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(54) **ELECTROLYSIS CELL FOR SYNTHESIZING PERCHLORIC ACID COMPOUND AND METHOD FOR ELECTROLYTICALLY SYNTHESIZING PERCHLORIC ACID COMPOUND**

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(57) **ABSTRACT**

The present invention provides an electrolysis cell for synthesizing a perchloric acid compound, the electrolysis cell having: a feedstock solution containing a chloride or a chloric acid compound; a cathode; and an anode having an electroconductive diamond as an anode substance, the anode electrolytically oxidizing the chloride or the chloric acid compound to synthesize the perchloric acid compound, and a method for electrolytically synthesizing a perchloric acid compound.

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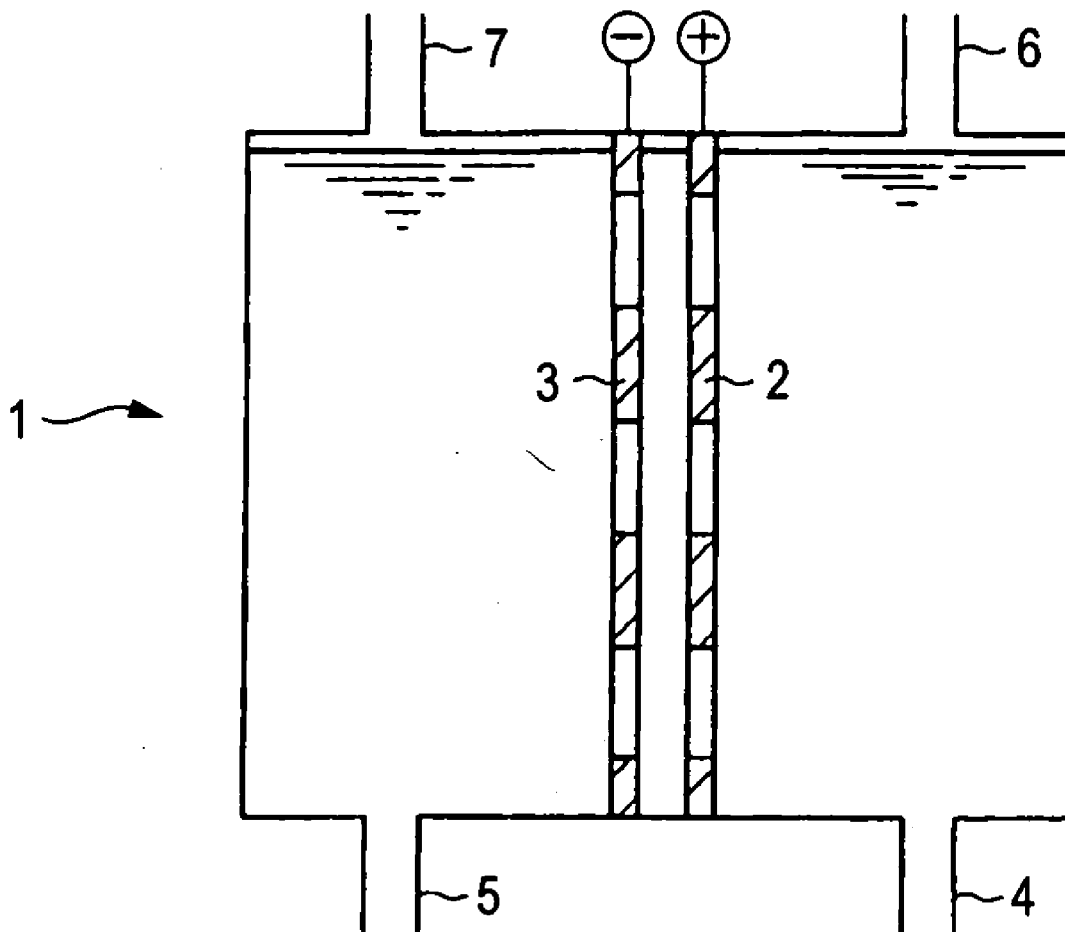


FIG. 1

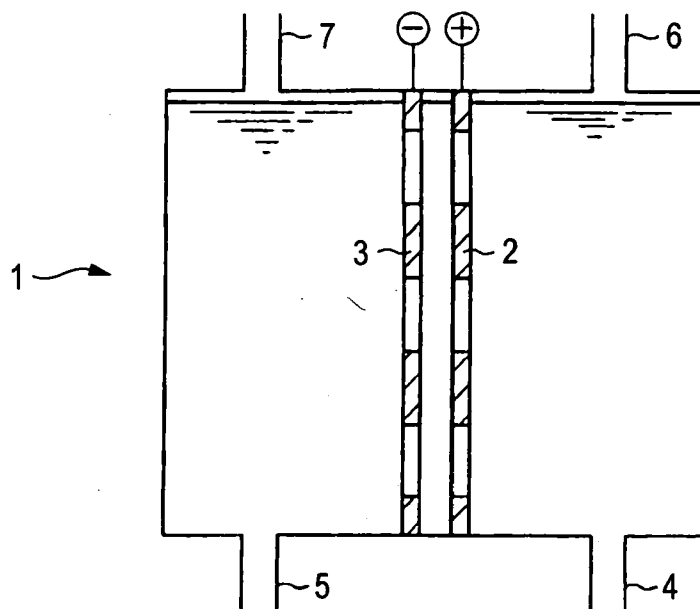
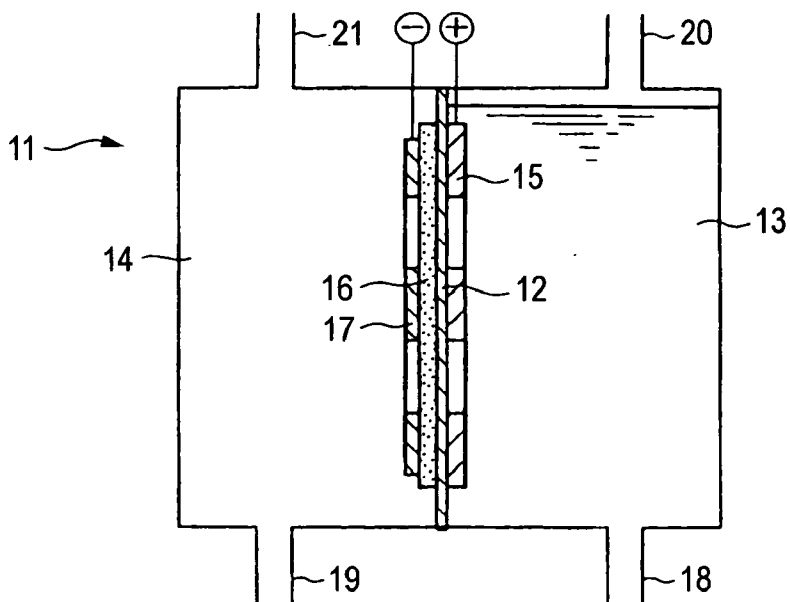


FIG. 2



**ELECTROLYSIS CELL FOR SYNTHESIZING
PERCHLORIC ACID COMPOUND AND METHOD
FOR ELECTROLYTICALLY SYNTHESIZING
PERCHLORIC ACID COMPOUND**

FIELD OF THE INVENTION

[0001] This invention relates to an electrolysis cell of which an object is to obtain a perchloric acid compound effectively as a high purity product, which is an industrially important oxidant and used as a feedstock for an explosive and a reaction synthesis reagent, and a producing method of the perchloric acid compound with the electrolysis cell.

BACKGROUND OF THE INVENTION

[0002] Each of perchloric acid and perchlorate is one of useful materials for electrolytic synthesis and used for a rocket fuel, an explosive, and a reaction catalyst. Sodium perchlorate is obtainable by oxidizing, concentrating, and crystallizing sodium chlorate through electrolysis. Perchloric acid is synthesized by heat cracking distillation of sodium perchlorate with sulfuric acid and put on market and transported as a 60% to 70% aqueous solution to avoid risk of explosion. Potassium perchlorate is synthesized by adding potassium chloride to a solution synthesized by electrolysis, followed by double decomposition, cooling, crystallizing, and separation.

[0003] In the perchlorate electrolytic synthesis, an electrode having a Ti substrate on whose surface a platinum foil is formed is used, and the lower limit of a concentration the chloric acid compound (chlorate), which is a feedstock, is 0.01 M since deterioration of electrode material and deterioration of electrode can be accelerated when the concentration comes to 0.1 M or less. It is considered that such deterioration is caused since: an oxide film is formed on a surface of the platinum material under the above-mentioned electrolysis condition; performance of the electrode becomes unstable due to on/off of a current; and consumption is accelerated due to an additive such as a fluorine ion. Though lead oxide is excellent in decomposition characteristic and useful as a substitute, it has drawbacks such as management difficulty since it generates sludge to deteriorate product purity and makes performance of an electrode unstable. A substitute electrode material has been pursued for solving the problems.

[0004] Since diamond is excellent in heat conductivity, optical transparency, and resistance to high temperature and oxidation, and, particularly, since it is possible to control electroconductivity of the diamond by doping, diamond is considered promising as a next generation special semiconductor device or an energy converting element. Studies on application of an electroconductive diamond electrode to electrochemical fields have been conducted actively in recent years since the electroconductive diamond has, in addition to the mechanical and chemical stability, a good electroconductivity that is imparted by the doping.

[0005] Related patent publications are as listed below.

[0006] In U.S. Pat. No. 5,399,247, it is disclosed that it is possible to decompose an organic wastewater by using diamond as an anode material.

[0007] In JP-A-9-268395, it is disclosed that diamond is useful as an electrode for functional water (containing ozone).

[0008] In JP-A-2001-192874, it is disclosed that an electroconductive diamond is useful as an electrode for persulfuric acid synthesis.

[0009] In JP-A-2004-211182, it is disclosed that the electroconductive diamond is useful as an electrode for percarbonic acid synthesis.

[0010] In JP-2004-202283, it is disclosed that, when an ion exchange membrane is used for processing a wastewater containing an organic chlorine compound by electrolysis with the use of an electroconductive diamond electrode, it is possible to prevent negative ion transfer between two-chambers, thereby preventing a chlorine ion (negative ion) that has been dissociated at the cathode from transferring to the anode side and preventing a deterioration in organic compound decomposition efficiency otherwise caused by oxidation of the chlorine ion from a hypochlorous acid ion to a perchloric acid ion at the anode (paragraph [0023]).

[0011] However, any study for the purpose of improving a current efficiency and purity of the perchloric acid compound for the purpose of industrial electrolytic synthesis is not found in the above patent publications.

SUMMARY OF THE INVENTION

[0012] An object of this invention is to find an electrolysis cell and an electrolytic synthesis method capable of synthesizing a perchloric acid compound such as perchloric acid at high purity and with good power efficiency.

[0013] The present invention provides: an electrolysis cell for synthesizing a perchloric acid compound, the electrolysis cell comprising: a feedstock solution containing a chloride or a chloric acid compound; a cathode; and an anode having an electroconductive diamond as an anode substance, the anode electrolytically oxidizing the chloride or the chloric acid compound to synthesize the perchloric acid compound; and a method for electrolytically synthesizing a perchloric acid compound with the electrolysis cell.

[0014] Hereinafter, this invention will be described in detail.

[0015] It is possible to synthesize a perchloric acid compound which is an object product of this invention at high efficiency by electrolyzing a chloride or a chloric acid compound by using an electroconductive diamond electrode. In this invention, the perchloric acid compound includes perchlorate, perchloric acid, and a perchloric acid ion; the chloride includes a chloride, hydrochloric acid, and a chloride ion; and the chloric acid compound includes chlorate, chloric acid, and a chloric acid ion. When specifying the perchlorate, the perchloric acid, the perchloric acid ion, the chloride ion, the chlorate, the chloric acid, and the chloric acid ion, the above terms are used.

[0016] In conventional perchloric acid electrolysis synthesis methods, sodium chlorate is synthesized by electrolysis using sodium chloride as a feedstock, and then sodium perchlorate is produced in another cell by electrolysis using the thus-obtained sodium chloride as a feedstock. On the other hand, this invention enables one-stage electrolysis of the sodium perchlorate, and such device is advantageous from cost point of view. The one-stage electrolysis has not put into practical use in direct synthesis of the perchloric acid compound from the chloride ion.

[0017] In the case of obtaining a chloric acid ion from a chloride ion, a platinum anode is not practical since it is consumed rapidly. Also, a lead oxide anode has a large activation overvoltage and is suitable for perchloric acid synthesis; however, the lead oxide anode is problematic

from practical point of view since it reacts with the chloride ion used as a feedstock to cause precipitation and accelerates generation of sludge.

[0018] Though the diamond anode does not prevent chlorine gas generation, it achieves remarkably great perchloric acid compound synthesis efficiency at a low concentration since it is free from sludge generation, so that the electrode consumption which is the problem of the platinum electrode hardly proceeds.

[0019] A part of chloric acid used as the feedstock becomes a chlorine gas and is dissolved during electrolytic oxidation of hydrochloric acid, it is easy to separate a desired product from the chlorine gas by degassing as a post-treatment; therefore, it is possible to synthesize a high purity perchloric acid compound by the hydrochloric acid electrolytic oxidation. Though separation of chemicals such as sulfuric acid is essential in the above-described chemical synthesis method, the separation is not required or easily performed in this invention. Thus, this invention is advantageous for synthesizing the perchloric acid compound at high purity.

[0020] In the case of using the anode having the electroconductive diamond as the anode substance for electrolytic synthesis of a perchloric acid compound from a chloric acid compound, it is possible to obtain the desired compound at higher yield and purity as compared to the case of using the conventional anode.

[0021] Further, when an anode chamber and a cathode chamber are partitioned by a membrane such as a cation exchange film, it is possible to prevent transfer of the chloride ion and the chloric acid ion, thereby contributing to improvement of efficiency and purity.

[0022] When a gas electrode suitable for reducing an oxygen gas is used as the cathode, it is possible to lower a power source unit by 0.5 V or more as a cell voltage.

[0023] The use of the gas electrode for hydrochloric acid electrolysis for obtaining a chlorine gas has been known, but use of the gas electrode in the above-described perchloric acid electrolytic synthesis has not been known.

[0024] This invention relates to an electrolysis cell and a method for synthesizing a perchloric acid compound which is an industrially important oxidant at high purity, and, more specifically, this invention relates to an electrolytic synthesis of a desired perchloric acid compound by using a solution containing a chloride compound or a chloric acid compound as a feedstock electrolysis liquid and electrolyzing the electrolysis liquid with the use of an anode having an electroconductive diamond as an anode substance.

[0025] Unlike the conventional methods wherein perchloric acid compound electrolytic synthesis is performed after obtaining sodium chlorate by using hydrochloric acid used as a feedstock, this invention enables to obtain the desired compound by one process step (chloric acid compound is produced as an intermediate product in one case, and chloric acid compound is not produced as the intermediate compound in the other case) by using the chloride as the feedstock. Further, when hydrochloric acid is used as the feedstock becomes a chlorine gas and is dissolved, and it is possible to synthesize the high purity perchloric acid compound by degassing the chlorine gas since it is easy to separate the chlorine gas from the desired product as a post-treatment.

[0026] When the chloric acid compound is used as the feedstock, it is possible to obtain the desired compound at higher yield and purity as compared to the case of using the conventional anode. This method is applicable to improvement of product quality by electrolyzing an electrolysis liquid containing residual chloric acid of 0.1 M or less, which has been synthesized by the conventional electrolysis cell in the electrolysis cell having the diamond electrode, and such combined method is industrially effective.

[0027] When the electrolysis cell is partitioned by using an ion exchange membrane to obtain an anode chamber and a cathode chamber, it is possible to prevent ions or the like from migrating from one chamber to the other chamber, thereby enabling to obtain the desired compound at higher purity.

[0028] Also, when a gas electrode suitable for reduction of an oxygen gas is used as the cathode, it is possible to lower a cell voltage, thereby enabling to obtain the desired compound at a lower cost.

[0029] Though it is essential to separate chemicals such as sulfuric acid in the conventional chemical synthesis methods, this invention has the advantage of synthesizing high purity perchloric acid conveniently and contributes to industrial safety and environment.

BRIEF DESCRIPTION OF THE DRAWINGS

[0030] FIG. 1 is a schematic vertical sectional view showing a separating-membraneless electrolysis showing an electrolysis cell for synthesizing a perchloric compound according to this invention.

[0031] FIG. 2 is a schematic vertical sectional view showing a separating-membrane electrolysis.

[0032] The reference numerals used in the drawings denote the followings, respectively.

[0033] 1, 11: electrolysis cell

[0034] 2, 15: anode

[0035] 3, 16: cathode

[0036] 12: positive ion exchange membrane

DETAILED DESCRIPTION OF THE INVENTION

[0037] Hereinafter, each of elements of this invention will be described in detail.

Anodic Reaction

[0038] In the case where hydrochloric acid is used as a feedstock in an anodic reaction in an electrolysis cell of this invention (acidic region), the reaction is represented by the following Expression (1), and perchloric acid ions are generation by the reaction.



[0039] Further, a side reaction represented by Expression (2) proceeds in addition to the direct electrolytic oxidation process of Expression (1), so that the generated chlorine reacts with water to generate perchloric acid ions in accordance with Expression (3).



[0040] Further, chloric acid ions are generated as a byproduct in accordance with Expression (4), and the thus-generated chloric acid ions react with water in accordance with Expression (5), so that a perchloric acid compound (perchloric acid ions) that is the desired compound is generated.



[0041] A water electrolysis represented by Expression (6) occurs as a side reaction.



[0042] Generation of OH radicals is considered to occur as an elementary step of the reaction, and there is a pathway wherein the radicals react with the chlorine ions to generate the perchloric acid compound that is the desired product.

Cathodic Reaction

[0043] A Cathodic reaction differs depending on presence/absence of oxygen supply.

[0044] In the case where oxygen is not supplied, hydrogen is generated in accordance with Expression (7).



[0045] In the case where oxygen is supplied, water is generated in accordance with Expression (8), or hydrogen peroxide is synthesized in accordance with Expression (9).



Electrode Substrate of Diamond Electrode

[0046] As an electrode substrate, Si is generally used from the view point of chemical stability such as thermal expansion rate consistency, stability under synthesis conditions such as a hydrogen atmosphere (brittleness due to hydrogen occlusion, etc.). However, since Si is a semiconductor material, it is necessary to dope boron or the like to achieve good electroconductivity. A surface of the electrode substrate may preferably be uneven in order to enhance mechanical strength. It is desirable to perform polishing and nucleation with the use of diamond particles in order to accelerate precipitation of diamond. It is possible to use Nb, Ta, Zr, Ti (valve metal) or Mo, W, graphite, or various carbides other than Si.

Diamond Electrode

[0047] An electroconductive diamond electrode is generally produced by forming an electroconductive diamond layer on an electroconductive substrate by thermal filament CVD or microwave plasma CVD. By activating a mixture gas having an appropriate composition containing hydrogen, carbon, and boron (or nitrogen) materials under a pressure of 1 to 100 kPa on a hot filament (1,800° C. to 2,600° C.), carbon and hydrogen radical species are generated. A volumetric ratio between the hydrogen and the carbon gas feedstock is controlled to about 0.05 to 0.1. Though a gas feedstock such as methane and diborane is ordinarily used, use of alcohols and boron oxide is preferred from the view point of safety in production site. By maintaining a substrate temperature to 600° C. to 900° C., a carbon radical precipitation reaction is started on the substrate. Since a non-diamond component is etched by the hydrogen radicals, only a diamond layer is grown. A precipitation rate is 0.1 to 5 μm/H. It is considered that the stable carbide layer that is

generated on the substrate under the precipitation conditions contributes to improvement of adhesion strength. A doping amount is from 100 ppm to 10,000 ppm, and resistivity changes in inversely proportional to the doping amount (10 to 0.01 Ωcm). An optimal thickness is 1 to 10 μm from the view points of electrode endurance (protection of substrate) and a production cost.

[0048] As a result of SIMS analysis, it is confirmed that B/C ratios of the supply gas and the generated layer are substantially identical to each other. It is possible to confirm that the coating layer formed by the CVD is diamond by raman spectrum. It is possible to confirm a precipitation state of polycrystalline structure having particle diameter of 0.1 to 10 μm by SEM picture.

[0049] The electrode to be used in this invention is not particularly limited to that prepared by the CVD, and it is possible to use an electrode having an electrode substrate on which powder particles are fixed.

Cathode

[0050] In the case where the feedstock is chloric acid in the cathodic reaction involving the hydrogen generation of Expression (7), the cathode is required to have chemical endurance since it is used in the acidic atmosphere. A substrate of graphite, zirconium, or the like is suitably used. In order to lower a voltage, it is preferable to coat a surface of the cathode with a component (platinum group metal, other oxides) excellent in catalyst activity.

[0051] In the case where the chloride is used as the feedstock, a substrate of graphite, stainless, nickel, or the like is suitably used since the cathode is used in the alkali atmosphere. In order to lower a voltage, it is preferable to coat a surface of the cathode with a component (platinum group metal, other oxides) excellent in catalyst activity.

[0052] The use of the diamond electrode for the cathode is preferable since the diamond electrode has the chemical stability.

[0053] In turn, it is possible to lower a cell voltage by supplying oxygen to proceed the oxygen gas reduction reaction of Expression (8) as the cathodic reaction. When a specific catalyst is used, the oxygen gas reduction reaction preferentially proceeds to generate the hydrogen peroxide of Expression (9). In this case, the chloride is generally used as the feedstock since efficient production is achieved in the alkali aqueous solution atmosphere.

[0054] As the catalyst for the oxygen gas cathode suitable for hydrogen peroxide, a platinum group metal, a noble metal, oxides thereof, sulfide, or carbon such as graphite, or electroconductive diamond may preferably be used. These catalysts may be used alone in the form of a plate or may be formed on an anti-corrosion plate such as a stainless plate and a carbon plate; a metal mesh; a powder sintered material; or a metal fiber sintered material by a thermal decomposition method, a fixing method using a resin, composite plating, or the like in an amount of 1 to 1,000 g/m².

[0055] As a cathode power feeder, a metal such as carbon, nickel, and stainless, alloys thereof, or oxides thereof may preferably be used. In order to promptly supply and remove the reaction generation gas and liquid, it is preferable to disperse a hydrophobic material or a hydrophilic material. Formation of a hydrophilic sheet on a backside of the cathode that is opposite to the anode is effective, since it is possible to control the gas supply to the reaction surface by using the sheet.

[0056] An amount of oxygen to be supplied may preferably be 1.1 to 10 times that of a theoretical value. The oxygen gas used as the feedstock may be the air or oxygen prepared by separating and concentrating the air may be used, and it is possible to use commercially available oxygen bombs. Oxygen is supplied to a gas chamber at the rear of the electrode when such gas chamber is equipped, but it is possible to inject oxygen into water to be treated in advance of the electrolysis.

Ion Exchange Membrane

[0057] In the case where a cation exchange film is used as the membrane to be used in this invention so as to obtain a two-chamber type cell having an anode chamber and a cathode chamber, it is possible to prevent transfer of the chloride ions and the chloric acid ions, thereby contributing to improvement in synthesis efficiency. Also, in the case where the concentration of the electrolysis solution is lowered, the cation exchange film is necessary for maintaining ion conductivity.

[0058] The ion exchange membrane may be either one of a fluorine resin-based film or a hydrocarbon resin-based film, and the former one is preferable from the view point of anti-corrosion property. Examples of a resin excellent in chemical resistance include a fluorinated resin having a sulfone group as an ion exchange group (Nafion as a commercially available product of Du Pont). Nafion is produced from a copolymer of tetrafluoroethylene and perfluoro[2-(fluorosulfonylethoxy)-propyl]vinylether.

Cell Structure

[0059] As a material for the electrolysis cell, a glass lining material, carbon, titanium excellent in anti-corrosion property, stainless, a PTFE resin, and the like are preferred from the view point of stability.

[0060] The electrolysis cell is constructed by using the above materials. It is possible to use a non-partitioned cell (FIG. 1) having two porous electrodes (anode and hydrogen cathode) which will be described later in this specification, a two-chamber cell provided with a membrane sandwiched between the anode and the hydrogen cathode of the cell of FIG. 1, a two-chamber cell (FIG. 2) having a porous anode and an oxygen gas cathode and a membrane sandwiched between the anode and the cathode, or the like.

[0061] In the two-chamber cell, it is necessary to keep a distance between the electrodes as small as possible in order to lower a resistivity loss, and it is preferable to keep the distance to 0 to 2 mm in order to regulate a pressure loss of a pump for supplying water as small as possible as well as to maintain uniform pressure distribution.

Operational Conditions

[0062] An initial concentration of hydrochloric acid, other chlorides and chloric acid, or chlorate, which are feedstocks, may preferably be 0.1 M or more. In the case where hydrochloric acid or other chlorides are used as the feedstock, chloric acid ions are generated as an intermediate product by the electrolysis, and a chloric acid ion concentration will be lowered while a perchloric acid ion concentration is increased. By continuing the electrolysis, it is possible to lower the feedstock concentration to 0.01 M or less. Though it is possible to decompose whole feedstock ultimately, it is preferable to lower the concentration to an appropriate value and to perform separation by another

separation method since a current efficiency is lowered and an excessive power is used for decomposing the whole feedstock.

[0063] Though the reaction speed is increased to reach an equilibrium value in a short time when a temperature is high among the electrolysis conditions, the decomposition rate is increased when the temperature is too high. Therefore, an appropriate temperature range under the atmosphere is from a room temperature to less than 100° C. A current density may preferably be 1 to 100 A/dm².

[0064] In the case of using hydrochloric acid as the feedstock, it is preferable to proceed the desired reaction by increasing solubility in order to increase the current efficiency since chlorine is the intermediate product during the perchloric acid synthesis as indicated by Expression (7). Therefore, a pressure may preferably be as high as possible, and an appropriate pressure is 0.1 to 1 MPa.

[0065] In the case where a chlorine gas remains, it is possible to separate and remove the chlorine gas by degassing operation.

[0066] Shown in FIGS. 1 and 2 are electrolysis cells for synthesizing a perchloric acid compound according to this invention, wherein shown in FIG. 1 is one example of a membraneless cell using a gas generation electrode for each of an anode and a cathode, and shown in FIG. 2 is one example of a membrane electrolysis cell using a gas generation electrode as an anode and an oxygen gas electrode as a cathode.

[0067] The electrolysis cell 1 shown in FIG. 1 does not use any membrane, and a porous anode 2 obtained by coating a substrate which is made from silicon or the like with an electroconductive diamond and a porous cathode 3 obtained by coating a substrate which is made from nickel or the like with a cathode material such as platinum and a platinum group metal oxide are disposed in the electrolysis cell 1 with a gap being defined therebetween.

[0068] Reference numerals 4 and 5 denote feed stock water inlets, and reference numerals 6 and 7 denote generated gas outlets formed on an upper surface of the electrolysis cell.

[0069] When the electrodes are electrically conducted to each other with a chloride solution or a chloric acid compound solution of sodium chlorate or the like being supplied to the electrolysis cell 1, perchloric acid ions are synthesized by electrolysis in accordance with Expressions (1), (3), and (5).

[0070] The electrolysis cell 11 shown in FIG. 2 is divided into an anode chamber 13 and a cathode chamber 14 by a perfluorosulfonic acid-based positive ion exchange membrane 12. A porous anode 15 that has a substrate and an electroconductive diamond coated on the substrate is in close contact with one side of the positive ion exchange membrane 12 in the anode chamber 13, and a gas diffusion cathode 16 is in close contact with the other side of the positive ion exchange membrane 12 in the cathode chamber 14. A porous cathode power feeder 17 is connected to the gas diffusion cathode 16 in a closely contacted fashion for power supply and reinforcement.

[0071] Reference numeral 18 denotes an anodic liquid inlet formed on a bottom surface of the anode chamber of the electrolysis cell; reference numeral 19 denotes a cathode gas inlet formed on a bottom surface of a cathode gas chamber of the electrolysis cell; reference numeral 20 denotes an

anode gas outlet formed on the upper surface of the electrolysis cell; and reference numeral **21** denotes a cathode gas outlet formed on the upper surface of the electrolysis cell.

[0072] When the electrodes are electrically conducted to each other with a chloride solution of hydrochloric acid, sodium chloride, or the like or a chloric acid compound solution of sodium chlorate or the like being supplied to the anode chamber **13** of the electrolysis cell **11** while supplying an oxygen-containing gas to the cathode gas chamber **14**, the perchloric acid ions are synthesized by electrolysis in the same manner as in the electrolysis cell of FIG. 1.

EXAMPLES

[0073] Hereinafter, examples and comparative examples relating to perchloric acid compound electrolytic synthesis according to this invention will be described, but it should be understood that the present invention is not to be construed as being limited thereto.

Example 1

[0074] An anode obtained by forming a thickness of 5 μm of an electroconductive diamond doped with boron of 5,000 ppm on electroconductive silicon having a thickness of 1 mm serving as a substrate was used. As a cathode, a platinum-plated titanium plate having a thickness of 1 mm was used. An electrode area of each of the anode and the cathode was 20 cm^2 .

[0075] These electrodes were disposed in the membraneless cell (capacity: 1000 ml) shown in FIG. 1 in such a fashion that they are opposed to each other with a gap of 1 cm being defined therebetween. The cell was charged with 500 ml of a 0.02 M sodium chlorate solution, and electrolysis was performed for one hour with a current of 1 A (current density: 0.05 A/cm^2) with stirring with a water temperature being kept to 35° C.

[0076] Analysis was performed by using an ion chromatography device, and a current efficiency of the perchloric acid ion generation was calculated from the concentration. Sodium hypochlorite was measured by KI titration. It was confirmed that the current efficiency of perchloric acid ion generation was 20%.

[0077] Further, the electrolysis was continued for 4 hours to find that sodium chlorate used as the feedstock was lowered to 2 mM, while the hypochlorous ions generated as a byproduct were lowered to 0.1 mM.

Comparative Example 1

[0078] Electrolysis was conducted for a total of 5 hours in the same manner as in Example 1 except for using a platinum titanium plate in place of the diamond electrode, and generation of perchloric acid ions was not confirmed. The result was different from the conventional ones, but it is estimated that the perchloric acid ion was short of the detection limit since a feedstock concentration and a current density were small.

Comparative Example 2

[0079] Electrolysis was conducted for a total of 5 hours in the same manner as in Example 1 except for using a titanium plate on which lead oxide is precipitated in place of the diamond electrode, and a current efficiency of perchloric acid ion generation was about 2%.

Example 2

[0080] An anode obtained by forming an electroconductive diamond doped with boron of 5,000 ppm on a substrate which was a niobium mesh having an original plate thickness of 2 mm was used. As a cathode, a platinum-plated titanium mesh having an original plate thickness of 1 mm was used. An electrode area of each of the anode and the cathode was 20 cm^2 , and a two-chamber cell wherein an ion exchange membrane (117 Nafion) was sandwiched between the anode and the cathode of the membraneless cell of FIG. 1 is assembled (gap between electrodes: 1 mm), followed by injecting 200 ml of a 0.5 M hydrochloric acid solution to the anode chamber and the cathode chamber. By performing electrolysis for one hour under conditions of 40° C. and 20 A (current density: 1 A/cm^2), perchloric acid ions were obtained at a current efficiency of 12%. A chloric acid concentration during the electrolysis was lowered to 0.05 M. A cell voltage was 5.5 V. A residual chlorine gas was removed by degassing.

Comparative Example 3

[0081] Electrolysis was conducted in the same manner as in Example 2 except for using a platinum-plated titanium mesh having an original plate thickness of 1 mm as an anode, and a current efficiency of perchloric acid ion generation was about 0.2%.

Example 3

[0082] The anode of Example 2 was used, and a sheet having the size of 2 $\text{cm} \times 2 \text{ cm} \times 0.5 \text{ mm}$ of thickness obtained by: mixing a graphite powder (product of Tokai Carbon Co., Ltd.; TGP-2), which was used as a catalyst, with a PTFE resin; applying the mixture on a core material which was a carbon cloth (product of Zoltek Corporation; PWB-3), and baking at 330° C. was used as the cathode. A platinum-plated titanium mesh was used as a cathode power feeding support, and a two-chamber cell wherein the ion exchange membrane of FIG. 2 (Nafion 117) was disposed between the chambers was assembled. As a result of electrolysis for one hour at 60° C. and 20 A in the same manner as in Example 2 with oxygen being supplied to the cathode chamber, perchloric acid ions were obtained at a current efficiency of 10%. A hydrochloric acid concentration during the electrolysis was lowered to 0.05 M. A cell voltage was 4.7 V.

[0083] While the present invention has been described in detail and with reference to specific embodiments thereof, it will be apparent to one skilled in the art that various changes and modifications can be made therein without departing from the spirit and scope thereof.

[0084] The present application is based on Japanese Patent Application No. 2006-015201 filed on Jan. 24, 2006, and the contents thereof are incorporated herein by reference.

What is claimed is:

1. An electrolysis cell for synthesizing a perchloric acid compound, the electrolysis cell comprising:
 - a feedstock solution containing a chloride;
 - a cathode; and
 - an anode having an electroconductive diamond as an anode substance,
 the anode electrolytically oxidizing the chloride to synthesize the perchloric acid compound.

2. An electrolysis cell for synthesizing a perchloric acid compound, the electrolysis cell comprising:

a feedstock solution containing a chloric acid compound;
a cathode; and

an anode having an electroconductive diamond as an anode substance,

the anode electrolytically oxidizing the chloric acid compound to synthesize the perchloric acid compound.

3. The electrolysis cell according to claim 1,

wherein an ion exchange membrane is disposed between the anode and the cathode.

4. The electrolysis cell according to claim 2,

wherein an ion exchange membrane is disposed between the anode and the cathode.

5. The electrolysis cell according to claim 3,

wherein the cathode is a gas diffusion cathode for electrolytically reducing an oxygen.

6. The electrolysis cell according to claim 4,

wherein the cathode is a gas diffusion cathode for electrolytically reducing an oxygen.

7. A method for electrolytically synthesizing a perchloric acid compound, the method comprising electrolyzing a feedstock solution containing a chloride with an anode having an electroconductive diamond as an anode substance to synthesize the perchloric acid compound.

8. A method for electrolytically synthesizing a perchloric acid compound, the method comprising electrolyzing a feedstock solution containing a chloric acid compound with an anode having an electroconductive diamond as an anode substance to synthesize the perchloric acid compound.

9. The method for electrolytically synthesizing a perchloric acid compound according to claim 7,

wherein the concentration of the feedstock solution is 0.1 M or more, and the concentration is reduced to 0.01 M or less through the electrolyzing.

10. The method for electrolytically synthesizing a perchloric acid compound according to claim 8,

wherein the concentration of the feedstock solution is 0.1 M or more, and the concentration is reduced to 0.01 M or less through the electrolyzing.

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