1		Study on high-temperature resistance, salt/calcium resistance of						
2	environment-friendly colloidal gas aphron drilling fluid							
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12 Abstract: The Colloidal Gas Aphron (CGA) drilling fluid successfully solved the problems of lost 13 circulation and reservoir damage that are faced by drilling in depleted oil/gas reservoirs and 14 low-pressure areas. However, the lack of high-temperature resistance, salt/calcium resistance are 15 the key problems that restrict its application in complex formations. This study provides an 16 environmentally friendly and non-toxic CGA formula based on self-developed reagents. 17 Microscopic tests showed that stable aphrons were successfully generated in 36%NaCl-CGA or 18 7.5% CaCl<sub>2</sub>-CGA aged at 150°C, with stabilization of  $\geq$ 2 hours. The Herschel-Bulkely model 19 accurately describes the rheological behavior of CGA fluids containing NaCl/CaCl<sub>2</sub>. The addition 20 of NaCl increases CGA fluid viscosity, while CaCl<sub>2</sub> is the opposite. However, CGA fluid 21 maintains appropriate rheological parameters and shear thinning behavior, which means good 22 cutting carrying capacity. With the addition of NaCl/CaCl<sub>2</sub>, CGA has low filtration volumes, 23 which meets API requirements. NaCl/CaCl<sub>2</sub> reduces the lubrication coefficient and increases the 24 adhesion of the mud cake. Moreover, the anti-cuttings pollution ability of 150°C aged CGA can 25 reach 10%. CGA, 36%NaCl-CGA, and 7.5%CaCl<sub>2</sub>-CGA all have low linear expansion rates 26 (<28%) and high rolling recovery rates (>84%). Therefore, the CGA system has good inhibitory 27 performance and is compatible with easily hydrated formations.

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Key words: CGA drilling fluid; environmentally friendly; underbalanced drilling; 29 high-temperature resistance; Ca<sup>2+</sup>/Na<sup>+</sup> contamination tolerance. 30

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### 32 1. Introduction

Recently, a near-balanced drilling technology, the colloidal gas aphron (CGA) drilling fluid 33 34 has been successfully applied in drilling depleted oil/gas reservoirs and other under-pressure 35 areas[1]. It is a near/under-balanced technology which is composed of independent ridgid 36 micro-foams with bubble size  $\sim 100 \mu m$ , and has the following significant advantages: a) As shown 37 in Figure 1, the aphrons are composed of one core and three films. The unique structure makes it 38 have high stability and strong pressure-bearing capacity, which is nearly 10 times that of ordinary

39 foam[2]. Through cyclic pressure/relief experiments and pressure sealing experiments, Xie et al. 40 confirmed that under a pressure of 20MPa, CGA has compressibility and recovery properties, and 41 can effectively seal sandstone formations of 40-60 mesh[3]. Pasdar et al. monitored the behavior 42 of CGA under high pressure using a high-pressure microscope and studied the single bubble 43 behavior and bubble size distribution of CGA. Observations indicate that CGA survived at 44 pressures as high as 13.8MPa may support the idea for field applications of CGA as drilling 45 fluids[4]. Zheng et al. combined microscopy and HTHP filtration experiments to further 46 demonstrate the sealing performance of CGA fluids under conditions of 120-200°C and 3.5MPa[5]; 47 b) Approve the used as elastic plugging materials to alleviate or avoid lost circulation. Also, 48 aphrons have little affinity with each other or the formation rock surface, and can be easily 49 removed by formation fluid backflow during the production stage[6,7]; c) The construction 50 process is simple and don't need the air compressor. Aphrons are generated by mechanical 51 agitation of surfactants and biopolymers at a speed of higher than 5000rpm or shearing of 52 pipelines[8].

53 In theoretical research, scholars mainly focus on the aphrons stability and bubble size 54 distribution, the rheology and plugging properties in porous media, and have obtained the 55 following conclusive results:

- 56 In terms of the optimization of foaming agents and foam stabilizers, scholars have a) 57 successively evaluated a variety of foam stabilizers (XG, starch, CMC, etc.) and multiple types of foaming agents (SDS, CTAB, X-100, CAPB[9], plant root extraction, 58 59 etc.). It is proposed that the CGA system prepared by 0.3~0.8% xanthan gum (XG) and 60  $\sim 0.5\%$  sodium dodecyl sulfate (SDS) is the preferred system, with high stability and 61 good rheology[10-15]. The polymer concentration greatly influenced the stability and 62 bubble size of CGA fluids. The most stable CGAs were formed at higher concentrations 63 of polymer[16].
- b) The average size of the aphron is affected by many factors such as the type and concentration of surfactant/polymer, temperature, pressure, agitation rate, and more. As the concentration of the foaming agent increases, the aphrons size increases; with the increase of the foam stabilizer, the size of the aphrons decreases, and the stability is significantly enhanced[17]. Alizadeh et al. established the mathematical relationship between the bubble size and temperature-pressure, and pointed out that temperature is the main factor affecting the size of aphrons[18].
- 71 In 2005, Popov and Growcock took the lead in proving that aphrons would form a c) 72 plugging zone at the front of the fluid through radial flow experiments[19]; In 2012, 73 Nareh et al. pointed out that more attention should be paid to the size distribution of 74 aphrons rather than its average size under porous media[20]; In 2019, the flow and 75 plugging properties of CGA fluid in heterogeneous porous media were visually observed by Mohsen et al. by using the etched glass plate model and microscope[21]. By 76 77 analyzing the injection pressure and backflow permeability data, it was confirmed that 78 CGA fluid can significantly control the flow of fluid to fractures.
- d) In terms of rheology, Arabloo et al. used eight kinds of rheological models to describe
  the rheological behavior of a typical CGA drilling fluid m prepared by XG and SDS at
  25-45°C, and selected the Herschel-Bulkley, Mizhari-Berk, Sisko, Power-Law and the
  Robertso-Stiff model[17]. Ehsan et al. further proved that the Herschel-Bulkley model

has high applicability to the CGA system, the goodness of fit is higher than 0.999[13]. In the latest research, nanocomposite CGA fluids have attracted much attention. Herschel-Bulkley, Mizhari-Berk, Power Law, and Robertson-Stiff models' predictions have strong consistency with the experimental data of nano-enhanced colloidal gas aphron (NCGA)-based fluids[22]. The CGA fluids have also been used as adsorbents that are easily removed from contaminated wastewater[23,24].

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89 Table 1 lists the field application research progress of CGA drilling fluid in recent years. The 90 CGA drilling fluid has successfully solved the problems of serious lost circulation, reservoir 91 damage, and difficult drilling that often occurred in drilling depleted oil/gas reservoirs and 92 low-pressure areas. The early development of the CGA drilling fluid was mainly to optimize and 93 compound commercial foaming agents and stabilizers to meet the needs of the site. In the past five 94 years, the research direction has gradually changed to the independent synthesis of new treatment 95 agents. The stability, high-temperature resistance, and anti-pollution ability of the system are 96 improved. However, at present, the temperature resistance of CGA drilling fluid is still within 97 150°C, the salt resistance is  $\leq 20\%$  and the calcium resistance is generally less than 1%. Further 98 breakthroughs must be made in the aspects of high-temperature salt and calcium resistance and its 99 environmental properties[25,26].

100 In this paper, a novel CGA drilling fluid system was constructed by the self-developed 101 environment-friendly foam stabilizer EST, foaming agent AGS-8, and lubricant ChCl-PEG. A 102 comprehensive evaluation of the stability, rheology, lubricity, filtration, anti-cuttings pollution, 103 inhibition, environmental protection was carried out in high temperature high salt (150°C, 104  $\leq$ 36%NaCl) or high temperature high calcium (150°C,  $\leq$ 7.5%CaCl<sub>2</sub>) conditions.

## 105 2. Technical requirements for CGA drilling fluids

Combined with literature research, this study puts forward the technical requirements forCGA drilling fluid.

(1) Stability: After high-temperature aging, the CGA drilling fluid needs to maintain good
stability, which is the premise for safe drilling. In the laboratory test, the change of aphrons
morphology can be observed by microscope and combined with the change of drilling fluid
density, the drainage, coalescence, and defoaming of aphrons in the drilling fluid, the stability can
be analyzed. Generally, one cycle of drilling fluid is usually no more than 2h.

(2) Good rheology: After high-temperature aging, the drilling fluid needs to maintain good rheology to solve the problems of suspension and carrying cuttings in the drilling process, as well as keep the wellbore clean, and ensure downhole safety. Generally, it is suitable to keep the value of flow behavior index (n) at 0.4~0.7 and the value of YP/PV higher than 0.36. In addition, the LSRV is a concerned rheological parameter index in CGA drilling fluids. The LSRV value of the CGA drilling fluid system after high-temperature aging should be higher than 10000mPa·s[10,27].

(3) Low fluid loss: Studies have shown that near/underbalanced drilling still has the
possibility of filtration[28]. Therefore, the high-temperature filtration property of the CGA drilling
fluid cannot be ignored, and the fluid loss volume (FL<sub>API</sub>) should be controlled within 15mL.

122 (4) The pull-up and rotation resistance of the drill string increases greatly with the increase of 123 the well depth, and the drilling tools wear seriously during the drilling process. Drilling fluids and 124 mud cakes need to maintain high lubricity (extreme pressure lubrication coefficient  $\leq 0.20$ ) to 125 ensure low friction and torque, reduce sticking, and protect drilling tools[29,30].

126 (5) Inhibitory: For areas with high mudstone content, prone to collapse and clay hydration

problems, the drilling fluid needs to have low fluid loss and strong inhibition to ensure wellborestability[31].

(6) Cuttings resistance: Solid powder with large specific surface areas, such as cuttings, bad
soil, sand, etc., will be produced during drilling. Some Solid dust will defoam or inhibit foaming.
Therefore, CGA drilling fluid is also required to have a certain ability to resist cuttings pollution
during on-site construction[32,33].

(7) Environmentally friendly: the CGA drilling fluid should not only achieve the due
auxiliary drilling effect but also consider environmental protection issues, including non-toxic and
easy degradation of raw material, and meeting the discharge standard and easy degradation after
waste. Hence, the biodegradability and toxicity of the CGA drilling fluid system were
evaluated[34].

## 138 **3. Materials and Method**

## 139 **3.1 Materials**

In this paper, foam stabilizer (EST) is a graft-modified starch prepared by inverse emulsion polymerization. The synthesis method and high-temperature foam stabilization properties of EST have been reported in the previous literature[35]. EST plays the role of stabilizing foam and reducing filtration loss at a dosage of  $\geq$ 1%. With the increase of EST concentration, the application effect will be enhanced. However, the dosage of EST should be controlled within 5% to avoid the adverse effect of high-concentration EST on slurry preparation.

146 The foaming agent is an alkyl glycine-type zwitterionic surfactant (AGS-8), which is 147 prepared with sodium chloroacetate and n-octylamine. The surface activity, of AGS-8 can be 148 referred in Table 2 and the published article for details[36]. Research has proved that AGS-8 has a 149 good synergistic stabilization effect with EST, the high-temperature resistance is  $\geq 180^{\circ}$ C, and the 150 salt resistance reaches saturation (36%NaCl).

A deep eutectic solvent (DES) ChCl-PEG synthesized by choline chloride and polyethylene 151 152 glycol was added to the system as a lubricant. DES is a new environmentally friendly, non-toxic 153 and biodegradable solvent discovered, which usually consists of two or three kinds of hydrogen 154 bond donors (HBD) and hydrogen bond acceptors (HBA) [37,38]. The HBA of ChCl-PEG is 155 choline chloride (ChCl) for it contains a lot of positive charges, which is expected to form a 156 physical adsorption film on the surface of the friction pair through electrostatic action. The HBD ChCl-PEG is polyethylene glycol (PEG). PEG can effectively reduce the wear scar and roughness 157 158 between friction pairs and is a good water-based lubricant.

As Table 3 shows, the biodegradability indexes (Y) of EST, AGS-8, and ChCl-PEG are allgreater than 25, and the materials are all easily biodegradable.

### 161 **3.2 Methods**

### 162 **3.2.1 Preparation and observation of CGA drilling fluid**

163 CGA drilling fluid was prepared with 3% bentonite base mud, EST, AGS-8, and ChCl-PEG. 164 First, the base mud was prepared by mixing freshwater, 0.25%Na<sub>2</sub>CO<sub>3</sub>, and 3% bentonite. After stirring for 1h, the mud was stood for 16h. Second, add EST and ChCl-PEG to the base slurry 165 (300 mL) in turn, mix and stir at 8000 rpm for 20 min by using a high-speed mixer (Model 166 167 WT-2000C, China). The polymer is fully dispersed in the base slurry. Add AGS-8 continuously 168 and stir at 10000rpm for 3min to obtain the CGA drilling fluid at normal temperature. Then, CGA drilling fluid was put into a roller furnace (Model XGRL, China) and aged at 150°C for 16h. After 169 170 that, high-temperature CGA drilling fluid was obtained by stirring at 10000rpm for 3min again.

171 The salt-resistance and calcium-resistance of CGA drilling fluid were evaluated by adding a 172 certain concentration of NaCl or CaCl<sub>2</sub>.

### 173 **3.2.2 Evaluation of drilling fluid properties**

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174 Microscopic observation of aphrons: The CGA drilling fluid sample was placed on the 175 glass slide, and the microbubbles in the system were visually observed by a polarizing microscope 176 with CCD high-speed camera (Model Olympus BX51, Japan). The microbubbles size in the 177 images was measured by Nano-measurer software, and then statistically analyzed in the Origin 178 software[39].

Rheology: The rheological properties of CGA drilling fluids were studied using two 179 180 viscometers. A Brookfield viscometer (Model Brookfield DV-II) was used to test the LSRV of 181 CGA drilling fluids at 0.3 rpm. The six-speed rotational viscometer is used to test the shear stress 182 data at different shear rates of the CGA system. Four commonly used rheological models are used 183 to describe the rheological behavior of CGA fluid under high-temperature, high-salt, or 184 high-calcium, and the optimal model with highest accuracy is selected[40].

- Binham Model:  $\tau = \tau_0 + \mu_P \gamma$ (1)
- *Power-law Model:*  $\tau = K\gamma^n$ 186 (2)
- *Casson Model:*  $\tau^{1/2} = \tau_c^{1/2} + \eta_{\infty}^{1/2} \gamma^{1/2}$ 187 (3)
  - *Herschel-Bulkely Model:*  $\tau = \tau_v + K \gamma^n$ (4)

Where,  $\tau$  is the shear stress,  $\theta$  is the reading of six-speed viscometer,  $\gamma$  is the shear rate,  $\tau_0$  is 189 190 the yield point,  $\mu_P$  is the plastic viscosity, K is the consistency index, n is the flow behavior index,  $\tau_c$  is the yield point of the Casson model,  $\eta_{\infty}$  is the ultimate high shear viscosity,  $\tau_v$  is the yield 191 192 point of the H-B model.

193 The rheological parameters were calculated according to formulas (5~7). Stir the drilling 194 fluid at 600rpm for 10s, and after standing for 10min, multiply the maximum reading of the dial at 3rpm by 0.511, which is the gel strength ( $GS_{10}$ min). The ratio of YP and PV is defined as the 195 196 YP/PV.

Apparent viscosity (AV)=
$$\theta_{600}$$
\*0.5 (5)

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$$Plastic \ viscosity \ (PV) = \theta_{600} - \theta_{300} \tag{6}$$

Yield point (YP)=0.511\*(
$$\theta_{300}$$
 -PV) (7)

200 Filtration: According to the American Petroleum Institute (API) standard, the API filtration volume of CGA drilling fluid is tested in the atmosphere of 0.69mpa  $N_2$  by using a medium 201 202 pressure fluid loss instrument (Model SD -6, China), and the filtration volume ( $FL_{API}$ ) of the sample within 30min is recorded[41]. 203

204 Lubricity: The lubrication performance of drilling fluid includes the lubricity of mud cake 205 and the lubricity of fluid. The mud cake adhesion coefficient (f) and extreme pressure lubrication 206 coefficient (K) are the two main technical indexes to evaluate the lubricity of drilling fluid. They 207 are evaluated by mud cake adhesion coefficient instrument (Model EP, China) and extreme 208 pressure lubrication instrument (Model NF-2, China) respectively. The indexes are calculated 209 according to Formula 8-11[42].

210	Adhesion coefficient (f)=Maximum torque value (N)×0.845/100	(8)
211	Correction factor $(F)=34/Friction$ coefficient with water as calibration	(9)
212	Friction coefficient (M)= The reading of friction coefficient/100	(10)
213	<i>Lubrication coefficient</i> $(K) = F \times M$	(11)
214	Inhibitory: A linear dilatometer and rolling recovery experiment were used to analy	ze the

**Inhibitory:** A linear dilatometer and rolling recovery experiment were used to analyze the

215 inhibition of drilling fluid. Immerse the sample into the standard bentonite block that is pressed by 216 the hydraulic instrument, connect the linear dilatometer and computer software, and test the 217 expansion amount and expansion rate of the bentonite block within 16h. Rolling recovery experiments were carried out by using a roller furnace (Model XGRL, China). Take about 20g of 218 219 sandstone rock samples (6~10 mesh) and 350mL of drilling fluid sample into an aging jar, heat it 220 at 150°C for 16h. After taking out, rinse and dry, the sample was sieved with a 40-mesh sieve. 221 Collect and weigh the cuttings that do not pass through the 40-mesh sieve, and the ratio of mass to 222 the initial rock sample mass is the rolling recovery rate[43].

## 223 **3.2.3 Environmental testing**

BOD<sub>5</sub>/COD method is an important evaluation method for the discharge and treatment of industrial wastewater containing organic matter. Dissolve the sample in deionized water, and test the five-day biochemical oxygen demand (BOD<sub>5</sub>) and chemical oxygen demand (COD) of the sample to evaluate the biodegradability of the material. The biodegradability evaluation index (Y) was defined as the percentage rate of BOD<sub>5</sub> and COD.

According to the Standard "SY/T 6788-2010", the biotoxicity test of the CGA system is carried out by the luminescent bacillus method[44]. When the luminescent ability of luminescent bacteria decreases by half, the concentration of oilfield chemicals is recorded as  $EC_{50}$ , which is used to characterize the acute toxicity level of water. The greater the  $EC_{50}$  value, the lower the biological toxicity. The corresponding relationship between  $EC_{50}$  and the toxicity of drilling fluid is listed in Table 4.

## 235 **4. Results and Discussion**

# 4.1 Orthogonal experiment: determining the optimal concentration of the treatment agents

A three-factor four-level orthogonal experiment was designed to determine the optimal concentration of each reagent in the formula of CGA. The high-temperature stability parameters of the system were used as the evaluation index, recording the time ( $T_0$ ) when CGA begins to discharge liquid after aging at 150°C for 16h. The results are shown in Table 5.

242 Based on the results of 16 sets of experiments, the average values  $P_1$ ,  $P_2$ ,  $P_3$ ,  $P_4$  for each factor 243 and level were calculated, as well as the difference between the maximum average and the 244 minimum average (range R). Results indicate that  $R_A > R_B > R_C$ , that is, the effects of the three 245 reagents on the stability of the CGA system are EST, AGS-8, and ChCl-PEG in descending order. 246 For factor A (EST dosage),  $P_3 > P_4 > P_2 > P_1$ , that is, when the dosage of EST is 3%, the system 247 stability is optimal; For factor B (AGS-8 dosage),  $P_1 > P_4 > P_3 > P_2$ , that is, when the dosage of 248 AGS-8 is 3%, the system stability is optimal; For factor C (ChCl PEG dosage),  $P_2 > P_4 > P_3 > P_1$ , that 249 is, when the dosage of ChCl-PEG is 3%, the system stability is optimal.

Therefore, based on the high-temperature stability of the CGA drilling fluid system as the evaluation standard, the optimal formula for the CGA system was determined through orthogonal experiments as follows:

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## 3% Bentonite mud+3% EST+3% AGS-8+3% ChCl PEG4.2 Bubble size distribution and stability of aphrons

The system of CGA, CGA with 36%NaCl, and CGA with 7.5%CaCl<sub>2</sub> are taken out after 150°Caged. After stirring at 10000rpm for 3min, the densities of the drilling fluid decreased from 1.03 g/cm<sup>3</sup> to 0.78, 0.75, and 0.66 g/cm<sup>3</sup>, respectively. Figure 2(a) and Figure 3(a) show aphrons and the bubble size distribution in the 150°C aged CGA system. Through the statistical analysis, it is found that the bubble size of aphrons in the figure is between  $27.08 \sim 265.45 \mu m$ , about 87% of the aphrons have a bubble size of  $10 \sim 150 \mu m$ , and the average bubble size is  $99.15 \mu m$ .

Figure 2(b) and (c) show that in the CGA system containing 36%NaCl or 7.5%CaCl<sub>2</sub>, the aphrons always maintain a stable spherical shape with a thick liquid film, and keep independent of each other, which is highly consistent with the description of aphron structure by Sebba et al[45]. In other words, aphrons with stable structures have been proved to be generated at high-temperature high salt, and high-temperature high calcium.

266 Figure 3(b) shows that the bubble size of the aphron s in the CGA system with 36%NaCl 267 ranges from 33.86 to 266.52µm, the proportion of aphron s in the range of 10~150µm exceeds 93%, and the average diameter is  $97.35\mu$ m. Figure 3(c) shows that the bubble size of aphrons in 268 269 CGA with 7.5% CaCl<sub>2</sub> ranges from 21.81 to 285.39 µm, the proportion in the range of 10~150 µm 270 exceeds 96%, and the average diameter is 74.65µm. Under the same conditions, NaCl had little 271 effect on the density and aphron size of the CGA system. The addition of CaCl<sub>2</sub> increased the 272 proportion of small-diameter microbubbles in the CGA system and decreased the average 273 diameter.

In general, the bubble size distribution of aphrons in this study is highly consistent with the that of the previous reference, which intuitively proves that CGA drilling fluid is successfully generated under the conditions of high temperature, high salt, or high calcium by using EST, AGS-8, and CHCI-PEG[8].

278 Figures 4 (a), (b), and (c) show the time-dependent image of aphrons in the CGA system, 279 CGA containing 36%NaCl, and CGA containing 7.5%CaCl<sub>2</sub>, respectively. The distribution of 280 aphrons is shown in Figure 5. There was no coalescence between foams, and no Plateau boundary 281 in the whole observation period, which indicates that the pressure difference drainage and 282 coalescence combination do not occur violently. As time goes by, gas diffusion takes place 283 between aphrons of different diameters. The small diameter bubble becomes smaller and smaller 284 until it disappears, and the large diameter bubble increases. According to Table 6, the average 285 diameters of CGA and CGA systems containing 7.5% CaCl<sub>2</sub> after standing for 2 hours increase by 286 20.85 and 18.85 $\mu$ m, respectively. The proportion of aphrons with a diameter <100 $\mu$ m decreased 287 by 11.27% and 10.97%, respectively, which is within an acceptable range. The average diameter 288 and bubble size distribution of CGA systems containing 36% NaCl did not show significant 289 changes. In short, the CGA system and CGA containing NaCl or CaCl<sub>2</sub> have high stability during 290 an observation period of at least 2 hours.

## 291 **4.3 Rheology**

The rheological properties of the CGA system and the CGA system containing NaCl/CaCl<sub>2</sub> before and after aging at 150°C were tested. The fitting results of the four rheological models were analyzed.

The goodness of fit value  $(R^2)$  and root mean square error (RMSE) are parameters used to 295 evaluate fitting accuracy. The closer the  $R^2$  value is to 1, the smaller the RMSE value, and the 296 better the fitting of the model. As shown in Table 7, the Bingham model has the lowest fitting 297 accuracy. For the CGA system under different test conditions, the value of  $R^2$  is between 0.909 298 and 0.973, and the value of RMSE is high, which is between 3.07 and 7.93. The Power-law model 299 and the Carson model have better fitting accuracy. The R<sup>2</sup> value of the Power-law model 300 (0.991-1.000) is closer to 1 than that of the Carson model (0.970-0.997). But the RMSE value of 301 302 the Power-law model  $(0.29 \sim 1.83)$  is higher than the Carson model.

Among the four models, the  $R^2$  value of the Herschel-Bulkely model is greater than 0.998 and has a lower RMSE value (0.14~1.04). Therefore, the Herschel-Bulkely model is the optimal model to describe the rheological behavior of the CGA system.

Table 8 lists the rheological parameters of the CGA system. The results show that the apparent viscosity (AV) and plastic viscosity (PV) of the CGA system increase with the increase of NaCl before and after aging at 150°C. The YP/PV of the CGA system with different concentrations of NaCl fluctuated between 0.456 and 0.772, and was always higher than 0.36. The flow behavior index (n) was between 0.451 and 0.691. At this time, the drilling fluid has a high cuttings-carrying capacity and strong shear-thinning properties.

The addition of CaCl<sub>2</sub> reduced the viscosity of the system to a certain extent after hightemperature aging. The values of AV and PV of the CGA system containing CaCl<sub>2</sub> before and after aging at 150°C remained above 47.5mPa·s and 30mPa·s, respectively. The value of YP/PV  $(0.444\sim0.699)$  and n (0.500 $\sim$ 0.689) both fluctuated within appropriate ranges.

In addition, the CGA system always has higher gel strength ( $GS_{10min}$ >5.11 Pa) and low shear viscosity (LSRV>29393mPa·s) under different conditions. To sum up, the CGA system has appropriate rheological parameters and high LSRV values under high temperature, high salt and high calcium environment, which shows high cuttings-carrying ability and strong shear-thinning behavior.

## 321 **4.4 Filtration and lubricity**

322 Table 9 lists the filtration and lubricity parameters of CGA and CGA systems containing 323 different concentrations of NaCl and CaCl<sub>2</sub> before and after aging at 150°C. The CGA system maintains a stable low filtration volume under the condition of a high concentration of NaCl/CaCl<sub>2</sub>. 324 325 With the increase of NaCl/CaCl<sub>2</sub>, the FL<sub>API</sub> of CGA at room temperature fluctuates in the range of 326 4.8~6.5mL. After 150°C aging, the filtration property of the CGA system is slightly improved, and 327 the FL<sub>API</sub> fluctuates within 2.5~4.5mL, which always meets the API requirements[46]. In addition, 328 a low value of fluid loss minimizes hydration expansion and maintains formation stability when 329 drilling into mudstone formations.

With the increase of NaCl/CaCl<sub>2</sub>, the lubrication coefficient of the CGA system decreased, and the adhesion coefficient of the mud cake increased slightly. In general, with different concentrations of ion contamination, the CGA system maintained a low adhesion coefficient (0.057~0.123) and lubrication coefficient (0.098~0.132) before and after high-temperature aging. In summary, CGA has good filtration properties and lubricity under conditions of high temperature, high salt, and high calcium.

### 336 **4.5 Cuttings resistance**

Add 5%, 10%, and 15% cuttings to the CGA system in turn, and the property changes after 337 338 aging at 150°C for 16h are shown in Table 10. After adding 5% or 10% cuttings, the viscosity and 339 GS10min changed within an acceptable range, the fluid loss increased slightly, and the lubricity 340 decreased. In general, all parameters met the design requirements. The 15% cuttings significantly reduced the performance of the drilling fluid. The dispersion of rock powder led to a significant 341 increase in the apparent viscosity (AV) and plastic viscosity (PV). The AV and PV values of the 342 343 CGA system reached 112.5 and 75 mPa·s, respectively, which may lead to difficulty in pump 344 starting and solid-phase removing. In addition, the lubrication coefficient of the CGA system is 345 also far beyond the design requirements. In conclusion, the anti-cuttings pollution ability of CGA 346 after aging at  $150^{\circ}$ C is not less than 10%.

### **4.6 Inhibitory**

348 As shown in Figure 6 (a), CGA drilling fluid always maintains a low linear expansion rate 349 (4.29~6.24%) at high-temperature or high-concentration ion pollution, which is equivalent to a variety of strongly inhibitory water-based drilling fluid systems (Table 11). The filtration fluid of 350 351 the CGA system is also obtained with the medium-pressure filtration instrument. The linear 352 expansion rates of freshwater and filtration fluid are tested and compared, which is more in line with the actual drilling conditions. As shown in Figure 6 (b), the bentonite block has a significant 353 354 hydration expansion in the freshwater, with an expansion rate of 51.8%. While the expansion rate of the filtration fluid of CGA, CGA+36% NaCl, and CGA+7.5% CaCl<sub>2</sub> system, which is between 355 356 20.11~27.05%. Therefore, the CGA system has good inhibition properties and compatibility with 357 easily hydrated formation.

According to the rolling recovery experiment (Figure 7), the sandstone particles in freshwater are broken and become fine after hot rolling at 150°C for 16h. Most of the particles with a particle size of more than 40 mesh are screened out, and the rolling recovery rate is only 43.41%. In the CGA system, the cuttings recovered after aging still maintain a good coarse particle shape and have a high recovery rate (87.98%). The rolling recovery rate of the CGA system with 363 36%NaCl or 7.5%CaCl<sub>2</sub> decreases slightly but remains at a high value (84.94% and 84.26%).

Combined with Table 11 and the test results, it can be found that the inhibition of the CGA system is equivalent to a variety of strongly inhibitory water-based drilling fluids. The CGA system has excellent inhibition at high temperature, high salt, and high calcium conditions, and is suitable for collapse-prone formations like shale or loose sandstone.

## 368 **4.7 Environmental testing**

The biodegradability of the CGA system was tested by the BOD<sub>5</sub>/COD method. Results show that the BOD<sub>5</sub> and COD value of CGA drilling fluid is 66.3mg/L and 209mg/L, respectively. The biodegradability evaluation index (Y) was 31.72. Therefore, the CGA system is biodegradable.

Figure 8 shows the "Concentration-Relative luminous intensity" curve of the filtration fluid of the CGA system. The  $EC_{50}$  value is 72116mg/L. Based on Table 3, it can be determined that the CGA system is an environmental-friendly and non-toxic drilling fluid system.

## 375 **5. Conclusion**

This study constructs a CGA drilling fluid system with high-temperature resistance (150°C), salt-resistance, and calcium-resistance (36% NaCl or 7.5% CaCl<sub>2</sub>). The stability, rheology, filtration, lubricity, anti-cuttings pollution ability, inhibition, and biological toxicity of the CGA system were evaluated. The following conclusions can be drawn:

1) Using the high-temperature stability of the CGA system as an indicator, the formula of the
CGA system was optimized through three-factors four-levels orthogonal experiments, which is: 3%
Bentonite mud+3% EST+3% AGS-8+3% ChCl PEG.

Microscopic tests showed that stable aphrons were successfully generated in
 36%NaCl-CGA or 7.5%CaCl<sub>2</sub>-CGA system aged at 150°C. Aphrons maintain a stable morphology
 throughout a 2-hour observation period without significant coalescence.

386 3) The Herschel-Bulkely model accurately describes the rheological behavior of CGA fluids 387 containing NaCl/CaCl<sub>2</sub>. The addition of NaCl increases the viscosity of the CGA fluid, while 388 CaCl<sub>2</sub> is the opposite. The CGA fluid maintains appropriate rheological parameters (fluidity index, 389 gel strength, and yield point) and shear thinning behavior, which means good cuttings carrying 390 capacity. 391 4) With the addition of NaCl/CaCl<sub>2</sub>, the CGA has a low filtration volume (<7mL) and low 392 value of lubrication coefficient (0.107~0.132), which meets API requirements of filtration and 393 lubricity properties.

394 5) The anti-cuttings pollution ability of 150°C aged CGA can reach 10%. CGA, 395 36%NaCl-CGA, and 7.5%CaCl<sub>2</sub>-CGA all have low linear expansion rates (<28%) and high rolling 396 recovery rates (>84%). The CGA system has good inhibitory performance and is compatible with 397 sandstone, shale and other easily collapsed formations.

398 6) The CGA system has been proven to be a biodegradable and non-toxic drilling fluid 399 system.

400 In summary, this study breaks through the limitations of high temperature, high salinity, and 401 high calcium conditions on the CGA properties, providing possibilities for the application of CGA 402 drilling fluids in complex formations.

403

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Table 1. Application status of CGA drilling fluid in the field.

Year	Main	Drilling fluid	Field application			
	components	properties				
2005[47]	Foaming agent HYF and foam stabilizer CMC	ρ: 0.75~0.96 g/cm <sup>3</sup> ; FL<10mL; YP/PV: 0.62~0.95.	It effectively solves the problems of serious lost circulation in northeastern Sichuan. Compared with similar wells in adjacent areas, the drilling speed is increased by $3\sim7$ times, the drilling time is shortened by 29.5 days, and the direct economic benefit is more than 1 million ¥.			
2016[48]	Foaming agent LF-2 and foam stabilizer HMC-1, viscosity increasing agent HT-XC, fluid loss agent	<ul> <li>ρ: 0.85~0.95 g/cm<sup>3</sup>;</li> <li>FL&lt;7.4 mL;</li> <li>Strong inhibitory and blocking ability;</li> <li>Temperature</li> <li>resistance: 150°C.</li> </ul>	The system is used in 21 wells in the oilfields of Cicai, Shencai, and Shenleng, with a maximum well depth of 4005m and a maximum temperature of 141.5°C. Compared with adjacent wells, the ROP in the same layer is increased by 70% after the use of microbubble drilling fluid.			

	KH-93 SMP	- II	3					
2018[	Foaming TSB and stabili HV-P	g agent Str l foam c izer a AC S	oam er and inhibition:		It is applied to the well section (2542-2837m Kizivolda gas field in Kazakhstan. The system h density and stable wellbore, and there is no scrat obstruction when drilling large sections of m gypsum, and gypsum mudstone.			
Foaming agen LHPF-1 and BS-12, foam stabilizer XG		1 and a foam er XG	g/cm <sup>3</sup> ; ae plugging rate nd penetration recovery rate >90%; Cuttings esistance: 7%.	complica coincider		accidents. I bore stabilit	lt has a high g y, good inhibit	
53 54		Tabl	2 Eundomont	al curfactant	parameters of A			
54	Sample	CMC (mn			$\Gamma_{\rm max}$ (nmol/cm <sup>2</sup> )		$(nm^2)$	
	AGS-8	9.69	• • •	38.1	0.15		.10	
55								
56			Table 3. Bio	odegradabilit	y of materials			
	Sample	BO	$D_5 (mg/L)$	COD (mg	/L) Y=	(BOD <sub>5</sub> /COI	D) *100	
	EST		55.8	168		33.21		
	AGS-8		70.7	224		31.56		
	ChCl-PEC	j	99.2	285		34.81		
57 58			l is difficult to ily degradable	e	.0≤Y<25.0, th	e material is	degradable; If	
59		Table /	Biological to	vicity classifi	cation of drillin	o fluid		
59	Toxicity		4. Biological to		cation of drillin	g fluid	Emission	
59	Toxicity classification	Table 4       Extremely       high toxic	<b>4.</b> Biological to High toxic	xicity classifi Moderate toxic	cation of drillin Micro toxic	g fluid Non-toxic	Emission standard	
59 50	-	Extremely		Moderate		-		
59	classification EC <sub>50</sub>	Extremely high toxic <1	High toxic	Moderate toxic 100~1000	Micro toxic 1000~10000	Non-toxic >10000	standard	
59 50 51	classification EC <sub>50</sub>	Extremely high toxic <1 Table 5. Orth	High toxic 1~100 ogonal experin	Moderate toxic 100~1000	Micro toxic	Non-toxic >10000 CGA system	standard	

1		3%	2%	75
2	10/	4%	3%	68
3	1%	5%	4%	60
4		6%	5%	54
5		3%	2%	98
6	20/	4%	3%	75
7	2%	5%	4%	88
8		6%	5%	95
9		3%	2%	120
10	20/	4%	3%	95
11	3%	5%	4%	108
12		6% 5%		135
13		3%	2%	150
14	40/	4%	3%	90
15	4%	5%	4%	110
16		6%	5%	92
P <sub>1</sub>	64.25	110.75	87.5	
$P_2$	89	82	102.75	
<b>P</b> <sub>3</sub>	114.5	91.5	91.25	$R_A > R_B > R_C$
$P_4$	110.5	94	96.75	
R	50.25	28.75	15.25	

 Table 6. Data statistics of the bubble size distribution.

Formula and Conditions	Average diameter (μm)	Percentage of bubble size in the range of <100μm (%)	Percentage of bubble size in the range of >100µm (%)
150°C aged CGA	99.15	59.16	40.84
150°C aged CGA2hours	120.00	47.89	52.11
150°C aged CGA+36% NaCl	97.35	59.65	40.35
150°C aged CGA+36% NaCl2hours	96.32	56.16	43.84
150°C aged CGA+7.5%CaCl <sub>2</sub>	74.65	78.18	21.82
150°C aged	93.50	67.21	32.79

565 566		Table 7	Fitting res	sults of CGA	system to four	r rheological	models			
	~ ~ ~ .	Bingham Model			Power-law Model		Casson Model		Herschel-Bulkely Model	
Formula	Condition	Fitting parameter	R <sup>2</sup> /RMSE	Fitting parameter	R <sup>2</sup> /RMSE	Fitting parameter	R <sup>2</sup> /RMSE	Fitting parameter	R <sup>2</sup> /RMSE	
<u></u>	Before aging	$\tau_0 = 8.752$ $\mu_P = 0.053$	0.973/ 3.13	K=1.123 n=0.571	0.991/1.83	$\tau_c = 4.193$ $\eta_{\infty} = 0.033$	0.997/0.10	$\tau_y=4.270$ K=0.464 n=0.691	0.999/0.68	
CGA	150°C	$\tau_0=13.788$ $\mu_P=0.071$	0.941/ 6.29	K=2.448 n=0.503	0.999/0.91	$\tau_c = 5.973$ $\eta_{\infty} = 0.047$	0.984/0.29	τ <sub>y</sub> =2.809 K=1.723 n=0.550	1.000/0.14	
+10%NaCl	Before aging	$\tau_0=8.523$ $\mu_P=0.063$	0.972/ 3.74	K=0.983 n=0.612	0.996/1.39	$\tau_c=3.441$ $\eta_{\infty}=0.043$	0.996/0.14	$\tau_y=3.079$ K=0.572 n=0.685	0.999/0.58	
+10%1\aC1	150°C	$\tau_0=13.193$ $\mu_P=0.074$	0.938/ 6.74	K=2.252 n=0.519	1.000/0.44	$\tau_c = 5.118$ $\eta_{\infty} = 0.052$	0.981/0.33	$\tau_y$ =1.277 K=1.933 n=0.539	1.000/0.17	
	Before aging	$ au_0=9.070$ $\mu_P=0.068$	0.966/ 4.50	K=1.122 n=0.604	0.998/1.00	$\tau_c=3.233$ $\eta_{\infty}=0.049$	0.991/0.23	$\tau_y=2.163$ K=0.799 n=0.649	1.000/0.52	
+20%NaCl	150°C	$ au_0 = 16.752$ $\mu_P = 0.077$	0.942/ 6.68	K=3.251 n=0.475	0.996/1.68	$\tau_c = 8.118$ $\eta_{\infty} = 0.047$	0.986/0.27	$\tau_y$ =5.118 K=1.807 n=0.553	1.000/0.58	
+30%NaCl	Before aging	$ au_0 = 14.021$ $\mu_P = 0.060$	0.911/ 6.59	K=3.109 n=0.446	0.999/0.66	$\tau_c = 6.330$ $\eta_{\infty} = 0.038$	0.970/0.36	$\tau_y$ =0.670 K=2.865 n=0.457	0.999/0.64	
+30/014401	150°C	$ au_0 = 13.888$ $\mu_P = 0.084$	0.952/ 6.66	K=2.116 n=0.546	0.998/1.30	$\tau_c = 5.497$ $\eta_{\infty} = 0.059$	0.986/0.30	$\tau_y$ =2.908 K=1.513 n=0.591	0.999/0.80	
+36%NaCl	Before aging	$\tau_0=17.117$ $\mu_P=0.067$	0.909/ 7.48	K=4.047 n=0.427	0.999/0.80	$\tau_c = 8.251$ $\eta_{\infty} = 0.041$	0.970/0.37	$\tau_y$ =1.715 K=3.381 n=0.451	0.999/0.69	
+307011401	150°C	$ au_0 = 16.827$ $\mu_P = 0.085$	0.934/ 7.93	K=3.112 n=0.494	0.999/0.81	$\tau_c = 7.174$ $\eta_{\infty} = 0.056$	0.981/0.35	$\tau_y$ =2.538 K=2.409 n=0.528	1.000/0.22	
+5%	Before aging	$\tau_0=13.100$ $\mu_P=0.055$	0.927/ 5.41	K=2.871 n=0.445	0.997/1.00	$\tau_c = 6.381$ $\eta_{\infty} = 0.033$	0.977/0.30	$\tau_y=2.882$ K=1.892 n=0.500	0.999/0.64	
CaCl <sub>2</sub>	150°C	$\tau_0 = 7.258$ $\mu_P = 0.062$	0.963/ 4.26	K=0.885 n=0.622	1.000/0.29	$\tau_c = 2.102$ $\eta_{\infty} = 0.048$	0.989/0.25	τ <sub>y</sub> =0.620 K=0.796 n=0.636	1.000/0.15	
+7.5%CaCl <sub>2</sub>	Before aging	$ au_0 = 7.464 \ \mu_P = 0.042$	0.959/ 3.07	K=1.097 n=0.543	0.993/1.26	$\tau_c=3.448$ $\eta_{\infty}=0.027$	0.993/0.14	$\tau_y=2.844$ K=0.557	0.998/0.69	

150°C	0		=0.549 0.684 0.4	998/1.04	$\tau_c = 1.213$ $\eta_{\infty} = 0.049$	0.991/0.2	$\begin{array}{c} n=0. \\ \tau_y=0. \\ 2 & K=0. \\ n=0. \end{array}$	203 528 0.998/
568 <b>Table 8.</b> Rh	eological para		•			ations of Na	aCl/CaCl <sub>2</sub>	
569			and after ag	ging at 150		DI /	VD	
Formula	Condition	LSRV (mPa·s)	GS <sub>10min</sub> (Pa)	n	AV (mPa·s)	PV (mPa·s)	YP (Pa)	YP/PV
CGA-2	Before aging	84583	7.154	0.691	59.5	39	20.951	0.537
	150°C/16h	51189	7.665	0.550	80.5	49	32.193	0.657
CGA-2+10%NaCl	Before aging	63394	6.132	0.685	68.5	47	21.973	0.468
	150°C/16h	38995	5.621	0.539	82.5	51	32.193	0.631
CGA-2+20%NaCl	Before aging	63995	5.621	0.649	74	51	23.506	0.461
	150°C/16h	39995	8.176	0.553	89	56	33.726	0.602
CGA-2+30%NaCl	Before aging	71790	7.665	0.457	69	40	29.638	0.741
	150°C/16h	42796	6.132	0.591	94	65	29.638	0.456
CGA-2+36%NaCl	Before aging	75392	9.198	0.451	79	45	34.748	0.772
	150°C/16h	46595	6.132	0.528	96	57	39.858	0.699
CGA-2+5%CaCl <sub>2</sub>	Before aging	47991	7665	0.500	64	38	26.572	0.699
	150°C/16h	36195	5.11	0.636	66	46	20.440	0.444
CGA-2+7.5%CaCl <sub>2</sub>	Before aging	38500	6.132	0.634	47.5	30	17.885	0.596
	150°C/16h	29393	5.11	0.689	62	42	20.440	0.487

**Table 9.** Filtration and lubricity of CGA system with different concentrations of NaCl/CaCl<sub>2</sub>

before and after aging at 150°C

Formula	Condition	FL <sub>API</sub> (mL)	Adhesion coefficient	Lubrication coefficient
CGA	Before aging	6.2	0.057	0.116
CUA	150°C/16h	4.2	0.063	0.122
CGA+10%NaCl	Before aging	6	0.059	0.120
COATIO70INaCI	150°C/16h	4.5	0.078	0.132
CGA+20%NaCl	Before aging	6	0.068	0.117
CUA+20%INaCI	150°C/16h	3.5	0.073	0.122
CGA+30%NaCl	Before aging	5.5	0.072	0.108

	150°C/16		C/16h	5h 3		0.076		0.114		
	CGA+36%NaCl	Before	e aging	4.	8	0.073		0.1	04	-
		150°	C/16h	2.	5	0.08	6	0.1	07	
	CGA+5%CaCl <sub>2</sub>		e aging	-		0.097		0.098		
			C/16h	4		0.09		0.1		_
	CGA+7.5%CaCl	CGA+7.5%CaCl <sub>2</sub> Before agin		5.		0.10		0.1		
		150°	C/16h	3.	5	0.12	.3	0.1	16	_
573										
574				• Cuttings re				E T		
	Formula		$GS_{10\min}$	AV	PV	YP (Da)	YP/PV	FL <sub>API</sub>	f	Κ
	CGA	(mPa·s) 51189	(Pa) 7.665	(mPa·s) 80.5	(mPa·s) 49	(Pa) 32.19	0.657	(mL) 4.2	0.063	0.122
C	GA+5%Cuttings		6.132	80.3 71	49 42	29.64	0.037	4.2 5.2	0.005	0.122
	GA+10% Cuttings		7.154	71 89	42 49	40.88	0.700	5.2 6.2	0.100	0.155
	GA+15% Cuttings		12.264	112.5	75	38.33	0.511	4.6	0.112	0.191
575		01//0	12.201	112.0	10	50.55	0.011		0.102	0.210
576	Та	ble 11. Inhit	nition ne	erformance c	of various d	rilling flu	id system	s		
570	14			inomianee e		near expan	-	olling re	coverv	ı
		Formula	ì			rate (%)		rate (	•	
		CGA				5.63		87.98		-
		CGA+36%1	NaCl	1 6.24				84.94	%	
	(	CGA+7.5%C	CaCl <sub>2</sub>	l <sub>2</sub> 4.29 84.269			<b>i%</b>			
	Water-based drill	ing fluids ba	sed on H	KCl, polyme	rs,					
	amine-based polyalcohols and na			no-wetting		6.6~7.2		80.8~8	35.5	
		modifiers[	-							
	Solid-free low dama	•	•	~5 *				85.3	3	
	dril	ling fluid sys	stem[53]	]		_			-	
577										
578	Appendix									
	AGS-	8			n Amino Ac					
	API		American Petroleum Institute							
	AV					nt Viscos	•			
	BOD			the Five	-day Bioch			mand		
	CaCl					m Chlorid				
	CGA			Colloidal Gas Aphron						
	ChCl-Pl			G		bricant	1.0.			
	CAPI			Coc	onut Oil Ar	-	•	e		
	CMC			Carboxymethyl Cellulose						
	COD			Chemical Oxygen Demand Cetyltrimethylammonium Bromide						
	CTAE EC <sub>50</sub>		the	-	-				ical	
	$EC_{50}$		une	Concentrati			icais III (fi	C DIOIOg	icai	
	EST			ΔΝ	lodified Sta	icity Test	Stabiliza	r		
	f				Mud Cake A					
	I			uie r	Tuu Cake F	1011031011	Coemciel	n		

FL <sub>API</sub>	Filtration Volume
GS <sub>10min</sub>	10 minutes Gel Strength
HTHP	High Temperature High Pressure
k	the Extreme Pressure Lubrication Coefficient
LSRV	Low Shear Rate Viscosity
n	the Flow Behavior Index
$\mathbf{R}^2$	the Goodness of Fit Value
ROP	the Borehole Footage Per Unit Time
RMSE	the Root Mean Square Error
SDS	Sodium Dodecyl Sulfate
XG	Xanthan Gum
X-100	Triton X-100
YP	Yield Point
τ	the Shear Stress
θ	the Reading of Six-speed Viscometer
γ	the Shear Rate
$ au_0$	the Yield Point of Bingham Model
$\mu_{ m P}$	The Plastic Viscosity
$ au_{ m c}$	the Yield Point of the Casson Model
$\eta_\infty$	the Ultimate High Shear Viscosity
$ au_{ m y}$	the Yield Point of the H-B Model
Verve	Surface Tension Corresponding to the Critical Micelle
үсмс	Concentration
$\Gamma_{\max}$	the Saturated Adsorption Capacity
$A_{min}$	the Minimum Surface Area



Figure 1. Schematic diagram of the structure of the aphron.



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Figure 2. The figures of aphrons in the CGA system aged at 150°C. (a) CGA; (b) CGA+36% NaCl;
(c) CGA+7.5% CaCl<sub>2</sub>



- Figure 3. The bubble size distribution of aphrons in the CGA system aged at 150°C. (a) CGA; (b)
   CGA+36%NaCl; (c) CGA+7.5%CaCl<sub>2</sub>







Figure 4. Observation image of Aphrons in CGA aged at 150°C after standing for 2 hours
(one cycle of drilling fluid): (a)CGA system; (b) CGA+36% NaCl; (c) CGA+7.5% CaCl<sub>2</sub>.







**Figure 5.** The bubble size distribution of aphrons in the CGA system aged at 150°C and standing



Bubble size (µm)

Q

(a)





(b) the filtration fluid of CGA drilling fluid.





Figure 7. Sandstone samples from rolling recovery experiments: (a) initial samples; (b) After boiling in water; (c) After hot rolling in the CGA system; (d) After hot rolling in CGA+36% NaCl; (e) After hot rolling in CGA+7.5% CaCl<sub>2</sub>.





