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Amr El-Shamy
Gesr AlSuez, amrsalah1980@gmail.com

Mostafa Emara
Department of Chemistry, Faculty of Science (Boy&rsquo;s branch), Al-Azhar University, Cairo, Egypt., scdreh@yahoo.com

Rabab Abou Shahba
Department of Chemistry, Faculty of Science (Girl&rsquo;s branch), Al-Azhar University, Cairo, Egypt., mmbelsabbah@gmail.com

Aya Shoeeb
Department of Chemistry, Faculty of Science (Girl&rsquo;s branch), Al-Azhar University, Cairo, Egypt., ayashoeeb2000@gmail.com

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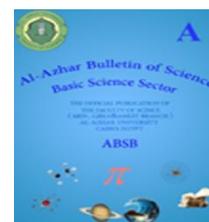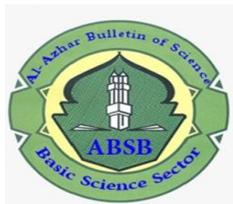
APPLICATION OF ELECTRODIALYSIS TECHNIQUE IN REMOVAL OF SOME HEAVY METAL IONS FROM DISCHARGE WASTEWATER IN PAPER INDUSTRY

Aya Sh. Mohamed [a], Amr S. El-Shamy [b,*], Mostafa M. Emara [c] and Rabab M. Abou Shahba [a]

[a] Chemistry department, Faculty of Science (Girl's branch), Al-Azhar University, Cairo, Egypt.
[b] Chemistry department, Freshwater and Lakes Division, National Institute of Oceanography, Fisheries (NIOF), Cairo, Egypt.
[c] Chemistry department, Faculty of Science (Boy's branch), Al-Azhar University, Cairo, Egypt.

* Corresponding Author: amrsalah1980@gmail.com

ABSTRACT

Electrolysis (ED) is a modern technology for separating pollutants using membranes located in an electric field and has therefore been used in industrial wastewater treatment. The cornerstone of an ED cell is a stack of membranes whose planar plate is composed of cation (CM) and anion (AM) selective membranes. To investigate design requirements such as limiting current density (LCD), current efficiency and membrane resistance by laboratory experimental scale using an ED cell with electrodes, stainless steel 316 [cathode (-) and anode (+)], value of pH equal 6.8 and low voltage supply energy of 24.8 V is installed to remove about 100mg/L for each metal (Iron, Manganese, Nickel, Copper, Zinc, Lead, and Cadmium) ions out of solution of salts. The modified membranes and electrodes to upgrade its durability and conductivity, the recycle flow was 90 and 34 L/hr for concentrate and product, respectively, which are 25 and 9.4 ml/s and with consumed 7 to 11 kWh/m^3 for a continuous operation. For industrial wastewater; the results are obtained a best and encouraging specific with removal efficiency (up to 91.87%) during the 4 hr operating time. The result of removing heavy metal ions was 1.521, 0.96, 0.123, 1.41, 0.94, 0.12 and 0.097 mg/L as initial concentrated and after passing through ED cell became finial concentration 0.23, 0.11, 0.01, 0.22, 0.14, 0.02 and 0.014 with removal efficiency 84.88, 88.54, 91.87, 84.40, 85.11, 83.33 and 85.57% for heavy metal ions under study respectively. The next is being acted on large scale for long operating system.

Keywords: Industrial wastewater; Electrodialysis technology; Heavy metals; Anion membrane; Cation membrane.

1. INTRODUCTION

Industrial wastewater treatment is a very urgent task to be taken care of, which leads to the public's expectation to produce clean water that is free from odor, color, taste, turbidity and other harmful metal ions. Water containing heavy metals must be properly managed, otherwise it may lead to severe damage to the environment, resulting in a long-term impact on the health of living organisms and humans [1, 2]. Electrochemical processes have received much attention in recent years as an effective technology in industrial wastewater treatment. Electrochemical systems have many properties compared to others [3] such as operating under various conditions, including pressure and ambient temperature as effective performance with the ability to adapt to changes in composition and flow rate. It is also capable of treating a wide range of contaminants,

including refractory carboxylic acids [4,5] and perfluorinated carboxylic acids [6,7]. Electrodialysis (ED) is stationary membrane separation processes in which ions are pushed across an ion-selective membrane under the action of an electric field [8]. For some applications, ED has become more interesting in the past decade, such as: reverse osmosis concentration treatment [9], brackish water treatment [10], organic acid production [11], and removal of hardness [12,13], etc. Furthermore, electrodialysis reversal (EDR) occurs when the polarity of the electrodes in the system is reversed, and the greatest benefit of EDR performance is conventional pressure or compression. ED membrane. The processes are long-term self-cleaning of the membranes, ensuring a constant flow and desalination rate [14]. Practical designs for ED stations have been devised by [15,16] and co-workers [17] in many scientific studies. It has been modified by [18]. This is to reduce the number of undefined properties. Reverse dialysis (EDR) has been activated to treat and avoid organic contamination problems [19]. During the past 20 years, EDR has gained wide popularity as it is an outstanding membrane desalination process that saves and reuses water when properly operated.

ED/EDR is applied in reducing the concentration of inorganic substances such as radium [20], perchlorate, bromide, fluoride, iron, manganese and nitrate [21, 22 & 23] in water Drinkable. Additionally, this technology can be used for municipal and industrial wastewater recovery, reverse osmosis rejection, well desalination, surface water treatment, and effluent treatment in cooling towers for reuse [24,25]. This technology has shown the best efficiency of hydraulic recovery and also its cost before other membrane technologies, particularly when compared to reverse osmosis (RO) [26]. Thus bottom residues produced during the ED/EDR process, is another important characteristic of this technology [27]. Furthermore, electrodialysis has a high cost if used in seawater desalination and does not have

any effective effect against biological pollutants. [14]. The major work of this study is to perform and improve the design of ED processes for using in recovery of industrial wastewater.

2.MATERIALS AND METHODS
2.1. Chemicals and Preparations

A stock solution of (Iron, Manganese, Nickel, Copper, Zinc, Lead, and Cadmium) ions was prepared by dissolving calculated quantities of salts in deionized water. Heavy metals in solution were tested, by a GBC atomic absorption reader (Flame and GF 5000 Graphite Furnace his model SavantAA AAS). The pH adjustments were done using 1.0 M NaOH and 1.0 M HCl [28].

2.2. Sampling

Samples of water were collected from the points discharged into surface water stream of the Egyptian Co. for Paper industry in Abu Zaabal city in Al-Qalyubia governorate.

2.3. Methods of water analysis

The methods discussed in the American Public Health Association [29] were used for the determination of the abiotic parameters except where noted. pH and conductivity (EC, $mScm^{-1}$) were in-situ measured using hydrolab model (Multi Set 430i WTW) after previous calibration. Total solids (TS) were measured by evaporating a known volume of well mixed sample. Total dissolved solids (TDS) were determined by filtrating a volume of sample with glass microfiber filter (GF/C) and a known volume of filtrate was evaporated at 180 °C. Total suspended solids (TSS) equal the difference between TS and TDS. Dissolved oxygen was measured using modified Winkler method, BOD was measured using 5-days method. COD was carried out using potassium permanganate method. Sulphides were measured using Iodometric method. Sulphate ($SO4^{-2}$) determined using turbidimetric method. Nitrogen-Ammonia ($N-NH_3$) was determined by phenate method. Nitrogen-Nitrite ($N-NO_2$) was determined using colorimetric method with

formation of reddish purple azo-dye. Nitrogen-Nitrate (N-NO$_3$) was measured as nitrite after cadmium reduction. Orthophosphate (P-PO$_4$) was estimated by using ascorbic acid molybdate method. Silicate (SiO$_4$) were determined using molybdosilicate method. Water alkalinity was determined immediately after sampling collection using phenolphthalein and methyl orange indicators.

2.4. Lab experimental scale

An Electrodialysis (ED) cell was designed to treat the volume of a 6000 mL solution has 100 mg/L of (Iron, Manganese, Nickel, Copper, Zinc, Lead, and Cadmium) ions. Setup experimental is shown schematically and experimentally in Figure (1 a & b). The stack contains the following key parts: electrodes stainless steel 316 [cathode (-) and anode (+)], low density polyethylene spacers and ion-exchange membranes arranged in the way of obtaining four separated streams. Table 1 provides the details of RALEX ion exchange membranes AM (2 anion membranes) and CM (3 cation membranes) (produced in Ralex® Membrane AMHPP – Mega- EU) were used.

2.4.1. Pretreatment: Removing suspended solid on the sample by filtration process using multi-micron filter to give ED good performance.

2.4.2. Process sequence: Starting the current feed of the stack, the cations and anions are motive to the cathode and anode, respectively. They reach a membrane, some reject, and others pass, depending on its size, charge, and hydrophilic effects. At the same time, water compound also pass through the membrane as a solubility shield for ions and flow occurs between the membranes [30,31].

2.5. Batch experiment

The batch experiments were occurring in lab experimental scale has 4 vessels with 3 L of volume, 1 pump KSA NBBD-22 (10 L/ min), and a rectifier Siemens SM 3004-D with automatic range of 0–70 V/0–20 A. The total area of a single cell was equal 0.08 m^2 of activated membrane area has thickness of 3µm in each room. The batch of experiments recycle motive with 3 L of wastewater stream in the concentrate and product sides. The solution of electrolyte (2 L of 0.5-1 M NaCl) was circulating in a closed cycle between the electrodes chambers. Through each experiment, temperature, pH, concentrations, voltage, volume level and conductivity were measured.

Table 1: RALEX ion exchange membranes properties

	Material Ion-exchange groups	Binder	Thickness (mm) Dry	Swelled	Resistance (Ω cm^2)	Counter ion transference number	Exchange capacity (mval/g)
AM	R-(CH$_3$)$_3$N$^+$	Matrix Polyethylene	0.45	0.85	<7	0.95	1.8
CM	R-SO$_3^-$	Cross-linked Polystyrene	0.45	0.70	<8.5	0.95	2.2
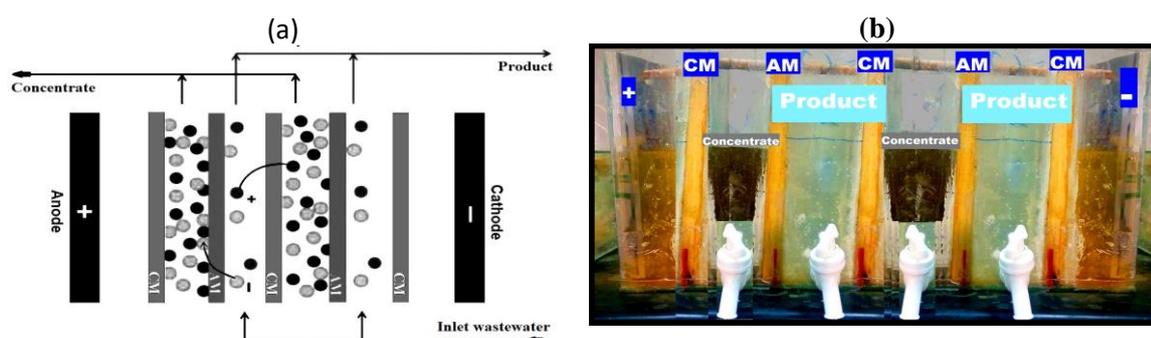
Fig. 1. **(a)** Schematic diagram of experimental electrodialysis (ED) cell, **(b)** Lab system of experimental electrodialysis (ED) cell

2.6. Measurement of limiting current density (LCD)

Figure 2 explains the current-voltage curve, in simple electrochemical cells [32], LCD in ED is usually determined by experimental, because polarization layer of concentration depends on several specification which difficult the theoretical prediction [13,15,31]. An Eq. (1) has accepted empirical procedure, by concentration and solution flow velocity with the LCD (i_{lim}) in the stack in an exponential function.
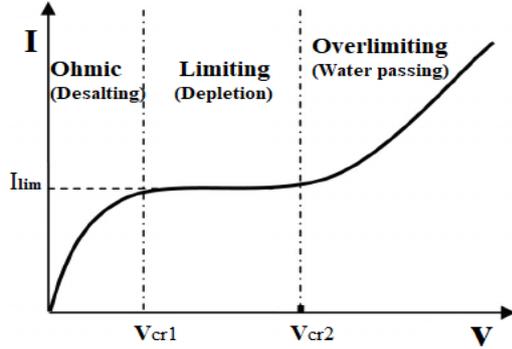
Fig. 2. I–V curve in voltage of wide range

$$i_{lim} = nc^a v^b \tag{1}$$

Where C = Ion concentration (mol/m³),

v = Flow velocity in the stack (m/s) and

n,a and b = The coefficients in the equation

2.7. Measurement of resistances and cell voltage drop

To measurement the resistance changes through the batch on two paramerters: resistance of wet membrane, the product and concentrate of cells. The theoretical voltage of the stack can be calculated by Eqs. (2) And (3) as shown in Eq. (4) for cells used (3 cells): find two practical resistances are contributed in overall voltage development:

(1) Voltage of bulk solution :

$$\Delta U_{sol} = i(\sum_k \frac{h}{k_k}) \tag{2}$$

(2) Voltage of membrane:

$$\Delta U_{mem} = i(\sum_k R_{mw}) \tag{3}$$

Where; k_k = logarithmic average conductivity in each k compartment (S/m),

h = Thickness and

R_{mw} = The resistance of the swollen membranes in Ω m².

$$\Delta U_{stack} - \Delta U_{ov} = N \left(\Delta U_{sol} + \Delta U_{mem}\right) = N[i(\sum_k \frac{h}{k_k})$$

$$i(\sum_k R_{mw})] \tag{4}$$

The total change in cell voltage (ΔU_{cell}) calculated through Eqs. (2) and (3).

And resistance of membranes obtained in Eq. (5) (Lee et al 2002).

$$\Delta U_{cell} = \Delta U_{sol} + \Delta U_{mem} = i(\sum_k \frac{h}{k_k})$$

$$+ i(\sum_k R_{mw}) \tag{5}$$

3. RESULTS AND DISCUSSION

3.1. Effect of Na⁺ concentration on LCD (i_{lim}) measurements:

Figure 3 shows the Na⁺ concentration with LCD values which is of one major component, where C and i_{lim} units are g/L and A/m², respectively and can be calculated by Eqs. (6). When the correlation coefficients (R^2) equal 0.915 indicate good linear eq. these results are nearly to the results of [33] and the LCD values record in Table 2 that were used to select the applied voltage in batch.

$$i_{lim} = 52.025C^{0.28} \tag{6}$$
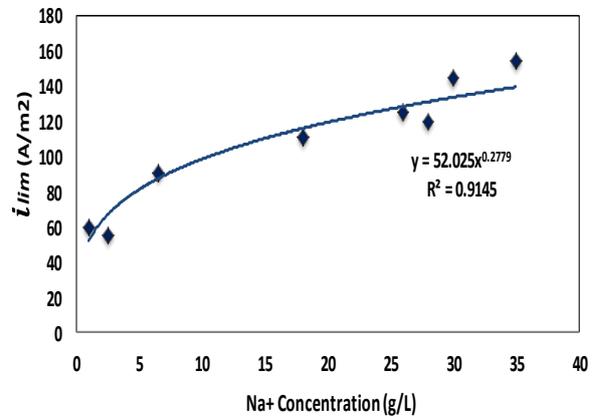
Fig. 3. Correlation of LCD as Na⁺ concentration

3.2. Batch Conductivity studies

Figure 4 shows the conductivity of product side (200 µs/cm) was less than 78.5 % of initial value (930 µs/cm). Table 2 was shown the change of the conductivity on both cells with the time. pH changes were measured and the average values for each cell were: Product: 7.2 ± 0.5, Concentrate: 6.8 ± 0.5 and Electrolyte: 7.6 ± 0.5. The electrolyte concentration and conductivity nearly stable during 8 hour of batch experiments due to closed cycle flow between two electrodes.
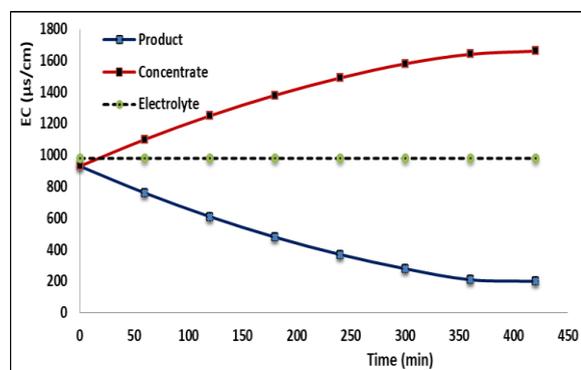
Fig. 4. The change in conductivity through the batch experiments

Table 2: Comparison between LCD and current applied

Time	60	120	180	240	300	360	420	480
Voltage(V)	8.3	7.6	15.8	20.3	24.8	28	34.8	47
Current density (A/m^2)	60	55	91	111	125	120	145	155
LCD (A/m^2)Eq.(2)	52	67	88	117	130	132	135	141

3.3. Effect of resistances on flow rate L/min

Figure 5 explains the change in the flow rate of product (90-34 L/h) due to increase resistance in system (0.14-0.30 $\Omega.m^2$) on wet membrane this is due to the factors like increase in concentration and pollute with organic material with the particles in the sample increased gradually as they were deposited on the membranes, so flow rate decreases with the passage of time. the recycle flow was 90 and 34 L/hr for concentrate and product, respectively, which are 25 and 9.4 ml/s

and with consumed 7 to 11 kWh/m³ for a continuous operation. At each membrane the average resistance values were 20 Ω cm² and the overvoltage potential was 24.8 V at lower concentration when measuring direct voltage as described in the study by [34,35].
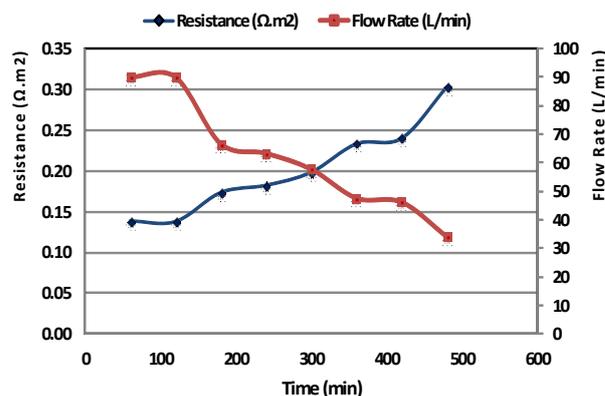
Fig. 5. The effect of flow rate with change in resistance through the batch studies.

3.4. Removal of Iron, Manganese, Copper, Cobalt and Cadmium ions

Table 3 contains analysis of ion concentrations at the beginning and end of the batch test. The ion concentration was determined according to techniques described in experimental section. Metal ions Iron, Manganese, Nickel, Copper, Zinc, Lead, and Cadmium had 100 mg/L initial concentration and after passed through ED cell became finial concentration 2, 1.5. 0.5, 0.8 with removal efficiency 93, 98.5, 99.5, 99.2 and 97% respectively.

Table 3: Heavy metal analysis in batch experiments and removal efficiency after 4 h

Metal	Initial Conc. (mg/L)	Final Conc. (mg/L)	Removal (%)
Fe^{2+}	100	2	98
Mn^{2+}	100	1.5	98.5
Ni^{2+}	100	0.8	99.2
Cu^{2+}	100	0.50	99.5
Zn^{2+}	100	4.50	95.5
Pb^{2+}	100	3.5	96.5
Cd^{2+}	100	3.0	97

3.5. *Application of our ED cell in removal of Iron, Manganese, Nickel, Copper, Zinc, Lead and Cadmium ions from wastewater of paper industry which submerging on Surface water*

The selected sampling station depended mainly on the points discharged into surface water stream of the Egyptian Co. for Paper industry in Abu Zaabal city in Al-Qalyubia governorate. The obtained results of industrial discharge wastewater before and after treatment according to Table (4, 5) were completely removed from all tested industrial wastes. The efficiency of physicochemical characteristics of industrial discharge wastewater before, after Physical-Separation and after ED-Treatment is presented in Table 4. After treatment we noted that high efficiency in removal and decrease in concentration of parameters EC, TDS, TSS, TS, TH, BOD and COD (91.33, 91.35, 99.76, 95.51, 97.56, 99.9 and 99.98.09%) respectively; according to allowable range of Egyptian law.

Table 4: Physicochemical characteristics of Egyptian Co. for Paper Industry; industrial discharge wastewater before and after treatment.

Parameters (mg/L)	Allowable range*	Industrial Wastewater			Removal (%)
		Before	After Physical-Separation	After ED-Treatment	
Color	Color less	brownish	Yellowish	Color less	97.44
EC µS/cm	2000	2430.26	2110	183	91.33
TDS	1200	1506.76	1330	115	91.35
TSS	30	7522	1232	3	99.76
TS	1230	9028.76	2562	115	95.51
pH	6.0-9.0	7.52	7.32	6.7	8.47
T.H	500	586.37	382.44	9.35	97.56
DO	> 5.0	0.04	9.3	9.50	-----
BOD	30	4350.78	3760	0.10	99.90
COD	15	16030.0	14789	135	99.09
SO_4^{2-}	1.0	43.37	40.22	0.53	98.68
Cl^-	1.0	73.40	63	0.85	98.65
NO_2^-	1.0	0. 38	0.32	0.12	62.50
NO_3^-	30	6.132	5.113	0.17	96.68
NH_4^+	-------	0.6062	0.374	0.08	78.61
SiO_2	1.0	11.22	10.113	0.014	99.86
PO_4^{3+}	-------	0.198	0.175	0.02	88.57
Tp	-------	0.268	0.214	0.02	90.65
Na^+	1.0	80.10	72.15	0.07	99.90
K^+	1.0	16.20	13.42	0.112	99.17
Mg^{2+}	150	55.5	52.49	5	90.47
Ca^{2+}	200	143.30	124.51	3.11	97.50

* Law No. 92 /2013 for Allowable range for industrial wastewater

Table 5 explains high efficiency after the treatment in removal and decrease in concentration of parameters Fe, Mn, Ni, Cu, Zn, Pb and Cd according to allowable range of Egyptian law.

Table 5: Some heavy metals concentration of Egyptian Co. for Paper industry; industrial discharge wastewater before and after treatment.

Parameters (mg/L)	Allowable range*	Industrial Wastewater		
		Before	After Physical-Separation	After ED-Treatment
Fe^{2+}	1.0	1.954	1.521	0.23
Mn^{2+}	0.5	0.98	0.96	0.11
Ni^{2+}	0.1	0.149	0.123	0.01
Cu^{2+}	1.0	1.42	1.41	0.22
Zn^{2+}	1.0	0.97	0.94	0.14
Pb^{2+}	0.05	0.13	0.12	0.02
Cd^{2+}	0.01	0.01	0.097	0.014

* Law No. 92 /2013 for Allowable range for industrial wastewater

Table 6 provides the details of the metal ions Iron, Manganese, Nickel, Copper, Zinc, Lead and Cadmium (1.521, 0.96, 0.123, 1.41, 0.94, 0.12 and 0.097 mg/L as initial concentration and after passed through ED cell became finial concentration 0.23, 0.11, 0.01, 0.22, 0.14, 0.02 and 0.014 with removal efficiency 84.88, 88.54, 91.87, 84.40, 85.11, 83.33 and 85.57 % respectively.

Table 6: Removal efficiency of some heavy metal analysis of Egyptian Co. for Paper industry in and removal rate after 4 h

Metal	Initial Conc. (mg/L)	Final Conc. (mg/L)	Removal (%)
Fe^{2+}	1.521	0.23	84.88
Mn^{2+}	0.96	0.11	88.54
Ni^{2+}	0.123	0.01	91.87
Cu^{2+}	1.41	0.22	84.40
Zn^{2+}	0.94	0.14	85.11
Pb^{2+}	0.12	0.02	83.33
Cd^{2+}	0.097	0.014	85.57

3.6. The evaluation of the progress of ED

3.6.1. Economic impact: about 90% Saving city water consumption by reused industrial wastewater after treatment, as well as recovery cellulose fibers lost in wastewater reused in paper making.

3.6.2. Environmental and social impact: we succeed in water conservation, decrease consumption; the treatments are done in an environmentally friendly manner and help reduce unemployment.

4. CONCLUSION

The Na^+ concentration with LCD values which is one major component, where C and i_{lim} units are g/L and A/m^2, respectively. Where the correlation coefficients (R^2) equal 0.915 indicate good linear eq. And through the batch experience, LCD values were used to select the applied voltage. The conductivity of product side (200 µs/cm) was less than 78.5 % of the initial value (930 µs/cm). The change of the conductivity on both cells (product and concentrate). During the experiments, pH changes were measured and the average values for each cell were: produced: 7.2 ± 0.5, concentrate: 6.8 ± 0.5 and electrolyte: 7.6 ± 0.5. The electrolyte concentration and conductivity nearly stable during 8 hours of batch experiments due to closed cycle flow between two electrodes. The change in the flow rate of product (90-34 L/h) due to increase resistance in the system (0.14-0.30 Ω.m^2). The recycle flow was 90 and 34 L/hr for concentrate and product, respectively, which are 25 and 9.4 ml/s and with consumed 7 to 11 kWh/m^3 for a continuous operation. At each membrane the average resistance values were 20 Ω cm^2 and the overvoltage was 24.8 V used to remove 100 mg/L for each metal (Iron, Manganese, Copper, Cobalt and Cadmium) ions out of solution of salts. The removal efficiency: 84.88, 88.54, 91.87, 84.40, 85.11, 83.33 and 85.57 % respectively. Economic impact: about 90% Saving city water consumption by reusing industrial wastewater after treatment, as well as recovery cellulose fibers lost in wastewater reused in paper making.

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تطبيق تقنية الأغشية الكهربية (الديلزة) في إزالة بعض أيونات المعادن الثقيلة من مياه الصرف في صناعة الورق

أيه شعيب محمد[1], عمرو صلاح الشامي[2,*], مصطفي محمود عمارة[3], رباب محمد أبو شهبه[1]

1. قسم الكيمياء, كلية العلوم (فرع البنات), جامعة الأزهر, القاهرة — مصر.
2. قسم الكيمياء, شعبة المياه العذبة والبحيرات, المعهد القومي لعلوم البحار والمصايد, القاهرة - مصر.
3. قسم الكيمياء, كلية العلوم (فرع البنين), جامعة الأزهر, القاهرة — مصر.

الملخص العربي

يمثل استخدام الأغشية الكهربية (الديلزة) تقنية حديثة لعمليات الفصل واكتسبت مؤخرًا اهتمامًا متزايدًا في معالجة مياه الصرف الصناعي. من خلال هذا البحث ، نود أن نقدم طريقة لا تعالج بشكل فعال المعادن الثقيلة فحسب ، بل تنفذ أيضًا عملية الإزالة بمعدل انتقائي ملحوظ. يمثل اللب الرئيسي لخلية (الديلزة) مجموعة غشائية تتكون من أنيون صفيحة مستوية (AM) وأغشية انتقائية كاتيون (CM) . ولتحديد عوامل التصميم الرئيسية مثل مقاومة الغشاء والكفاءة الحالية و كثافة التيار المحدودة (LCD) من خلال تجارب على نطاق معملي باستخدام خلية (الديلزة) مع أقطاب من الفولاذ المقاوم للصدأ 316 [كاثود (-) وأنود (+)] ، جهد منخفض إمداد طاقة 24.8 فولت ، وقيمة 6.8 pH مصممة لإزالة 100 مجم / لتر لكلا من أيونات من محلول الأملاح الاتية (حديد ، منغنيز ، نيكل ، نحاس ، زنك ، رصاص وكادميوم). تم إدخال تعديلات على الأقطاب الكهربائية والأغشية لتحسين الموصلية والمتانة ، وكان تدفق إعادة التدوير 34 و 90 لترًا / ساعة للمنتج والمرفوض على التوالي ، والذي يمثل 9.5 و 25 مل / ثانية مع استهلاك من 7 إلى 11 كيلو واط ساعة / م 3 لعملية مستمرة. وبأخذ عينات من الصرف الصناعي علي المسطح المائي للشركة المصرية لصناعة الورق. تظهر النتيجة التي حصلنا عليها بيانات جيدة وواعدة مع كفاءة إزالة عالية (تصل إلى 93.29٪) خلال 4 ساعات من استمرار العمل نتيجة إزالة أيونات المعادن الثقيلة (1.521 ، 0.96 ، 0.123 ، 1.41 ، 0.94 ، 0.12 و 0.097 ملغم / لتر كتركيز أولي وبعد المرور عبر خلية (الديلزة) أصبح التركيز النهائي 0.23 ، 0.11 ، 0.01 ، 0.22 ، 0.14 ، 0.02 و 0.014 مع كفاءة الإزالة 84.88 و 88.54 و 91.87 و 84.40 و 85.11 و 83.33 و 85.57٪ على التوالي. الأثر الاقتصادي: توفير حوالي 90٪ من استهلاك المياه البلدية عن طريق إعادة استخدام مياه الصرف الصناعي بعد المعالجة ، وكذلك استعادة ألياف السليلوز المفقودة في مياه الصرف الصناعي في صناعة الورق.