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# ASSESSMENT OF TEMPERATURE AND ELECTRIC FIELD EFFECT ON NICKEL RECOVERY FROM SPENT CATALYST VIA ELECTRO-LEACHING

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***Abstract.*** *Catalysts from the oils and fats hydrogenation process are a matrix composed of spent catalysts and organic materials, such as free fatty acids and vegetable oil. These wastes can become an environmental problem if they do not receive a suitable solid waste treatment. However, when disposed these materials carry within themselves some species that have economical value, such as nickel. Aiming to comply with environmental standards of waste disposal, and also to recover nickel from spent catalysts, electro-leaching arises as a feasible recovering technique. Therefore, the aim of the present work was to evaluate the synergetic effect of electric field and temperature on nickel ions from spent catalysts from the hydrogenation of oils and fats process. Four experiments were conducted, in which samples were previously calcinated at 400 °C. In experiments 1 and 2 (EL-01 and El-02), each sample was added to a solution of sulfuric acid (1.5 mol $L^{-1}$), remaining in the solution for 48 h. Subsequently, in the test EL-01, the mixture was submitted to the electro-leaching process for 192 h, applying an electric field of 1.0 V $cm^{-1}$ at room temperature. Test EL-02 was conducted analogously to EL-01, however, the electric field was not applied. In experiments 3 and 4 (EL-03 and EL-04), samples remained in a sulfuric solution (1.5 mol $L^{-1}$) for 16 h. After that, in test EL-03, the mixture underwent the electro-leaching process for 48 h at 45 °C. In experiment EL-04 the mixture remained in solution, without electric field application. The results showed that test EL-03 presented the highest recovery, removing 42.5% of $Ni^{2+}$. On the other hand, in experiment EL-04, the $Ni^{2+}$ recovery was 21.1%. Tests EL-01 and EL-02 presented the lowest removal, resulting in 15.7% and 10.3% of $Ni^{2+}$ removal respectively. In light of these results, it was observed that the synergetic effect between temperature and electric field favored the removal of $Ni^{2+}$ from catalysts, enabling the species removal using a less pollutant technique than the conventional leaching process.*

***Keywords:*** *electro-assisted leaching, metallic ions recovery, electromigration*

## 1. INTRODUCTION

The fats and oils industry in Brazil has grown rapidly throughout the years driven by soy oil processing reaching an average production of 202.337 t/day (Abiove, 2022). However, a high amount of residue is generated during the process. Hydrogenation is one of the steps of the chemical process to produce margarine, which consists in the saturation of the double bonds in the unsaturated fatty acids chains through the addition of hydrogen atoms (Coenen, 1976), modifying the physicochemical characteristics of the oil and increasing its stability against oxidation and decomposition (Choo et al., 2001). This process is accomplished by applying metallic catalysts, where nickel (Ni) is the active agent for the occurrence of the hydrogenation reaction (Yadav and Sharma 2019; Razavian et al., 2020).

Although Ni catalysts are economic and highly active, due to the poisoning of the active sites the catalytic activities reduce after excessive use, promoting the deactivation of the catalyst (Idris et al., 2010; Bartzas et al., 2021). The spent Ni catalyst originated from fats and oils hydrogenation is a matrix composed by spent catalyst and organic material, such as free fatty acids (FFA), vegetable oil and other products from unaccomplished hydrogenation (Esmaeili and Rahimpour 2017). Eventually, this material becomes an environmental issue due to its nickel and oil content (Akcil et al., 2015). Furthermore, the disposal of such residue without previous treatment is forbidden by Law 12,305/10, which institutes the national policy for solid residues.

Techniques for recuperation of Ni in solid residues from industrial processes have been studied throughout the years, such as: metal extraction by acid or alkaline leeching acids, hydrometallurgy, pyrometallurgy, ion exchange, and electrokinetic remediation (Idris et al., 2010; Sheik et al., 2013; Esmaeili and Rahimpour, 2017; Pires et al. 2019; Pradhan et al., 2020; Maidel et al., 2022). Among these techniques, electroremediation stands out for its use of low concentrations of acids and low operating temperatures. This technique is also known as electric field-assisted leaching (EFAL). Although the technique is widely used for soil contaminant removal (Pires et al., 2019), EFAL has also shown promising

results in extracting metallic elements from spent fluid catalytic cracking (FCC) catalysts in the petroleum industry (Valt et al., 2015; Maidel et al., 2022).

EFAL is an electrokinetic technique that utilizes the electric field for the removal of species in ionic form. Additionally, electrolytes are used to assist in mass transport of the species, which align themselves along the electric field lines, promoting ion extraction (Acar et al., 1995; Song et al., 2019). In addition to species transport, parallel reactions can occur on the electrode surfaces, such as water electrolysis, which is responsible for pH variation in the anodic and cathodic regions (Cameselle, 2014).

Although nickel (Ni) has economic value and is considered a critical material by the European Commission (2023), when catalysts from oil and fat processes are discarded without prior treatment, the simultaneous disposal of Ni occurs. Thus, the removal of Ni from this secondary source contributes to the supply of consumer demand, also favoring the circular economy. Therefore, the aim of this study was to evaluate the synergistic effect of electric field and process temperature variables on the recovery of nickel ions ($Ni^{2+}$) from spent catalysts derived from oil and fat hydrogenation using the electro-leaching technique.

## 2. MATERIAL AND METHODS

Table 1 presents the composition of the spent catalyst, which was characterized using the semi-quantitative X-ray fluorescence (XRF) method.

Table 1. Composition of the Ni spent catalyst

| **Compound** | $NiO_2$ | $SiO_2$ | MgO | $Al_2O_3$ | $Fe_2O_3$ | $K_2O$ | $Na_2O$ | $SO_3$ | $P_2O_5$ | LOI* |
|---|---|---|---|---|---|---|---|---|---|---|
| **(%)** | 31.6 | 21.0 | 1.9 | 1.9 | 0.7 | 0.7 | 0.6 | 0.6 | < 0.2 | 40.8 |

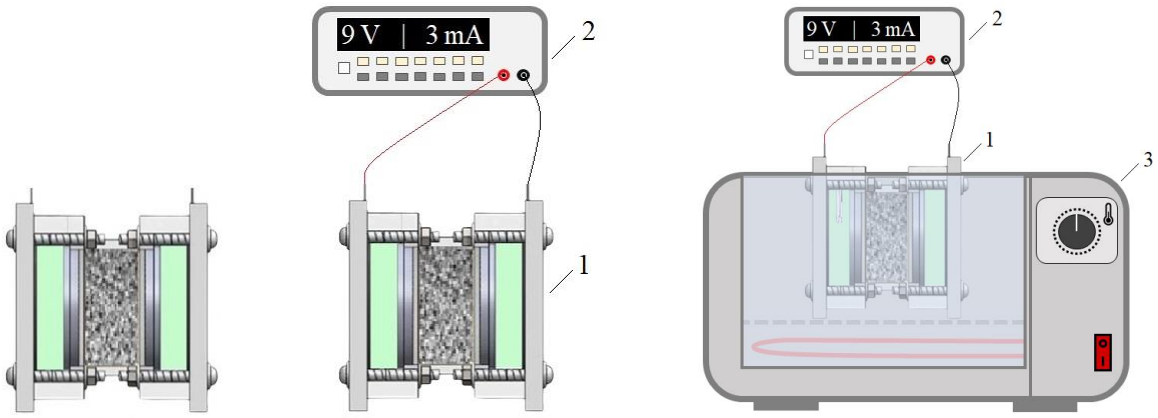
**LOI: Loss-on-ignition

### 2.1 Electro-leaching

In order to remove contaminants and free fatty acids from the spent Ni catalyst particles, the sample was calcined at 400°C for 3 h in a hermetically sealed 20 L stainless steel (AISI 316) vessel. During heating, the organic matter and water present in the catalyst are evaporated, and the vapors are condensed in a water-cooled heat exchanger, installed at the gas outlet. After cooling, water could be separated from fat through phase differentiation. To perform the electro-leaching experiments, an electrochemical reactor was used (Figure 1a). The reactor used has a length of 9 cm, with a cylindrical bed of 8 cm internal diameter, and an internal volume of 452 $cm^2$. The electrolytic chambers have an internal volume of 150 $cm^3$. In each experiment, 60 g of spent Ni catalyst were added to the reactor bed. Subsequently, the electrolyte solution was pumped into the system until it filled the internal volume of the cell. The flow of this solution occurred from the anode chamber (AC) towards the cathode chamber (CC). The electrodes used were made of titanium (grade 2), which were attached to the ends of the reactor. To provide the electric field, the electrodes were connected to a power supply (Agilent - E3645A).

Four electro-leaching experiments were conducted. In the first experiment (Figure 1b), named EL-01, the spent Ni catalyst initially rested for 48 h in a sulfuric acid ($H_2SO_4$) solution (95% NEON - CAS 7664-93-9) at a concentration of 1.5 mol $L^{-1}$ in the electrochemical reactor bed. Subsequently, the sample underwent electro-leaching process for 192 h with the same electrolyte used during soaking, applying an electric field of 1 V $cm^{-1}$. This test was conducted at room temperature (23°C ± 2°C). The second experiment (EL-02) was carried out similarly to the first one, except that no electric field was applied, as shown in Figure 1a.

In the third experiment (EL-03), the spent Ni catalyst rested for 16 h in a sulfuric acid solution at a concentration of 1.5 mol $L^{-1}$. Immediately after, the sample was submitted to an electro-leaching process for 48 h using the same electrolyte, and applying an electric field of 1 V $cm^{-1}$. In this experiment, the reactor was placed in a thermostatic bath at a temperature of 45°C (Figure 1c) during both the soaking and electro-leaching stages. Finally, the fourth experiment (EL-04) was conducted following the same procedure as the third test, however, without the application of an electric field (Figure 1a).




(a) (b) (c)

Figure 1. Experimental apparatus with: (a) electrochemical reactor used in experiments EL-02 and EL-04, (b) electrochemical reactor (1) and power supply (2) used in experiment EL-01, and (c) electrochemical reactor (1), power supply (2) and thermostatic bath (3) used in used in experiment EL-03

In all experiments, aliquots of the anolyte and catholyte were collected to determine the concentration of $Ni^{2+}$ ions during the experiments. For the EL-01 and EL-02 tests, samples were collected every 24 h. For the EL-03 and EL-04 experiments, samples were collected every 6 h. The electric current was monitored at each sample collection. The quantification of $Ni^{2+}$ ions was performed using the analytical method of polarography (Methrom - 797 VA). To investigate the presence of possible oxide deposits on the electrode surfaces, scanning electron microscopy (SEM) was used, and energy-dispersive spectroscopy (EDS) was employed for the quantification of these species.

### 2.2 Electro-leaching efficiency process

The removal efficiency ($\xi$) of the electro-leaching process for Ni was calculated from the ratio between the final recovered mass ($m_r$) of the species, which migrated to the anode (AC) and cathode (CC) chambers, and the initial mass ($m_0$) of Ni present in the catalyst before treatment. Therefore,

$$\xi = \frac{m_r}{m_0} 100\% . \tag{1}$$

To obtain the recovered mass of $Ni^{2+}$, the concentration of the species in the electrolytic chambers (AC and CC) and the volume of the chambers ($V_{AC} = V_{CC} = 0.15$ L) were considered. Therefore, Eq. (1) can be rewritten as follows:

$$\xi = \left( \frac{C_{AC} V_{AC} + C_{CC} V_{CC}}{m_0} \right) 100\% . \tag{2}$$

## 3. RESULTS AND DISCUSSION

After the resting time of the samples in the $H_2SO_4$ solutions, a green color was observed in the electrolytic chambers in all cases studied. According to Randhawa et al. (2016), the green color can be attributed to the presence of $Ni^{2+}$ ions in the electrolyte. Furthermore, the authors Al-Mansi and Abdel Monem (2002) claim that nickel oxide in the presence of $H_2SO_4$ can undergo the following reactions:

$$NiO + H_2SO_4 \rightleftarrows NiSO_4 + H_2O , \tag{3}$$

$$Ni^{2+} + H_2SO_4 \rightleftarrows NiSO_4 + H_2 , \tag{4}$$

$$Ni^{2+} + H_2SO_4 + H_2O_2 \rightleftarrows NiSO_4 + 2H_2O , \tag{5}$$

and

$$Ni^{2+} + S^{2-} \rightleftarrows NiS\,. \tag{6}$$

Another factor that may be related to the green coloration is the formation of a cationic complex, where $Ni^{2+}$ might be solvated, forming the hexaaquanickel (II) species, according to Habib et al., (2021):

$$Ni^{2+} + 6H_2O \rightleftarrows Ni\left(H_2O\right)_6^{2+} \tag{7}$$

The presence of $Ni^{2+}$ ions after the resting period of the catalyst in the $H_2SO_4$ solutions was confirmed by the polarography quantification method. Subsequently, the removed mass of Ni was calculated for each experiment, i.e., the removal by conventional leaching. The average mass obtained for the 48-hour resting period at room temperature was 0.0018 g, and for the 16-hour resting period at 45°C was 0.0028 g. Therefore, the initial mass ($m_0$) of remaining Ni in the catalyst for the EL-01 and EL-02 tests was 0.2242 g, and for the EL-03 and EL-04 ones was $m_0$ = 0.2432 g. After this step, the electro-leaching experiments were initiated.

During electro-leaching, the water electrolysis occurs on the surface of the electrodes, where the anodic reaction is given by:

$$H_2O \rightarrow 2H^+ + \frac{1}{2}O_2 + 2e^-\,, \tag{8}$$

and the cathodic reaction is:

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2\,. \tag{9}$$

Considering the reaction from Eq. (8), the presence of hydroxyl ions $(OH)^-$ can lead to the formation of other nickel compounds during treatment time, such as the hydroxides: $Ni(OH)^+$ and $Ni(OH)_2$. However, considering that the reaction medium is acidic, the insoluble species $Ni(OH)_2$ can be redissolved, making $Ni^{2+}$ available again for removal via electromigration (Randhawa et al., 2016), according to:

$$Ni\left(OH\right)_2 + 2H^+ \rightleftarrows Ni^{2+} + 2H_2O\,. \tag{10}$$

### 3.1 Electro-leaching efficiency

The efficiency of $Ni^{2+}$ removal for each experiment was obtained using Eq. (2). Figure 2 shows the behavior of $Ni^{2+}$ removal over time for the experiments. Electro-leachings EL-01 and EL-03 were conducted with the assistance of the electric field, while the others were not subjected to it. In all cases, the extraction of $Ni^{2+}$ was observed in both the anode (AC) and cathode (CC) chambers. Therefore, to obtain the total extraction, the extractions in both chambers, AC and CC, were summed, resulting in a $Ni^{2+}$ extraction of 15.7% for EL-01, 10.8% for EL-02, 39.5% for EL-03, and 21.5% for EL-04.

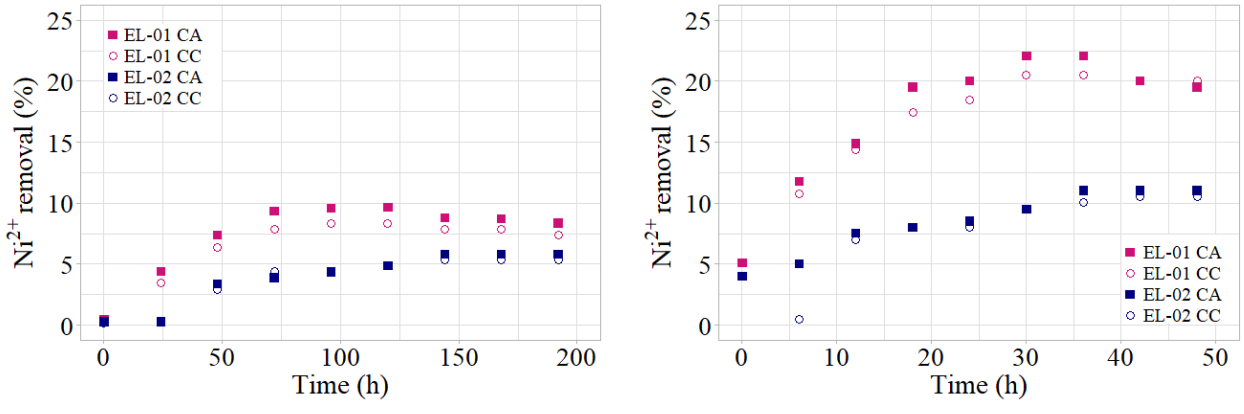
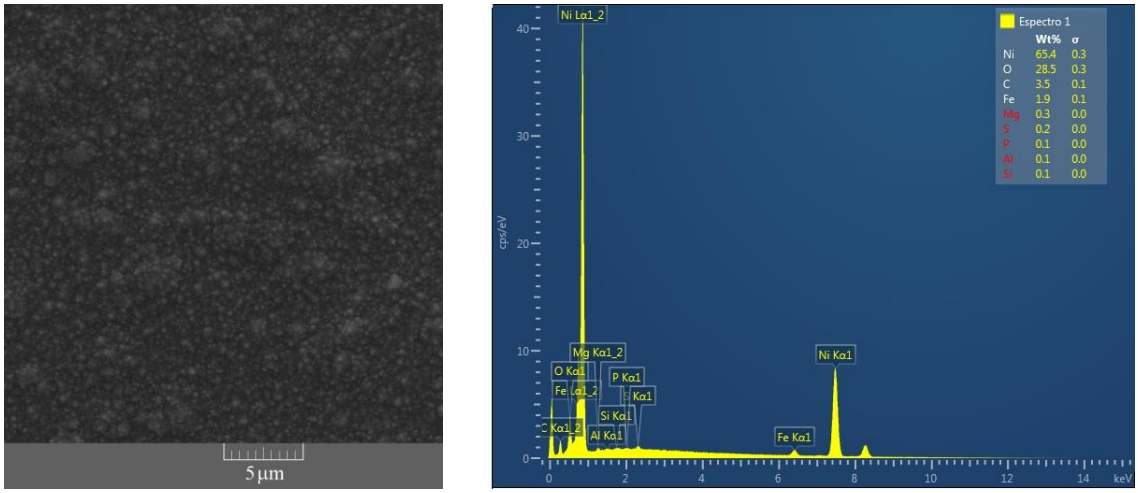



Figure 2. $Ni^{2+}$ removal efficiency of electro-leaching experiments. The removal in both chambers (anodic and cathodic) was considered

According to Figure 2, it can be observed that electro-leaching EL-01 presented an increasing removal profile up to 96 h of treatment. The same trend was observed in EL-03, however, up to 30 h of testing. After these periods, in both cases, the removal efficiency decreased until the end of the experiments. One of the factors that may be related to this reduction in $Ni^{2+}$ removal was the formation of a nickel oxide film on the surface of the cathodic electrode (Figure 3). Consequently, $Ni^{2+}$ ions present in the solution were transferred to the electrode surface in the form of nickel oxide. Hence, the presence of this film reduces the effectiveness of the electric field over time due to the increased resistance of the medium resulting from the deposition of a low-conductivity film (Park et al., 2015).




(a) (b)

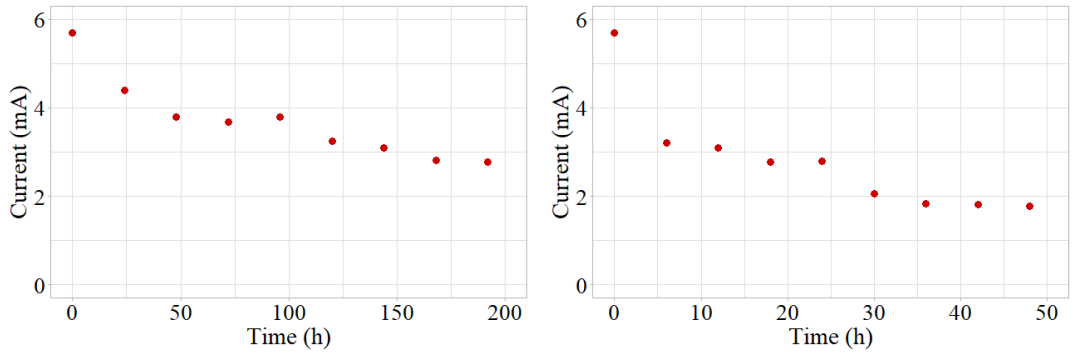
Figure 3. Characterization of the nickel oxide film formed on the cathodic electrode obtained by (a) SEM micrograph via SE (secondary electrons), and (b) EDS spectrum

When comparing the experiments in pairs, EL-01 to EL-02, and EL-03 to EL-04, the effectiveness of the electric field in the extraction of species can be confirmed. However, when comparing the electro-leaching EL-01 with EL-03, a different behavior in the extraction of $Ni^{2+}$ was observed. In the EL-03 experiment, the removal of $Ni^{2+}$ was higher, resulting in an extraction efficiency of 39.5%, considering both electrolytic chambers. This result is associated to the temperature at which the test was conducted (45 °C), which promotes mass transfer of the species from the catalyst particle to the solution (Al-Abbad and Dwairi, 2021).

### 3.2 Electric current

During the EL-01 and EL-03 experiments, the electric current was monitored. In both tests, a decreasing profile of the variable was observed, as shown in Figure 4. Therefore, the explanation for the higher values of electric current in the first hours of the experiment is associated to the initial stabilization of the titanium oxide electrodes ($Ti/TiO_2$). In both experiments, oscillations in the electric current were observed throughout the tests. This trend may be associated with the development of parallel reactions on the surface of the electrodes, such as the formation of a nickel oxide film on the cathode surface, as shown in Figure 3.

Although the electro-leaching process was conducted with sulfuric acid at a concentration of 1.5 mol $L^{-1}$, the electric current dissipated in the process remained below 6 mA (Figure 4), contributing to a low removal cost. Additionally, the decreasing trend in the electric current value indicates that there was no corrosion on the anodic electrode, favoring the preservation of the electrodes.




(a) (b)

Figure 4. Profile of electric current during the experiments: (a) EL-01 and (b) EL-03

## 4. CONCLUSION

In the present study, the effect of electric field and temperature on the removal of $Ni^{2+}$ from spent catalysts derived from the oils and fats industry was evaluated. For this purpose, different experiments were conducted. The results indicated that the experiments conducted without the application of an electric field showed a removal of 21.1%. On the other hand, the experiment that showed the highest removal of $Ni^{2+}$ was conducted using an electric field of 1.0 V $cm^{-1}$, with sulfuric acid at a concentration of 1.5 mol $L^{-1}$ as the electrolyte, and an operating temperature of 45 °C. This electro-leaching resulted in an extraction of 39.5% of $Ni^{2+}$, representing an 87.2% increase in removal efficiency. Therefore, the results confirm the effectiveness of the electric field and temperature in improving the species removal.

When comparing the technique used in this study with other conventional methods of metal species leaching, electro-leaching emerges as a more attractive alternative due to its lower energy consumption and lower environmental impact, for it employs milder conditions to promote the extraction of species.

## 5. ACKNOWLEDGEMENTS

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