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A twin chamber up-flow bio-electrochemical pumparound system for sequential nitrification and denitrification of reject water

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ABSTRACT

In this study, a modified twin chamber up-flow bioelectrochemical reactor (TCUBER) was investigated during sequential bioelectrochemical nitrification and denitrification of reject water experimentally. A twin chamber up-flow bioelectrochemical pump-around system (TCUBEPS) makes it possible to investigate the effects of different pH values (6.5 ± 0.1, 7.5 ± 0.1, 8.5 ± 0.1, and 9.5 ± 0.1) on the system efficiency at different applied current intensities (I = 20–50 mA) and HRT (6–24 h) values. The results show that the pump-around system succeeded to stabilize the pH and the most favorable values of pH for the activation of nitrifying and denitrifying bacteria in both compartments were 7.5 and 8.5, respectively, whereas the removal percentage of NH_4-N was 96% at higher applied HRT (24 h) and constant I (50 mA). This study contributed to a better understanding of the function of variables, namely HRT, pH, and I to treat reject water with high concentration of ammonium.

Keywords: Reject water; Nitrification; Denitrification; Autohydrogenotrophic denitrification; Bio-electrochemical; Pumparound system

1. Introduction

The inappropriate discharge of untreated and treated wastewater (e.g. reject water) containing high concentration of inorganic nitrogen, such as ammonium, nitrate and nitrite into receiving waters leads to an excessive growth of algae and eventually promotes eutrophication of lakes and rivers and decreases the quality of drinking water resources [1,2].

In order to protect consumers from the unpleasant effects of nitrogen components, standards have been established by different organizations for different concentration levels of nitrogen in drinking-water and discharged wastewaters into receiving water. The World Health Organization in 1985 declared that the acceptable concentration of nitrate in drinking water to be 10 mg N/l [3–5]. The US Environmental Protection Agency set the maximum contaminant level of nitrate at 10 mg N/l [6,7]. The acute and chronic toxicities of NH_3-N are 0.05–0.35 mg/L and 0.01–0.02 mg/L for the protection of sensitive aquatic animals [8]. Therefore, to

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achieve the standards mentioned above and to decrease the release of nitrogen components, different methods for water and wastewater have been accomplished.

Authors have formerly argued about different biotic and abiotic methods applied to eliminate nitrogen, and also noted superiority associated with autotrophic bacteria using bioelectrochemical method (BER) [1]. As mentioned by researchers, hydrogen gas preparation for biomass can be performed by two main methods: the injection of hydrogen gas into the reactor was the initial method of H_2 supply to autohydrogenotrophs, which was found to have a limitation, mainly due to the low solubility of H_2 in aqueous solution (1.6 mg/L at 20°C). This reduces accessibility and contact time for microbial reactions, thus resulting in lower nitrate removal rates compared to heterotrophic denitrification. Other drawbacks in the application of hydrogen are the costly supply of hydrogen gas, flammability, and explosive mixtures of H_2 with air, transportation, and storage problems. *In situ* generation of hydrogen was suggested as an effective technique to overcome the above problems [1,9,10].

The theory of BER has been well described in a previous published review, which allowed simultaneous or sequential nitrification and denitrification (SND) due to the production of suitable electron donors by cathode reaction (H_2) and electron acceptors by anode reaction (O_2) as expressed in Eqs. (1) and (2) [1,9,10]:

$$5H_2O \rightarrow 2.5O_2(g) + 10H^+ + 10e^- \quad (1)$$

$$10H_2O + 10e^- \rightarrow 5H_2(g) + 100H^- \quad (2)$$

Furthermore, simultaneously generation of oxygen and hydrogen in this reactor makes possible to run reactor as SND. Eqs. (1) and (2) show the electrolysis of water, where oxygen and hydrogen gases are produced on the anode and cathode, respectively. The two-step reduction processes of ammonium to nitrate are show in reactions (3) and (4), which follows by sequential reactions of nitrate reduction to nitrogen gas according to reactions (5)–(8). Moreover, the net reaction (Eq. (9)) indicates that the reduction of 1 mol nitrate to nitrogen gas needs 5 mol electron [7,11–13]:

Steps of ammonium oxidation to nitrate:

$$NH_4^+ + 3O_2 \rightarrow 2NO_2^- + 2H_2O + 4H^+ \quad (3)$$

$$2NO_2^+ + O_2 \rightarrow 2NO_3^- \quad (4)$$

Sequential reactions of nitrate reduction to nitrogen gas:

$$NO_3^- + H_2(g) \rightarrow NO_2^- + H_2O \quad (5)$$

$$NO_2^- + 0.5H_2 + H^+ \rightarrow NO(g) + H_2O \quad (6)$$

$$2NO(g) + H_2 \rightarrow N_2O(g) + H_2O \quad (7)$$

$$N_2O(g) + H_2 \rightarrow N_2(g) + H_2O \quad (8)$$

Net reaction:

$$2NO_3^- + 6H_2O + 10e^- \rightarrow N_2(g) + 12OH^- \quad (9)$$

A twin chamber up-flow bioelectrochemical reactor (TCUBER) was developed in previous investigation. Former investigation showed a significantly suitable elimination on nitrogen components when reactor ran without pump-around system at semi-batch mode [14]. Nevertheless, the application of this integrated process has been limited in practice because of its complexity and energy consumption [15]. Most previous studies on bioelectrochemical systems focused on the denitrification of contaminated synthetic water and wastewater, which is much easier to operate than sequential nitrification and denitrification in a BER for reject water treatment. Advantages of this modified reactor over previous reactors are the control of pH by a pump-around system for both compartments to cope with the problem of pH decrease in nitrification chamber, using palm shell granular activated carbon (PSGAC) as third electrode and biocarrier and applying this system to treat reject water with high concentration of ammonia. In addition to the main operational variables (HRT and I), by sequential nitrification and denitrification of reject water in continuous mode, the effect of pH values on system efficiency was also investigated.

2. Material and methods

2.1. Experimental set-up

The applied TCUBEPS in this research work has been used as TCUBER without pump-around system (PS) in previous authors research work to treat synthetic wastewater without controlling of pH [14]. Reactor was built up in two columns from Plexiglas at effective liquid volume of 1.8 L for each column (42 cm in height with the inner diameter of 10 cm). Each column was equipped with a mesh plate stainless steel at the bottom

(type 316) and 12 stainless steel rods (40 mm in height) to enhance the supply of electricity in the palm shell-GAC (22 cm in high) as third electrode in anode and cathode compartment (Fig. 1).

A Nafion 117 cation permeable membrane (Dupont Wilmington, DE) was inserted between anode and cathode compartment through a flange and O-ring type joint. The applied O rings at the bottom and middle of chambers provided the possibility of maintaining and replacing of plates and membrane. The electrodes and Nafion were pretreated and cleaned before installing and after each run in all experiments according to applied method by Chae et al. [16].

The reactor was equipped with a pump-around system for anode and cathode compartments under the continuous mode (Fig. 1). The main object of applying pump-around system was to adjust of the pH at the wanted values (6.5 ± 0.1, 7.5 ± 0.1, 8.5 ± 0.1, and 9.5 ± 0.1) using two set of fermenters, which were equipped with DO meter (METTLER TOLEDO, O_2-sensor, Switzerland) and pH meter (METTLER TOLEDO, pH-sensor, Switzerland). The pH adjustment was carried out through automatic injection of acid (H_2SO_4; 1 N) or alkaline (NaOH; 1 N) solution by controllers. Experiments were conducted at a current intensity of 50 mA and HRTs of 6, 12, and 24 h at mentioned pH values in both compartments. After finding out the best value of HRT and pH, the efficiency of anode and cathode compartment is investigated at different values of current intensity.

2.2. Biomass and wastewater components

The mixed culture of acclimatized nitrifying bacteria and hydrogenothrophic denitrifying bacteria, which originated from Pantai Dalam Sewage Treatment Plant IWK inoculated into the TCUBEPS with initial mixed liquor suspended solids (MLSS = 3,000 mg/L), through different sampling ports from the opposite sides of each compartment.

Reject water was collected from Pantai Dalam Sewage Treatment Plant IWK, Pantai Dalam, Kuala Lumpur, Malaysia. The wastewater was relatively strong, which the evaluations of chemical and physicochemical parameters are shown in Table 1 (after five repetitions). The TUBEPS was fed from the inlet of anode compartment with reject water according to Table 1. The effluent of first compartment was used to feed cathode compartment continually.

2.3. Analytical methods

The samples were analyzed immediately or were stored at cold temperature (4°C) prior to analysis. The

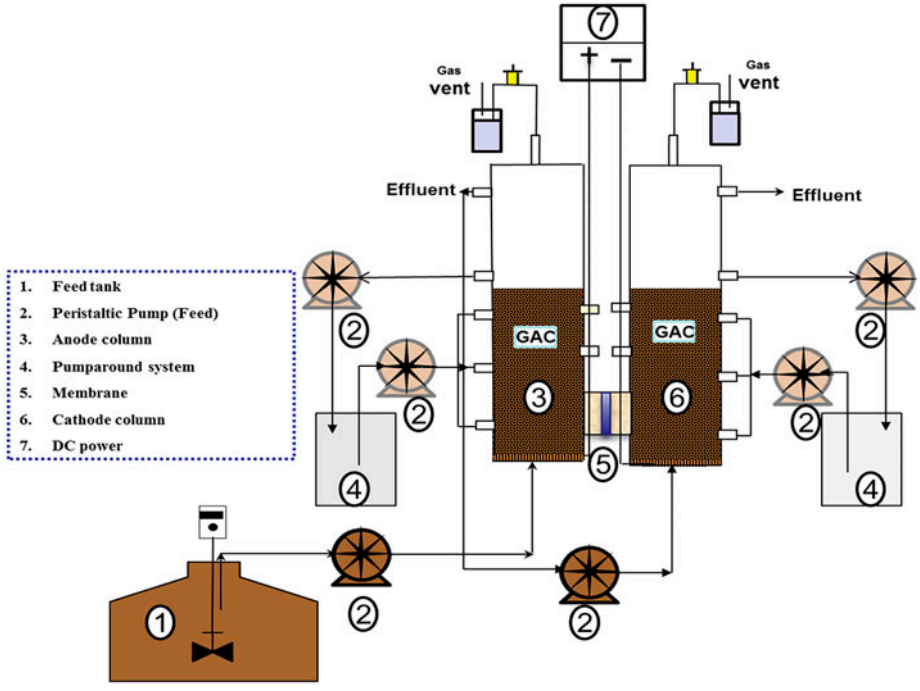

Fig. 1. Schematic of a twin chamber up-flow bio-electrochemical pump-around system (TCUBEPS).

Table 1
The characteristics of reject water

Constituents	Mean value (mg/L)	SD[a]
COD	160	±30
Alkalinity	750	±50
Conductivity	1,725	±120
PH	7	±0.2
NH_4^+-N	200	±20
NO_2^--N	5	±2
NO_3^--N	10	±3
TKN	200	±25
TSS	124	±16
VSS	114	±11
PO_4^--P	85	±10

[a]SD: Standard of deviation.

samples for the determination of dissolved components, e.g. ammonium, nitrate, and nitrite concentration, were analyzed using an Advanced Compact Ion Chromatograph IC 861 (Metrohm® Ltd, Herisau, Switzerland). Samples were filtered before the analysis using a 0.2 μm filter. Temperature, pH, and DO were monitored continuously online. In addition, other experimental tests were determined using standard methods [17]. The repetition of analysis was considered when an error higher than 5% was observed in the samples during the experiment. The ability of system for ammonium removal can be expressed in term of current efficiency (CE) according to Eq. (10) [14,18,19]:

$$CE = \frac{[Q(C_{inf} - C_{eff})] \times n}{1/F} \times 100 \quad (10)$$

where Q is the volumetric flow (cm^3/s), C_{inf} and C_{eff} are the concentration of ammonium in influent and effluent (mol/cm^3), n is the stoichiometric coefficient (2), and F is Faraday's constant (96,485.3365 C/mol).

3. Results and discussion

3.1. Effect of pH

Results showed that pH perceptibly affected nitrification and denitrification by controlling the growth and activity of nitrifying and denitrifying bacteria. At the first stage of experimental works, the reject water treatment was achieved under different pHs and HRT values at a constant I value. The results showed that pH is related to many factors to achieve successful nitrification and denitrification. Therefore, finding the optimum range of pH is important in SND. Previous studies reported that the optimum ranges of pH value for nitrifying and denitrifying bacteria are in ranges of 7.5–8 and 7–8, respectively [20,21]. The pH values that were applied higher and lower than the aforementioned values indicated a significant deterioration in process efficiency. The nature of nitrification and denitrification, electrolysis of aqueous solutions as a function of HRT and I, and the mode of flow (plug flow mode) make pH control more complicate in this integrated biological and electrochemical system. In the present study, an attached system was employed with the up-flow mode. Hence, at the same operational conditions without applying the pump-around system and pH controllers in both columns, the collected samples from different sampling ports showed different pH values when an abiotic test has been conducted. The result of the pH value monitoring during 24 h showed that adjusting pH to 7.8 was impossible. The pH in the anode zone decreased to 7.1, and in the cathode zone elevated from 4.6 to 9.7, both of them are undesirable pH values for nitrification and denitrification. Therefore, controlling of pH in reactor by adjusting the injection of acid and alkaline through a pH controller "pump-around system" was considered as the main challenge for improving the reactor performance. Results showed that the system has the ability to control pH at wanted values at a constant circulation of effluents from each compartment (20 mL/min) and injection of different values of acid and alkaline.

Fig. 2 shows that the most favorable value of pH for the high level activation of nitrifying and denitrifying bacteria in both compartments was 7.5–8.5, whereas the percentage removal of NH_4-N was 96% at higher applied HRT (24 h) and constant I (50 mA). As noted, the nitrification process was pH sensitive and the ammonium consumption rate declined significantly to 33% at pH 6.5 because the nitrifying activity showed a tendency to fall within the low pH range.

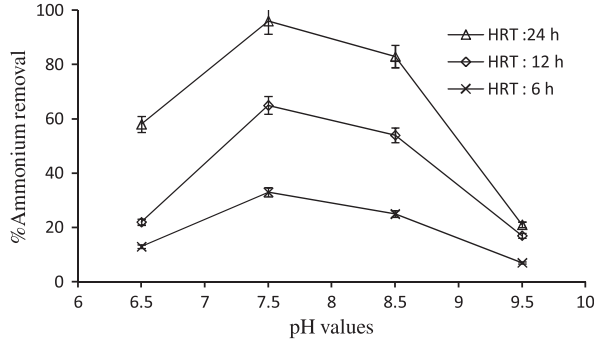

Fig. 2. Ammonium removal during the performance of TCUBEPS at different pH value. Condition: HRT (6, 12, and 24 h), I (50 mA) (Y error bar is less than 5%).

Meanwhile, the activity of the nitrifiers was inhibited when pH increased to 9.5, thereby decreasing nitrification rate and reducing the efficiency of ammonium oxidation to 21%, which is the lowest among the experiments at HRT = 24 h.

The findings of this part of research work was in agreement with the reports by previous researchers that nitrification will be limited at pH values <6.5 or >10 [22–24].

Regarding the denitrification in cathode compartment by autohyrogenotrophic denitrifying bacteria, during this stage, fluctuations of the ammonium oxidation efficiency were cause of different observations for nitrate and nitrate concentrations in the effluent at different pH values. The concentrations of nitrate and nitrate in the effluent were low at pH 6.5 in all runs because of the decreased consumption of ammonium (Fig. 2). Based on theoretical oxidation of ammonium to nitrite and nitrate through nitrification, the rate of denitrification was low as compared to when pH was increased to 7.5 and then to 8.5. This finding indicates significant changes in the concentrations of nitrite and nitrate in the effluent at HRT = 24 h between days 77 and 94.

The cathode zone was more alkaline, particularly when the pH level was 9.5, indicating less efficiency of the system at HRT = 24 h and increasing the discharge of intermediate (NO_2) from the reactor, which could be the result of incomplete nitrification and denitrification processes. Except for the unsuitable value of pH, the inefficiency of the system could be attributed to the disintegration of the carbon source (carbonate ions) that limited the carbon [25].

3.2. Effect of hydraulic retention time

In addition to the adequate amount of oxygen and hydrogen that are required to cope with the susceptibility of the nitrification and denitrification processes, an appropriate HRT should also be included to provide micro-organisms with sufficient reaction time. Fig. 3 demonstrates the significant effect of HRT on system efficiency. The concentration of ammonium in the effluent decreased from 135.3 to 7.1 mg NH_4-N/l as HRT was increased from 6 to 24 h, corresponding to a total removal efficiency of more than 96% at a total HRT of 24 h, which this result is in agreement with achieved results of author's previous research works. The results showed that the novel twin-chamber up-flow bio-electrochemical reactor had ability to remove 95% of ammonium at current intensity (50 mA) and HRT (24 h) [14]. The obtained results revealed the positive effect of increasing HRT on the consumption of ammonium in different applied pH values.

Fig. 3 shows that the removal efficiency of nitrogen components in the effluent was dependent on HRT through the bioelectrochemical process, which has been confirmed by researchers at the previous studies [14,18]. Nitrate and nitrite removal decreased as flow rate increased from 75 to 300 ml/h, when HRT was 24 and 6 h. This finding may be attributed to an increase in the nitrogen loading with a shorter HRT while oxygen and hydrogen generation rate did not change under constant electric current. At a flow rate of 300 ml/h, the enhancement of system efficiency was insignificant, whereas the concentrations of ammonium, nitrate, and nitrite in the effluent increased consistently. The SND rate likewise reached to its maximum at a flow rate of 75 ml/h at HRT 24 h, when the concentrations of nitrate and nitrite decreased in the effluent. This result indicates that at HRTs <12 h, the sequential nitrification and denitrification rates were limited by the flow rate. However, beyond this point, the nitrification and denitrification rates were decreased by the oxygen and hydrogen gas concentrations. Thus, a further increase in the flow rate would only lead to the incomplete nitrification and denitrification as well as the accumulation of nitrite in both compartments. Therefore, longer retention time could guarantee high removal rate [19]. By contrast, shorter retention time leads to less efficiency for ammonium oxidation, NO_2-N accumulation in the system, and increased nitrite concentration in the effluent.

3.3. Effect of current intensity

This part of experimental works was conducted at different values of I (20, 35, and 50 mA) at the best pH and HRT values to treat reject water in a TCU-BEPS (Fig. 3). The results indicate that nitrification proceeded within the anodic biofilm by utilizing the oxygen derived from the electrolysis in steady state during all of the experiments. The amounts of produced nitrite and nitrate were significantly dependent on the current intensity. The nitrification rate, as well as nitrate and nitrite concentrations in the effluent, increased with an increase in the current value. Moreover, the results indicated that the ammonium oxidation decreased at low I (20 mA). Consequently, nitrate and nitrite concentrations were very low at the effluent of system. However, at a higher I of 50 mA (the voltages were 9.01–12.11 V, with an average of 10.42 V), significant improvement in the nitrification process performance could be observed because of the

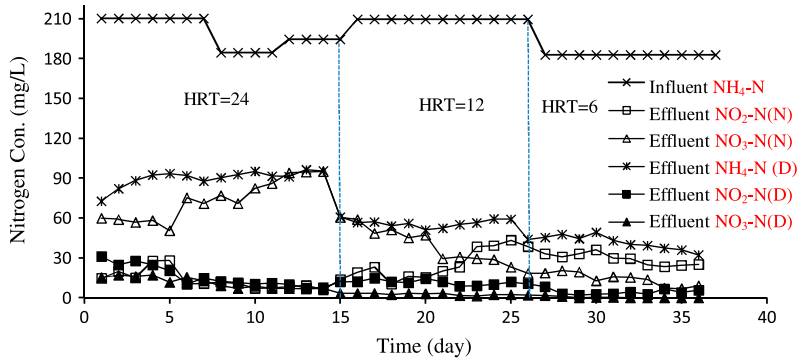

Fig. 3. Accumulation of nitrite, nitrate and ammonium removal during the performance of TCUBEPS at flow rate (75, 150, and 300 mL/h), condition: pH (7.5), *I* (50 mA), (N: nitrification; D: denitrification).

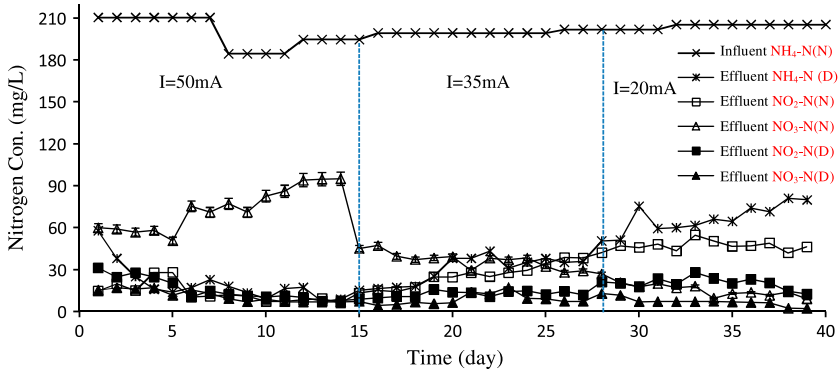

Fig. 4. Effect of current intensity on nitrification and denitrification performance. Conditions: HRT (24 h), *T* (29 ± 1), pH 7.5 (*Y* error bar is less than 5%), (N: nitrification; D: denitrification).

high oxygen generation(about 3 mg/L) that was mentioned in the previous studies [14,15]. The nitrite accumulation at a low *I* (20 mA) was more than when higher *I* was applied in the reactor and differed from the results obtained at the previous author's research work when carbon was prepared continually by using $NaHCO_3$ for SND in a TCUBER [14]. This difference could be attributed to the lack of carbon source in reject water and the lack of O_2 and H_2 production in nitrification and denitrification chambers at the low current. Moreover, adjusting the current value was found to be beneficial in achieving partial nitrification at lower *I* values.

As shown in Fig. 4, the results obtained differed from those in former study, where denitrification was improved with increasing *I*. At this stage, the efficiency of the system decreased at *I* of 50 mA because of high ammonium oxidation, which was cause to increase of nitrite and nitrate concentrations in the effluent. This outcome could be explained by the so-called oxygen depression effect. The high concentration of oxygen (0.5–1 mg/l) in the effluent of the nitrification compartment had an adverse effect on the denitrification process and inhibited the activities of denitrifying bacteria at the beginning of process.

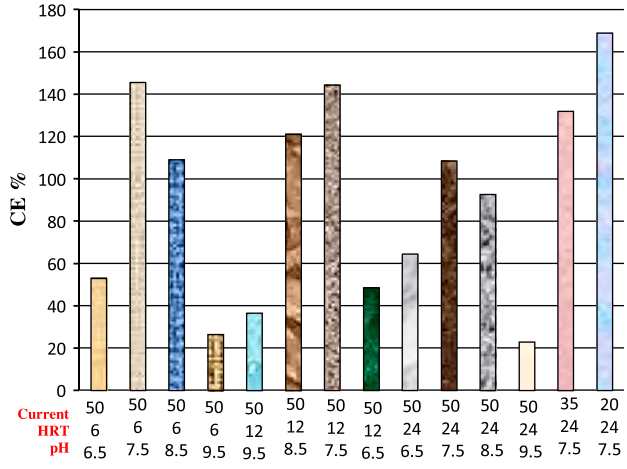

Fig. 5. The system CE during different runs.

Table 2
Comparison for operational parameters (Current intensity, pH, and HRT) achieved in different studies

Reactor type	Influent nitrogen (mg/l)	Current intensity	pH	HRT (h)	Removal	Refs.
DCBER-CB[a]	49 ± 0.8	0.5 (mA/cm^2)	≈7	48	56%	[10]
BER-ASE[b]	TN: 37.2–68	80 (mA)	NA	6	75%	[11]
TCUBER	200	50 (mA)	≈7	24	95%	[14]
S-BER[c]	TN = 50	0.3 (mA/cm^2)	NA	10	5 g N/m^2/d	[15]
MC-BER[d]	15–40	40 (mA)	≈7	6	86.5%	[18]
TCUBEPS	200 ± 20	50 (mA)	7.5	24	96%	Present study

[a]Divided cell bio-electrochemical reactor packed with Celite biocarrier.
[b]Biofilm-electrode reactor and activated sludge-electrode.
[c]Single BER.
[d]Multi-cathode BER.

However, the efficiency of the system increased after several days (between days 5 and 15). The adverse effect could be due to the shock caused by the presence of oxygen at the bottom of the cathode zone, but the biological consumption and electrochemical reduction of DO may result in the formation of an anoxic region in the cathodic biofilm [15]. A run with same environmental and operational condition without the application of electric current was conducted and its results showed that nitrogen removal was negligible.

The CE of TCUBEPS was calculated using various values of *I*, HRT, and pH for ammonium elimination according to Fig. 5. However, with an increase in *I*, the efficiency of the system for ammonium removal gradually increased because of high production of O_2, but applying low values of HRT and *I* at constant pH value (7.5) leads to high CE. The values higher than 100%, as mentioned in previous studies [14,19,26], for the obtained results cannot be sufficient; it is probably due to some remained oxygen from previous runs, which trapped in PSGAC bed. Furthermore, results revealed that at low (6.5) and high (9.5) value of pH the CE was more than 100% due to decrease of bacteria activity and subsequently accumulate of O_2 in reactor.

Some novel bioelectrochemical reactors (BER) with different configurations have been reported within the last decades, which amongst rarely exploited the GAC as cathode material or biocarrier. Furthermore, only a few novel reactors have focused on nitrification and denitrification, simultaneously. In this case, BERs were applied for syntactic water and wastewater at low concentration of nitrogen components compared to the employed TCUBEPS in this study. Therefore, their functions and results are presented in Table 2 to make a comparison.

4. Conclusion

As earlier investigation by applying, the TCUBER showed that pH decrease can be cause of nitrification rate decrease, by this present research work was revealed that a fitting modification reactor as TCUBEPS can dominate on mentioned shortcomings. The equipped anode compartments with a pump-around system make possible the pH adjustment by pH controllers. Results showed that the pump-around system succeeded to stabilize the pH and the most favorable value of pH for the nitrifying and denitrifying bacteria was 7.5 and 8.5, whereas the percentage removal of NH_4^+-N was 96% at higher applied HRT (24 h) and constant *I* (50 mA).

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