

# Electrolytic treatment of Standard Malaysian Rubber process wastewater

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## Abstract

A new method of Standard Malaysian Rubber (SMR) process wastewater treatment was developed based on in situ hypochlorous acid generation. The hypochlorous acid was generated in an undivided electrolytic cell consisting of two sets of graphite as anode and stainless sheets as cathode. The generated hypochlorous acid served as an oxidizing agent to destroy the organic matter present in the SMR wastewater. For an influent COD concentration of 2960 mg/L at an initial pH  $4.5 \pm 0.1$ , current density  $74.5 \text{ mA/cm}^2$ , sodium chloride content 3% and electrolysis period of 75 min, resulted in the following residual values pH 7.5, COD 87 mg/L, BOD<sub>5</sub> 60 mg/L, TOC 65 mg/L, total chlorine 146 mg/L, turbidity 7 NTU and temperature  $48^\circ\text{C}$ , respectively. In the case of 2% sodium chloride as an electrolyte for the above said operating condition resulted in the following values namely: pH 7.2, COD 165 mg/L, BOD<sub>5</sub> 105 mg/L, TOC 120 mg/L, total chlorine 120 mg/L, turbidity 27 NTU and temperature  $53^\circ\text{C}$ , respectively. The energy requirement were found to be 30 and 46 Wh/L, while treating 24 L of SMR wastewater at 2 and 3% sodium chloride concentration at a current density  $74.5 \text{ mA/cm}^2$ . The observed energy difference was due to the improved conductivity at high sodium chloride content.

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**Keywords:** Electrolytic treatment; Standard Malaysian Rubber process wastewater; Hypochlorous acid; Chlorine; In situ oxidation; Electrochemical

## 1. Introduction

The agro-based industrial sector played a very significant role in Malaysia's economy until 1980s. Thereafter, due to industrialization large tracts of rubber land which were initially located in the outer periphery of urban areas were converted for industrial, commercial and supporting residential uses. The above changing scenario in the last two decades was responsible for a gradual decline in raw natural rubber production, and Malaysia lost its position as the world's largest natural rubber producer which it held for the past many decades. Presently, Malaysia is the fourth largest rubber producer in the world—after Thailand, Indonesia and India. The raw natural rubber produced in Malaysia during 1960s were ribbed smoked sheet and air dried sheet, while in 1970s it was crumb rubber. The current bulk of Malaysian rubber produced is in the form of technically specified crumb rubber called as Standard Malaysian Rubber (SMR). As the current method of rubber wastewater treatment are based on the following options namely: anaerobic-cum-facultative lagoon system,

anaerobic-cum-aerated lagoon system, aerated lagoon and oxidation ditch system [1,2]. While the applicability of treatment scheme like coagulation [3], stabilization ponds [4], ponding and activated sludge system [5], rock bed filtration [6] and ultra-filtration [7,8] in treating rubber wastewater were also been investigated.

The electrochemical methods of treatment are favored as they are neither subjected to failures due to variation in wastewater strength nor due to the presence of toxic substance and require less hydraulic retention time. The most promising way to carry out electrolytic treatment in a convenient and cost effective method is to generated chlorine or hypochlorous acid as and when required using sea water or brine liquor. The electrochemical method of waste treatment came into existence by treating sewage generated from ship onboard by mixing sewage and seawater in the ratio 3:1 and subjecting them to electrolysis [9]. Thereafter, the application electrochemical treatment was widely received in treating industrial wastewaters like textile [10–14], and olive mill [15,16], tannery [17–19], swine manure [20], distillery [21] and beer effluent [22], respectively. The stability of anodic electrode during electrochemical oxidation of phenol and its compounds were investigated using porous carbon felt [23], boron-doped diamond [24], ruthenium mixed oxide as

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Table 1  
Characteristics of raw Standard Malaysian Rubber process wastewater

Parameters <sup>a</sup>	Concentration
pH	7.5
BOD	1380
COD	2960
Suspended solids	310
Ammoniacal nitrogen	57

<sup>a</sup> Except pH all other parameters are in mg/L.

electrode [25] dimensionally stable anode (DSA) and graphite felt [26] and Ti/TiO<sub>2</sub>–RuO<sub>2</sub>–IrO<sub>2</sub> [27].

In this article electrolytic oxidation based on in situ hypochlorous acid generation is being proposed as a method of treatment for Standard Malaysian Rubber process wastewater. The hypochlorous acid is generated using graphite anode and stainless sheet as cathode in an undivided electrolytic reactor. The SMR wastewater was obtained from the Kota Trading Co. Sdn, Negeri Sembilan, Malaysia.

## 2. Materials and methods

### 2.1. SMR wastewater characteristics

The characteristics of SMR wastewater are presented in Table 1. The SMR wastewater was preserved at a temperature less than 4 °C, but above freezing point in order to prevent the wastewater from undergoing biodegradation due to microbial action [28].

### 2.2. Electrolytic reactor setup

The electrolytic reactor had a circular shape with the following dimensions viz: 300 mm inner diameter and 450 mm height with a liquid volume of 24 L as shown in Fig. 1. A graphite rod of 270 mm in length and 60 mm in diameter was used as an anode. Perforated stainless steel sheets 270 mm long, 50 mm wide and

having a thickness of 0.8 mm were used as cathodes. Two sets of anode and cathode were employed during the electrolysis. Each set of graphite anode was surrounded by two cathode sheets. The distance between anode and cathode was 20 mm. A rectifier having an input of 230 V and variable output of 0–20 V with a maximum current of 100 A was used as a direct current source. The reactor content was kept under mixed condition using recirculation pump having a capacity of 3 L/min. Provisions are provided for removing the floating sludge from the top outlet zone and settled sludge from the bottom sludge line.

### 2.3. Analytical method

The organic strength of the wastewater was determined by COD and TOC method [29], while its biodegradability was measured based on BOD<sub>5</sub>. The total chlorine concentration in the sample was determined by iodometric method, while the ammonia–nitrogen was determined by titrimetric method. The clarity of the wastewater was determined by Nephelometric method. The suspended solids were determined by drying the filtered solids at 105 °C [28].

### 2.4. Experimental method

The SMR wastewater feed into the reactor was maintained at a complete mixed condition. During the electrolysis, chlorine was produced at the anode and hydrogen gas at the cathode. As the anode and cathode were kept in undivided electrolytic reactor the generated chlorine undergoes disproportionation reaction resulting in hypochlorous acid ( $\text{Cl}_2 + \text{H}_2\text{O} \rightarrow \text{HOCl} + \text{HCl}$ ). Further disproportion of  $\text{OCl}^-$  to  $\text{ClO}_3^-$  was favored at high temperature (approximately 75 °C) and under alkaline conditions ( $3\text{OCl}^- \rightarrow \text{ClO}_3^- + 2\text{Cl}^-$ ). At 20 °C the hypochlorous acid dissociates to the extent of 3% at pH 6, 30% at pH 7 and 80% at pH 8 [30]. The floating sludge rose to the top of the reactor zone and was removed through the floating sludge outlet. Samples of effluent were drawn periodically and were analyzed to

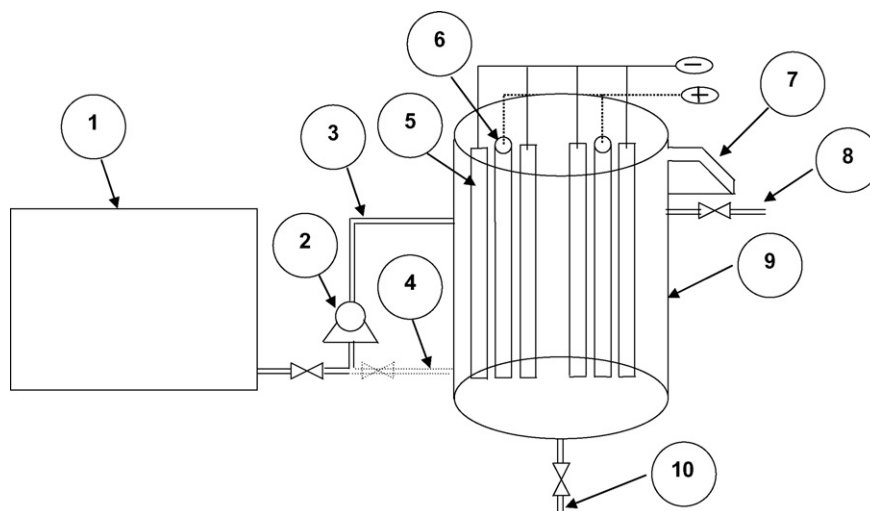


Fig. 1. Schematic diagram of electrolytic reactor. (1) Feed tank; (2) feed pump; (3) inlet line; (4) recirculation line; (5) stainless sheet as cathode; (6) graphite as anode; (7) floating sludge outlet; (8) treated effluent outlet line; (9) electrolytic reactor; (10) settled sludge outlet.

assess the performance of the electrolytic system. Experimental variables such as current densities (37.2 and 74.5 mA/cm<sup>2</sup>), pH (4.5–7.5) and sodium chloride concentration (2 and 3%) were evaluated systematically. The initial experiments were carried out using potable water at 3% sodium chloride concentration for a pH range 4.5–7.5 in steps of 0.5, in order to determine the amount of total chlorine generated.

### 3. Results and discussion

#### 3.1. Hypochlorous acid generation from potable water

The variation on total chlorine production in potable water was investigated for an initial sodium chloride concentration of 3% at a fixed current density of 50 mA/cm<sup>2</sup> by varying the initial pH from 4.5 to 7.5. The results showed that irrespective of the initial pH the total chlorine production was more or less the same for the given electrolysis period. For example, during 15 min of electrolysis period at a fixed current density of 50 mA/cm<sup>2</sup> for an initial pH value of 4.5, 5.5, 6.5 and 7.5, the total residual chlorine concentration was found to be 568, 572, 580 and 592 mg/L, respectively. For the above said operating condition the corresponding residual pH was found to be 5.8, 6.7, 7.3 and 8.1, respectively. As the pH value increased higher than 7.5, the generated hypochlorous acid will be converted into hypochlorite ion which is a weak oxidizing agent. Hence the reactor pH determines the efficiency of generated chlorine towards oxidizing the organic matter. In the case of electrolytic treatment of raw SMR process wastewater it was subjected to initial screening through a clear opening of 2 mm, which was then followed by pH adjustment to 4.5 ± 0.1.

#### 3.2. Electrolytic treatment of SMR wastewater

The electrolytic treatment of SMR process wastewater was conducted based on the optimized pH value by varying the current densities in order to ascertain the COD removal efficiency during the treatment process. For example at a current density of 37.2 mA/cm<sup>2</sup> and sodium chloride content of 2 and 3% for an electrolysis period of 75 min resulted in a residual COD value of 405 and 365 mg/L, respectively. In the case of electrolysis at a current density 74.5 mA/cm<sup>2</sup> for the above said sodium chloride content and electrolysis period, the residual COD were found to be 165 and 87 mg/L, respectively. Fig. 2 shows the residual COD concentration during the electrolysis period with 2 and 3% sodium chloride as electrolyte for a fixed current density 74.5 mA/cm<sup>2</sup>, respectively. As the electrolysis period increased the residual COD concentration decreased with 3% sodium chloride as electrolyte, as when compared to 2%. The reason for this type of behavior was due to the presence of more chloride ion in the 3% electrolyte solution which resulted in more hypochlorous acid generation during electrolysis. The energy requirement while treating SMR wastewater at varying sodium chloride concentration (2 and 3%) and at a fixed current density of 74.5 mA/cm<sup>2</sup> resulted in 46 and 30 Wh/L, respectively. The observed low energy requirement at 3% electrolyte was due to the improved conductivity, in comparison to the 2%

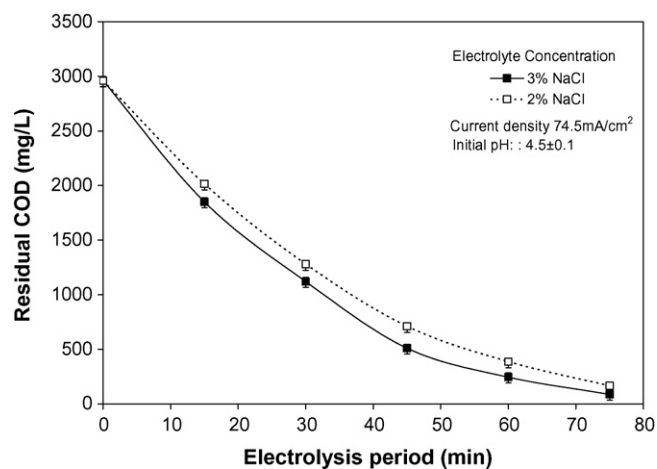


Fig. 2. Residual COD concentration vs. electrolysis period.

electrolyte addition. Hence, further experiments were carried out using 3% sodium chloride as electrolyte at a fixed current density 74.5 mA/cm<sup>2</sup>.

The residual BOD<sub>5</sub> concentration versus the electrolysis period is shown in Fig. 3 for a current density 74.5 mA/cm<sup>2</sup> at a sodium chloride concentration of 2 and 3%, respectively. As shown in Fig. 3 with the increase in electrolysis period the residual BOD<sub>5</sub> concentration resulted in lower value. For example at the end of 15, 45 and 75 min of electrolysis period with 2% sodium chloride as electrolyte for a current density 74.5 mA/cm<sup>2</sup>, the residual BOD was found to be 980, 390 and 105 mg/L, respectively. Whereas in the case of 3% sodium chloride as electrolyte for the above said condition the residual BOD resulted in a value of 870, 295 and 60 mg/L, respectively.

The total organic carbon (TOC) removal during the electrolysis period is shown in Fig. 4 for a current density 74.5 mA/cm<sup>2</sup> at a sodium chloride content of 2 and 3%. As the electrolysis period increased the total organic carbon also decreased, and a higher removal in TOC was observed with the increase in electrolyte concentration. For example at the end of 15, 45 and 75 min of electrolysis with 2% sodium chloride as electrolyte for a current density 74.5 mA/cm<sup>2</sup>, the residual TOC was found to be

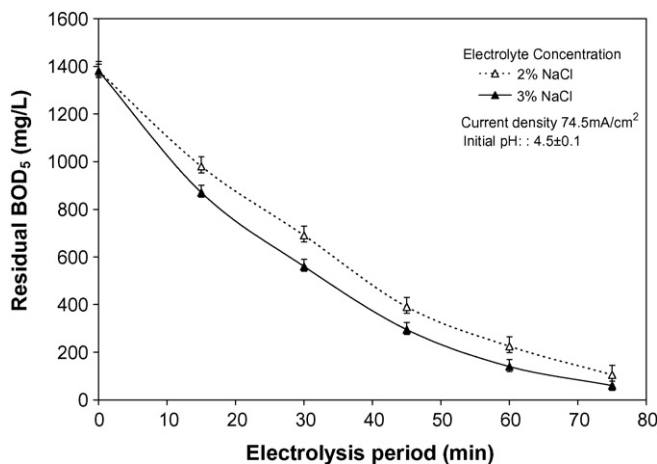


Fig. 3. Residual BOD<sub>5</sub> concentration vs. electrolysis period.

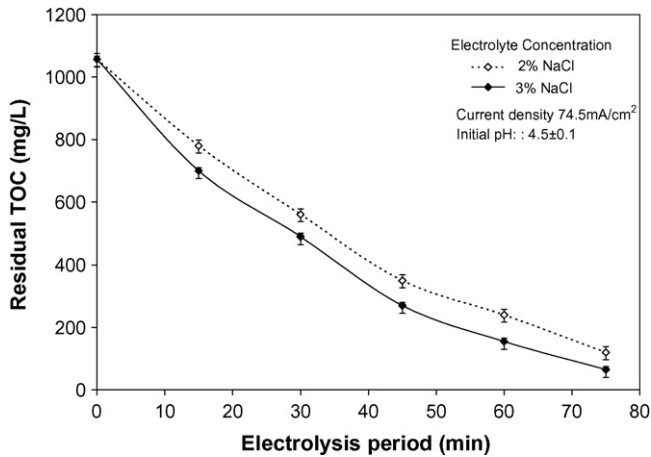


Fig. 4. Residual TOC concentration vs. electrolysis period.

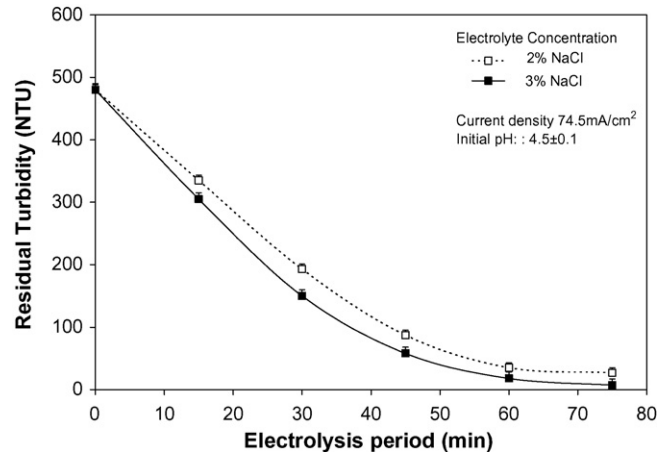


Fig. 6. Residual turbidity vs. electrolysis period.

780, 350 and 120 mg/L, respectively. In the case of 3% sodium chloride as electrolyte for the above said electrolysis period and current density the residual TOC value was found to be 618, 270 and 65 mg/L, respectively. The COD/TOC ratio decreased from 2.8 to 1.3 at the end of 75 min of electrolysis period for a current density 74.5 mA/cm<sup>2</sup>, respectively. The decrease in COD/TOC ratio shows that the carbon was destroyed due to the oxidizing action of the generated hypochlorous acid. In the case of phenol–formaldehyde resin wastewater treatment based on hypochlorous oxidation resulted in a decrease in COD/TOC ratio from 4.3 to 1.3 [27]. The electrochemical oxidation of flavor manufacturing effluent showed a decrease in COD/TOC ratio from 3.3 to 1.7 [31].

The total residual chlorine concentration during the electrolysis period is shown in Fig. 5, for an initial COD concentration of 2960 mg/L at a current density of 74.5 mA/cm<sup>2</sup> for varying sodium chloride content (2 and 3%). For example at the end of 15, 45 and 75 min of electrolysis period for a sodium chloride content of 2% and current density of 74.5 mA/cm<sup>2</sup> the total residual chlorine concentration was found to be 12, 70 and 102 mg/L, respectively. In the case of 3% sodium chloride as electrolyte for the above said condition resulted in a total residual chlorine concentration of 30, 103 and 146 mg/L, respectively. The reason for

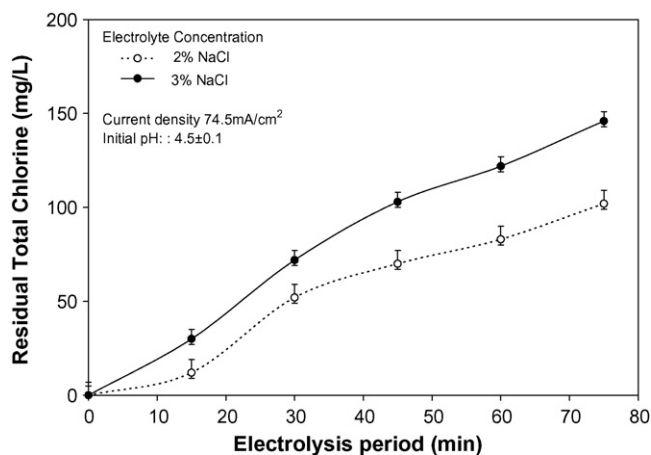


Fig. 5. Residual total chlorine concentration vs. electrolysis period.

the rise in total residual chlorine concentration during the electrolysis with 3% sodium chloride as electrolyte when compared with 2% at a fixed current density of 74.5 mA/cm<sup>2</sup> was due to the presence of more chloride ions in the electrolyte solution. During the initial period of electrolysis the residual chlorine level was found to low, thereafter with the further increase in electrolysis period the chlorine level in the electrolyte showed a gradual rise. Moreover the accumulation of hypochlorous acid was relative low as it has been utilized to destroy the organic content of the SMR wastewater as shown in Fig. 2. During electrolysis the chloride was converted into chlorine gas at the graphite anode. As a result of disproportionation reaction the generated chlorine was converted into hypochlorous which oxidizes the organic matter and reduces to chloride. In the absence of organic substance the accumulation of residual chlorine was remarkable as the same was illustrated when electrolysis was conducted using potable water.

Fig. 6 shows the residual turbidity level during the electrolysis period for varying sodium chloride content (2 and 3%) and at a fixed current density of 74.5 mA/cm<sup>2</sup>. For example at the end of 15, 45 and 75 min of electrolysis period with 2% electrolyte concentration the residual turbidity was found to be 335, 87 and 27 NTU, respectively. In the case of 3% electrolyte concentration the residual turbidity was found to be 305, 58 and 7 NTU, respectively.

The effluent temperature during the electrolysis period is shown in Fig. 7, under varying sodium chloride content (2 and 3%) and at a fixed current density of 74.5 mA/cm<sup>2</sup>. For example at the end of 15, 45 and 75 min of electrolysis period with 2% electrolyte concentration the residual effluent temperature was found to be 28, 42 and 53 °C, respectively. In the case of 3% electrolyte concentration for the above said condition resulted in an effluent temperature of 27, 38 and 48 °C, respectively. As the effluent temperature was below 75 °C during the electrolysis period the conversion of OCl<sup>-</sup> to ClO<sub>3</sub><sup>-</sup> is less pronounced [32].

The residual pH during the electrolysis period is shown in Fig. 8 for an influent COD concentration of 2960 mg/L under varying sodium chloride content (2 and 3%) and at a fixed current density 74.5 mA/cm<sup>2</sup>. For example at the end of 15, 45 and

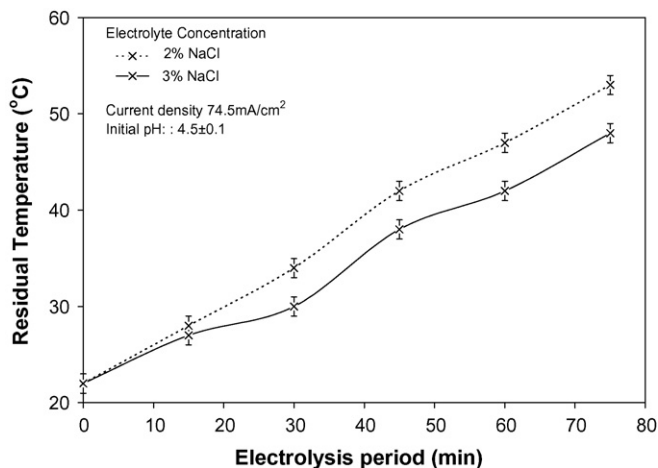


Fig. 7. Effluent temperature vs. electrolysis period.

75 min of electrolysis period with 2% sodium chloride content the electrolyte the residual pH was found to be 4.7, 6.0 and 7.2, respectively. In the case of 3% electrolyte concentration the residual pH values were found to be 4.8, 6.6 and 7.5, respectively. The rise in pH during the electrolysis period could be due to the loss of hydrogen gas at the cathode, resulting in hydroxide ion accumulation. As the electrolyte pH reached a maximum of 7.5 during the electrolysis the formation of  $\text{ClO}_3^-$  was negligible in the present investigation [32]. The ammoniacal nitrogen was found to be below detectable limit at the end of 15 min of electrolysis period for a current density  $74.5 \text{ mA/cm}^2$  and sodium chloride content of 2 and 3%, respectively.

### 3.3. Stability of graphite electrode

The graphite anode used in this present investigation belongs to category used for industrial arc electrode. The carbon loss during the hypochlorous generated from potable water and during treatment of SMR wastewater was negligible. As the initial weight of the carbon electrodes was found to be 2060.92 g, while after electrolysis at the end of whole experiment resulted in 2059.86 g (the electrolysis were carried out for 25 cycles).

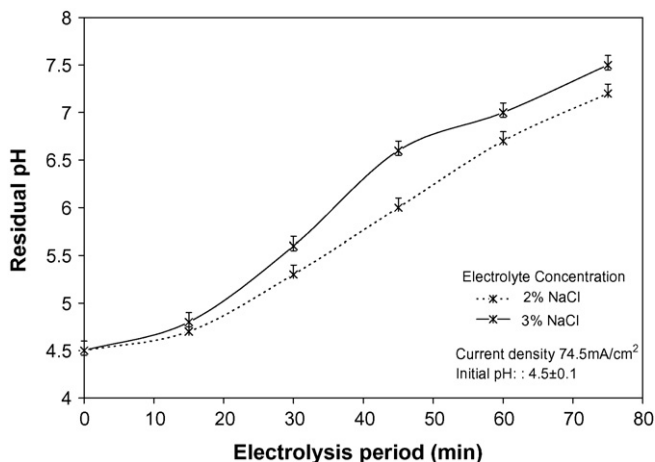


Fig. 8. Residual pH vs. electrolysis period.

Hence it could be confirmed that the graphite electrode was considerably stable.

## 4. Conclusion

The present investigation revealed that in situ generation of hypochlorous acid based on electrolysis was effective towards the treatment of Standard Malaysian Rubber process wastewater. For an influent COD concentration of  $2960 \text{ mg/L}$  at an initial pH  $4.5 \pm 0.1$ , current density of  $74.5 \text{ mA/cm}^2$ , sodium chloride content 3% and electrolysis period of 75 min, resulted in the following residual values pH 7.5, COD  $87 \text{ mg/L}$ , BOD<sub>5</sub>  $60 \text{ mg/L}$ , TOC  $65 \text{ mg/L}$ , total chlorine  $146 \text{ mg/L}$ , turbidity 7 NTU and temperature  $48^\circ\text{C}$ , respectively. During the electrochemical oxidation process the SMR wastewater undergoes in situ disinfection due to generated hypochlorous acid. Moreover the excess chlorine concentration can be reduced either by the addition of bisulfite. The chlorinated organics formed due to the partial oxidation in the electrolytic treatment can be removed by passing through activated carbon, while the excess salt can be removed by reverse osmosis before the discharging the treated effluent.

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