

Study on the experiment of processing oily wastewater by bubble column flotation

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Abstract: In the petroleum industry, a large quantity of oil-contained wastewater is produced every day. This kind of wastewater normally contains oil droplet from 100 to 1000ppm, which is characterized by tiny oil droplets, dispersion, stable, uneasy to reunite float. The conventional methods such as demulsification agent and heat treatment will undoubtedly increase the cost of production. Bubble column flotation was attracted more attention by oil companies for its characters of cost-effective, simple structure, high efficiency and no secondary pollution, etc. Oil removal efficiency is affected by many parameters. In this paper, experimental study mainly attempts to find the key factors affecting the separation efficiency and utilized to improve bubble column structure and increase the separation rate. Two new physical quantities, total ventilation per unit volume of wastewater and aeration intensity were defined. Two key parameters about the degree of salinity and aeration intensity were studied separately.

Introduction

During the progress of crude oil production, processing, and consuming, a large quantity of oil-polluted wastewater is produced. Oil in water forms the emulsion. Emulsions are suspensions of droplets, greater than 0.1 μ m, consisting of two completely immiscible liquids, one of which is dispersed throughout the other. According to existence of oil droplets in water, emulsions are divided into four categories which are shown in Table 1.

Table 1

Emulsion types according to existence of oil in water

Free oil	Mechanically emulsified oil	Chemically emulsified oil	Dissolved oil
Non-miscible with water, rapidly rises to water surfaces. Forming a film or large droplets.	Present in water due to high shear by pump, stabilized by electrical charges.	Miscible with water, stabilized by surfactants, having hydrophobic end.	Water-soluble oil, water is translucent and transparent. Removal by filtration, gravity settling is impossible.
>100 μ m	20-100 μ m	<20 μ m	<5 μ m
Macro emulsion	Micro emulsion	Micro emulsion	Mini emulsion

In horizontal-flow sedimentation tank, the size of oil droplets that can be removed effectively is

above 50 μm . But when there is stream-flow in the processing device, droplets about 30 μm are difficult floating up. Especially highly dispersed state droplets between 1 μm to 30 μm or so, even keep in a static state, due to the Brown movement, cannot go up. Some are extremely difficult to remove as the diameter below 20 μm .

The principle of air flotation method is to produce a large amount of micro bubbles in the water, so three-phase mixture (oil/gas/water) is formed. Under the joint action of interfacial tension, flotation and hydrostatic pressure difference, micro bubbles adhere to the oil droplet's interface. The integrated density of oil droplets is decreased after adhesion so they can float up rapidly to the water surface. That's the process of oil/water separation.

Micro bubble diameter in 30-50 μm called conventional flotation, without agents it can only remove the oil droplets above 30 μm . After adding flocculants, more than 10 μm oil drops can be removed. With the help of chemical demulsification, most of the oil droplets could be separated from oily water. Due to micro size and complex surface properties, emulsified oil has become the difficulty in the wastewater treatment.

Generally, the oil concentration in wastewater is 100-1000ppm. Oil droplets are dispersed in the water to form very stable emulsions, which increase the difficulty of the separation. Contrast with fine filtration, chemical demulsification, reverse osmosis membrane, biological treatment, active carbon adsorption, electrochemical etc. method on the treatment of oil-bearing wastewater, air flotation technology has obvious advantages in cost control and processing efficiency. The free floating velocity is no more than 0.001mm/s as oil droplets with diameter of 1.5 μm . While the droplets adhered on the air bubble, the floating velocity is 0.9mm/s, 900 times are increased [1, 2].

There are many factors that affect the air flotation efficiency, such as micro bubble size, gas phase volume fraction in the water, pressure, temperature, pH value, the degree of sewage water salinity and so on [3]. According to the condition of laboratory, we can change any one of the factors to test the influence of the oil removal. In this paper, the salinity, aeration intensity will be taken as the key factor, through the experimental comparison, trying to find its effect on the oil removal efficiency of the general rule.

Experiment program

The main equipment of this experiment is a total height of 2300mm, 50mm inner diameter sealed Plexiglas column, which can withstand the working pressure of 0.5MPa. The column was placed vertically as shown in Fig. 1. The total wastewater inlet is arranged at the position of 1600mm high from the bottom. Air floating treated water discharges from outlet A which is designed 100mm high above column's bottom. Outlet B as an oil draining port is located in 2000mm position near the column top. Micro bubble generator is directly mounted on the column's bottom. A pressure sensor and an exhaust device are arranged on the top cover of the bubble column. Multiple sampling ports have been settled at appropriate places.

By manually adjusting the top exhaust valve opening, internal pressure level can be controlled in a certain range, ensure the top of gas space existing 220mm at least.

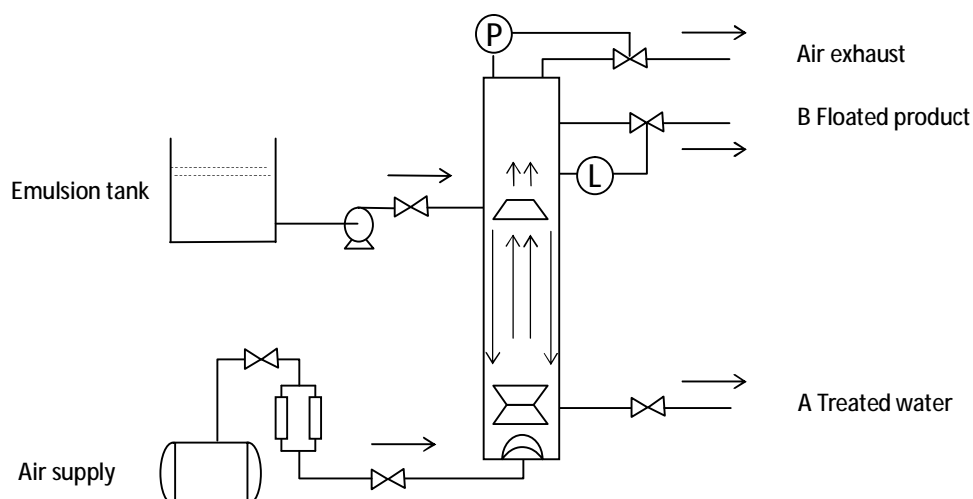


Fig.1 bubble column Schematic diagram

Static and dynamic flotation experiments have been carried out separately. Static flotation experiments are treated by emulsified liquid filling, air floating, sampling, draining and pipeline cleaning, and then repeat the test. Continuous filling and keeping a balance between imports and exports are the key characters of dynamic flotation, which is different from static. Bubble generating device was made up of metal filter cloth or microporous membrane. For the safety of experiment, cooking oil was used to produce emulsions. The parameters of emulsion preparation are shown in Table 2. The prepared emulsions was produced 4L every time for one experiment and can keep stable for 3 hours at least. The key operating conditions employed in this study are listed in Table 3.

Table 2

Emulsion preparing conditions

Parameters	Conditions	Parameters	Conditions
Oil type	Olive oil	Water volume(ml)	4000
Temperature(°C)	20	Agent volume(ml)	1.5
Oil density(g/cm ³)	0.91	Homogenizer	Ultra turrax
Viscosity(mPa·s)	11-13	Rotate speed(rpm)	13500-24000
Emulsify agent	Tween-20	Mixing time(min)	2-5

Table 3

Operating conditions

Parameters	Conditions	Parameters	Conditions
Operate temp(°C)	20	Column effective volume(L)	3.9
Air supply	Pressed air	Feeding rate(L/Min)	0-5
Air pressure(MPa)	0.1-0.5	Inspection instrument	Malvin 2ES3600
Bubble size(mm)	0.8-2	Analysis software	Mastersize 2000

Result and Discussion

The effect of bubble column flotation is influenced by many factors. In this paper, the degree of salinity and aeration intensity was selected as the key parameters which influenced oil removal efficiency. Thus relevant experiments were carried out.

Effect of salinity

Mineralization degree is an important index for the evaluation of water chemical composition, which is used to evaluate the total salt content in water. Oilfield production salinity is from 1×10^3 mg/L for minimum to a maximum of 1.4×10^5 mg/L. In general indoor experiments, emulsions were prepared by distilled water or tap water, the salinity was easily to be ignored. This paper specially designed the flotation experiments for different salt emulsions, therefore to find the law affected by salinity.

Sodium chloride was used as mineral substance in flotation experiments. From 5×10^3 to 20×10^3 ppm, five different kinds of salt emulsions were produced. Six flotation experiments were carried out including one without salt and kept the other parameters constant. Experimental results are shown in Fig. 2.

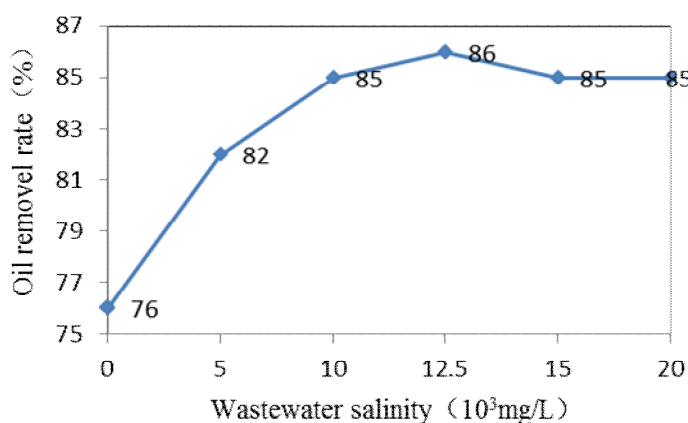


Fig. 2 Effect of salinity

Through the contrast experiments, after adding mineral salt, the maximum oil removal efficiency was obviously increased over 10%. The rate was initially rose up with the increase of the salinity. But over 10×10^3 mg/L, the removal rate tended to be stable. So there is an inflection point of salinity to achieve the maximum removal rate.

The reason analysis: First, the addition of electrolytes can affect the bubbles formation and growth. Bubble diameter was reduced due to the existence of electrolyte in the liquid. Second, when the bubble surface adsorbed electrolyte ions, the coalescence of bubbles were restrained [4]. Last, the thickness of oil-water interface layer can be compressed by salt electrolyte, bubble surface energy was increased and the adsorption capacity was improved too. But when the salt content is above 1.25%, the flotation efficiency tends to be stable. More salinity may restrict the gas holdup, which could be the reason.

Effect of aeration intensity

In flotation process, the air bubbles generating is the key factor. Optimized nozzle structure and proper gas flow rate joined together to produce effective bubbles, which will influence the

efficiency of oil removal. In this experimental system, a gas flowmeter can measure air flow which filled in flotation column. Sparger was made up of multilayer metal filter cloth. Bubble size is controlled by changing the overlap layer. In this paper, bubbles size was mostly from 0.8mm to 2mm. Further smaller bubble can be produced, but higher pressure of air supply is needed. If aeration cannot reach certain intensity, the oil removal will not be fully processed by air flotation.

This paper introduced the concept of total ventilation per unit volume of wastewater and calculation formula is given by Eq. 1:

$$h = \frac{Q_g \times t}{V_l} \times 100\% \quad (1)$$

In Eq. 1, the dimensionless quantity η is for total ventilation per unit volume of wastewater, Q_g for air flowrate, t for flotation duration time, V_l for the total volume of treated wastewater. In static air flotation experiments, the V_l is equal to the effective volume of V_1 (3.9L), and in dynamic experiments, V_l is equal to the total amount of treated wastewater. For The experimental column in this paper, by the volume of liquid tank and pump flow limit, V_l is approximately equal to 2 times the air flotation column effective capacity (7.8L).

In order to measure the strength of air flotation treatment in unit time, the ratio of η and t is defined as K , the symbol of aeration intensity. The correlation was shown in Eq. 2:

$$k = \frac{h}{t} = \frac{Q_g}{V_l} \times 100\% \quad (2)$$

Static and dynamic experiments were carried out separately to study the influence on oil remove efficiency by K . 3.9L oily wastewater was treated every time in static experiment, by constant air flowrate into column, repeat 5 times to take samples from outlet A every 1.5 minutes. In dynamic experiment, flotation column was loaded fully by oily wastewater in advance, open the air supply valve to start flotation, run the pump to feed column with wastewater, total amount of wastewater is 7.8L. Outlet A and outlet B are opened at same time to keep the liquid level in column stable, flowrate of outlet B is 30% of inlet flow. Experimental results are shown in Fig. 3 and Fig. 4.

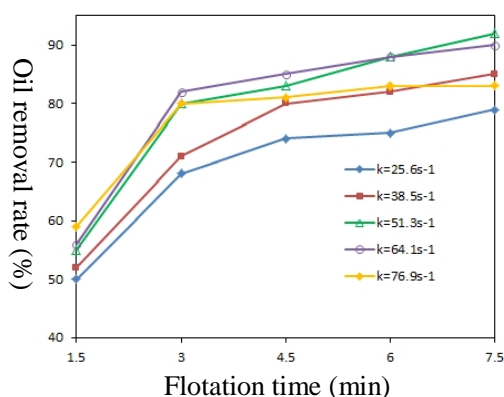


Fig. 3 Effect of aeration intensity in static tests

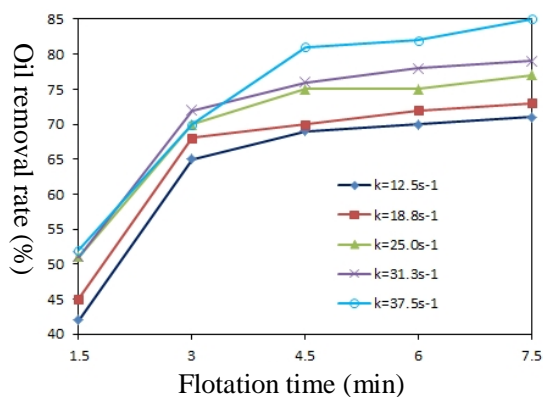


Fig. 4 Effect of aeration intensity in dynamic tests

Static experimental results were illustrated in Fig. 3, all oil removal rate trend line increased with flotation time. Oil removal rate could be infinitely close to 100% as long as the duration is enough, which means satisfactory water quality always can be gotten. The curve in Fig. 3 also shows that, oil removal rate increased obviously at begin, and gradually reached a slowly growth stage, this tendency increased with the K value. As $k=25.6$ and 38.5 , the time of slowly growth stage was about 4.5 minutes. As $K=51.3$ to 76.9 , the time was 3 minutes, earlier than the other two. From the experimental curve, when $K=38.5$ and 51.3 , Oil removal rate can be maintained at a high growth rate in the later period. As the value of aeration intensity K was exceed 51.3 , oil removal rate increased slowly, and even smaller than $K=25.6$ and 38.5 at last. The reason is: with the aeration intensity increase, the number of micro bubbles increased to a limit, and the diameter of the bubble began to increase. The transition from homogeneous flow to turbulent had a negative effect on the air flotation too.

The dynamic experimental process shown in Fig. 4 was different from static, but it can be concluded that the value of the two different experiments was correlative. Compared the K values of two different experiments, the growth curve of oil removal rate trend to be the same as K value was close. Fig. 4 demonstrated that nearly all curves grew steadily after 3 minutes, which is related to the dynamic air flotation. Appropriate K values (such as $K=37.5$) can be guaranteed in both dynamic and static experiments, which can ensure that the oil removal rate has an ideal increasing with time.

Conclusions

In order to optimize the design of bubble column flotation, working parameters were studied by experiments separately. Two new physical quantities, total ventilation per unit volume of wastewater and aeration intensity are defined. The two key parameters about the degree of salinity and aeration intensity were studied separately. The following conclusions stand out:

- 1 The salinity of the wastewater has a significant effect on the oil removal rate, and there is a turning point in the relationship curve between the degree of salinity and the oil removal rate;
- 1 The proper aeration intensity can ensure the rapid growth of the oil removal rate in a short time, and keeping a reasonable growth rate during the post flotation process;
- 1 Excessive aeration intensity is bad for flotation;
- 1 Data shows that the oil removal rate curves are basically close in static and dynamic experiments for same K , and the K value is an important physical quantity of the air flotation process.

There are many factors that affect the efficiency of oil removal by bubble column. In practice, to achieve the best oil removal effect, synthesize variety of factors and find the key parameters which can be operated in design.

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