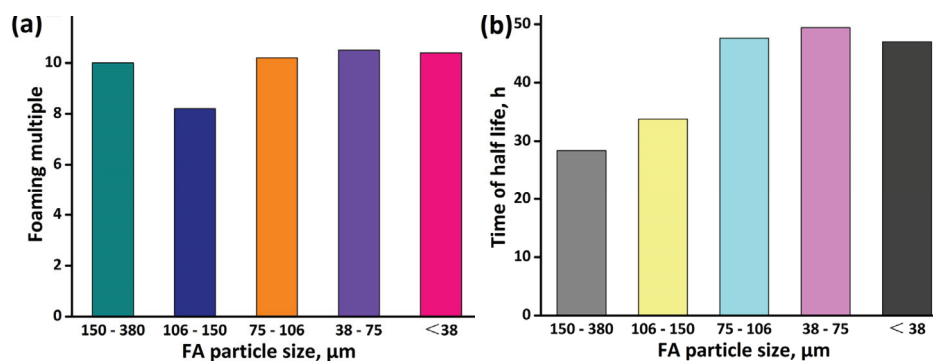


Fig. 5. Influence of particle size on the properties of three-phase foam.

No significant differences were observed in values of the  $F$  among smaller particle fractions (<75  $\mu\text{m}$ ). Thus, smaller particles can keep good dispersion in



the dispersion liquid, and hence the results can be grained large area. The larger particles are not suitable for stabilization because they are dispersed difficultly and the gravity in the aqueous foam leads to foam decay.

### *pH*

In actual application in coal mines or oil fields, foam is often used in areas where coal or oil are prone to spontaneous combustion. In such places, the pH value differs from respective levels in the laboratory. Therefore, it is necessary to study the influence of pH value factors on the characteristics of the foam, and hence, the pH value of the foaming system was adjusted to about 4, 5, 6, 7, 8, 9, and 10. Fig. 6 shows that with increasing pH value, both the foaming ability and foam stabilization first increase and then decrease. This is mainly because at a low pH value, the foaming solution is acidic, and the  $H^+$  in the solution will react with the anionic foaming agent (such as:  $-SO_3^-$ ), resulting in a decreased effective content of the foaming agent and weakened foaming ability.

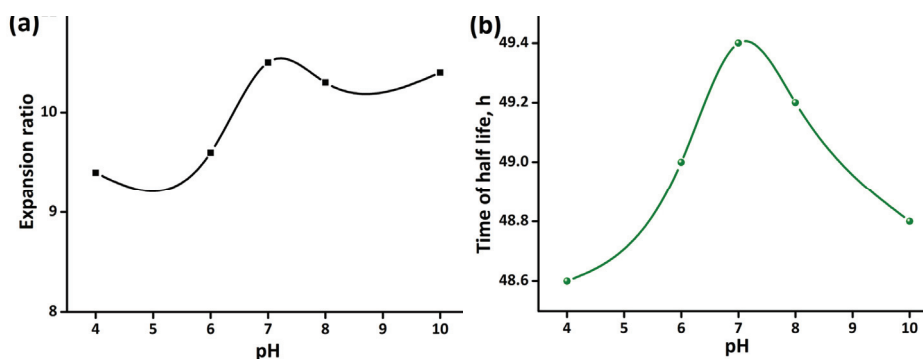


Fig. 6. Influence of pH on the performance of three-phase foam.

With increasing pH value, the relative effective content of the foaming agent increases, and foaming capacity is increased. When the pH value exceeds 7, the  $OH^-$  combines with the hydrophilic group in the foaming agent, which affects the foaming effect. However, changes of the  $F$  and  $t_{1/2}$  were not obvious, so they can be used for wider application fields.

### CONCLUSIONS

This study combines the advantages of foam fire-extinguishing technologies, and a new type of mine fire-extinguishing foam was prepared. Firstly, the optimum ratio of foaming agents was obtained by orthogonal experiment and variance analysis for different factors and levels. Then, FA particles were used as the solid phase to generate three-phase foams. The effects of solid to liquid ratio, FA size distribution and the system pH on the foaming multiple and stabilization time of the foam were assessed. Under the optimized operating conditions of 1:4

solid–liquid ratio, smaller particle fractions ( $<75 \mu\text{m}$ ) and from weak acid to weak base, the fly ash-based three-phase foam showed satisfactory stability and foaming ability. These investigations provided a novel route for preparing high-efficiency fire-extinguishing foams applicable in the confined space of underground coal mines.

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## ИЗВОД

ЕКСПЕРИМЕНТАЛНО ИСПИТИВАЊЕ ФАКТОРА КОЈИ УТИЧУ НА ПРИПРЕМУ ТРОФАЗНЕ ПЕНЕ

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Трофазна пена се сматра једним од перспективних напредних материјала за гашење пожара. Међутим, услови припреме, цена и ефекат су кључни фактори за индустријску примену. У овој студији предложен је нови трофазни систем пене са летећим пепелом и комплексним сурфактантом. Пет типова сурфактаната: алкохол полиокси-етилен-етар, натријум-сулфат, диетаноламин кокосово уље, натријум-лаурил-сулфат, полиакриламид и емулзија силиконске смоле модификоване полиетаром су одабрани као средства за пењење. Кроз лабораторијске експерименте, испитан је утицај на степен експанзије и стабилност пене типа/концентрације сурфактанта, концентрације/величине честица летећег пепела и рН. Стање пене је одређено нумеричком оптимизацијом. Резултати ове студије могу послужити као референца за разумевање припреме нове трофазне пене. Надамо се да овај рад може да пружи корисне смернице за припрему ефикасне трофазне пене за гашење пожара за безбедно чување безбедности процеса у области хемијске производње, транспорта и складиштења.

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