



Produced Water Deoxygenation via Nitrogen Purging Scheme– Parametric Study – Part 2

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HIGHLIGHTS

- A gas lift column was examined to de-oxygenate produced water collected from Omani oil fields.
- Semi batch and counter-current purging packed column schemes were operated. effectively for the de-oxygenation process
- Complete de-oxygenation of produced water grafted with polyacrylamide was achieved within two minutes.
- Achieving complete DO removal indicates the feasibility of using the proposed approach for large-scale industrial applications.

ARTICLE INFO

Handling editor: Qusay F. Alsathy

Keywords:

Produced water treatment; Dissolved oxygen; N₂ purging; Gas lift column.

ABSTRACT

In this following up paper, we present our findings by examining a pilot scale gasification column and applying nitrogen purging for samples of produced water grafted with different polyacrylamide concentrations (100 – 500 ppm). Upon applying a semi-batch, counter-current scheme for a series of experiments on packed gas-lift column, zero ppm level of dissolved oxygen (DO) was reached within less than 1 minute of nitrogen purging from the start time applied for solutions with viscosity less than 10 mPa.s and using the inline measuring scheme. However, zero ppm DO level was not reachable when purging produced water (PW) samples grafted with fresh polyacrylamide with a viscosity higher than 10 mPa.s. Nonetheless, the residues of DO were detected by offline measuring after examining the higher viscosity samples in the shallow limit (less than 0.4 ppm DO) and reached zero ppm when applying the inline measuring scheme. Two operation mode schemes, circulation, and once-through, were applied. Upon investigating the once-through contact scheme, the adopted nitrogen purging method was effective in reaching zero ppm level in less than 2 minutes, which is an excellent result compared with other well-known treatment techniques.

1. Introduction

Oil production wells generate huge quantities of water mixed with hydrocarbons, solids, and salts. The take-out is known to be produced water (PW) which is unsuitable for surface discharge due to the high content of hydrocarbons and ions [1]. PW is therefore injected into deep ground layers as a disposal method [2]. However, the increased demand for water in industrial, agricultural, and domestic sectors, especially in water-stressed regions, makes PW's treatment worth functional investment and reuse [3]. On the other hand, the high content of dissolved oxygen (DO) in the produced water is considered a big challenge to the ample reuse of PW, as dissolved oxygen promotes microbiological activity leading to corrosion in the reservoirs and plugging of the injection wells [4].

Produced water (PW) forms wastewater, continuously found with petroleum in reservoirs [5]. Large quantities of wastewater are produced with oil production, approaching 80-95% [6-7]. Around 300 million barrels of produced water per day from oilfields [8], from which 40% is discharged into the environment [5, 8-9]. PW is considered high salty formation water (TDS = 5000 to 250,000 mg/L) with low TSS content and not heavily emulsified oil in water. The global efforts towards preserving water resources suggest the need to reuse a huge quantity of produced water, commonly wasted in water bodies

randomly. However, the major challenge towards reusing PW is the high content of dissolved oxygen (DO). DO is a corrosive agent that converts H_2S or FeS into S compounds [10]. Moreover, DO promotes microbiological growth in the reservoirs, undesired for effective oil recovery processes [11]. Both chemical methods (e.g., sodium sulfite and ammonium sulfite as oxygen scavenging agents) and physical treatments were applied to remove dissolved oxygen from produced water [12, 13]. Chemical methods were shown with various deficiencies such as water contamination, high capital and operating cost, low production rate, and low efficiency.

On the other hand, physical methods were experimented with, and the results indicated better performance in this sense. N_2 purging was the most practical/ effective for de-oxygenating deionized water [14]. However, the application of nitrogen purging for DO removal from produced water was not scarce mentioned in the literature due to difficulties resulting from various contents in the produced water (e.g., chemicals and salts).

In our previous study [11], de-oxygenation of PW grafted with various polyacrylamide concentrations [100-500 ppm] was conducted in unpacked and packed gas lift columns applying semi-batch, counter-current as well as once through contact modes and adopting an offline measuring scheme [i.e., DO was measured, after a while, for samples collected from the column]. A considerable reduction in the dissolved oxygen content up to shallow limits (less than 1 ppm) was detected; however, residues of DO (≈ 0.5 ppm) were unable to remove even when elongating the purging duration to 300 minutes. This was attributed to the used DO measuring device limitation (3 minutes to display results in the offline scheme) and other reasons related to the column design and operation (e.g., packing arrangement). This finding protrudes a difficulty in achieving complete DO removal and suggests the need for further investigations. The current phase of the study (Part2) is to conduct thorough investigations in the shallow DO limits towards attaining complete DO removal.

2. Materials and Methods

The work in this phase was based on the gasification column principle used in Part1 of the study [11]. A schematic diagram is illustrated in Figure 1. An atmospheric cylindrical lab-scale gas-lift column consisted of a PVC tube [3 mm thickness, 1.3 m height, 10 cm diameter, fitted with 6 cm in diameter spherical shape gas distributor of fine holes (i.e., average hole size of 3 mm)], was designed and fabricated in the labs of the university of Nizwa, the photo is depicted in Figure 2. The applied fine diffuser secured the generation of fine nitrogen bubbles, which acted positively in attaining better DO removal efficiency compared with the results obtained in part1 [11] of the study. The experimental sets were designed to test the purging of tap water (TW), produced water (PW), and artificial samples prepared from produced water grafted with a polymer (anionic polyacrylamide) (PWP) in a range (100-500 ppm). Other operational parameters were set to be the nitrogen flow rate (in the range 2-6 L/min resulting in superficial gas velocity in the range 25.5 – 76.4 m/min) and the liquid stream feed rate (in the range 1-3 L/min). It's worth mentioning that the dynamic level in the column was set to be 30 cm from the tower bottom and applied in all experimental sets. It is also important to mention that temperature considerably affects the gas stripping process. However, we have not considered the effect of the temperature increase in the current study due to some limitations. It will be considered in future studies. Furthermore, all experimental sets were conducted at atmospheric pressure to resemble industrial conditions.

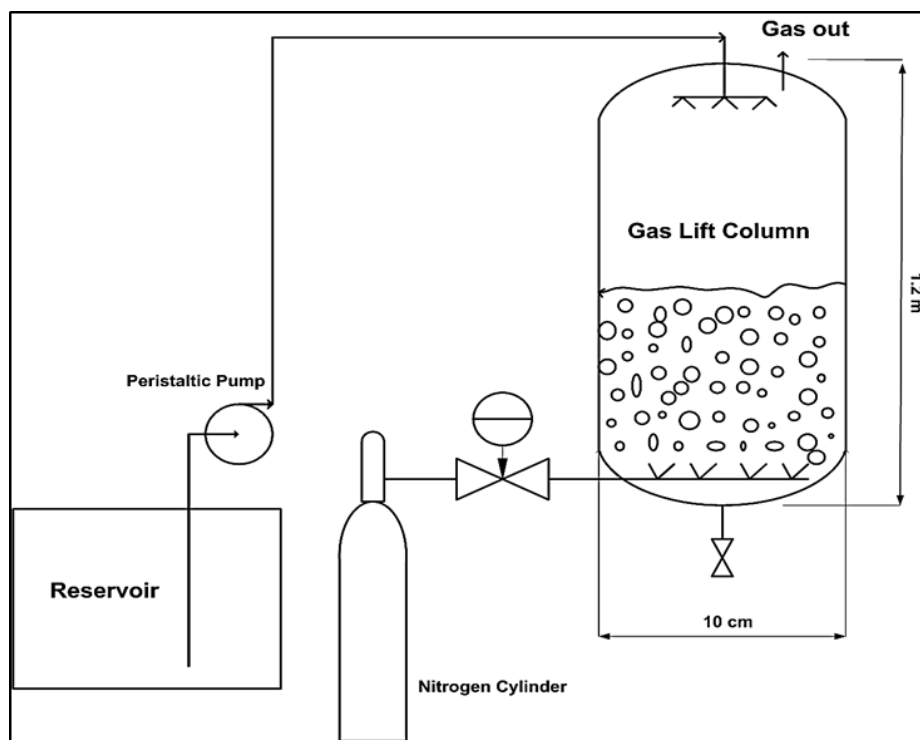


Figure 1: Schematic diagram for the cylindrical gas lift column [5]

To mimic industrial PW samples, anionic polyacrylamide was selected to prepare artificial solutions of produced water at different concentrations, exceeding, in some cases, the real produced water found in the oil industry. Polyacrylamide is a water-soluble polymer, insoluble in most organic solvents, and has great flocculation results. As a result, it can reduce the frictional resistance between liquids; therefore, its commonly used for viscosity reduction, which is highly important for applications in the oil industry.

The PWP samples, in a range (of 100-500 ppm), were prepared by dissolving the hydrolyzed polyacrylamide powder received from Petroleum Development Oman (PDO) in the produced water, applying appropriate agitation (via a magnetic stirrer running at 700 rpm) for 5 hours. The selected range for PWP samples was based on a viscosity profile standard, where higher solution viscosity is proportional to the polyacrylamide concentration. After completing dissolution, the prepared PWP samples were left stagnant for more than 12 hours to ensure no separation for the dissolved polyelectrolyte. Nitrogen gas, purchased from “Muscat gas co.” was supplied through gas cylinders. The gas flow rate was controlled through a gas rotameter (Tucsen/China).



Figure 2: Picture for the gas lift PVC column packed with glass balls and connected to the DO sampling head from the bottom

Various amounts of produced water supplied from PDO were loaded to the column and purged through nitrogen according to a configured experimental plan, using semi-batch and counter-current operational schemes. It is worth mentioning that the viscosity of the examined produced water was measured using a viscometer (MYR VR 3000 rotational viscometer) while the dissolved oxygen was measured throughout the experimental runs using a DO sensor (Hanna DO Model OX-2P), applying two approaches. The first was implemented by taking the sample from the column and measuring the DO instantly on the bench (offline DO measuring scheme). This has resulted in an inconsistency in some studied cases, in which measured DO residues were kept in the shallow region (< 0.4 ppm) even when applying long purging durations. In later experiments, the DO sensor was located in a custom-made sampling head for this purpose (named inline DO measuring scheme). This approach led to a clear trend for DO reduction, approaching zero ppm level in most of the investigated cases. Hence, our results are presented and discussed according to the applied contact approach (semi-batch/counter-current), and the applied DO measuring scheme (inline/offline). To avoid varied DO levels at the start point, all experimental sets in this study were implanted by using water samples that reached the DO saturation point. This has been done by subjecting the examined water sample to a compressed air purging overnight, reaching oxygen saturation level (≈ 6 ppm).

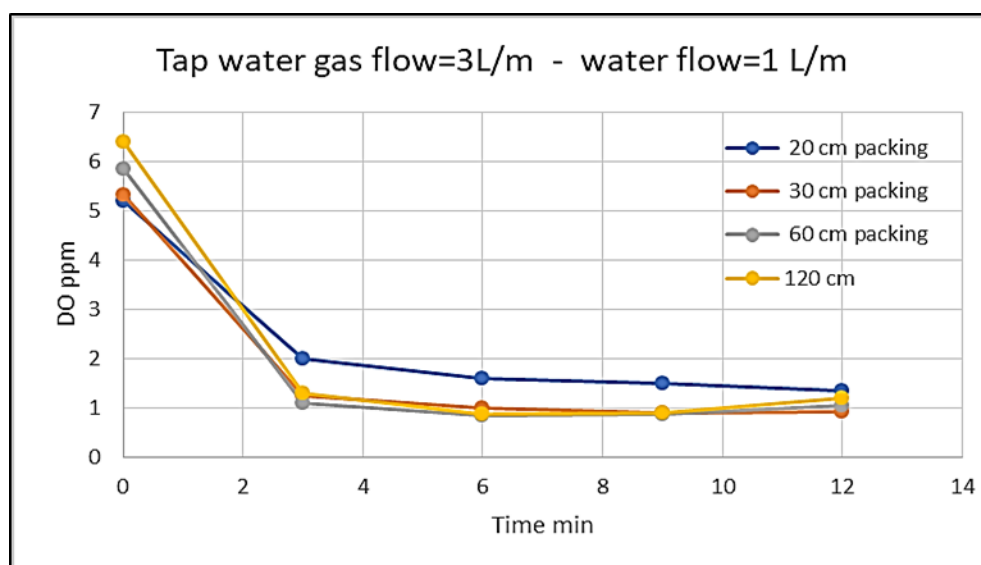
3. Results and Discussion

The experimental sets were dedicated to measuring the efficiency of the applied N_2 purging method for reducing DO content in the examined samples. Both semi-batch and counter-current operational schemes were applied for the PW purging with N_2 in a packed column equipped with a spherical glass balls layer, applying offline and inline DO measuring schemes.

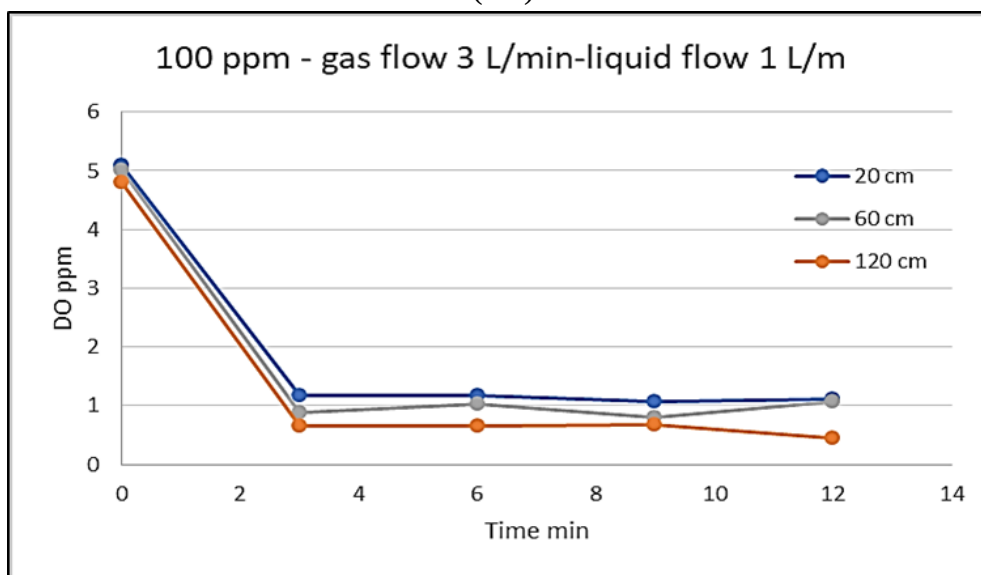
3.1 Counter Current Purging in A Packed Column Applying Offline DO Measuring Scheme

The offline DO measuring mode results were presented in our previous work [5], demonstrating considerable DO removal in all applied schemes. In contrast, DO in the shallow limits (≈ 0.5 ppm) could not remove even when applying nitrogen

purging for 300 minutes. Therefore, the packed layer height was increased to 120 cm in further investigations considering DO offline measuring mode. An experimental set was conducted to evaluate the enhancement in the DO removal efficiency. The PVC column, Figure 2, for this experimental set was used as it proves more reliable when packed with heavy-weight glass balls. Figure 3 (a-b) compares the offline DO measure upon implementing different packing layer heights using tap water and 100 ppm PWP. The tap water results indicate a limited enhancement achieved when increasing the layer height to 120 cm. As per logic, the better removal efficiency was expected from doubling the packing layer height. However, PWP reduced the DO to 0.45 ppm at 120 cm. This could be attributed to a reduction in the interfacial tension upon the existence of chemicals in the stripped solution. This may indicate a DO measuring issue (offline) exists when reaching the shallow limits. An extra set was implemented at 120 cm packing height, fixing the liquid flow rate at 1 L/min and examining different nitrogen purging flow rates (2-6 L/min) and different concentrations of PWP samples (100-500 ppm). The results are presented in Figure 4 (a-e), which demonstrate incomplete DO removal. Hence, all previous schemes applied with experimenting with various operational parameters could not reduce the dissolved oxygen in the shallow region (less than 0.4 ppm), which makes it a questionable mark.



(3-a)



(3-b)

Figure 3: (a-b) Effect of using various heights of packing layer. Counter current contact scheme applied at 3 L/min (N_2) and 1 L/min liquid flow (offline DO measuring scheme) for tap water (3-a) and for PWP grafted with 100 ppm polyacrylamide (3-b)

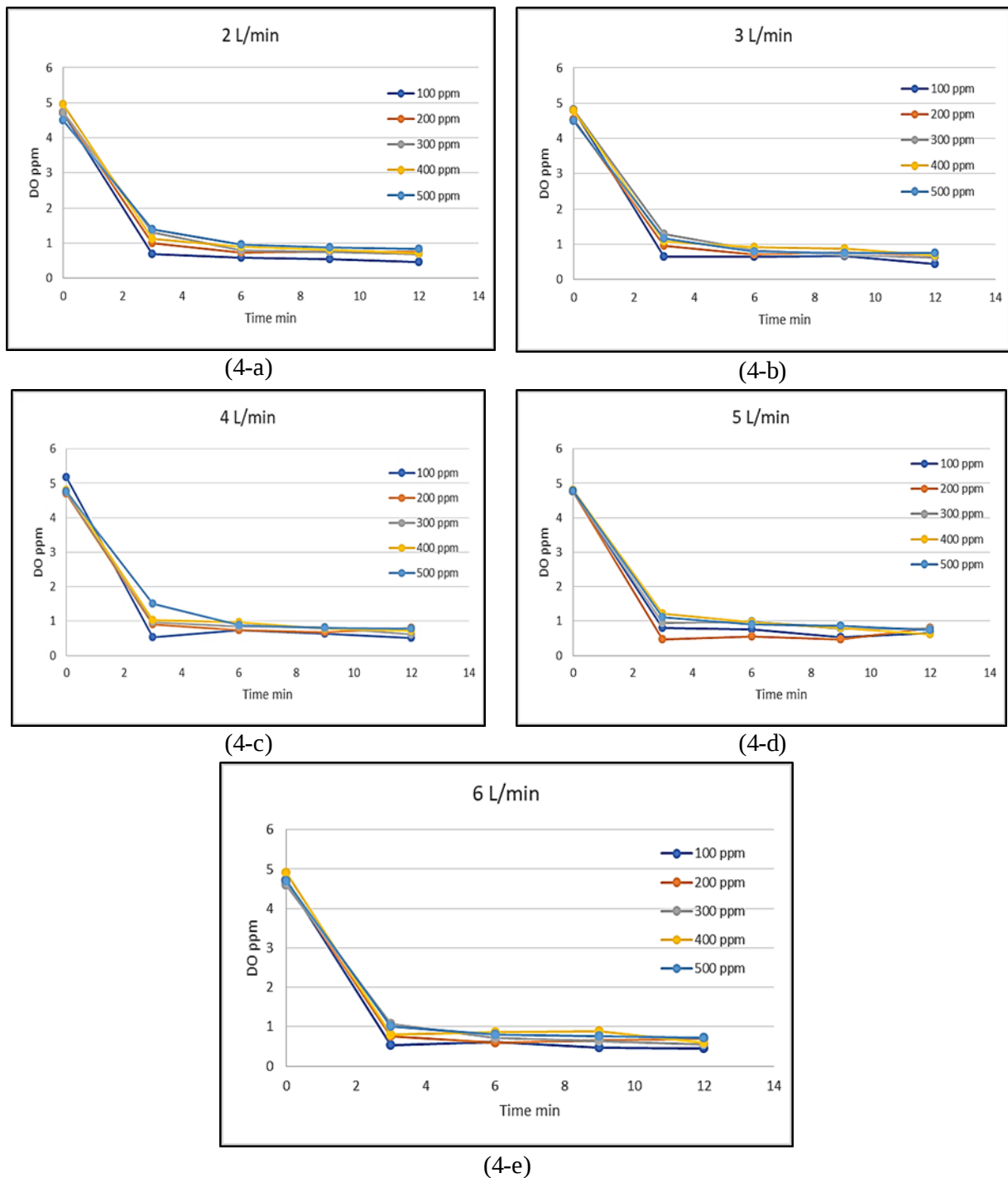


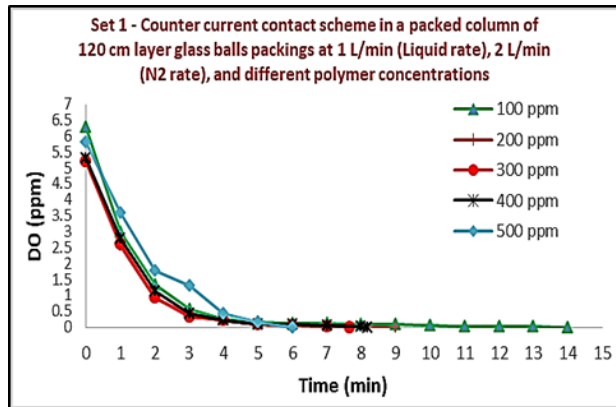
Figure 4: (a-e) Comparison between the results of applying purging for a counter-current contact scheme applied at 1 L/min liquid flow, various N_2 purging rates, and different PWP concentrations (offline DO measuring scheme)

The aforementioned observation in the offline mode suggests a need for a thorough study targeting the shallow DO region. However, PWP reduced the DO to only 0.45 ppm when applying 120 cm packing layer. Upon applying deep investigations, we observed that the DO reading had reached zero ppm level on applying the inline measuring scheme, as shown in the next section. This indicates that during collecting the sample from the column and preparing it for offline DO measurement (on the bench), it absorbs oxygen immediately from the atmosphere, subject to the sample shaking/agitation rate, leading to a sudden increase in the DO and considerable deviation from the real value. This became clear when a sample from the column was put in a beaker, and the DO was measured before and after sample shaking. We observed a significant increase in the measured dissolved oxygen after shaking the beaker for just a few seconds.

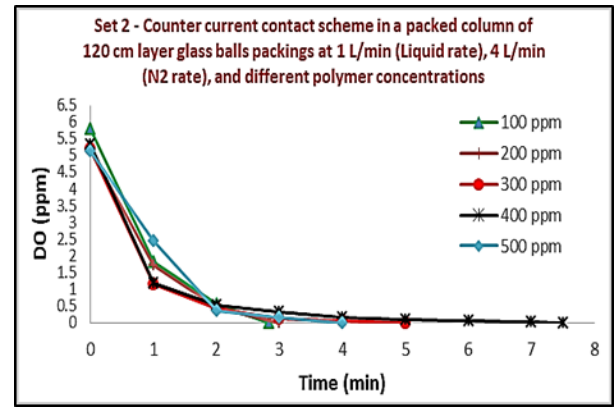
Hence, the coming section discusses the results of further investigations conducted when applying the inline DO measuring scheme.

3.2 Counter Current Purging in A Packed Column Applying Inline DO Measuring Scheme

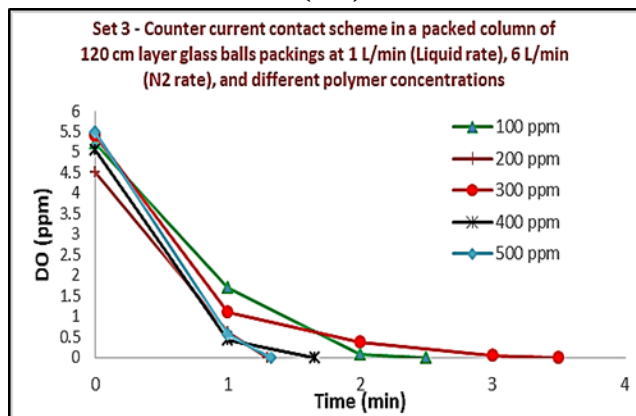
As per the thorough investigations reported in our previous study [5], a counter-current contact scheme led to better DO removal efficiency. Therefore, the experimental sets were implemented in this section considering the inline DO measuring scheme. The PVC column Figure 2 was used in all sets, proving more reliable when packed with heavy-weight glass balls. Nine experiments were conducted considering various operational parameters applying the inline measuring scheme. The first set of results is illustrated in Figure 5 (a-i), which indicates the achievement of zero DO ppm level within a few minutes of N₂ purging, regardless of the polyacrylamide concentration value commensurate with adopting the inline measuring scheme for the future stages, including scale-up tries. The obtained results prove the success of the adopted nitrogen purging scheme for attaining 100% DO removal efficiency from extreme cases of produced water samples extracted from different resources.



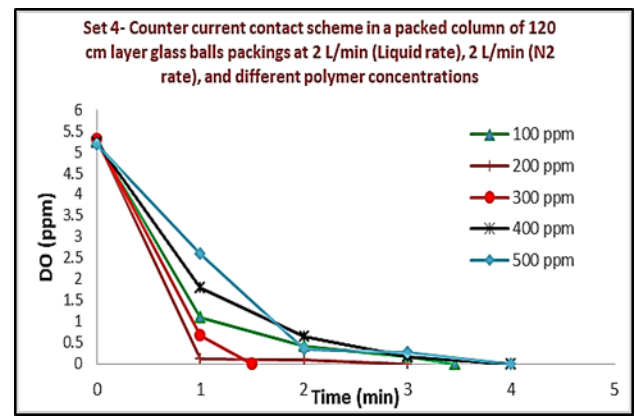
(5-a)



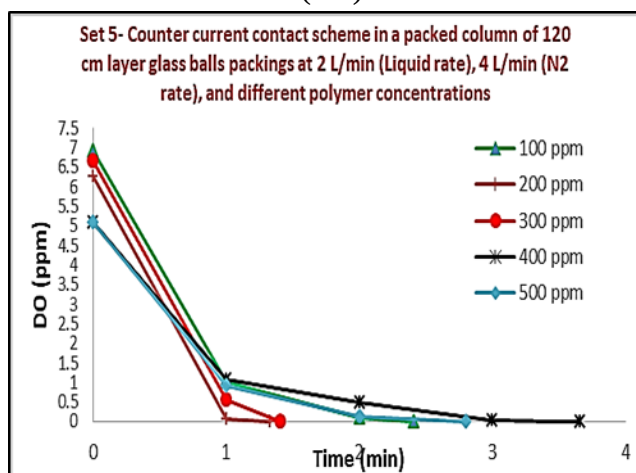
(5-b)



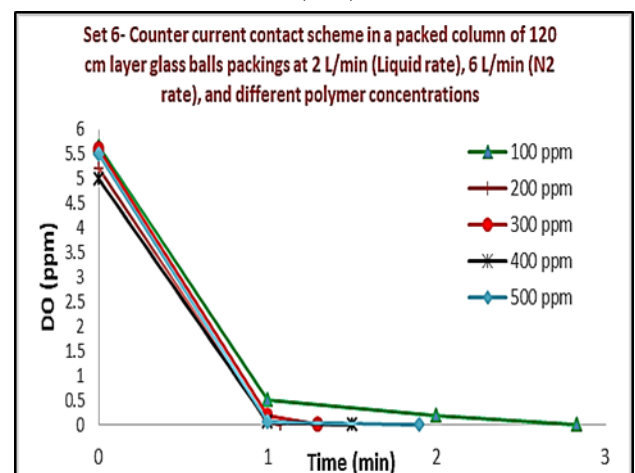
(5-c)



(5-d)



(5-e)



(5-f)

Figure 5: Continued

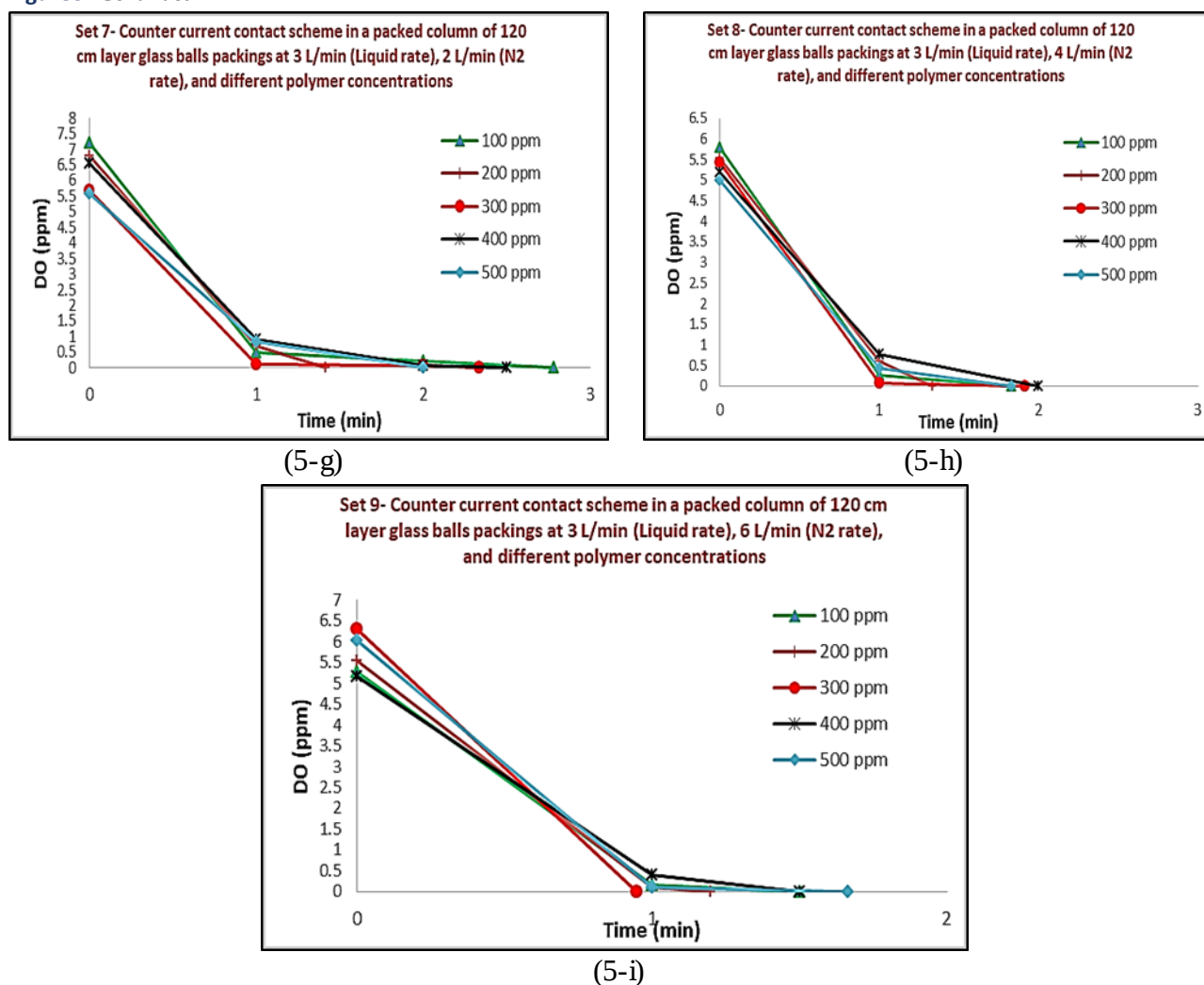


Figure 5: (a-i) Comparison between the results of applying purging for a counter-current contact scheme applied at various N_2 purging rates, liquid flow rates, and different PWP concentrations (inline DO measuring scheme)

For optimization purposes, the results were summarized in Table 1 and Figure 6, which clearly indicate the optimum ratio (liquid/gas) is 1/3 at different polymer concentrations.

Table 1: Purging time required to reach zero ppm DO at different liquid/gas ratios applied for different polymer concentrations

Polymer conc (ppm)	Ratio (Liquid /Gas)								
	Time (min) for zero Do 1 L/m Liquid and 2,4,6 L/m Gas			Time (min) for zero Do 2 L/m Liquid and 2,4,6 L/m Gas			Time (min) for zero Do 3 L/m Liquid and 2,4,6 L/m Gas		
	1/2	1/4	1/6	2/2	2/4	2/6	3/2	3/4	3/6
100	14	2.83	2.5	3.46	2.4	2.83	2.785	1.833	1.5
200	9	3	1.3	3	1.33	1.08	1.416	1.333	1.2
300	7.66	5	3.5	1.5	1.41	1.3	2.333	1.916	0.95
400	8	7.5	1.66	4	3.66	1.5	2.5	2	1.5
500	6	4	1.33	4	2.8	1.9	2	1.833	1.66

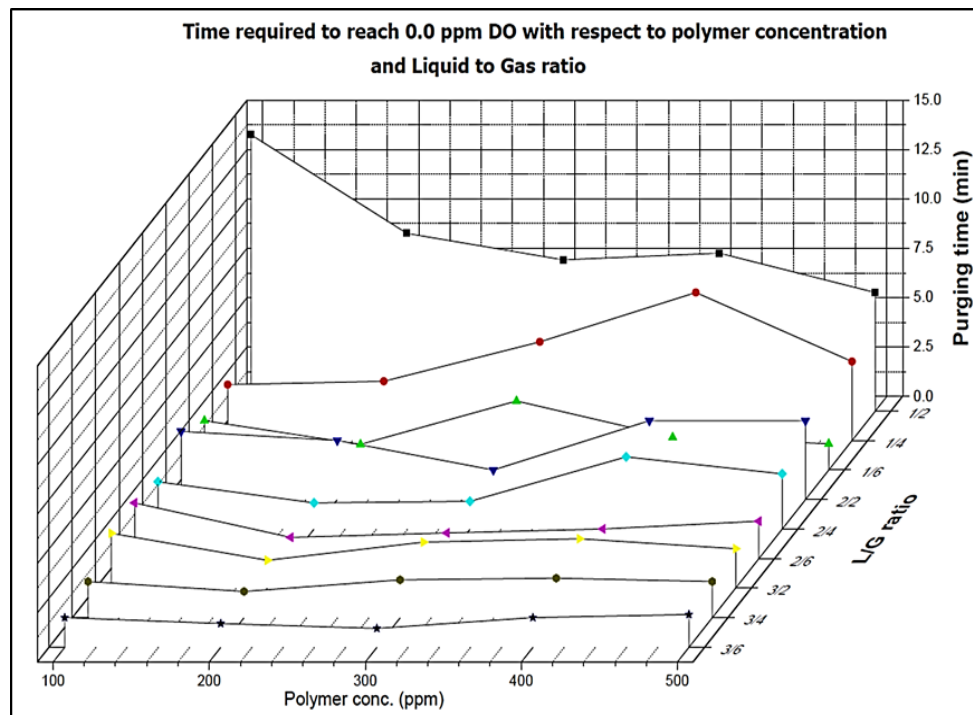


Figure 6: Purging time required to reach zero ppm DO at different liquid/gas ratios applied for different polymer concentrations

It's worth noting that the calculated standard deviation for the time needed to reach zero ppm DO level was shown to be 0.0399 for a randomly selected experimental case, which indicates the certainty of the obtained results, as illustrated in Figure 7.

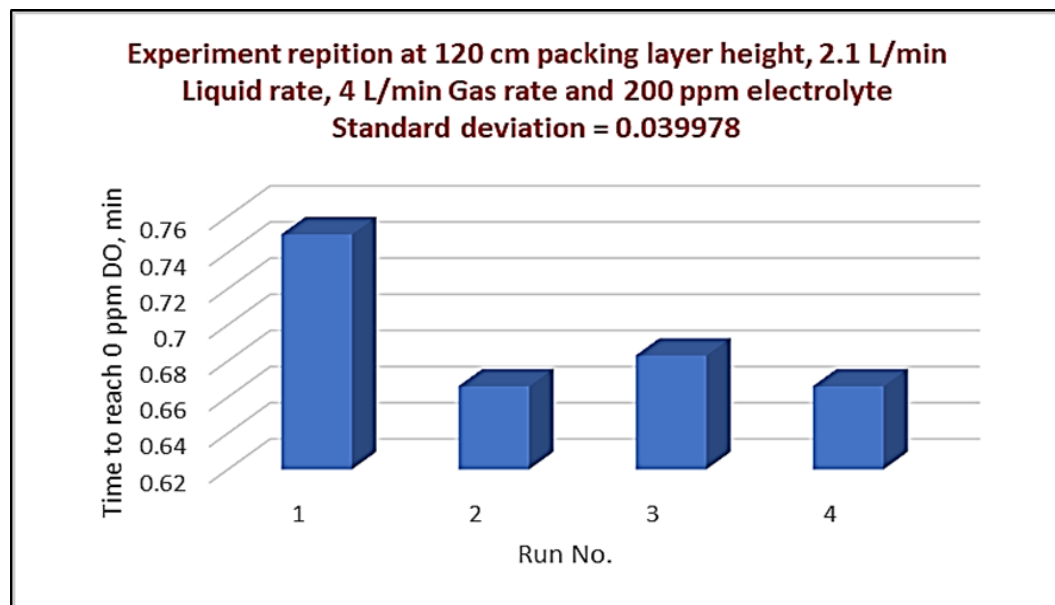


Figure 7: Measured time recorded to reach zero ppm level and the standard deviation calculated for 4 iterations of the studied experimental cases (inline DO measuring mode)

An important observation for the conducted experimental investigation was the formation of foams, especially for higher liquid viscosity. Figure 8 shows pictures of the formed foaming reaching the column top when applying a higher gas flow rate (4 & 6 L/min). The foam formation was reduced and stopped from leaking outside the column top by lowering the liquid flow rate in the cases of 400 ppm and 500 ppm to 2.7 L/min and 2.5 L/min, respectively.

Looking carefully at the results, it can be observed that the stripping became more efficient when holding up liquid in the column, especially at higher liquid flow rates and higher polymer concentrations. The attained integrated effect resembles the semi-batch scheme in terms of efficiency. It could be attributed to an increase in the viscosity of the liquid at the bottom part, which resists the liquid streaming out from the column, offering many opportunities for contact between the gas and liquid streams.

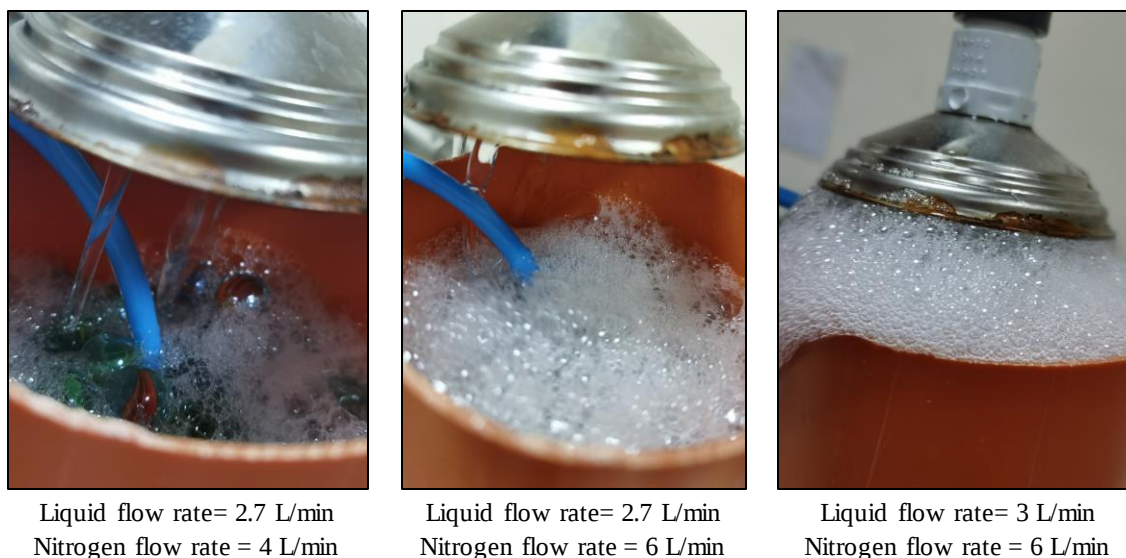


Figure 8: Foam formation when applying higher viscosity liquid (400 ppm polymer)

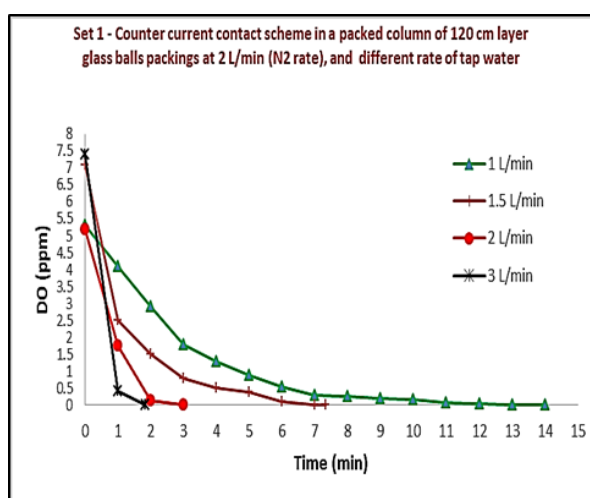
Another important observation is the polyelectrolyte viscosity which was partially reduced when subjected to multi runs of nitrogen purging. The viscosity reductions for various PWP solutions are shown in Table 2, in which huge reductions (more than 40% after 5 experimental runs or 5-10% upon a single run) occurred for higher concentrations of polymer. This is obviously attributed to the partial vaporization of the polymer upon multi-usage.

Table 2: Viscosity was measured for solutions of various polymer concentrations before and after being subjected to nitrogen purging

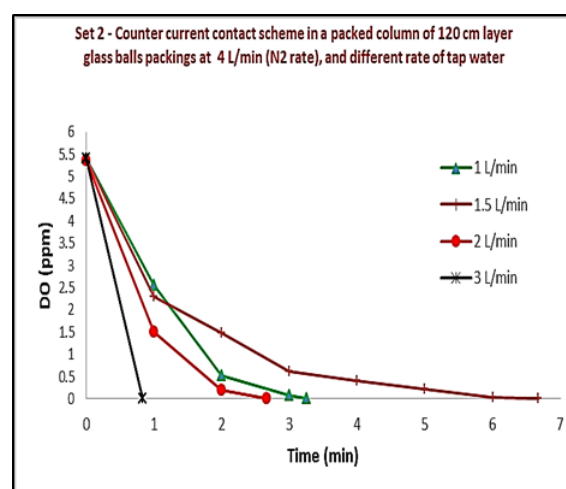
Source of water	Initial Viscosity (mpa.s)	Viscosity after N2 purging (mpa.s)
PW original	3-4	3
PW/polymer 100	8	5
PW/ polymer 200	10	6
PW/ polymer 300	12	7
PW/ polymer 400	15	9
PW/ polymer 500	17	10

Further optimizing investigations were conducted and aimed to measure the fastest period required to reach zero ppm DO. Figures 9 and 10 illustrate that zero ppm DO level was reached in less than 2 minutes for both tap water and produced water, respectively, when applying higher liquid and gas flow rates.

It is noteworthy that when applying lower liquid flow rates, where no holdup liquid is attained, channeling may be generated, causing a longer time for DO removal.

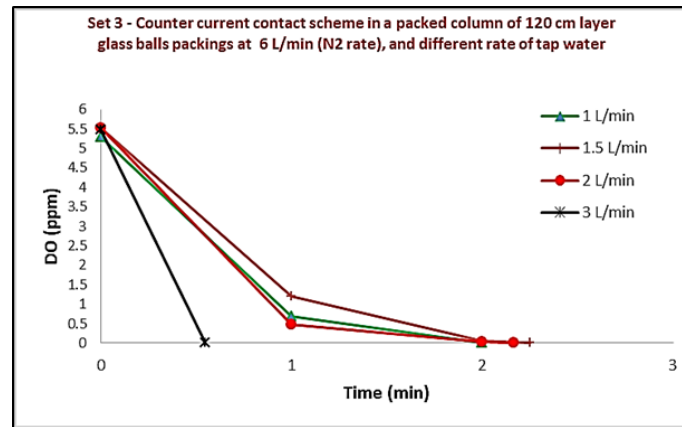


(9-a)

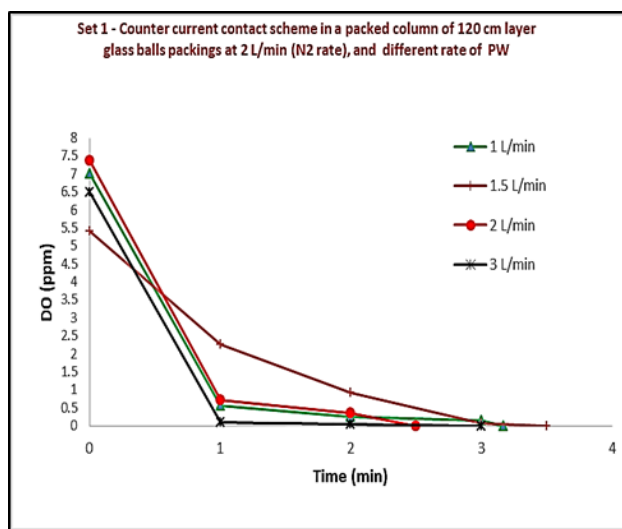


(9-b)

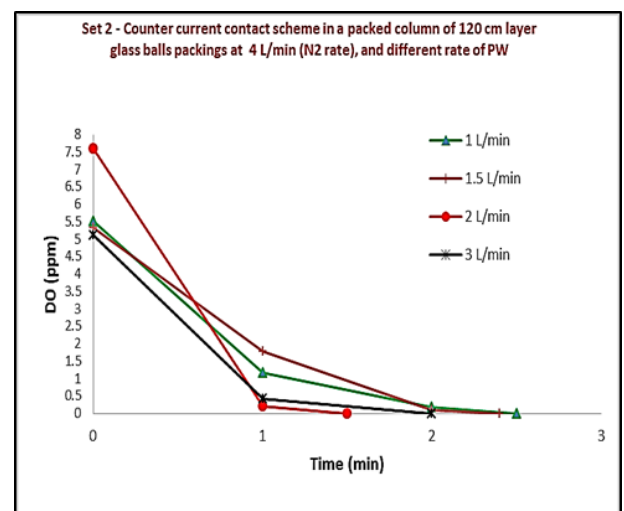
Figure 9:Continued



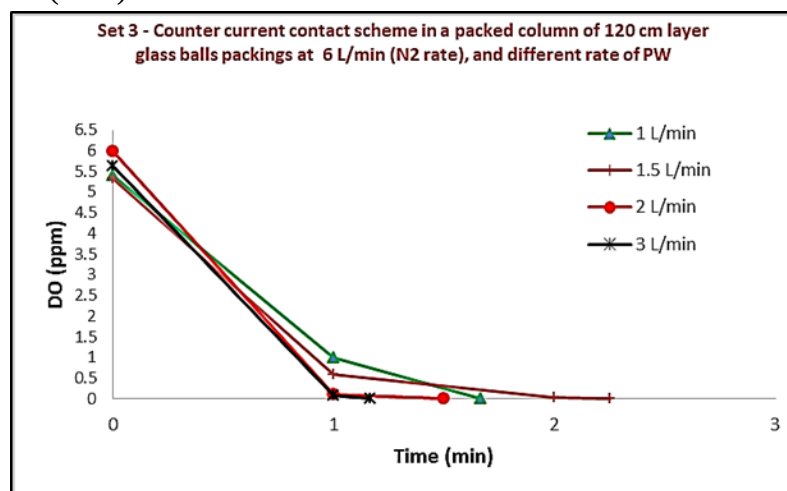
(9-c)

Figure 9: (a-c) Comparison between the results of applying purging for a counter-current contact scheme applied at different liquid flow rates, various N₂ purging rates for examining tap water (inline DO measuring scheme)

(10-a)



(10-b)



(10-c)

Figure 10: (a-c) Comparison between the results of applying purging for a counter-current contact scheme applied at different liquid flow rates, various N₂ purging rates for examining produced water (inline DO measuring scheme)

3.3 Counter Current Purging in A Packed Column Applying Once Through Liquid Flow and Inline DO Measuring Scheme

Based on the unique performance accomplished (i.e., achieving 100% DO removal within 2 minutes in a circulation mode scheme), another challenge was overwhelmed within this part of the study in which we targeted the attainment of 100% removal when applying once through the liquid flow scheme. Two operation modes were tested for tap water, and PW grafted with polyacrylamide (PWP). In the first instance, tap water was streamlined to the column top at 2 L/min simultaneously with nitrogen entered to the column bottom at different purging flow rates. In the second approach, the column was purged with nitrogen for a few minutes to ensure total oxygen removal from the system. Then, tap water was streamlined to the column top while continuing nitrogen purging from the column bottom. The results for the two cases are illustrated in Figures 11 and 12. It can be observed that the second approach has resulted in faster DO removal and could be attributed to the shrinkage that occurred in the oxygen content before the contact between the liquid/gas streams.

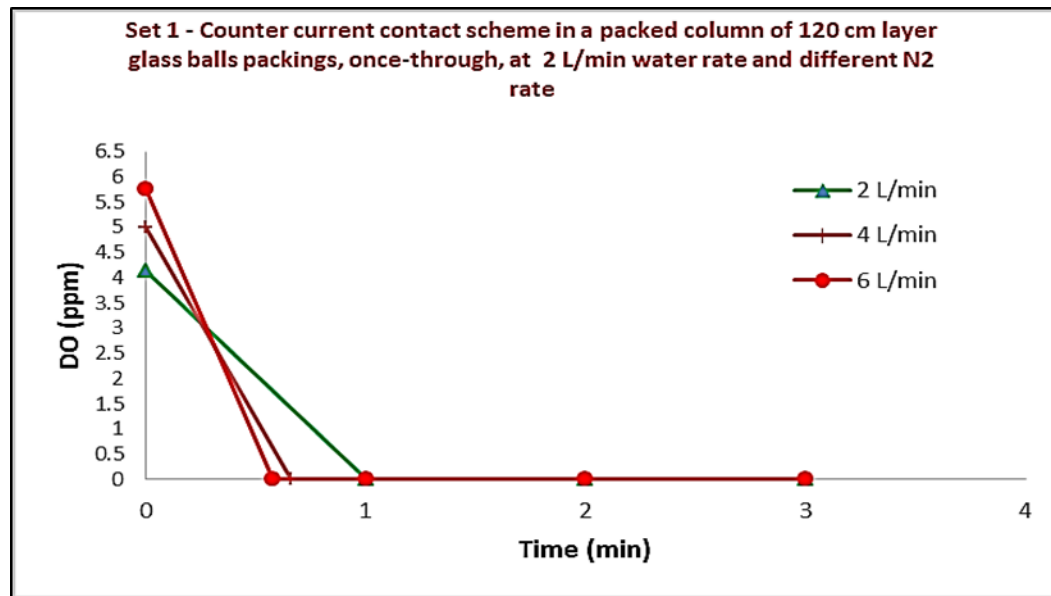


Figure 11: Applying once through counter current contact scheme for tap water without pre-nitrogen purging, and inline DO measurement

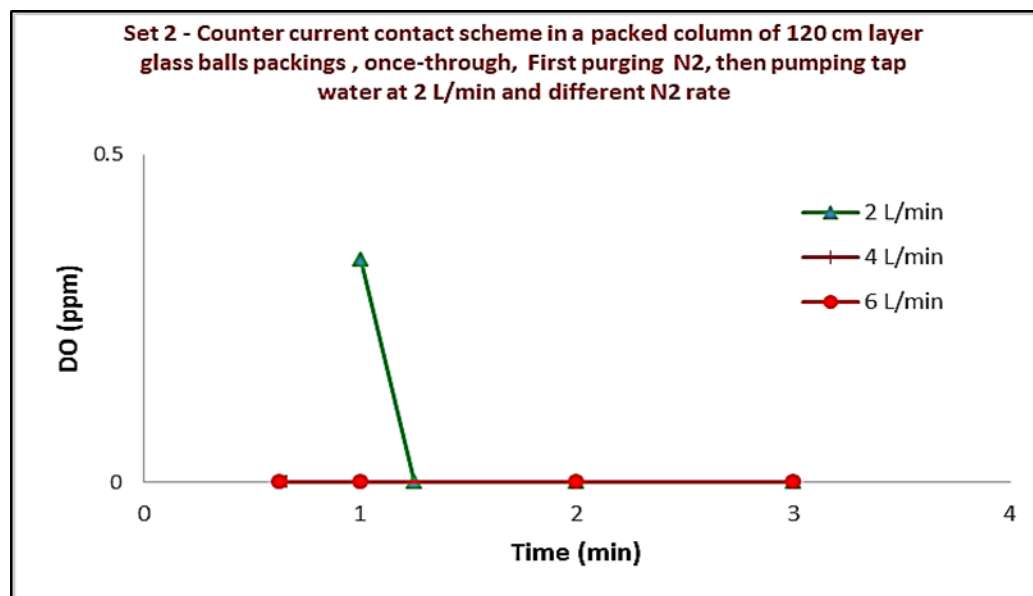


Figure 12: Applying once through counter current contact scheme for tap water with pre-nitrogen purging, and online DO measurement

The investigations were further extended to include PWP samples (produced water grafted with polyacrylamide). As previously mentioned, the viscosity of the solutions is reduced proportionally after being subjected to N₂ purging. Hence, we adopted the measured viscosity for each applied solution for terminology rather than only considering the grafted polymer concentration. In this sense, the results of applying different experimental schemes are illustrated in Figures 13 to 18.

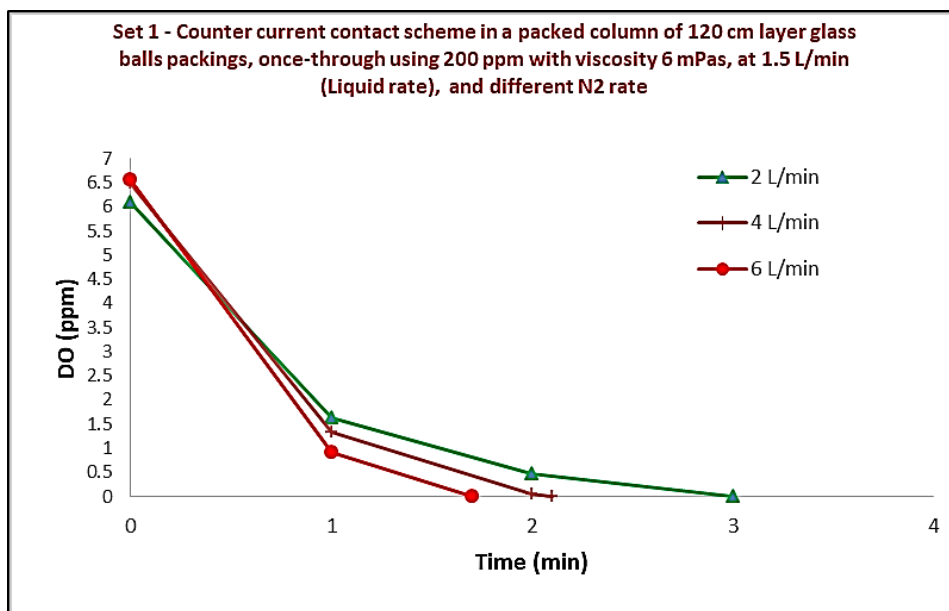


Figure 13: Applying once through counter current contact scheme for a PWP grafted with 200 ppm polymer (measured viscosity = 6 mPa.s) without pre-nitrogen purging, and inline DO measurement

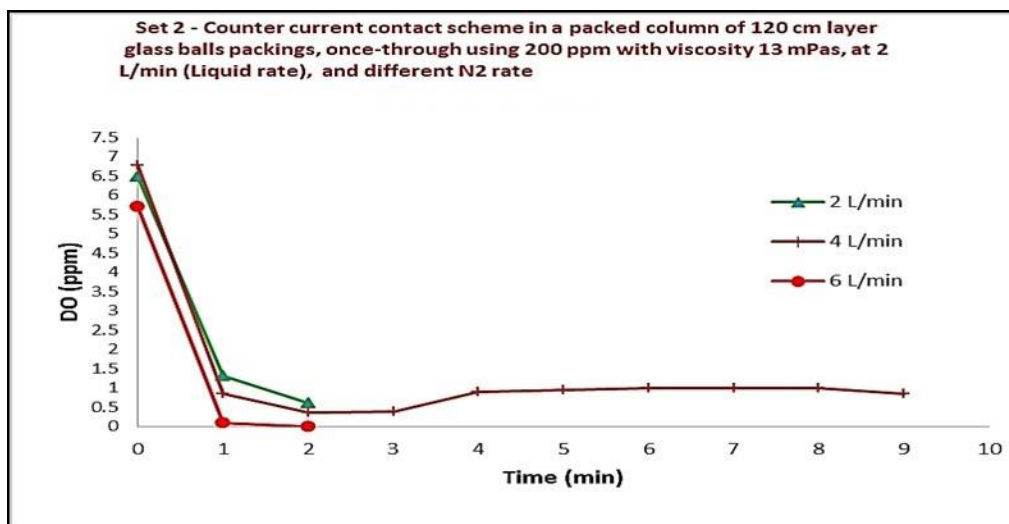


Figure 14: Applying once through counter current contact scheme for a PWP grafted with 200 ppm polymer (measured viscosity = 13 mPa.s) without pre-nitrogen purging for 2 & 4 L/min, and with pre-nitrogen purging for 6 L/min (inline DO measurement)

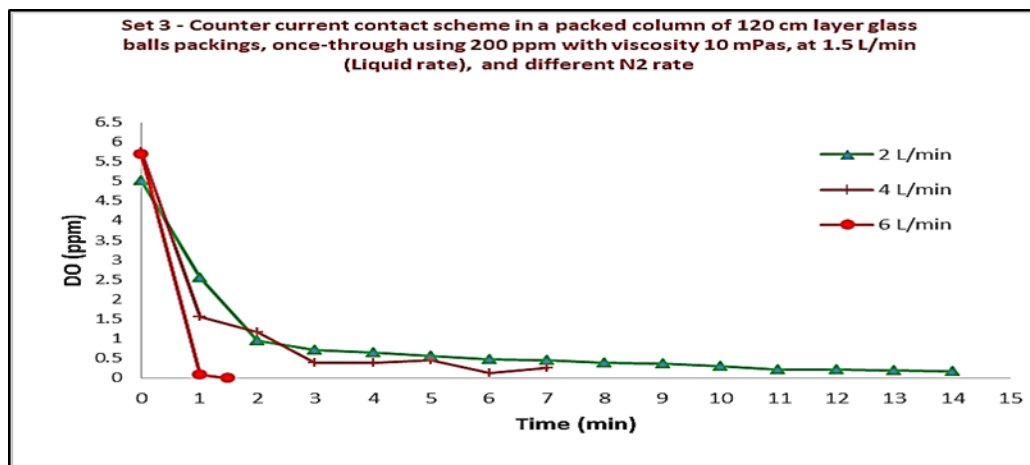


Figure 15: Applying once through counter current contact scheme for a PWP grafted with 200 ppm polymer (measured viscosity = 10 mPa.s) at 1.5 L/min PWP flow, and inline DO measurement

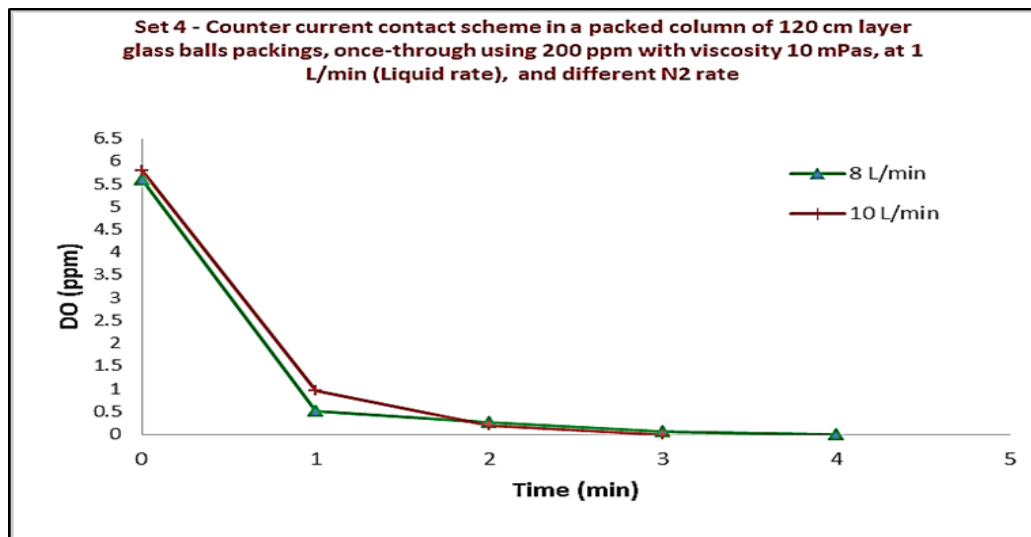


Figure 16: Applying once through counter current contact scheme for a PWP grafted with 200 ppm polymer (measured viscosity = 10 mPa.s) at 1.0 L/min PWP flow, and higher nitrogen flow rate (inline DO measurement)

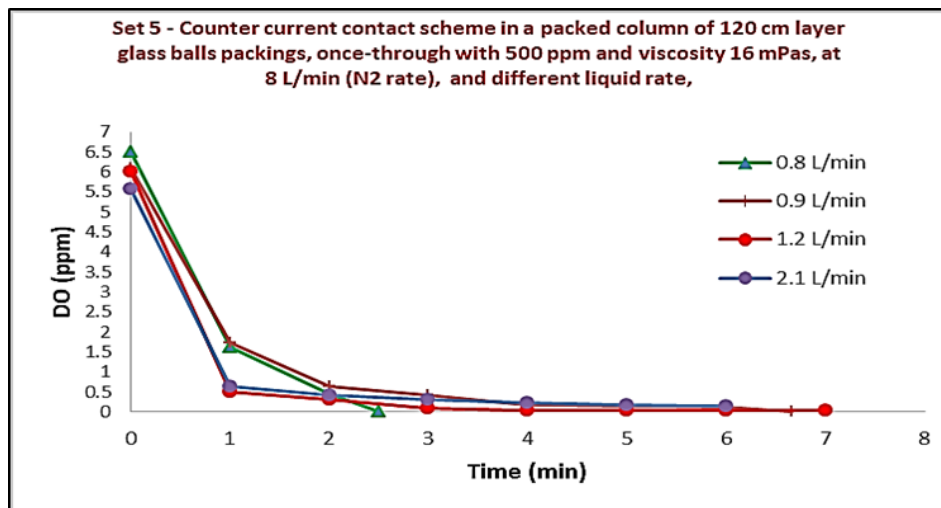


Figure 17: Applying once through counter current contact scheme for a PWP grafted with 500 ppm polymer (measured viscosity = 16 mPa.s) at 8.0 L/min nitrogen flow, and different liquid flowrates (inline DO measurement)

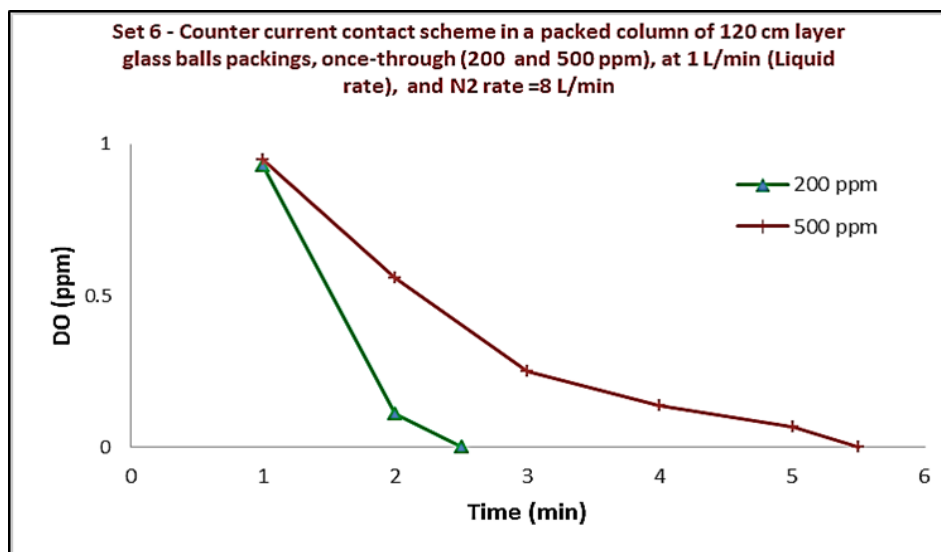


Figure 18: Applying once through counter current contact scheme for a PWP grafted with 200 & 500 ppm polymer at 8.0 L/min nitrogen flow and 1 L/min liquid flow rates (inline DO measurement)

It can be concluded that when the solution viscosity increases, higher gas flow rates and lower liquid flow rates will affect properly reaching zero ppm level in a shorter purging duration.

To sum up, the attained results from all investigated protocols in the current phase [Part 2] are accounted highly promising and competitive to the results reported in the literature [15], in which DO level approached zero ppm (ppb level) within not less than 20 minutes of nitrogen purging. This makes it worth to be investigated on an industrial scale. Hence, the next phase of the study will be dedicated to examining the effect of applying finer bubble size on the DO removal efficiency in a pilot-scale gas lift tower for industrial applications.

4. Conclusions

In this work (part 2 of the study), expanded experimental investigations were conducted to examine the nitrogen purging scheme for de-oxygenation-produced water grafted with different polyacrylamide concentrations. The investigated samples resemble extreme cases PW specimens commonly found in the oil industry. The adopted scheme proved useful in all examined cases, where zero ppm level of DO residues was reached within 2 minutes of stripping start. The nitrogen purging approach was also proved effective when applying the once-through scheme, which is important for extreme treatment cases. The outcomes of this study indicate the feasibility of adopting the nitrogen purging scheme for produced water de-oxygenation on an industrial scale.

Acknowledgments

The authors thank Petroleum Development Oman (PDO) at the Sultanate of Oman for sponsoring this project (project ref. UCT8).

Funding

Research is funded by Petroleum Development Oman (PDO) at the Sultanate of Oman (project ref. UCT8).

Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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