Enhanced foam-stabilizing performance by adding magnesium aluminum silicate compared with sodium bentonite independent of temperatures

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Abstract

High-temperature-resistant foam stabilizers are desperately demanded for high-temperature conditions, such as geothermal well. In this study, nano-magnesium aluminum silicate (NMAS) and micro-magnesium aluminum silicate (MMAS) are initially studied and compared with sodium bentonite (Na-BT) as high-temperature-resistant foam stabilizers. We find that increasing temperature could facilitate aggregation of clay dispersion, and the increase of particle size results in significant positive effect on apparent viscosity (AV), interface dilational modulus (E), foam film thickness, and eventually drainage half-life ($T_{0.5}$) of the foam. Simultaneously, it leads to a negative impact on the initial foam volume (V_0) and

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foam diameter. Moreover, NMAS even possesses excellent foam stabilization effect after being aged at 320 °C for 16 h that it could still dramatically extend $T_{0.5}$ to 9.78 h, whereas $T_{0.5}$ of MMAS- and Na-BT-stabilized foam are merely 45.52 and 13.78 min, respectively. Furthermore, V_0 of Na-BT-stabilized foam cannot resist the influence of NaCl and CaCl_2 even when only 1.0 wt% NaCl or 0.1 wt% CaCl_2 is added. By comparison, V_0 of NMAS- and MMAS-stabilized foam drilling fluids are comparatively not sensitive to NaCl and CaCl_2 until the concentration of 3.0 wt% and 0.2 wt%, respectively. Not only has our study provided a guideline for using clay mineral as a foam stabilizer in various high-temperature conditions, but also find an excellent high-temperature-resistant foam stabilizer, NMAS, which could bring considerably satisfying foam-stabilizing performance even at temperatures as high as 320 °C.

Keywords: aqueous foam, foam stabilizers, magnesium aluminum silicate, sodium bentonite, high temperature

1 Introduction

Geothermal energy is an environmentally friendly and renewable resource. Owing to the huge economic potential and bright application prospect, geothermal energy has attracted extensive attention worldwide. Lund et al. claimed that the geothermal energy has contributed about 592,638 TJ (164,635 GWh) in 2015, which is increased by about 39.8% over 2010, resulting in a growing rate of 6.9% annually¹.

However, for medium- or low-temperature geothermal resources ($< 150\text{ }^{\circ}\text{C}$), current exploration equipment and technologies cannot achieve efficient utilization. After being transported to the surface, those medium- or low-temperature geothermal resources are only capable of heating, spa treating, and planting². However, high-temperature geothermal resources ($> 150\text{ }^{\circ}\text{C}$) can be implemented in power generation industry, which not only meets diverse needs and brings enormous economic profit, but also makes a great contribution to avoiding environmental pollution of using conventional method^{3, 4}. Thus, high-temperature geothermal resources ($> 150\text{ }^{\circ}\text{C}$) deserve preferably to be exploited. In addition, geothermal resources with higher temperatures are more appealing due to the enormous potential, while they probably face much more difficulties in the drilling process. To construct safe and effective high-temperature geothermal wells, drilling fluids should be designed strictly based on the characteristics of geothermal wells.

The abnormally low formation pressure developed in geothermal resources easily leading to severe leakage should be primarily paid attention to. To solve this problem, foam drilling fluid is thought the most suitable drilling fluid and has been utilized in some drilling geothermal wells owing to the low density and Jamin effect⁵. However, in the perspectives of thermodynamics and dynamics, foams are essentially unstable especially in severe high temperature and high pressure condition, which is disadvantageous for safe and efficient drilling⁶. High-temperature is also another biggest challenge for drilling geothermal wells. Therefore, it is necessary to introduce foam stabilizers to enhance foam stability. Currently, foam stabilizers mainly cover

macromolecular substance and nonionic surfactant, such as polyacrylamide, carboxymethyl cellulose, hydroxyethyl cellulose, and N, N-dimethyldodecylamine-N-oxide^{7, 8}. However, as temperature increases, thermal degradation is inevitable for these macromolecular substances and they will completely degrade when the temperature rises up to 180 °C. As regards nonionic surfactant, cloud point effect greatly limits the scope of use mainly at high temperatures⁹. Therefore, it is desperately necessary to develop new high-temperature-resistant foam stabilizers that can be applied in high-temperature geothermal wells.

In recent years, solid particles are widely used as foam stabilizers in food¹⁰, cosmetics¹¹, enhanced oil recovery^{12, 13} and fracturing¹⁴. Binks et al. fabricated a stable foam by utilizing silica nanoparticles as foam stabilizer¹⁵. Gonzenbach et al. grafted short-chain amphiphiles to the surface of particles by in-situ hydrophobization and these particles were found to be able to stabilize foams¹⁶. Sun et al. added partially hydrophobically-modified SiO₂ nanoparticles into sodium dodecyl sulfate solution to enhance foam stability¹⁷. Faure et al. modified the surface of hydrophilic alumina particles via introducing short fluorinated phosphoric acid and found fluorinated particles at a concentration of 100 g·L⁻¹ could dramatically extend drainage half-life ($T_{0.5}$) to 10 h¹⁸. Our group fabricated Janus particles that can extend the drainage half-life to 668 s after hot rolling at 280 °C for 16 h and resist CaCl₂ concentration of 0.8 wt%¹⁹. Thus solid particles seem to be promising candidates for using as foam stabilizers under high temperature conditions.

Montmorillonite is a group of layered phyllosilicate solid particles and able to disperse well in water. Owing to super dispersion ability in water, low cost and environmentally friendly property, Na-bentonite (Na-BT), naturally occurring and mainly consisting of montmorillonite, has been widely utilized as a basic material in water-based drilling fluid for viscosifying and filtration control purpose^{20, 21}. Nevertheless, only very few researches have used clay particles as foam stabilizers. Luo et al. found 5 wt% Na-BT could extend $T_{0.5}$ to 6000 s and diameter of the bubble will decrease as Na-BT increasing²². Besides, Na-BT can increase apparent viscosity (AV) and interface dilational modulus (E) of base fluid at room temperature, which are favorable for slowing down the process of drainage and coalescence. Thus, montmorillonite is expected to be used as an effective foam stabilizer. However, the stabilizing performance at high temperatures has not been studied, although it is of vital importance for deep wells including geothermal wells.

Actually, the utilization of natural clay was restricted owing to the variability of the composition and properties of natural minerals depending on occurrence. In order to prepare montmorillonites with reproducible and homogeneous characteristics, including size and surface, as well as mineral composition and chemical composition, many synthetic smectite clays were developed in chemical industries, such as nano-magnesium aluminum silicate (NMAS) and micro-magnesium aluminum silicate (MMAS). They are composed of two silicon-oxygen tetrahedral sheets surrounding either a magnesium-oxygen or aluminum-oxygen octahedral sheet^{23, 24}. Thus, they belong to the group of (2:1) phyllosilicates, similar to Na-BT. The

difference between NMAS and MMAS is that the former are completely synthetic and composed of monolayered disc-shaped particles with a diameter of 30 nm and a thickness of 1 nm, while the latter is usually purified from natural saponite and montmorillonite minerals, and thus the composition, particle distribution, and morphology of MMAS are similar to Na-BT. After being dispersed in water, they all display negative charged face and positive charged edge of the layer. Owing to the large specific surface area, the synthetic clays possess more excellent viscoelasticity and thixotropy performance compared with Na-BT²⁵. Therefore, they are usually applied to suspend and stabilize medicines and pigments^{26, 27}. Jiang et al. demonstrated NMAS is a high-performance rheological modifier in water, exhibiting better shear-thinning performance, higher gel strength, and quicker recovery ability of gel structure compared with Na-BT²⁸. Thus, magnesium aluminum silicate (NMAS and MMAS), in our opinion, is expected to be a prospective candidate for foam stabilizer.

In this study, the performance of NMAS and MMAS as high temperature-resistant foam stabilizers were studied and compared with Na-BT. After being hot rolled at a series of temperatures, the initial foam volume (V_0) and $T_{0.5}$ of NMAS, MMAS or Na-BT base fluid were evaluated utilizing the Waring blender method. Other properties of each base fluid sample and the corresponding foam were systematically measured. On the basis of these results, the underlying foam stabilization mechanism of NMAS, MMAS, and Na-BT were uncovered. The results indicate MAS-stabilized foams, especially that stabilized by NMAS possess more

excellent stability and salt tolerance compared with Na-BT-stabilized foam.

2 Experimental section

2.1 Materials

Nano-magnesium aluminum silicate (NMAS) was purchased from Sheng Xin Chemical Technology Co., Ltd. (Guangzhou, China). Micro-magnesium aluminum silicate (MMAS) was obtained from Peng Tai Technology Co., Ltd. (Hunan, China). Na-BT was supplied by Alfa Aesar (Shanghai, China). The anionic foaming agent AD300 was provided by Aoda Petrochemical Co., Ltd. (Shandong, China). All chemicals were directly used without further purification.

2.2 Characterization of NMAS, MMAS, and Na-BT

X-ray fluorescence (XRF) (AxiosmAX, Netherlands) was adopted to analyze the chemical compositions of the NMAS, MMAS, and Na-BT. The mineral compositions were determined by X-ray diffraction (XRD) (Bruker D8 Advance, Germany)²⁸.

The size distribution of NMAS was measured by a Malvern Zetasizer Nano series (Malvern, UK), while that of MMAS and Na-BT were conducted on a Mastersizer 2000 analyzer (Malvern Instruments Ltd., UK). All samples were homogeneously dispersed in deionized (DI) water for 24 h in advance and ultrasonically dispersed again for 30 min before the measurement. The average size was obtained by measuring each sample 2 times.

The ζ potentials of NMAS, MMAS, and Na-BT in water were measured using a

Malvern Zetasizer Nano series (Malvern, UK). The concentrations of all samples were $0.3 \text{ g}\cdot\text{L}^{-1}$. Prior to the measurement, all samples were prepared by sonication dispersion method for 30 min. The average ζ potential was obtained by repeating the measurements 3 times.

The microstructures of the NMAS, MMAS and Na-BT particles dispersed in water were observed by a Leica DM4M optical microscope (Leica Microsystems, Germany) and an F20 transmission electron microscope (TEM) (FEI Corporation, America) with a 20 kV accelerating voltage. As for TEM sample, the NMAS, MMAS and Na-BT suspensions were diluted to 0.1 wt% using DI water. After an ultrasonic dispersing procedure for 30 min, they were dropped onto a carbon film-coated copper grid. The samples were immediately observed when they were thoroughly dried naturally in air.

Langmuir specific surface area were measured using a Kubo X1000 (Builder, China). Before the measurements, the samples were degassed under vacuum for 2 h at 200°C .

2.3 Preparation of the NMAS, MMAS and Na-BT base fluids

At room temperature, NMAS, MMAS, and Na-BT dispersions in DI water at concentrations of 1.5, 3.0, and 3.0 wt%, respectively, were prepared by dispersing the powder into DI water by mechanical stirring at 600 rpm for 24 h. After being fully hydrated, 0.6 wt% AD300 was added into each suspension. Hereafter, the mixture dispersions were nominated as NMAS, MMAS, and Na-BT base fluids and studied in

the following experiments.

To simulate the high-temperature environment, the hot rolling experiments were adopted to study the influences of the high-temperature aging on base fluid properties. According to the American Petroleum Institute (API) guidelines, 350 mL of NMAS, MMAS or Na-BT base fluid was individually poured into an aging cell (Tuochuang Instrument Company, China) and subsequently rolled at a certain temperature (25, 80, 120, 140, 160, 200, 240, 280, 300, or 320 °C) for 16 h in a roller oven (Tuochuang Instrument Company, China). After cooling down, the thermally aged base fluids were obtained.

2.4 Performance of NMAS, MMAS, and Na-BT on foaming ability and foam stabilization before and after being aged at different temperatures

The foam was tested by the Waring blender method as follows. 100 mL of base fluid was vigorously stirred at 11,000 rpm for 1 min. After stirring, foams were prepared and quickly poured into a measuring cylinder within 10 s. The V_0 and $T_{0.5}$ were recorded, describing the foaming ability and foam stability, respectively. To be noted, $T_{0.5}$ corresponded to the time required for drainage of 50 mL base fluid from foams in this study.

About 100 μL of foam stabilized by NMAS, MMAS or Na-BT was sprayed on the slide and gently covered by a coverslip. Then, they were observed by the optical microscope mentioned above, and the photos were taken by a Leica DM4M electron multiplying CCD camera (Leica Microsystems, Germany). Then, the bubble diameter and film thickness were obtained after analysis of the photos by utilizing ImageJ

software (National Institutes of Health, USA).

2.5 Mechanism exploration

The rheological properties of base fluid were measured by a ZNN-D6L rotational viscometer (Qingdao, China). The rotational viscometer was operated at a fixed rate of 600, 300, 200, 100, 6, and 3 rpm. The apparent viscosity (AV), plastic viscosity (PV) and yield point (YP) was calculated according to the following formulas:

$$AV = \theta_{600} / 2 \quad (1)$$

$$PV = \theta_{600} - \theta_{300} \quad (2)$$

$$YP = 0.51(\theta_{300} - PV) \quad (3)$$

$$Gel_{10s} = \theta_3 / 2 \quad (4)$$

Where θ_{600} , θ_{300} , θ_3 are the readings of the viscometer at 600, 300 and 3 rpm, respectively.

The surface tension and interfacial dilational modulus (E) were determined by a tensionmeter (Tracker-H, TECLIS, France) at a relative volume variation of 10% and a sinusoidal oscillation frequency of 0.1 Hz²⁹.

The particle size distribution of based fluids was measured in the same method as above (2.2 Characterization of NMAS, MMAS, and Na-BT).

2.6 Performance of NMAS, MMAS, and Na-BT as foam stabilizers after being aged at 320 °C

2.6.1 Stability of the foam

100 mL of base fluid after being aged at 320 °C was stirred at 11,000 rpm for 1

min to fabricate foam. Then the disproportionation and coalescence of bubbles over time were observed by the optical microscope mentioned above. In addition, foam was poured into a measuring cylinder to observe drainage and collapse degree of foams at fixed intervals.

2.6.2 Salt tolerance evaluation

100 mL of base fluid after being aged at 320 °C was stirred at 11,000 rpm for 1 min to fabricate foam. Then, NaCl and CaCl₂, at a series of concentrations, were added into the foam and stirred at 11,000 rpm for 1 min again. Then V_0 and $T_{0.5}$ were recorded in the same way as described above.

3 Results and discussions

3.1 Characterization of NMAS, MMAS, and Na-BT

NMAS is nano-sized synthetic clay, and has been washed and purified thoroughly; MMAS is micro-sized and produced after a series of fine processing such as purification, modification, activation, repair, and gelation from natural saponite and montmorillonite minerals; whereas Na-BT is mainly comprised of natural montmorillonite. For a complete understanding of NMAS, MMAS and Na-BT, mineral and chemical compositions were firstly characterized (Figure S1 and **Figure S2**). Based on the XRD quantitative analysis (Table 1), we find that NMAS contains 98.8 wt% smectite and 1.2% quartz; MMAS is mainly composed of 52.9 wt% smectite and 27.8 wt% plagioclase; while the constituent of Na-BT includes 74.8 wt%

smectite and 23.3 wt% calcite in our study. Thus, NMAS possess more smectite than MMAS and Na-BT. It can be seen that smectite is the main component of NMAS, MMAS and Na-BT, which can swell and disperse well in water and make these three chemicals play an important role in various fields. In addition, according to the results of XRF, there are 3.68, 4.09 and 28.31 wt% MgO in Na-BT, MMAS and NMAS, respectively (Table 2), which is in fact, is attributed to the different saponite mineral content. Thus, Na-BT and MMAS possess similar percentage of saponite, which is much lower than NMAS. Compared with montmorillonite, saponite is easier to produce nanoscale size, owing to lower hardness and superior cleavage.

Table 1. Mineral compositions of the NMAS, MMAS, and Na-BT (wt%).

	Smectite	Quartz	Potassium feldspar	Plagioclase	Calcite	dolomite	Magnesite	Hematite	Anhydrite
NMAS	98.8	1.2	-	-	-	-	-	-	-
MMAS	52.9	7.4	6.4	27.8	2.9	1.8	0.8	-	-
Na-BT	74.8	-	-	-	23.3	-	-	1.4	0.5

“-”: no presence of the item.

Table 2. Chemical compositions of the NMAS, MMAS, and Na-BT (wt%).

	SiO ₂	Na ₂ O	Al ₂ O ₃	MgO	CaO	Fe ₂ O ₃	SO ₃	K ₂ O	P ₂ O ₅	TiO ₂
NMAS	66.23	3.27	0.37	28.31	0.04	0.04	0.36	-	0.01	-
MMAS	73.21	3.73	15.40	4.09	0.46	1.16	0.02	1.64	0.02	-
Na-BT	64.68	4.63	18.71	3.68	0.95	5.07	0.44	0.77	0.21	0.59

“-”: no presence of the item.

Most of clay minerals generally disperse in water to form plate-like particles³⁰. The characteristics of the suspensions, such as particle size distributions and ζ potential, are important for the bulk properties. Despite the lamellar structure, the DLS can tell the difference of size in general. Just as Figure 1(a) shown, the median particle size (d_{50}) of NMAS is 19.691 nm and much smaller than that of MMAS and

Na-BT, which are 4.219 μm and 4.655 μm , respectively. It can be seen NMAS and MMAS display different size distributions and the latter has similar distribution with Na-BT, following the sequence: $\text{NMAS} < \text{MMAS} < \text{Na-BT}$, which is also observed in optical microscopy images (Figure 1(d1-d3)). There is nothing could be discerned in NMAS dispersion because of its nano-size. By contrast, MMAS and Na-BT particles were large enough to be observed. Owing to electrostatic interaction between negatively charged face and positively charged edge of the plates, three-dimensional network structure resembling card palace would be created by these plates throughout the dispersion³¹. The ζ potential of NMAS, MMAS, and Na-BT is measured -28.2, -26.4, and -21.3 mV, respectively as shown in the inserted image (Figure 1b). The higher ζ potential indicates the larger electrostatic repulsive force between face and face, inferring easier to form a three-dimensional network structure through edge-face attraction according to literatures^{32, 33}. Apparently, NMAS is the most liable to form a three-dimensional network structure and exhibit thixotropic property. This can also been verified in TEM experience. As shown in Figure 1(e1), there are numerous round discs with a diameter of approximately 30 nm. In regard to MMAS and Na-BT, big and thick layers are observed, with a two orders of magnitude larger size than NMAS (Figure 1(e2 and e3)). Langmuir specific surface area of NMAS is 21.9 m^2/g and much larger than that of MMAS and Na-BT, which are 16.7 and 5.1 m^2/g , respectively. Owing to a smallest size and largest specific surface area, NMAS normally only requires a low concentration to form a three-dimensional network structure. As for MMAS and Na-BT, it requests a higher particle concentration to gain

a certain gel strength³⁴, which has been confirmed in Figure 1(c1-c3). 3.0 wt% NMA suspension could form a gel and still hold on to the bottle bottom after inversion. However, 3.0 wt% MMA and Na-BT suspension cannot achieve this.

Figure 1. (a) Particle size distribution, (b) ζ potential, (c1-c3) optical microscope images, (d1-d3) digital photographs, and (e1-e3) TEM images of NMA, MMA, and Na-BT particles.

3.2 Performance of NMAS, MMAS, and Na-BT on foam stabilization before and after being aged at different temperatures

In this study, we have evaluated the performance of NMAS, MMAS, and Na-BT on foam stabilization before and after being aged at different temperatures. Firstly, we selected an anti-high temperature anionic surfactant AD300 as the foaming agent. Figure 2(a) shows that the critical micelle concentration (CMC) of AD300 is 0.35 wt%. Due to the adsorption of surfactant molecules onto the surface of clay and tendency of hydrophilic clay to suspend in liquid phase, a concentration above CMC is needed for considerable foam quality. Thus, the concentration of 0.6 wt% for AD300 was determined for all following experiments. From Figure 2(b), we can see that V_0 of NMAS-stabilized foam decreases, whilst $T_{0.5}$ increases as the concentration of NMAS increases from 1.0 to 3.0 wt%. The stabilizer has a contrary effect on foaming ability and foam stability, similar with the previous reports^{35, 36}. It is difficult for the base fluid to generate foam when the concentration of NMAS exceeds 2.0 wt%, and the corresponding V_0 is smaller than 400 mL. It might be interpreted that when the concentration of NMAS is too high, the base fluid easily forms a strong three-dimension network structure, which impedes the foaming process. Thus, the concentration of NMAS was adjusted to 1.5 wt% to compare the foam stability performance with base fluids containing 3.0 wt% MMAS and Na-BT, and the latter ones were determined according to clay tolerance of drilling fluids at high temperature³⁷. Combined with the mineral composition results, it is worth mentioning the percentage of smectite in NMAS base fluid is the lowest (1.48 wt%), while that in

Na-BT base fluid is the highest (2.48 wt%), and that in MMAS base fluid is in-between (1.59 wt%).

Subsequently, the foaming ability and foam stability of base fluids were compared with blank one (only contain 0.6 wt% AD300) after being separately aged at 25, 80, 120, 140, 160, 200, 240, 280, 300 and 320 °C for 16 h. As shown in Figure 2(c), V_0 of blank one slowly decreases with the temperature rising and always larger than 590 mL, while V_0 of NMA base fluid decreases from 425 to 350 mL as the hot rolling temperature increases from 25 to 160 °C and then begins to increase with the temperature rising. Similarly, V_0 of MMAS base fluid decreases from 510 to 375 mL when the hot rolling temperature increases from 25 to 240 °C, and then increases continually when the temperature is higher than 240 °C. However, it exhibits two inflection points at 140 and 280 °C on the curve of V_0 versus temperature for Na-BT base fluid, respectively. Apparently, the effect of temperature on the foaming ability of base fluids added with Na-BT, MMAS and NMA are different, which can be used as a reference for drilling high-temperature wells. Additionally, V_0 of MMAS base fluid is much larger than that of Na-BT and NMA base fluids, indicating the better foaming ability of MMAS base fluid, when the temperature is lower than 200 °C. When the temperature rises to 320 °C, a reversal phenomenon occurs that NMA base fluid generate the largest V_0 of foam and MMAS base fluid possesses the smallest V_0 . Correspondingly, foam quality has similar trend as V_0 . Owing to lower quality, sphere bubble tends to appear in NMA-stabilized foams. Thus, the addition of particle will reduce the foaming ability. The difference in foaming ability may be

related to surface tension and AV of base fluids, which together decide the foaming efficiency under the same stirring condition.

When it comes to drainage half-life, $T_{0.5}$ of blank one always keeps at around 7 min. However, $T_{0.5}$ of NMAS-stabilized foams increases quickly from 189 to 1105 min with temperature increasing from 25 up to 160 °C and then $T_{0.5}$ gradually decreases to 586 min at 320 °C. In sharp contrast, $T_{0.5}$ of MMAS-stabilized foams rises slowly as the temperature increases up to 200 °C and abruptly reaches its maximum of 211 min at 240 °C and then it gradually falls down. Interestingly, the curve of $T_{0.5}$ versus temperature for Na-BT-stabilized follows a fluctuating character. In detail, $T_{0.5}$ of Na-BT-stabilized foams reaches its two peaks at 140 °C and 280 °C, respectively and experienced the valley at 200 °C. Obviously, NMAS exhibits excellent foam stabilization performance compared with MMAS and Na-BT at all temperatures, though containing the lowest concentration of smectite. In addition, the foam stabilization performance of NMAS in our study is much better than 5% Na-BT reported by others, whose $T_{0.5}$ is 6000 s at 25 °C²².

Film thickness and bubble diameter are basic characteristics of foam. By analyzing lots of photographs as shown in Figure S3 and Figure S4, the average film thickness and bubble size were depicted in Figure 2(e) and Figure 2(f), respectively. It can be seen that that the average film thickness of NMAS-stabilized bubbles drastically increases as temperature rises up to 160 °C and then gradually declines. The average film thickness of MMAS-stabilized bubbles slowly increases as temperature rises up to 160 °C and it abruptly increases to the maximum at 240 °C

before a gradual descent. Different from above foams, the average film thicknesses of Na-BT-stabilized bubbles reach peaks at 140 and 280 °C, respectively, and in this temperature range, the minimum thickness appears at 200 °C. It is easy to find that trends of average film thicknesses are so similar to $T_{0.5}$, indicating a positive relationship between film thicknesses and $T_{0.5}$. In fact, thick film could prevent or slow down foam fracture in the case of liquid drainage and air penetration³⁸. Thus, thicker film is beneficial for slower liquid drainage and air penetration. Finally, disproportionation and coalescence of foams will be greatly mitigated and foams become considerable stable.

From Figure 2(f), we can see that the average diameter of NMAStabilized bubbles decreases continuously when temperature increase from 25 to 160 °C and then begin to increase with the temperature rising. The average diameter of MMAS-stabilized bubbles gradually decreases as temperature increasing from 25 to 280 °C, with a plateau at around 80 - 120 °C, and then increases when aging temperature exceeds 280 °C. As for average diameters of Na-BT-stabilized bubbles, the largest average diameter occurs at 25 °C. As the aging temperature increases, the average bubble diameter decreases rapidly from 406 to 263 μm at 80 °C. Then the average diameter trough appears at 140 and 280 °C and a crest forms at 200 °C. The variation of average bubble diameter may be related to surface tension and AV of base fluids, which are two key factors in the foaming process.

It is further concluded that the effect of temperature on $T_{0.5}$ is quite opposite to V_0 (Figure 2(c) and 2(d)). Given that both V_0 , $T_{0.5}$, average film thickness and average

bubble diameter vary in such different trends for NMAS, MMAS, and Na-BT base fluids when the temperature rises from 25 to 320 °C, it should be figured out what effects have high temperature have brought to these base fluids.

Figure 2. (a) The surface tension of AD300 solutions at various concentrations ranging from 0 to 0.8 wt%. (b) V_0 and $T_{0.5}$ of NMAS-stabilized foams at various concentrations ranging from 1.0 to 3.0 wt%. (c) V_0 and foam quality, (d) $T_{0.5}$, (e) average film thickness, and (f) average bubble diameter of NMAS-, MMAS-, and Na-BT-stabilized foams after being aged at 25, 80, 120, 140, 160, 200, 240, 280, 300 and 320 °C, respectively.

3.3 Mechanism analysis

3.3.1 Characterization of base fluids

Properties of base fluid exert a decisive influence on V_0 and $T_{0.5}$ of foam. Accordingly, surface tension, AV, E and particle size distribution of base fluids were investigated in this study. From Figure 3(a), it is found that surface tensions of base fluids added with NMAS, MMAS, or Na-BT decrease slightly initially and then increase a little with temperature increasing, although the foaming ability and foam stability change significantly as temperature increases. Therefore, the influence of temperature on foaming ability is not attributed to surface tension⁶.

It is reported that the rheological properties of the base fluids also play an important role in foaming ability and foam stability³⁹. It can be seen from Figure 3(b) and Table 3, the AV of base fluids added with NMAS gradually increases from 9.25 to 13.5 mPa·s, as the temperature increases up to 160 °C and then decreases with temperature rising. It behaves in quite opposite trend to V_0 of foam as a function of temperature. Similar phenomena are found for MMAS or Na-BT base fluids. It is reported that the increasing AV would hinder the migration of the surfactant molecule to the water/air interface and reduce the foaming efficiency, resulting in decreasing of average bubble diameter and V_0 ^{22, 40}. We also further investigate the PV, YP, and Gel₁₀ (Table 3). In this study, with the increasing of temperature, PV possesses similar trend with AV. However, YP varies irregularly and manifests a decreasing trend substantially, inferring the initial gel structure has been broken. It is also evident by the results that all the Gel₁₀ almost approach 0 after thermal aging, which is on

behalf of the gel strength in static condition. Thus, one of the main effects of NMAS, MMAS and Na-BT particles is to change the rheological properties of base fluid. Accordingly, we speculate the effect of temperature on rheological properties, foam stability and foaming ability to be the result of smectite particle hydration expansion, dehydration, aggregation, and dispersion, which are affected by hot rolling condition.

As we all know, foam stability essentially stems from the stability of the liquid film⁶. In recent years, the interfacial dilational modulus (E) is used to characterize the resistance ability and the self-repair ability of liquid film. The greater E is, the stronger resistance and self-repair ability film will possess, in other words, the stronger the film will be³⁵. As shown in Figure 3(c), E of NMAS base fluid drastically increases to 13.951 from 2.789 mN/m as the temperature increases up to 160 °C from 25 °C and then decreases with temperature rising. Even though the aging temperature is as high as 320 °C, it still remains 3.463 mN/m. By comparison, no significant impact of temperature can be found on E of MMAS and Na-BT base fluids. In detail, E of MMAS base fluids stepwise increases until 240 °C and then declines slowly, with a maximum of 2.865 mN/m, whereas E of Na-BT base fluids increases as temperature rises to 140 °C and then gradually falls till 200 °C. Then it climbs again to 2.634 mN/m at 280 °C before declining again. It is obvious that the E of NMAS base fluids is much higher than MMAS and Na-BT base fluids at any temperature. Besides, E of MMAS base fluids is lower than that of Na-BT base fluids when the temperature is below 160 °C. However, when the temperature is over 160 °C, E of MMAS base fluids becomes higher than that of Na-BT base fluids on the contrary. It

is obvious that the influence of temperature on E has the same trend with $T_{0.5}$. The results verify that the foam with the greater E has better stability, which has been reported in other researches^{35,41}. Although AV seems to have a certain positive effect on foam stability, we could come to the conclusion that E is the decisive factor for foam stability as temperature rising, considering the more matching trend between E and $T_{0.5}$.

In order to reveal the underlying mechanism of AV and E of base fluids varying with temperature rising, particle size distribution was studied. After adding AD300, d_{50} of NMAS, MMAS, and Na-BT dispersions at room temperature increase from 19.691 nm, 4.219 μm , and 4.655 μm to 348.2 nm, 10.8 μm , and 27.4 μm , respectively. Anionic AD300 molecules adsorb onto the particles by static electronic, hydrophobic and hydrogen bonding interaction. As a result, bridges would be built among the particles and the particle size therefore becomes larger. Thermal aging temperature would further impact the aggregation state, including the particles size distribution. As for NMAS base fluid, d_{50} abruptly increases from 0.348 to 24.1 μm after hot rolling at 80 °C for 16 h. With the temperature rising, d_{50} of NMAS base fluid primarily increases to its maximum of 97.2 μm gradually at 160 °C and then decreases slowly (Figure 3(d)). Such significant change of d_{50} leads to an increase of turbidity in NMAS base fluids after aging at high temperature, probably attributed to its enormous specific surface area, which endows particles high surface energy and supplies sufficient reaction area to aggregation⁴⁴. The particle size varied in the similar manner as E and especially AV. Similar phenomena are found in particle size distributions of

MMAS and Na-BT base fluids. Thus there is a positive relationship among d_{50} , AV. Aggregation and dispersion are the main factors for variation of AV. As the aging temperature increases, highly hydrated and well dispersed particles would gradually release hydrated water and crystal water, tending to aggregate by face to face connection in a relatively loose form, consequently displaying larger d_{50} and higher AV^{45, 46}. Once temperature exceed a critical point, particle dehydration would become exacerbate and result in compacted aggregates with consolidation of the particle surface and the decline of aggregation size, leading to decrease of AV(Figure 4)⁴⁷. The critical point is related to the composition and interlayer cations of particle, thus NMAS, MMAS, and Na-BT exhibit different inflection points⁴⁸. The interaction between NMAS aggregates is so strong that high speed stirring (11000 rpm) during foaming process has little destructive effect on it, quite different from a half reduction on aggregate size of MMAS and Na-BT particles, which may be one of the reasons for the best foam stabilization of NMAS particles. The aggregates distributed in film enlarge film thickness and enhance viscoelasticity and mechanical strength, which slow down the speed of liquid drainage and air penetration. Besides, the large aggregates also tend to distribute in Plateau border to delay disproportionation and slow down liquid drainage furtherly⁶. Thus the temperate aggregation of clay particles is beneficial for foam stability. In addition, during the high-temperature aging process, there may be some chemical and mineral composition change, which is beyond the scope of this article and would not be discussed in this study.

Table 3. Rheological properties of base fluids after being aged at 25, 80, 120, 140,

160, 200, 240, 280, 300, and 320 °C.

	Aging temperature (°C)	AV (mPa•s)	PV (mPa•s)	YP (Pa)	Gel _{10 s} (Pa)
0.6 wt% AD300+1.5 wt% NMA	25	9.25	7.5	1.75	1.5
	80	11	8	3	1.25
	120	12	11	1	0
	140	13	12	1	0
	160	13.5	12	1.5	0
	200	11	10.5	0.5	0
	240	9.75	10	0	0
	280	9	9	0	0
	300	8.25	9	0	0
	320	6.5	8	0	0
0.6 wt% AD300+3.0 wt% MMS	25	4	2	2	1.5
	80	5.5	5	0.5	0
	120	6	5.5	0.5	0
	140	7	7	0	0
	160	8.5	7	1.5	0
	200	12.5	10	2	0
	240	21	20	1	0
	280	17.5	17	0.5	0
	300	11	10	1	0
	320	7	7	0	0
0.6 wt% AD300+3.0 wt% Na-BT	25	2	3	0	0
	80	9	8	1	0
	120	10	9	1	0
	140	11.5	10	0.5	0
	160	10	9	1	0
	200	9.5	9	0.5	0
	240	10.5	10	0.5	0
	280	11	11	0	0
	300	7.5	7	0.5	0
	320	3.5	4	0	0

Figure 3. (a) Surface tension, (b) AV, (c) E, and (d) d_{50} of NMAS, MMAS, and Na-BT base fluids after being aged at 25, 80, 120, 140, 160, 200, 240, 280, 300, and 320 °C.

Figure 4. Illustrative picture of clay particles after being aged from 25 to 320 °C.

3.3.2 Relationship between hot rolling temperature and foam properties

In our study, NMAS, MMAS and Na-BT display different foam stabilizing performance and also affect the foaming ability at different extent. After being aged at different temperatures for 16 h, the clay particles aggregate together and the particle size distribution changes with the increasing of aging temperature. When the particle aggregation size increases, AV and E increase. In the foaming process, the increment of AV might impede the migration of surfactant molecule from base fluid to the water-air interface, which thus decreases the foaming ability, bringing reduced initial foam volume and bubble diameter, as well as increased foam film thickness^{22, 40}. At the same time, the increment of AV will slow down liquid drainage and air penetration and the increment of E will enhance the resistance ability and the self-repair ability, which are beneficial for foam stability^{35, 43}. On the contrary, when the aggregate size decreases as aging temperature increase, the AV and E decrease as a result. Nevertheless, irrespective of NMAS or MMAS or Na-BT, d_{50} , AV, E, foam film thickness, and $T_{0.5}$ manifest similar variation trend. Simultaneously, bubble average diameter and V_0 also exhibit similar variation trend, which is approximately reverse to the trends of above aforementioned parameters. In addition, all the curves for these parameters always display the same inflection points as aging temperature increases. Our study has figured out the effect of base fluid properties on foam and these finds will give guides to the future researches of clay particles used as foam stabilizer, especially in high-temperature condition.

3.4 Performance of NMAS, MMAS, and Na-BT as foam stabilizers for 320 °C and high salt condition

From above experiments, NMAS displays a good foam stabilization performance at high temperature. In this section, we further investigate the performance of NMAS, MMAS, and Na-BT as foam stabilizers for extra high temperature (320 °C) condition. Foam stability is evaluated by observing the microscopic bubbles and macroscopic foam volume. Additionally, during circulation in the annulus, foams usually would probably get contaminated by inorganic salts, such as NaCl and CaCl₂. These inorganic salts will inhibit the activity of the foaming agent and flocculate clay mineral, which could cause a giant effect on foaming ability and foam stabilization. Thus, salt tolerance was investigated.

3.4.1 The stability of the foam evaluated by optical microscope and digital photographs

Owing to the difference of Laplace pressure, smaller bubbles will keep shrinking until they disappear and bigger bubbles will keep expanding before bursting. This phenomenon has vividly exhibited in Figure 5. With time elapsing, disproportionation and coalescence of blank foams proceed quickly. The number of small bubbles obviously decline and big bubbles appear rapidly after 5 min. Compared with blank foams, disproportionation and coalescence of foams stabilized by Na-BT slow down a lot in the first 10 min, indicating Na-BT do have a certain foam stabilizing effect. However, the foam stabilizing effect cannot sustain for a long time. After 30 min, the

bubbles become similar with the blank ones. As for MMAS-stabilized foam, speeds of disproportionation and coalescence have been slowed down as well, confirming a competitive foam stabilizing effect. When it comes to NMAS-stabilized foam, disproportionation and coalescence have been dramatically inhibited, even after a long time. There is almost no change between 30 min and 40 min since the bubbles are prepared, displaying an excellent foam stabilization performance. In a word, it demonstrates the stability of the foam follows the sequence of $\text{NMAS} > \text{MMAS} > \text{Na-BT} > \text{Blank}$. The results agree well with the macroscopic observation. Just as Figure 6 has presented, the liquid drains rapidly from blank foams and more than 50 mL have been drained in 10 min. The foams gradually become transparent, especially after 1 h. And only about 50 mL foams still exist after 5 h. Therefore, blank foam has poor stability. As for foams stabilized by Na-BT, about 45 mL of liquid has drained in 10 min and there are still 325 mL foams exist after 5 h and about 290 mL foams exist after 10 h. Definitely, Na-BT improves the stability of foams, although the effect is moderate. The liquid drained slowly from MMAS-stabilized foam and only about 8 mL have drained in 10 min. The volume of foams decreased from 395 to 260 mL after 10 h and still remained 90 mL after 15 h. When it comes to NMAS-stabilized foam, there is no liquid drained from until 1 h. Moreover, it is until 10 h that about 50 mL liquid drained from NMAS-stabilized, indicating an outstanding foam stabilizing effect. Even after 15 h, 300 mL foams still retained. Thus, from both of microscopic and macroscopic perspective, the stability of the foam follows the sequence of $\text{NMAS} > \text{MMAS} > \text{Na-BT} > \text{Blank}$.

Figure 5. Optical microscope images of blank one, Na-BT-stabilized foams, MMAS-stabilized foams, and NMAStabilized foams over time. All samples have been aged at 320 °C for 16 h.

Figure 6. Digital photographs of blank one, Na-BT-stabilized foams, MMAS-stabilized foams, and NMAS-stabilized foams over time. All samples have been aged at 320 °C for 16 h.

3.4.2 Salt tolerance evaluation

We evaluated the NaCl and CaCl₂ tolerance of NMAS-, MMAS- and Na-BT-stabilized foams (Figure 7). As the concentration of NaCl increases, all the V_0 of the NMAS-, MMAS-, and Na-BT-stabilized foams decrease continuously. At the same time, $T_{0.5}$ increase gradually, which is attributed to the flocculation of clay particles and verify that aggregation is advantageous for foam stability mentioned

above. Remarkably, NMAS-stabilized foam always manifests the largest V_0 and longest $T_{0.5}$, regardless of the salt concentration. In addition, V_0 of NMAS- and MMAS-stabilized foam decrease slowly when NaCl concentration is below 3 wt%. In contrast, V_0 of Na-BT-stabilized foam decreases from 425 to 350 mL when only 1 wt% NaCl is added. Thus, NaCl tolerance follows the sequence of NMAS > MMAS > Na-BT.

The similar phenomenon appears in CaCl_2 tolerance investigation. The adding of CaCl_2 sustainably decrease all the V_0 of the NMAS-, MMAS-, and Na-BT-stabilized foams, increasing $T_{0.5}$ contrarily. V_0 of Na-BT foam decreases to 315 mL when only 0.1 wt% CaCl_2 is added, showing poorest CaCl_2 tolerance. While V_0 of NMAS- and MMAS-stabilized foam decrease slowly when CaCl_2 is less than 0.2 wt% and NMAS-stabilized foam always possesses the best foaming ability and foam stabilization performance. Thus, NMAS-stabilized foam displayed prominent NaCl and CaCl_2 tolerance.

Figure 7. (a) V_0 at different concentrations of NaCl, (b) $T_{0.5}$ at different concentrations of NaCl, (c) V_0 at different concentrations of CaCl₂ and (d) $T_{0.5}$ at different concentrations of CaCl₂. All samples have been aged at 320 °C for 16 h.

4 Conclusion

Magnesium aluminum silicate (NMAS and MMAS) were adopted as anti-high temperature foam stabilizers. NMAS performs the best foam stabilization effect at room temperature and elevated temperature even as high as 320 °C. It is found that increasing temperature could change aggregation state of particle. Increasing aggregate size results in gigantic positive effect on AV, E, foam film thickness, and $T_{0.5}$ and vice versa. Simultaneously, it leads to a negative effect on foam diameter and the V_0 . Moreover, NMAS-stabilized foam manifests excellent foam stability and salt tolerance compared with MMAS- and Na-BT-stabilized foam after being aged at 320 °C. Thus, NMAS are expected to use as foam stabilizer in extra-high-temperature

and high salt concentration conditions, such as geothermal well drilling, as well as enhanced oil recovery, fracturing, and mineral flotation.

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Enhanced foam-stabilizing performance by adding magnesium aluminum silicate compared with sodium bentonite independent of temperatures

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Increasing temperature could facilitate aggregation of clay dispersion, and the increase of particle size results in significant positive effect on apparent viscosity (AV), interface dilational modulus (E), foam film thickness, and eventually drainage half-life ($T_{0.5}$) of the foam. Simultaneously, it leads to a negative impact on the foam diameter and initial foam volume (V_0).

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