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Effects of EOR chemicals and superficial gas velocity on bubble size and gas holdup of a bubble column

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Abstract. Chemical Enhanced Oil Recovery (EOR) can boost oil extraction in offshore operations, however one of the main concerns regarding its application is how the efficiency of flotation units for treating produced water is affected. The present work thus focuses on investigating the impact of EOR chemicals on the physical properties of EOR effluents and how this can affect flotation performance parameters such as bubble size and gas holdup. Design of experiments has been used to assess the influence of polymer, surfactant and sodium chloride concentrations on bubble size and gas holdup of a laboratorial bubble column. The influence of superficial gas velocity has also been assessed together with chemicals concentrations, yet at low levels in order to avoid clusters, swarms and foam. The characterization of the synthetic effluent containing polymer, surfactant and sodium chloride has indicated that the fluid behaves as a non-Newtonian fluid, what makes separation processes in flotation cells challenging. Results showed that polymer concentration of 2000 mg/L can lead to significant increases in fluid viscosity, promote a growth of more than 40% in bubble size and only increases gas holdup when surfactant is present at high concentration. Therefore, polymers are expected to be detrimental to produced water treatment. Surfactants decrease both fluid surface tension and bubble size, increasing gas holdup. For the range studied, superficial gas velocity favors gas holdup and sodium chloride concentration seems to weakly influence bubble size and gas holdup. This work highlights the fact that changes in physical properties of produced water do modify bubble size distribution and gas holdup and this must therefore be taken into account when flotation-like systems are designed to deal with EOR effluents.

1 Introduction

Enhanced Oil Recovery (EOR) encompasses several methods for increasing the production of petroleum reservoirs [1]. According to Thomas [2], EOR methods consist of chemical, electrical, miscible, steam and thermal techniques. These methods have proven to boost oil recovery from mature reservoirs when conventional methods (water flooding) no longer work [3]. Chemical enhanced oil recovery comprehends water injection with different additives to enhance the removal of oil from the reservoir. Among the chemical methods, the main techniques are polymer flooding and Surfactant/Polymer (SP) flooding [4].

Polymer flooding mechanism operates by decreasing the mobility of the water phase, reducing the incidence of fingering effects [5]. Besides, polymers are responsible for increasing the viscosity of the water and, therefore, improving the efficiency of oil sweeping [6].

Surfactants have the function of reducing the interfacial tension between water and oil phases, enhancing the mobility of the oil, as well as modifying the wettability of the porous rock [7]. The combined injection of surfactants and polymers into the reservoir (SP flooding) may increase sweep efficiency [8].

Due to this ability to improve the oil/water mobility ratio, in the past few years, chemical methods for EOR have been implemented at various onshore sites [9]. Ma *et al.* [10] reported the success of the polymer flooding technique that enhanced oil recovery by more than 12% in the oil industry.

Such results suggest that applying chemical EOR methods in the offshore environment would increase oil recovery. On the other hand, some researchers point out obstacles to the effective use of EOR methods in offshore oilfields. One such restriction is the inability of available technologies to process the produced water containing EOR [11–13].

The produced water system in offshore installations is usually equipped with skim tanks, gas flotation/degassers and hydrocyclones [14]. In the case of EOR effluents,

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Wang *et al.* [15] reported that the use of hydrocyclones was an inefficient method for treating the water produced by polymer flooding.

Although Chen *et al.* [16] confirm the benefits of the polymer flooding in offshore operations, they have also pointed out the difficulty of treating produced water for reinjection or disposal. As polymers tend to concentrate in such streams, oil/water separation becomes more difficult and larger separators are required to increase residence time [17]. Furthermore, if the produced water is meant to be discharged into the environment, then the oil content of the effluent should be within the established offshore environmental regulations.

Flotation is the last treatment stage in offshore oil production units and is responsible for suiting water discharge regulatory specifications [18]. Hence, maintaining flotation units at a high level of efficiency is essential to protect the environment and make oil production sustainable.

It has been reported though that EOR chemicals may decrease flotation efficiency. According to Zheng *et al.* [19], the presence of surfactants and polymers can profoundly affect the performance of flotation units. Argillier *et al.* [12] have stated that these products may adversely influence bubble size distribution. Polymers may cause oil–water emulsion stabilization and affect the performance of the flotation unit [13]. The presence of surfactants in EOR produced water can also contribute for oil–water emulsion stabilization. However, on the other hand, surfactants can inhibit bubble coalescence [20], affecting positively the gas holdup of the flotation units.

Flotation is a gravitational separation method that employs gas bubbles in such a way that they attach to oil droplets, enhancing their buoyancy and allowing them to be skimmed from the surface of produced water [21]. To accomplish this goal efficiently, Moosai and Dawe [22] point out the vital importance of bubble size in gas flotation. Robinson [23] clarifies that, according to Stokes' Law, the smaller the bubble diameter in produced water, the smaller the bubble rises and therefore the more frequently bubbles collide with the oil droplets, increasing flotation efficiency.

Saththasivam *et al.* [24] confirmed the importance of reducing gas bubble size to increase flotation efficiency, finding that size reduction makes collision more likely. In their water/air/*n*-dodecane system, Oliveira *et al.* [25] increased the water salinity in order to decrease the size of air bubbles. Their results showed that the probability of collision between bubbles and *n*-dodecane increased, thus the flotation efficiency improved. Rajak *et al.* [26] have indeed confirmed the abovementioned relations between bubble size and water salinity. They have enhanced flotation efficiency to separate oil from oil-in-water emulsions by increasing water salinity.

The above-mentioned makes it clear how important is to measure the effects of EOR chemicals (polymer and surfactant) and produced water composition (salinity) on bubble size distribution in order to understand and improve gas flotation when processing EOR effluents.

Bubble columns, often used to perform chemical reactions and for wastewater treatment [27], have also been used to assess the hydrodynamics of multiphase flows

because of their simple geometry and lack of moving parts [28–31]. These advantages allow researchers to easily measure the size of gas bubbles using image acquisition and data analysis [32–34]. Another important factor that can be measured is the gas holdup [35–38]. Unlike bubble size measurement, which is a local measurement, gas holdup is a global measurement and can be used to calculate residence time. It can also be used to assess interfacial area, mass and heat transfer between the phases involved in the multiphase flow [39, 40].

Li *et al.* [38] studied the effect of process variables on gas holdup of a cyclone-static micro-bubble flotation column for treatment of oily water. According to their results, varying holdup from 9.7% to 10.5%, improved oil removal from 95.2% to 99%. Therefore, a marginal improvement in holdup can generate considerable increases in equipment performance, while the opposite is also true: a reduction in holdup can significantly affect the efficiency of flotation units.

The purpose of this work is thus to evaluate the influence of polymers, surfactants and produced water salinity on gas bubble size and holdup. A laboratory bubble column has been used and the influence of superficial gas velocity has also been assessed. Factorial design technique has been applied to identify the main factors and interactions that effectively impact bubble size and gas holdup. Results help to characterize physical properties of EOR effluents and establish how bubble size and gas holdup are affected by EOR chemicals and superficial gas velocities.

2 Materials and methods

2.1 Synthetic produced water

Synthetic produced water has been prepared by adding sodium chloride, polymer and surfactant at different concentrations. As the focus of the present work is the gas–water interface, no oil has been added to the synthetic produced water.

Based on the works of Liu [41], Zhao [42], Zhao *et al.* [43], Flaaten [44], Barnes *et al.* [45], and Solairaj *et al.* [46] the adopted range for salinity in the experimental set was from 60 to 200 g/L of sodium chloride (NaCl).

The polymer used is a copolymer of acrylamide and 2-Acrylamido-2-Methylpropane Sulfonate (AMPS). Its molar mass is about 12×10^6 g/mol [47]. It has been supplied by *SNF* and is used in real EOR applications. According to Liu *et al.* [48] and [49], polymer concentration in EOR produced water varies from 100 to 2000 mg/L and this is the range investigated in the present work.

The surfactant used in the synthetic produced water was Sodium Dodecylbenzenesulfonate (SDBS), supplied by *LABSYNTH*. According to Olajire [1] and Miao *et al.* [5] such surfactant has been used in advanced EOR systems and is therefore of interest for industrial application. In accordance to the work of Liu *et al.* [49], the surfactant concentration lies in the range of 0–100 mg/L and so it has been varied in the present work.

Brine has been prepared by dissolving sodium chloride in tap water at the desired concentration. At the same time,

a polymer stock solution has been prepared by gently shaking the product in tap water till it dissolved. For polymer–injection-like effluents, volumes of the brine and polymer stock solution were mixed. For surfactant–polymer–injection-like effluents, surfactant was added to the mixture of brine and polymer stock solution, according to the desired concentration.

2.1.1 Physical properties of synthetic produced water

Density, viscosity and surface tension measurements have been performed to characterize the different compositions of synthetic produced water for the tests in the bubble column. All analyses have been performed at 20 °C, since tests in the column have also been conducted at this temperature.

Density analyses have been performed in an Anton Paar DMA 4500 digital density meter based on ASTM D4052-09.

Viscosity analyses have been carried out in an Anton Paar Physica MCR 501 rheometer based on DIN 13343, DIN 1342, DIN 3219, DIN 51810, DIN 53019, DIN 54458, ISO 3219, ISO 6721-1 and USP 912 international standards.

Surface tension analyses have been performed on a Teclis Tracker S. tensiometer using the procedures described in ISO 304/1985, ISO 6889/1986, and ASTM D1331-14 international standards.

2.2 Bubble column

The bubble column used for the experiments is 2 m high and has 5.4 cm of internal diameter. At the bottom of the column, air is injected through a metal distributor with 57 holes of 0.5 mm in diameter, evenly spaced by 6 mm.

The column was operated at 20 °C in semi-batch mode. It has been filled with synthetic produced water and air bubbles have been injected at different superficial gas velocities.

Preliminary experiments in the lab column have shown that the presence of polymers accelerated the transition to the heterogeneous flow regime, as also noted by Ruzicka *et al.* [50], which makes it more difficult to evaluate bubble sizes. Therefore, following Bordel *et al.* [51], the present work has then been limited to the bubbly flow regime, where the formation of clusters and swarms is low. Besides, surfactants are expected to form foam which also disturbs the bubble column operation and its data acquisition. Air superficial velocities have been set between 0.25 and 0.5 cm/s in order to avoid clusters, swarms and foam.

Figure 1 shows the schematic of the bubble column in which the tests were performed. Air flow has been controlled using needle valves and has been measured using a Coriolis meter – FQIT (Micromotion, model CMF10M, with flow transmitter Model 2700). Air-supply pressure has been measured with an absolute pressure gauge in the bottom of the column (Yokogawa, model EJA510E). Image acquisition for bubble size assessment has been carried out at the point A (Fig. 1). In order to calculate gas holdup, two Pressure Indicators (PIT) have been installed. The first one

has been placed 260 mm from the air distributor and the second, from 1740 mm, as can be seen in Figure 1.

2.3 Assessing bubble size

The Sauter mean diameter is commonly used in bubble column and flotation studies. According to Pérez-Garibay *et al.* [52], its use is justified by the fact that the flotation rate is easily related to the surface area of the bubbles.

The mean Sauter diameter of the bubbles has been obtained by image acquisition and analysis at the point A (Fig. 1). An IDT NanoSense MKIII high speed camera with Nikon Nikkor 60 mm F2.8 lens have been used. At each section photos were taken using three different exposure times (150, 170 and 200 μs). For each exposure time, 200 images were obtained and have been processed using the Shadow sizing module tool of the *Dantec DynamicStudio*® software.

2.4 Assessing gas holdup

According to Li *et al.* [38], equation (1) can be used to calculate the gas holdup ε:

$$\varepsilon = 1 - \left(\frac{\Delta P}{\rho_l \times g \times h} \right), \tag{1}$$

where ΔP is the differential pressure measured between the PITs in the column, ρ_l is the specific mass of the liquid phase, g is gravity acceleration and h is the distance between pressure measurements.

2.5 Design of experiment

In order to assess the influence of salinity, polymer concentration, surfactant concentration and superficial gas velocity in bubble size and gas holdup, the 2^k complete factorial design has been used. Therefore, each of the aforementioned variables have been evaluated at two values, these being the extreme values of interest. These values have been defined above and are now summarized in Table 1. In coded variables, the lowest value is associated to −1 and the highest to +1, as also shown in Table 1. A generic variable φ is related to its coded counterpart ϕ through:

$$\emptyset = \frac{\varphi - (\varphi_{\text{high}} + \varphi_{\text{low}})/2}{(\varphi_{\text{high}} - \varphi_{\text{low}})/2}. \tag{2}$$

Bubble diameter and gas holdup assays have been carried out in duplicates and the average of these experimental values have been taken. Additionally, for holdup assessment, two more experiments have been performed at the central point of experimental design, that is, with 130 g/L of NaCl, 1050 mg/L of polymer, 50 mg/L of surfactant and superficial gas velocity of 0.375 cm/s.

Analysis of Variance (ANOVA) has been used to identify the independent variables and their interactions that were statistically significant in affecting bubble size and gas holdup. The statistical evaluation has been carried out with the *MINITAB*® statistical software.
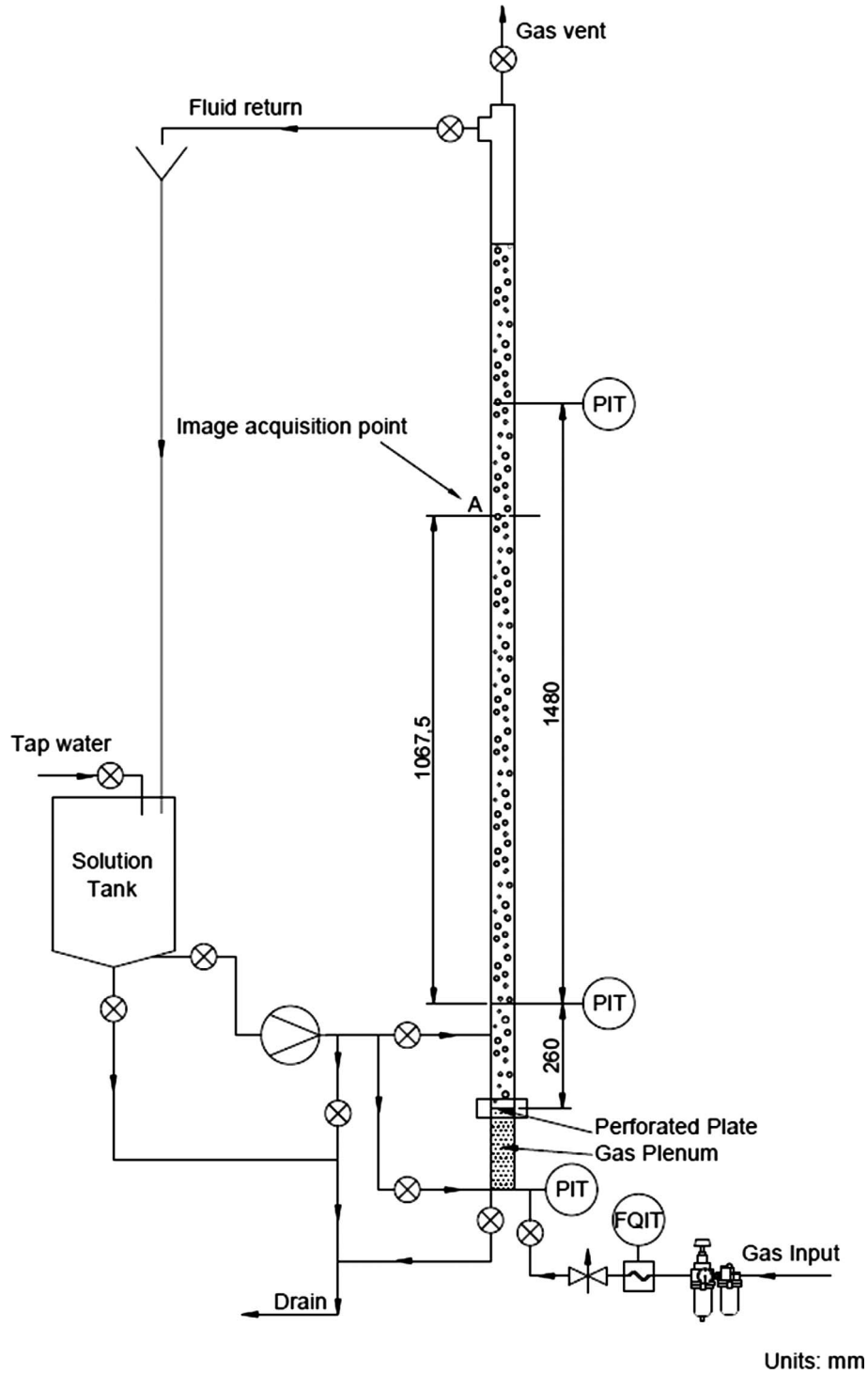
Fig. 1. Experimental setup to assess bubble size and gas holdup.

3 Results and discussion

Table 2 shows how EOR chemicals affect physical properties of the synthetic produced water.

Apparent viscosity μ_{app} has been evaluated for a general power law fluid and can be expressed as in equation (3):

$$\mu_{app} = K\gamma^{n-1}, \tag{3}$$

where K stands for the consistency index in dynamic viscosity units, γ is the shear rate and n is a non-dimensional parameter known as the flow behavior index, since its value indicates whether the flow is Newtonian ($n = 1$),

Table 1. 2^4 complete factorial design for bubble diameter and gas holdup.

Factors	Low level (-1)	High level ($+1$)
Salinity (NaCl) (g/L)	60	200
Polymer concentration (mg/L)	100	2000
Surfactant concentration (mg/L)	0	100
Superficial gas velocity (cm/s)	0.25	0.50

Table 2. Physical properties of synthetic produced water.

NaCl (g/L)	Polymer (mg/L)	Surfactant (mg/L)	Specific mass (kg/m^3)	Surface tension (dina/cm)	Consistency index, K (mPa s^n)	Flow behavior index, n
60	100	0	1041.6	70.8	2.04	0.915
60	2000	0	1042.3	72.8	22.87	0.779
200	100	0	1126.7	72.8	1.79	0.978
200	2000	0	1125.3	74.2	24.05	0.791
60	100	100	1039.7	33.5	0.98	1.032
60	2000	100	1040.8	30.7	24.64	0.761
200	100	100	1124.8	42.5	4.19	0.831
200	2000	100	1123.2	42.3	21.66	0.790
130	1050	50	1083.4	35.7	9.33	0.827

non-Newtonian pseudoplastic ($n < 1$) or non-Newtonian dilatant ($n > 1$).

It can be seen in Table 2 that high concentration of polymer increases viscosity significantly and is the main factor responsible for a non-Newtonian pseudoplastic fluid behavior. Density is mainly affected by high concentrations of NaCl and surface tension is drastically reduced with high concentrations of surfactant. Such evidences support how challenging can be the separation processes in flotation cells.

Table 3 shows how EOR chemicals and superficial gas velocity affect bubble size (Sauter mean diameter) and gas holdup. It can be seen that as superficial gas velocity increases, both bubble size and gas holdup increase; an increase in polymer concentration increases bubble size, but gas holdup decreases when surfactant is not present; the presence of surfactant decreases bubble size, and increases gas holdup even at high polymer concentrations; salinity seems to not have a significant effect on bubble size and gas hold up.

In order to quantify how bubble size and gas holdup are affected by superficial gas velocity and EOR chemicals, regression analysis and ANOVA combined with F-test have been carried out. This also allows evaluation of non-significant terms ($p > 0.05$).

A first-order model with interactions has been fitted for bubble size and a model reduction was performed to neglect the non-significant factors and interactions. All interactions and salinity have been considered non-significant, being excluded from the model. The corresponding ANOVA data is presented in Table 4, confirming previous analysis of Table 3: polymer concentration, surfactant concentration,

and superficial gas velocity have strong effect on bubble diameter, as confirmed by their respective null p-values and by Figure 2, where the main effects of the variables on bubble size are highlighted.

It can be seen in Figure 2 that increasing only polymer concentration from 100 to 2000 mg/L can lead to a growth of more than 40% in bubble diameter; that 100 mg/L of surfactant only can decrease bubble size by 20%; that only doubling superficial gas velocity increases bubble diameter by less than 10%; and that salinity only does not play a significant role in the range studied.

The model obtained for bubble Sauter mean diameter (in mm) is presented in equation (4):

$$d = 5.82 + 0.98P - 0.53S + 0.24V, \qquad (4)$$

where d stands for bubble Sauter mean diameter and P, S, and V correspond to the values of polymer concentration, surfactant concentration and superficial gas velocity, respectively, in coded variables (from -1 to $+1$). Since a reasonable fit between predicted and experimental bubble diameter has been obtained ($R^2 = 0.9537$/adjusted $R^2 = 0.9422$), one can assume that the developed model predicts experimental data satisfactorily.

A first-order model with interactions has also been fit to relate salinity, polymer concentration, surfactant concentration and superficial gas velocity with gas holdup. Then, a model reduction has been performed to neglect the non-significant terms. The corresponding ANOVA data is presented in Table 5.

It can be seen in Table 5 that the parameters that significantly affect gas holdup are superficial gas velocity

Table 3. Results of factorial design for bubble diameter and gas holdup.

NaCl (g/L)	Polymer (mg/L)	Surfactant (mg/L)	Superficial gas velocity (cm/s)	Bubble diameter (mm)	Holdup (%)
60	100	0	0.250	4.8	1.3
60	100	0	0.500	5.2	2.4
60	2000	0	0.250	7.3	1.1
60	2000	0	0.500	7.9	1.6
200	100	0	0.250	5.3	1.0
200	100	0	0.500	5.7	2.1
200	2000	0	0.250	6.8	1.1
200	2000	0	0.500	7.8	1.9
60	100	100	0.250	4.3	1.2
60	100	100	0.500	4.4	2.5
60	2000	100	0.250	5.7	1.8
60	2000	100	0.500	6.4	2.9
200	100	100	0.250	4.5	1.2
200	100	100	0.500	4.5	2.5
200	2000	100	0.250	5.9	1.5
200	2000	100	0.500	6.6	2.7
130	1050	50	0.375	–	2.2

Table 4. ANOVA of the first-order model obtained for bubble size data.

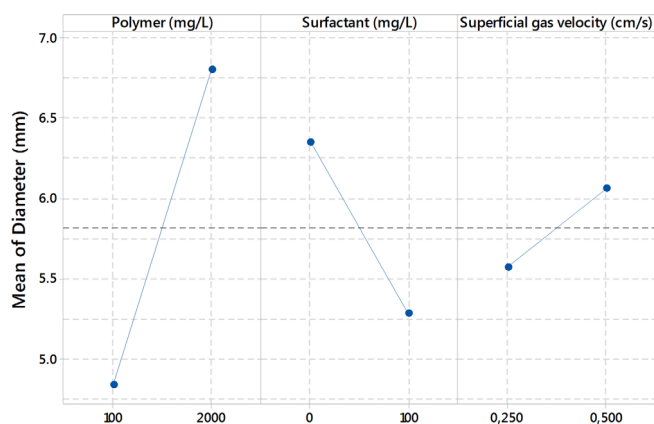| Source	df	Sum of squares	Mean square	F-value	p-value
Model	3	20.8719	6.9573	82.46	0.000
Linear	3	20.8719	6.9573	82.46	0.000
Polymer	1	15.4056	15.4056	182.59	0.000
Velocity	1	0.9506	0.9506	11.27	0.006
Surfactant	1	4.5156	4.5156	53.52	0.000
Error	12	1.0125	0.0844		
Total	15	21.8844			

ANOVA, Analysis of Variance.

Fig. 2. Individual main effects for bubble diameter.

and surfactant concentration ($p < 0.05$). Polymer concentration does not affect directly holdup, as a main factor, however its influence on this parameter occurs through its interaction with surfactant.
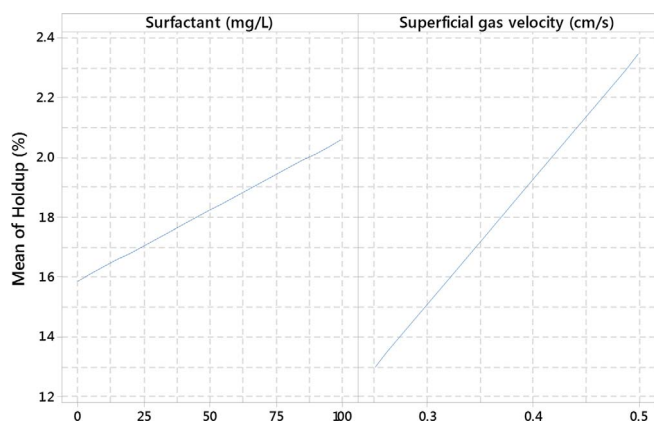
Figure 3 confirms the importance of superficial gas velocity and surfactant concentration on holdup. It can be seen that only the addition of only 100 mg/L of surfactant causes an increase in holdup that exceeds 25%. It can also be noted that only doubling superficial gas velocity produces an increase of roughly 70% in holdup.

Figure 4 shows the influence of variable interactions on holdup. It can be seen that polymer–surfactant interaction affects holdup differently, depending on surfactant concentration: when the surfactant concentration is low, an increase in polymer concentration decreases holdup; at a concentration of 50 mg/L of surfactant, holdup is not

Table 5. ANOVA of the first-order model obtained for gas holdup data.

Source	df	Sum of squares	Mean square	F-value	p-value
Model	3	5.7350	1.91167	41.73	0.000
Linear	2	5.3125	2.65625	57.98	0.000
Surfactant	1	0.9025	0.90250	19.70	0.001
Velocity	1	4.4100	4.41000	96.26	0.000
Two-way interaction	1	0.4225	0.42250	9.22	0.010
Polymer × Surfactant	1	0.4225	0.42250	9.22	0.010
Error	13	0.5956	0.04581		
Total	16	6.3306			

ANOVA, Analysis of Variance.

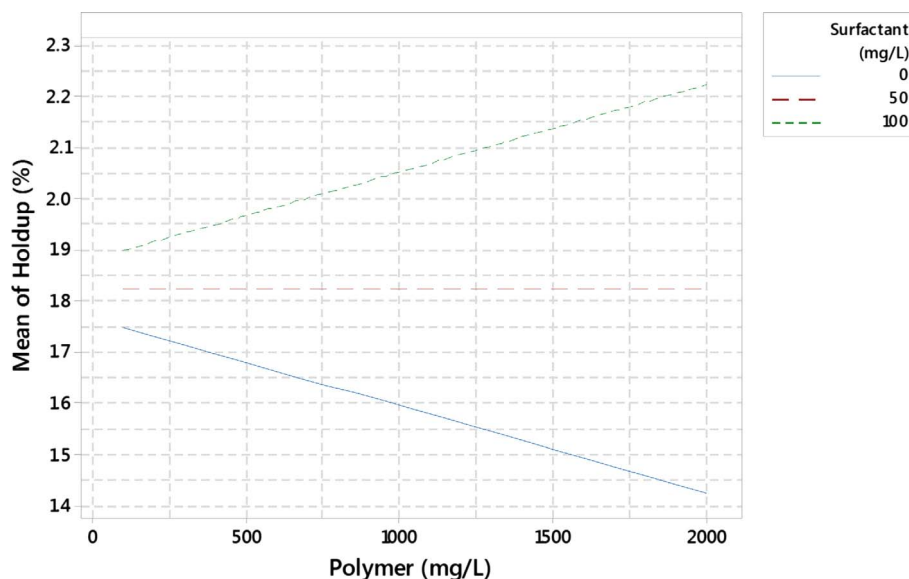**Fig. 3.** Individual main effects for gas holdup.

affected by polymer concentration; while the presence of 100 mg/L of surfactant, increases holdup when polymer concentration is increased. This behavior may be associated

with the fact that bubble diameter decreases in the presence of high concentration of surfactant.

The first-order model obtained for gas holdup is presented in equation (5) for coded variables. The coefficient of determination (R^2) is 0.9059, which ensures the model can explain 90% of the variability. The adjusted R^2 (0.8842) is in reasonable agreement with the R^2 value and therefore the gas holdup model can also be considered satisfactory:

$$\varepsilon\,(\%) = 1.82 + 0.24\,S + 0.52\,V + 0.16P \times S. \qquad (5)$$

It is worth to relate the effect of EOR chemicals on bubble size and gas holdup with changes in the physical properties of synthetic produced water. As polymer increases fluid viscosity, as discussed in Table 2, it can be inferred that viscosity is responsible for increasing bubble size and affects gas holdup differently, depending on surfactant concentration. As surfactant reduces surface tension, this physical property is responsible for decreasing bubble size and increasing gas holdup. Thus, the presence of surfactant inhibits the effect of polymer in increasing bubble size and makes

Fig. 4. Effects of the interaction in the first-order model for gas holdup.

it increase gas holdup. For the range studied, salinity seems to weakly influence bubble size and gas holdup.

4 Conclusion

The present work has used a bubble column to assess the impact of EOR chemicals and superficial gas velocity on bubble size and gas holdup. Factorial experiments have been designed to identify the main factors and their interactions that significantly affect these operational parameters of bubble columns.

The resulting synthetic produced water samples have been characterized and their main physical properties have been determined. It has been shown that they behave as non-Newtonian fluids.

Experimental results did show that both the increase in superficial gas velocity and the presence of surfactant favor a significant increase in gas holdup. On the other hand, an increase in polymer concentration only increases gas holdup when surfactant is present at high concentrations.

Polymer concentration favors an increase in bubble size, while the presence of surfactant decreases bubble diameter. Salinity seems to weakly influence bubble size and gas holdup.

The effects of EOR chemicals are eventually related to changes in physical properties of the continuous phase, the synthetic produced water. The presence of polymer increases viscosity and intensifies non-Newtonian behavior and surfactants drastically decrease surface tension.

Experiments have provided clear insights of how EOR chemicals influence bubble size and gas holdup in a laboratory bubble column. As a whole, it can be said that polymers are expected to be detrimental to produced water treatment, since they tend to decrease gas holdup and increase bubble size in the absence of surfactants, rendering flotation-like systems less efficient. These results are therefore of crucial importance for proper treatment of EOR effluents, since changes in the composition of produced water can severely impact the efficiency of unit operations that rely on gas bubble size for the separation process. Further experiments in the presence of oil droplets would allow to confirm the present conclusions and this remains for future work.

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