International Journal of Minerals, Metallurgy and Materials
Volume 19, Number 6, June 2012, Page 561
DOI: 10.1007/s12613-012-0596-0

Generation of sodium hypochlorite (NaOCl) from sodium chloride solution using C/PbO_2 and Pb/PbO_2 electrodes

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(Received: 24 April 2011; revised: 11 May 2011; accepted: 9 July 2011)

Abstract: Two modified electrodes (Pb/PbO_2 and C/PbO_2) were prepared by electrodepositing a lead oxide layer on lead and carbon substrates. These modified electrodes were used as anodes for the generation of sodium hypochlorite (NaOCl) from sodium chloride solution. Different operating conditions and factors affecting the treatment process of NaOCl generation, including current density, pH values, conductive electrolytes, and electrolysis time, were studied and optimized. By comparison the C/PbO_2 electrode shows a higher efficiency than the Pb/PbO_2 electrode for the generation of NaOCl.

Keywords: sodium hypochlorite; sodium chloride; lead oxide; electrodeposition; electrodes

1. Introduction

Sodium hypochlorite (NaOCl) is used on a large scale for surface purification, fabric bleaching, odor removal, and water disinfection [1-2]. The *in-situ* produced hypochlorite is used for the anodic oxidation of dye molecules [3-6] and phenols [7] in wastewater. It has numerous advantages, namely simple dosage, safe storage and transportation, and no residual effluent [8].

Both hypochlorous acid and hypochlorite ions in water undergo a decay process, and the decay rate depends on exposure to light (UV), temperature, pH values, initial available chlorine concentration, and the presence of catalysts, atmospheric carbon dioxide organic matter and metal ions [9-10]. Studies indicated that the decay rate increases at higher concentrations and higher temperatures [11-12]. The shelf-life or stability of NaOCl solution has been investigated previously [13-16].

Krstajić *et al*. [17] established a model for producing hypochlorites. Measurements showed a good agreement with the value predicted by the model. Cheng and Kelsall [18] developed two models to predict hypochlorite ($HOCl+OCl^-$) production by the electrolysis of near-neutral aqueous sodium chloride (NaCl) solutions, in reactors with (a) an anode and a cathode in the form of plates, and (b) a lead dioxide-coated graphite felt anode and a titanium plate cathode.

Electrochemical synthesis of NaOCl in a membrane electrolysis cell with an alumina/zirconia ceramic membrane and a titanium anode coated with cobalt oxide has been investigated [19]. Under optimum conditions, the current efficiency (CE) for NaOCl is 77% [20]. Petkov *et al*. [21] described the relationship between the quantity of the obtained hypochlorite and the electrical energy consumption from one part and the current value and the concentration of the initial chloride solution from the other. Both IrO_2 and MnO_2 with their rutile structures have been widely used in industries [22-23]. Ruthenium-tin binary (RS) oxides [$(Ru+Sn)O_2$] and ruthenium platinum (RP) oxide [$(Ru+Pt)O_x$] were coated on titanium substrates by thermal decomposition. The effects of electrolysis conditions for the CE of hypochlorite production on binary $(Ru+Sn)O_2$ and $(Ru+Pt)O_2$ electrodes and the treatment of a high salt-containing dye wastewater using this hypochlorite were also investigated [24-25].

In this study, two modified electrodes (Pb/PbO_2 and C/PbO_2) were prepared by the electrodeposition of a lead oxide layer on lead and carbon substrates. They were used

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as anodes for the generation of NaOCl from NaCl solution. Different operating conditions and factors affecting the treatment process of NaOCl generation were studied and optimized.

2. Experimental

2.1. Electrodeposition of doped lead dioxide on different substrates

2.1.1. Preparation of the Pb/PbO_2 modified electrode

(1) Lead surface treatment. Pretreatments of the lead substrate were carried out before anodization to ensure good adhesion to the lead dioxide coating. Lead was first roughened to increase the adhesion of PbO_2 deposit *via* subjecting its surface to mechanical abrasion by sandpapers of different grades, down to 40/0. Then, it was cleaned by acetone to remove sand particles or any other particles lodged in the metal surface. This process has a great application. Then, it was treated with an alkali solution (a mixture of sodium hydroxide (50 g/L) and sodium carbonate (20 g/L)) to remove any organic materials in the surface and tri-sodium orthophosphate (20 g/L) and sulfuric acid (2 g/L) to remove any oxides. Uniform and well adhesive deposit necessitates a smooth surface with no oxide or scales. To confirm our preparation, the lead substrate was soaked for 2 min in a pickling solution consisting of nitric acid (400 g/L) and hydrofluoric acid (5 g/L) and then chemically polished in a boiled oxalic acid solution (100 g/L) for 5 min [26].

(2) Electrochemical deposition of PbO_2. PbO_2 was deposited galvanostatically on the pretreated lead substrate by electrochemical anodization of lead in an oxalic acid solution (100 g/L). This acid solution was electrolyzed galvanostatically for 30 min at ambient temperature using an anodic current density of 100 mA·cm^{-2}. The cathode was stainless steel (austenitic type), and the two (lead and stainless steel) electrodes were concentric axial. This arrangement gave the formation of a regular and uniform deposit [26].

2.1.2. Preparation of the modified C/PbO_2 electrode

(1) Carbon surface treatment. Pretreatment of a carbon rod (8 mm×25 cm) was carried out following the procedure applied by Narasimham and Udupa [27]. The carbon rod was soaked in a 5% NaOH solution, washed with distilled water, dried in a furnace at 105°C, and cooked with linseed oil to reduce the porosity of the rod. After that, the electrode is ready to receive doped PbO_2.

(2) Electrochemical deposition of PbO_2. The electrodeposition of PbO_2 was performed at a constant anodic current of 20 mA·cm^{-2} in a 12% $Pb(NO_3)_2$ solution containing 5% $CuSO_4{\cdot}5H_2O$ and 3% surfactant. The role of the surfactant is to minimize the surface tension of the solution. Electrodeposition was carried out for 60 min at 80°C by continuous stirring [27].

2.2. Chemicals

NaCl, sodium fluoride, sodium carbonate, sodium phosphate, sodium sulfate, calcium chloride, potassium chloride, sodium hydroxide, sulfuric acid, and potassium iodide were of analytical grade and were purchased from Merck.

2.3. Electrolysis for the generation of NaOCl

The experiments were carried out in a 100 mL Pyrex glass cell with the prepared (C/PbO_2 and Pb/PbO_2) electrode as anodes and the titanium sheet as cathodes.

The operating electrolysis was carried out under the following condition: current density, 0.5-2.5 A; pH 1-13; potential, 2-12 V; temperature, 5-40°C, and the concentration of NaCl solution, 5-60 g/L. The time of electrolysis ranged from 5 to 80 min. The distance between the two electrodes (anode and cathode) varies from 1 to 4 cm, and the area of the cathode differs from 1 to 6 cm^2. The investigations of this study were carried out in the presence of 15 g/L NaCl and 5 g/L of different types of electrolytes (NaCl, $CaCl_2$, KCl, Na_2CO_3, NaF, Na_3PO_4, and Na_2SO_4).

2.4. Analysis

The hypochlorite content was determined by the following two methods: (1) reacting liquid bleaches with iodides and treating iodine produced with a measured excess of thiosulfate solution previously standardized calorimetrically [28]; (2) measuring the absorption of investigated electrolytes using a double-beam UV-visible spectrophotometer from Shimadzu at λ_{max}=292 nm.

3. Results and discussion

3.1. Effect of conductive electrolyte type

The effect of conductive electrolyte type on the hypochlorite generation was investigated by using NaCl, $CaCl_2$, KCl, Na_2CO_3, NaF, Na_3PO_4, and Na_2SO_4 on both Pb/PbO_2 and C/PbO_2 electrodes. The operating conditions of the treatment process were as follows: current density, 1 A·cm^{-2}; temperature, 10°C; electrolysis time, 90 and 60 min for Pb/PbO_2 and C/PbO_2 electrodes, respectively; pH 12; and the distance between the two electrodes, 1 cm. As shown in

Fig. 1, it is clear that NaCl is the most effective conductive electrolyte for the generation of NaOCl. For this reason, NaCl was chosen as the conductive electrolyte in the following series of experiments.

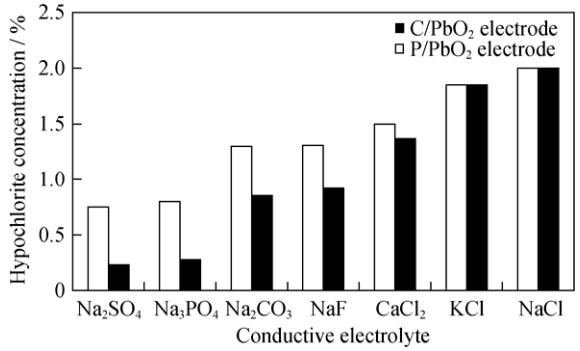

Fig. 1. Effect of conductive electrolytes on the hypochlorite generation from NaCl solution using C/PbO_2 and Pb/PbO_2 electrodes.

Different reactions of NaOCl production occurring at the electrodes and in the bulk solution are described in various reviews [29].

(1) Primary reactions.

At the anode:

$$2Cl^- - 2e^- \rightarrow Cl_2 \quad (1)$$

At the cathode:

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \quad (2)$$

In the bulk:

$$Cl_2 + 2H_2O \leftrightarrow HClO + Cl^- + H_3O^+ \quad (3)$$

$$HClO + H_2O \leftrightarrow ClO^- + H_3O^+ \quad (4)$$

The equilibrium constants for the two reversible reactions are 4×10^{-4} $mol^2 \cdot L^{-1}$ and 3×10^{-8} mol^{-1}, respectively, at 25°C [30].

(2) Side reactions.

At the anode:

$$6ClO^- + 3H_2O - 6e^- \leftrightarrow ClO_3^- + 5Cl^- + 6H^+ + 3O_2 \quad (5)$$

$$2H_2O \rightarrow O_2 + 4H^+ + 4e^- \quad (6)$$

At the cathode:

$$ClO^- + H_2O + 2e^- \rightarrow Cl^- + 2OH^- \quad (7)$$

In the bulk:

$$2HOCl + ClO^- \rightarrow ClO_3^- + 2Cl^- + 2H^+ \quad (8)$$

$$2ClO^- \rightarrow 2Cl^- + O_2 \quad (9)$$

The observed behavior of NaCl as the most effective electrolyte for NaOCl generation may be due to the small ion size of Na^+, which increases the ion mobility. The opposite behavior observed when using Na_2CO_3, NaF, Na_3PO_4, and Na_2SO_4 might be related to the formation of an adherent film on the anode surface, which poisoned the electrode process. Also, the mentioned electrolytes do not contain chloride ions, which are the main source of OCl^-. $CaCl_2$ is a less effective electrolyte than NaCl in NaOCl production because of the formation of insoluble calcium hypochlorite $Ca(OCl)_2$.

3.2. Effect of current density

As the current density increases, hypochlorite production also increases. However, the cell temperature also increases with the increase in current density. Fig. 2 shows that the concentration of NaClO increases up to 1 $A\cdot cm^{-2}$ by using Pb/PbO_2 and C/PbO_2 electrodes. At current densities higher than 1 $A\cdot cm^{-2}$ the concentration of NaClO decreases due to the increase in temperature and the decomposition of hypochlorite. The optimum current density obtained at the maximum hypochlorite concentration is 1 $A\cdot cm^{-2}$. It is evident that the decrease of energy consumption is due to the increased CE [12]. The treatment processes were carried out under the following conditions: pH 12; temperature, 10°C; NaCl concentration, 20 g/L; electrolysis time for Pb/PbO_2 and C/PbO_2 electrodes, 90 and 60 min, respectively; distance between the two electrodes, 1 cm.

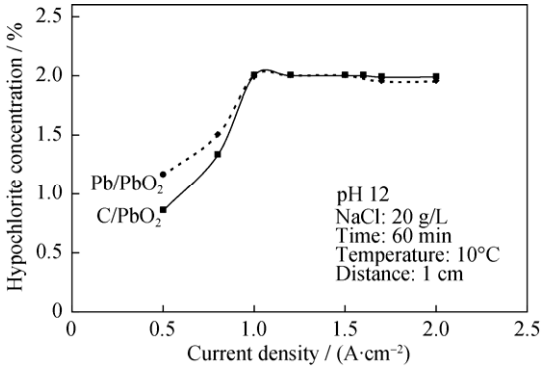

Fig. 2. Effect of current density on the hypochlorite generation from NaCl solution using C/PbO_2 and Pb/PbO_2 electrodes.

At temperatures above 35°C, NaOCl tends to chemically decompose to sodium chlorate.

$$3NaClO \rightarrow NaClO_3 + 2NaCl \quad (10)$$

3.3. Effect of temperature

Temperature plays a vital role in the electrogeneration of hypochlorite using Pb/PbO_2 and C/PbO_2 electrodes. Fig. 3 illustrates that the increase of temperature up to 10°C de-

creases the NaClO concentration as well as the current efficiency (CE) of the reaction. Low temperature favors the generation of NaClO. A higher temperature leads to the chemical decomposition of the hypochlorite formed as explained earlier. The electrolyser has to be maintained at ambient temperature (10°C) at which the maximum NaClO concentration is about 2.0 for Pb/PbO_2 and C/PbO_2 electrodes, respectively. These experiments were carried out under the same conditions mentioned above.

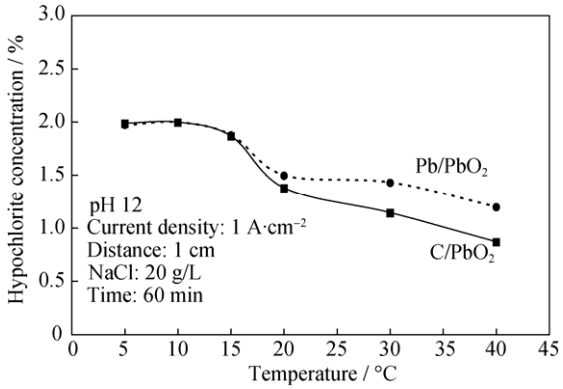

Fig. 3. Effect of temperature on the hypochlorite generation from NaCl solution using C/PbO_2 and Pb/PbO_2 electrodes.

The NaOCl production rate depends on temperature [31]. At temperatures between 23 and 30°C, there is a drop in the production of the hypochlorite. Over this small temperature range, the NaOCl production rate is lowered and NaOCl tends to chemically decompose to sodium chlorate at 35°C [32]. It is clear from Fig. 3 that the optimum temperature of NaOCl production is 10°C for Pb/PbO_2 and C/PbO_2 electrodes. At low temperature, the loss of chlorine gas decreases, so NaOCl is increased.

3.4. Effect of electrolysis time

Fig. 4 represents the effect of time on hypochlorite generation by using both Pb/PbO_2 and C/PbO_2 electrodes. Electrolysis was carried out at different time intervals between 10 and 300 min under the previous experiment conditions. An increase of electrolysis duration up to 90 and 60 min leads to the increase in hypochlorite generation for Pb/PbO_2 and C/PbO_2 electrodes, respectively. Further increase in time has no significant effect.

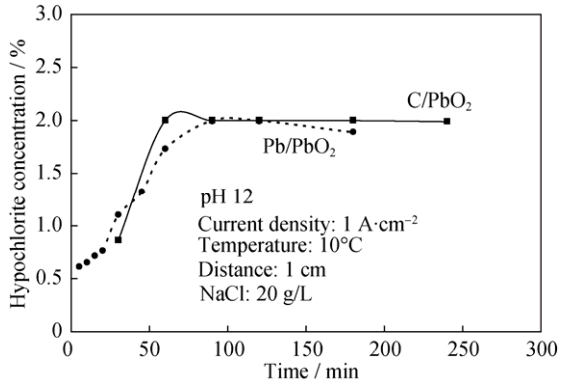

Fig. 4. Effect of time on hypochlorite generation from NaCl solution using C/PbO_2 and Pb/PbO_2 electrodes.

3.5. Effect of NaCl concentration

Fig. 5 represents the effect of NaCl concentration on NaOCl generation. The results indicate that an increase in NaCl concentration leads to an increase in hypochlorite production up to 20 g/L for both Pb/PbO_2 and C/PbO_2 electrodes. A further increase in NaCl concentration has no effect on the hypochlorite generation. For this reason, 20 g/L NaCl is considered as the optimum concentration.

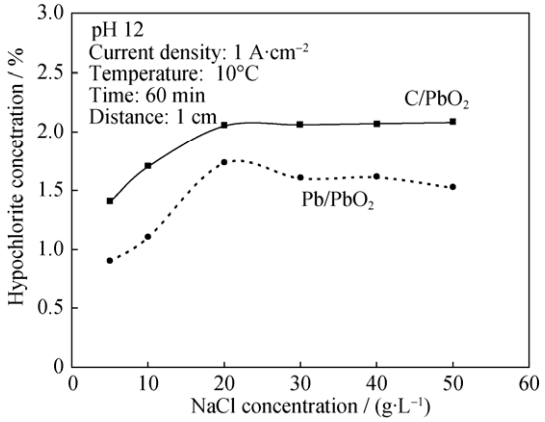

Fig. 5. Effect of NaCl on hypochlorite generation from NaCl solution using C/PbO_2 and Pb/PbO_2 electrodes.

3.6. Effect of pH values

The pH value has an important effect on NaOCl generation. Fig. 6 shows that the rate of NaOCl generation increases as the pH value increases up to 12 for both Pb/PbO_2 and C/PbO_2 electrodes.

The basic medium initiates the absorption of chlorine

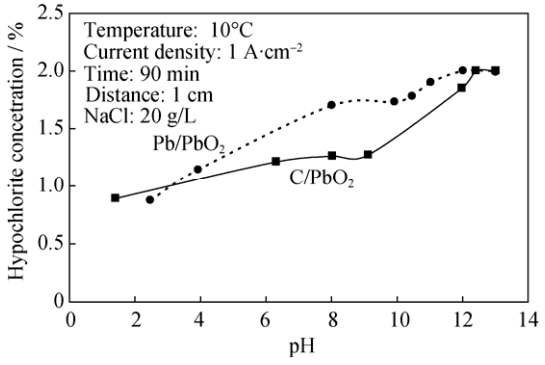

Fig. 6. Effect of pH values on hypochlorite generation from NaCl solution using C/PbO_2 and Pb/PbO_2 electrodes.

produced on the anode, which increases the generation of the hypochlorite. The reaction between chlorine and NaOH forms NaOCl according to the following reaction:

$$NaOH + Cl_2 \rightarrow NaOCl + Cl^- + H^+ \quad (11)$$

The most stable NaOCl solutions are those at pH values between 11.5 and 13 [33].

3.7. Effect of the distance between two electrodes

Fig. 7 shows the effect of the distance between the cathode and the anode on NaOCl production for both Pb/PbO_2 and C/PbO_2 electrodes. It is clear that the NaOCl production rate increases with the distance decreasing to 1 cm. This is due to the electrolyte ohmic potential drop and hence the cell potential [34]. The highest hypochlorite production was achieved with the narrow distance between the cell electrodes of 1 cm.

Table 1 shows the stability of NaOCl prepared by using Pb/PbO_2 and C/PbO_2 electrodes at 25 and 4°C in light and dark. Degradation occurred very slowly (60 d) in dark at 25 and 4°C. However, in light (at 25°C), nearly complete degradation was achieved and reached 1.8% and 1.5% using C/PbO_2 and Pb/PbO_2 electrodes, respectively.

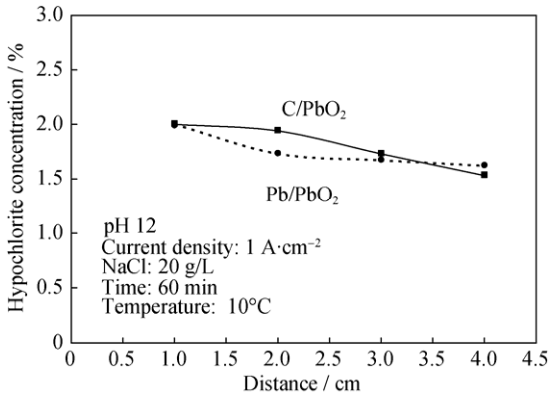

Fig. 7. Effect of the distance between two electrodes on the hypochlorite generation from NaCl solution using C/PbO_2 and Pb/PbO_2 electrodes.

Table 1. Stability of NaOCl prepared on Pb/PbO_2 and C/PbO_2 electrodes in different conditions (temperature and light)

Time / d	Concentration of NaOCl prepared on the C/PbO_2 electrode / %			Concentration of NaOCl prepared on the Pb/PbO_2 electrode / %		
	4°C in dark	25°C in light	25°C in dark	4°C in dark	25°C in light	25°C in dark
0	100	100	100	100	100	100
1	100	96.6	99.9	100	81.5	99.9
5	100	80.1	97.8	100	75.8	97.7
8	100	79.9	97.4	99.7	47.7	95.3
16	99.5	68.2	95.4	99.5	35.0	95.0
26	99.5	27.5	94.2	97.3	15.0	94.6
36	99.6	12.8	94.2	96.9	10.3	94.0
50	97.4	5.1	94.1	95.3	4.2	93.8
60	96.8	1.8	94.0	94.8	1.5	93.1

Table 2 represents the efficiency of Pb/PbO_2 and C/PbO_2 electrodes for NaOCl generation and electrical energy consumption. It is clear that C/PbO_2 is more efficient than Pb/PbO_2 in the generation of NaOCl.

Table 2. Percent yield of Pb/PbO_2 and C/PbO_2 electrodes on NaOCl generation

Electrode	Percent yield in NaCl solution / %	Cost
C/PbO_2	80.0	USD $\$4.8\times10^{-4}$
Pb/PbO_2	69.2	USD $\$7.2\times10^{-3}$

The electrical energy consumed for NaOCl using Pb/PbO_2 and C/PbO_2 electrodes was calculated by the following equation:

Electrical energy consumed = $IEt/1000\times3600$ kW·h,

where I is the applied current, A; E the voltage of the electrolytic cell; and t the electrolytic time, s [7].

The yield of generating NaOCl using Pb/PbO_2 and C/PbO_2 electrodes was 2% and that using dimensionally stable anodes (DSA) was 0.8%. This indicates that Pb/PbO_2 and C/PbO_2 electrodes are more efficient in generating NaOCl than DSA. Both the investigated electrodes are cheaper and more anticorrosive than platinum electrodes [35].

4. Conclusions

(1) The C/PbO_2 electrode is more efficient in generating NaOCl than the Pb/PbO_2 electrode from NaCl solution.

(2) The yield of generating NaOCl using Pb/PbO_2 and C/PbO_2 electrodes was 2% and that using DSA was 0.8%.

(3) The C/PbO_2 electrode is cheaper than DAS and is more resistant to corrosion than the platinum electrode.

(4) Factors for the optimal generation of NaOCl using the modified electrodes, including conductive electrolyte, NaCl concentration, current density, pH values, temperature, and the distance between the cathode and the anode, were investigated.

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