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STUDY OF THE EFFECT OF HERMETICITY OF THE INSTALLATION DURING ELECTROLYSIS OF SODIUM CHLORIDE SOLUTION

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Abstract

This article examines the effect of the tightness of the installation during the electrolysis of sodium chloride solution in a periodic mode. The influence of parameters (temperature, duration, tightness) on the process of obtaining sodium hypochlorite in diaphragm less electrolysis of a solution of table salt is studied.

Keywords: Water disinfection, electrolysis, current density, active chlorine, hydrogen, sodium hypochlorite, tightness, cathode, anode, temperature, continuous, periodic.

Introduction

The most common method of water disinfection in the world using gaseous chlorine [1, 2], being reliable (with prolonged action) and relatively cheap, raises doubts among specialists and consumers regarding the environmental safety of its use [1 – 5]. The toxicity of chlorine, increased by the high concentration of the reagent, as well as the need to transport containers with chlorine in densely populated urban areas and its subsequent storage at stations, usually located near residential areas, determines the high risks of its use.

Sodium hypochlorite (SHC) is a generally recognized alternative disinfectant to gaseous chlorine; it is a safe-to-handle, low-toxic, and easy-to-use reagent containing active chlorine.

Sodium hypochlorite can be obtained on-site by electrolysis of artificially prepared solutions of table salt or natural mineralized chloride waters.

During the electrolysis of sodium chloride solution, in addition to the main electrochemical reactions:

on the cathode $2H_2O + 2e^- \rightarrow H_2\uparrow + 2HO^-;$

on the anode $2Cl^- - 2e^- \rightarrow Cl_2\uparrow;$

or total $2NaCl + 2H_2O + Cl_2 \rightarrow H_2\uparrow + 2NaOH;$

both at the cathode and at the anode, side reactions and secondary processes occur that reduce the current output of active chlorine [6]:

– electrochemical oxidation of hypochlorite to chlorate ClO3–:

$$6Cl^- - 6e^- \rightarrow ClO_3 + 1.5O_2 + 5Cl^-;$$

– chemical decomposition of hypochlorite anion:

$$2ClO^{-} \rightarrow O_2 + 2Cl^{-};$$

– electrochemical reduction of hypochlorite and chlorate at the cathode:

$$ClO^{-} + 2H_2O + 2e^{-} \rightarrow 2HO^{-} + Cl^{-}.$$

As is known [7], a number of factors that influence the yield of active chlorine include: the concentration of chlorides in the initial solution, the anode current density, the nature of the movement of the electrolyte in the electrolyzer, the temperature and carbonate hardness of the solution, the material of the anodes, and the electrolysis mode.

Chlorine oxides obtained by electrolytic decomposition of NaCl solutions are distinguished by an inactivating capacity that somewhat exceeds the activity of chlorine-containing preparations. The prospects of the electrolytic method of water disinfection are due to its reliability in operation, simplicity and sufficient cost-effectiveness. By now, the patterns of electrolytic disinfection of water containing bacteria and viruses have been studied quite fully, designs of electrolysis units have been developed, their industrial production and application at water supply stations have begun. Usually, during the electrolytic decomposition of table salt, the solution environment is within 10-11, which promotes the interaction of chlorine with the alkali formed under these conditions. Electrolysis in such parameters is accompanied by the production of chlorine forms in their activity, identical to the products present during the treatment of water with bleach [8].

Studying the process of electrolysis of table salt in a diaphragmless installation in search of ways to obtain a solution of sodium hypochlorite, we decided to assemble a sealed laboratory installation. At the same time, we conducted research on the effect of the sealing of the installation on the process of formation of highly concentrated sodium hypochlorite (potassium) in a periodic mode.

Material and Methods

The determination of active (equivalent) chlorine in sodium hypochlorite solution was carried out based on the following standard method [9].

The method is based on the oxidation of potassium iodide with active chlorine to iodine, which is titrated with a specific solution of sodium thiosulfate. To eliminate interfering effects (the presence of ozone, nitrites, iron oxide, etc. in the water), the solution samples are acidified with a buffer solution with a pH of 4.5.

Preparation of Reagents

– 0.1 N sodium thiosulfate solution, dissolve the ampoule of fixanal in distilled water and bring the volume to 1 l;

– potassium iodide in crystals, 10 g of salt is dissolved in distilled water and the volume is brought to 100 ml;

– 0.01 N sodium thiosulfate solution, 100 ml of 0.1 N $Na_2S_2O_3$ solution are diluted with distilled water and the volume is brought to 1 l. The solution is used when the active chlorine content in the sample is more than 1 mg / l;

– 0.005 N sodium thiosulfate solution, 50 ml of 0.1 N $Na_2S_2O_{3\ solution}$, brought to 1 l with distilled water, used when the active chlorine content in the sample is less than 1 mg/l;
– 0.5% starch solution, 0.5 g of soluble starch is mixed with 15-20 ml of distilled water, added to 100 ml of boiling distilled water and boiled for 1-2 minutes;
– a buffer solution with pH 4.5, 102 ml of 1 M acetic acid (60 g of glacial acetic acid in 1 l of water) and 98 ml of 1 M sodium acetate solution ($CH_3COONa\ 3H_2O$ in 1 l of water).

Conducting an Analysis

A 2 ml sample of hypochlorite is taken into a conical flask and 150-200 ml of distilled water is added, then 5-10 ml of a 5 N solution of hydrochloric acid is added and, while mixing the sample, 1-2 g of potassium iodide is added. The released iodine is titrated with a 0.1 N solution of sodium thiosulfate until a light yellow color is obtained. After this, about 1 ml of starch solution is added and titrated until the blue color disappears. The calculation of the concentration of active (equivalent) chlorine X g/l is determined by the formula:

$$X_{\text{г/л}} = \frac{V_{\text{ТСН}} \cdot 0{,}003545 \cdot 1000}{10 \cdot V_2(2\text{мл})} = V_{\text{ТСН}} \cdot 0{,}17725$$

where V_{TSN} is the volume of sodium thiosulfate solution used for titration, ml; N_1 is the normality of the $Na_2S_2O_3$ solution; V_2 is the volume of the analyzed sodium hypochlorite solution, ml.
Throughout the experiments, the current density was maintained at 10 A/dm2 at an operating voltage of 3.5 V. The process temperature was measured with a mercury glass thermometer manufactured according to TU 25-2021.010-89 TT. When conducting electrolysis, a German training stand (power supply) of the GUNT brand was used. Geratebau GmbH CE – 105.

Experimental Part

The process of electrolysis of a solution of table salt can be carried out in continuous (flow) or periodic modes. The creation of a laboratory setup for carrying out a continuous process requires complex expensive equipment, so it was decided to conduct experiments in a periodic setup. For this, a 1-liter glass jar with a metal screw cap was selected. Having opened two holes in the lid, electric wires were passed through, sealing them with silicone auto sealant manufactured according to TU 20.17.10-016-58646534-2014, article 11225-060. Two electrodes measuring 3x7 cm with an area of 0.2 dm^2 were made of dense graphite, a block with an interelectrode distance of 5 mm was assembled from them, the exposed parts of the fasteners and wires were sealed with the same silicone sealant, and the block was immersed in a 25% solution of sodium chloride. In the cell, the electrodes were installed horizontally, and the lower electrode was connected to the positive pole (where molecular chlorine gas is formed and its maximum dissolution in cool water (11-14 ^{0}C) occurs). The electrolysis cell was installed in a 10-liter plastic bath with running water (10-11 ^{0} C) to maintain the required process temperature.

Results

Experiment No. 1 duration 1 hour, temperature at the beginning 15 ^{0}C, at the end 20 ^{0}C, current density 10 A/dm ^{2,} $Na_2S_2O_3$ consumption – 2.8 ml, concentration of active chlorine C $_{A.X}$ = 4.96 g/l.

Experiment No. 2 was continued for another hour at a temperature of 12 ^{0}C at the beginning and 18 ^{0}C at the end, with the consumption of $Na_2S_2O_3$ being 3.0 ml and the concentration of active chlorine C $_{A.X}$ = 5.31 g/l. The setup with the sample was stored with a hermetically sealed lid in a water bath at 15-18 ^{0}C. Experiment No. 3 was carried out with this sample after 24 hours.

Experiment No. 3 duration 3 hours, temperature at the beginning 13 ^{0}C, at the end 20 ^{0}C, current density 10 A/dm^2 consumption of $Na_2S_2O_3$ – 5.3 ml, concentration of active chlorine C A.X $_{=}$ 9.39 g/l. The setup with the sample was stored hermetically sealed with a lid in a water bath at 15-18 ^{0}C. After 24 hours, experiment No. 4 was carried out with this sample. In this case, the content of active chlorine was determined first, consumption of $Na_2S_2O_3$ – 5.0 ml.

Experiment No. 4 duration 8 hours, temperature at the beginning 13 ^{0}C, at the end 14 ^{0}C, current density 10 A/dm2 consumption of $Na_2S_2O_3$-5.8 ml, concentration of active chlorine C A.X $_{=}$ 10.28 g/l. The setup with the sample was stored hermetically sealed with a lid in a water bath at 12-14 ^{0}C. The next day, experiment No. 4 was carried out with this sample. In this case, the content of active chlorine was determined first, consumption of $Na_2S_2O_3$ – 5.8 ml.

Experiment No. 5 duration 8 hours, temperature at the beginning 12 ^{0}C, at the end 13 ^{0}C, current density 10 A/dm^2 consumption of $Na_2S_2O_3$– 6.2 ml, concentration of active chlorine C A.X = 10.99 g/l. The setup with the sample was stored hermetically sealed with a lid in a water bath 12-14 0 C. The next day , experiment No. 5 was carried out with this sample. In this case, the content of active chlorine was determined first, consumption of $Na_2S_2O_3$ – 6.1 ml.

Experiment No. 6 duration 8 hours, temperature at the beginning 11 ^{0}C, at the end 14 ^{0}C, current density 10 A/dm^2 $Na_2S_2O_3$ consumption – 6.5 ml, concentration of active chlorine C $_{A.X}$ = 11.52 g/l.

No. of Experiments	Duration, hours	t $_{start}$, ^{0} C	t $_{con}$, ^{0} C	Consumption, ml $Na_2S_2O_3$	Loss of active chlorine during storage	With $_{A.H}$, g/l	t $_{storage}$, ^{0} C
1	1	15	20	2.8	2.8	4.96	
2	1	12	18	3.0	3.0	5.1	15-18
3	3	13	20	5.3	5.0	9.59	15-18
4	8	13	14	5.8	5.8	10.28	12-14
5	8	12	13	6.2	6.1 ml	10.99	12-14
6	8	11	14	6.5		11.52	

Discussion

1. The resulting gaseous chlorine and hydrogen are released onto the surface of the solution and will be collected in the upper part of the installation, while chlorine gas, being heavier than hydrogen, will be located in the lower part of the mixture and will be maximally absorbed by the aqueous solution, increasing the formation of sodium hypochlorite in the solution.
2. In the upper part of the electrolysis unit, the maximum amount of gaseous hydrogen was collected above the solution and left the unit when the lid was opened to collect samples for active chlorine analysis.
3. The release of hydrogen from the reaction mixture leads to an increase in the pH of the medium, which is favorable conditions for the formation of sodium hypochlorite.
4. When the electrolysis lasts more than 8 hours, a sufficient amount of hydrogen (increase in pressure) accumulates above the reaction mixture, so that the absorption of chlorine in the reaction mixture shifts towards its maximum and this leads to the formation of a sufficient amount of sodium hypochlorite.

Conclusion

To obtain maximum hypochlorites, the process of electrolysis of chloride solutions must create conditions for the removal of hydrogen and for the absorption of chlorine by the reaction solution.

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