Regressional modeling of electrodialytic removal of Cu, Cr and As from CCA treated timber waste: Application to wood chips

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SUMMARY

The volume of waste wood treated with chromated copper arsenate (CCA) is expected to increase over the next few decades. Therefore, alternative disposal options to landfilling are becoming attractive, especially those that promote re-use. The authors modeled the electrodialytic removal of Cu, Cr and As from CCA-treated timber waste. The method uses a low-level direct current as the "cleaning agent", in the presence of an extracting solution. The technique was tested in thirteen experiments on wood chips of out-of-service CCA-treated *Pinus pinaster* Ait. poles. The experiments differ in the extracting solution and initial current intensity, which may be considered as different treatments. Statistical techniques were used to compare the results of the different treatments, in order to select the best conditions for overall removal of the three metals. First, the five best treatments were selected, on the basis of the efficiency of removal of Cu, Cr and As. Then a multi-treatment regression approach was applied to the selected treatments, which points to the choice of oxalic acid with a concentration of 2.5%.

Key words: CCA treated timber waste, Electrodialytic remediation, Wood chips, Copper, Chromium, Arsenic, Regressional modeling, Multi-treatment regression design

1. Introduction

Chromated copper arsenate (CCA) is the most common formulation that has been used to preserve wood from insects, fungi and marine borers (Humphrey, 2002). In spite of its usefulness, due to its strong fixation in wood, extending its useful lifetime to 30 or more years, chromium (Cr) and arsenic (As) are hazardous to human health and present a potential threat to the environment.

Electrodialytic remediation is an emerging method that uses a direct electric current as cleaning agent (Probstein and Hicks, 1993; Ottosen et al., 1997, Ribeiro and Mexia, 1997; Ottosen et al., 2003). In our previous studies, the movement of charged particles in an electrical field was applied to a new object, CCA-treated wood waste, to assist Cu, Cr and As removal. The removed metals might be re-used and the "cleaned" timber further recycled, making the process an environmentally promising solution.

The first remediation experiments were performed with sawdust, applying the same current density, the same experiment duration, and different extracting solutions (water, oxalic, formic and citric acids) (Ribeiro et al., 2000a). The encouraging removal percentages of 93% of Cu, 95% of Cr and more than 99% of As, obtained at the end of 30 days' experiments, for 2.5% (w/w) oxalic acid, led to the study of other percentages of this extracting solution (e.g. 0.5%, 1.0%, 1,75%, 2.5%, 5.0%, 7.5%) (Ribeiro et al. 2000b). However, the neighborhood of 2.5% (w/w) oxalic acid was selected as the optimal percentage (Moreira et al., 2004). To widen the scope of the study to include the particle size of the treated wood waste subjected to electrodialytic remediation, experiments were conducted with wood chips. In this new set of experiments, both dialytic (no external power supply) and electrodialytic conditions (with external power supply at different current densities) were tested (Velizarova et al., 2002) and, as expected, an electric potential difference applied between electrodes increased the ionic fluxes. The nature of the extracting solutions (several organic acids, single or combined, EDTA as a chelating agent, NaCl as a "pure" current-carrying medium, and water as a reference) were found to control the direction of the fluxes through the ion exchange membranes to the two electrode compartments and the individual metal removal efficiencies (Velizarova et al., 2004). For wood chips, the maximum overall removal efficiencies obtained were 81% for Cu, more than 87% for Cr and more than 95% for As. However, more than 94% of Cu, 91% of Cr and 98% of As were obtained individually. This clearly shoes the potential of the method, encouraging further study of electrodialytic remediation.

For the statistical analysis of data collected from experiments, the treatments to be compared were defined by: a) the type and percentage of the extracting solution used to saturate the wood waste and b) the initial current passing through the electrodialytic cell.

After the first selection of treatments based on removal efficiencies, to achieve optimization, a mathematical modeling was applied and then a multi-treatment regression design approach (Mexia, 1987) was used to compare different treatments.

2. Experimental section

2.1 CCA-treated timber waste

A set of experiments was carried out using wood chips prepared from CCA-treated *Pinus pinaster* Ait. Poles, which had been out of service for 20 years. The CCA formulation and the treatment scheme used for the pole are unknown. The initial estimated concentrations of Cu, Cr and As in the chips was 1.68±0.07 g Cu/kg^{-1}, 1.49±0.08 g Cr/kg^{-1}, and 1.95±0.01 g As/kg^{-1}. The "total" Cu, Cr and As content was determined according to BS 5666: Part 3: Method 1 (BS 5666, 1979).

2.2 Laboratory cell

All experiments were run in a batch laboratory electrodialytic (ED) cell, developed at the Technical University of Denmark (Ottosen and Hansen, 1992). This type of cell is described in detail elsewhere (Ribeiro and Mexia, 1997; Ribeiro et al., 2000b; Moreira et al., 2004).

2.3 Electrodialytic experiments

Electrodialytic experiments carried out with this cell may use different extracting solutions, initial currents, and durations. Each different combination of those factors will define a treatment of the wood chips.

Thirteen experiments corresponding to different treatments were carried out for 14 days and one more was carried out for 28 days (Table 1).

Six extracting solutions were used: distilled water; oxalic acid 2.5% (w/w); mixture of oxalic acid 2.5% and formic acid 5% in the ratio 1:1 (w/w); formic acid 5% (w/w); EDTA 2.5% diosodium salt and sodium chloride (NaCl) 1.25% (w/w). The initial current passing through the electrodialytic cell also varied: 0, 20, 40, 60, 80 and 120 mA.

2.4 Analytical methods

During each experiment, samples of the electrolyte solutions (catholyte, nd anolyte) were periodically collected and analyzed for Cu, Cr and As determination. At the end of each experiment, the concentration of these target metals in the ion exchange

Table 1. Treatments (Experimental conditions)

Treatments	Extracting solution	Initial current (mA)	Duration (days)
1	Water	0	14
2	Water	11.6	14
3	2.5% oxalic acid	0	14
4	2.5% oxalic acid	40	14
5	2.5% oxalic acid	60	14
6	2.5% oxalic acid	80	14
7	2.5% oxalic acid	120	14
8	5% formic acid:2.5% oxalic acid	0	14
9	5% formic acid:2.5% oxalic acid	40	14
10	5% formic acid	40	14
11	2.5% EDTA	40	14
12	1.25% NaCl	40	14
13	2.5% oxalic acid	20	28

membranes was determined, and the metals deposited on the electrodes were analyzed in solutions. The final "total" amount of Cu, Cr and As in the wood chips after the experiments was also analyzed according to Method 1 of the British Standard (BS 5666, 1979). Copper and chromium were determined by Atomic Absorption Spectrometry (Perkin-Elmer 300-AAS) and arsenic by Inductively Coupled Plasma (ISA Jobin – Yvon 24 - ICP).

3. Results and discussion

3.1 Preliminary analysis of data

As in experiments with sawdust (Moreira et al., 2004), three different types of data were collected from the experiments with wood chips: 1) Concentration of Cu, Cr and As in both electrolytes (anolyte and catholyte) during the experiments; 2) Percentage of removal efficiency derived from the concentrations of Cu, Cr and As in the wood chips before and after the experiments; 3) Amounts of Cu, Cr and As accumulated at the end of the experiments in both electrolytes, ion exchange membranes and electrodes.

In Fig. 1a, 1b and 1c the three available types of data: a) Percentages of removal efficiency; b) Maximum concentration in the electrolytes and c) Amounts at the end of the experiments in ion exchange membranes and electrodes, for Cu, Cr and As, are jointly presented for each treatment.

As with the experiments using sawdust (Moreira et al., 2004), in these treatments with wood chips initial discussion will be centered on removal efficiency.

Copper. Treatments with oxalic acid 2.5% and with current (treatments 4-7 and 13) showed good removal efficiencies (around 68-84%), as did treatments 9 (mixture of oxalic acid 2.5% and formic acid 5%) and 11 (EDTA 2.5%) (77% and 80% respectively). Treatments 10 (formic acid 5%) and 12 (NaCl 1.25%) demonstrated the highest Cu removal efficiencies: 92% and 94.5% respectively. Treatments without current applied showed low removal efficiencies (less then 55%).

Chromium. As with Cu, treatments with oxalic acid 2.5% showed good removal efficiencies (around 72-87%). Treatment 9 (mixture of oxalic acid 2.5% and formic acid 5%) showed the highest Cr removal efficiency (90.5%). However, treatments 10, 11 and 12, unlike in the case of Cu, showed low removal efficiencies (less then 44%).

Arsenic. The removal efficiencies of arsenic and chromium are in similar, with higher values for As. Treatments with oxalic acid led to removal efficiencies in the range 80-96%. Treatment 9 (mixture of oxalic acid 2.5% and formic acid 5%) presented the highest efficiency removal for As (98%), while treatments 10, 11 and 12 showed a relatively low efficiency removal (less then 67%).

A correlation coefficient of 0.94 between the Cr and As removal efficiencies was found and a linear relation ship $y = 0.77x + 30.3$ between y (As removal efficiency) and x (Cr removal efficiency) was fitted, with $R^2 = 0.89$. This linear relation is similar to the one previously obtained for the experiments with sawdust (Moreira et al., 2004), supporting the claim that, after an initial amount of As has been removed, its removal becomes dependent on Cr removal. The initial amount of As removed, approximately 30% of the total, apparently corresponds to As that is not bonded and is "easily" mobilized into the electrolytes.

Taking into consideration the removal of the three metals as a whole, the treatments that revealed the best removal efficiencies were 4, 5, 7 and 13 (treatments with oxalic acid 2.5% and initial currents of: 40, 60, 120 and 20 mA) and also treatment 9 (1:1 mixture of oxalic acid 2.5% and formic acid 5%, and initial current of 40 mA) (Fig. 1a, 1b, 1c). Therefore, the treatment selection will be between the initial currents 20, 40, 60 and 120 and between the use of oxalic acid 2.5% and the mixture of this with formic acid 5%.

Since the time span for treatment 13 (28 days) was double that of treatments 4, 5, 7 and 9, in the following section, we started by comparing treatments 4, 5, 7 and 9, and then go on to compare the best of that group with treatment 13

3.2 Time evolution of metal concentrations in the electrolytes

To refine the comparison of treatments 4, 5, 7, 9 and 13, a multiple regression approach (Mexia, 1987) was used, first for the first four treatments, and then comparing the treatment selected from these with treatment 13.

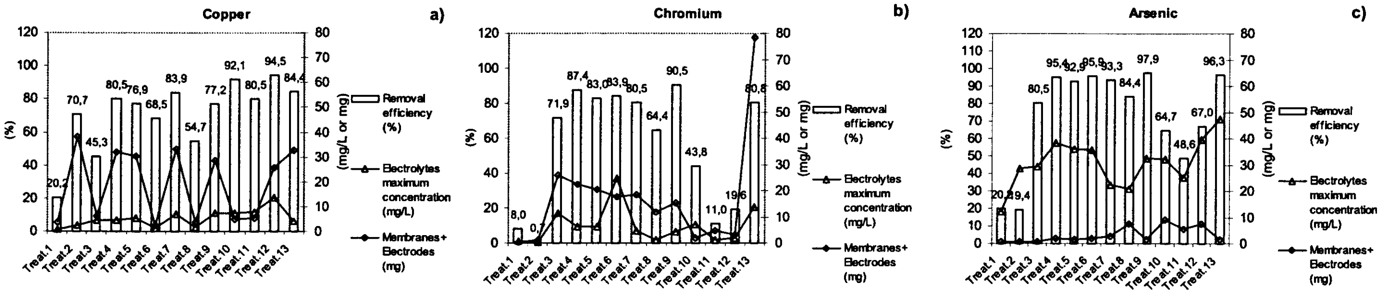

Fig. 1. Removal efficiency (%), maximum concentration in the electrolytes and amounts in membranes and cathode

As with the experiments using sawdust (Moreira et al., 2004), a preliminary observation of the time evolution of metal concentrations in the electrolytes led us to fit polynomial regressions. Thus using standard statistical techniques, namely the least squares method and significance tests for regression coefficients (Montgomery, 1997), a 4th-degree polynomial regression

$$y = \alpha_1 t + \alpha_2 t^2 + \alpha_3 t^3 + \alpha_4 t^4 \tag{1.1}$$

was applied for Cu and Cr, while for As a 3rd-degree polynomial regression

$$y = \alpha_1 t + \alpha_2 t^2 + \alpha_3 t^3 \tag{1.2}$$

was applied. In the expressions (1.1) and (1.2), t represents time measured in days and the $\alpha_j, j = 1,2,3,4$ are the regression coefficients. The adjusted regression coefficients $\tilde{\alpha}_j, j = 1,2,3,4$ for treatments 4, 5, 7, 9 and 13 and the values of R^2 are presented in Table 2.

The next step was to apply the multi-treatment regression designs (Mexia, 1987) described elsewhere (Moreira et al., 2004), and the well known Scheffé multiple comparison method (Scheffé, 1959; Mexia, 1987) to find significant differences between corresponding coefficients. These will be the coefficients for the same powers of time (t, t^2, t^3, t^4).

The coefficient of least degree (α_1) is especially important since it will be relevant in determining the initial speed of mobilization of metals into the electrolytes, and this study aims to find the most efficient treatment for removal of all heavy metals in as short a time as possible.

As stated above, after selecting the best treatment from the first group (treatments 4, 5, 7 and 9), a comparison will be carried out between it and treatment 13.

3.2.1 Copper

Using the Scheffé multiple comparison method, at 5% confidence level, it was shown that:

- For $\tilde{\alpha}_1$, there are significant differences between treatments 4 and 9, 5 and 9, and 7 and 9;
- For $\tilde{\alpha}_2$, there are significant differences between treatments 4 and 9, 5 and 9, and 7 and 9;
- For $\tilde{\alpha}_3$, there are significant differences between treatments 4 and 9 and 7 and 9;
- For $\tilde{\alpha}_4$, there are significant differences between treatments 4 and 9.

The results enable us to separate significantly, at the 5% level, treatment 9 from the other three treatments.

Table 2. Regression coefficients from the adjusted polynomial regressions, for treatments 4, 5, 7, 9 and 13 (T- treatment)

T	Copper					Chromium					Arsenic			
	$\tilde{\alpha}_4$	$\tilde{\alpha}_3$	$\tilde{\alpha}_2$	$\tilde{\alpha}_1$	R^2	$\tilde{\alpha}_4$	$\tilde{\alpha}_3$	$\tilde{\alpha}_2$	$\tilde{\alpha}_1$	R^2	$\tilde{\alpha}_3$	$\tilde{\alpha}_2$	$\tilde{\alpha}_1$	R^2
4	-0.0013	0.0356	-0.3929	2.0786	0.76	-0.0010	0.0408	-0.5964	3.3636	0.97	0.0326	-1.0471	10.9503	0.91
5	-0.0004	0.0128	-0.1670	1.3255	0.96	-0.0017	0.0570	-0.6978	3.5140	0.99	0.0162	-0.6186	8.0090	0.94
7	-0.0010	0.0300	-0.2964	1.2301	0.90	-0.0019	0.0606	-0.6683	2.9682	0.81	0.0445	-1.1114	8.7016	0.90
9	0.0006	-0.0232	0.2398	0.0896	0.97	-0.0004	0.0211	-0.3711	2.0888	0.86	0.0071	-0.4782	7.5179	0.90
13	0.0002	-0.0033	0.0045	0.3410	0.99	-0.0023	0.0880	-1.2319	6.9161	0.97	0.0136	-1.1873	12.2329	0.74

The maximum value of $\tilde{\alpha}_1$ is attained for treatment 4, which is equivalent to saying that treatment 4 (2.5% percentage of oxalic acid and 40 mA) is the treatment with the highest initial speed, followed by treatments 5, 7 and 9 (Table 2).

It can be observed that the value of the $\tilde{\alpha}_1$ coefficient for the 4 treatments follows a regular pattern, where treatments 4 and 9 occupy extreme positions and treatments 5 and 7 occupy intermediate ones. The same happens with $\tilde{\alpha}_2, \tilde{\alpha}_3$ and $\tilde{\alpha}_4$ (Table 2). There are 24 possible ways of ordering 4 coefficients, but only 4 of them are compatible with this pattern. Thus for each coefficient, the probability of such a pattern occurring, due to chance is $(4/24)=(1/6)$. When the 4 coefficients are considered this probability will be $(1/6)^4 < 0.001$, so this agreement is highly significant. Thus it may be concluded that, in case of Cu, treatment 4 is the best one, since it has the highest $\tilde{\alpha}_1$ coefficient, meaning a higher initial speed of mobilization of Cu into the electrolytes.

Using t tests, significant differences were found for treatments 4 and 13. It is important to note that the $\tilde{\alpha}_1$ coefficient was significantly higher for treatment 4 than for treatment 13 (Table 2), so the former should have significantly higher initial speed of removal.

In Fig. 2, the curves of the adjusted polynomials are presented for the Cu concentrations corresponding to treatments 4, 5, 7, 9 and 13 and also the curves for the instantaneous speed of Cu removal into the electrolytes, which is given by the first derivatives of those polynomials.

Treatments 4 and 5 did not present inflection points inside the time range of 14 days (Fig. 2). For those treatments, the process was in deceleration from the beginning until the end of the 14 days of experiments. Treatment 7 presents a first inflection point at time 4.9 (measured in days) and a second inflection point at time 10 (Fig. 2). At this first inflection point, the process changed from decelerating to accelerating, and a minimum speed of removal was attained at this time. At the second inflection point, the process changed from accelerating to decelerating and the maximum speed of removal was attained. For treatment 9, just one inflection point was found inside the time range, at time 4.4, where the maximum speed of removal was attained. For treatment 13, again just one inflection point was found, at time 10, corresponding to the minimum speed of removal (Fig. 2).

3.2.2 Chromium

Applying the Scheffé multiple comparison method to the coefficients at the 5% level, it was found that, for $\tilde{\alpha}_1$ there are significant differences between treatment 4 and 9 and treatment 5 and 9, while for $\tilde{\alpha}_2$, $\tilde{\alpha}_3$ and $\tilde{\alpha}_4$ there are no significant differences between treatments. Since $\tilde{\alpha}_1$ has much more weight in the regression than the other coefficients, it can be concluded from these results that there are significant differences between treatment 9 and treatments 4 and 5, but treatment 7 is not significantly different from 9.

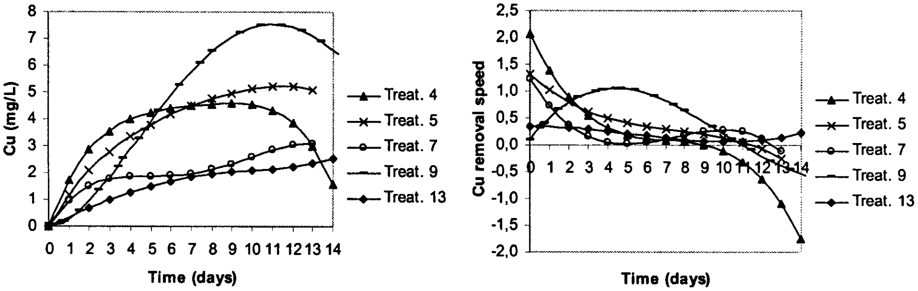

Fig. 2. Adjusted polynomials for Cu concentration and Cu removal speeds, for treatments 4, 5, 7, 9 and 13

It can be observed that the maximum value for $\tilde{\alpha}_1$ is attained for treatment 5, followed closely by treatment 4, after which come treatments 7 and 9 (Table 2).

The comparison between treatments 4, 5 and 13 (Table 2) presents a considerably higher $\tilde{\alpha}_1$ coefficient for treatment 13. At the 5% level, the t test showed that all corresponding coefficients for treatment 4 and 13, and for treatments 5 and 13, were significantly different.

Fig. 3 presents the curves of the adjusted polynomials for Cr concentrations and for instantaneous speed of Cr removal, for treatments 4, 5, 7, 9 and 13.

Treatments 4, 5, 7, 9 and 13 present a first inflection point at times 7.7, 6.8, 5.7, 8 and 7.8 respectively (Fig. 3). At these first inflection points, the process changed from decelerating to accelerating, attaining a minimum speed of removal. For the treatments with oxalic acid 2.5% (4, 5, 7 and 13), a correlation coefficient of -0.98 between the initial current and the first inflection point was found, and the linear relation ship $y = -0.02x + 8.33$, with an R^2=0.96, was fitted, between x (initial current (mA)) and y (time of the first inflection point). A second inflection point was found for treatments 4, 5, 7 and 13 at times 13.3, 10.3, 10.2 and 11.5 respectively. At these second inflection points, the process changed from accelerating to decelerating, attaining a maximum speed of removal (Fig. 3).

3.2.3 Arsenic

Using the Scheffé method at 5% level, no significant differences for the $\tilde{\alpha}_1$, $\tilde{\alpha}_2$ and $\tilde{\alpha}_3$ coefficients were found, between treatments. However, the maximum value for $\tilde{\alpha}_1$ is attained for treatment 4, followed by treatments 7, 5 and 9, following the pattern described for Cu. Thus treatment 4 can also be selected with some confidence, from the group of treatments of 14 days' duration, as being the treatment with the best initial speed of As removal.

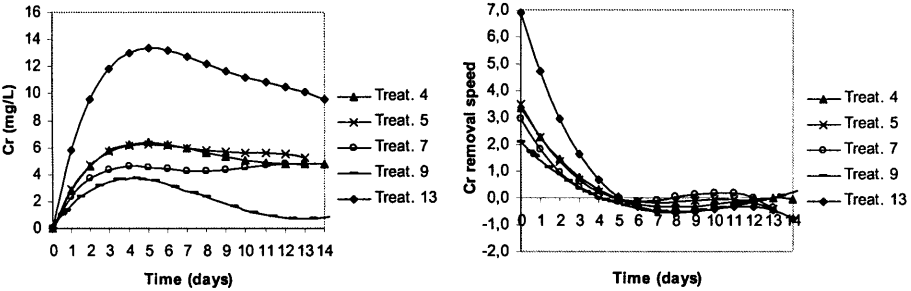

Fig. 3. Adjusted polynomials for Cr concentration and Cr removal speeds for treatments 4, 5, 7, 9 and 13

The comparison between treatments 4 and 13 (Table 2) shows a higher $\tilde{\alpha}_1$ coefficient for treatment 13, however no significant (at the 5% level) differences between corresponding coefficients were found when comparing treatments 4 and 13. Fig. 4 presents the curves of the adjusted polynomials for As concentrations in the electrolytes and the instantaneous speed of As removal, for treatments 4, 5, 7, 9 and 13.

Treatments 4, 5, 7 and 13 present one inflection point at times 10.7, 12.7, 8.3 and 8.7 respectively (Fig. 4). At these inflection points, the process changed from decelerating to accelerating, attaining a minimum speed of removal. Treatment 9 did not present any inflection point inside the time range, since the process was in deceleration until the end of the experimental time range (Fig. 4).

3.3 Joint comparison

The results obtained for global removal efficiencies of Cu, Cr and As showed that oxalic acid, and possibly a mixture between oxalic acid and formic acid, are the best extracting solutions. As for the initial current, treatments with 40 mA, 60 mA and 120 mA and possibly 20 mA were those with the best global removal efficiencies.

For the selected treatments (4, 5, 7, 9 and 13), polynomial regressions were found for the time evolution of electrolyte concentrations for Cu, Cr and As, and multiple comparison methods were applied. The results obtained for the first group of the selected treatments (4, 5, 7 and 9) allowed us to conclude, mainly on account of the initial speed of removal, that:

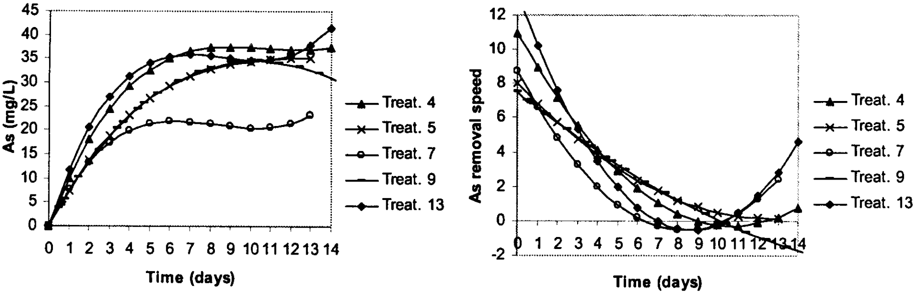

Fig. 4. Adjusted polynomials for As concentration and As removal speeds, for treatments 4, 5, 7, 9 and 13

- For Cu, oxalic acid was the best extracting solution and 40 mA the best initial current;
- For Cr, oxalic acid was the best extracting solution and 60 mA and 40 mA were the best initial currents;
- For As, oxalic acid was the best extracting solution and 40 mA the best initial current.

Thus, taking in consideration the global results for the three metals, one can conclude that the best extracting solution, as in the experiments with sawdust (Moreira et al., 2004), is in fact oxalic acid 2.5% (w/w), and the best initial current is 40 mA.

The results of the comparison between the initial currents of 20 mA and 40 mA are not conclusive, since for Cr and As 20 mA is the best current but for Cu the best is 40 mA. This suggests that the range from 20 mA to 40 mA contains the best options.

4. Conclusions

The main conclusions of this study are that for the electrodialytic remediation of CCA- treated wood waste in the form of chips, oxalic acid 2.5% (w/w) is the best extracting solution and the best initial currents to be applied lie in the range from 20 to 40 mA.

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