Evaluation of Produced Water Treatment Using Advanced Oxidation Processes and Sodium Ferrate(VI)

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Abstract—Oil and gas exploration is an essential activity for modern society, although the supply of its global demand has caused enough damage to the environment, mainly due to produced water generation, which is an effluent associated with the oil and gas produced during oil extraction. It is the aim of this study to evaluate the treatment of produced water, in order to reduce its oils and greases content (OG), by using flotation as a pre-treatment, combined with oxidation for the remaining organic load degradation. Thus, there has been tested Advanced Oxidation Process (AOP) using both Fenton and photo-Fenton reactions, as well as a chemical oxidation treatment using sodium ferrate(VI), $Na_2[FeO_4]$, as a strong oxidant. All the studies were carried out using real samples of produced water from petroleum industry. The oxidation process using ferrate(VI) ion was studied based on factorial experimental designs. The factorial design was used in order to study how the variables pH, temperature and concentration of $Na_2[FeO_4]$ influences the O&G levels. For the treatment using ferrate(VI) ion, the results showed that the best operating point is obtained when the temperature is 28 °C, pH 3, and a 2000 $mg.L^{-1}$ solution of $Na_2[FeO_4]$ is used. This experiment has achieved a final O&G level of 4.7 $mg.L^{-1}$, which means 94% percentage removal efficiency of oils and greases. Comparing Fenton and photo-Fenton processes, it was observed that the Fenton reaction did not provide good reduction of O&G (around 20% only). On the other hand, a degradation of approximately 80.5% of oil and grease was obtained after a period of seven hours of treatment using photo-Fenton process, which indicates that the best process combination has occurred between the flotation and the photo-Fenton reaction using solar radiation, with an overall removal efficiency of O&G of approximately 89%.

Keywords—Advanced oxidation process, ferrate(VI) ion, oils and greases removal, produced water treatment.

I. INTRODUCTION

OVER the last century, oil exploration has become an essentially important activity for modern society, and it remains nowadays. However, the supply of its world demand has caused great damage to the environment. One of the main environmental concerns related to the oil production process is the produced water, which is the largest liquid waste generated by the oil and gas industry both from onshore and offshore production operations [1].

In addition to the amount generated, another important factor to be considered regarding produced water is its complex composition, once it is a mixture of a variety of organic and inorganic compounds. Produced water generally has as its main constituents; oils and greases, dissolved solids, organic and inorganic salts, dissolved gases and bacteria, chemical additives, heavy metals, and sometimes even radionuclides can be found [2], [3]. Thus, the potential effect associated with discharging this wastewater on the environment without an appropriate treatment has become an important source of issue. Besides, its disposal requires meeting the environmental regulations. On the other hand, produced water reinjection also requires treatment and planning to meet the standards required to avoid formation damage. The traditional technologies frequently used, such as hydrocyclones, air flotation and filtration, are not capable of achieving the separation efficiency required for beneficial use of produced water. Thus, many technologies and methods are being studied and improved, aiming to treat produced water, such as membranes and ultrafiltration, adsorption, as well as Advanced Oxidation Processes (AOP) [1], [2]. In this context, AOP have recently emerged as a viable technology in the wastewater treatment area since it is possible to mitigate environmental problems through the mineralization of toxic and harmful contaminants, producing carbon dioxide and mineral salts. Some of the main advantages of using these processes are the high applicability and low energy costs, which make them sustainable. AOP efficiency is based on highly reactive radical's generation, normally hydroxyl radicals (•OH) which are strongly oxidizing and nonselective species [4].

In addition to the well-known AOP, the use of strong oxidants, such as Ferrate(IV), has attracted many attention to its use for environmental remediation, due to its great oxidant potential (E° = 2.2 V in acid medium) [5], [6].

The aim of this work is to evaluate the produced water treatment, in order to reduce its oils and greases content (OG), firstly by using flotation as a pre-treatment, combined with AOP for the remaining organic load degradation. Thus, there will be tested and compared both Fenton and photo-Fenton processes. Also, a chemical oxidation treatment $Na_2[FeO_4]$ as a strong oxidant will be studied later on. The oxidation process using ferrate(VI) ion will be studied based on factorial

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experimental designs used in order to evaluate how the variables pH, temperature, and oxidant concentration influence the OG levels.

II. Methodology

A. Produced Water Characterization

The produced water used has been collected from a three-phase separator from a Petrobras Waste Water Treatment Plant in Brazil. Samples of the collected water were analyzed, in order to obtain some of the physical and chemical properties, which consisted in pH determination, conductivity, turbidity, Total Dissolved Solids (TDS), and Oils and OG. In addition, the cations Ca^{+2}, Mg^{+2}, Na^+, K^+, and Fe^{+2} have been quantified, as well as the anions chlorides, sulfides, sulfates, carbonates, bicarbonates and nitrites. All methods used to determine these parameters were followed according to the Standard Methods for the Examination of Water and Wastewater [7]. The main parameter used for the evaluation of the treatments efficiency was the OG.

B. Pre-Treatment Using Flotation

All the batch flotation test has been performed at 28 °C by adding 300 mL of produced water to a beaker, under a constant aeration of 1.76 L.h^{-1}, for 2.5 hours, under a 1050 rpm constant stirring. A magnetic stirrer with heating plate was responsible for shaking the sample during flotation, as shown in Fig. 1.

Fig. 1 Experimental set-up of the flotation process used to treat produced water

C. Fenton-Like and Photo Fenton-Like Using Solar Radiation

The Fenton-like reaction was used aiming to reduce the OG levels of the produced water once a high concentration of iron was verified after the water characterization. An H_2O_2 concentration of 0.5 mmol.L^{-1} was used at this stage. Thus, 700 mL of the produced water sample was acidified using H_2SO_4 until the pH was equal to 3. Then, 100 mL of the sample was placed in seven Erlenmeyer flasks each, adding H_2O_2 to each one of these systems. After one-hour intervals, the inhibitor solution (0.1 M NaOH, 0.1 M KI, and 0.1 M Na_2SO_3) was added to each of the systems in order to stop the Fenton reaction.

The samples were filtered for the precipitated iron removal, and then taken for final OG analysis. Therefore, the OG degradation was monitored up to seven hours of treatment, in one-hour intervals. The same experimental procedure was then repeated. In order to test the effect of adding iron to the system, the same procedure was performed, however, by adding 0.4 mM of Fe^{+2} to each of the erlenmeyers, using $FeSO_4.7H_2O$.

Regarding the photo Fenton-like process using solar radiation, a concentration of 160 mM H_2O_2 was used, and no iron source was needed. The reaction system consisted of using Petri dishes exposed to the sun for a period of up to 7 hours, containing 30 mL of produced water with pH equal to 3. The produced water acidification process was also performed using H_2SO_4.

D. Ferrate(VI) Ion

All Na_2FeO_4 solutions used in this research were prepared at the time of their use to avoid ferrate decomposition. In this case, the method used for the sodium ferrate synthesis was wet oxidation. Nevertheless, the washing and purification steps were not performed in order to minimize the production costs. The source of Fe^{+3} was a commercial ferric chloride ($FeCl_3$) solution at 41% w/w. On the other hand, the sodium hypochlorite (NaClO) used was an alkaline solution (3/4 of sodium hypochlorite at 10% w/w and ¼ of caustic soda at 50% w/w) supplied by the company Beraca Sabará Chemicals and Ingredients S/A.

Due to the high organic load of the working effluent and also to the recalcitrance of the pollutants, the oxidant manufacturer's indicated working with a sodium ferrate concentration range between 1000 and 2000 mg.L^{-1}.

The performance of ferrate(VI) as an oxidant is influenced by several factors, such as the pH of the medium, the oxidant solution's concentration, as well as the temperature of the process. To investigate the effects of these variables and to optimize the process, a 2^3 factorial experimental design was performed in duplicate, each one with two replicates at the central point. These variables were considered as the input variables, and the response variable was the final OG level of the treated produced water. Table I shows the factors and levels of the factorial experimental design studied.

TABLE I
FACTORIAL DESIGN LEVELS FOR THE OXIDATION PROCESS USING THE FERRATE(VI) ION

Coefficients	Levels		
	-	0	+
pH	3	5	7
Temperature (°C)	28	64	100
$Na_2[FeO_4]$ concentration (mg.L^{-1})	1000	1500	2000

Regarding the pH variable, its measured value for the crude effluent was not high (pH=7), indicating a neutrality. However, according to the literature, ferrate(VI) ion presents better results as oxidant, when used in acidic solutions (pH=3). For the temperature, the chosen minimum and maximum levels were, respectively, the ambient temperature

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(28 °C) and 100 °C, due to technical and economical limitations since this is an easily reached temperature.

All batch oxidation experiments were performed by adding 100 mL of produced water to 125 mL Erlenmeyer flaks, adjusting the pH with H_2SO_4, as necessary, according to what was required for each experiment. To each of the Erlenmeyer flasks, were added the appropriate quantities of freshly prepared Na_2FeO_4, at the required temperature (all predicted by the experimental design). After 24 hours, all samples were filtered. Then, the final OG levels of each sample, after the water treatment, were analyzed. It is important to note that, for each experiment, a corresponding white test was also performed.

III. RESULTS AND DISCUSSION

A. Produced Water Characterization

The results obtained during the characterization of produced water physicochemical parameters are shown in Table II.

B. Pre-Treatment via Flotation

After the flotation experiment has been performed as described above, the corresponding OG value of 42.7 mg.L^{-1} was measured after treatment, representing a removal efficiency of approximately 47% of oils and greases. This result can be considered as satisfactory when the simplicity of the process, as well as its cost-effectiveness, is considered. Therefore, it is interesting to evaluate the use of this process as a pre-treatment for the oxidation process in order to reduce the contaminant content of the effluent that is destined to the oxidation treatment since the oxidation process could present higher cost than flotation.

C. Fenton and Fenton-Like Experiments

The results when the Fenton reaction was used to reduce the OG levels in produced water are shown in Fig. 2. Analyzing the graph, it is possible to notice that the results obtained during the experiments using both Fenton and Fenton-like reactions were quite similar, which indicates there are no advantages in adding iron into the process. In both processes, the OG removal efficiency was around 20%.
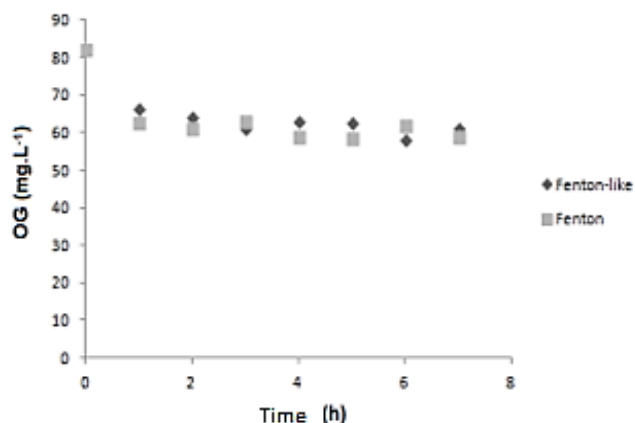
Fig. 2 Reduction of the OG levels in the produced water as a function of time

Thus, it is important to notice the need for the use of radiation, in order to increase the efficiency of the process, which was already expected according to the literature [12]-[14].

TABLE II
RESULTS FOR THE CRUDE PRODUCED WATER CHARACTERIZATION

Parameters	Measured values	Values according to the literature
pH	7.01	5.2 – 7.4 [8]
Conductivity (mS.cm^{-1})	4.1	3.702 [9]
Turbidity (NTU)	180	204.0 [9]
Chlorides (mg.L^{-1})	2100	80 – 200000 [10]
TDS (mg.L^{-1})	3580.00	2130 [11]
Sulphides (mg.L^{-1})	19.0	11.0 [11]
Total Hardness of water (mg.L^{-1})	750.0	-
Ca^{+2} (mg.L^{-1})	150.3	13 – 25800 [10]
Mg^{+2} (mg.L^{-1})	91.2	8 – 6000 [10]
Na^+ (mg.L^{-1})	1240.0	132 – 97000 [10]
K^+ (mg.L^{-1})	60.10	24 – 4300 [10]
Fe^{+2} (mg.L^{-1})	34.5	<0.1 – 100 [10]
SO_4^{-2} (mg.L^{-1})	259.00	2 – 1650 [10]
CO_3^{-2} (mg.L^{-1})	9.6	-
HCO_3^- (mg.L^{-1})	273.12	77 – 3900 [10]
NO_2^- (mg.L^{-1})	0.09	40 – 100 [9]
OG (mg.L^{-1})	81.0	2 – 565 [8]; 29.32 [11]

D. Photo Fenton-Like Experiments

For the photo-Fenton-like process using solar radiation, the results shown in Fig. 3, indicated that at the end of the 7-hour treatment, the OG level was 16.33 mg.L^{-1}, which represents an OG removal percentage of approximately 80.5% for this process.
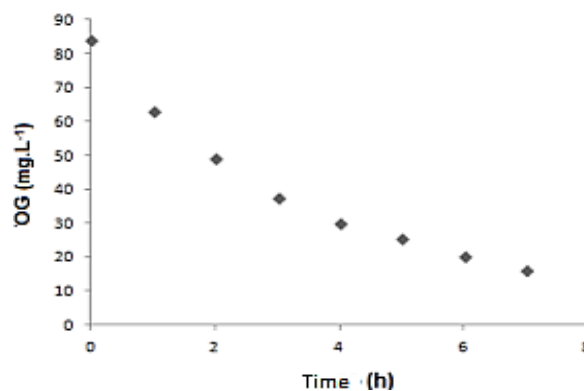
Fig. 3 OG reduction in produced water as a function of the reaction time

Based on the results already discussed, it was decided to evaluate the use of flotation as a pre-treatment only for the photo-Fenton reaction using solar radiation. This choice can be justified by the fact that this process presented the best results among the Fenton/photo Fenton experiments performed in the present work. Thus, when photo-Fenton-like reaction was used after flotation, as the second stage for produced water treatment, a final OG level of 8.7 mg.L^{-1} was obtained, which therefore indicates an overall OG removal efficiency of approximately 89%.

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E. Ferrate(VI) Ion: Evaluating the Influence of pH, Temperature and Oxidant Concentration on the Oxidation Process

STATISTICA for Windows version 7.0 software was used to evaluate the experimental data obtained in the factorial experimental design. The results obtained for each corresponding experimental design experiment are presented in the Appendix in Table IV.

Analyzing Table IV, it can be observed that higher efficiency in the reduction of the oils and greases by the oxidation using the ferrate(VI) ion, was obtained in the experiment with pH at its lower level and the variables temperature and concentration at their higher levels, which corresponds to a final OG of 3.42 $mg.L^{-1}$, and an average OG removal efficiency of 95.78%. However, when comparing this result with the experiment in which pH and concentration were maintained at the same levels, but the temperature was at its lowest level, an OG removal efficiency of 94.23% was observed. Then, it can be stated that in economic terms, it could be more feasible to work at 28 °C. Therefore, the experiment with pH and temperature at its lower levels (pH=3 and T=28 °C) and concentration at its upper level (C=2000 $mg.L^{-1}$) will be considered as the optimal point.
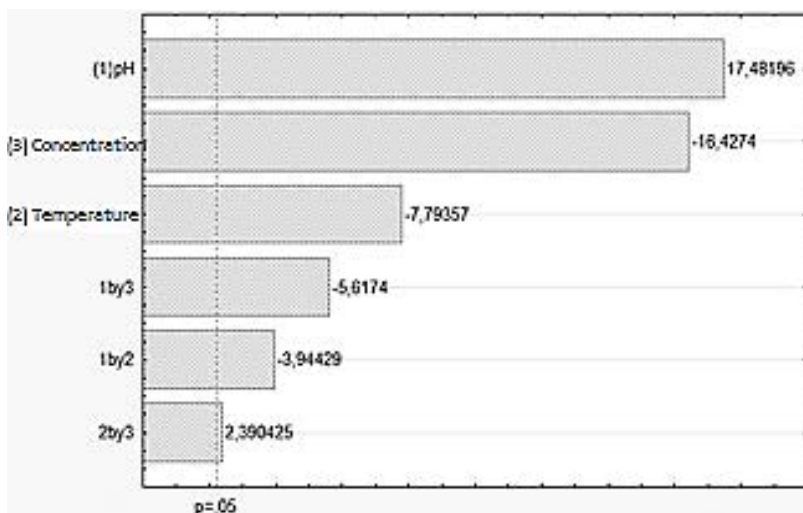

Fig. 4 Pareto diagram representing the effects estimation caused by pH, temperature and $Na_2[FeO_4]$ concentration, and its interactions in the treatment of produced water using the ferrate ion(VI) as oxidant, for a linear adjustment
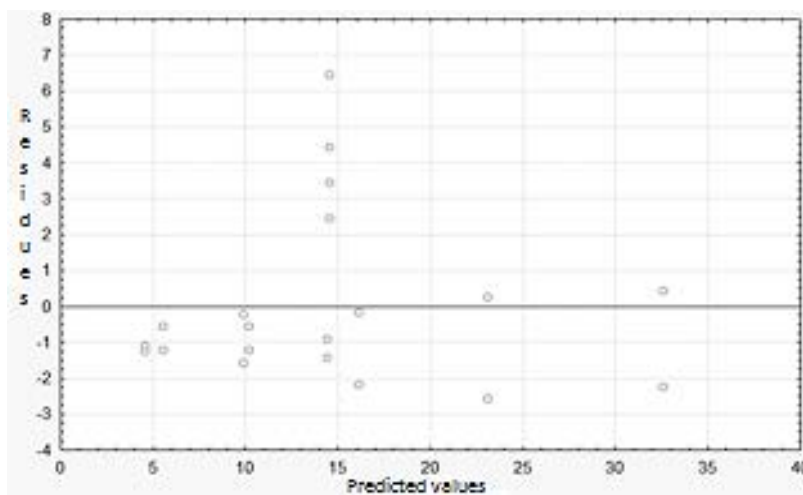

Fig. 5 Predicted values in the produced water treatment vs. residual values

From the data shown in Table IV, it was evaluated the possibility of a linear correlation between the process variables within the studied range. Fig. 4 presents the Pareto Diagram, which shows the main effects estimation for the main variables, as well as the corresponding interaction effects. In this graph, the variables whose rectangles are after the vertical line are those with significant effects at 95% confidence. Thus, it is easy to observe that all the three variables isolated presented significant effects on the wastewater OG removal. It can be also noticed that combined

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action between the variables also have statistical significance. Their adjustments were discussed through Analysis of Variance (ANOVA), according to Table III, together with the evaluation of residues graphs, Figs. 5 and 6.

Analyzing Table III, it can be seen that regression shows 96.13% of the variance around the mean, of a maximum percentage of 98.53% that could be explained. Therefore, 2.4% of the variance around the mean cannot be explained due to the model lack of fitting, at 95% confidence.

The F-test has been done in order to assess whether the regression model is statistically significant. As the calculated value, equal to 25.06, was much higher than the tabulated value, $F6.13=2.98$ at 95% confidence, then the null hypothesis can be rejected, and the regression equation is statistically significant [15]. However, it is also important to evaluate the fit quality of model.

Observing Fig. 4, it can be seen that most of the residues are settled far from the zero-centered average, indicating a possible lack of fit for the chosen model. In addition, Fig. 5 shows the experimental values versus the predicted values, and that also indicates the model's lack of fit since several points are outside the line. Faced with this evidence, a new F test was performed to confirm the lack of fit. Using the data in Table III, the calculated value equal to 24.29 is much higher than the tabulated value, $F2.11=3.98$, at 95% confidence, which confirms the lack of fit. Thus, an option to eliminate the lack of adjustment, is to add a quadratic term. The adjustment of a quadratic model, however, generates an equation with higher number of parameters, which consequently requires a greater number of levels. The data required for the quadratic model adjustment can be obtained through the factorial design expansion.
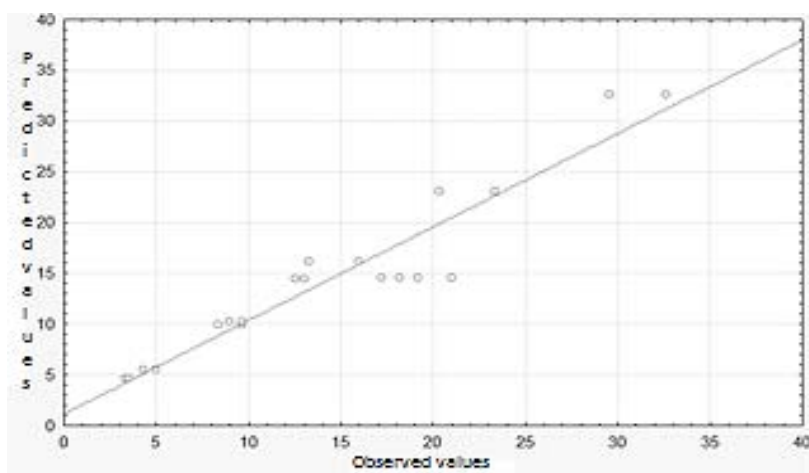

Fig. 6 Observed values vs predicted values by the linear model in the treatment of the production water using ferrate(VI) ion

TABLE III
ANOVA FOR THE LINEAR MODEL ADJUSTMENT

Variation source	Quadratic sum	Degrees of freedom	Quadratic average	Explained variation	Maximum explained variation
Regression	1240.598	6	206.7663	96.13%	98.53%
Residue	107.273	13	8.251769		
Lack of adjustment	87.468	2	43.73415		
Pure error	19.805	11	1.800459		
Overall	1290.55	19			

As in this research, there are economical limitations regarding reducing the pH to values below 3, and also for increasing the sodium ferrate(VI) concentration to values higher than 2000 mg.L^{-1}, so it was decided to perform face-centered planning only by adding new points within the studied range. Therefore, the realization of more experiments should provide results which will allow the adjustment to a quadratic model, predicting, within the studied range, the OG value according to the studied variables. Nevertheless, as already discussed, the data already obtained by the factorial design indicate as optimal point the experiment with pH and temperature at its lower levels (pH = 3 and T = 28 °C) and

sodium ferrate concentration at its upper level (C = 2000 mg.L^{-1}), resulting in an average final OG level equals to 4.67 mg.L^{-1}, representing an overall OG removal efficiency of 94.23%, even without using the proposed pre-treatment.

IV. CONCLUSION

The flotation process used as a pre-treatment for produced water resulted in an OG reduction of approximately 47%. Both Fenton and Fenton-like reactions did not show a satisfactory OG reduction (both around 20%), as expected, indicating that photo-Fenton processes using solar radiation would possibly show better results. Thus, using photo-Fenton-like process with solar radiation, by the end of the seven-hour treatment, the OG removal percentage obtained was approximately 80.5%. In addition, it was found that the combination of flotation and photo Fenton-like using solar radiation achieved an overall OG removal efficiency of approximately 89%.

With respect to produced water treatment using sodium ferrate, the results indicated a large influence of Ph, as well as Na$_2$[FeO$_4$] concentration in the medium, while the observed effect of temperature was not significant. Due to the discrete

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influence of temperature, it was concluded that increasing the temperature from 28 °C to 100 °C is not economically interesting. Thus, it was considered that the optimal result is obtained when using pH and temperature at the lowest level, and the sodium ferrate(VI) concentration at the highest level (C=2000 mg.L^{-1}), which corresponded to a removal efficiency of approximately 94% in the final OG levels.

Regarding the linear adjustment, it was concluded that the data did not fit well at the studied model, and therefore, it was decided that proceeding with new experiments is necessary in order to try to adjust the data to a quadratic model.

APPENDIX

TABLE IV
COMPLETE 2³ FACTORIAL DESIGN MATRIX FOR PRODUCED WATER TREATMENT USING FERRATE(VI) ION

Experiment	pH	Temperature	Na$_2$[FeO$_4$] concentration	OG (mg.L^{-1})
01	-	-	-	13.00
02	+	-	-	39.33
03	-	+	-	9.00
04	+	+	-	23.33
05	-	-	+	4.33
06	+	-	+	14.00
07	-	+	+	3.33
08	+	+	+	9.67
09 (C)	0	0	0	19.00
10 (C)	0	0	0	22.00
11	-	-	-	13.50
12	+	-	-	33.00
13	-	+	-	9.67
14	+	+	-	20.50
15	-	-	+	5.00
16	+	-	+	16.00
17	-	+	+	3.50
18	+	+	+	8.33
19 (C)	0	0	0	17.00
20 (C)	0	0	0	18.00

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