

Treatment of Spent Caustic from Sodium Dithionate Industry Effluent using Electrochemical Membrane Cell

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ABSTRACT: In this paper treatment of caustic from Sodium dithionate industry effluent is presented. Sodium dithionate industry effluent very harmful to environment in many ways. Effluent can contaminate soil and ground water if landfilling is adopted. It will also decrease dissolved oxygen in water bodies and harmful to aquatic organisms. Effluent will affect the agricultural field, irrigation water source as well as drinking water. Incineration of residues from industry will leads to acid rain because they contain sulfur compounds. So the treatment before disposal of effluent is necessary . From the characterization study concluded that treatment is necessary for the effluent before disposal. Among various methods for industrial waste water treatment electrochemical membrane process was selected because of its attractiveness & economical method. The study was carried out using TSIA anode (System I) and Pt/Ti anodes (System II) . In system I, experiment was carried out in a constant current density of 50 mA/cm² at 4 hour duration. The variation of potential, pH, concentration of sodium thiosulfate, sodium formate and sodium hydroxide with respect to time were analysed. Since chemical and electrochemical reactions takes place in system I, recovered products from system I were caustic, sulfate and sulfur. Characterization studies has also been carried out The percentage reduction of sodium thiosulfate, sodium formate and yield of sodium hydroxide , sulfate were found out..

KEYWORDS: Sodium dithionate industry effluent, TSIA anode, Pt/Ti anodes electrochemical membrane,

I. INTRODUCTION

Over last three decades the developments and advances in the field of electrochemical and environmental engineering helped to investigate efficient methods to control and prevent the emission of pollutants which cause threat to environment. The concept of zero waste during production was introduced 30 years ago. It has been regarded that dilution was best solution for pollution. But in dilution process only the transfer of waste from one environmental medium to other is taking place. Likewise ideal pollution control strategy is to recover, recycle and reuse the substances that may become pollutants. So clean production technologies like zero/low effluent technology is need of the hour. Even though it has been a long time objective for almost all industries, only a few were able to achieve it. Sometimes it may involve high cost. The actual concept of zero effluent is to convert feed stock to product without significant by product.

SODIUM DITHIONATE INDUSTRY EFFLUENT

It is a waste industrial caustic solution that has become exhausted and is no longer useful (or spent). Sodium dithionate industry effluents are made of sodium thiosulfate, sodium formate, sodium hydroxide, water, and contaminants. The contaminants have consumed the majority of the sodium (or potassium) hydroxide and thus the caustic liquor is spent, for example, in one common application H₂S (gas) is scrubbed by the NaOH (aqueous) to form NaHS (aq) and H₂O (l), thus consuming the caustic. Spent caustics are malodorous wastewaters that are difficult to treat in conventional wastewater processes. The effluent from sodium dithionate industry is a sulfurous spent caustic. Typically the effluent is disposed of by high dilution with biotreatment, deep well injection, incineration, wet air oxidation, Humid Peroxide Oxidation or other speciality processes.

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ENVIRONMENTAL IMPACTS OF SODIUM DITHIONATE INDUSTRY EFFLUENT

Water pollution by organic compounds arising from many industrial, agricultural and human activities is one of the main concerns in present world. The vast majority of these compounds are persistent organic pollutants, owing to their resistance to conventional treatments such as coagulation, biological oxidation, adsorption, ion exchange and chemical oxidation. As a result, they have been detected in rivers, lakes, oceans and even drinking waters all over the world. This constitutes a serious environmental health problem mainly due to their toxicity and potential hazardous health effects (carcinogenicity, mutagenicity and bactericidal) on living organisms, including human beings (Francisca et. al,2016). Effluent from dithionate industry can contaminate soil and ground water if landfilling is adopted. It will also decrease dissolved oxygen level in water bodies and harmful to aquatic organisms pollution. Effluent will affect the agricultural field, irrigation water source as well as .drinking water. Incineration of residues from industry will leads to acid rain because they contain sulfur compounds.

To avoid the environmental issues and save the ground water and agriculture field, need of treatment of sodium dithionate industry effluent is necessary. Electrochemical membrane process is more efficient than other measures. More attractive & economical method is recovery of the valuable chemicals from effluent , and subsequently reusing or selling the valuable compounds thus recovered. The recovered sulfur can be used in energy storage device while recovered caustic can find application in sodium dithionate industry itself.

II. MATERIALS AND METHODS

Chemicals and Glasswares

All the chemicals used in this study were of analytical reagent (AR) grade and were supplied by SRI Chemicals Ltd., India. Glass wares used in the present study was manufactured by M/S Borosil Glass Works Ltd.(Bombay, India) and marked under the brand name 'Borosil'. They were washed with diluted sulphuric acid followed by distilled water.

Reagents used

1. 1 N Standard Sodium Hydroxide Solution (NaOH)
2. 20% (W/V) Neutralised Formaldehyde solution (HCHO)
3. N/10 Standard Hydrochloric acid (HCl)
4. 1% Standard Hydrochloric acid (HCl)
5. 1% (V/V) Acetic Acid Solution (CH₃COOH)
6. N/10 Standard Iodine Solution (I₂)
7. N/10 Standard Sodium Thiosulfate Solution (Na₂S₂O₃)
8. 20% (V/V) Hydrochloric acid (HCl)
9. Starch Indicator Solution
10. Phenolphthalein Indicator Solution
11. Crystal violet indicator
12. 0.622 N Perchloric acid
13. 30% Hydrogen Peroxide solution (H₂O₂)
14. 30% Standard Sodium Hydroxide Solution (NaOH)

Sample

The effluent sample was collected from Sodium dithionate processing industry near Karaikudi, Tamilnadu, India.

METHODOLOGY

The flow chart showing methodology is shown in fig.1.

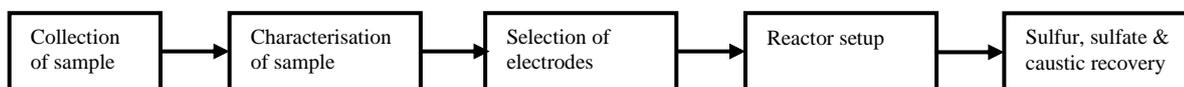


Fig 1 Flow diagram showing Methodology

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Water

All the solutions and samples were prepared by using double distilled water

EXPERIMENTAL STUDIES

This study was carried out to investigate the efficiency of the technique.

Characterization of effluent.

Determination of pH

The pH of the sample waste water was determined using portable pen type pH meter (Water proof pH tester 30n, Eutech instrument IP67) at room temperature. The pH meter was calibrated as per the standard procedure. The meter is switched on and the pH electrode about 2-3 cm was dipped into the pH standards buffer solution. pH calibration mode was activated by pressing CAL/MEAS key. The CAL icon appeared in the LCD. The upper display shows the default (uncalibrated) pH measurement of pH electrode while the lower display indicates the pH standard buffer solution which was automatically recognized by the meter. The solution was swirled gently and the meter reading was allowed to stabilize. The upper display value is calibrated to the pH standard calibration points. The same procedure was repeated with other two standard buffers for better accuracy of the calibration. The electrode was rinsed in tap water before dipping into next standard buffer. Then the electrode was dipped about 2 to 3cm into the test solution. The test solution was stirred gently and the meter reading was allowed to stabilize till the display shows 'ready'. Then the reading on the display was noted as pH of the test solution.

Estimation of Free Alkali

1 ml of mother liquor was pipetted into a 250 ml conical flask and titrated it against N/10 HCl using Phenolphthalein as indicator. The titre value was noted as 'a' ml. The titre value was noted as 'c' ml and free alkali was calculated as per eqn. (3.1).

$$\text{Free Alkali as NaOH (g/L)} = a \times F \times 400 \quad (3.1)$$

Where F – Factor of N/10 HCl and 40 is molecular weight of sodium hydroxide.

Estimation of Sodium Sulphite

5 ml of 20% HCHO was added to the above titrated solution and titrated it against N/10 HCl using Phenolphthalein as indicator. The titre value was noted as 'b' ml. The titre value was noted as 'c' ml and sodium sulphite was calculated as per eqn. (3.2).

$$\text{Sodium Sulphite content as Na}_2\text{SO}_3 \text{ (g/L)} = b \times F \times 126 \quad (3.2)$$

Where F – Factor of N/10 HCl and 126 is molecular weight of sodium sulphite.

Estimation of Sodium Thiosulfate

15 ml of 1 % CH₃COOH was added to the above titrated solution and titrate it against N/10 Iodine as starch indicator. The titre value was noted as 'c' ml and sodium thiosulfate was calculated as per eqn. (3.3).

$$\text{Sodium Thiosulfate as Na}_2\text{S}_2\text{O}_3 \text{ (g/L)} = c \times F \times 158 \quad (3.3)$$

Where F – Factor of N/10 iodine and 158 is molecular weight of sodium thiosulfate

Estimation of Sodium formate

Sodium hydroxide and sodium sulfite were estimated by acid base titration. Sodium thiosulfate was estimated by Iodometric titration method. Sodium formate was estimated by the following procedure, 1 ml Sample was taken into 100 mL conical flask and the sample was weighed using weighing balance (Model). 15mL of glacial acetic acid was added into the sample and heated with warm condition. With hot condition crystalline violet indicator was added and the solution becomes violet colour. The coloured solution was titrated with 0.6227N of per chloric acid. The end point is violet to green colour.

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$$\% \text{ of sodium formate} = \frac{\text{perchloric acid consuming (ml)} \times \text{perchloric acid (N)} \times \text{Eq. wt}}{\text{Density}}$$

% to g/L conversion = % of HCOONa X Density X 10

Determination of Electrical Conductivity, resistivity and TDS

Cyberscan series 600 water proof potable meter Eutech instrument was used to measure conductivity, resistivity and TDS. The instrument calibrated with standard 0.01M KCl solution of conductivity 14.12 μmhos at 25°C. Then the cell was rinsed thoroughly with deionised water and carefully wiped with a tissue paper. The rinsed cell was dipped into the sample solution, the solution was swirled and the reading was noted down when it reached the steady state along with the temperature.

Reactor set up

A double compartment electrochemical membrane reactor was constructed by perspex sheet thickness about 10mm. The compartments were separated by nafion 961 membrane of dimension 7.5 x 4 cm which is in contact between the anolyte and catholyte in the electrochemical membrane reactor. The working volume of the electrochemical cell operated in the experiment was 250 ml. The experiment was carried out using two systems. In system I, TSIA was used as anode and expanded Ti mesh as cathode and in system II, platinised titanium was used as anode and expanded Ti mesh as cathode. Inter electrode distance was about 1cm. The schematic representation of reactor is shown in the fig. 2(a) (b) and original cell setup is shown in fig.2(c)

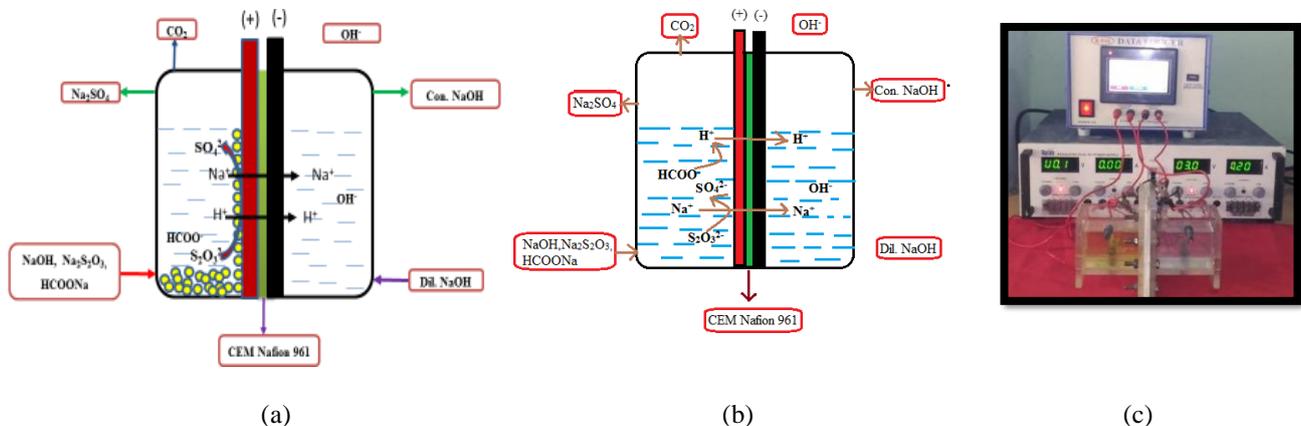


Fig.2. Schematic diagram of (a) system I (b) system II (c) original cell setup

Experimental procedure

Initial characterization of effluent was carried out. After that highly concentrated effluent was diluted with water of 1: 10 ratio. It was used as anolyte. 0.2 N Sodium hydroxide was used as catholyte. The study has been carried out for two different systems. System I which consist of TSIA as anode and system II consist of Pt/Ti anode. Anode and cathode were immersed in the respective electrolyte.

For system I, experiment was performed in a constant current density of 50 mA/cm². The cell voltage and current were monitored using data logger (KPAS Instronics Engineers India Pvt. Ltd. Model no.KM-784 S1 no. 5294). The individual anode and cathode potential were recorded for every 30 minutes interval using multimeter (Keysight U1232A). Corresponding change in current as the time progress was noted down. During the process anolyte and catholyte pH were also measured at 30 minutes time interval. Concentration of sodium thiosulfate, sodium formate NaOH was also noted down. Sodium hydroxide of 0.2 N was filled in catholyte compartment. The reactor was run until the pH of anolyte reaches up to 1. The anolyte and catholyte were collected separately. After filtration Sulphur was recovered from anolyte by adding CS₂ to the precipitate. Sulphate is obtained by precipitation with by BaCl₃ solution.

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Caustic was also recovered from catholyte compartment. After that estimation of Free alkali, Sodium sulfite, Sodium thiosulfate, Sodium formate and Sodium sulfate were carried out.

For system II it was done in potentiostatic mode with constant voltage of 3 V applied between the electrode by aplab. The cell voltage and current were monitored using data logger (KPAS Instronics Engineers India Pvt. Ltd. ,Model no.KM-784 SI no. 5294) Individual potential was maintained as 1.1 V and 1.5 V respectively and are measured using multimeter (Keysight U1232A) .The variation in current, pH of anolyte and catholyte were noted at 1 hour interval. Concentration of sodium thiosulfate, sodium formate NaOH was also noted down. Sodium hydroxide of 0.2 N was filled in catholyte compartment . The reactor was run until the pH of anolyte reaches up to 5. The anolyte and catholyte were collected separately. Sulphate is obtained by precipitation with by BaCl₃ solution. Caustic was also recovered from catholyte compartment. After that estimation of Free alkali, Sodium sulfite, Sodium thiosulfate, Sodium formate and Sodium sulfate were carried out.

III. RESULTS AND DISCUSSION

The thiosulfate oxidation was investigated using two types of experimental processes, system I and system II. During the course of electrolysis in both the systems, the colour of the anolyte gradually disappeared from deep yellow, indicating a decrease in the sodium ion concentration and the occurrence of thiosulfate oxidation in the anolyte chamber.

System I

In system I, TSIA mesh was used as anode and Ti mesh was used as cathode. TSIA anodes are technically and economically viable substitute for the conventional graphite electrode. With coating of electrocatalytic materials of Pt,Ir or its oxides they have better conducting property and dimensional stability, lower voltage and long life. They are characterised by good chemical and mechanical stability. The result in lower consumption and purer products.

Variation of voltage

The graph shown below clearly explains the effect of current density and voltage with respect to time. The constant current density of 50 mA/cm² was applied between the electrodes and individual anode and cathode potentials were noted at 30 minutes time interval. The reaction time was about 4 hours. If we increase the current density, the reaction takes place at faster rate. During the course of electro oxidation thiosulfate was converted as sulphate and slufur while sodium formate as CO₂.The fig. 4.1 shows the variation of voltage.

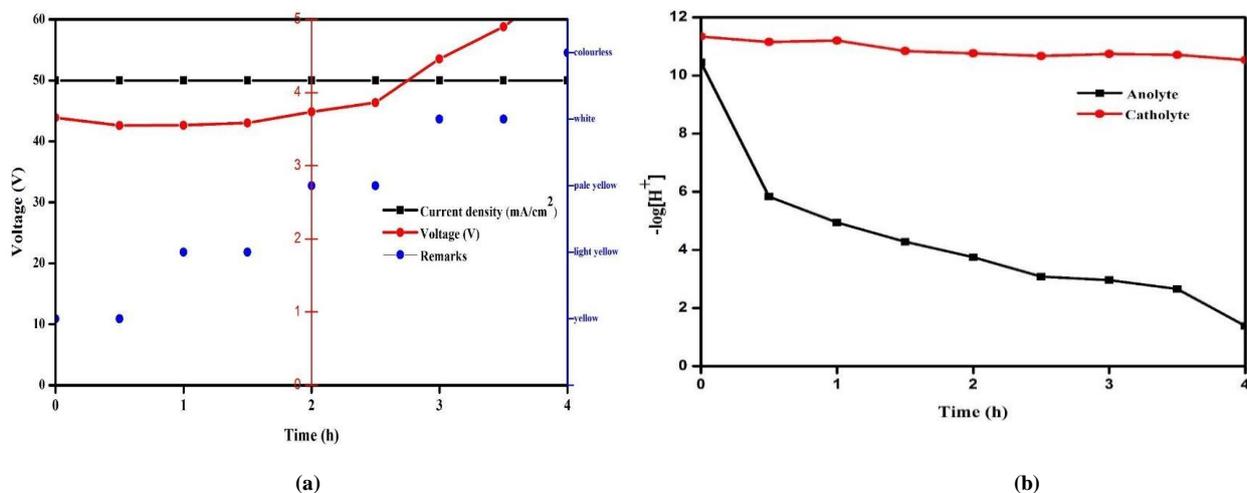


Fig 3 Variation of (a) voltage (b) pH

Variation of pH

The change in pH of anolyte and catholyte solution during the course of reaction is shown in the fig.4.2 below. The anode pH was initially alkaline in nature and it changes to acidic at the end of reaction. The pH was gradually decreased

from 10.45 to 1.38. During this period the acidic nature of H⁺ ions also decompose the thiosulfate to form sulphate and sulphur particles because thiosulfate is stable at alkaline pH. Catholyte pH initially increased and remains constant due to increase in concentration of sodium hydroxide. The colour change of the solution can also be analysed from the graph. The disappearance of yellow colour occurred because of loss of sodium ions from anolyte compartment. Initially the effluent was light yellow in colour then it changes to pale yellow, white and finally colourless. Hosimin et al. (2016) also explains that the change in pH of the anolyte and catholyte at different time intervals. During the sulphide oxidation in the anolyte pH range gradually decreases due to the migration of sodium ions to the catholyte where water is oxidized to produce H⁺ ions. In his study at neutral pH, the yellow colour reappeared due to the formation of polysulfides where the sulfide was oxidized as sulfur but this was not observed in this study. Here the yellow colour of the anolyte completely disappeared below neutral pH

Reduction in concentration sodium thiosulfate and sodium formate

During the course of reaction thiosulfate concentration was found to be decreasing and it is shown in the fig. 4.3. Thiosulfate initially converted to tetrathionate and further oxidised to form sulphate and sulphur. The initial concentration of thiosulfate was 34.24g/L and final concentration was 12.64g/L. The percentage reduction of thiosulfate was 64.77% Concentration of sodium formate was also found to be decreasing as it get oxidised to form CO₂. The initial concentration of formate was 34.685 g/L and final concentration was 16.06 g/L The percentage reduction of formate was 53.69%. CO₂ gas was liberated.

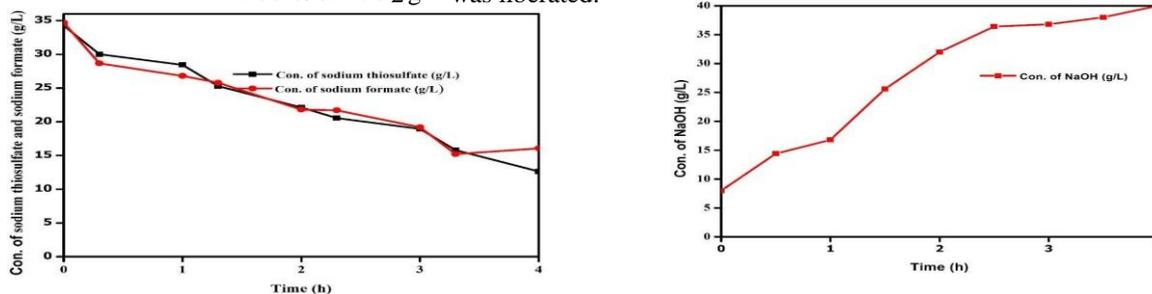


Fig.4 (a) Reduction in concentration of thiosulfate and formate (b) Sodium hydroxide concentration in catholyte

Recovery of caustic

The yield of caustic (NaOH) obtained after the reaction is clearly depicted in the fig. 4.4 as shown below. In the cathodic compartment reduction of water takes place and the OH⁻ ions thus formed will combine with Na⁺ ions which passes through the cation exchange membrane from anodic compartment resulting in NaOH formation. Initially the concentration of NaOH was 0.2 N (8g/L) and its concentration increased to 1 N (40g/L) after the reaction ,so that an yield of 32 gpL was obtained.

Recovered products



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(a) (b)

Fig.5 (a) Colour of effluent before and after reaction (b)Recovered sulfur and sulfate

After carrying out the experiment using system I, the yellow coloured effluent becomes colourless. This colour change may occur due to the removal of sodium ions from anodic compartment. The fig below shows the colour change. In system 1 sulphate and sulfur particles were settled in anodic compartment. In anodic compartment, water splitting takes place and the effluent becomes acidic as a result of reaction. Sulfur was produced because of oxidation of thiosulfate in acid environment. Along with electrochemical reaction, chemical reaction also took place. The sulphate was estimated by precipitation while adding 20% barium chloride in acidic environment. In system 1 sulphate and sulfur particles were settled in anodic compartment. In anodic compartment, water splitting takes place and the effluent becomes acidic as a result of reaction. Sulfur was produced because of oxidation of thiosulfate in acid environment. Along with electrochemical reaction, chemical reaction also took place. The sulphate was estimated by precipitation while adding 20% barium chloride in acidic environment. Sulfur is obtained after recrystallization process by adding carbon disulphide. It was confirmed by using SEM and XRD. SEM analysis was used for determining size of the particle. while XRD was used to determining the crystalline structure of the element.

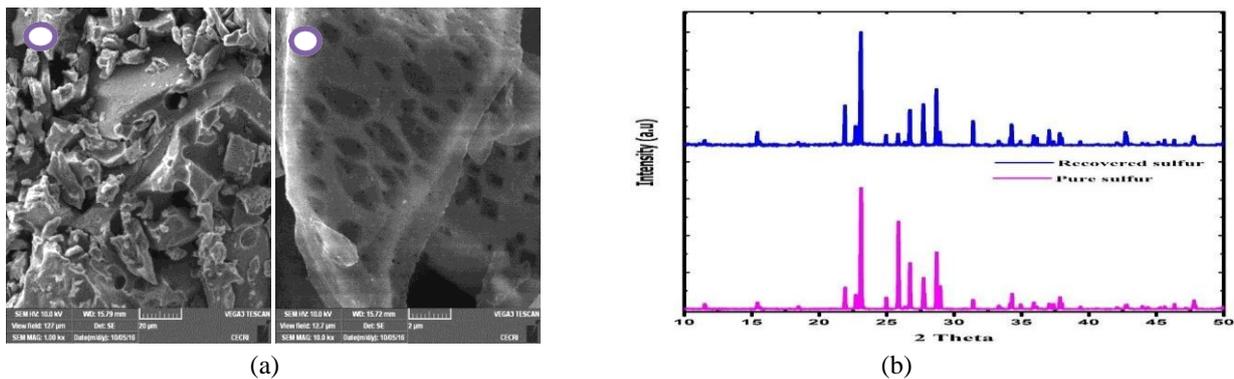


Fig.6(a) SEM image of recovered sulfur from sodium dithionate industry effluent .(b) XRD spectrum of crystalline Pure and recovered sulfur partials (022), (026), (135)

Effluent characterization

The decrease in parameters after treatment are shown in the table 1

Sl. No.	Parameter	Effluent		10 Times dilution Before treatment		Effluent after treatment	
		system I	system II	System I	System II	(TSIA)	(Pt/Ti)
1	pH	12	12	10.47	10.47	1.30	5.09
2	Free alkali (gpL)	16	16	4	4	-	-
3	Sodium sulfite (gpL)	8.82	8.82	1	1	-	-
4	Sodium formate (gpL)	372.69	372.69	34.685	34.685	16.06	10.40
5	Sodium thiosulfate (gpL)	300.99	300.99	34.24	34.24	12.64	1.56
6	Conductivity (m S)	108.2	73.66	73.66	108.2	30.70	69.34
7	Resistivity (Ω)	6.520	9.650	9.650	6.520	23.03	10.17
8	TDS (ppt)	76.69	76.69	51.96	51.96	21.75	49.61

From the table we can understand that the pollutional characteristics decreased after carrying the reaction using both systems. Free alkali and sodium sulfite contents are purely eliminated. Effluent which was alkaline in nature changed to acidic nature. For system I it reaches about 1.3. But in case of system II, the pH was maintained up to 5.09. In both systems conductivity and TDS values were found to be decreased and resistivity is found to be increased. The percentage reduction in thiosulfate and formate was effective for system II than that of system I. So we can conclude that Pt/Ti is more effective anode than TSIA for carrying out this process.

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IV. CONCLUSION

Sodium dithionate industry effluent is rich in thiosulfate. If it is discharged to the waterbodies without treatment, it cause decrease in dissolved oxygen content. So pollution control technologies like zero/low effluent technology is need of the hour. Moreover ideal pollution control strategy is to recover, recycle and reuse the substances that may become pollutants. Since, the initial characterization of the effluent demanded water treatment, an electrochemical membrane process was developed. The experiment was carried out with two systems. System I was a two compartment cell with TSIA anode, Ti cathode and nafion 961 cation exchange membrane. Free alkali and sodium sulfite contents are purely eliminated. Sodium thiosulfate was decreased from 34.24 to 12.64 g/L with percentage reduction 64.77% and sodium formate concentration was decreased from 34.685 to 16.06 g/L with percentage reduction 53.69% .Thiosulfate was oxidised to sulfate and sulfur while formate was oxidised to CO₂. The yellow coloured effluent become colourless as a result of migration of sodium ions to cathode compartment. Effluent which was alkaline in nature changed to acidic nature i.e., pH about 1.3. This decrease in pH was due to formation of H⁺ ions. So here, chemical reaction i.e., splitting of water also takes place along electrochemical oxidation. The conductivity was found to be decreased because of decrease in concentration of ions and resistivity is found to be increased. TDS value also decreased and it was within the permissible limit. From anodic compartment, sulphate was recovered by precipitation with Barium chloride in acid environment and Sulfur was obtained by recrystallization process carried out using carbon disulfide. The SEM, EDAX and XRD analysis confirms that recovered product is sulfur itself. The yield of NaOH obtained from cathodic compartment was 32 gpl while yield of sulphate was 37.9 gpl. The yield of Sulfur was about 2 gpl. The current efficiency was found to be 37.12 %. But at the end of reaction the coating of the electrode get peeled off because of the nature of effluent and lack of stability.

In system II was a two compartment cell with Pt/Ti anode, Ti cathode and nafion 961 cation exchange membrane. The concentration of thiosulfate decrease from 34.24 g/L to 1.56 g/L with percentage reduction about 95.44% .The formate get oxidised and carbon dioxide is produced. The concentration of sodium formate decrease from 34.685 g/L to 10.40 g/L with percentage reduction about 70%. pH decrease up to 5. Here also yellow coloured effluent becomes colourless due to migration of sodium ions to cathode compartment. The yield of NaOH obtained for system II was 36 gpl .Yield of sulphate for system II was 31.37 gpl. No Sulfur was obtained here since direct oxidation of thiosulfate to sulphate takes place. The current efficiency was found to be 70.296 % and energy consumption of the reaction was about 29.029 KW h/g. Cost efficiency was 203.364 for system II. The reaction followed first order with rate constant (-5.334 X 10⁻³ /h). From this study it was concluded that Pt/Ti was the suitable anode material for carrying electrochemical membrane process of dithionate industry effluent which was conducted in potentiostatic mode. The percentage reduction of thiosulfate, formate, yield of caustic and sulfate, current efficiency and cost efficiency are high for Pt/Ti than that of TSIA. Energy consumption was also less. More over there was no damage to the coating of Pt/Ti after the reaction. So Pt/Ti is more suitable material.

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