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# Removal of Chromium from Spent Liquor of Tannery Waste Water by Electro floatation Method

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ABSTRACT: RO treatment plants use semi-permeable membranes and pressure to separate salts from water. These systems typically use less energy than thermal distillation, leading to a reduction in overall desalination costs. The reverse osmosis process enables now the massive production of water with a moderate cost, providing flexible solutions to different necessities within the fields of population supply, industry and agriculture. The great development of reverse osmosis (RO) technology has been a consequence of several factors such as reduction in energy consumption and membrane cost. Nevertheless, the major problem of RO desalination plants is the generation of a concentrate effluent (brine) that must be properly managed. Disposal of such brines presents significant costs and challenges for the desalination industry due to high cost and environmental impact of brine disposal. The reject brine from desalination plants not only contains various types of salts at higher concentration but also other types of wastes like pretreatment chemicals (antiscalents, antifouling,...etc). Also, if the feed water includes harmful chemicals such as heavy metals or others, these chemicals are concentrated in the reject brine. Improper disposal of reject brine from inland plants results in several environmental problems. These facts demand study of the effect of pH for electrolytic hardness removal the effect of current density for electrolytic hardness removal, the efficiency of suitable electrode in electrolytic hardness removals from R.O reject wastewater. In this paper removal efficiency of hardness and other factors are compared by using nickel foam and carbon felt as cathode in tannery waste water sample.

KEYWORDS: reverse osmosis, hardness, carbon felt electrode, nickel foam electrode, desalination.

#### I. INTRODUCTION

Water is a common substance that is essential to all known forms of life. Water covers 70 percent of the planet's surface. On the planet Earth water is mostly found in oceans and large bodies of water. 1.6 percent of the Earth's water is found underground in aquifers. Water purification is the process of removing undesirable chemicals, biological contaminants, suspended solids and gases from contaminated water. The goal of this process is to produce water fit for a specific purpose. Most water is disinfected for human consumptiona) but water purification may also be designed for a variety of other purposes, including meeting the requirements of medical, pharmacological, chemical and industrial applications. In general the methods used include physical processes such as filtration, sedimentation, and reverse osmosis, biological processes such as slow sand filters or biologically active carbon, chemical processes such as flocculation and chlorination and the use of electromagnetic radiation such as ultraviolet light.

Water is the key and most precious natural resource, no wonder it is called the elixir of life. With the increasing water scarcity, skyrocketing competition occurs among various sectors, such as industry, agriculture, and domestic sector. Especially for a developing and agrarian country like India, the demand for water supply is insusceptible. In addition, raising public awareness on water conservation strategies from both the state and central governments along with stringent environmental norms significantly increased the monitoring of water use in industries at all possible levels. Industrial water treatment seeks to manage four main problem areas: scaling, corrosion, microbiological activity and disposal of residual wastewater. Disposal of residual wastewaters from an industrial plant is a difficult and costly problem. Most petroleum refineries, chemical and petrochemical plants have onsite facilities to



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treat their wastewaters so that the pollutant concentrations in the treated wastewater comply with the local and/or national regulations regarding disposal of wastewaters into community treatment plants or into rivers, lakes or oceans.

#### Reverse osmosis

Reverse osmosis is a process that industry uses to clean water, whether for industrial process applications or to convert brackish water, to clean up wastewater or to recover salts from industrial processes. Reverse osmosis will not remove all contaminants from water as dissolved gases such as dissolved oxygen and carbon dioxide not being removed. But reverse osmosis can be very effective at removing other products such as trihalomethanes (THM's), some pesticides, solvents and other volatile organic compounds (VOC's). In the reverse osmosis process cellophane-like membranes separate purified water from contaminated water. RO is when a pressure is applied to the concentrated side of the membrane forcing purified water into the dilute side, the rejected impurities from the concentrated side being washed away in the reject water. RO can also act as an ultra-filter removing particles such as some micro-organisms that may be too large to pass through the pores of the membrane.

The great development of reverse osmosis (RO) technology has been a consequence of several factors such as reduction in energy consumption and membrane cost. Nevertheless, the major problem of RO desalination plants is the generation of a concentrate effluent (brine) that must be properly managed. Disposal of such brines presents significant costs and challenges for the desalination industry due to high cost and environmental impact of brine disposal. The reject brine from desalination plants not only contains various types of salts at higher concentration but also other types of wastes like pretreatment chemicals (antiscalents, antifouling,...etc). Also, if the feed water includes harmful chemicals such as heavy metals or others, these chemicals are concentrated in the reject brine. Improper disposal of reject brine from inland plants results in several environmental problems

RO treatment plants use semi-permeable membranes and pressure to separate salts from water. These systems typically use less energy than thermal distillation, leading to a reduction in overall desalination costs. The reverse osmosis process enables now the massive production of water with a moderate cost, providing flexible solutions to different necessities within the fields of population supply, industry and agriculture. The great development of reverse osmosis (RO) technology has been a consequence of several factors such as reduction in energy consumption and membrane cost. Nevertheless, the major problem of RO desalination plants is the generation of a concentrate effluent (brine) that must be properly managed. Disposal of such brines presents significant costs and challenges for the desalination industry due to high cost and environmental impact of brine disposal. The reject brine from desalination plants not only contains various types of salts at higher concentration but also other types of wastes like pretreatment chemicals (antiscalents, antifouling,...etc). Also, if the feed water includes harmful chemicals such as heavy metals or others, these chemicals are concentrated in the reject brine. Improper disposal of reject brine from inland plants results in several environmental problems.

## **RO Reject Water Characteristics and Recovery Process**

As a kind of semi-permeable membranes, RO membranes intercept ions and these ions are contained in the reject water. Concentration factor is relevant to system recovery rates. The higher the recovery rate of RO, higher the concentration factor. That is why RO reject contains high TDS, high hardness, high alkalinity, high silica content and high organic content. To reuse RO reject water, a second stage RO is always necessary to further remove TDS. However recovery RO has to face scaling matter such as Calcium, Magnesium and Silica, which result in low recovery rate of this second stage RO



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Electrolysis: Splitting water with electricity to produce hydrogen and oxygen:

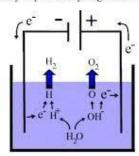


Fig 1.2 Electrolysis

Here chemical decomposition produced by passing an electric current through a liquid or solution containing ions. Water is capable of undergoing oxidation by releasing oxygen at anode and reduction by producing hydrogen gas at cathode.

At anode 
$$H_2O$$
 \_\_\_\_\_  $\frac{1}{2}O_2 + 2H^+ + 2e^-$  (1.1)

At cathode 
$$2H_2O + 2e^ \longrightarrow$$
  $H_2 + 2OH^-$  (1.2)  
 $OH^-$  ions converts  $HCO_3^-$  into  $CO_3^{2-}$  ions  
 $HCO_3^- + OH^ \longrightarrow$   $CO_2^{2-} + H_2O$  (1.3)

$$HCO_3^- + OH^ \longrightarrow CO_3^{2-} + H_2O$$
 (1.3)

These carbonate ions reacts with calcium ions present in hard water to form calcium carbonate which is deposited on the cathode surface.

$$\operatorname{Ca}^{2^{+}} + \operatorname{Co}_{3}^{2^{-}} \qquad \longrightarrow \qquad \operatorname{CaCo}_{3} \tag{1.4}$$

High ph condition in the vicinity of the cathode also promotes the precipitation of magnesium hydroxide Mg(OH)<sub>2</sub> Electrochemical scale removal offers many advantages: environmental compatibility, no need to handle and dose chemicals, accessibility to automation and convenient process control (David Hasson et.al. (2010). The major limitation of this electrochemical process are the high cathode area requirement, existence of a limiting current density beyond which the precipitation rate remains unchanged and the requirement of periodical cleaning of cathode surface (Irina Zaslavschi et.al., 2011). So far in desalination field only plate electrodes are been used, inorder to increase the efficiency the surface area of cathode should be increased. Thus the goal of this investigation is to increase the surface area by using 3D electrodes thereby increasing the cathodic surface area for deposition.

#### II. MATERIALS AND METHODS

#### **GENERAL**

The main focus of the present study was the treatment and reuse of brine by Electrochemical process – electrolysis in terms of improving the desirable water quality and recyclable parameters. To study the effect of pH for electrolytic hardness removal, the effect of current density for electrolytic hardness removal and the efficiency of 3D electrode in electrolytic hardness removal from R.O reject wastewater. Hence, to achieve these objectives, the following methodology was formulated and the same was discussed in this chapter.

# COLLECTION OF SAMPLE

Sample is collected from the R.O unit present inside CSIR-CECRI, karaikudi, Sivaganga district. Sample is collected from this place, as Sivaganga districts groundwater is rich in calcium than other districts in Tamilnadu.



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#### MATERIALS USED

#### CHEMICALS AND GLASSWARE

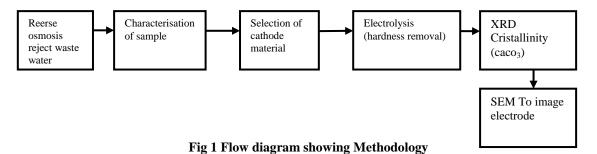
All the chemicals use in this study were of analytical reagent(AR) grade and were supplied by Sigma Aldrich chemicals Ltd, India. Glassware used in the present study was manufactured by M/S Borosil Glass Works Ltd.(Bombay, India), marketed under the brand name 'Borosil'. They were washed with diluted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

#### REAGENTS USED

Standard calcium chloride solution (0.01N), EDTA (0.01N), Ammonium hydroxide – Ammonium chloride buffer pH:10, Erichrome Black T indicator, Standard Magnesium chloride (0.01N), Sodium hydroxide (10%), Silver nitrate, Muroxide indicator were used in this research work

## METHODOLOGY

The methodology is shown in flow chart fig.1.



#### Water

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

## **EXPERRIMENTAL STUDIES**

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

### Characterisation of sample

The RO brine sample was characterized in terms of pH, Total dissolved solids, Electrical conductivity, Resistivity, Chlorides, Calcium, Magnesium, Sodium sulphate and Total alkalinity..

## III. REACTOR SETUP

### SINGLE CHAMBERED REACTOR

Electrochemical removal of calcium from RO reject was studied in a continuous flow system. The cell of dimension 10 x 15 x 6cm with a capacity of 200ml was used. 1200ml of RO reject was taken in a cylindrical vessel. By using peristaltic pump the sample was continuously supplied to the cell with 135 rpm. Here 3D electrode of area  $50 \text{cm}^2$  was used as cathode and TSIA electrode of area  $50 \text{cm}^2$  was used as anode. The cell was operated for a current density of  $10 \text{mA} / \text{cm}^2$ . The cell run time was 2hr 45min. The hardness, pH, conductivity, TDS, anode potential and cathode potential were measured for every 15 min. The hardness was measured by standard EDTA titrimetric method and others were measured by Eu-tech multimeter. This system was repeated for another 10 cycles for better efficiency and accuracy in data. Single chambered reactor is shown in Fig 3.5

## III. RESULTS AND DISCUSSION

The aim of this work and the methodology formulated to carry out this study were presented in the previous chapter. This chapter presents the results that were obtained during this experimental work.



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#### Initial characteristic values of R.O sample

For R.O reject wastewater the initial characterisation was done and thus obtained values are tabulated

	1 able1	
Sl. No.	Parameter	Values
1	pН	6.9
2	Total dissolved solids	868 ppm
3	Electrical conductivity	946.7 μs
4	Resistivity	575.7 Ω
5	Chlorides	180 mg/l
6	Total hardness	350 mg/l
7	Calcium hardness	250 mg/l

#### EFFECT OF CURRENT AND VOLTAGE ON HARDNESS REMOVAL

The reactor was operated with a current density was  $10\text{mA/cm}^2$ . Anode, cathode and total cell potential were 0.918v, 1.198v and 6-8v respectively. Total cell potential was higher due to the resistance offered by electrodes and the electrolyte. The progressive occupancy of the surface by the scale induces an increase of the local current density on the bare areas and, therefore, an increase of both the cathodic polarization and the local pH.( C. Gabrielli et al, 2006) . Electrochemical process tends locally to stabilizes the reduced water through the partially clogged electrode, even if, at the beginning, the oxygen reduction was the only activated reaction. The voltage delivered by the power unit and applied between the terminals slowly increases because of the increase of both the cathodic overvoltage and the water resistivity during the decarbonisation process. . Increase of the current density is found to augment the precipitation rate, which was in the range  $8\text{-}12\text{ mA/cm}^2$  after which the rate reaches an asymptotic leve ( David hasson et al, 2010). Based on this as maximum removal was at  $10\text{mA/cm}^2$  current density, it was fixed throughout this work.

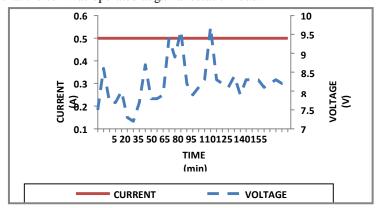


Fig 4.1 Effect of current and voltage on hardness removal for nickel foam

In case of nickel foam the current was maintained constant and the potential varied between 7-9.5 V. Voltage increases gradually due to decrease in ions conducting current. The high isotropy of the carbon felt, higher than that observed for porous materials like Ni foam. The apparent electrical resistivity of carbon felts and, in general, of carbon three-dimensional electrodes, has values 100 times higher than metallic three-dimensional electrodes. The potential inside the three-dimensional electrode depends on the electrical conductivity of both the porous medium and the solution (Coeuret et al, 1984).



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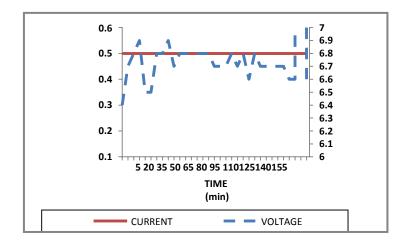


Fig.4Effect of current and voltage on hardness removal for carbon felt

In case of electric cost carbon felt proved to be the wise option. Total cell potential was higher due to the resistance offered by electrodes and the electrolyte

#### EFFECT OF T.D.S AND RESISTIVITY ON HARDNESS REMOVAL

During the operation, Ca and Mg ions contributing hardness were removed in the form of CaCO<sub>3</sub> and Mg(OH)<sub>2</sub>. Due to precipitation of these ions, TDS decreases and resistivity increases.

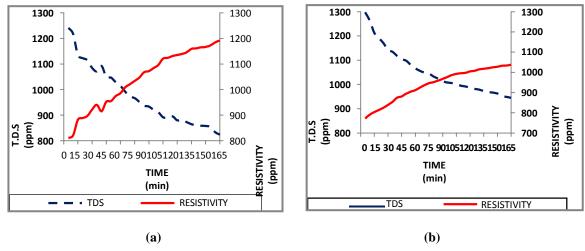


Fig 4.3 Effect of T.D.S and resistivity on hardness removal for(a) nickel foam (b) carbon felt

Fig 4.3 and 4.4 shows variation of TDS and resistivity of water with respect to treatment time. As time progresses, TDS decreases and resistivity increases. Main parameter determining TDS and resistivity in RO reject is Ca &Mg ions. Due to removal of these scaling ions in the form of deposits, TDS decreases and resistivity increases.

## EFFECT OF CHLORINE EVOLUTION

The chlorine generated at the anode is partially released as gas into atmosphere and partially reacts with water to form hydrochloric and hydrochlorous acids.



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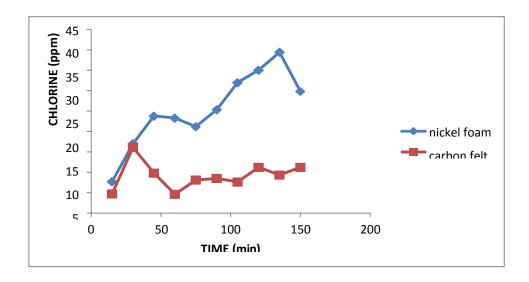


Fig 4.5 shows evolution of chlorine in system with Nickel foam and Carbon felt as cathodes. Evolution of chlorine was more in case of Nickel foam, which was in the range of 8-40 ppm. But in carbon felt it was in the range of 5- 20 ppm comparatively lower.

Chlorine evolution is a main drawback in nickel foam system as it results in black colour precipitate due the reaction of hypochlorous acid with nickel present in nickel foam. This drawback makes nickel foam a failure cathode for hardness removal as post treatment of effluent is required which may also change the water quality. Solution for this problem was discussed in a journal by I.Zaslavschi in 2013 (i.e.) use of oxygen evolution electrodes which will suppress the chlorine generation by promoting oxygen evolution. This solution can avoid 90% of chlorine generation Thus based on this carbon felt was selected as alternative option for this problem, which was conformed with experimental results.

## EFFECT OF CATHODIC AREA AND pH ON HARDNESS REMOVAL

Cathode area is a key factor determining the efficiency and percentage of CaCO<sub>3</sub> and Mg(OH)<sub>2</sub> deposition as it is on cathode. So far, In the research works of desalination field they have used plate electrodes. Literature on single compartment electrolysis system for calcium removal was reviewed in which Gabrilli 2006, David Hasson 2010, Kai Zeppenfeld 2011, Xinyang Li 2014 have used DSA as anode and Stainless steel as cathode. So far in single compartment electrolysis system DSA was used as anode and stainless steel was used as cathode under this case both the electrodes are plate electrodes. Novelty of this work is to increase the cathodic area by using 3D electrodes like nickel foam and carbon felt.

In case of Nickel foam shown in fig 4.6, it is a lustrous silvery metal. Active surface area of nickel foam is 1500 times more than that of nickel solid plate. Hydrogen evolution is higher in case of nickel foam, so OH ions production at cathode is higher which enhances deposition of CaCO<sub>3</sub> and Mg(OH)<sub>2</sub>. As a result hardness removal efficiency is higher.



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(a) Fig 4.6(a) Nickel foam cathode

(b) Carbon felt cathode

Carbon felt shown in fig 4.7 is a Porous light weight material . The carbon felt was reported to have porosity around 0.98, a specific surface area of 22100-22700~m-1,  $2.7\times10-3~V$  m electrical resistivity and a tortuosity between 5 and 6.. (Gonzalez- Garcia et.al., 1998). At alkaline environment the deposition of  $CaCO_3$  and  $Mg(OH)_2$  was enhanced. Initially the pH of the sample was 7.9 then gradually decreases to 6.5 during the process . During the cycle the pH plays a significant role, once it reduces the deposition rate decreases. In 2hr45min 60-70% was reduced. Initially the removal was high for the first 75 min after that the removal rate is gradually decreases and then it reaches a asymptotic limit. The cycle run time was confined to 2hr45min as it reaches asymptotic limit

# COMPARISION OF SINGLE CHAMBERED REACTOR, THREE CELL -STALKED REACTOR AND SIX ELECTRODE REACTOR

## NICKEL FOAM

Nickel foam of area 50 cm², 150 cm² and 180 cm² was used as cathode along with TSIA-Titanium substrate insoluble anode. Thus the hardness was removed and efficiency varied with respect to cathodic area. Hardness removal for the ten cycles is shown in the figure 4.8 and 4.9. For every 15 minutes, sample was collected and hardness was found to be decreased to an average value of 180 mg/l in all 10 cycles. Percentage removal of hardness for single chambered reactor is 57.14%, three single chambered cell stalked reactor is 65.62%. These differences in percentage removal for three reactors are because of gradual increase in cathodic surface area. The main drawback in case of nickel foam is dissolution of nickel from cathode in the form of nickel oxide black colour precipitate. This precipitate is due to chlorine evolution followed by formation of hypochlorous acid which in turn reacts with cathode material resulting in black colour precipitate

## **CARBON FELT**

Carbon felt of area 50 cm<sup>2</sup>, 150 cm<sup>2</sup> and 180 cm<sup>2</sup> was used as cathode along with TSIA-Titanium substrate insoluble anode. Thus the hardness was removed and efficiency varied with respect to cathodic area. Hardness removal for the ten cycles in various systems is shown in the figure 4.10, 4.11 and 4.12. Compared to Nickel foam, carbon felt has many advantages no dissolution of cathode material takes place.

Using this three reactor setup was operated and proved to have better efficiency. Cathode area is a key factor determining the efficiency and percentage of CaCO<sub>3</sub> and Mg(OH)<sub>2</sub> deposition as it is on cathode. So far, In the research works of desalination field they have used plate electrodes. So as a 3D electrode it proves to have greater surface area. For every 15 minutes, sample was collected and hardness was found to be decreased to an average value of 180 mg/l in all 10 cycles. Percentage removal of hardness for single chambered reactor is 54.4%, three single chambered cell stalked reactor is 69.23% and 6 electrode reactor is 70.45%. These differences in percentage removal for three reactors



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are because of gradual increase in cathodic surface area. For 50 cm<sup>2</sup> of carbon felt the removal was comparatively greater to that of nickel foam. From the graph it is observed that the cathode maintains it capacity to remove same amount of hardness without any decline in its efficiency. Proved to have long life even after 10 cycles.

## CHARACTERISATION OF CATHODE AFTER DEPOSITION USING SEM

After 10 cycles under galvanostatic condition the cathode was examined using SEM. SEM result shows that the CaCO3 was deposited in crystalline form. Calcium carbonate scaling in galvanostatic condition was observed and discussed by Gabrielli in 2006, (i.e.) calcium carbonate deposition was made in pure calcite crystals and in second layer dendrites were formed for several millimetre long. In current work no dendrites were found only crystalline layer due to calcite and aragonite was formed. In addition the gaseous overpressure at the cathode surface will also provoke detachment of top layer of crystals. In fact, when the electrolyser is powered at a constant DC voltage, the situation is not very different. A great part of the voltage is consumed by the ohmic drop in the bulk water and inside the porous crust. Therefore the current intensity varies very little, whereas the electrochemical potential becomes progressively more negative in the open area of the cathode. Fig 4.13 shows SEM image for nickel foam cathode after 10 cycles.

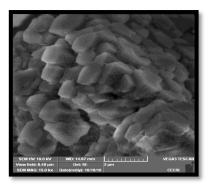


Fig 4.13 SEM for Nickel foam.

## IV. CONCLUSION

Based on the above discussion, the quality parameters of the R.O reject were analysed and its results are presented in the table. It shows that the R.O reject contains high TDS, high hardness and high alkalinity. All these result in scaling problem so all the above mentioned factors should be reduced. Electrolysis was carried out at lab scale using nickel electrode and carbon electrode were cathode in two systems whereas in both the system TSIA electrode was used as anode. During the operation the TDS decreases and the resistivity increases as calcium ions are removed. The cathodic surface area was increased by using 3D electrodes. Alkaline pH increases the deposition of CaCO<sub>3</sub>. XRD AND SEM has to be done for carbon electrode after 10 cycles. The removal efficiency was found to be higher in case of 6 electrode reactor. The efficiency for Nickel foam was 67.23% and for Carbon felt was 70.45%. The precipitation of CaCO<sub>3</sub> was in the form of calcite and aragonite which was confirmed by X-ray diffraction spectroscopy. The porous CaCO<sub>3</sub> was observed in Scanning electron microscope. Chlorine evolution is a main drawback in nickel foam system as it results in black colour precipitate due to the reaction of hypochlorous acid with nickel present in nickel foam. So as a alternative oxygen evolution electrode like carbon felt was used. Carbon felt proved to be best option with efficiency of 70.45% with complying all drinking water standards.

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