

# **SURFACE QUALITY AND CORROSION RESISTANCE OF 316L STAINLESS STEEL ELECTROPOLISHED USING PHOSPHORIC – SULFURIC ACIDS**

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## **Abstract**

*Electropolishing is an electrochemical surface finishing technique. It is commonly applied to equipment that requires a gleaming finish. This surface property is frequently required in 316L stainless steel (SS) medical implants. Electropolishing removes a thin layer from the metal's surface through electrochemical processes. This results in a very clean, smooth, and bright metal surface. The process parameters, such as electrolyte solution, electrical current, and electropolishing time, influence surface roughness and glossiness. The dissolution of metallic ions during the process may also affect the corrosion resistance of the treated material in addition to producing a shiny surface. This study investigated the surface glossiness, surface roughness, and corrosion of electropolished 316L SS. Electropolishing experiments on 316 SS were carried out using various H<sub>3</sub>PO<sub>4</sub> (50%) and H<sub>2</sub>SO<sub>4</sub> (32%) electrolyte solution compositions. The influences of electrolyte solution composition, electric current, and electropolishing time were studied. The results showed that increasing the H<sub>2</sub>SO<sub>4</sub> content of the mixture and electropolishing the 316L SS for a longer period of time improved the surface roughness and glossiness. Under 10 Amp electric currents, the best surface glossiness was discovered. A corrosion test revealed that the electropolishing produced a Cr and Ni-rich layer that improved the corrosion resistance of the samples.*

**Keywords:** 316L, Stainless Steel, Electropolishing, Glossiness, Corrosion

## **1. INTRODUCTION**

316L stainless steel (SS) is a low-carbon austenitic SS. This material possesses excellent corrosion properties and exhibits good chemical and mechanical stability, high mechanical strength, good machinability, and biocompatibility <sup>[1,2]</sup>. Because of its good properties, steel is widely applied for many industrial applications involving chemicals or food processing <sup>[2]</sup>. In medical devices, it is the most widely used among metallic materials <sup>[3,4]</sup>. Hence, this material is commonly used for medical devices, *i.e.*, cardiovascular stents, orthopedic and dental implants, and a variety of surgical tools such as scalpels and forceps <sup>[1,2,5-9]</sup>.

Surface quality is frequently required for a product made of SS. Smoother surfaces are considered more cleanable. This is an important characteristic of material for food processing <sup>[10-12]</sup>. Surface roughness is also critical for medical device applications <sup>[1,5,8]</sup>. Electropolishing is an effective method to provide an excellent surface for SS. It reduces micro roughness and decreases the risk of dirt adhering to the surface. This technique is also

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used for deburring, brightening, and passivating. Electropolishing provides an undisturbed-metallurgically clean surface <sup>[13]</sup>. The process also improves metal surface smoothness, appearance, surface corrosion resistance, and removal of contaminations and refinement of surface oxidation layer <sup>[14]</sup>. Compared to mechanical polishing, electropolishing produces a surface free from deformations without interfering with the crystalline network structure and superficial stresses <sup>[10]</sup>.

Two step electropolishing was attempted to electropolish 316L SS using a sulfuric acid-free electrolyte <sup>[15]</sup>. The work observed that more material removal rate was increased with low water concentration, while the surface roughness was reduced under higher water concentration. Chromium enrichment was indicated in the formed passive layers when electropolishing austenitic SS using a commercial  $H_2SO_4 + H_3PO_4$  electrolyte <sup>[16]</sup>. However, the electropolishing provided poor corrosion properties of material compared with a mechanically-ground specimen. This was claimed to be due to the presence of sulfate and phosphate in the passive layers. Addition of  $Cr_2O_3$  on  $H_2SO_4 + H_3PO_4$  electrolyte produces maximum gloss of the specimen's surface. While 14 minutes electropolishing time was found to give the maximum gloss, this study also proved that increasing the time higher would worsen the surface <sup>[17]</sup>. Electropolishing temperature that resulted in lower roughness parameters and minimized the possibility of bacterial attachment was identified <sup>[18]</sup>.

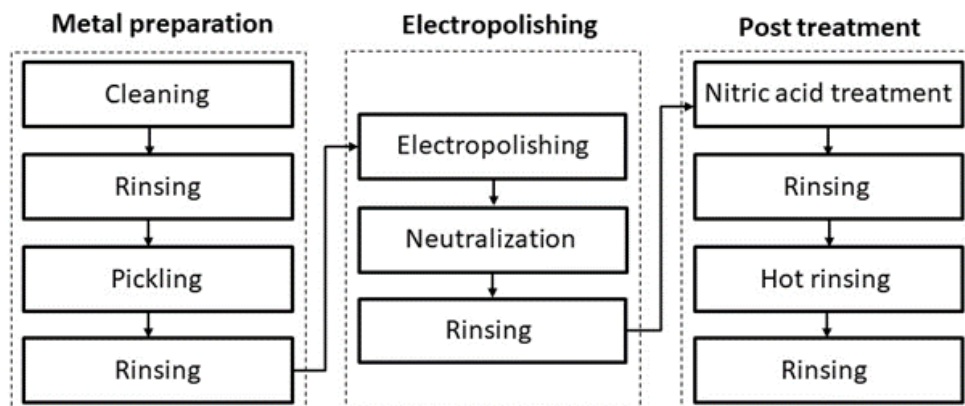
In the present work, 316L SS is electropolished using a mixture of  $H_2SO_4 + H_3PO_4$  acid as a case for orthopedic implant application of the steel. Previous works <sup>[2]</sup> employed similar liquid mixtures to evaluate the characteristics of stainless steel manufactured in different ways, i.e., thermal spray coating (TSC), additive layer manufacturing (ALM), and cast 316L stainless steel. The surface quality of the electropolished metals was evaluated based on the surface roughness produced by the process. The study was also carried out on the crystallographic structure of the surface. However, the work did not study the effect of the electrolyte composition and other electropolishing parameters. In the present work, a study of the electropolishing of 316L SS using a different mixture composition of  $H_2SO_4$  and  $H_3PO_4$  acid was carried out on the 316L SS sheet. This was a study of a case in the surface finishing process in orthopedic implant manufacturing. To provide a practical reference for electropolishing 316L SS, the influence of the electropolishing parameters on the surface quality is scrutinized. The glossiness of the surface and corrosion of the material were studied since those were important properties for the orthopedic implant product. To achieve the objective, experiments on electropolishing 316L SS were performed under different conditions of acid mixture ratio, electric current, and time. This was to study the influence of electropolishing conditions on surface glossiness, surface roughness, and corrosion resistance.

## 2. METHOD AND MATERIAL

Material for the experiments was a commercial-grade AISI 316L SS plate with a thickness of 5 mm manufactured by Huaigang Special Steel, Co., China. The steel had the brand of Qiyuan Metal, produced by Shandong Qiyuan Metal Co., China. The material was cut into approximately 40 mm X 35 mm. The electrode was a stainless-steel cylinder of 10 mm diameter and 30 mm length. The specimens were immersed in NaOH for 5 minutes and ground with 120, 240, and 360 grits of sandpaper. Each specimen was weighed before being electropolished under the determined setting condition.

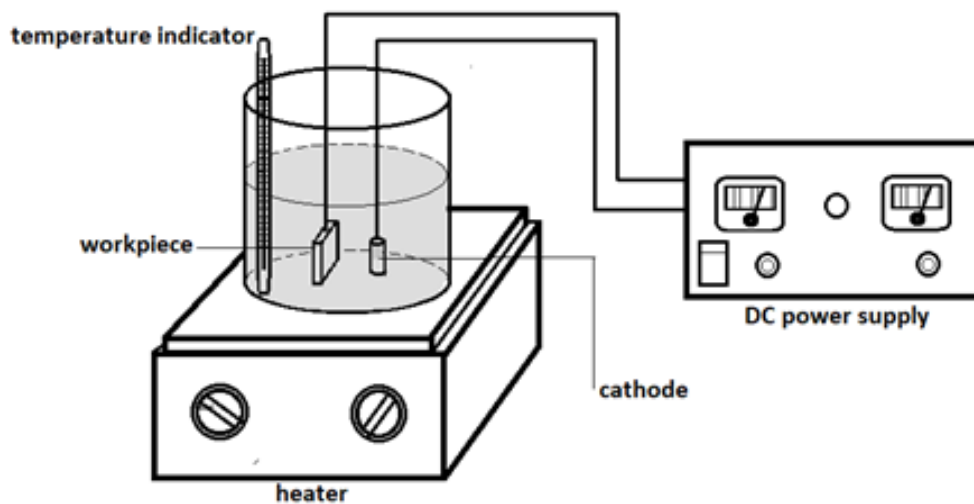
The treatment is illustrated in Figure 1. The process was initiated with pretreatment of the material. This stage was carried out to remove oil, dirt, or any other substance on the surface that may disturb the electropolishing process. It consisted of cleaning the sample using NaOH, pickling, and rinsing. Pickling was done using  $HNO_3$  (20%) and HF (5%) for 5

minutes. After the electropolishing, post-treatment was accomplished to clean all chemicals that might remain in the surface.



**Figure 1.** Stages of electropolishing process

Electropolishing was performed using  $\text{H}_3\text{PO}_4$  (50%) and  $\text{H}_2\text{SO}_4$  (32%) with a composition ratio of 1:1; 2:3; and 3:7. Table 1 presents the solutions that are used for the treatment. Setting up the electropolishing process is given in Figure 2. After the electropolishing, specimens were weighed to measure the weight loss and their surface quality was checked with BGD 512 glossmeter and MITUTOYO SJ201 surface roughness tester.



**Figure 2.** Experimental set up of electropolishing

**Table 1.** Chemical solutions for electropolishing and pre/post treatment

<i>Treatment</i>	<i>Liquid</i>	<i>Conc.</i>
Cleansing	NaOH	25%
Rinsing	Cold water	-
Pickling	$\text{HNO}_3$	20%
	HF	5%
Rinsing	Cold water	-
Electropolishing	$\text{H}_2\text{SO}_4$ 1 : 1; 2 : 3; 3 : 7	50%

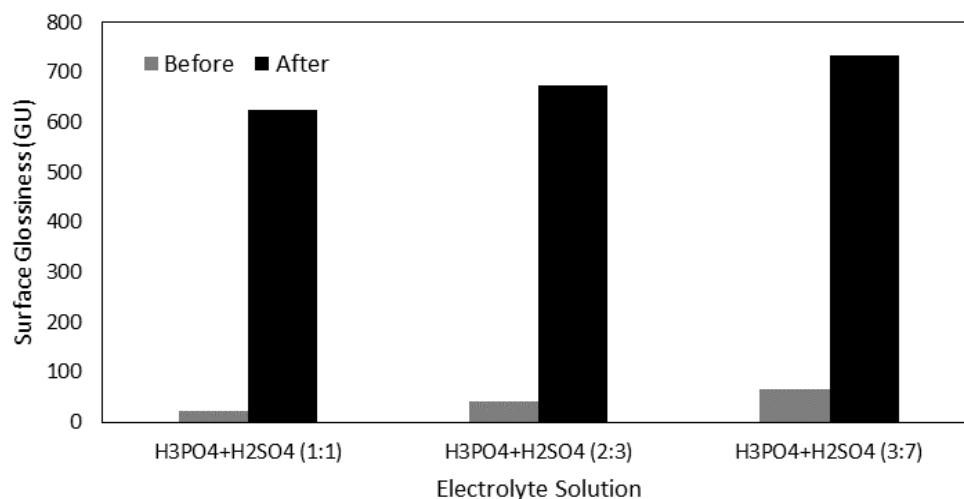
	H <sub>3</sub> PO <sub>4</sub>	32%
Neutralization	NaOH	25%
Rinsing	Cold water	-
Nitric Acid Treatment	HNO <sub>3</sub>	25%
Rinsing	Cold water	-
Hot Rinsing	Hot water	-
Drying	-	-

### 3. RESULT AND DISCUSSION

#### 3.1. Surface Glossiness

Glossiness of the electropolished samples under different electrolyte solutions, electric current, and electropolishing time is presented in Figures 3, 4, and 5 respectively. Figure 3 shows the results of electropolishing under different ratios of H<sub>3</sub>PO<sub>4</sub> and H<sub>2</sub>SO<sub>4</sub> in the electrolyte solution mixture. The electropolishing was conducted under 20 Amp electric current for five minutes. Figure 3 reveals that variation of electrolyte solution composition (H<sub>3</sub>PO<sub>4</sub> + H<sub>2</sub>SO<sub>4</sub>) yields different surface glossiness of the electropolished samples. This was due to the dissolution of metallic elements into the electrolytic solution. The surface of electropolished 316L SS contained Fe and C, which dissolved into the H<sub>3</sub>PO<sub>4</sub> + H<sub>2</sub>SO<sub>4</sub> solution. Dissolution of the elements produces a more even and smoother surface. This surface reflected the light better than an un-electropolished sample, which subsequently results in better glossiness [19-21]. Several works reported that lower surface roughness commonly created better glossiness.

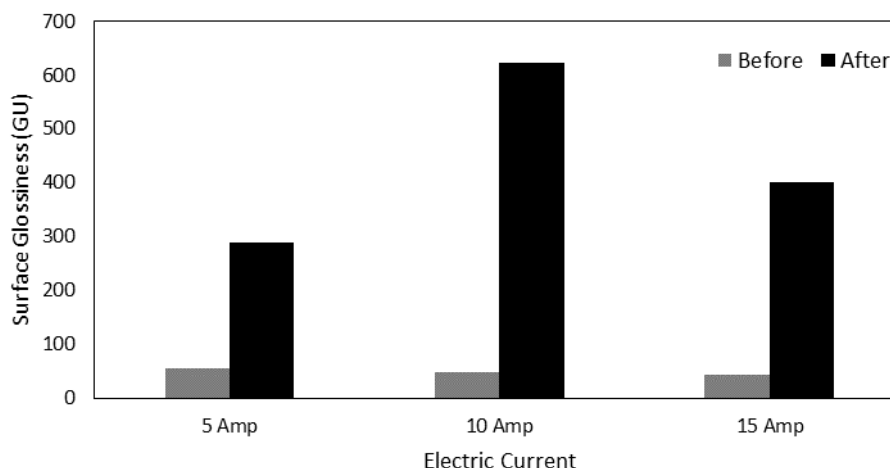
Compared to the electropolished surface, the untreated samples were rougher and consisted of more Ca, Fe, and Si. Therefore, this material had a poor glossy surface. The ratio of H<sub>3</sub>PO<sub>4</sub> and H<sub>2</sub>SO<sub>4</sub> in the electrolytic solution produces a different ability to dissolve Fe and C. This explains the influence of the electrolyte solution composition on the glossiness of the electropolished surface.



**Figure 3.** Improvement of glossiness of the specimen's surface after electropolishing under different composition of electrolyte solution

As illustrated in Figure 3, electropolishing treatment greatly improved the quality of the surface. The figure discloses that increasing the composition of H<sub>2</sub>SO<sub>4</sub> in an electrolyte

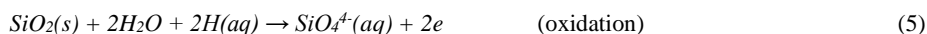
solution of  $H_3PO_4 + H_2SO_4$  made better glossiness improvement. Conversely, it is also seen in the figure that the ability of  $H_3PO_4$  without addition of  $H_2SO_4$  in dissolving metallic elements on the surface is very weak. Referring to the figure, the best glossiness is produced by  $H_3PO_4 + H_2SO_4$  solution with a composition ratio of 3 : 7. This improvement was enabled due to more corrosive and stronger oxidizer properties of  $H_2SO_4$  than  $H_3PO_4$ . Therefore, adding  $H_2SO_4$  increases the dissolution ability of the electrolyte solution. These properties allow the electrolyte solution to dissolve metallic elements or metallic oxide ( $Fe/Ni/Cr/CaO/SiO_2$ ) into ion of  $Fe(III)/Ni(II)/Cr(III)$ .



**Figure 4.** Improvement of glossiness of the specimen's surface after electropolishing under different electric current

Dissolution process of metallic elements in the specimens' surface follows reduction and oxidation bellow:

Anode:



Cathode:



Surface glossiness of the electropolished sample was also affected by the electric current applied in the treatment. To study this influence, electropolishing experiments were carried out under several electropolishing time. The experiments were executed using 3 : 7 ratio of  $H_3PO_4 + H_2SO_4$  and a five-minutes electropolishing time. Results of the experiment are given in Figure 4.

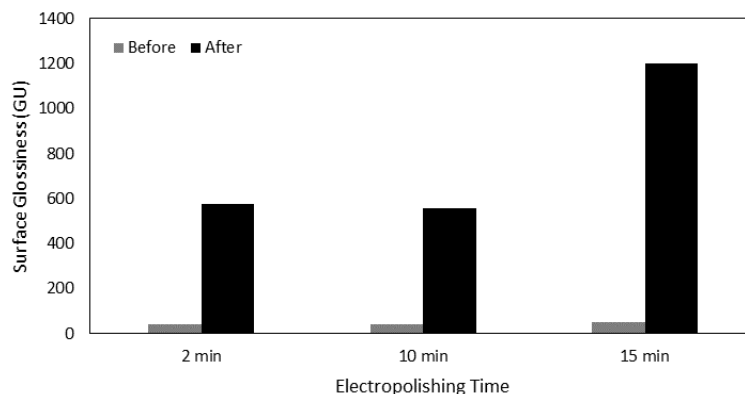
Figure 4 implies that increasing the electric current in electropolishing treatment tends to improve the surface glossiness. In this setting, the lowest improvement in surface glossiness was created when the treatment was carried out using 5 Amp electric current. Increasing the current to 10 Amp, the treatment improved the glossiness of the surface to 624 GU (Gloss Unit). This improvement was reduced when the current was enlarged to 15 Amp, thus an optimum condition was indicated at 10 Amps of electric current. The decrease in glossiness when the current was increased to 15 amps could be attributed to a higher current causing an excessive metal removal rate during the electropolishing process, thus worsening

the surface quality [22]. Similar to the result of the present work, the study also showed an optimum condition for surface quality. A leveling off in the electropolishing process was observed. It was found that increasing the current density improved the process' performance. However, adding more current led to a decrease in the improvement until it reached a plateau and eventually declined. The tendency of the optimum condition was also found in another research [23]. This work found that increasing current density improved the electropolishing performance; however, it remained fairly constant at a certain value of the electric current.

The improvement in the glossiness seemed to relate with weight loss of the samples after the electropolishing. Weight loss that was produced by the process with 5 Amp, 10 Amp, and 15 Amp were 0.032 g/Amp.sec, 0.0174 g/Amp.sec, and 0.012 g/Amp.sec respectively. Among the dissolution rates of treatment, electropolishing using 10 Amp electric current could be the optimum rate. A homogenous dissolution might be created on the surface samples by this treatment. Hence, a more even surface was resulted. This surface subsequently produced better glossiness. Higher or smaller dissolution rates as given by electropolishing under 15 Amp and 5 Amp could yield inhomogeneous dissolution of metallic impurities. Too high dissolution rates could deepen the scratch that might exist on the surface. Hence, it created a less-glossy surface.

The glossiness of the surface was also improved when electropolishing was done longer. To study the effect of electropolishing time on the surface quality, electropolishing treatments using variation of electropolishing time were conducted. The experiments were carried out using 3 : 7 ratio of  $H_3PO_4 + H_2SO_4$  under 10 Amp electric current. Glossiness of the surface samples after treatment is given in Figure 5.

Figure 5 shows that the treatment produced glossiness as high as 1200 GU when it was carried out for 15 minutes. Improvement in glossiness was already achieved in electropolishing as short as 2 minutes. Lengthening electropolishing to 10 minutes had yet to make further improvement. A high difference in the glossiness enhancement was obtained with 15 minutes electropolishing time. Referring to the discussion formerly on the influence of electric current on surface glossiness, an optimum condition that gave the highest gloss improvement was also indicated. This condition was correlated with the dissolution rate of metallic impurities in the samples' surface as formerly discussed. Based on this hypothesis, there might be an optimum electropolishing time that yields the best glossiness improvement. In contrast with this prediction, increasing electropolishing time was claimed to continuously decrease surface roughness which reflects better glossiness of 316L SS under constant electric voltage [24].



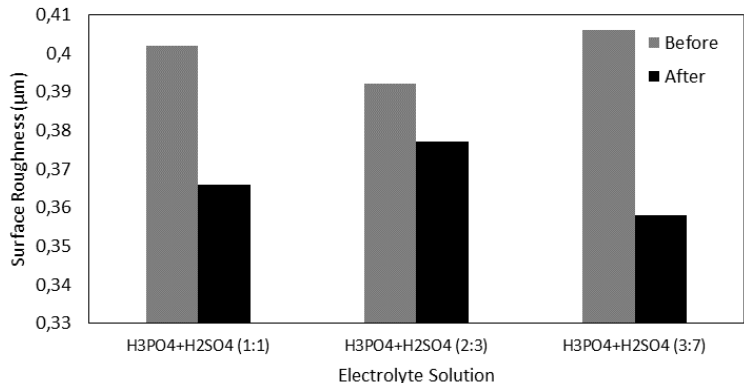
**Figure 5.** Improvement of glossiness of the specimen's surface after electropolishing under different electropolishing time

To explore further the influence of electropolishing time on the surface quality, experiments with longer electropolishing time should be attempted. This was not conducted in the present study. An optimum electropolishing time giving the best specular reflectivity was observed in electropolishing 304 SS using a different electrolyte bath [25]. This work observed an optimum electropolishing time at 9 – 15 minutes. Another work reported that a maximum gloss of 316L was achieved in electropolishing for 14 minutes [17], while lengthening the process beyond this time worsened the surface. Referring to these observations, as indicated in this present study, 15 minutes of electropolishing could be the optimum setting to produce the best gloss surface maintaining other parameters constant.

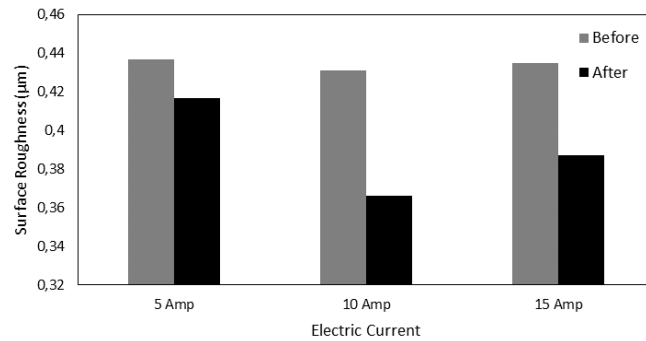
**3.2. Surface Roughness.**

Improvement of the samples’ surface roughness with respect to electrolyte solution, electric current, and electropolishing time were presented in Figure 6, 7, and 8. The figures exhibit the difference in the surface roughness before and after electropolishing treatment. Figures 6 – 8 reveal that the electropolishing reduced the surface roughness of the surface. An increase in the H<sub>2</sub>SO<sub>4</sub> composition in the electrolyte solution mixture did not significantly improve smoothness of the surface (see Figure 6). However, the lowest surface roughness is indicated in the figure, *i.e.*, 3:7 ratio of H<sub>3</sub>PO<sub>4</sub> and H<sub>2</sub>SO<sub>4</sub>. Thus, the dissolution rate of the metallic impurities is enhanced with the increment of H<sub>2</sub>SO<sub>4</sub> composition on the electrolyte solution. This phenomenon was discussed previously in the relation of the electrolyte solution with surface glossiness of the electropolished sample.

Surface roughness of the electropolished surface was also influenced by electric current applied in the treatment. Figure 7 illustrates that increasing electric current tends to decrease the surface roughness. An optimum setting that provided the smoothest surface was indicated at 10 Amp electric current. This is slightly like the effect of electric current on the surface glossiness. Nevertheless, the optimum electric current setting for the treatment in Figure 7 is strongly indicative. This optimum condition might be relevant to an analysis of energy required for the ionization that was proposed to explain the metallic dissolution in electropolishing [10]. The lowest energy for ionization of a superficial atom during anodic dissolution occurred on peaks which were the highest part of the surface indentation. However, too low electric current might not be able to generate sufficient energy for the ionization. It resulted in less dissolution of metal, which yielded to poor surface smoothness. On the other hand, this work also suggested that excessive current might cause intensive emission of oxygen that could result in surface defects and poor surface roughness. Thus, an electric current that generates the optimum energy might exist in between the two extreme conditions.



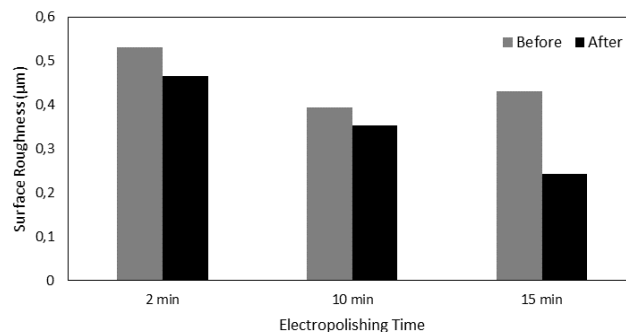
**Figure 6.** Surface roughness of the specimen’s surface after electropolishing using different solutions composition.



**Figure 7.** Surface roughness of the specimen's surface after electropolishing under different electric current

Observations of the surface roughness with respect to electropolishing time produced the results as given in Figure 8. The figure shows the difference in surface roughness before and after electropolishing treatment in different electropolishing durations. The results implied that within the applied electropolishing time, lengthening the process continuously decreased the surface roughness. Longer electropolishing time caused longer dissolution, hence more mass dissolution was made. Comparing the results with surface glossiness (Figure 5) showed that reducing the surface roughness reflects the improvement of surface glossiness. This relation was also disclosed in the surface produced by variations of electropolishing time and electric current.

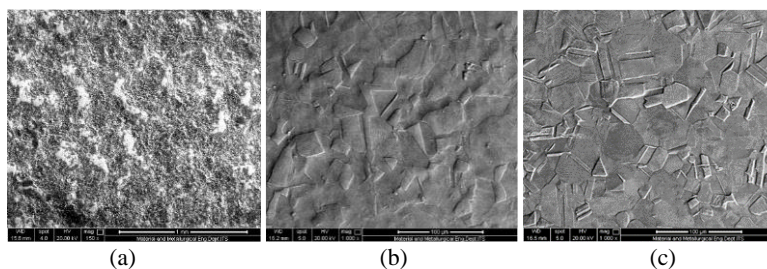
Figure 9 presents morphology of the untreated sample (Figure 9a) and the electropolished materials (Figures 9b and 9c). A significant improvement of the surface was shown in Figures 9b and 9c. Figure 9 (a) shows the surface of untreated material as rough and uneven. It also contains particles and oxide impurities in the form of silicon oxide and calcium oxide. These oxides were also indicated by the result of elemental analysis using EDX, which gave 9.53% and 1.58% of Si and Ca composition respectively. These impurities were not found in the electropolished samples (Figures 9b and 9c). Si and Ca were not detected by EDX analysis on the surface of the electropolished samples.



**Figure 8:** Surface roughness of the specimen's surface after electropolishing with different electropolishing time

The electropolished samples showed an even and cleaner surface as seen in Figures 9 b and c. Austenitic grains clearly appeared on the surface. The electropolishing process using 15 Amp electrical current seems to give a better surface than the electropolished material using 10 Amp current. As shown by Figure 9 c, a better surface was indicated than the other surfaces. The austenite crystal grains were also presented more clearly. Metallic impurities could present in the samples electropolished with 10 Amp electrical current (Figure 9 b). This makes unclear the grain boundary in the figure.



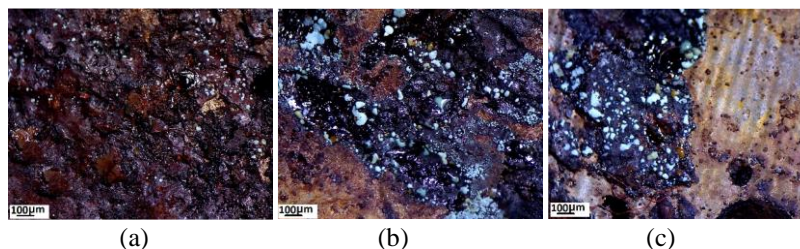


**Figure 9.** SEM image of the surface; (a) untreated material, (b) electropolished under 10 Amp electrical current, (c) electropolished under 15 Amp electrical current

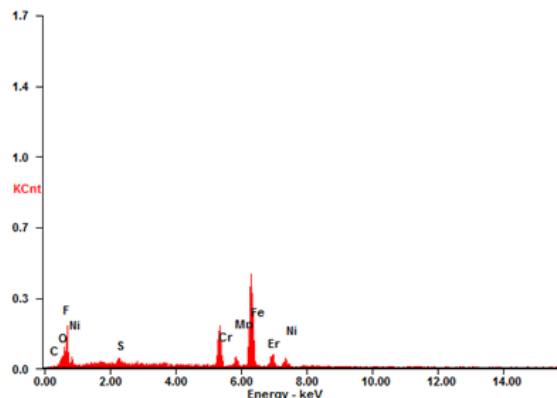
### 3.3. Corrosion Resistance

Weight loss of the samples during the corrosion test is depicted in Figure 10. A higher corrosion rate is experienced by all samples at the early stage of corrosion. Further growth of the corrosion in the untreated and electropolished samples were similar. It was shown by the approximately same slope of the weight loss curve. As shown in the figure, the electropolishing process produced the highest weight loss in the untreated sample. The treatment seemed to improve the corrosion resistance as indicated by the smaller weight loss yielded by the electropolished treatment. The smallest weight loss was given by the samples which were electropolished under 15 Amp electric current. Thus, electropolishing under 15 Amp electrical current brings the best corrosion resistance.

The corroded surface of the electropolished samples after the corrosion testing is shown in Figure 10. The figure illustrates uniform corrosion that occurs throughout the surface. The corrosion was observed on the entire surface of the untreated samples (Figure 10.a) indicating severe corrosion, while some light corroded zones were found in the electropolished samples (Figure 10.b and 10.c). The scale of oxide consists of discrete oxide particles (small white particles as seen in the figure) with a continuous uniform oxide scale as the matrix. A different appearance was shown on the surface of the electropolished samples. Some areas of uncorroded surface could be observed. These were indicated by the light-brown colored zone in Figures 10.a and 10.b. The other part of the surface was occupied with blackened brown and dark purple substances.



**Figure 10.** Surface of untreated material (a), electropolished - 10 A (b), and electropolished - 15 A (c)



**Figure 11.** EDX profile of the sample electropolished under 15 Amp electrical current

**Table 2.** EDX elemental analysis on the surface of electropolished sample (15 Amp electric current)

C	O	F	S	Cr	Mn	Fe	Er	Ni
1.30%	3.33%	13.94%	1.56%	11.92%	2.88%	30.12%	29.98%	4.97%

Improvement of the corrosion resistance of the electropolished materials was due to higher content of Cr and Ni at the surface. Higher content of Cr and Ni improved the resistance of the dissolution of iron (Fe). The dissolution of Fe was related to corrosion and electrolysis processes. Cr and Ni elements at the surface created a non-active electrolysis layer, therefore the anode-cathode function was not formed. This avoids the corrosion process as it requires an electrolysis mechanism.

#### 4. CONCLUSION

From the experiments and analysis of the results, the following conclusions were drawn:

- Surface quality and corrosion of electropolished 316L SS was studied with respect to electrolyte solution, electropolishing time, and electric current. The investigation found that a higher composition ratio of  $H_2SO_4$  in the mixture of  $H_3PO_4 + H_2SO_4$  electrolyte solution improved the dissolution rate of the metallic elements, which produced better surface glossiness of the material.
- This work also observed that the longest electropolishing (i.e., 15 minutes) time resulted in the best surface roughness and glossiness. This implied that better dissolution of metallic elements was produced by longer electropolishing. In the time range of the experiment, the present research did not observe any optimum condition in terms of the electropolishing time. Extending the electropolishing in the experiment might be required in further study to scrutinize if any optimum condition exists.
- Investigation on the effect of electric current on surface glossiness showed that too low or too high electric current produced poor surface glossiness. The optimum electric current for the electropolishing was identified at 10 Amp electric current.

A scale of oxide consisting of discrete oxide particles with a continuous uniform oxide scale was formed on the surface of the material during the corrosion test. Study on the corrosion of the samples revealed that a Cr-rich layer was formed on the surface of the electropolished

materials. It was identified by the existence of high Cr content at the surface. As compared with the untreated sample, the electropolished surface had a lower weight loss rate due to uniform corrosion. Thus, electropolishing has been proven to improve the corrosion resistance of 316L SS.

## ACKNOWLEDGMENT

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