

# Towards A Green and Sustainable Electropolishing Technology: Feasibility Studies on the Application of Choline Chloride-based Solvents for Electropolishing Aluminum

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**Abstract** – *In this study, electropolishing of aluminum was performed using ethaline, an ionic liquid mixture of choline chloride and ethylene glycol, as polishing electrolyte. Polishing was conducted using a two-electrode system at electrode potentials of 4.5 V and 6.0 V. Electropolishing removed pre-treatment artefacts such as scratches and yielded a smooth aluminum surface. Surface analysis revealed the presence of nodular or hemispherical features in the electropolished metal. Surface roughness measurements indicate optimum electropolishing time of 25 minutes and 15 minutes at potentials of 4.3 V and 6.0 V, respectively. Ethaline is a promising ionic liquid electrolyte for electropolishing aluminum.*

*Keywords* – *electropolishing, deep eutectic solvent, aluminum, choline chloride*

## I. INTRODUCTION

Aluminum (Al) is an important technological metal due to its strength, lightweight, corrosion-resistance, non-toxicity, ductility, and malleability [1, 2]. These properties allow Al to exhibit a high degree of stability, long service lifetime, good machinability, and excellent formability [2, 3]. It is used in a variety of applications including structural (e.g. window frames), commercial (e.g. cans, foils), household (e.g. pans, kitchen utensils), and transport and aerospace (e.g. lightweight panel structures) [1, 2].

One particular Al alloy of importance is 6061 (UNS designation: A96061). 6061 is a precipitation-hardened Al (95.8 wt%) alloy that contains magnesium (0.8-1.2 wt%) and silicon (0.4-0.8 wt%) as major alloying element [4, 5]. Minor impurities include iron, copper, chromium, zinc, titanium and manganese [5]. 6061's tensile strength can range from 205 to 290 MPa, while % elongation can be from 4 to 18% [5]. It is one of the most common of the Al alloys for general use, and finds use in automotive (e.g. chassis), aerospace (wings, fuselage), marine (yacht, boat construction) and household (food packaging, bike frames) applications [2]. The microstructure is a two-phase system consisting of Al-rich  $\alpha$  phase with finely dispersed  $Mg_2Si$  serving as the precipitation-hardening phase [6].

The demand for Al and its alloys with unique surface properties such as high glossiness and homogeneity has significantly increased within the past decades due to its importance in

high technology industries and precision engineering. Electropolishing is considered one of the most popular approach in imparting a highly polished surface to aluminum [7]. Aside from decorative purposes, electropolishing can increase a metal's corrosion resistance as a direct result of the ultra-smooth surface produced in this process.

Electropolishing is the controlled dissolution of a metal surface by the passage of electric current at a particular potential in an electrolytic medium, wherein the material is removed from the metallic workpiece to polish, deburr, or passivate [7, 8]. It reduces the roughness of metal through preferential dissolution in high current density regions, leaving a smooth and reflective metal surface [8, 9]. In contrast with mechanical polishing, this process does not introduce mechanical and thermal stresses in the surface and allows the material to preserve its identity and composition.

Electropolishing traditionally uses strong concentrated acids such as nitric acid, perchloric acid, phosphoric acid, and sulfuric acid to deburr, brighten, and polish metal workpieces electrochemically [7, 10-16]. Several problems arise from the use of these corrosive liquids. One example is extensive gassing, which occurs in acidic mixtures as a result of the reduction of hydronium ions into hydrogen gas. The bubbles produced temporarily act as an insulating material that impedes electron flow resulting in a slow process. Additionally, since electropolishing is performed at high temperatures [9, 16-18], then electrolytes are rendered more aggressive. These chemicals not only pose a serious threat to process infrastructures (via corrosion), but, more seriously, endanger humans and harms the environment [16, 18]. Hence, future studies are geared towards looking for alternative technologies that could do aluminum polishing using cost-effective, non-toxic, and biodegradable electrolytes.

One promising approach is the use of a novel and unique material called ionic liquids (ILs) [19-22]. Ionic liquids consist primarily of salts in liquid form [23]. Due to their unique properties, ILs have found use in different applications including gas and wastewater treatment, energy, and surface modification processes such as electroplating, electroless plating, and electropolishing [23-25].

Deep eutectic solvents (DES), a type of ILs, has recently gained research interest, particularly as an electrolyte that could replace traditional aqueous solution for electrochemical processes. Some of the advantages of using DES in electrochemical processes include (i) wide potential window, (ii) high solubility of metal salts, (iii) avoidance of water/metal chemistry, and (iv) high conductivity [24, 26]. One of the most critical advantages of DES is that it is non-toxic and biodegradable [27]. This implies that DES offer a truly clean and 'green' alternative to the traditional electrolytes that consist of environmentally harmful aqueous ('water-based') solutions (e.g. Cr(VI), cyanide, acids and alkaline solutions) [25-27].

Saleem [28] successfully electropolished aluminum using different types of first-generation ionic liquids and concluded positively on the viability of ILs technology as an alternative route for aluminum electropolishing. Recently, several works have reported on the application of DES in the electropolishing of Ni and its alloys and cobalt [29, 30], and stainless steels [19, 31, 32]. However, only few studies have reported on the application of DES on

aluminum electropolishing [33-35]. Hou et al. [33] used chloroaluminate-based IL and they obtained electropolishing results very similar to those obtained using traditional strong acid solutions. They further observed the occurrence of an optimum electropolishing time, beyond which surface roughness increases. Kityk et al. [34] investigated Al electropolishing using choline chloride-based DES. They reported that increasing the electrode potential during the anodic treatment result in a decrease in surface roughness and improvements in the surface brightness. Clearly, more studies are necessary to ascertain the possibility of commercial adoption of IL technology for Al electropolishing.

In this paper, we report on the use of ethaline, which is a mixture of choline chloride and ethylene glycol, as an electrolyte for Al electropolishing. The influence of electropolishing parameters, such as applied voltage and electropolishing time, on the ensuing surface qualities of the metal was evaluated. The work will also describe a surface feature that has not been previously reported for electropolished Al and recommends future areas of study.

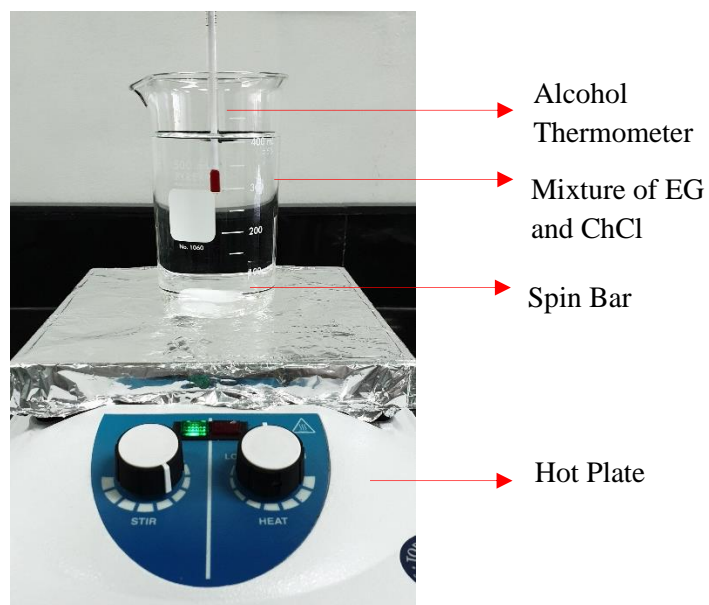
## II. METHODOLOGY

### 2.1 Materials and Chemicals

As-received choline chloride ( $\text{ChCl}$ ;  $(\text{CH}_3)_3\text{NCH}_2\text{CH}_2\text{OH}^+[\text{Cl}^-]$ ) (Sigma Aldrich, 98%) and ethylene glycol (EG,  $(\text{CH}_2\text{OH})_2$ ) (Technical Grade) were used to prepare ethaline. The electrodes used in the experiments were 6061 grade Al as working electrode, stainless steel (SS 316) as counter electrode, and platinum rod (Pt, 99.99% pure) as reference electrode. The working electrode has an exposed surface area of  $1 \text{ cm}^2$ . The Al substrates were polished using 800 grit SiC papers to ensure that all substrates have the same initial surface roughness. The substrates were rinsed with deionized water and kept in a desiccator prior to use.

### 2.2 Ethaline synthesis

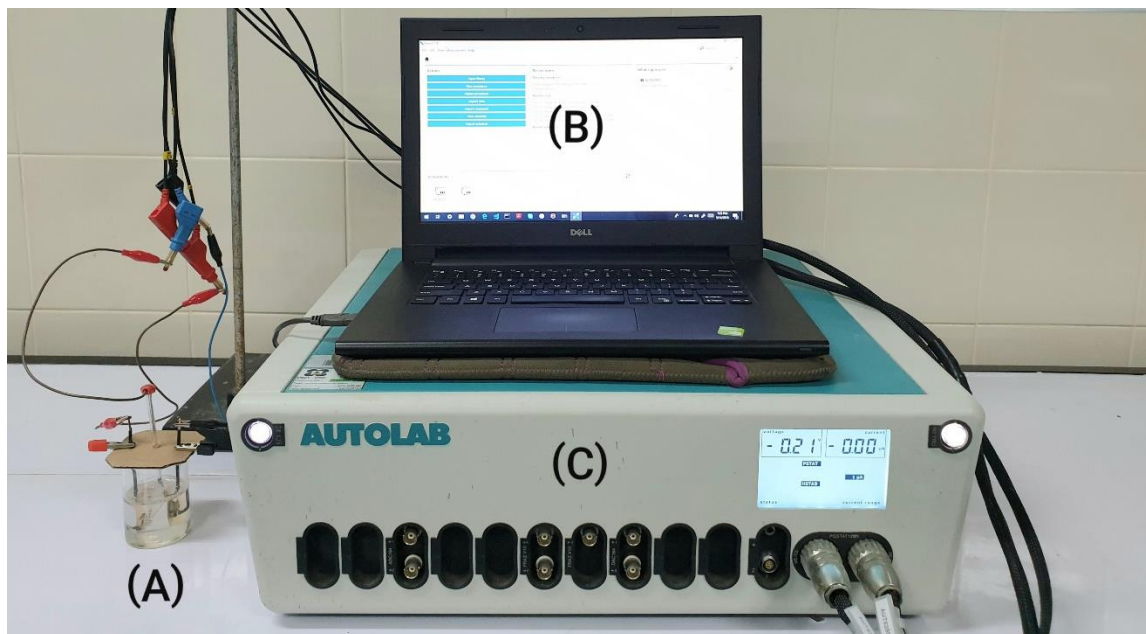
Ethaline was synthesized by adding the proper amounts of ChCl to EG to create a 1:2 molar ratio mixture. The mixture was heated at  $70\text{-}80^\circ\text{C}$  and constantly stirred until a colorless and homogenous solution was obtained, as shown in Figure 1. The synthesized solvent was allowed to cool to room temperature and stored in a tightly sealed glass container to prevent moisture absorption.



**Figure 1.** Experimental set-up for ethaline synthesis.

### 2.3 Electrochemical evaluation

Linear sweep voltammetry (LSV) was performed using a potentiostat (Autolab Potentiostat Model AUT 83997) and a three-electrode apparatus, as shown in Figure 2. The three-electrode apparatus consists of Al as the working electrode, SS as the counter electrode, and Pt as the reference electrode. Fifty (50) mL of ethaline was placed in a beaker, and the three electrodes were subsequently immersed in the solvent. The electrodes were attached to the potentiostat and the voltammetry test was initialized using the Nova 2.1 software. LSV was done in potentiostatic mode, at a potential range of 0V to 5V, and a scan rate of 20 mV/s. Three replicates were performed, and the corresponding data points were averaged.



**Figure 2.** Components of the voltammetry /electropolishing set-up: (A) electrodes immersed in ethaline, (B) laptop Computer with Nova 2.1 Software, and (C) potentiostat.

#### 2.4 Electropolishing experiment

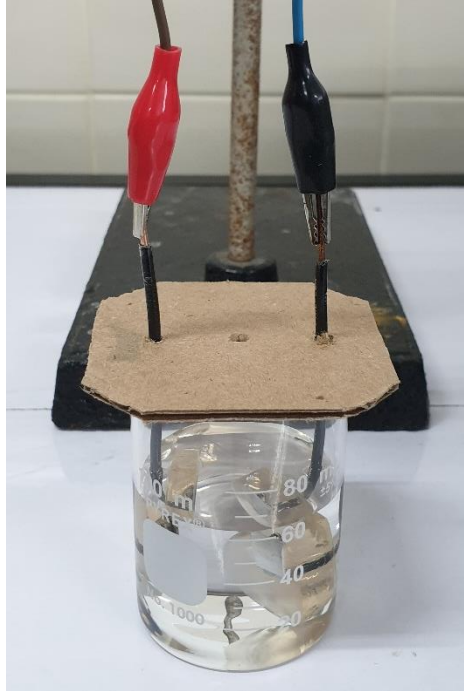
Electropolishing of Al was conducted following the settings for the applied voltage and time shown in Table 1. The applied voltage was based on the results of LSV analysis. The anticipated plateau region characteristic of electropolishing occurred between 4.0V to 4.5V, and the electropolishing potential of 4.3 V was chosen from this potential range. In contrast, the high potential setting of 6.0V was selected to determine the upper limits of the electropolishing operation.

The ranged of values used for the electropolishing time was based on the study by Hou et al. [35] where it was suggested that the optimum electropolishing time for Al in an analogous solvent was 25 minutes.

**Table 1.** Experimental settings for the operating voltage and electropolishing time.

Sample	Applied Voltage (V)	Electropolishing Time (mins)
Control	0	0
L15	4.3 V	15
L25		25
L35		35
H15	6.0	15
H25		25
H35		35

A two-electrode system was used in the electropolishing experiments, as shown in Figure 3. The Al plate was used as the cathode, and the SS plate was used as the anode.



**Figure 3.** Two-Electrode System: SS as Working Electrode and Al as Counter Electrode

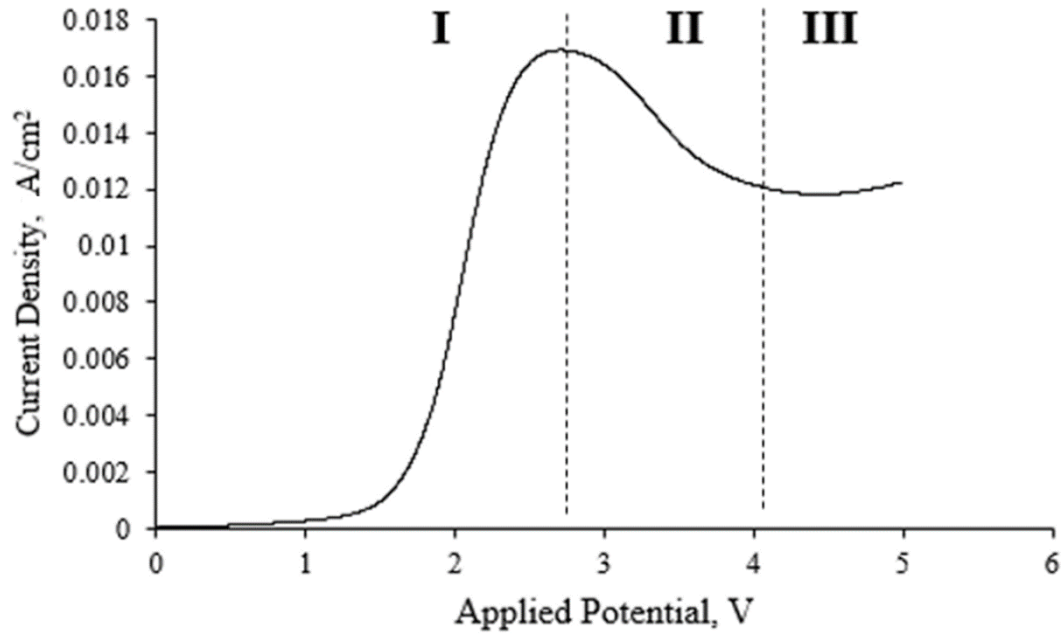
### 2.5 Characterization

The surface features of the control (unpolished) and electropolished Al specimens were inspected using a high-powered compound microscope (Carl Zeiss Axios Scope A1). Surface roughness was measured using atomic force microscopy (AFM, Park Systems NX10).

## III. RESULTS

### 3.1 Electrochemical characterization of ethaline

The anodic polarization curve of Al in ethaline is shown in Figure 4. The three regions characteristic of electropolishing systems in aqueous solutions were also observed in the ethaline system; namely, etching region (*I*: 0-2.7V), passivation region (*II*: 2.7-4.0V), and polishing region (*III*: 4.0-5.0V). The maximum current density, which occurs at the peak between the etching and passivation region was  $16.9 \text{ mA cm}^{-2}$  while the limiting current density of the polishing process was  $12 \text{ mA cm}^{-2}$ . More importantly, the existence of the polishing region (region III) in the polarization curve indicates the possibility of conducting Al electropolishing in ethaline.

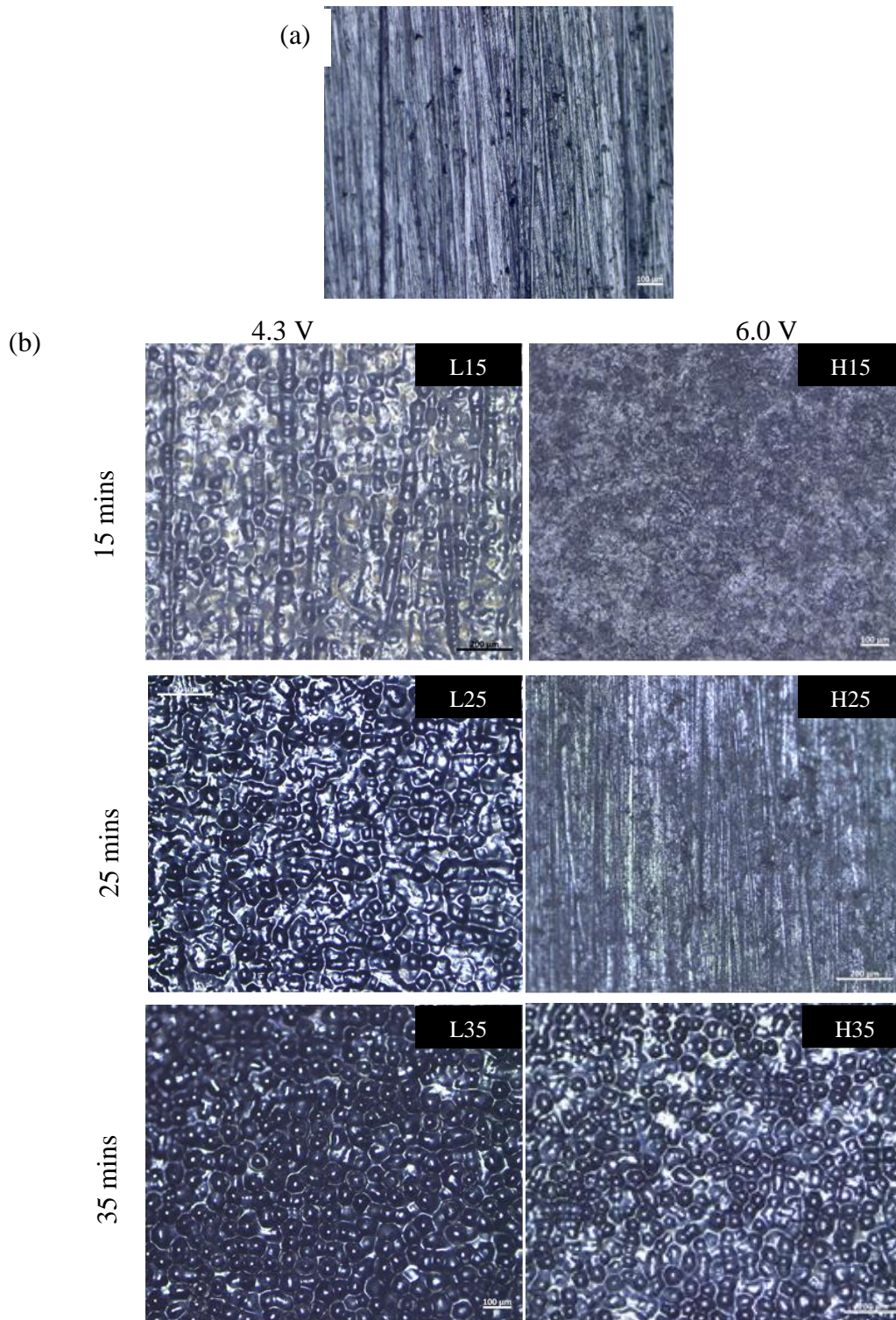


**Figure 4.** Linear sweep voltammogram of Al 6061 vs Pt in ethaline.

### 3.2 Electropolishing experiments: SEM Analysis

After electropolishing, the test specimens were analyzed using the SEM to detect changes in surface morphology. Figure 5 shows metallurgical microscope images of the unpolished Al, alongside micrographs for the specimens electropolished at different conditions.





**Figure 5.** Photomicrographs of the surface of the (a) unpolished aluminum and (b) aluminum specimens after different electropolishing treatments. Scale bar represents 100  $\mu\text{m}$ .

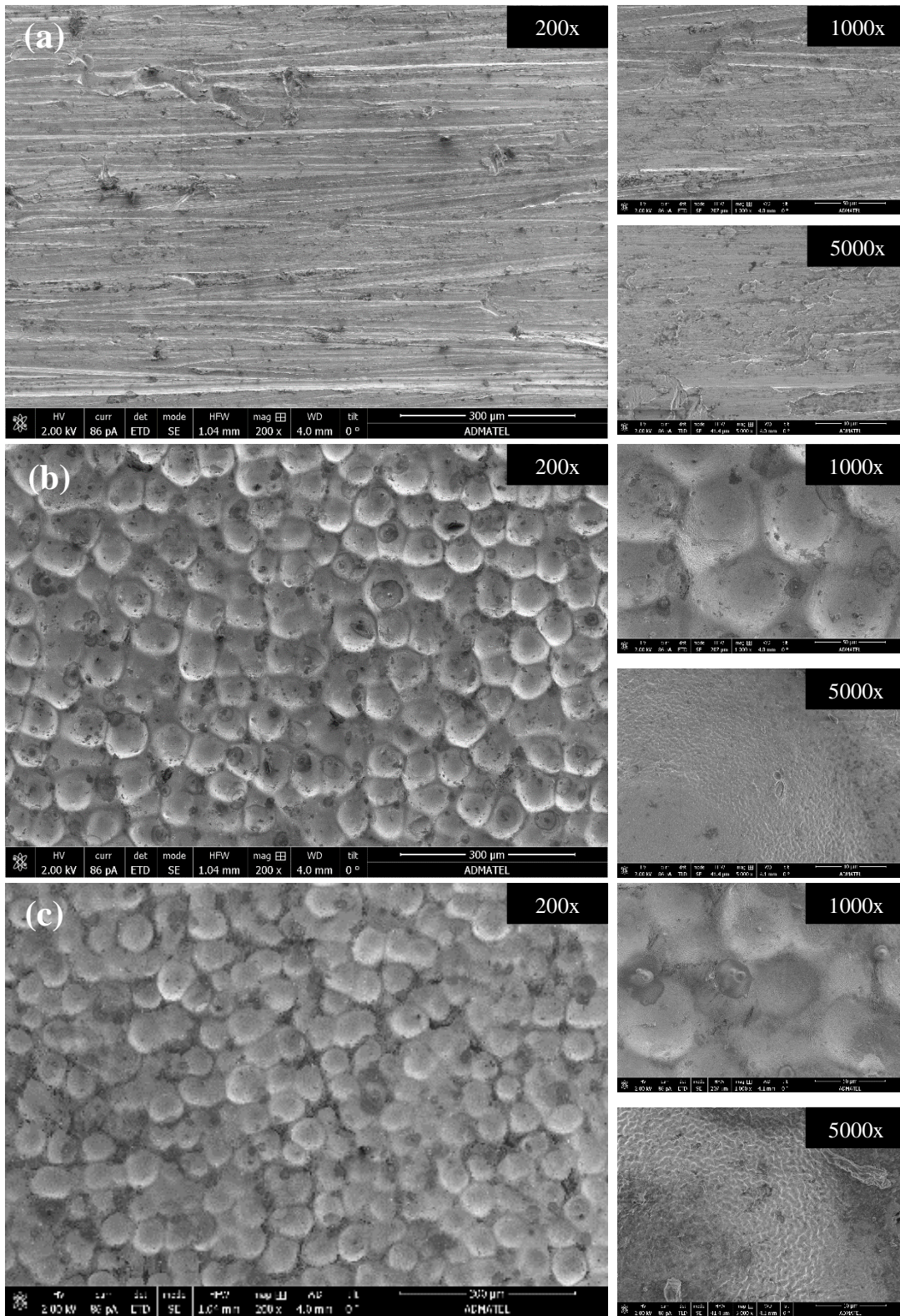


The surface of the unpolished Al exhibited scratch marks brought about by mechanical grinding, as seen Figure 5(a). Pits, indicated by dark spots, were also visible on the surface. This feature may have resulted from abrasive (silicon carbide) embedding on the Al surface. Alternatively, superficial impurities could similarly create this feature.

For the specimens polished at 4.3 V, a change in the surface quality was observed. Initial visual inspection indicates some improvement in surface shine or glossiness. A closer look at the surface revealed that the scratches from the previous surface preparation step was removed and replaced by smooth, nodular (or hemispherical) features at the surface as seen in Figure 5(b). This surface morphology has not been previously reported for electropolished Al. The diameter of the nodules ranges from 20 to 80  $\mu\text{m}$ . With increasing polishing time (i.e., from L15 to L35), the nodules become more prominent and well-defined, this observation confirms that electropolishing is indeed feasible at 4.3V, as predicted by the by the LSV curve.

For the specimens polished at 6.0V, the features came out differently compared to those seen in the specimens polished at the lower potential (i.e. 4.3 V). At 15 min electropolishing time (H15), the surface appears amorphous, with no definite features standing out. At 25 min (H25), light scratches were made visible. At 35 minutes (H35), the surface appeared polished and the same nodular structure that was previously seen in the specimens polished at 4.3 V.

Results indicate that only the Al electropolished at 6.0V and 35mins (H35) exhibited a polishing effect. H15 (6.0V, 15mins) and H25 (6.0V, 25mins), although possessing surface features different from the control, did not exhibit polished surface characterized by the presence of smooth grains. It possible that only passivation instead of material dissolution occurred for these specimens.

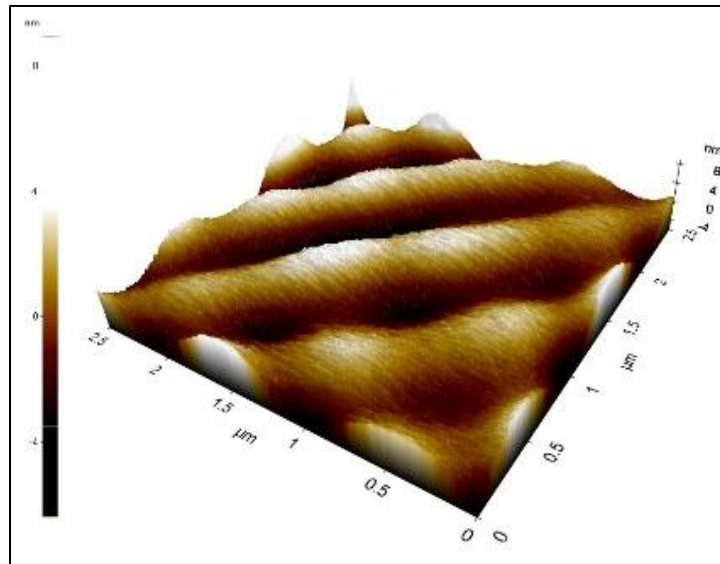


**Figure 6.** SEM images (200x, 1000x, and 5000x magnification) of the (a) unpolished Al, and Al specimens electropolished at (b) 4.3V for 35 mins (L35), and at (c) 6.0V for 35mins (H35).

Figure 6 presents the SEM images of the unpolished specimen, and the Al specimens electropolished at 4.3V for 35 mins (specimen L35), and at 6.0V for 35mins (specimen H35). In the control, as seen in Figure 6(a), only scratches from the previous surface preparation treatment are visible. In contrast, the two electropolished specimens possess very similar morphology containing the nodular or hemispherical surface features. Specimen L35 appears to have a more defined structure than H35. Additionally, at higher magnifications (5000X) presented in Figure 6(b) and (c), the surface of H35 contained pits and was found to be rougher than L35.

### 3.3 Electropolishing experiments: SEM Analysis

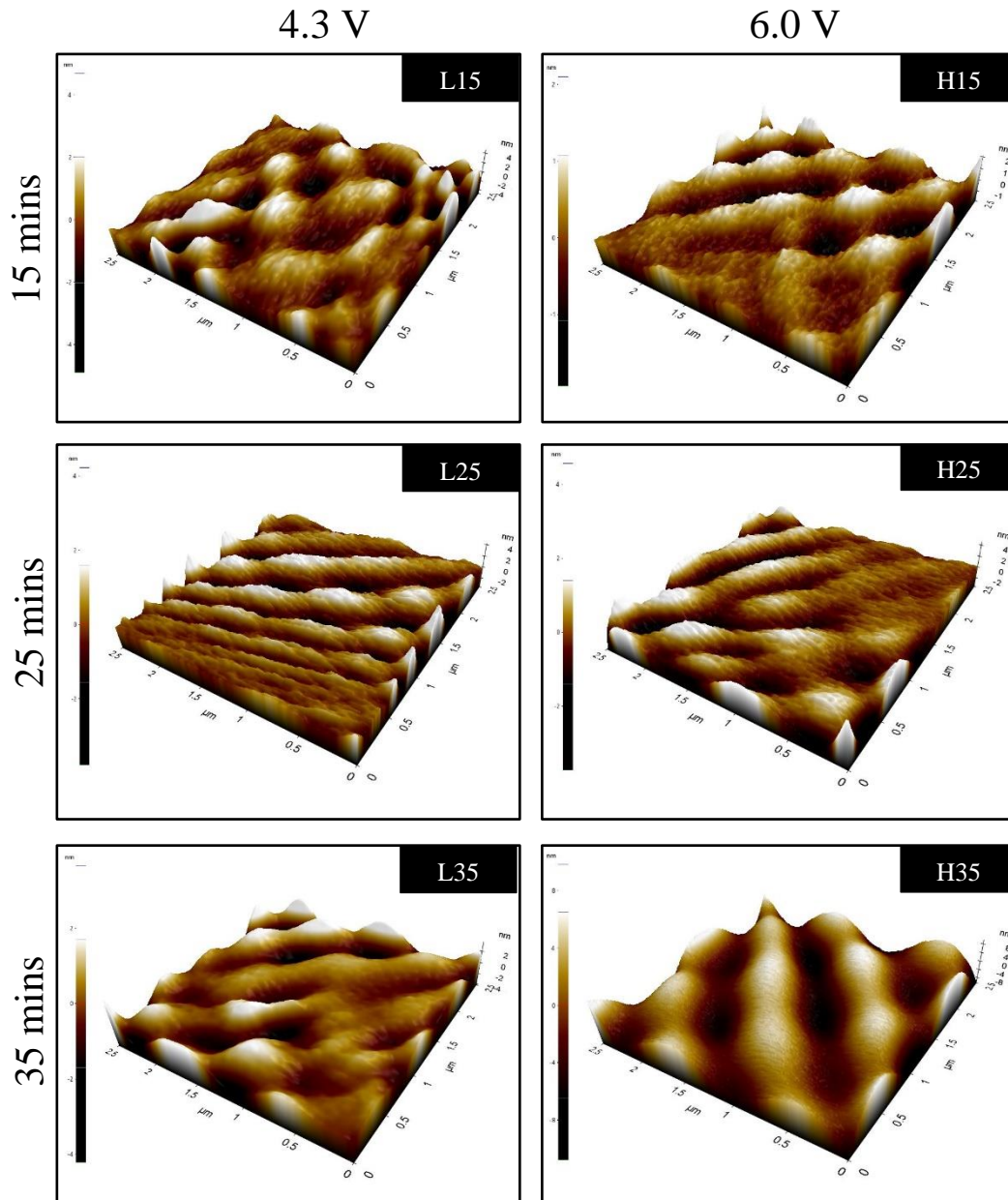
AFM analysis was conducted to gather quantitative information of the specimen's surface roughness. Figure 7 shows the results of AFM imaging of the control or the unpolished specimen. The surface exhibited alternating, parallel ridge-like structures. This feature likely relates to the surface scratches created by the initial mechanical grinding.



**Figure 7.** AFM image of the unpolished aluminum.

Figure 8 presents the AFM images of the Al electropolished at different settings. Remnants of the ridge-like features seen in the control are still evident in these specimens. However, the surface appears relatively smoothed and the presence of hemispherical protrusions are also noticeable, which agrees with the nodular features observed under the SEM.

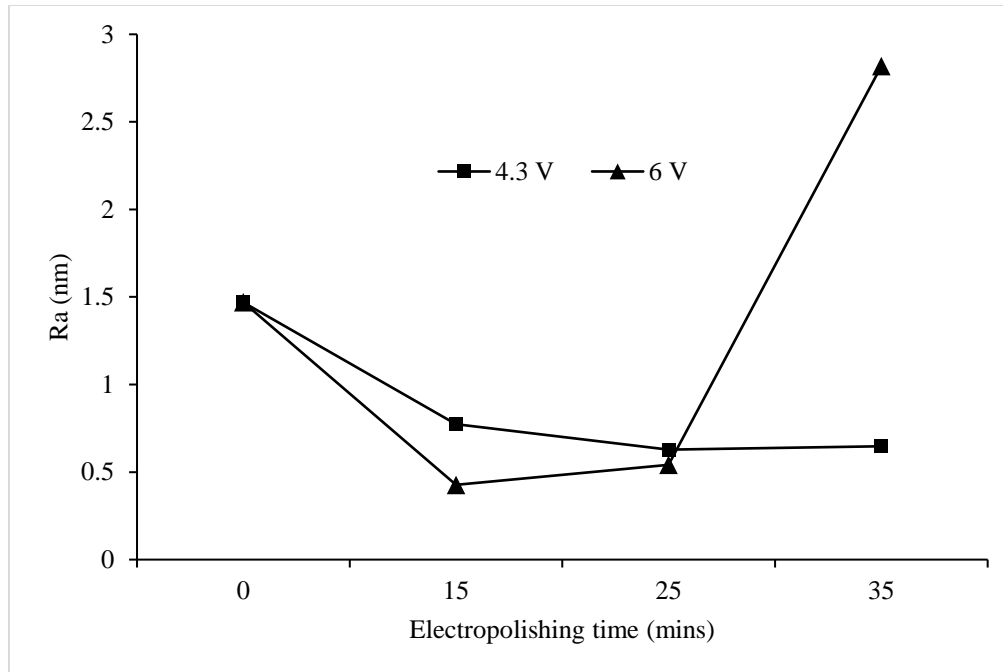




**Figure 8.** AFM images of Al polished at different test conditions. Scanned area is  $2.5 \times 2.5 \mu\text{m}^2$ .

Figure 9 shows the plot of the average roughness (Ra) versus electropolishing time for the specimens polished at 6.0 and 4.3 V. It can be observed that the lowest surface roughness is obtained at 25 minutes for 4.3V polishing time and 15 minutes for 6.0V polishing time. In both voltage settings, increasing the processing time decreases the roughness until achieving the optimum electropolishing time. Beyond the optimum time, Ra increases.

Interestingly, the roughness of the Al specimens electropolished at 6V were lower than that polished at 4.3 V at the 15- and 25-min electropolishing times. However, the initial surface analysis of these specimens indicated no polishing effect on these specimens.



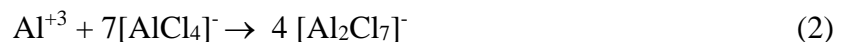
**Figure 9.** The plot of surface roughness (Ra) versus electropolishing time (mins) as a function of polishing potential.

#### IV. DISCUSSION

The results of this study suggest the feasibility of using the DES ethaline for Al electropolishing. The anodic LSV test results clearly indicated a range of potential at which polishing can be conducted, and the subsequent experiments confirm successful electropolishing at such potentials after certain polishing times. The LSV also suggests that dissolution occurs in a single step process, consistent with Kityk et al. [34]:



This reaction likely occurs simultaneous with the breakdown of the surface oxide [33]. Abbot et al. [36] further suggests that the  $\text{Al}^{+3}$  may also react with some  $\text{AlCl}_4^-$  if present in the DES:



Interestingly, the observed current density at the polishing potential range is relatively low, i.e.  $1.2 \text{ Adm}^{-2}$ . For example, Hou et al. [33] used a current density of  $2.85 \text{ Adm}^{-2}$  to  $4.28$

$\text{Adm}^{-2}$  for Al electropolishing in  $\text{AlCl}_3$ /Trimethylamine hydrochloride DES. This low current density suggests poor conductivity of the electrolyte. A low current density is unwanted as it results in low electropolishing rate and increases process time. Additives may be used in the future to increase electrolyte conductivity and improve polishing current.

With regard to the occurrence of an optimum polishing time, Hou et al. [33] observed similar trends, i.e. the surface roughness (measured as Ra) increases as the polishing time until achieving the optimum setting, then eventually decreasing thereafter. Also, the Ra values obtained in the current study, as shown in Figure 8, are comparable to the Ra range of 0.947 to 1.01 achieved by Hou et al. [33].

As mentioned, the occurrence of the relatively macroscopic nodular or hemispherical surface feature in the electropolished Al has not been previously reported.

Bandyopadhyay et al. [37] reported finding different nanoscale morphologies in electropolished aluminum using an organic electrolyte consisting of perchloric acid, butyl cellosolve, ethanol and water. Different surface morphologies were produced by varying the electropolishing potential from 37 to 65 V. They described one pattern created at 65 V as ‘random hills’, which closely matches the nodular structure seen in this work. However, the difference is that nodules in the current study ranges from 20 to 80  $\mu\text{m}$  in size, while the ‘hills’ observed by Bandyopadhyay et al. were less than 100 nm in diameter. Bandyopadhyay et al. [37] explained that these shapes resulted from the preferential adsorption of organic molecules on the convex portion of the electrode due to enhanced electric fields induced by natural surface curvatures on the double-layer potential.

It is likely that the mechanism that produced the observed nodular features in the electropolished Al is a product of the interaction between the ionic liquid molecule and the double layer structure produced at the interface. As of yet, this interaction is not yet well understood, though several models have been proposed [38]. The hemispherical shape suggests localized, non-uniform polishing or etching rate, i.e. the perimeter of the hemisphere has been etched faster than its middle. This would mean that certain areas bear enhanced electric fields that encourages faster metal dissolution. Additional studies are necessary to understand this mechanism further. Future research may look at (i) the electrical double layer characteristics as a function of electrolyte composition, and (ii) the influence of surface preparation on the substrate. There is a possibility that these nodules outline Al grains and grain boundaries, and it is recommended that future work may also consider this prospect.

## V. CONCLUSION

Electrochemical polishing of Al was successfully conducted using Ethaline. Surface analysis revealed the presence of scratches on the unpolished Al electrode. After electropolishing, the aluminum surface became smooth and was replaced with nodules. The formation of the nodular structure was favored at the 4.3 V than at 6.0 V. Increasing polishing times created a more defined and prominent nodular structure. Results of AFM analysis agreed well with the results of SEM imaging, confirming ridge-like structures in the unpolished



specimens, and the occurrence of hemispherical protrusions after electropolishing. The AFM results indicated optimum electropolishing times (minimum Ra) per voltage setting, which are 25 minutes for 4.3 V and 15 minutes for 6.0 V. Non-uniform current distribution, likely due to the interaction of the ionic molecule and the electrical double layer, may explain the nodular structure formed at the surface.

## VI. ACKNOWLEDGEMENTS

The authors would like to acknowledge the assistance of Mr. John Raymund Brusas of the Sustainable Electrochemical Laboratory (SETLab) – DMMME UP Diliman. This work also acknowledges the Office of the Vice Chancellor for Research and Development (OVCRD) University of the Philippines Diliman, for funding support through the Ph.D. Incentive Award.

### Nomenclature:

<i>Symbol</i>	<i>Description</i>	<i>Units</i>
Ra	Surface roughness	nm

### *Subscripts, superscripts, and abbreviations*

AFM	Atomic Force Spectroscopy
Al	Aluminum
ChCl	Choline Chloride
Cr	Chromium
DES	Deep Eutectic Solvent
EG	Ethylene Glycol
ILs	Ionic Liquids
LSV	Linear Sweep Voltammetry
Ni	Nickel
Pt	Platinum
SEM	Scanning Electron Microscope
SS	Stainless Steel

### *Units*

$\text{Adm}^{-2}$	ampere per decimeter squared
$^{\circ}\text{C}$	Celsius
$\mu\text{m}$	micrometer
$\mu\text{m}^2$	micrometer squared
$\text{mA cm}^{-2}$	milliamperes per centimeter squared
$\text{mV/s}$	millivolts per second
mins	minutes
nm	nanometer
V	voltage

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