(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
22 November 2007 (22.11.2007)

PCT

(10) International Publication Number
WO 2007/133535 A2

(51) International Patent Classification:
F25D 19/00 (2006.01) *F25D 19/02* (2006.01)

(21) International Application Number:
PCT/US2007/011091

(22) International Filing Date: 8 May 2007 (08.05.2007)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
60/746,703 8 May 2006 (08.05.2006) US

(71) Applicant (*for all designated States except US*): **SIEMENS WATER TECHNOLOGIES CORP.** [US/US]; 181 Thorn Hill Rd., Warrendale, Massachusetts 15086 (US).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **MARTIN, Barry, L.** [US/US]; 8132 South Chariot Street, Concord, Ohio 44060 (US). **ZOLOTARSKY, Vadim** [US/US]; 62 Henshaw Avenue, Springfield, New Jersey 07920 (US). **IVANTER, Irina, A.** [RU/US]; 14 Pero Court, Sayreville, New Jersey 08872 (US). **ANTONIOU, Michael** [US/US]; 220 Spruce Mill Lane, Scotch Plains, New Jersey 07076 (US).

(74) Agents: **MUSACCHIO, Pasquale** et al.; Siemens Corporation- Intellectual Property Dept., 170 Wood Avenue South, Iselin, New Jersey 08830 (US).

(81) Designated States (*unless otherwise indicated, for every kind of national protection available*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (*unless otherwise indicated, for every kind of regional protection available*): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:
— *as to the identity of the inventor (Rule 4.17(i))*

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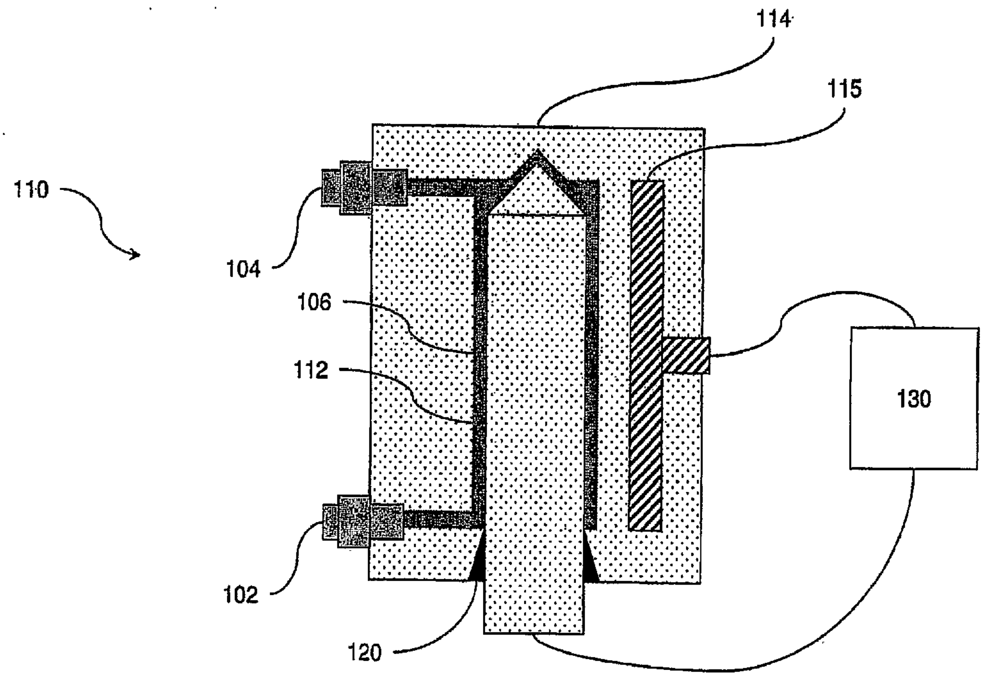
(54) Title: ELECTROLYTIC APPARATUS WITH POLYMERIC ELECTRODE AND METHODS OF PREPARATION AND USE



(57) Abstract: The invention provides low cost methods and apparatus that utilize at least one carbon-filled polymeric electrode to electrolytically generate desirable products.

WO 2007/133535 A2

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*
— *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))*

Published:
— *without international search report and to be republished upon receipt of that report*

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ELECTROLYTIC APPARATUS WITH POLYMERIC ELECTRODE AND METHODS OF PREPARATION AND USE

BACKGROUND OF INVENTION

1. Field of Invention

This invention relates to electrolytic apparatus having at least one polymeric electrode and to methods of construction and use thereof and, in particular, to electrolytic apparatus comprising at least one polymeric electrode that electrolytically generates oxidizing species.

2. Discussion of Related Art

Electrolytically-generated disinfecting solutions have been disclosed. For example, Barger, et al., in U.S. Patent No. 6,255,270, disclose cleaning and disinfecting compositions with an electrolytic disinfecting booster. Tremblay, et al., in U.S. Patent Application Publication No. 2003/0042134, disclose a high efficiency electrolysis cell for generating oxidants in solutions. Indeed, Logan, in U.S. Patent No. 2,163,793, disclose electrolytically producing chlorine dioxide.

Kadlec, et al., in U.S. Patent No. 6,869,518, disclose the electrochemical generation of chlorine dioxide. Chen et al., in U.S. Patent No. 6,921,521, disclose a method of producing chlorine dioxide that employs alkaline chlorate in a mineral acid medium and urea as a reducing agent. Scheper, et al., in U.S. Patent No. 6,921,743, disclose automatic dishwashing compositions containing a halogen dioxide salt and methods for use with electrochemical cells and/or electrolytic devices. Price, et al., in U.S. Patent Application Publication No. 2003/0213503, disclose signal-based electrochemical methods for automatic dishwashing.

Scheper, et al., in U.S. Patent Application Publication No. 2003/0213704, disclose a self-contained, self-powered electrolytic device for improved performance in automatic dishwashing.

Herrington, in U.S. Patent No. 7,008,523, discloses an electrolytic cell for surface and point of use disinfection. Tremblay, et al., in U.S. Patent Application Publication No. 2004/0149571, disclose electrolytic cell for generating halogen dioxide in an appliance. Tremblay, et al., in U.S. Patent No. 7,048,842, disclose an electrolytic cell for generating chlorine dioxide.

Roensch, et al., in U.S. Patent No. 7,077,995, disclose a method for treating aqueous systems with locally generated chlorine dioxide.

SUMMARY OF THE INVENTION

In accordance with one or more embodiments, the invention relates to an electrolytic apparatus comprising an electrolytic cell having at least one carbon-filled polymeric electrode.

In accordance with one or more embodiments, the invention relates to a method comprising providing an electrolytic cell having at least one carbon-loaded polymeric electrode.

BRIEF DESCRIPTION OF THE DRAWING

The accompanying drawing is not intended to be drawn to scale. For purposes of clarity, not every component may be labeled in the drawing.

In the drawing, FIG. 1 illustrates an electrolytic apparatus in accordance with one or more embodiments of the invention.

DETAILED DESCRIPTION

The present invention can provide electrolytic apparatus, systems utilizing one or more electrolytic apparatus, as well as techniques that involve such electrolytic devices. In accordance with some aspects, one or more embodiments of the invention involve electrolytic devices having at least one polymeric electrode. In some cases, the electrolytic device of the invention can comprise a plurality of polymeric electrodes. For example, the electrolytic device can have at least one electrode serving as a cathode comprised of a polymeric material and, optionally, one or more electrodes serving as an anode comprising the same or different polymeric material. In some cases, the polymeric electrode can be considered to be loaded with electrically conductive components. In some aspects, the invention provides relatively low cost components compared to conventional electrodes but with comparable performance. The polymeric electrode can be a carbon-filled polymeric electrode. Further embodiments can involve utilizing other components that facilitate conduction or conveyance of an applied electric current through the one or more polymeric electrodes of apparatus of the invention. The polymeric electrode can further comprise one or more electrical cores or components that provide electrical conductivity throughout the body of the electrode. For example, the electrolytic cell can have an anode and a cathode, any one or both can be a polymeric electrode with at least one metallic core embedded therein. The metallic core thus serves to electrically conduct current and reduces the likelihood of resistive gradient through the electrode.

The electrolytic devices of the invention can be utilized for electrocatalytic generation of one or more products from one or more precursor species. Some aspects of the invention involving the various electrolytic devices of the invention can be directed to generating an oxidizing species. In particular, some aspects of the invention can involve electrolytically generating one or more halogenated oxidizing agent. For example, one or more electrolytic embodiments of the invention can involve electrolytically generating a chlorinated, brominated, or fluorinated compound, or mixtures thereof, that can oxidize one or more target compounds, non-limiting examples of which include, bacteria. In accordance with some particularly advantageous embodiments, the various electrolytic embodiments of the invention can be used to generate one or more of chlorine, hypochlorous species, and chlorine dioxide. Further, some embodiments of the invention provide an oxidizing species that can be present or carried in a disinfecting solution generated in situ, typically for immediate delivery and use. As used herein, disinfecting refers to at least partially rendering organisms biologically inactive or inert or incapable of further reproduction or colony propagation.

Further particular embodiments of the invention involve providing an electrolytic apparatus comprising at least one polymeric electrode. The following discussion involves generation of chlorine dioxide but the invention is not limited as such and can be used to generate other desirable species. The electrolytic apparatus, exemplarily illustrated in FIG. 1, in one or more embodiments of the invention can have one or more electrolytic cells 110, one or more of which can comprise at least one cathode 112 and at least one anode 114. Electrolytic apparatus 100 can further comprise one or more sources of electrolyte in fluid communication or at least capable of being in fluid communication with electrolytic cell 110. A plurality of sources of electrolytic fluids can be utilized to flexibly provide functionalities selectively chosen by an operator of the apparatus. Thus, for example, the electrolytic apparatus can comprise a first or primary source of a first electrolyte comprising one or more precursor compounds and an alternate or supplemental source of a second electrolyte comprising one or more alternative precursor compounds. The electrolyte can further comprise at least one oxidizing agent selected from the group consisting of chlorates, perchlorates, hypohalites, permanganates, chromates, and peroxides. In other cases, the electrolyte can consist of or consist essentially of a halite or a halide such as a chlorite or a chloride.

The electrolytic cell can be fluidly connected to a source of electrolytic fluid through inlet port 102. Cell 110 further comprises at least one outlet port 104 for delivery of a generated product to a point of use. Cell 110 has a body 114 containing a cavity 106 which during operation is filled with the electrolyte from the one or more sources of electrolyte having at least one precursor species therein. Cell 110 further has an anode 112 connected to a power source 130. Body 114 can also serve as a cathode and is illustrated as electrically connected to power source 130 through one or more metallic, conductive cores 115 at least partially embedded within body 114. Typically, a member 120 can secure and electrically insulate anode 112 from cathodic body 114. Thus, some electrolytic cells of the invention can comprise a body 114 that serves to contain electrolyte and facilitates electrolytic conversion of a precursor compound into one or more generated oxidizing compounds. In alternative embodiments, the body of the electrolytic cell can serve as the anode.

This aspect of the invention facilitates production of electrolytic cell especially where the polymeric materials are employed. Thus, some aspects of the invention can provide a castable or moldable electrode configured to contain electrolyte.

During operation, an applied current is conducted through the electrolytic cell to generate one or more desired compounds from the one or more precursor compounds in the electrolytic fluid transferred into cell. For example, chlorine dioxide can be generated at or near an electrode, typically at or near the anode, of the electrolytic cell, from a precursor chlorite species in the electrolyte. In some particular embodiments of the invention, halite compounds can be electrolytically converted to provide a disinfecting or deodorizing solution comprising chlorine dioxide. Other suitable desirable or ancillary reactions may be facilitated including those that generate hypochlorous species, chlorine, and other oxidizing species. As noted herein, however, ancillary oxidizing species may also be present in the electrolyte.

The electrical current can be provided by one or more electrical sources. In some cases, the electrical source provides direct current potential of less than about 6 volts but in some cases, less than about 4.5 volts and in still other cases, less than about 3 volts.

Depending on service, lower potentials can be used to provide sufficient generation of the desired product. Typically, however, a minimum potential, such as at least about 2 volts, may be preferred to provide at least partial conversion of the precursor compounds into one or more desirable oxidizing products. Particularly advantageous embodiments of the electrical source can involve conventional primary cells such as zinc-carbon, alkaline, or lithium based electrochemical cells or batteries as well as secondary or rechargeable batteries such, but not limited to, nickel cadmium or nickel metal hydride, or lithium ion cells. Preferably, the electrical source can comprise one or more cells such as those having size designations of "AA," "AAA," "C," and "D."

Where the electrolytic apparatus comprises a plurality of electrolytic cells, one or more of the at least one sources of electrolytic fluid can be fluidly connected, or be connectable to the any one of the electrolytic cells. In some embodiments of the invention, one or more components of the electrolytic apparatus can be removed and replaced. For example, the source of electrolyte can be removed, accessed or otherwise filled with fresh electrolyte. Similarly, the power source can be replaced or recharged or otherwise be re-energized and thereby further provide electrical current for the cell.

At least a portion of the generated product solution can then be used to at least partially disinfect or deodorize a point of use such as a surface. Delivery of the product solution comprising the one or more generated oxidizing agents can be effected utilizing any suitable technique. For example, the generated disinfecting or deodorizing solution can be rendered airborne as an aerosol or be sprayed on at least a portion of the surface desired to be decontaminated. In other cases, the generated solution comprising the one or more oxidizing agents can be transferred into a bath comprising the same or different desirable oxidizing agents. The target article can then be immersed therein so as to facilitate oxidizing or inactivation of the target compounds by the one or more oxidizing species.

The polymeric electrode can utilize any suitable binding component or matrix. For example, the electrode can comprise thermoplastic or thermosetting polymeric materials with electroconductive or electroactive components, non-limiting examples include graphite and electrically conductive carbon. The polymeric electrode can be formed by injection molding or similar techniques utilized to fabricate polymeric components. Indeed, moldability of the polymeric materials can provide low cost electrodes having increased surface areas, relative to conventional non-polymeric based electrodes, thereby reducing the effective current density and, in some cases, lowering the operating voltage. For example, the polymeric electrodes can be configured to have ribs that increase the effective surface area. Further, improved cell fluid dynamics can be realized by casting at least a portion of the cell with smooth surfaces and a surface profile that improves mass transfer through the cell and, in some cases, also reduces any tendency for precipitation of calcerous deposits. Non-limiting examples of polymeric binders include polyethylene, polypropylene, polystyrene, polytetrafluoroethylene, polyethyleneterephthalate, polyvinylchloride, polycarbonate, nylon, polymethylmethacrylate, and blends or copolymers thereof. The polymeric binder can further comprise reinforcing agents such as fibers. In some cases, the reinforcing component can also serve to facilitate electrical conductivity. For example, the electrode can comprise a reinforcing metallic core, comprised of for example, copper, connected to the power source. In some cases, at least a portion of the polymeric electrodes can further comprise an electrocatalytic coating. Non-limiting examples of which include valve metals, precious metals, platinum group metals, as well as their oxides and mixtures thereof. Other coating materials that can be utilized include, for example, CoO_2 and MnO_2.

The electrode can further utilize a reticulated or mesh substrate in the molded electrode. For example, the polymeric material can be molded with a titanium mesh. The conductive characteristics of the cell can be improved by facilitating further electrical contact points by, for example, using high pressure points and also by utilizing reticulated structures to further enhance contact characteristics. The reticulated structures can be comprised of, for example, copper, nickel, aluminum, and silver.

The various system and techniques of the invention can be used in applications other than in the exemplarily disclosed chlorine dioxide-generating embodiments including, for example, in electrochlorination, generation of mixed oxidants, and swimming pool chlorinators. Other applications include use as electrodeionization cathodes.

Examples

The function and advantages of these and other embodiments of the invention can be further understood from the examples below, which illustrate the benefits and/or advantages of the one or more systems and techniques of the invention but do not exemplify the full scope of the invention.

Example 1

This example compares the performance of an electrolytic cell comprising a carbon-loaded polyethylene electrode, from Covalence Specialty Materials Corp. (Franklin, Massachusetts), relative to a cell with a bare titanium electrode. The anode in both cells was a titanium mesh electrode with OPTIMA® RUA-SW electroactive coating, from Siemens Corporation (Union, New Jersey). The electrode gap was 2 mm. The electrolyte solution used was 1 M sodium chloride.

Table 1 lists the measured potential at various operating current densities. The data shows higher specific resistivity of carbon-loaded polyethylene electrodes relative to titanium cathodes.

Table 1. Measured cell voltage, in volts, using a carbon-loaded polyethylene cathode or a titanium cathode.

Current Density (kA/m^2)	Carbon Loaded Polyethylene	Titanium
0.5	3.77	3.44
1.0	4.3	4.01
2.0	5.13	5.13

Example 2

In this example, the polymeric electrodes of Example 1 was modified to improve performance by utilizing metallic components, four aluminum bars, and having several contact points. The anode used was a titanium sheet with OPTIMA® RUA electroactive coating, from Siemens Corporation. The electrodes gap was 1.6 mm and the electrolyte solution was 1 M sodium chloride.

Table 2 lists the measured potential with various contact points and shows that increased points of contact can improve cell characteristics because the carbon-loaded can, in some cases, be considered as a polyethylene filled with carbon particles in contact with each other to provide a conductive pathway and to the surface of the electrode.

Table 2. Measured cell potential using a carbon-loaded polyethylene cathode with several contact points.

Current Density (kA/m^2)	Metallic Component, Contacts	Voltage (volts)
0.5	Al bars 4	4.6
	Al bars 4, 2	4.48
	Al bars 4, 3	4.34
	Al bars 4, 3	4.3

Example 3

This example shows the performance of a cell utilizing the carbon-loaded polyethylene cathode of Example 1 and further comprising a silver reticulated structure to facilitate electrical conduction. The anode was comprised of titanium sheet catalyzed with OPTIMA® RUA coating. The electrodes gap was 1.6 mm and the electrolyte was 1 M sodium chloride.

Table 3 lists the measured potential with and without the reticulated conductor and shows the improved performance.

Table 3. Measured cell potential using a carbon-loaded polyethylene cathode with and without a reticulated conductor.

Current Density (kA/m^2)	Contacts	Voltage (volts)
0.5	Al bars 4, 3 contacts	3.75
	Al bars 4, 3 contacts with reticulated silver conductor	2.5

Example 4

To further improve the performance, the carbon-loaded polyethylene cathode of Example 1 was treated with agents that reduce surface tension. The anode used was a titanium sheet catalyzed with OPTIMA® RUA coating. The electrodes gap was 1.6 mm and the electrolyte was sodium chloride. The carbon-loaded polyethylene cathode was used with two aluminum bars and two silver strips of reticulated foam and the data presented in Table 4 show that such components facilitate electrical contact by lowering the cell potential.

Table 4. Measured cell potential using a carbon-loaded polyethylene cathode with various surface treatments.

Current Density (kA/m^2)	Treatment	Voltage (volts)
0.5	none	3.16
	1 hour of operation electrolytically	2.68
	1 hour of operation electrolytically and 15 minutes of contact with n-propanol	2.54
	3 hours of operation electrolytically and 15 minutes of contact with n-propanol	2.24
	5 hours of operation electrolytically and 15 minutes of contact with n-propanol	2.2
	5 hours of operation electrolytically and 15 minutes of contact with n-propanol	2.29

Example 5

In this example, an alternative carbon-based material, GRAFCELL® graphite plate, from GrafTech International Ltd. (Parma, Ohio) was used as the cathode. It is believed that the greater carbon content, relative to the carbon-loaded electrode, should provide improved specific resistivity, and lower contact resistance. The anode was comprised of titanium sheet catalyzed with OPTIMA® coating. The electrodes gap was 1.6 mm and the electrolyte solution was sodium chloride.

Table 5 shows the performance of this configuration.

Table 5. Cell performance using GRAFCELL® graphite plate cathode over time.

Current Density (kA/m^2)	Cell Voltage (volts)	Electrolysis at 0.5 kA/m^2 (hr)
0.5	3.02	none
	2.96	4.5
	2.85	20.5
	2.88	

Example 6

In this example, the anode and cathode of Example 4 were modified to evaluate alternative contact configurations. As in Example 4, the anode comprised of titanium sheet and catalyzed with OPTIMA® coating was sandblasted and two connected at two contact points, without using aluminum contact bars. The GRAFCELL® graphite plate cathode was similarly configured to have two contact points. A titanium sheet was also used to provide a comparative basis.

Table 6 lists the measured potentials at various current densities using GRAFCELL® graphite sheet cathode and titanium sheet cathode. The electrolyte was 3 M sodium chloride solution. The GRAFCELL® sheet was pretreated for 40 hours at 0.5 A/m^2. The data shows comparable performance between the graphite sheet cathode and conventional titanium cathode.

Table 6. Cell potential, in volts, using GRAFCELL® graphite plate cathode compared to titanium cathode.

Current Density (kA/m^2)	GRAFCELL® graphite sheet cathode (volts)	Titanium cathode (volts)
0.125	2.67	2.7
0.25	2.76	2.73
0.5	2.88	2.92

Example 7

In this example, cell was assembled using GRAFCELL® graphite sheet as the cathode and also as the anode. The electrodes gap was 1.6 mm and the solution was sodium chloride. At 0.2 kA/m^2, the operating potential was 3.37 volts. This example thus shows that cells comprising carbon-loaded electrodes can be used in accordance with some aspects of the invention.

Example 8

In this example, the cell as in Example 7 was further modified by platinizing the graphite sheet anode. Under the same operating configuration, the operating voltage was measured to be about 2.99 volts thereby lowering the overvoltage potential.

Having now described some illustrative embodiments of the invention, it should be apparent to those skilled in the art that the foregoing is merely illustrative and not limiting, having been presented by way of example only. Numerous modifications and other embodiments are within the scope of one of ordinary skill in the art and are contemplated as falling within the scope of the invention. For example, the anode of the electrolytic cell can comprise a carbon-filled polymeric material. In particular, although many of the examples presented herein involve specific combinations of method acts or system elements, it should be understood that those acts and those elements may be combined in other ways to accomplish the same objectives.

Those skilled in the art should appreciate that the parameters and configurations described herein are exemplary and that actual parameters and/or configurations will depend on the specific application in which the systems and techniques of the invention are used. Those skilled in the art should also recognize or be able to ascertain, using no more than routine experimentation, equivalents to the specific embodiments of the invention. It is therefore to be understood that the embodiments described herein are presented by way of example only and that, within the scope of the appended claims and equivalents thereto; the invention may be practiced otherwise than as specifically described.

Moreover, it should also be appreciated that the invention is directed to each feature, system, subsystem, or technique described herein and any combination of two or more features, systems, subsystems, or techniques described herein and any combination of two or more features, systems, subsystems, and/or methods, if such features, systems, subsystems, and techniques are not mutually inconsistent, is considered to be within the scope of the invention as embodied in the claims. Further, acts, elements, and features discussed only in connection with one embodiment are not intended to be excluded from a similar role in other embodiments.

As used herein, the term "plurality" refers to two or more items or components. The terms "comprising," "including," "carrying," "having," "containing," and "involving," whether in the written description or the claims and the like, are open-ended terms, i.e., to mean "including but not limited to." Thus, the use of such terms is meant to encompass the items listed thereafter, and equivalents thereof, as well as additional items. Only the transitional phrases "consisting of" and "consisting essentially of," are closed or semi-closed transitional phrases, respectively, with respect to the claims. Use of ordinal terms such as "first," "second," "third," and the like in the claims to modify a claim element does not by itself connote any priority, precedence, or order of one claim element over another or the temporal order in which acts of a method are performed, but are used merely as labels to distinguish one claim element having a certain name from another element having a same name (but for use of the ordinal term) to distinguish the claim elements.

CLAIMS

1. An electrolytic apparatus comprising an electrolytic cell having at least one carbon-filled polymeric electrode.

2. The electrolytic apparatus of claim 1, wherein the at least one carbon-filled polymeric electrode comprises electrically conductive carbon disposed in a thermoplastic polymeric binder.

3. The electrolytic apparatus of claim 2, wherein the polymeric binder comprises polyethylene.

4. The electrolytic apparatus of claim 1, further comprising a body encasing at least a portion of the electrolytic cell, at least a portion of the body comprising carbon disposed in a polymeric binder.

5. The electrolytic apparatus of claim 4, wherein at least a portion of the body serves as at least one electrode of the electrolytic cell.

6. The electrolytic apparatus of claim 1, further comprising a source of an electrolyte comprising at least one of a halite and a halide.

7. The electrolytic apparatus of claim 6, wherein the electrolyte comprises a chlorite.

8. The electrolytic apparatus of claim 6, wherein the electrolyte comprises a chloride.

9. The electrolytic apparatus of claim 6, wherein the electrolyte further comprises at least one oxidizing agent selected from the group consisting of chlorates, perchlorates, hypohalites, permanganates, chromates, and peroxides.

10. The electrolytic apparatus of claim 1, further comprising a source of electrical potential connected to the at least one carbon-filled polymeric electrode and providing less than about 3 volts to the electrolytic cell.

11. The electrolytic apparatus of claim 10, further comprising a circuit constructed to regulate the electrical potential to the electrolytic cell to at least about 2 volts.

12. The electrolytic apparatus of claim 1, wherein at least one carbon-filled polymeric electrode serves as a cathode.

13. The electrolytic apparatus of claim 1, wherein the at least one carbon-filled polymeric electrode comprises at least one metallic core.

14. The electrolytic apparatus of claim 1, wherein the at least one carbon-filled polymeric electrode comprises an electrocatalytic coating disposed on at least a portion of a surface thereof.

15. A method comprising providing an electrolytic cell having at least one carbon-loaded polymeric electrode.

16. The method of claim 15, further comprising establishing an electrical current through the at least one carbon-loaded polymeric electrode.

17. The method of claim 16, wherein establishing the electrical current comprises providing current with a potential of less than about 3 volts.

18. The method of claim 17, wherein establishing the electrical current comprises providing current with a potential of at least about 2 volts.

19. The method of claim 16, wherein establishing the electrical current comprises connecting a terminal of an electrical source to a carbon-loaded polymeric cathode of the electrolytic cell.

20. The method of claim 16, wherein establishing the electrical current further comprises connecting a terminal of an electrical source to a carbon-loaded polymeric electrode having an electroactive coating disposed on at least a portion a surface thereof.

21. The method of claim 16, wherein establishing the electrical current comprises connecting an electrical source to a carbon-loaded polymeric electrode having a metallic core.

22. The method of claim 16, wherein establishing the electrical current comprises conducting current from an electrical source to a carbon-loaded polymeric electrode through an electrically conductive reticulated member contacting at least a portion of the carbon-loaded polymeric electrode.

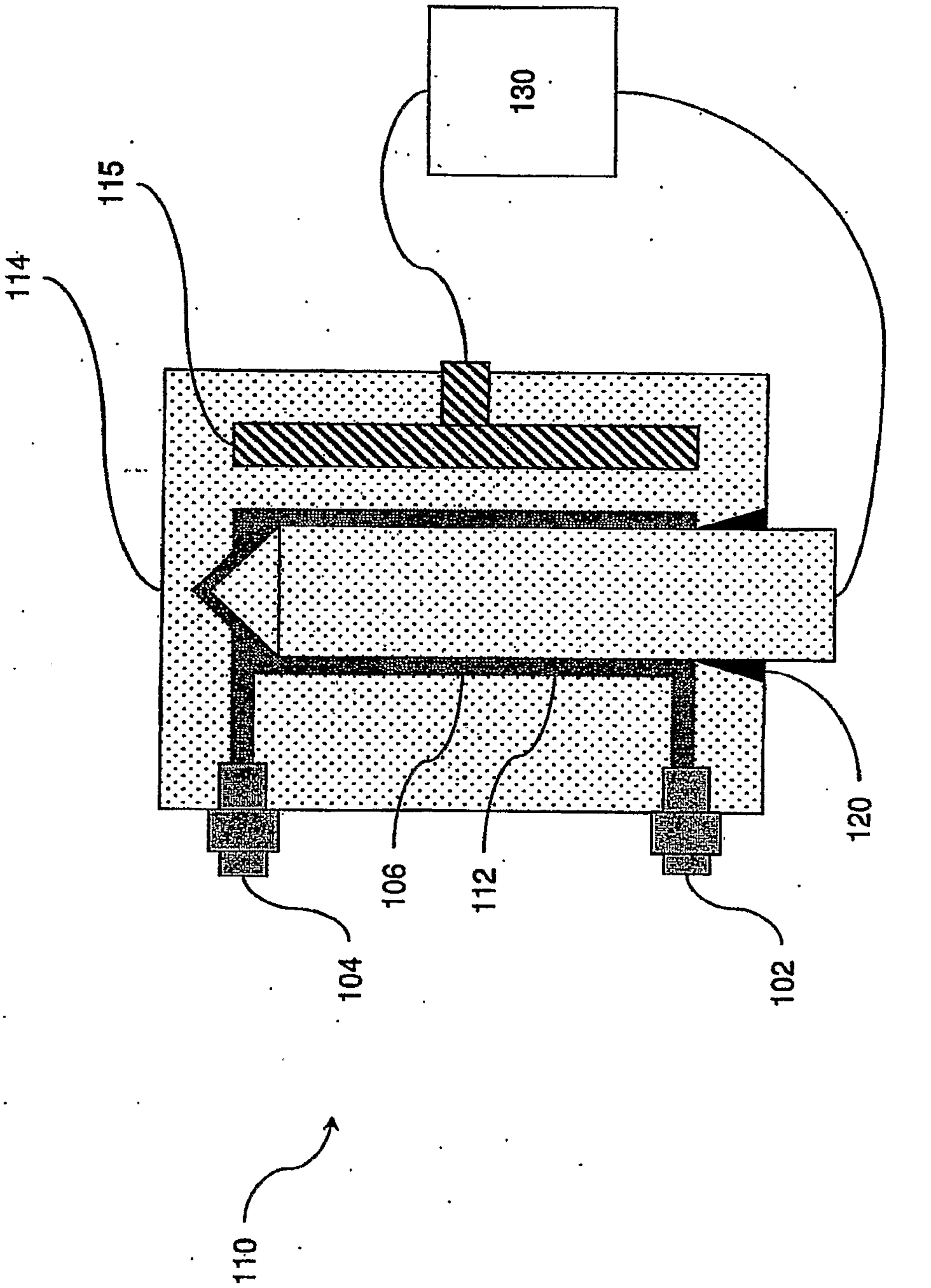

FIG. 1