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Declarations under Rule 4.17:

- as to the identity of the inventor (Rule 4.17(i))
- as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))
- as to non-prejudicial disclosures or exceptions to lack of novelty (Rule 4.17(v))

(54) **Title:** APPARATUS FOR DUAL PHASE CHROMIUM REMOVAL AND ENERGY RECOVERY AND METHODS THEREOF

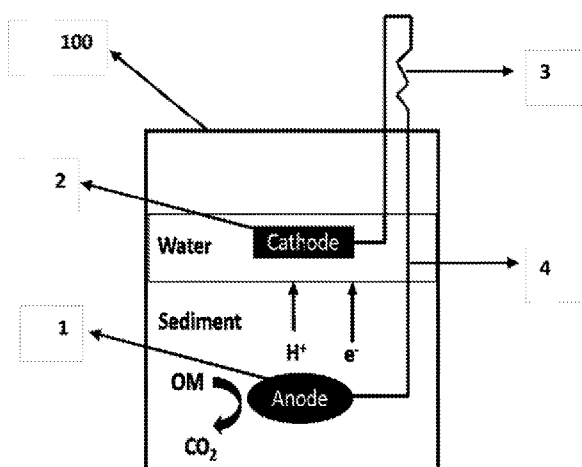


FIG. 1

(57) **Abstract:** An electrolytic cell configuration to simultaneously treat a nitrogenous organic waste in the sediment as well as reduce a carcinogenic metal ion from both the sediment and the overlying supernatant phase. Apparatus and methods are disclosed to reduce hexavalent chromium (Cr^{+6}) to a lower nontoxic state i.e. Cr^{+3} with an organic waste. The source of urea could be from the urea manufacturing plant, or the fecal discharge from any mammalian body (both human and animals), dialysis unit and fertilizer or agricultural runoff.

Published:

- with international search report (Art. 21(3))
- in black and white; the international application as filed contained color or greyscale and is available for download from PATENTSCOPE

FIELD OF THE INVENTION

[0001] The present invention relates to wastewater and sediment remediation particularly to an apparatus and methods of reduction of Cr(VI) to Cr(III) from dual phases.

BACKGROUND OF THE INVENTION

[0002] The sediment strata play a vital role in predicting the quality of lakes, streams and other water bodies. Often, these waterlogged sediments (typically mimicking a wetland ecosystem) contain both organic and inorganic pollutants discharged from industrial effluents or other wastewater or agricultural runoff streams, which deteriorate the quality of the sediments. These sediments act as a principal source of scavenging the pollutants and thereby tend to bio accumulate them for longer periods. As time progresses, the sediments release back the pollutants to the overlying water phase, thereby causing contamination in the dual stratum. The contaminations observed in a representative marshland are primarily heavy metals such as Cr, Cu, Zn, As, etc. It poses a great challenge in remediating such sites as these clayey soils sorb the pollutants and retain for longer time periods.

[0003] Dredging is the most common traditional ex-situ method widely reported in the literatures for removing the contaminated sediments. In recent times, several physico-chemical and biological technologies such as chemical precipitation, pump

and treat, in-situ solidification /stabilization, soil flushing, electro kinetic remediation and bioremediation have emerged in depolluting the sediments. The process limitation that exists with several of these techniques is that either they cause secondary contamination, or expensive or require lengthy time scales for complete remediation. Thus, the feasibility of the existing techniques on a real scale application seems highly questionable. An alternative to this, could contemplate reduction of metal ions from dual phase (i.e. both the underlying sediment phase and overlying water phase) with an economically viable technology to overcome the existing scenario.

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[0004] Another challenge is the effective utilization of waste generated with organic matter as an alternate to conventional energy resources. The generated waste with high organic loading find their way to the sediment and or the water phase increasing in the ratio of N, P, K leading to eutrophication many a times. A typical example to this can be animal waste management, which aims in best utilizing the nutrients for energy recovery. 20
The essential nutrients in theses wastes, if not managed properly can deteriorate the quality of water and soil. The combined process described above may undermine the resilience in the societal face as environmental threats, if not remediated. 25
In order to strike a balance between the energy and the environment, attempts have been made by several 30

pioneers to implement microbial fuel cells (MFC) as a green technology for the reduction and recovery of pollutants from wastewater. Prima facie, the MFC's employ the microorganisms as the biocatalyst to oxidize a natural organic matter at anode and simultaneously reduce a metal ion at cathode with energy output. More recently as an extension of MFC, sediment-MFC (SMFC) has been explored as an alternate for power generation. SMFC's have been investigated over the decades as an alternate source of energy especially in the remote areas where the access to basic power is denied. The greater challenge here, which still remains unanswered is that, the twofold benefit of these fuel cells to bridge the gap between the energy and the environment sectors.

[0005] Existing reported literatures have focused the application of SMFC's for the energy sector and very few studies have reported to remediate the organic contaminated sediments such as toxic refractory organic pesticides (Cao et al. 2015), PAHs (Sherafatmand et al. 2015), phenol (Huang et al. 2011), polychlorinated biphenyls (Xu et al, 2015) with organic matter as electron donor and correspondingly reported power densities as 77.5 mW m^{-2} , 6.02 mW m^{-2} , 29.45 mW m^{-2} , 18.30 W m^{-3} , respectively. Kelvin et al. 2005, demonstrated the feasibility of *Geobacter species* to effectively reduce soluble U (VI) to insoluble U (IV) from groundwater. Wang et al. 2016, reported the removal

of Cr (VI) contaminated soil using dual chambered MFC. Ashoka et al. 2012, reported the comparison of different electrodes in MFC with cow dung as substrate and Nylon as PEM and methylene blue as mediator. Recently, Habibulet.al, 2016 demonstrated the feasibility of plant-MFC for the bio electrochemical removal of Cr(VI) from the contaminated water with electricity generation.

[0006] Few other reported literatures which have primarily focused on deriving power from organic contaminated SMFC's are the following: Song et al. 2011, Hunag et al. 2012, An et al. 2013, Xu et al. 2016 and Zhao et al. 2016.

[0007] Though, several studies have been reported for the soil organic matter degradation, the need for the removal of simultaneous heavy metals from the dual stratum with energy recovery still remains an unexplored area, which is investigated in this art.

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OBJECTS OF THE INVENTION

- 20 **[0009]** The main object of the invention is to disclose an apparatus and methods for reduction of Cr(VI)to Cr(III)from both sediment and water phase through an electrochemical driven process without the use of additional power.
- 25 **[0010]** It is yet another object of the invention is to reduce a carcinogenic metal ion from both the sediment and the overlying supernatant phase.
- [0011]** It is yet another object of the invention to generate power from contaminated wastewater and
30 sediment matrices.

SUMMARY OF THE INVENTION

[0012] To meet the objects of the invention and overcome the drawbacks of the prior art it is disclosed here an electrochemical apparatus for simultaneous power production and remediation of Cr contaminated wetland having a bottom sediment layer and an overlying water surface comprising of:

an anode;

a cathode

the said anode submerged in the wetland soil, the cathode suspended in the water phase of the wetland, the said anode and cathode connected to a constant load; characterised in that, the natural dissolved oxygen separates the anode and cathode, the anaerobic oxidation of an organic material (electron donor) in the soil phase generates the necessary electrons and protons, the electrons travel via the conductive wire and the protons are exchanged via the soil-water interface, the electrons extracted from the sediment treat wastewater contaminated with metals, and the Cr (VI) in the sediment scavenge partial electrons released from the organic matter oxidation and accepts them to reduce to Cr (III).

[0013] It is herein disclosed an electrochemical method for simultaneous power production and remediation of Cr contaminated wetland having a bottom sediment layer and an overlying water surface comprising the steps of:
submerging an anode in the wetland soil;

suspending a cathode in the water phase of the wetland; and connecting the anode and cathode to a constant load; characterised in that, the natural dissolved oxygen separates the anode and cathode, the anaerobic oxidation of an organic material (electron donor) in the soil phase generates the necessary electrons and protons, the electrons travel via the conductive wire and the protons are exchanged via the soil-water interface, the electrons extracted from the sediment treat wastewater contaminated with metals, and the Cr (VI) in the sediment scavenge partial electrons released from the organic matter oxidation and accepts them to reduce to Cr (III).

BRIEF DESCRIPTION OF THE DRAWINGS

[0014] FIG. 1 Schematic representation of single chambered membrane free soil water electrolytic cell.

FIG. 2 Effect of urea concentration on Cr removal from (a) electrolyte phase (b) sediment phase

FIG. 3 Effect of initial metal ion concentration and removal efficiency at optimized urea concentration as organic matter (a) from electrolyte phase (b) from soil phase

FIG. 4 Power density curves with 1000 ppm Cr in dual phase (a) urea as organic matter (b) cow dung as organic matter

DETAILED DESCRIPTION OF THE INVENTION

[0015] To meet the stated objects, it is disclosed here an integrated sustainable electrochemical technology of depolluting the metal laden sediment and the overlying water phase with simultaneous energy recovery. Absolutely no power is used as source to the contaminated matrix, rather the existing energy in these matrixes is being tapped and self-utilized for the reduction of Cr(VI) from both the sediment and the supernatant.

[0016] The schematic of the sediment water electrolytic apparatus is depicted in Figure 1. The apparatus (100) has an anode (1) and a cathode (2). The anode (1) submerged in the wetland soil, the cathode (2) suspended in the water phase of the wetland, the said anode and cathode are in-turn connected to a constant load (3). The natural dissolved oxygen separates the anode and cathode. The anaerobic oxidation of an organic material (electron donor) in the soil phase generates the necessary electrons and protons, the electrons travel via the conductive wire and the protons are exchanged via the soil-water interface. The electrons extracted from the sediment treat wastewater contaminated with metals, and the Cr (VI) in the sediment scavenge partial electrons released from the organic matter oxidation and accepts them to reduce to Cr (III).

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[0017] The anode (1) and cathode (2) may be optionally connected to the current collector(4) and storage means through the resistor(3) to collect and store current.

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[0018] The instant invention also has an added advantage of oxidizing a wasteful organic matter as an electron source, achieving the benefit of cleaning up sediments rich in organic loading. In an exemplary case, urea is chosen an organic contaminant, as an electron donor in the soil. Cow urine, cow dung etc. can also be used in place of urea.

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[0019] It is disclosed here a sediment water electrolytic cell (SWEC) for decontaminating metal laden sediment, where the sediment matrix has an overlying water phase. This SWEC is a single chambered cell without any membrane for separation of sediment and water phase. The anode is submerged in the soil and the cathode is suspended in the overlying water phase. The SWEC essentially relies on the natural dissolved oxygen for separating the anode and cathode compartments rather than the use of membranes. The anaerobic oxidation of the urea/ cow dung added in the sediment (electron donor) generates electrons and protons, additionally exhibits an effective way of managing animal waste as a fuel. The generated electrons partially travel via the conductive wire where the Cr in the water phases accepts them; and the protons are exchanged

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via the soil -water interface. We hypothesize here that electrons could as well be consumed by Cr in sediment phase. The electrons are thus harvested from the sediment to treat wastewater contaminated with metals. The SWEC could be a major interest for power supply in the remote or rural areas where power shortage prevails. Catalyst free electrodes: Ni based electrodes (Ni foam) acts as anode and carbon-based electrodes (carbon felt) as cathode.

[0020] The present invention reduces hexavalent chromium (Cr-VI) with an organic waste as electron donor through a unique sediment-water membrane free electrolytic cell (SWEC). The carcinogenic metal ion is reduced from both the sediment and the overlying supernatant phase. Low cost Ni foam and carbon felt is employed as anode and cathode, respectively. Urea is chosen as a representative nitrogenous organic waste which is added as an electron source in the sediment. The source of urea could be from the urea manufacturing plant, or the fecal discharge from any mammalian body (both human and animals), dialysis unit and fertilizer or agricultural runoff. The primary objective is to depollute the Cr(VI) contaminated sediment and the overlying water phase through an electrochemical driven process.

[0021] Initial experiments were conducted to decouple the chemical and electrochemical phenomenon to achieve simultaneous metal ion

reduction from both phases. Results revealed that, the process is feasible only when the system is an electrochemical driven approach.

5 **[0022]** Further, experiments were as well conducted to discern if the system favors galvanostatic or potentiostatic in reduction of Cr(VI). From our preliminary investigations, we conclude that this proposed SWEC performs its best
10 on a potentiostatic mode. The principal mechanism involved here is the anaerobic electrochemical oxidation of an organic waste (urea herein) as an electron donor for the simultaneous removal of Cr(VI) from the sediment as well as the associated
15 supernatant. Additionally, the system was explored for the feasibility of discharging power for a 400 ppm Cr (VI) concentration in both the phases and was successful in achieving a power density of 2.5 mW cm⁻². The *in-situ* methodology can find its own
20 commercial value fitting in real grounds with the advantage of being self-driven, cost effective and an ecofriendly approach.

25 The invention is further described by means of an example.

Experimental section:

30 **[0023]** The electrodes, Ni foam and carbon felt were procured from Sigma Aldrich and AVCARD respectively. The fresh electrodes were rinsed with MilliQ water, oven dried at 80 °C and used. The Cr(VI) as the catholyte was prepared using K₂Cr₂O₇

(oven dried for 2 h at 110 °C) in 0.5 M H₂SO₄ with pH maintained between 1 and 2. Standard Cr solution of desired concentrations was prepared from a stock solution. Sodium bentonite was employed as the model clay component. Micro scale soil studies were conducted with soil packed in the reactor vessel and homogenized with freshly prepared Cr(VI) solutions of the desired concentrations (viz, 50 to 1000 ppm). To mimic the actual wetland ecosystem and to have the soil participation, the soil was equilibrated with the spiked Cr of desired concentration for 24 hours. Samples of sediment and the aliquot were drawn at equal time periods and the concentration of metal ion was calculated in the soil and liquid phases separately. The sediment samples were digested as per the USEPA 3060A protocol for the alkaline Cr (VI) digestion. Consecutively, both the sample phases were analyzed using calorimetric 1, 5 diphenylcarbazide method for absorption at 540 nm. All the experiments were carried out connecting a 1000 ohm resistance as a constant load with Ni wire as current collector at ambient temperature and pressure in triplicates and the mean values are reported. All the chemicals used were of analytical grade.

Cathodic efficiency for Cr (VI) in supernatant phase (mg/L) = (Initial concentration - Final concentration) / (Initial concentration) X 100 %.

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Cr in sediment phase (mg/g) = (Initial measured concentration (mg/L) * Volume made up (mL)) / (Mass of soil (g))

5 **Electrolytic cell fabrication:**

10 **[0024]** Different experimental framework was performed to decouple (i) chemical and electrochemical phenomenon (ii) at OCV (no load condition) (iii) when short circuited (primarily as
15 a current driven process) and (iv) with a constant load of 1000 ohm. Series of experiments were performed with submerging the anode in the contaminated sediment and suspending the cathode in the overlying water phase. The sediment-water
20 electrochemical cell relies on the naturally dissolved oxygen for separating the anode and cathode compartments rather than membranes. Both the electrodes were connected by means of a load to complete the circuit.

25 **[0025]** The anaerobic oxidation of the organic material (electron donor) in the soil phase generates the necessary electrons and protons. The electrons travel via the conductive wire and the protons are exchanged via the soil -water interface. The electrons are thus extracted from the sediment to treat wastewater contaminated with metals. Additionally, the Cr (VI) in the sediment scavenges partial electrons released from the organic matter
30 oxidation and accepts them to reduce to Cr (III).

Results and discussion:

[0026] Experiments were conducted to optimize the urea concentration for an initial metal ion concentration of 100 ppm. Results indicate that 100 % Cr reduction could be obtained with 1 M urea as organic matter from both electrolyte and soil phase (Figure 2 (a) & (b)). Following, the effect of different metal ion concentration (such as 100, 200 and 400 ppm) with 1 M urea as organic matter was further investigated. As discerned from our results, for an initial concentration of 100 ppm Cr in dual phase, the removal efficiency was 100 % and 98.31 % in electrolyte and soil phase, for 200 ppm the removal efficiency was 99.03 % and 83.49 % and 400 ppm the removal efficiency was 90.88 % and 76.70 % from electrolyte and soil phase in 8 h respectively with a constant load of 1000 ohm (Figure 3(a) & (b)).

[0027] The fundamental process mechanism for the reduction of Cr (VI) from both the phases may be attributed as follows. The quasi reversible oxidation of the transition metal anode Ni^{+2} to Ni^{+3} marks the spontaneous oxidation of urea (electron donor) to render electron flow from the submerged anode compartment. We contemplate that, the Cr(VI) in the sediment partially utilize these electrons and probably could have reduced to Cr(III). Simultaneously, the electron that travel across the sediment water interface are utilized by

the Cr(VI) in the supernatant. This marks the beginning of dual phase reduction of the metal ion.

5 **[0028]** Additionally, the aforementioned system was evaluated for the production of power. With a 1000 ppm Cr (VI) concentration in both the sediment and the supernatant phase, a power of 35 mW was observed with urea as organic matter and 50 mW with cow dung as organic matter with 6 cm² electrode
10 area (Figure 4 (a) & (b)).

[0029] The present invention is self-sustainable in nature and is absolutely eco-friendly.

We Claim:

1. An electrochemical apparatus for simultaneous power
production and remediation of Cr contaminated
5 wetland having a bottom sediment layer and an
overlying water surface comprising of:

an anode;

a cathode

the said anode submerged in the wetland soil, the
10 cathode suspended in the water phase of the wetland,
the said anode and cathode connected to a constant
load; characterised in that, the natural dissolved
oxygen separates the anode and cathode, the anaerobic
oxidation of an organic material (electron donor) in
15 the soil phase generates the necessary electrons and
protons, the electrons travel via the conductive wire
and the protons are exchanged via the soil-water
interface, the electrons extracted from the sediment
treat wastewater contaminated with metals, and the Cr
20 (VI) in the sediment scavenge partial electrons
released from the organic matter oxidation and accepts
them to reduce to Cr (III).

2. An apparatus as claimed in claim 1 wherein the said
25 anode is Nickel foam.

3. An apparatus as claimed in any of the preceding
claims wherein the said catalyst free cathode is
carbon felt.

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4. An apparatus as claimed in any of the preceding claims wherein the said electron donor is urea or animal excreta or waste organic matter or their combinations thereof.
- 5
5. An apparatus as claimed in any of the preceding claims wherein the anode and cathode may be optionally connected to the current collector and storage means through the resistor to collect and store current.
- 10
6. An electrochemical method for simultaneous power production and remediation of Cr contaminated wetland having a bottom sediment layer and an overlying water surface comprising the steps of:
- 15
- submerging an anode in the wetland soil;
 - suspending a cathode in the water phase of the wetland; and
 - connecting the anode and cathode to a constant
- 20
- load; characterised in that, the natural dissolved oxygen separates the anode and cathode, the anaerobic oxidation of an organic material (electron donor) in the soil phase generates the necessary electrons and protons, the electrons travel via the conductive wire
- 25
- and the protons are exchanged via the soil-water interface, the electrons extracted from the sediment treat wastewater contaminated with metals, and the Cr (VI) in the sediment scavenge partial electrons released from the organic matter oxidation and accepts
- 30
- them to reduce to Cr (III).

7. A method as claimed in claim 6 wherein the said anode is Nickel foam.
8. A method as claimed in claims 6 or 7 wherein the
5 said catalyst free cathode is carbon felt.
9. A method as claimed in claims 6, 7 or 8 wherein the said electron donor is urea or animal excreta or waste organic matter or their combinations thereof.
- 10
10. A method claimed in claims 6,7 ,8 or 9 as Cr wherein the Chromium is reduced from both sediment and water phase.

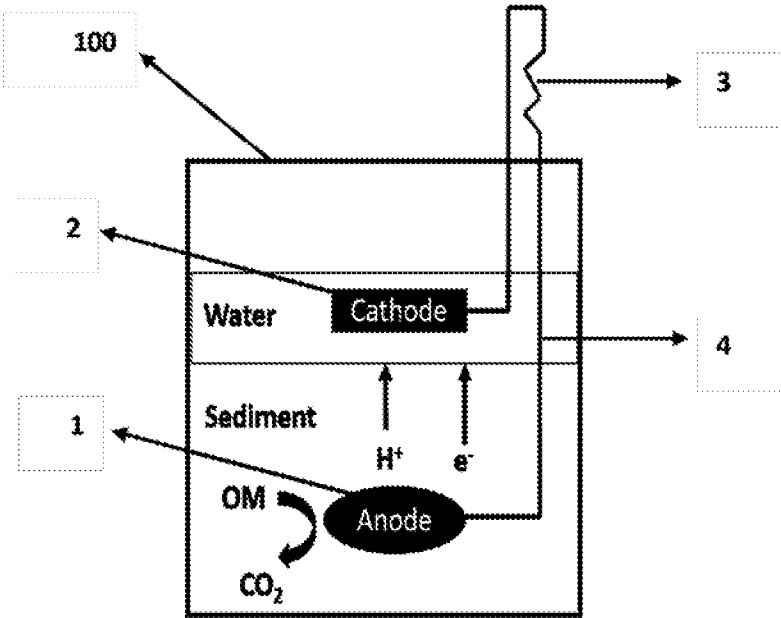


FIG. 1

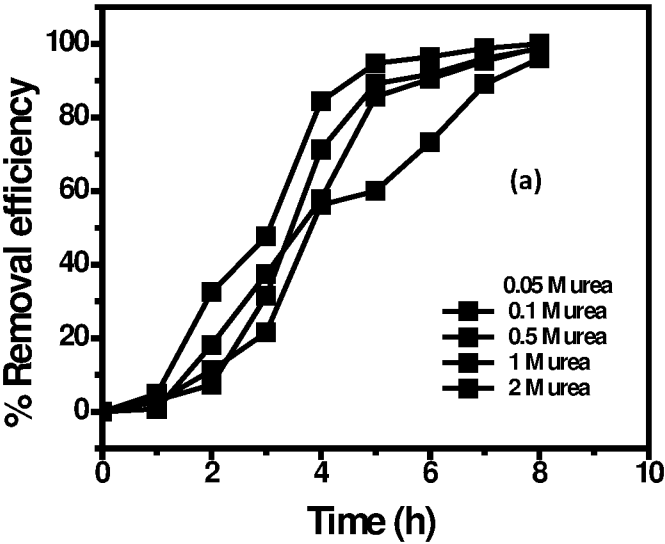


FIG. 2(a)

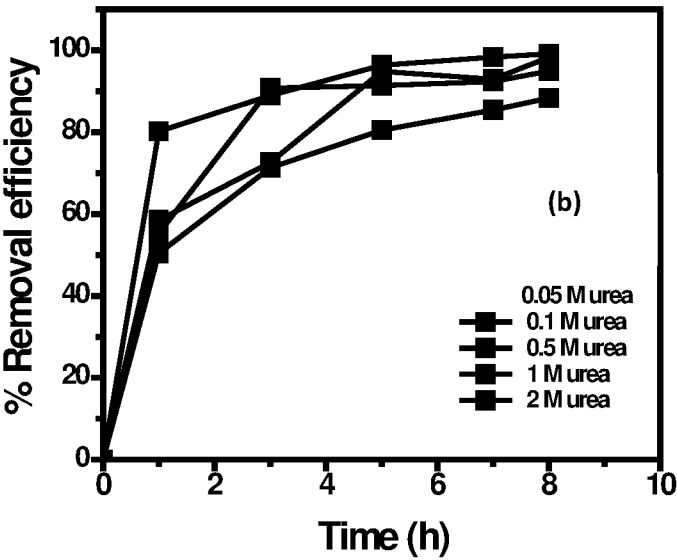


FIG. 2(b)

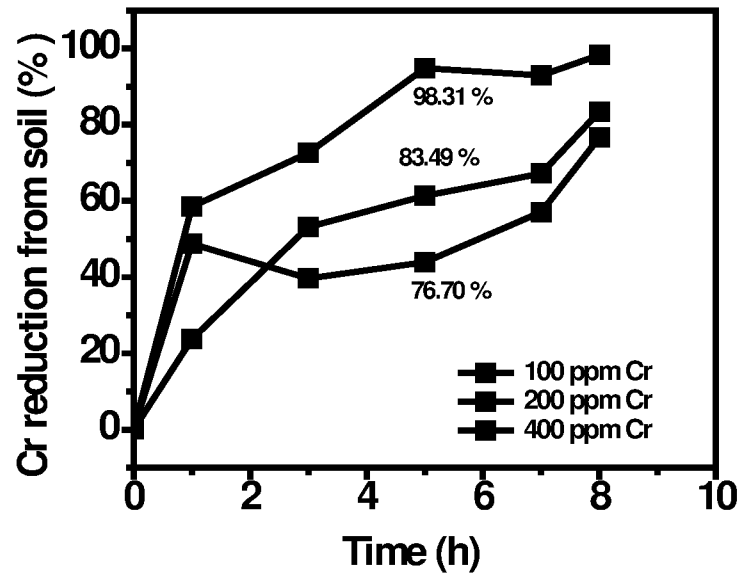


FIG. 3(a)

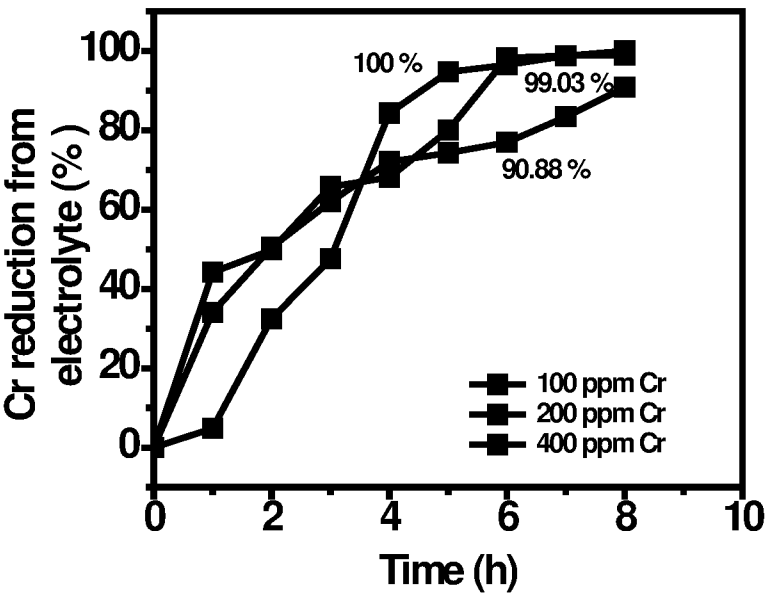


FIG. 3(b)

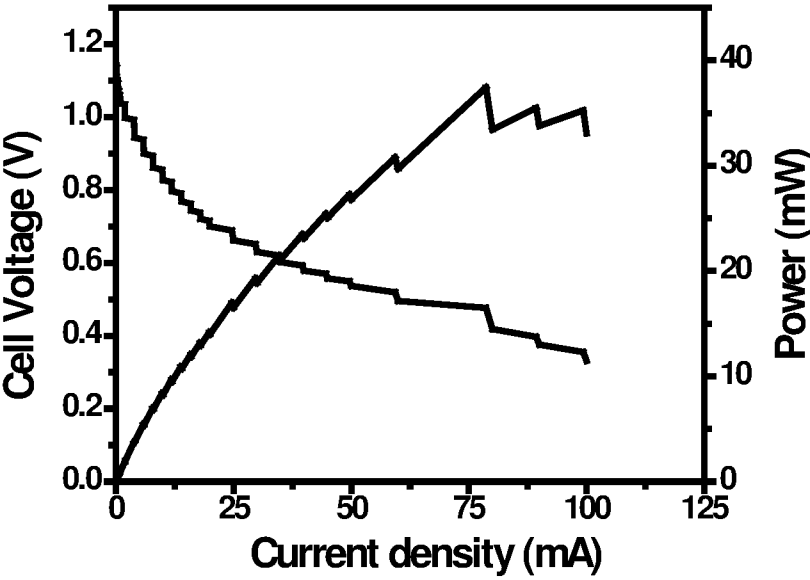


FIG. 4(a)

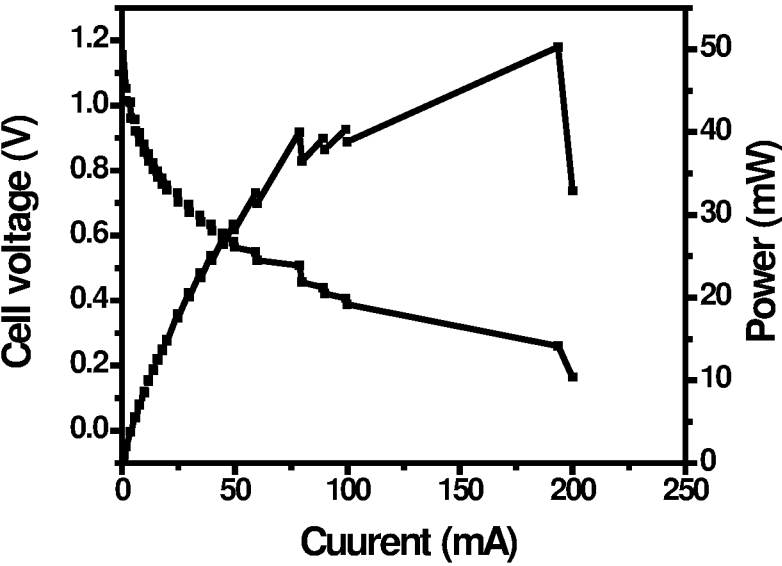


FIG. 4(b)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IN2018/050423

A. CLASSIFICATION OF SUBJECT MATTER
C02F1/46, C02F101/22 Version=2018.01

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C02F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

TotalPatent One, IPO Internal Database

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	Xu W Et Al: 'A urine/Cr(VI) fuel cell – Electrical power from processing heavy metal and human urine', Int. J. Environ. Sci. Technol. (2015) 12:2435-2446, Journal of Electroanalytical Chemistry, vol 764, 1 March 2016, pages 38-44, DOI: 10.1016/j.jelechem.2016.01.013, DOI: 10.1038/srep05860 whole document	1-10
Y	Yu B Et Al: 'Remediation of chromium-slag leakage with electricity cogeneration via a urea-Cr(VI) cell', Scientific Reports 4, Article number: 5860 (2014) whole document	1-10
Y	Xafenias N Et Al: 'Evaluating hexavalent chromium reduction and electricity production in microbial fuel cells with alkaline cathodes', Int. Journal of Environment Science and Technology, August 2015, Volume 12, Issue 8, pp 2435-2446, DOI: 10.1007/s13762-014-0651-7 whole document	1-10



Further documents are listed in the continuation of Box C.



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INTERNATIONAL SEARCH REPORT

International application No.

PCT/IN2018/050423

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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