Kinetics study on separation of cadmium from tellurium in acidic solution media using ion-exchange resins

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Abstract

The feasibility of using ion-exchange resins to separate cadmium from tellurium in acidic solutions of the two metals was investigated. We studied the competitive adsorption of cadmium and tellurium in such resins under varying acid strengths and contact time. We found that low sulfuric acid strength (i.e., 0.5 M) was most effective in removing cadmium from solutions. Different ion-exchange resins were tested for their affinity for cadmium and tellurium ions. In the selected systems, the ion-exchange rate of cadmium was rapid in the first 20 min, and reached equilibrium within 2 h. The Lagergren first-order model described the kinetic data with high coefficient of determination and correlation values. At room temperatures the ion-exchange for cadmium onto the resin followed the Freundlich isotherm model. The maximum removal of cadmium obtained from batch studies using resin A was 91%. Column studies with the same resin showed the removal of cadmium of 99.99% or higher.

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Keywords: Cadmium; Tellurium; Waste management; Ion-exchange; Recycling; Photovoltaics

1. Introduction

Cadmium (Cd) and tellurium (Te) are the major materials used in manufacturing cadmium telluride (CdTe) modules for photovoltaic (PV) cells. However, the content of these two metals in the PV module’s glass is extremely low, around 0.06% for each, according to our intensive leaching tests. Cadmium is a potentially hazardous metal and tellurium is a rare one; thus, both need to be treated and recycled. This demands prudent scrap-material management practices throughout the PV modules’ life cycle to avoid environmental contamination and health risks, and to comply with stringent regulations on cadmium discharges to the environment. Previous studies were limited to leaching with acidic solutions and extraction of cadmium and tellurium into the liquid phase [1–7]. None of the previous studies succeeded in completely separating Cd from Te. The concentrations of tellurium and cadmium in the leachate were ~1000–1100 ppm of each element. Then, multiple steps (including leaching, neutralization, precipitation, filtration/separation, and re-dissolving) were used to separate and recover both from solutions. Yet, the efficiency of separating cadmium from tellurium, and subsequent recovery were not satisfactorily high. The best separation was obtained from Bohland et al. who recovered about 80% of Te at 99.7% purity in lab-scale experiments [2]. Apparently, these methods were not sufficiently effective in recovering the metals from low-concentration waste solutions. We investigated alternative processes that have the potential to completely separate cadmium from tellurium in acidic solutions. Ion-exchange technology offers a viable alternative for treating such waste streams. By using an ion-exchange resin, either all metal-ions can be removed from a solution, or one specific metal ion can be selectively removed, thus, achieving their separation. By carefully choosing the type of ion-exchange resin, the heavy metal ions can be completely separated and removed from the waste solutions to meet stringent environmental regulations. Several researchers studied adsorption as a means to remove cadmium from aqueous solutions [8–24]. The tested adsorbents

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The quantities of cadmium and tellurium adsorbed onto the resins at equilibrium (q \( e \), mg/g) were calculated using the mass balance equation:

\[
q_e = \frac{(C_0 - C_e)V}{M}
\]

where \( C_0 \) and \( C_e \) are the initial concentration and equilibrium concentration of metals (mg/L), respectively, \( V \) the volume of solution (L), and \( M \) is the resin mass (g).

### 2.2. Ion-exchange resins

Several resins were tested and two were selected for further evaluation. Before using them, the resins were soaked in de-ionized water for 24 h and then were rinsed several times also with deionized water.

### 2.3. Batch studies

Batch studies of sulfuric acid strength, kinetics studies, and batch equilibrium isotherm studies were carried out in the same manner. In each type of studies, we prepared metal solutions containing tellurium and cadmium and transferred them into 250 mL wide-mouth polypropylene test bottles, with screw closure caps using a FINNIPET\textsuperscript{TM} pipette (accurate to \( \pm 0.3\% \) with a precision of 0.2%). Each bottle contained a known volume of the solution. A known amount of resin was then added into each bottle. Afterwards, the bottles were shaken continuously at 100 rpm on an INNOVA 2100 platform shaker manufactured by New Brunswick Scientific Co. Inc. At certain intervals of contact time, the solution samples were withdrawn and filtered through 0.70 \( \mu \)m filters. The tellurium and cadmium concentrations of the filtered liquid samples were measured with an ICP emission spectrometer. The experiments were conducted in duplicate, and mean values were used in analyzing the data.

### 3. Chemistry of leaching and ion exchange

Thermodynamic information on tellurium [25] and cadmium [27,28] showed the following: Cadmium is soluble in acid media, and insoluble in neutral and strongly alkaline media; tellurium (IV) is sparingly soluble in acid media, insoluble in neutral media, and soluble in alkaline media; tellurium (V) is soluble in acid media, and insoluble in alkaline media. Other studies showed that tellurium can be readily oxidized with hydrogen peroxide in acid media [1,2,25,26]. In preparing the initial tellurium-cadmium-containing solutions from the raw CdTe powder, a leaching procedure was employed. During leaching, in the presence of sulfuric acid and hydrogen peroxide, the Cd and Te are extracted and solubilized into the liquid phase based on the following

---

the concentration of $H^+$, or, in other words, the strength of
acidic the solution is, the more likely tellurium(VI) is in the
form of $Te^{6+}$. Tellurium(VI) would be in the form of $H_2TeO_6$ in
acidic media because $H_2TeO_6$ is a weak acid. Should any
tellurium(VI) ions exist in acid media, they would be nega-
tively charged. Therefore, it is unlikely that free $Te^{6+}$ exists
in the solution. In the present experiments, the only possi-
ble ions existing in sulfuric acid solution were $Cd^{2+}$, $H^+$,
$Te^{4+}$, and $TeO(OH)^+$. On adding the ion-exchange resin A
into the solution, the following reversible adsorption reac-
tions are possible:

$$2RSO_3^- + H^+ \leftrightarrow (RSO_3^-)_2Cd^{2+} + 2H^+ \quad (6)$$

$$4RSO_3^- + H^+ + Te^{4+} \leftrightarrow (RSO_3^-)_2Te^{4+} + 4H^+ \quad (7)$$

$$RSO_3^-(H^+ + TeO(OH)^+) \leftrightarrow RSO_3^-TeO(OH)^+ + H^+ \quad (8)$$

According to these reactions, low acidity will favor for-
ward reactions whereas high acidity will favor backward reac-
tions. Although all these listed adsorption reactions might
take place, their extent (completeness) for specific ion/species
is very different due to the different affinity and selectivity of
the resin to positively charged ions, which is the base for the
separation of ions in the solution.

4. Results and discussion

4.1. Batch acid strength studies

We investigated the effect of acid strength on the resin’s
adsorption capacity by using solutions containing 1060 ppm
of $Cd$ and 1240 ppm of $Te$ in sulfuric acid strengths of 0.5,
1–5 M. We mixed 100 mL of resin with 2, 5, and 10 g of resin
A for a total contact time of 24 h, which was sufficiently long
for the system to reach equilibrium.

As seen from the reversible adsorption reactions (6)–(8),
the concentration of $H^+$, or, in other words, the strength of
sulfuric acid, plays an important role in adsorption. As the
concentration of $H^+$ increases, the positively charged metal
ions compete to a lesser degree for exchange on the resin.
Fig. 1 shows the dependence of the adsorption of tellurium
and cadmium on the strength of the sulfuric acid. A very
low percentage of cadmium was removed at high $H_2SO_4$
concentration. For instance, at an initial 5 M $H_2SO_4$ con-
centrations, less than 10% of the cadmium was removed.
Yet, at that concentration, more cadmium was exchanged
from the solution. Overall, whatever the initial $H_2SO_4$
concentration was, many more cadmium ions were adsorbed
on the resin than were tellurium. This suggests that by carefully
controlling the acidity of the solution, cadmium can be selec-
tively adsorbed in the resin leaving tellurium in the liquid
phase. By selecting an appropriate strength of acid, 0.5 M in
our studies, the effluent liquid phase can be virtually free of
cadmium while tellurium retained in solution and cadmium
ions adsorbed on the resin. Elution of the resin will generate
concentrated cadmium solution, from which cadmium can be
recovered. Investigations on $Cd$ and $Te$ recovery are in
progress.

The manufacturer’s suggested operating pH range for resin
A is an all inclusive 0–14. For effectively removing cad-
mium in the tellurium–cadmium-resin systems we studied,
low acidity is preferable. However, according to our stud-
ies on the leaching behavior of CdTe containing PV glass
waste, a low acid concentration would have the following consequences: (1) incomplete extraction of tellurium and cadmium; (2) precipitation of TeO₂ from the leachate when the H₂SO₄ concentration was below 0.5 M. Accordingly, the goal of removing both tellurium and cadmium from PV glass waste would not be met. Investigations on the adsorption of cadmium and tellurium at concentrations below 0.5 M H₂SO₄ were beyond the scope of present studies.

Our studies also demonstrate that the amount of resin affects the percentage of cadmium removed. Fig. 1 shows that to remove more than 90% of 1060 ppm of cadmium from 100 mL solution with 0.5 M sulfuric acid, a minimum of 10 g of resin A is required at equilibrium. In comparison, only 83% and 62% of cadmium was removed when the amount of the resin was 5 and 2 g, respectively. As expected, the equilibrium concentrations of cadmium and tellurium in the liquid phase decrease with the increase in the amount of resin because the number of active H⁺ sites increases. However, this effect is not linear in high H₂SO₄ concentration solutions. As shown in Fig. 1, in 5.0 M H₂SO₄, the efficiency of cadmium removal was 11%, 7%, and 5% when the resin level was 10, 5, and 2 g, respectively.

4.2. Batch kinetic studies

The batch kinetic studies were conducted with the metal solutions containing initial concentrations of Cd 1000 ppm and Te 1270 ppm in 0.5 M sulfuric acid media. The amounts of resin A used were 5, 10, and 20 g, respectively. In each experiment, a total of 240 mL metal solution was used. The solution samples were withdrawn at contact times of 10, 20, 30, 40, 60, 80, 100, 120, and 720 min. Fig. 2(a) and (b) plot the ion-exchange kinetics for separating cadmium from tellurium with different amounts of resin A at an initial H₂SO₄ concentration of 0.5 M. It is clear from Fig. 2(a) and (b), that significant and rapid adsorption of cadmium took place within first 20 min, beyond which there was only a marginal increase in adsorption. Within 2 h, the concentrations of cadmium in the solutions reached a steady state, indicating that the ion-exchange reactions reached equilibrium. The adsorption equilibrium of tellurium was well established within less than 20 min. We confirmed that both the Cd and Te concentrations remained constant, by continuing sampling and analysis up for up to 12 h.

As discussed earlier, tellurium exists in the forms of Te(IV) and Te(VI) in sulfuric acid solutions. The only possible ions of tellurium in acidic media are Te(IV) since Te(VI) cannot be positively charged. Moreover, by using H₂O₂ in the leaching process, a large fraction of tellurium was oxidized to Te(VI). Consequently, only a small amount, if any, of positively charged Te(IV) may exist in the studied solutions. When in contact with resin A, positively charged Te(IV) ions, if any, would compete with cadmium ions. Evidently, the resin has a much higher affinity for cadmium, so unsurprisingly, the removal percentage of cadmium was much higher than that of tellurium. The two metals were completely separated in our column studies.

The mechanism of ion-exchange involves diffusion through a liquid film around the resin particle, and diffusion within the particle. The former is the rate-controlling mechanism at low concentrations, and the latter at high concentrations. However, in both cases, the size of the resin particles also is a determining factor.

We investigated the kinetics of cadmium and tellurium adsorptions for resin A at its optimum 0.5 M H₂SO₄ concentration at short intervals over 12 h. We expected that the kinetic studies would help in evaluating which classical models were suitable for assessing cadmium and tellurium adsorption. Fig. 3 shows the kinetics of cadmium adsorption on the resin.

Several mathematical models have been used to describe the adsorption kinetics of resin–solution systems. The most popular and frequently used models are Lagergren’s first order model [29,30] and Ho’s pseudo second-order model [31].

The Lagergren’s first order reaction model is expressed as follows [29,30]:

\[
\ln(q_e - q_t) = \ln(q_e) - Kt
\]

(9)

Fig. 2. (a) Cadmium removal percentage vs. time at initial H₂SO₄ concentration of 0.5 M (initial metal concentrations: Cd, 1090 ppm; Te, 1270 ppm, solution, 240 mL). (b) Tellurium removal percentage vs. time at initial H₂SO₄ concentration of 0.5 M (initial metal concentrations: Cd, 1090 ppm; Te, 1270 ppm, solution, 240 mL).
The above equation can be rearranged as follows:

\[
\ln\left(1 - \frac{q_t}{q_e}\right) = -K_L t
\]

(9a)

\[
q_t = q_e \left(1 - \exp\left(-K_L t\right)\right)
\]

(9b)

where \(K_L\) is the Lagergren’s rate constant for adsorption (min\(^{-1}\)), \(q_e\) the amount of metal ions adsorbed at equilibrium (mg/g), and \(q_t\) is the amount of metal ions adsorbed at any given time \(t\) (mg/g).

Ho developed a pseudo second-order kinetic expression for the competitive sorption systems of heavy metal ions by sphagnum moss peat [31]. It is expressed as follows:

\[
\frac{t}{q_t} = \frac{1}{(2Kq_e^2)} + \frac{t}{q_e}
\]

(10)

This can be rearranged to the form:

\[
q_t = (2Kq_e^2)\left(1 + 2Kq_e t\right)
\]

(10a)

where \(K\) is the pseudo second-order rate constant for adsorption (g/mg min), \(q_e\) the amount of metal ion adsorbed at equilibrium (mg/g), and \(q_t\) is the amount of metal ions adsorbed at any given time \(t\) (mg/g).

The batch kinetic study data for optimum initial H\(_2\)SO\(_4\) of 0.5 M was fitted to both models by nonlinear regression analysis using the software package ProStat (Version 3) for Windows. The analysis indicates that both models adequately described the kinetic data at the 95% confidence level. The Lagergren first-order model described the kinetic data quite well with higher coefficient of determination (COD) and correlation \(R^2\) values than Ho’s pseudo second-order model.

Figs. 4 and 5 show the kinetics of cadmium adsorption on resin A.

Table 1

Comparison of two models for cadmium adsorption on resin A

<table>
<thead>
<tr>
<th>Resin amount (g)</th>
<th>Model type</th>
<th>Model equation</th>
<th>COD</th>
<th>(R^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>First-order</td>
<td>(q_t = 3(1 - e^{-0.05t}))</td>
<td>0.997</td>
<td>0.998</td>
</tr>
<tr>
<td></td>
<td>Pseudo-second-order</td>
<td>(q_t = 1(2)0.006(1)^{33t} + 33)</td>
<td>0.973</td>
<td>0.991</td>
</tr>
<tr>
<td>10</td>
<td>First-order</td>
<td>(q_t = 2(1 - e^{-0.07t}))</td>
<td>0.998</td>
<td>0.999</td>
</tr>
<tr>
<td></td>
<td>Pseudo-second-order</td>
<td>(q_t = 1(2)0.037(21^3t) + 21)</td>
<td>0.977</td>
<td>0.992</td>
</tr>
<tr>
<td>20</td>
<td>First-order</td>
<td>(q_t = 12(1 - e^{-0.11t}))</td>
<td>0.999</td>
<td>0.999</td>
</tr>
<tr>
<td></td>
<td>Pseudo-second-order</td>
<td>(q_t = 1(2)0.031(12^3t) + 12)</td>
<td>0.989</td>
<td>0.996</td>
</tr>
</tbody>
</table>
mathematical models are widely used in describing ion-exchange
exchanger at constant temperature [20]. Two nonlinear math-
solution against the concentration of the same ion in the solid
equilibrium plot of the concentration of the exchanging ion in
at the end of the adsorption.
Both tellurium and cadmium concentrations were measured
sibility of leaching of tellurium and cadmium from the resin.
mixed and shaken with 5 g of the resin to determine the pos-
cient to reach equilibrium. A control solution with 100 mL of
each prepared solution and shaken for 24 h, which was suffi-
H₂SO₄ solution. Table 2 lists the concentrations of the pre-
and 1160 ppm Te in 0.5 M sulfuric acid, with virgin 0.5 M
tations were prepared by diluting a stock of 1040 ppm Cd
and 1160 ppm Te in 0.5 M sulfuric acid, with virgin 0.5 M
permanganate [8] and 0.055 min⁻¹ for cadmium adsorption on
vermiculite [8] and 0.055 min⁻¹ on perlite [23].
4.3. Equilibrium isotherm studies
Subsequent to determining the optimum strength of sul-
furic acid and the adsorption equilibrium time, equilib-
rium isotherm studies were conducted at room temperature
(23 ± 1 °C) by varying the initial concentrations of tellurium
and cadmium. A fixed 100 mL liquid volume was used in
each experiment. Solutions of different Cd and Te concen-
trations were prepared by diluting a stock of 1040 ppm Cd
and 1160 ppm Te in 0.5 M sulfuric acid, with virgin 0.5 M
H₂SO₄ solution. Table 2 lists the concentrations of the pre-
pared solutions. Then, 5 g of resin A were added to 100 mL of
each prepared solution and shaken for 24 h, which was suffi-
cient to reach equilibrium. A control solution with 100 mL of
0.5 M sulfuric acid solution, but without the metals, was also
mixed and shaken with 5 g of the resin to determine the pos-
sibility of leaching of tellurium and cadmium from the resin.
Both tellurium and cadmium concentrations were measured
at the end of the adsorption.
The ion-exchange equilibrium for metal ions can be con-
veniently analyzed by the ion-exchange isotherm, which is an
equilibrium plot of the concentration of the exchanged ion in
solution against the concentration of the same ion in the solid
exchanger at constant temperature [20]. Two nonlinear math-
ematical models are widely used in describing ion-exchange
isotherms: Freundlich’s model and Langmuir’s model.
The Freundlich’s model is often written as [32]:

\[ q = K_f C_e^1/n \]  \hspace{1cm} (11)

where \( q \) is amount of metal ions adsorbed per unit of weight of
resin (mg/g), \( K_f \) an equilibrium constant indicative of adsorp-
tion capacity, \( n \) the adsorption equilibrium constant whose
reciprocal is indicative of adsorption intensity, and \( C_e \) is the
concentration of metal ions in bulk solution at equilibrium
(mg/L).

The Langmuir model is mathematically written as follows
[33]:

\[ q = (q'_m bC_e)/(1 + bC_e) \] \hspace{1cm} (12)

where \( q' \) is amount of solute adsorbed per unit weight of resin
in forming a complete monolayer on the surface (mg/g), \( b \) the
constant related to the energy or enthalpy of adsorption,
and \( C_e \) is the concentration of metal ions in bulk solution at
equilibrium (mg/L).

The experimental results were analyzed using the software
package ProStat (Version 3). We found that the adsorption
data obtained were best fitted with the Freundlich isotherm
model. The obtained equation is \( q = (0.29C^0.7)^{1.26} \). The con-
stants obtained with this model were statistically significant
at 95% confidence level. The COD is 0.985, and the R² is
0.992. Fig. 5(a) shows plots of the Freundlich model as well
experimental results for cadmium adsorption on resin A.
The equilibrium isotherm of tellurium was also investi-
gated and the equilibrium plot of the concentrations of the
tellurium in solution against the concentrations of the tel-
urium in the resin is shown in Fig. 5(b). The adsorption of
tellurium on the resin did not fit the considered equilibrium
isotherm models. This suggests that the tellurium may be
adsorbed only physically onto the resin.
The experimental results also showed that, in the present
batch study conditions, resin A had an adsorption capacity
of 33 mg/g-resin for cadmium and 5 mg/g-resin for tellurium.
The equilibrium isotherm studies revealed that the selectivity
of resin A to cadmium is much higher than to tellurium. This
makes it possible to separate these two metals using ion-
exchange resin column.

4.4. Column studies
The column studies were conducted to elucidate the shape
of the breakthrough curves of cadmium and tellurium. In the
column studies, the effluent solution was collected at different
volume fractions and was analyzed by ICP for cadmium and
tellurium. The efficiency of adsorbing cadmium and tellurium
is presented by breakthrough curves showing concentration
ratios C/C₀ as a function of throughput volumes, where C is
the instantaneous concentration of effluents and C₀ is the
initial concentration of influent. Fig. 6 shows such curves for
tellurium and cadmium from an experiment on resin B. In this
test, the nominal resident time of the influent solution in
the resin column was 16 min. It is shown that the tellurium
breaks through the column almost immediately (at 50 mL,
0.5 bed volumes) after feeding and the column took approx-
imately 800 mL (8 bed volumes) before being completed.
Fig. 6. Removal of Cd from Cd- and Te-containing solution using resin B; volume, 100 mL. Influent concentrations: Te, 1160 ppm; Cd, 980 ppm; solution flow rate: 500 mL/h.

exhausted with tellurium. On the other hand, the cadmium was adsorbed for a substantial period of time, not achieving breakthrough until 1800 mL, and reached exhaustion after 2600 mL of throughput volume. Separation of cadmium from tellurium was fairly complete, as shown by the ICP analysis of the effluent fractions. In each of the effluent fractions which were collected prior to 1600 mL, the cadmium concentration was below 10 ppm showing that at least 99% of cadmium was removed by the resin, whereas tellurium was retained in the solution.

The steep increase in the concentrations of Cd and Te met- als in the effluent fractions indicates that a relatively small volume of solution would cause the column to reach complete exhaustion of the resin from the breakthrough point, or, in another words, very short mass transfer zone (MTZ). Mass balance calculations show that 2.1 g of cadmium were exchanged to 100 mL of swollen resin.

The column study results with resin A are plotted in Fig. 7. In this test, the nominal resident time of influent solution in the resin column was 27 min. The same trend is observed except that the breakthrough of cadmium did not occur until 2200 mL of influent was passed through the column. When a total of 3200 mL of influent solution was passed through the resin column, 2.7 g of cadmium were removed from solution. In the first 1800 mL of collected effluent, the cadmium removal percentage was always 99.9% or better.

When two ion-exchange columns were arranged in series, the removal of cadmium increased to 99.99% or better.

Fig. 7. Removal of Cd from Cd- and Te-containing solution using resin A; volume, 100 mL. Influent concentrations: Te, 1120 ppm; Cd, 942 ppm; solution flow rate: 300 mL/h.
Fig. 8. Removal of Cd from Cd- and Te-containing solution using resin A; two columns in series (100 mL each). Influent concentrations: Te, 1150 ppm; Cd, 989 ppm; solution flow rate: 750 mL/h.

5. Conclusion

The feasibility of removing and separating cadmium from tellurium in acidic media was studied using two ion-exchange resins. The following conclusions were drawn from the present studies:

- Significant and rapid adsorption took place within 20 min on both resins.
- Batch studies with resin A showed that approximately 91% of the cadmium was removed, while 21% tellurium was also adsorbed onto the resin.
- A kinetic study at 0.5 M H₂SO₄ showed that equilibrium for cadmium adsorption on resin A was attained within 2 h.
- The Lagergren first-order model described the kinetic data quite well, with higher coefficient of determination (COD) and correlation (R²) values than Ho’s model.

- The equilibrium isotherm analysis of the experimental data showed that the ion exchange for cadmium onto the resin follows the Freundlich isotherm.
- Column studies revealed that both resins A and B were effective in removing cadmium from tellurium. However, resin A showed the least leakage of cadmium in our tests.
- Column studies with two columns in series, showed a removal of cadmium higher than 99.99%, whereas 90–96% of the tellurium remained in the solution. It is expected that very high tellurium recovery (e.g., 99.99%) can be achieved in recirculating systems.
- Ion exchange was found to be an effective method for separating cadmium from tellurium in sulfuric acid media.

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References


