



Experimental study on characteristics of gas field mercury-containing sludge and the sludge reduction

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ABSTRACT

In order to obtain the basic characteristics of gas field mercury-containing sludge and provide a technical support for the sludge treatment, a detailed experiment was carried out. The experimental results showed the mercury content and moisture content of the sludge were very high, and the divalent mercury was the main existence form of mercury, and the little particle size was the main reason for the poor sedimentation performance of the raw sludge. The relations of moisture content and mercury content at different drying temperature indicated the mercury in the sludge was unstable, and the critical temperature to keep the mercury stable should below 80°C. Moreover, the volume of supernatant and the moisture content after centrifugal dehydration were used to evaluate the effect of sludge conditioning, and the proper coagulants and optimal dosages were determined. Additionally, the relations among moisture content, centrifugal rotational speed and centrifugation time showed the suitable centrifugal rotational speed was about 3,000 r/min, but the moisture content of centrifugation cannot meet the dehydration requirements of sludge packaging and transportation, thus the sludge drying should be used for further dewatering, and the reasonable drying temperature should below 80°C as also.

Keywords: Mercury; Sludge reduction; Moisture content; Stability; Drying temperature

1. Introduction

Mercury is a hazardous element, which would cause personnel poisoning, environmental pollution and even production accident [1–3], so the use and control of mercury requires extra care. Generally, the mercury does not blend and react with the common elements in the earth's crust and as a result mercury concentrates in the ore, but it is released into the environment as a result of the oil or gas well drilling into the mercury bearing rocks and soils. Currently, mercury was reported to be found in many gas

fields all around the world, such as the gas fields distributed in Thailand, Egypt, northwest China and so on [1,4–6], some mercury-containing gas fields and their mercury content are shown in Table 1. Under normal circumstances, in order to ensure the safety of pipeline transportation and improve the economic benefit, the dehydration and light hydrocarbon recovery would be conducted in the natural gas processing plant [7–9], and these separation treatments would produce multiple mercury-containing fluids, include the natural gas, condensate and gas field water. Then the mercury removal technology should be considered if the

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Table 1
Reported mercury-containing gas fields and its mercury content [1,7]

Gas field name	Natural gas ($\mu\text{g}/\text{m}^3$)	Condensate ($\mu\text{g}/\text{L}$)
Gulf of Thailand	100–400	400–1,200
Europe	100–150	–
South America	50–120	50–100
Africa	80–100	500–1,000
Gulf of Mexico (USA)	0.02–0.40	–
Overthrust Belt (USA)	5–15	1–5
North Africa	50–80	20–50

mercury content of these fluids exceed the requirement of standard, and the distribution of mercury in the gas field can be indicated by the data shown in Table 2. Moreover, due to the flow of mercury-containing fluid in the natural gas processing plant, the equipment and pipeline may suffer from the corrosion caused by mercury aggregation as also, especially the aluminum equipment [10–12].

The treatment of mercury-containing gas field water is one of the serious problems [5]. For one thing, the mercury content of the gas field water is very high. However, the local government usually has strict requirements on the mercury content in the discharge wastewater, the Integrated Wastewater Discharge Standard (GB 8978-1996) of China prescribes the maximum allowable emission concentration of mercury is $50 \mu\text{g}/\text{L}$, while the Thailand's regulatory discharge standards requires the concentration of mercury should be less than $10 \mu\text{g}/\text{L}$, these strict requirements pose great challenges to the mercury removal technologies. For another, the composition of gas field water is very complex, except the multiple forms of mercury, it contains oil, suspended solids, mechanical impurities and a variety of other non-metallic and metal ions, which bring additional difficulty to the water treatment process. The reported mercury removal technologies include adsorption, membrane separation, photo catalysis, ultrasound, etc. [13–16], and the

activated carbon is thought to be the most valuable absorbent [17]. Additionally, in contrast with the reinjection of mercury-containing gas field water to the gas reservoir, to satisfy the discharge requirements would need more strict treatment technology and economic investment, so the reinjection is a more reasonable way to choose.

Anyway, the treatment of mercury-containing gas field water would concentrate the mercury in the sludge. According to the National Catalogue of Hazardous Wastes (2016), the mercury-containing sludge belongs to solid waste for its toxicity, so the treatment of them needs to pay more attention. The U.S EPA (Environmental Protection Agency) recommends a method which depends on the mercury content to choose the treatment process, that is, adopting the pyrolysis process (mercury content $\geq 260 \text{ mg}/\text{kg}$) or solidification and extraction (mercury content $20\text{--}260 \text{ mg}/\text{kg}$). However, the specific treatment process also needs to consider the actual situation of the gas field. If an abandoned oil and gas well is present, it is a convenient method to inject mercury-containing sludge into the abandoned well, and this method has been applied in Gulf of Thailand [18]. Reversely, the EPA recommended method would be adopted if there is no abandoned well. Additionally, if the gas fields are scattered and the sludge throughput of single gas field is small, constructing a pyrolysis device for every mercury-containing gas field would cause more investment, so the centralized treatment of sludge would have more advantages after a sludge reduction in the scattered gas fields.

The main purpose of sludge reduction is to reduce the moisture content of sludge, so as to facilitate the transportation and subsequent treatment. The sludge reduction has close relations with the characteristics of mercury-containing sludge, including the stability of mercury, initial moisture content, particle size, etc., and how to improve the dewatering performance of the sludge and determine an appropriate drying temperature are two crucial problems to be considered. Due to the lack of experience in treatment of mercury-containing sludge in gas field, and the basic characteristics of sludge is not known, a detailed experiment was carried out to solve these problems.

Table 2
Mercury distribution in the gas field of northwest China^a

Sample	Gas field name			
	KeLa	YingMai	YaHa	DaBei
Raw gas ($\mu\text{g}/\text{m}^3$)	89.28–1,711.15	0.288–1.296	15.55–58.90	106.42–669.89
Commercial gas ($\mu\text{g}/\text{m}^3$)	0.14–0.75	0.0432–0.072	2.91–7.59	1.728–3.18
Stable light hydrocarbon ($\mu\text{g}/\text{L}$)	–	19.969–24.576	–	–
Condensate ($\mu\text{g}/\text{L}$)	–	77.952–93.312	47.6–50.2	2,226.8–2,262.62
Gas field water ($\mu\text{g}/\text{L}$)	128.4–1,712.7	18.7–34.45	8.4–140	83.82–435.3
Glycol-rich solution ($\mu\text{g}/\text{L}$)	17,781.42–34,793.6	–	–	2,115–5,030
Glycol-lean solution ($\mu\text{g}/\text{L}$)	165.09–543.06	–	–	33–56.5
Air (ng/m^3)	25–49	2–14	8–14	15–50
Soil ($\mu\text{g}/\text{kg}$)	18–34,100	8–138	87.5–12,800	6.05–20

^aThese data were detected by us in 2016. The mercury analyzer RA-915+ and its accessories RP-91NG, PYRO-915+ and RP-92 were used in the test.

2. Experiment

2.1. Sludge sample

The sludge was sampled from the mercury-containing gas field water treatment plant. The mercury removal method of the water treatment process was to oxidize the elemental mercury to ionic state at first, and then use MRA (mercury removal agent) to react with ionic mercury to form a complex with a small solubility. Lastly, the coagulation method was used to precipitate the mercury-containing complex into the sludge. The water treatment chemicals which were used to remove the mercury include sodium hypochlorite, PFC (polyferric chloride), PAM (polyacrylamide) and a sulfur-containing MRA.

2.2. Experimental procedures and apparatus

2.2.1. Testing of sludge basic characteristics

The basic parameters which had been detected include the moisture content, oil content, total mercury content, pH, relative density, settling velocity and SRF (specific resistance to filtration). Moreover, the existence forms of the mercury in the sludge were analyzed as also. The apparatus which were used in the test mainly include the infrared oil measuring instrument (OIL 460), atomic fluorescence spectrometer (AFS-2100), morphological analysis pretreatment device (SAP-20), auto sampler (LUMTECH 5500), drying box (101-005) and electronic balance (SQP). All tests were performed in strict accordance with national standards or industry regulations.

Moreover, in order to study the sedimentation performance of the mercury-containing sludge, 100 mL raw sludge was placed in a 100 mL measuring cylinder, and the volume of the sludge was recorded every 1 min. The laser particle size analyzer MASTERSIZER 2000 was used to measure the sludge particle size.

2.2.2. Testing the stability of mercury

This experiment was designed to study the relations among mercury content, moisture content and drying temperature. First, the relations of moisture content and mercury content were measured, the mercury-containing sludge was placed in an electric drying box with fixed temperature (25°C and 100°C, respectively), and the moisture content and mercury content of the sludge were measured every 10 min. Second, the relations between mercury content and drying temperature were studied, the raw sludge was placed in an electric drying box with fixed temperature (50°C, 60°C, 80°C, 100°C, 120°C, 140°C, 160°C, 180°C and 200°C, respectively), and then measure the mercury content if the sludge weight changes did not exceed 0.0005 g between two consecutive measurements.

2.2.3. Sludge conditioning experiment

In this experiment, the single-factor method was used to screen out the appropriate type of chemicals and determine the optimal dosages, and the volume of supernatant after 30 min sedimentation and the moisture content of

sludge after centrifugal dehydration at 3,000 rpm in 10 min were used to evaluate the treatment results. The chemicals used in this experiment included three inorganic coagulants, PAC (polyaluminum chloride), PFC and PAFC (polymeric aluminum ferric chloride), and nine different PAM (organic coagulant). All the dosages were fixed when screening the type of the chemicals, and only the dosage of organic coagulant (inorganic coagulant) was fixed when determining the dosages of inorganic coagulant (organic coagulant).

2.2.4. Sludge dewatering and drying experiment

The relations among the centrifugal rotational speed, centrifugation time and moisture content of sludge were studied in this part, the TD5Z desktop centrifuge was used to provide different centrifugal rotational speed. The studied centrifugal rotational speed included 2,000; 2,500; 3,000; 3,500 and 4,000 rpm, and the moisture content of sludge were measured at 2, 4, 6, 8, 10, 15 and 20 min, respectively. Moreover, in order to further decrease the moisture content of sludge, the sludge was placed in the 101 type electric blast drying box at a constant temperature (80°C, 100°C and 120°C, respectively), and the relations of drying time and moisture content can be determined.

3. Results and discussion

3.1. Basic characteristics of the sludge

The basic parameters of the sludge are shown in Table 3. It is obvious that the mercury content was more than 260 mg/kg, the divalent mercury was its main existence form, and a small amount of highly toxic methyl mercury was contained in the sludge, the ethyl mercury and alkyl mercury were not detected in the sludge. The existence form of mercury in the sludge indicated the mercury had been oxidized to ionic state in the water treatment process, and there was no doubt the existence form of mercury had close relation with the water treatment method. Due to the high mercury content in the sludge, it was reasonable to build a pyrolysis device in a centralized station to treat the sludge for there was no abandoned well. Moreover, the moisture content of the sludge was very high, which would cause difficult to transport them from a scattered water treatment plant to the centralized treatment center, so the sludge reduction should be considered. Additionally, the relative density, settling velocity and SRF shown in Table 3 indicate the dewatering performance of the sludge was poor, which brought difficult to the sludge reduction process.

The sedimentation performance of the raw sludge is shown in Fig. 1. It is clear the sediment process was very slow, the volume of sludge only changed from 100 to 86 mL in 30 min. Moreover, the particle size of the sludge was very little, and the percentage of sludge particle size below 100 µm accounted for 100% in our test, and it can be determined by the data shown in Table 4. The little particle size of sludge was the reason for its poor sedimentation performance, and it also indicated that the pore size of extrusion dewatering equipment was bigger than the sludge particle size, so the extrusion dewatering equipment was not suitable to use in the sludge dewatering process. Additionally, the

Table 3
Basic parameters of mercury-containing sludge

Measured parameters	Sample No.			
	1#	2#	3#	4#
Moisture content, %	99.31	99.24	99.4	99.14
Oil content, mg/kg	13,558	12,751	11,074	30,487
Total mercury content, mg/kg	585	118	115	498
Divalent mercury, mg/kg	557	92.0	95.3	467
Methyl mercury, mg/kg	0.274	4.38	6.30	0.65
Ethyl mercury, mg/kg	–	–	–	–
Alkyl mercury, mg/kg	–	–	–	–
pH	6.92	6.98	6.97	6.96
Relative density	1.0143	1.0142	1.0140	1.0142
SV (settling velocity), %	94.7	89.8	85.6	93.4
SRF, 10 ¹² m/kg	9.87	9.46	9.71	9.89

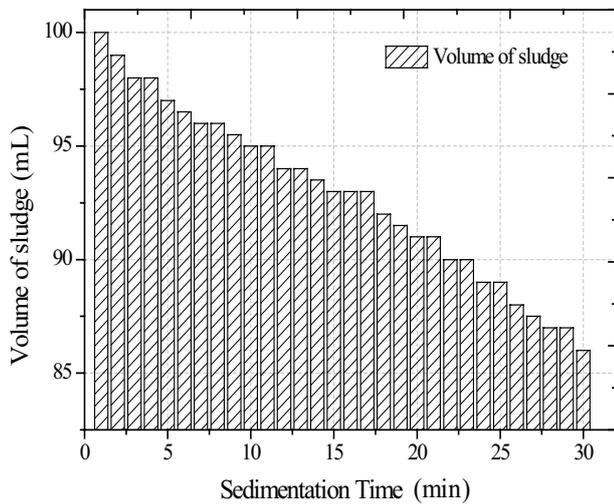


Fig. 1. Sedimentation performance of the sludge in 30 min.

poor sedimentation performance and little particle size of the sludge would cause difficult to the sludge reduction process, so the sludge conditioning should be adopted to improve its performance.

3.2. Stability of mercury in the raw sludge

The relations of moisture content and mercury content at different temperature are shown in Fig. 2. Due to the sludge was placed in the drying box, its moisture content was decreasing with the evaporation of water, and the mercury content of sludge would increase if the mercury was stable at the same time. The curve at 25°C was in line with this assumed trend, and the mercury content changed from 465 to 2,699.3 mg/kg when the moisture content was decreased from 85% to 10.5%. However, the curves at 100°C showed a different trend, the mercury content decreased from 498 to 33.6 mg/kg when the moisture content decreased from 84% to 7.22%. This phenomenon indicated the mercury in the sludge was unstable when the drying temperature was

Table 4
Particle size of the sludge

Measured parameters	Sample No.			
	1#	2#	3#	4#
Surface area mean particle size, μm	25.273	21.117	24.144	8.614
Volume average particle size, μm	35.804	33.954	33.555	29.377
Particle size < 30 μm , %	47	49	50	58
Particle size < 50 μm , %	78	81	83	87
Particle size < 100 μm , %	100	100	100	100
D10 ^a	14.615	13.620	14.187	6.152
D50 ^b	32.115	30.320	30.445	26.276
D90 ^c	63.011	60.207	58.021	56.473
Residual, %	0.501	0.537	0.576	0.978

^aCorresponding particle size when the cumulative particle size distribution reaches 10%.

^bCorresponding particle size when the cumulative particle size distribution reaches 50%.

^cCorresponding particle size when the cumulative particle size distribution reaches 90%.

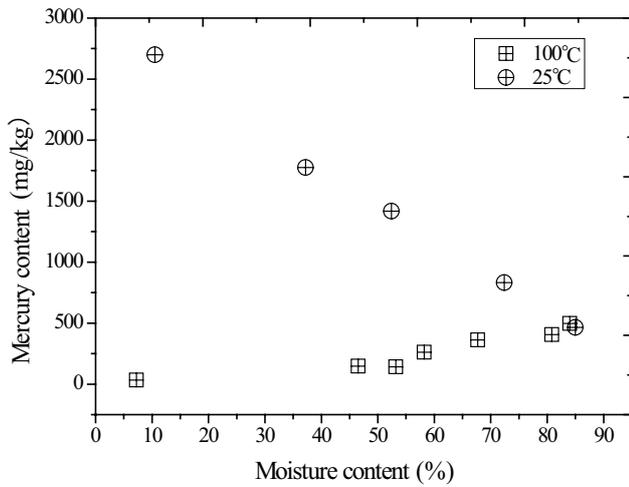


Fig. 2. Relations of moisture content and mercury content at different temperature.

high enough, though the divalent mercury was its main existence form, the mercury in the sludge can escape with the evaporation of mercury attached organic matter, such as the hydrocarbons and alcohols, and there should be a critical temperature below which the mercury in the sludge can keep its stability.

In order to determine the critical temperature of drying process, a detailed experiment was carried out, and the relations between mercury content and drying temperature are shown in Fig. 3. When the drying temperature changed from 80°C to 100°C, the mercury content of sludge had a sharp change, and it decreased from 1,782 to 69.33 mg/kg. Moreover, the mercury content changed a little when the temperature was higher than 100°C or below 80°C. The relations shown in Fig. 3 indicated the critical temperature was between 80°C and 100°C. Considering the fluctuation of the water treatment process in the actual operation, a small amount of elemental mercury may present in the sludge, so the drying temperature should not be too high, and it was better to keep the drying temperature below 80°C in engineering to ensure the stability of mercury.

3.3. Sludge conditioning result

3.3.1. Screening of inorganic coagulant

The chemicals were added to three beakers which filled with 70 mL mercury-containing sludge. The dosages of three inorganic coagulants were fixed at 150 mg/L, and the dosage of PAM was 25 mg/L. The screening results of the inorganic coagulant types are shown in Table 5. It is clear the treatment effect of PAC was better than the other two

Table 5
Screening results of the inorganic coagulant type

Measured parameters	PAC	PFC	PAFC
Volume of supernatant, mL	23	20	19
Moisture content, %	86.75	87.14	86.78

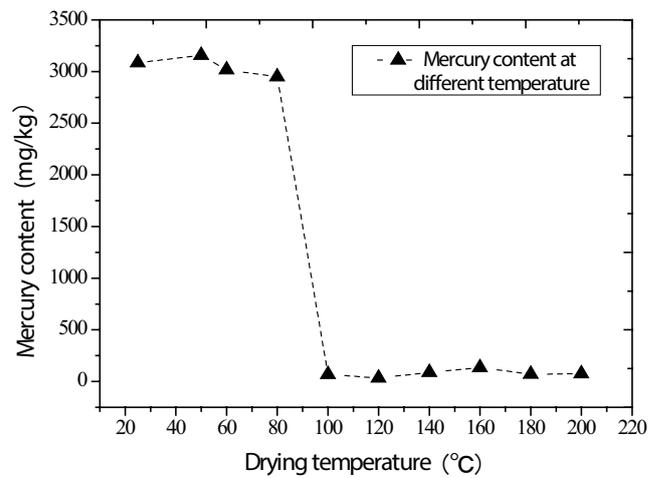


Fig. 3. Relations of mercury content and drying temperature.

inorganic coagulants, because the larger volume of supernatant and lower moisture content indicated the treated sludge had a much better sedimentation performance and dewatering performance. Moreover, the sludge which was treated by PAC had a bigger floc, and its supernatant was more transparent.

The optimal dosage of PAC can be determined from Fig. 4. In this experiment, 0, 100, 150, 200, 300, 400 and 500 mg/L PAC were added to the beakers filled with 50 mL raw sludge, and the dosage of PAM was fixed at 25 mg/L. The curves in Fig. 4 show that the optimal dosage of PAC was around 150 mg/L, because it had a lower moisture content and larger supernatant volume.

3.3.2. Screening of organic coagulant

The type and dosage of organic coagulant were determined as the above way used by the inorganic coagulant. The dosage of PAC was fixed at 150 mg/L, and then nine different organic coagulants were added to the beakers filled with 70 mL raw sludge, each of them control the dosage at 25 mg/L, the experimental results are shown in Table 6.

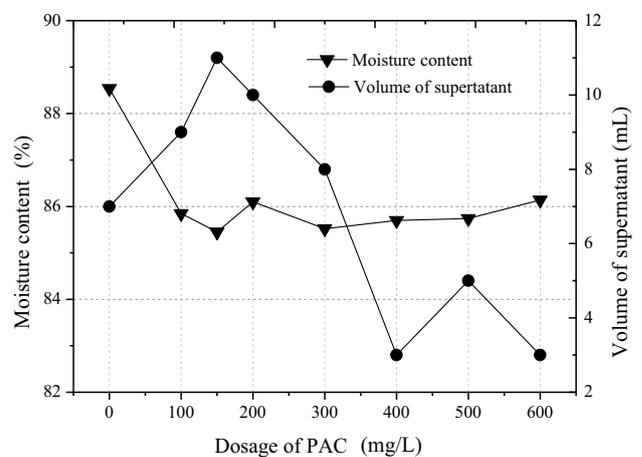


Fig. 4. Effect of the PAC dosage on the sludge treatment.

Table 6
Screening results of the organic coagulant type

PAM type	Volume of supernatant, mL	Moisture content, %
CPAM ^a , hydrolysis degree 5	18	86.51
CPAM, hydrolysis degree 20	19	86.44
CPAM, hydrolysis degree 40	17	86.35
CPAM, hydrolysis degree 50	17	86.32
NPAM ^b , hydrolysis degree 2	20	85.91
NPAM, hydrolysis degree 5	22	85.88
APAM ^c , molecular weight 1,600 million	25	85.31
APAM, molecular weight 1,700 million	23	85.45
APAM, molecular weight 1,800 million	26	85.24

^aCationic polyacrylamide.

^bNonionic polyacrylamide.

^cAnionic polyacrylamide.

Obviously, the APAM treated sludge had a lower moisture content and larger volume of supernatant, and the price of APAM was much cheaper, so the suitable type of organic coagulant was the APAM with a molecular weight 1,800 million.

Moreover, the optimal dosage of PAM was determined through experiment. 150 mg/L PAC were added to the beakers filled with 50 mL raw sludge at first, and then 0, 10, 15, 20, 25, 30, 35 and 50 mg/L PAM were added, respectively. The results shown in Fig. 5 indicate that the optimal dosage of PAM was around 25 mg/L, because the volume of supernatant was larger and the moisture content was lower. Additionally, the sedimentation velocity of the sludge was faster and the supernatant was clearer, it is because the larger and solid flocs were formed.

3.4. Sludge dewatering and drying

The above experimental results showed the sludge conditioning improved the dewatering performance of sludge, but the effect was not significant, the moisture content of sludge only decreased to about 84% at the optimal conditions, and it cannot satisfy the dehydration requirements for sludge packaging and transportation, so the sludge dewatering and drying should be further considered. Due to the little particle size of mercury-containing gas field sludge and the existence of unstable mercury, the centrifugal dewatering was recommended for its airtightness and continuity.

The initial moisture content of conditioned sludge was 95.24%, and the relations of moisture content with centrifugation time at different centrifugal rotational speed are shown in Fig. 6. The moisture content decreased with the centrifugation time at each centrifugal rotational speed, and the larger centrifugal rotational speed would cause lower moisture content. Moreover, the decreasing trend of moisture content was significant in the first 10 min, and it decreased to 87.47% at 2,000 rpm and 84.21% at 4,000 rpm,

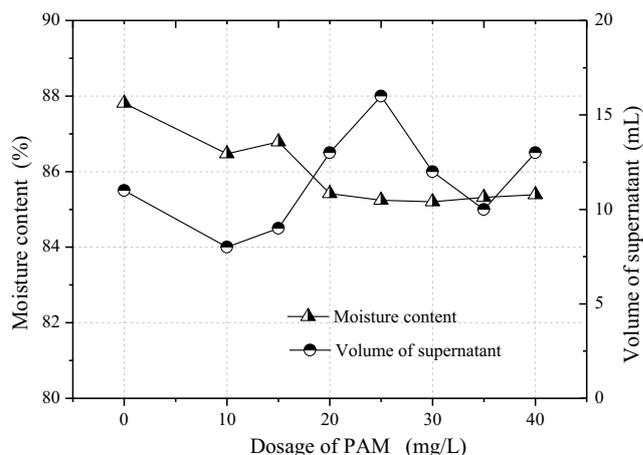


Fig. 5. Effect of the PAM dosage on the sludge treatment.

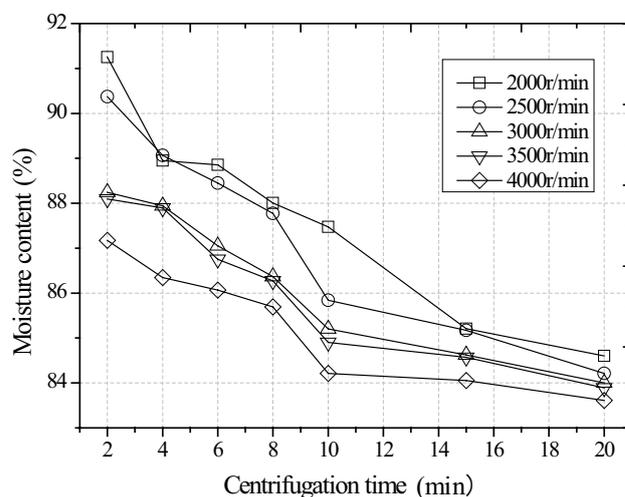


Fig. 6. Relations of moisture content with centrifugation time.

respectively. However, the decreasing trend was slow in the next 10 min, especially at the larger centrifugal rotational speed, so the suitable centrifugation time should be longer than 10 min. Additionally, the moisture content of sludge decreased from 95.24% to 84.6%, 84.21%, 84%, 83.89% and 83.61% in the 20 min, respectively. When the centrifugal rotational speed changed from 2,500 to 3,000 rpm, the moisture content had a larger decrease, so it was reasonable to control the centrifugal rotational speed at around 3,000 rpm.

Due to the centrifugation time was limit in the experiment and no doubt the longer centrifugation time would cause a lower moisture content, but the trend in Fig. 6 indicates that the moisture content cannot decrease to below 80%, and the 80% moisture content was still high for sludge packaging and transportation, because the ideal moisture content was about 60%, so the sludge drying experiment should be carried out to study the relations of moisture content and drying time at a suitable drying temperature. In this experiment, the sludge which had been dewatered by centrifuge was placed in three temperature fixed drying box (80°C,

Table 7
Relations of drying time and moisture content

Drying time, min	Moisture content, %		
	80°C	100°C	120°C
0	80.56	79.55	80.83
10	78.28	77.61	77.03
20	76.20	74.88	71.55
30	73.78	70.72	63.08
40	70.85	66.35	50.59
50	67.19	55.92	24.13

100°C and 120°C, respectively), and the relations of moisture content and drying time are shown in Table 7.

The data in Table 7 indicated that the higher drying temperature would cause a quicker drying rate. The moisture content of sludge decreased from 80.56% to 67.19% in 50 min at 80°C, but it only need about 40 and 20 min to attain the same moisture content at 100°C and 120°C, respectively. However, the higher drying temperature did not mean the better, because the mercury in the sludge was not stable, the higher drying temperature would cause the mercury volatilization. The experimental results had indicated the critical temperature to keep the mercury stable should below 80°C, so the drying temperature should not exceed this value. When the drying temperature was 80°C, it took about 70 min to decrease the moisture content of sludge from 80.56% to 60%. However, if the drying device was sealed and the volatilized mercury can be treated effectively, a higher drying temperature can be considered.

4. Conclusion

Due to the mercury-containing gas field was scattered and the sludge throughput of single gas field was small, the centralized treatment of sludge after the sludge reduction was a more reasonable method. In order to acquire the characteristics of gas field mercury-containing sludge, and provide a technical support for the sludge reduction, detailed experiments were carried out, and the main conclusion included: (1) The water treatment concentrated the mercury in the sludge, the total mercury content of raw sludge can attain 585 mg/kg, and the divalent mercury was the main existence form, and a small amount of highly toxic methyl mercury was existed as also, but no ethyl mercury and alkyl mercury were detected. Moreover, the existence form of mercury in the sludge was closely related to the water treatment method. (2) The moisture content of the sludge was above 99%, and its sedimentation performance was poor, which caused difficult to the sludge reduction and transportation. The percentage of sludge particle size below 100 µm accounted for 100%, and the little particle size was the main reason for its poor sedimentation performance. (3) The relations of moisture content and mercury content at different temperature indicated that the stability of mercury was dependent on the drying temperature, and the mercury can keep stable once the drying temperature was below 80°C. (4) The PAC and the APAM with the molecular

weight 1,800 million were the suitable organic coagulant and inorganic coagulant to condition the sludge, and the optimal dosages of them were 150 and 25 mg/L, respectively, because the joint use of them produced a larger volume of supernatant and lower moisture content. (5) The moisture content decreased with the centrifugation time at each centrifugal rotational speed, and the larger centrifugal rotational speed would cause lower moisture content, and the suitable centrifugal rotational speed was 3,000 r/min, but it can only decrease the moisture content to around 80%. (6) The higher drying temperature would cause a quicker drying rate, but the higher drying temperature did not mean the better. The sludge drying should consider the stability of mercury, and it is reasonable to keep the drying temperature below 80°C if there were no additional technical measures to control the instable mercury.

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