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Recovery of Al and Si from lunar regolith by low temperature electrochemical deposition from ethaline

ABSTRACT

In this study, the dissolution and electrochemical recovery of metals from regolith simulant using a choline chloride-based deep eutectic solvent (DES) and electrochemical recovery of metals from thus prepared electrolyte were investigated. For the first time, a designed lunar regolith representing Lunar Mare Soil (LMS-1) was dissolved in ethaline (choline chloride:ethylene glycol, ChCl:EG), and the solubility of the major oxide components of the regolith was determined using inductively coupled plasma–optical emission spectrometry (ICP–OES). The electrochemical reduction of dissolved metal species in the ethaline–regolith system involved multivalent redox transitions, which occurred at very close potentials or simultaneously. Based on electrochemical measurements and the determined equilibrium potentials of the metals, parameters for electrolysis were selected, and metal recovery was achieved by adjusting the electrode potentials. As a result, a deposit composed of Al, Cr, Fe, and Si is formed at a potential of -1.750 V vs. Pt, while at a more positive potential, the deposit consisted mainly of Si, according to EDS and ICP–OES analysis. The results indicate the potential of using DESs for the dissolution of lunar regolith and controlled electrochemical recovery of metals, introducing a previously unexplored strategy for extraterrestrial resource processing.

Keywords: Lunar regolith, Ethaline, Regolith dissolution in DES, Metals extraction from regolith

1.0 INTRODUCTION

To ensure sustainable space research and ultimately establish a human presence on the Moon, extraterrestrial mining is expected to become a primary objective [1,2]. The central aim of these efforts is to convert lunar resources into materials and components essential for sustaining human life [3–5]. Among the available resources, lunar regolith, originating from 4–5 m depths in Mare or 10–15 m depths in Highland regions, is particularly attractive due to its abundance and easy accessibility, making it a highly promising feedstock for such critical processes [3,6]. Therefore, it is reasonable to employ in situ resource utilization (ISRU) on the Moon to obtain oxygen and valuable metals from these essential resources [1,3,4,7].

The regolith is primarily composed of oxygen-bearing silicates and oxides, and its composition depends on whether it originates from the Mare or the Highland regions of the Moon [1,4,6]. The major oxide component of the lunar regolith is silica (SiO_2), ranging from 40.6 wt% to 48.1 wt%, followed by 4.7 to 24.5 wt% of FeO_x , 7% to 9.5 wt% MgO , from 6.3 to 28.0 wt% Al_2O_3 , and up to 8 wt% of TiO_2 [1,6,7]. It is evident that metals needed for construction, manufacturing, and life support systems in space can be extracted from lunar regolith [6,8]. Even now, coming into possession of genuine lunar regolith is difficult, so researchers use lunar regolith simulant, prepared based on information from the returned Apollo Lunar Soil samples, which represent adequate regions of the Moon [3]. Currently, most methods for metal extraction from lunar regolith are based on high-temperature molten salt electrolysis [7,9–15]. The main principle of the Magma process is that the regolith is melted directly at temperatures up to 1600°C [8,10,11,16]. Despite its widespread prospects, some issues remain unresolved, such as current efficiency when iron is present in the

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melt due to Fe^{2+} oxidation to Fe^{3+} [10], high operating temperature, and the selection of anode materials [8,11]. One of the most studied electrochemical methods for processing lunar regolith is the Cambridge FFC process (Fray, Farthing, Chen) [17]. This process is based on the solid-state electrochemical reduction of metal oxides to metals using a CaCl_2 electrolyte at temperatures around 900 °C [17]. In addition to chloride-based melts, molten fluoride salts have also been used to extract metals from lunar regolith [13,18]. An LiF – NaF eutectic mixture was used to dissolve the components of lunar regolith [13]. Another fluoride-based molten salt composed of NaF and AlF_3 was used for aluminothermic reduction and electrolysis of lunar regolith [18]. The product obtained from aluminothermic reduction at 980 °C was composed of Al, Si, and Al_5FeSi [18]. An approach using the same electrolyte system, after galvanostatic electrolysis, produced a cathodic product composed primarily of metallic Al and Si [7].

An alternative type of solvent capable of dissolving individual metal oxides present in the regolith has also been investigated. The ease of their synthesis, availability, and their low vapor pressure make these solvents, known as non-aqueous ionic liquids (ILs), or deep eutectic solvents (DESs), promising candidates for extracting metals from lunar regolith [19]. Despite certain limitations, the capacity of DESs to solubilize metal oxides has been proven, allowing the electrolysis process to be conducted at relatively low temperatures, typically in the range of 50–80 °C, which significantly reduces thermal challenges [19]. In the article [20], the authors studied metal extraction from lunar regolith EAC-1 using the ionic liquid 1-ethyl-3-methylimidazolium hydrogen sulfate as the solvent [20]. Potentiostatic electrolysis was applied to preferentially study oxygen extraction from lunar regolith, and it was emphasized that a metallic deposit was not produced [20]. In another article, the authors studied the dissolution of metals from two chondrites (H3, H5) as well as an iron IAB-MG meteorite, primarily composed of metallic Fe–Ni and troilite (FeS), in a deep eutectic solvent (DES). This led to the conclusion that using DES for sustainable metal recovery from lunar soil under extreme conditions is feasible [19].

The present work represents the first report on recovering the metals from the ethaline electrolyte containing dissolved regolith using potential controlled electrolysis. To address this, this study focuses on the dissolution of lunar regolith in ethaline and determines the reversible (equilibrium) potentials of dissolved metals in ethaline, as there is no existing data on this topic. To lay the

groundwork for the electrochemical extraction of valuable metals through ISRU, the electrolysis from a solvent containing dissolved regolith was performed by adjusting the deposition potential.

2.0. EXPERIMENTAL PART

2.1. Electrolyte preparation

Deep eutectic solvent (DES) precursors, choline chloride (ChCl , $\geq 98\%$) and ethylene glycol (EG, 99.8%), were obtained from Sigma-Aldrich and used without additional purification. Ethaline was synthesized by combining ChCl and EG in a 1:2 molar ratio under magnetic stirring at 50 °C until a transparent liquid was formed [21]. The electrolytes for electrochemical studies were prepared by dissolving selected amounts of LMS-1 (Lunar Mare Simulant, Space Resource Technology, Oviedo, FL, USA, 1g) into freshly prepared ethaline (50 ml). Dissolution of LMS-1 was carried out under argon atmosphere by mechanical stirring at 250 rpm for 6 h at 70 °C. LMS-1 is a high-fidelity lunar regolith simulant developed to mimic the composition of Lunar Mare regions, corresponding to the darker lunar surface areas. The simulant was synthesized using data derived from Apollo lunar soil samples, with a particle size distribution between 0 and 1 mm, and particle diameter with an average of 63 μm . The bulk density of LMS-1 was measured as 1.56 g/cm^3 . Since lunar regolith is a complex mixture of oxides, the LMS-1 simulant is well suited for investigating metal extraction. Its composition is dominated by major oxide phases, such as silicon, aluminum, titanium, and iron oxides, which are of primary interest in these investigations [20,22].

2.2. Electrochemical measurements

A laboratory-designed airtight Pyrex electrochemical cell with a single-compartment configuration was employed. The cell was fitted with a sealed lid containing openings for the electrodes, thermocouple, and gas inlet/outlet connections. Electrochemical investigations were conducted in the supernatant layer of the electrolyte using a three-electrode system. A copper electrode (Cu , 99.99%, Thermo Fisher Scientific, Oxford, UK,) served as the working electrode during electrochemical measurements, while a platinum (Pt , 99.99%, 4.5 cm^2) acted as the counter electrode and as the quasi-reference electrode (Pt wire 2mm diameter). Platinum materials were supplied by Sigma-Aldrich. Prior to each experiment, Cu and Pt electrodes were mechanically polished and chemically etched according to previously reported procedures [23,24]. Dissolved oxygen was eliminated from the system by argon purging before measurements, and a constant argon flow rate of 1 L min^{-1} was

maintained throughout the experiments. Electrochemical measurements and electrolysis were performed using a Gamry Instruments Interface 1010 Epotentiostat/galvanostat system. Cyclic voltammetry (CV) and square-wave voltammetry (SWV) were applied to record voltammograms in the supernatant layer of the ethaline electrolyte containing dissolved regolith-derived ions. Electrodeposition experiments were performed in a potentiostatic mode. Because these data are reported for the first time, the electrochemical experiments and ICP-OES analyses were repeated to assess the reproducibility of the results and quantify experimental variability.

2.3. Analysis experiments

Concentrations of dissolved metals in the DES supernatant, as well as those present in the

cathodic deposits, were quantified by inductively coupled plasma optical emission spectrometry (ICP-OES; SpectroSpectrogreen TL-FMT 46, SPECTRO Analytical Instruments, AMETEK Group, Berwyn, PA, USA). Prior to analysis, residual undissolved regolith particles were removed by vacuum filtration. The amounts of individual metals in the cathodic deposit were determined after dissolving the solid material in a mixture of concentrated HCl and HNO₃(1:3), after which ICP-OES analysis was performed. Surface morphology and elemental composition of the deposits were examined using scanning electron microscopy (SEM; TESCAN VEGA 3, Brno, Czech Republic) coupled with energy-dispersive spectroscopy (EDS; Oxford INCA 3.2, High Wycombe, UK). The schematic representation of the process is shown in Figure 1.

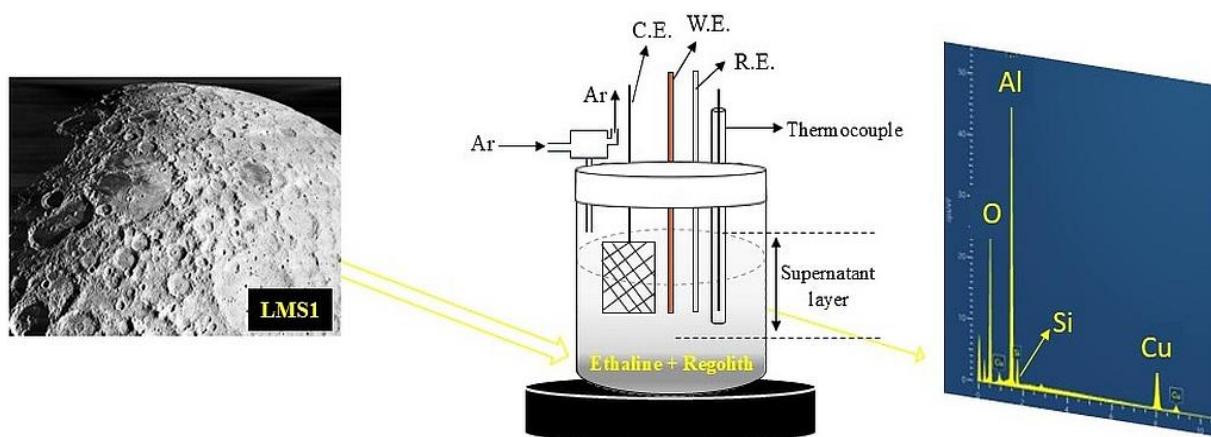


Figure 1. The schematic illustration of the process

3.0. RESULTS AND DISCUSSION

3.1. Regolith dissolution in Ethaline

To extract metals from lunar regolith by electrolysis in ethaline, the constituents of LMS-1 must be able to dissolve in the solvent. The lunar regolith simulant used in this study was composed of major metal oxides including 42.81 wt% SiO₂, 4.62wt% TiO₂, 14.13 wt% Al₂O₃, 0.21 wt% Cr₂O₃, 7.87wt% FeO_T, 18.89wt% MgO, and 0.15wt% MnO, among others which are particularly relevant in these investigations. After dissolving the regolith in ethaline, their solubility was determined by ICP-OES analysis. The dissolution effectiveness was assessed by measuring the metal concentration in the supernatant layer of the electrolyte. Once the metal concentration in the supernatant was known, the mass was converted to the corresponding oxides and compared with the metal mass ratio in the regolith. The calculation was performed according to the procedure described in [13,22].

The concentrations of the individual metals in the supernatant layer were converted to the corresponding metal oxide masses and then compared with the respective metal oxide mass fractions in the regolith. The solubility of FeO_T was calculated based on FeO as an estimate of all dissolved iron oxides, because FeO, Fe₂O₃, and Fe₃O₄ can occur simultaneously in the regolith, and their relative content distribution is unknown [2]. According to the ICP-OES analysis, the highest solubility was recorded for Cr₂O₃ with 4wt%. The solubility of Al₂O₃ was lower compared to other oxides 0.2wt%.

The measured solubilities of FeO_T, MgO and MnO were 1.2wt%, 3.7wt%, and 0.75wt% respectively. SiO₂, the major oxide component of the regolith, exhibited a solubility of 0.27wt%, indicating that the oxides composing the regolith do not dissolve completely. The solubility of TiO₂ was not determined because the obtained values of the

analytical signal were not considered reliable. Although the analysis indicates that dissolving major oxide components of the lunar regolith is feasible, the solubility rate was relatively low. Under these conditions, the individual solubility of any component of lunar regolith appears to be reduced by the combined solubility effect. Bessada et al. previously observed this effect in fluoride melts, attributing it to complex formation between oxide anions and metallic cations, and especially to the ability of free fluorine ions to form long chains by connecting different species [25]. Unlike fluoride melts, in ethaline, the chloride ligands tend to form coordinated complexes with metal ions [21]. This process reduces the concentration of free metal ions, which would generally be expected to enhance solubility. However, in this case, a plausible explanation is that in ethaline metals could also form complexes with ethylene glycol molecules $[M^{x+}(EG)_y]^{x+}$ instead of with chloride anions [26]. This behavior likely causes rearrangement of the solvent structure, and, along with competition between different metal ions for chloride ligands, leads to increased viscosity, which in turn reduces the individual solubility of the species present [26,27]. Nevertheless, these results indicate that metal recovery from this ionic liquid can be achieved at significantly lower temperatures compared to conventional processes, such as molten salt electrolysis or the FFC Cambridge process.

3.2. Electrochemical Measurements

After the dissolution of lunar regolith composed of various metal oxides, the electrolyte contains different metallic ions, and the electrochemical responses in such a system become highly complex, making it difficult to clearly identify the occurring reactions, especially the individual redox transitions. To address this, we first determined the individual reversible (equilibrium) potentials of the investigated metals in an ethaline electrolyte containing dissolved regolith. The key point is that, in this way, we can establish the order of electrochemical reduction potentials of the metals present in the electrolyte and understand their influence on the electrochemical behavior of the system. In this investigation, a platinum reference electrode was used, and a laboratory-made electrode of the selected metal served as the working electrode. The measured equilibrium potentials of metals are summarized in Table 1. The least negative potential was observed for Fe^{x+} , $E_{Fe} = -0.230$ V vs. Pt indicating that the Fe should be electrodeposited first. The equilibrium potentials of Cr, $E_{Cr} = -0.303$ V vs. Pt, and Si, $E_{Si} = -0.446$ V

vs. Pt, follow that of the Fe equilibrium potential, suggesting that the potential differences among these metals are very close, and their electrochemical reduction occurs more likely at very close potentials or in parallel. The equilibrium potential of Al was -0.770 V vs. Pt, implying that the electrodeposition of Al begins at slightly more negative potentials than the electrodeposition of Si(IV). The most negative potential was observed for Mg(II) $E_{Mg} = -1.517$ V vs. Pt, indicating the wide potential window to reach the onset for Fe, Cr, Si and Al electrodeposition. Under these conditions, the observed trend of equilibrium potentials aligns with the standard electrode potentials (vs. NHE) of the investigated metals [28]. Theoretically, metals can be obtained at the cathode from lunar regolith by electrolysis from ethaline in the following order: metallic Fe, Cr, Si, Ti, Al, and Mg. This proposition is likely valid because metal deposition begins at a potential very close to zero overpotential. Since the equilibrium potential for Mn was not determined in this case, it cannot be concluded that Mn deposition takes place.

Table 1. The equilibrium potentials of selected metals in ethaline at 70 °C measured vs. Pt, reference electrode.

Metal	Measured potential / V vs. Pt
Fe	-0.230
Cr	-0.303
Si	-0.446
Al	-0.770
Mg	-1.517
Mn	-

Cyclic voltammograms were obtained using a copper cathode in the ethaline supernatant layer at 70°C. As shown in Fig. 2.a), the voltammograms were recorded at different scan rates, revealing two reduction waves within the scanned potential range. It is evident that the cathodic peak potential either does not shift with increasing scan rates or becomes more pronounced. The reduction waves (I and II) are extended over a wide potential range, with both spanning more than 400 mV. These characteristics indicate that, within the defined potential window, the electrode reactions of metals dissolved in the system (Cu cathode, Pt counter and Pt reference, regolith dissolved in ethaline) involve complex, interconnected, simultaneous redox processes, and the simplified response to the applied CV does not reflect a well-defined redox transition.

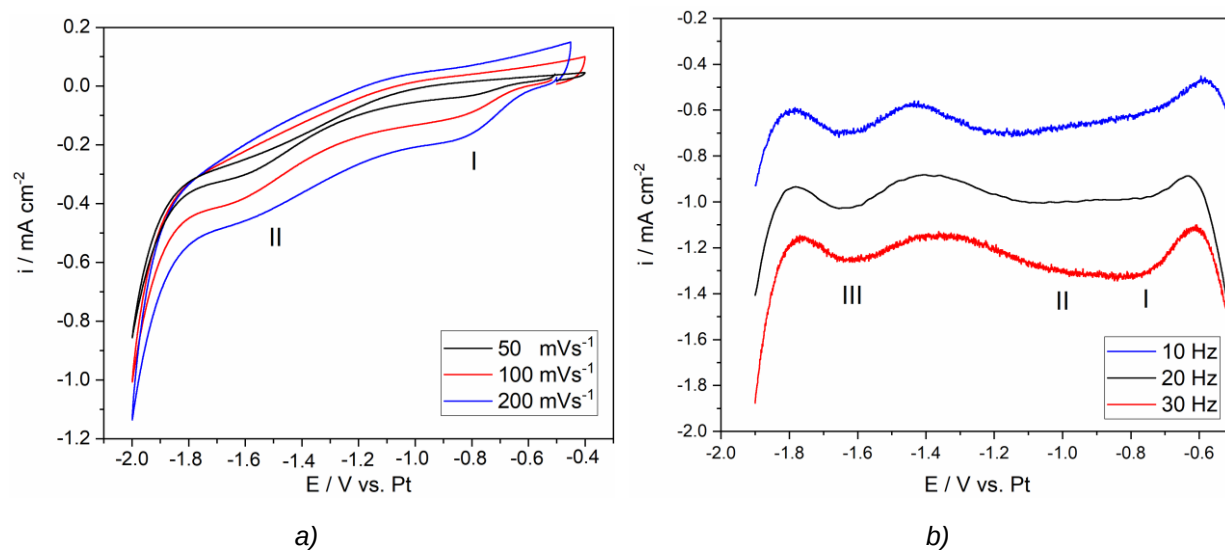


Figure 2. Voltammograms recorded on Cu working electrode in ethaline + regolith DES, 70 °C; (a) CV with different scan rates; (b) SWV with different frequencies, pulse height: 25 mV; potential step: 1 mV.

However, the anodic CV scans for the Cu substrate showed no anodic oxidation peaks of the metal electrodeposited after changing the potential sweep direction. One plausible interpretation of this behavior is that the electrochemical dissolution of the deposited metals is inhibited, and the oxidation processes are irreversible. In that case, the anodic reactions are more likely occurring at potentials beyond the electrochemical window of this electrolyte[29].

To understand the system complexity, SWV was used to clarify the underlying reduction pathway and the elementary steps, Fig. 2.b). This technique should provide more distinct signals corresponding to the selective redox reactions of metals in the electrolyte. However, the technique showed three cathodic current peaks, which are likely related to the electrodeposition of Fe, Cr, Si, and Al. Based on the results, the electrochemical reduction processes of the metals in this system are not only completely irreversible but also occur within a very narrow potential range, making it difficult to clearly distinguish the individual electrochemical reactions.

3.3. Electrolysis and deposit examination

Potentiostatic electrolysis was conducted on a Cu cathode using the supernatant layer of ethaline at potentials of -1.250 V and -1.700 V vs. Pt for two hours to determine whether metal electrodeposition occurs. The applied cathodic potential of -1.250 V vs. Pt corresponds to the end of cathodic wave (I) observed on the CV, while the second electrolysis was performed at the potential corresponding to the end of cathodic wave (II) on the CV (Fig. 2.a)).

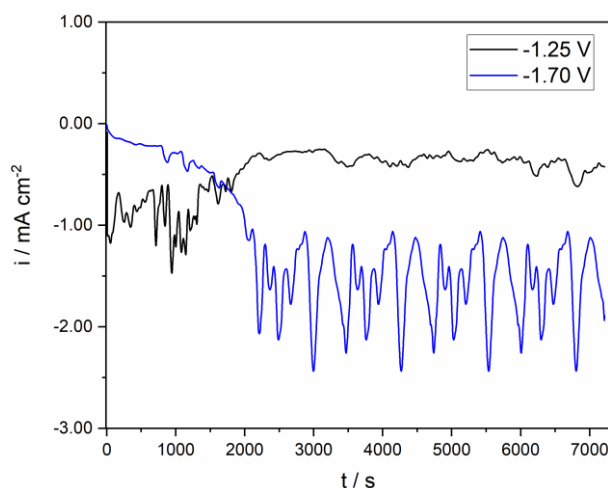


Figure 3. Potentiostatic deposition recorded on Cu WE in ethaline + regolith DES, 70 °C at different cathodic potentials for 2 h

As shown in Fig. 3, the typical relationship between current density and time during electrolysis at potentials of -1.250 V and -1.700 V vs. Pt for 2 h was recorded. According to the curve, electrolysis at -1.250 V was performed under relatively steady conditions, with a current density of approximately -0.6 mA cm^{-2} . During the electrolysis at higher overpotentials at -1.700 V vs. Pt the current density initially increased continuously for approximately 2000 s and then exhibited repeated increases and decreases. This behavior may be attributed to changes of the electrode surface during prolonged deposition time, depletion of electroactive ions near the electrode, or other time-dependent processes [30]. Nevertheless, the average current density during

the electrolysis at -1.700 V vs. Pt was -1.5 mA cm^{-2} . Based on these findings, by adjusting the electrode potential, selective metal recovery from regolith is envisaged.

Results analyzed by SEM/EDS, as shown in Fig. 4, indicate that the cathode deposit obtained by potentiostatic electrolysis for 2 hours at -1.250

V vs. Pt consists of the Cu substrate (80.9wt%), O (13.8wt%), Si (4.2 wt%), and Cl (1.1 wt%). The SEM image reveals that the deposit is randomly oriented on the working electrode substrate, as shown in Figs. 4.a) and 4.b). At higher magnification, Fig. 4.b) shows that the deposit does not adhere firmly to the substrate.

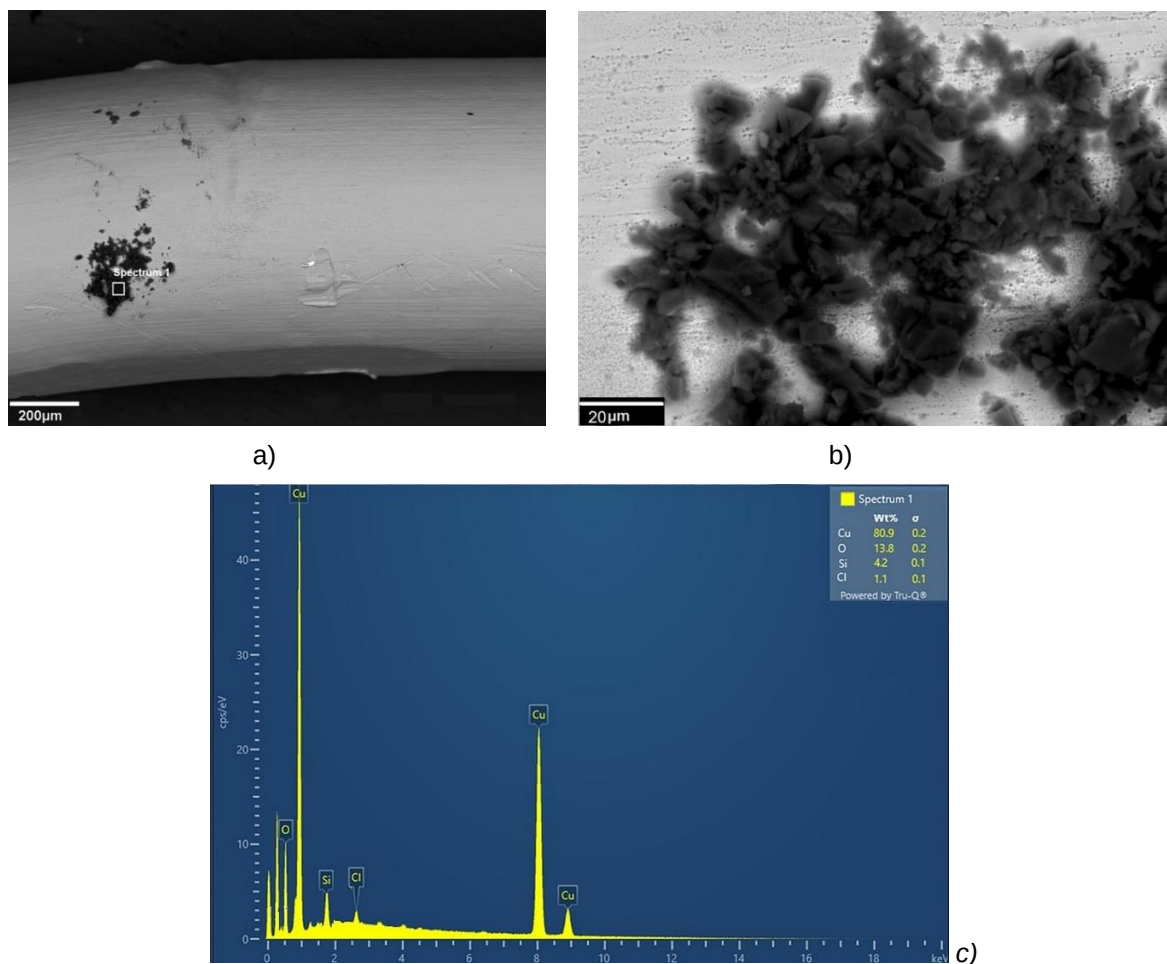


Figure 4. a-b) SEM micrographs of the obtained deposits on Cu substrate after potentiostatic deposition at -1.250 V vs. Pt for 2 h; with c) corresponding EDS analysis.

The images of the deposit after potentiostatic electrolysis at -1.700 V vs. Pt for 2 h at 70°C are shown in Fig. 5. As seen in Fig. 5.a) and b) the applied potential has a significant effect on the morphology of the deposit. The deposit is characterized by a compact layer well-adhered to the working substrate, with dark areas randomly distributed on the surface. Combined with EDS analysis, Figs. 5.c) and d) the area in the white rectangle of the deposit, Spectrum 16 is mainly composed of Cu (91.9 wt%) and Al (8.1wt%). In contrast, the dark area on the electrode surface (Spectrum 17), apart from the substrate and Al,

contains a substantial amount of electrodeposited Si (4.4wt%).

It is important to note that there are no EDS signals for other metals that should be electrodeposited, such as Cr or Fe. Characteristic EDS signals for Fe and Cr would also be expected because their onset deposition potentials are more positive than the onset deposition potential of Si, whose signal was detected. However, ICP-OES analysis of the deposit obtained at -1.250 V vs. Pt, confirmed that Fe and Cr were present, although only at a very small concentrations in the deposit. Their low concentration probably explains the

absence of characteristic Fe and Cr signals in the EDS analysis. Mg electrodeposition is not expected, as the equilibrium potential of Mg(II) is the most negative in the system. It is more likely that the substrate materials or complex structure in the electrolyte influence the onset potential for Mg(II) deposition, shifting it toward more negative potentials [26,31]. ICP-OES measurement of the deposit obtained by electrolysis at -1.700 V vs. Pt

was conducted to validate the proposed order of metal electrodeposition in ethaline containing dissolved regolith. ICP-OES analysis confirms that Cr, Fe, Si, and Al were extracted from the ethaline electrolyte after regolith dissolution. These findings support the proposition that measured equilibrium potentials of metals can be reliably used for their potential extraction from regolith using ethaline.

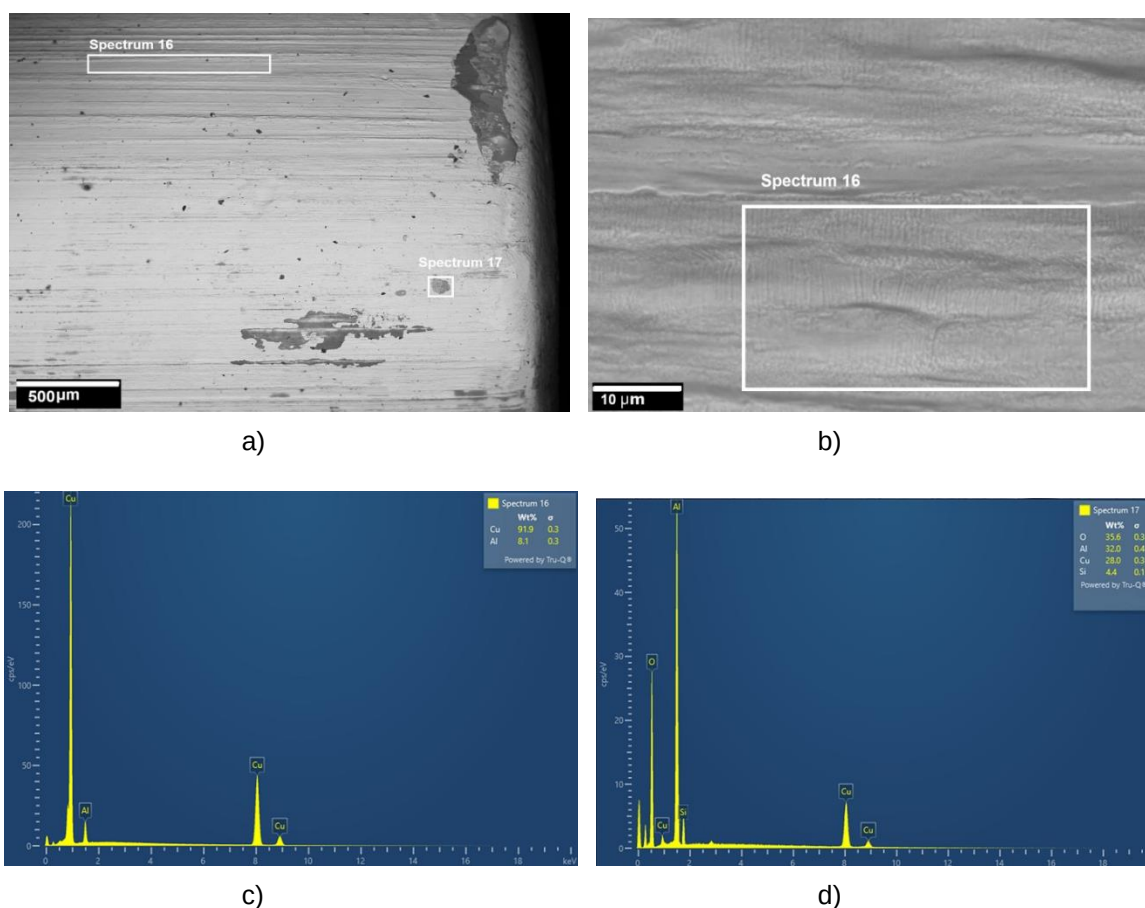


Figure 5. a-b) SEM micrographs of the obtained deposits on Cu substrate after potentiostatic deposition at -1.700 V vs. Pt for 2 h; with (c-d) corresponding EDS analysis

4.0. CONCLUSION

This study examined the dissolution of lunar regolith in ethaline and the recovery of its constituent metals by potentiostatic electrolysis. The approach revealed dissolution of metal oxides from lunar regolith, and, for the first time, ICP-OES analysis confirmed that the major metal oxides present in the regolith can be dissolved in ethaline. The order of metals equilibrium potentials (V vs. Pt) in an electrolyte containing dissolved lunar regolith was determined as $\text{Mg} > \text{Al} > \text{Si} > \text{Cr} > \text{Fe}$, which allows us to propose the order of metal electrodeposition on the cathode. Cyclic voltammetry and square-wave voltammetry

revealed multiple multivalent redox processes occurring at rather close potentials, resulting in overlapping reduction signals that made it challenging to identify the onset potential for metal deposition. Potential-controlled electrolysis experiments demonstrated that selective metal recovery from the DES-regolith system is feasible. By adjusting the electrode potential, and after analyzing the cathode product by SEM, EDS, and ICP-OES, we confirmed that the deposit was mainly composed of Al and Si. Si was recovered at more positive deposition overpotentials, whereas Al was deposited at more negative potentials. These findings provide a significant alternative for metal recovery from lunar regolith.

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IZVOD

OPORAVAK Al I Si IZ LUNARNOG REGOLITA ELEKTROHEMIJSKIM TALOŽENJEM NA NISKOJ TEMPERATURI IZ ETALINA

U ovoj studiji ispitani su rastvaranje i elektrohemijski oporavak metala iz simulatora regolita korišćenjem dubokog eutektičkog rastvarača (DES) na bazi holin hlorida i elektrohemijski oporavak metala iz tako pripremljenog elektrolita. Po prvi put, dizajnirani lunarni regolit koji predstavlja zemljište Lunar Mare (LMS-1) rastvoren je u etalinu (holin hlorid:etilen glikol, ChCl:EG), a rastvorljivost glavnih oksidnih komponenti regolita određena je korišćenjem induktivno spregnute plazma-optičke emisije spektrometrije (ICP-OES). Elektrohemijska redukcija rastvorenih metalnih vrsta u sistemu etalin-regolit uključivala je multivalentne redoks prelaze, koji su se dešavali pri veoma bliskim potencijalima ili istovremeno. Na osnovu elektrohemijskih merenja i određenih ravnotežnih potencijala metala, odabrani su parametri za elektrolizu, a oporavak metala je postignut podešavanjem potencijala elektroda. Kao rezultat toga, formira se naslaga sastavljena od Al, Cr, Fe i Si pri potencijalu od $-1,750\text{ V}$ u odnosu na Pt, dok se pri pozitivnijem potencijalu naslaga sastojala uglavnom od Si, prema EDS i ICP-OES analizi. Rezultati ukazuju na potencijal korišćenja DES-ova za rastvaranje lunarnog regolita i kontrolisano elektrohemijsko oslobađanje metala, uvodeći ranije neistraženu strategiju za preradu vanzemaljskih resursa.

Ključne reči: Lunarni regolit, Etalin, Rastvaranje regolita u DES-u, Ekstrakcija metala iz regolita

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