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Evaluation of Corrosion Inhibition Performance and Surface Characterization of PCL-Coated Aluminum Alloys

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Abstract: Corrosion is a natural and unavoidable process that must be controlled to minimize damage to metals across various industries. One effective method for corrosion inhibition is the use of polymer coatings. In this study, Al alloy coupons were coated with a 10% concentrated solution of compound polycaprolactone (PCL) using a dip coater. Different dip-coating parameters were tested, including drying time, dwell time, speed, and number of cycles. Both coated and uncoated Al alloy coupons were immersed in 1 M HCl solution for 1, 2, 3, 7, and 15 days. Corrosion behavior was evaluated using the weight loss method. Among the samples, Sample 3 (5 cycles, 200 mm/s speed, 60 s dwell time, 30 s drying time) showed the highest corrosion inhibition efficiency, while Sample 4 (5 cycles, 300 mm/s speed, 60 s dwell time, 60 s dry time) exhibited the poorest performance. In addition to the weight loss analysis, SEM-EDX and FTIR analyses were conducted on Sample 2 and an uncoated Fe alloy coupon before and after corrosion testing.

Keywords: Coating, Corrosion, FTIR

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1. INTRODUCTION

One of the most used metal for several industries is aluminum (Brough & Jouhara, 2020). The properties of aluminum, which have high mechanical strength, corrosion resistance and durability, are useful for many branches of engineering, especially marine, aviation and automotive (Sun, 2023). Although aluminum has high corrosion resistance, it can be exposed to pitting, galvanic and erosion corrosion in specific time interval (Kaufmann, 2019). A suitable environment should be prepared in the laboratory to study these corrosion types. Various corrosive fluids are used for corrosion assessment of aluminum at the laboratory stage. Acidic and alkaline aqueous medium can be used as corrosive fluids for evaluation of corrosion of aluminum, in addition ethanol is also used in some different implementations as corrosive fluid (Kruger et al., 2012, Rodic et al., 2019, Yanardag et al., 2020). In these experiments with corrosive fluids, studies are carried out on methods to increase the corrosion inhibition properties of metals. There are many different studying topics to prevent corrosion that may occur on the metal surface, such as material design, material selection or metal surface treatments (Zehra et al., 2022). One of these topics is coatings, which are one of the metal surface treatments, are used to improve the corrosion inhibition property of the metal (AlFalah et al., 2020). Although there are many different methods for producing coatings for metals, spin coating, dip coating and the more recently used electrospinning methods are among the frequently used methods. The effect of polymer coatings applied to the metal surface by the dip coating method, which is one of these coating methods, on corrosion inhibition has been evaluated in various articles (Knigge et al., 2023, Tran et al., 2023). In these dip coating implementations, either mostly polymer or polymer-

based composite structures are used as coating materials (AlFalah et al., 2020, Xu et al., 2022). Among these polymers, PCL is biocompatible and hydrophobic polymer which is frequently used various sectors for coating material (Aranugiri et al., 2021). Various evaluation methods are used to evaluate the corrosion inhibition efficiency of the applied coatings. For the assessment of corrosion inhibition performance, many new methods have emerged for corrosion detection and monitoring with the development of technology (Chin et al., 2020). On the other hand, weight measurement technique is one of the oldest and basic method for corrosion detection process (Mansfeld & Kenkel, 1975). While the application of this method does not require advanced equipment; this method has a straightforward methodology based on measurement of mass reduction over a period of time (Usman et al., 2015a). There are some studies that calculate the corrosion inhibition efficiency parameter used to evaluate corrosion performance, based on weight loss measurement technique (Amiery et al., 2020, Berrisoul et al., 2022). In the literature, in addition to weight loss measurement for corrosion exposure comparison of coated samples, examining the microstructural properties of samples via SEM is also seen as an important application (Vinodhini & Xavier, 2023, Hegde et al., 2023, Gnedenkov et al., 2023). FTIR analysis is also used as an evaluation tool in corrosion examinations additionally SEM. (Pesha et al., 2023, Nesane et al., 2023, Rajzer et al., 2014). In this study, PCL was used as the polymer, HCl as the corrosive solution, aluminum as the metal exposed to corrosion, dip coater as the coating method, and weight loss method as the corrosion evaluation method.

2. MATERIALS AND METHOD

The atomic composition of the aluminum alloy coupon is aluminum 92.8%, copper (Cu) 4.7%, magnesium (Mg) 1.5%, manganese (Mn) 0.5%, zinc (Zn) 0.25%, iron (Fe) 0.2%, nickel (Ni) 0.04%, and lead (Pb) 0.01%. Firstly, the used Al alloy coupon was cleaned with ethanol and distilled water for better coating process. Finally, the coupon was dried at oven at at 60 ℃. PCL, hydrochloric acid (HCl) and dichloromethane (DCM) were obtained from Sigma Aldrich. PCL's molecular weight was 80,000 g/mol. PCL solution was prepared at %10 concentration with DCM solvent while HCl solution was prepared from % 37 HCl main stock. Density of main stock HCl was 1.19 kg/L. % 37 HCl solution and distilled water were mixed for preparing 1 M HCl solution.

The used Al alloy coupon was immersed in the coating solution (10 % PCL) and slowly removed at a certain speed. This step ensured uniform adhesion of the coating material to the surface of the Al alloy coupon. Each coupon was immersed 1.5 cm depth into the PCL solution for coating. All samples were attached to the dip coater system with the help of a standard tape, keeping same the part of the metal which was immersed in the solution. Dwell time, drying time, and speed were adjusted according to the parameters given in Table 1. This process was repeated 5 times for each sample. Following the coating process, the samples were left to dry.

The used Al alloy coupon dried after the coating process. This step helped the coating material acquire its desired structural or chemical properties. Figure-1 shows the all steps for coating and corrosion test process. Figure-2 shows the dimensions of the coupon and the surface area which immersed in the polymer solution. PCL coating was applied on the Al alloy coupon under different immersion and drying conditions with MTI company's PTL-MM02-200 model dip coater, and after it was dried in oven, it was placed in 1 M HCl solution for corrosion test. Then, weighing was done on certain days to measure weight loss. Before weighing, samples were exposed drying procedure in the oven until the drying process is completed reliably. Corrosion inhibition efficiency was determined according to the following formula (Amiery et al., 2020):

$$IE\,\% = \frac{100 * w_0 - w_1}{w_0}$$

where $w0$ is referred to as the blank sample's weight loss and $w1$ matches with the coated samples' weight loss for different dip coater's parameters. Table - 2 shows the corrosion inhibition efficiency values for the samples.

A Scanning Electron Microscope (FEG 250 SEM - FEI-Quanta, Hillsboro, OR, USA) was employed to investigate the surface characteristics of variously coated Al samples. The measurement parameters included Accelerating voltage (HV) = 10 KV, HFW= 20.7µm and 82.9µm, detector= ETD, mode=SE, operating in High Vacuum Mode. Prior to SEM imaging, all samples underwent a coating process using a 40% gold and 60% palladium mixture. This coating procedure was executed with the Quanta FEG 250 model from the FEI brand, applying a 40 mA current for 60 seconds. Elemental compositions on the surface were analyzed using energy dispersive x-ray spectroscopy (EDX). Furthermore, Fourier-transform infrared spectroscopy (FT-IR) (Bruker model-ALPHA) was utilized to scrutinize chemical bondings at dipped and undipped PCL coating.

3. RESULTS AND DISCUSSION

The coupons coated with PCL were subjected to corrosion by immersion in a 1 M acidic solution for 15 days. After weighing each sample, they were placed in an HCl solution, and weight measurements were taken on the 1st, 2nd, 3rd, 7th, and 15th days. The results obtained are

presented in Table 2. Among the coated coupons, higher weight loss occurred in Sample 4.

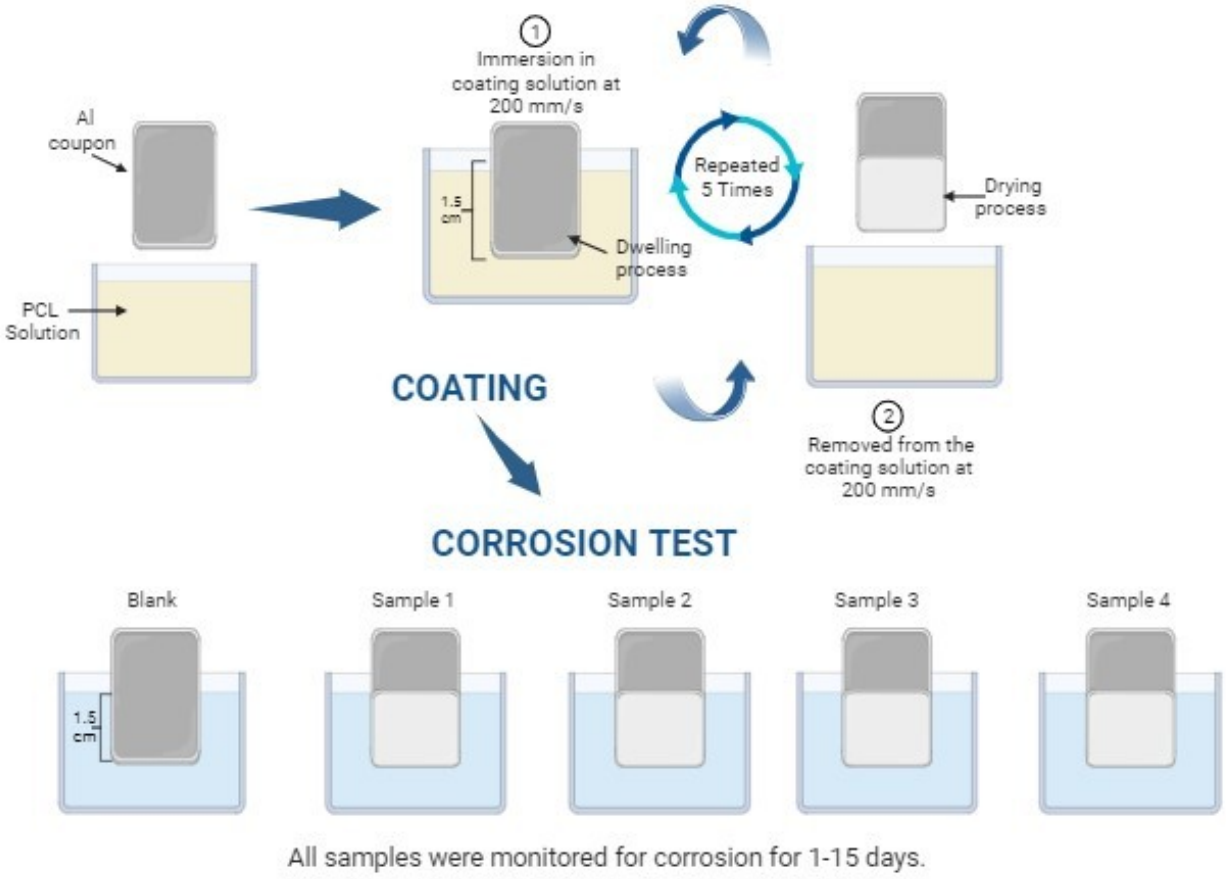


Figure 1: Al alloy coupon coating and corrosion test process.

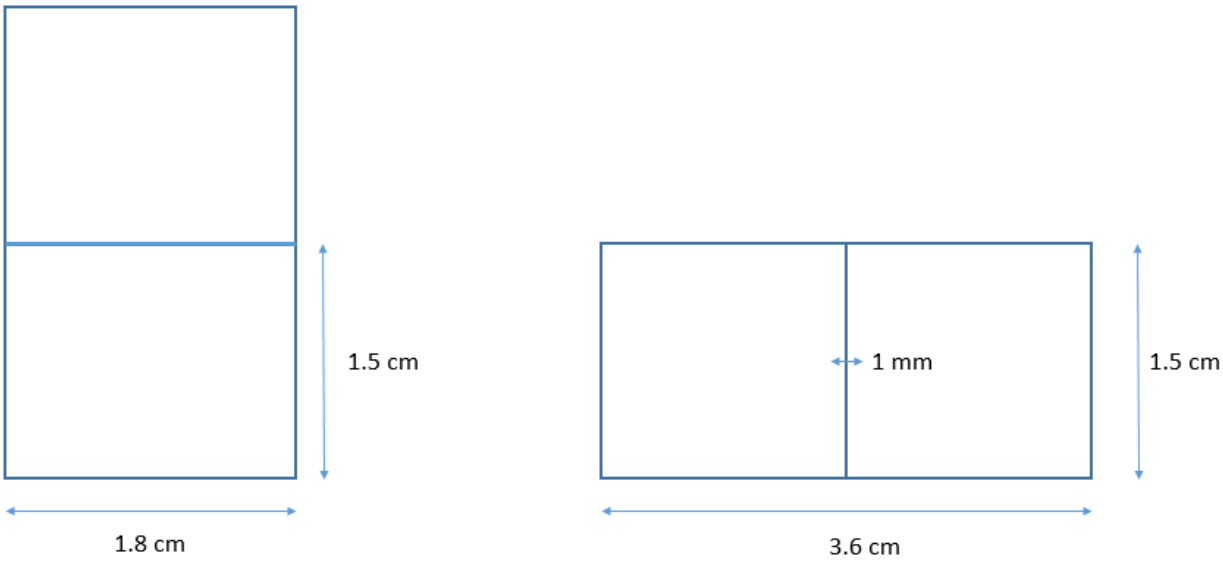


Figure 2: Representation of the surface area of the part of the metal coupon immersed in the polymer solution (thickness of coupon is 1 mm)

Table 1: Dip coater parameters of the coating process.

	Cycle	Dwell Time (s)	Drying Time (s)	Speed (mm/s)
Sample 1	5	20	60	200
Sample 2	5	20	30	200
Sample 3	5	60	30	200
Sample 4	5	60	60	200

Table 2: Samples' corrosion inhibition efficiency values.

	Blank (%)	Sample 1 (%)	Sample 2 (%)	Sample 3 (%)	Sample 4 (%)
Day 1	-	82	65	97	97
Day 2	-	90	87	85	77
Day 3	-	79	63	86	67
Day 7	-	68	82	77	40
Day 15	-	39	37	43	6

Figure 3 shows corrosion inhibition efficiency values for all samples. Vertical blue lines indicates the measurement days which are 1, 2, 3, 7, 15. The measurement was completed on the 15th day, it was observed that the corrosion inhibition efficiency reached its lowest level in all samples. Figure - 2 is derived from the data in Table -2.

In the review study on corrosion measurement due to weight loss conducted in 2015, in which many studies were reported using different metals including aluminum and different solutions including hydrochloric acid, many studies similar to this study were shown and the results obtained were similar to this study (Usman & Okora, 2015b).

Figure 4 shows the FTIR peaks for dipped and undipped PCL coatings. A comparison with solid PCL is also given in Table 3. As can be seen in the table, the peaks are close to each other. Looking closely at a few peak values, 2949 cm^{-1} peak shows asymmetric CH_2 stretching, 2865 cm^{-1} peak indicates symmetric CH_2 stretching, 1727 cm^{-1} peak exhibits carbonyl stretching, C-C and C-O stretching are seen at 1293 cm^{-1} peak and at 1157 cm^{-1} peak. 1240 cm^{-1} peak presents asymmetric COC stretching (Rajzer et al. 2014).

Figure - 5 show SEM images in 3 different magnification modes for the coated surface of the 2nd sample. SEM images were taken to visually detection the erosion of the coating in acid media. The worn structure of the coating on the sample surface after immersion in the acid media is clearly visible in 3 different magnification modes.

In another study, similar to this study, the corrosion of galvanized iron pipelines against water was evaluated using the weight loss method. SEM images of these pipelines were taken, as in our work, and wear on the metal surface was seen in these images (Kumar et al. 2023).

In another similar study, the corrosion of aluminum coupons in HCl, sulfuric acid and sea water was examined. As seen in the SEM images of aluminum coupons in the article, wear on the coupons is also clearly seen, as being similar to our study (Sabarirajan et al. 2025).

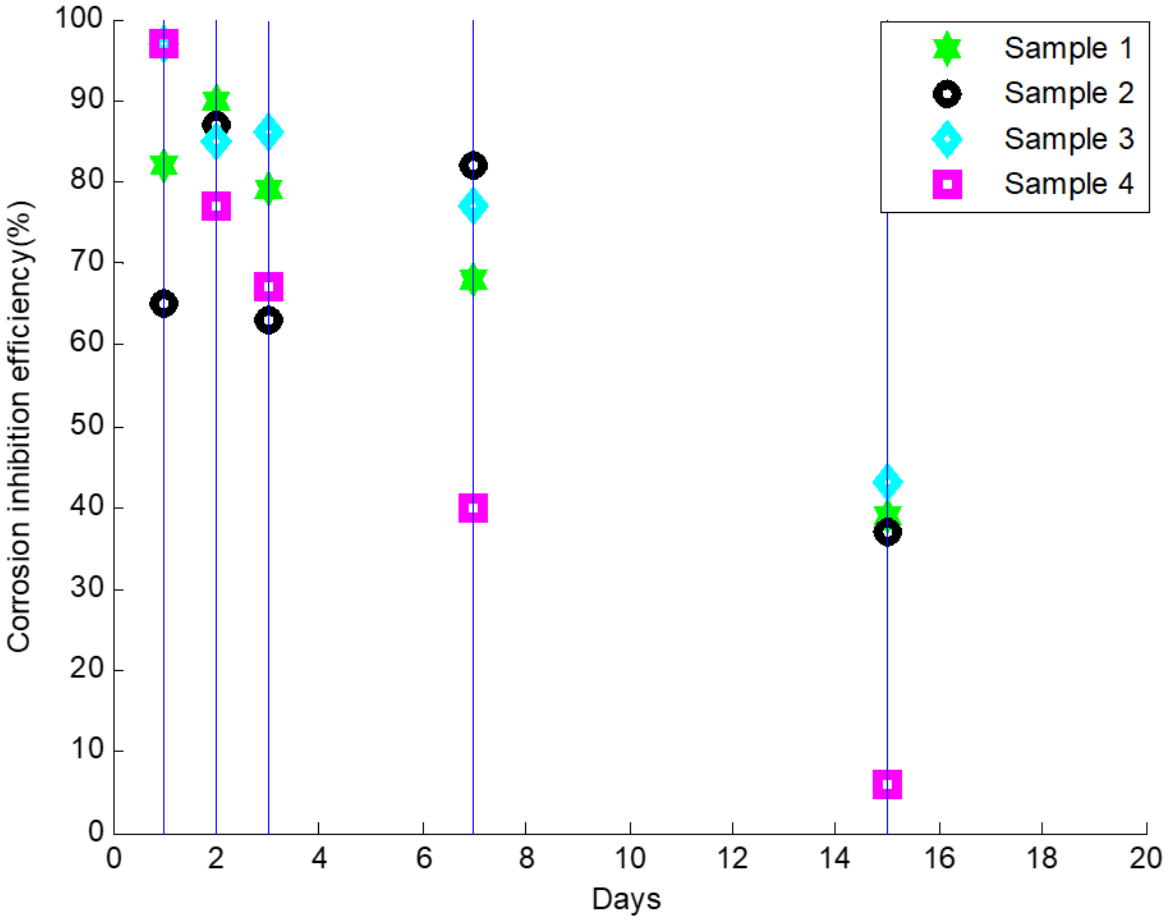


Figure 3: Corrosion inhibition efficiency values (%) for all samples versus days.

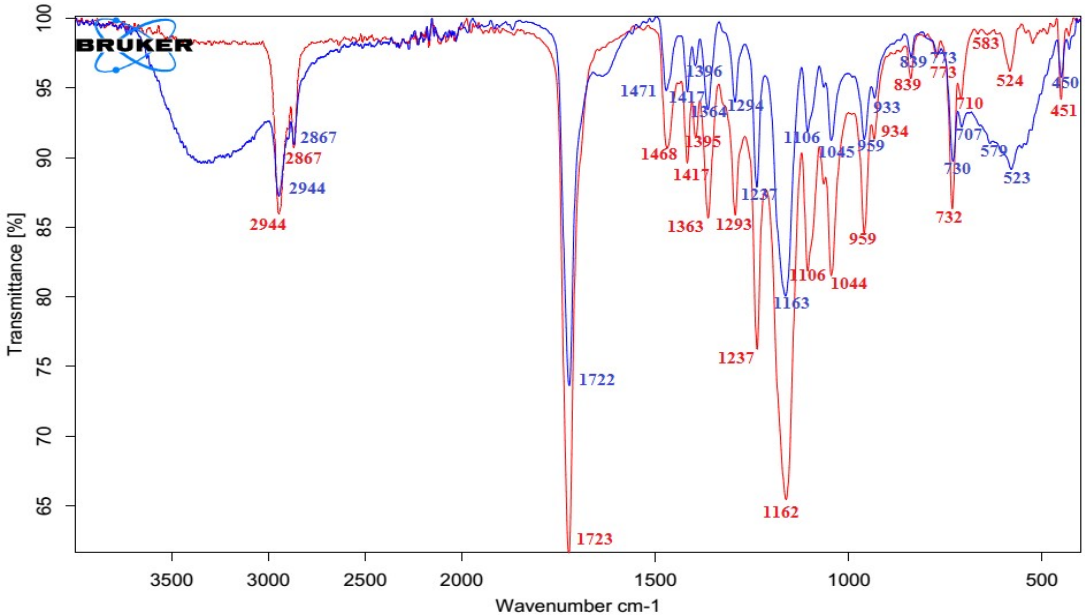


Figure 4: FTIR % transmittance peaks spectrum for dipped and undipped PCL coating (blue:dipped, red:undipped).

Table 3: FTIR peaks' values of solid, dipped and undipped samples.

Solid PCL wavenumber (cm^{-1}) peaks (Rajzer et al. 2014)	Undipped PCL coating wavenumber (cm^{-1}) peaks	Dipped PCL coating wavenumber (cm^{-1}) peaks
2949	2944	2944
2865	2867	2867
1727	1723	1722
1293	1293	1294
1240	1237	1237
1157	1162	1163

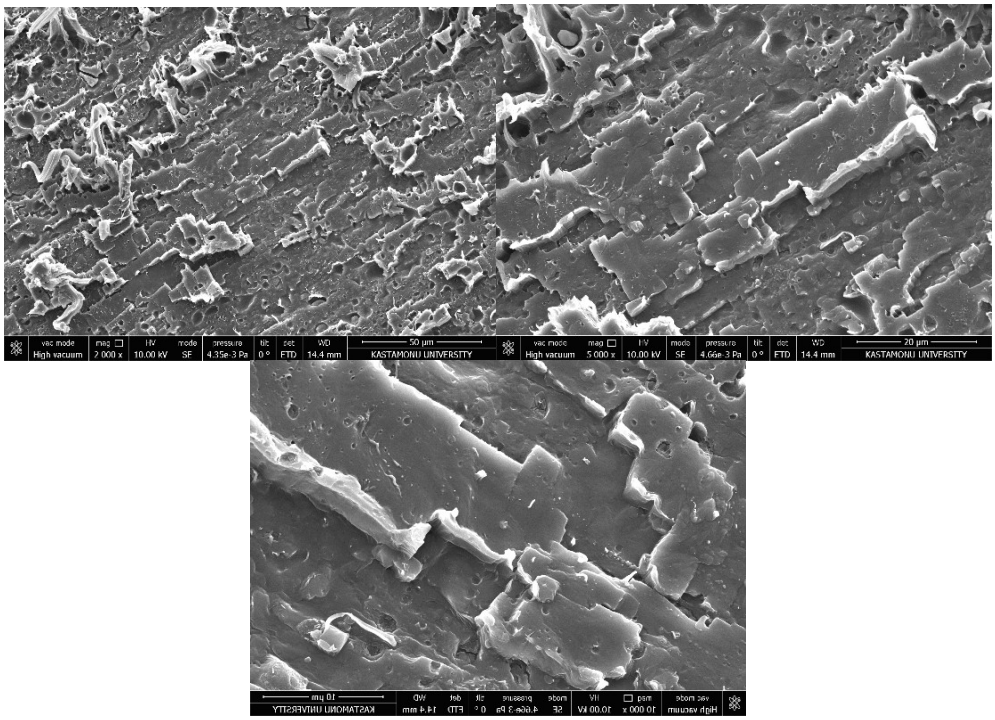


Figure 5: 2000x, 5000x, 10000x SEM images for coated surface of sample 2 after corrosion test.

Figure 6 and Figure 7 indicate the EDX measurements of the selected sample 2, showing the weight and atomic percentage of elements before and after acid immersion.

As a result of the EDX before the dipping process, the carbon percentage in the selected area of the coating was 74.92%, while the oxygen percentage was 25.08. After immersion, the carbon percentage for the same area was 72.42%, while the oxygen percentage was 27.05% and aluminum, which emerged due to wear on the Al coupon as a result of corrosion, was 0.52%.

4. CONCLUSION

In this study, PCL coated alloy coupons were tested for corrosion inhibition for PCL using different dip coater parameters. PCL coatings were compared among themselves according to their dwell time and drying time differences. Sample 4 had worse performance in corrosion inhibition efficiency values based on the 15-day data, whereas sample 3 had better performance than others'.

Peak values of solid undipped and dipped samples of PCL are very close for FTIR results examination. Therefore, it can be concluded that there is not much wear in the structure of PCL on the Al coupon. In the final analysis, according to the FTIR results, it can be said that PCL exhibits

corrosion inhibition properties in the form of coating used on the Al alloy coupon.

Fig-5 shows SEM images at different magnifications after corrosion, Fig-6 and Fig-7 show EDX results before and after corrosion for sample 2. The coating images before dipping into acid were smooth and homogeneous, but after dipping into acid, the coating images lost their homogeneity and turned into a worn-looking structure. As can be seen in Figure 6, in the measurement made before dipping into acid, while the majority of the elements in percentage belonging to the polymer (PCL) used were present on the coating surface, in Figure 7, the Al parts which worn out of the aluminum sample due to corrosion were also included in the atomic and weight percentage. According to the EDX results, for the measurements taken from the surface, the presence of Al atoms on the surface increased in percentage due to the corrosion.These SEM and EDX results show that the acid solution has caused some decays on the PCL coating.

According to high level changes for drying and dipping time for future works, may lead to differences in corrosion inhibition for the coated Al alloy coupon. It is thought that the study can be furthered by making changes to the parameters of dip coater.

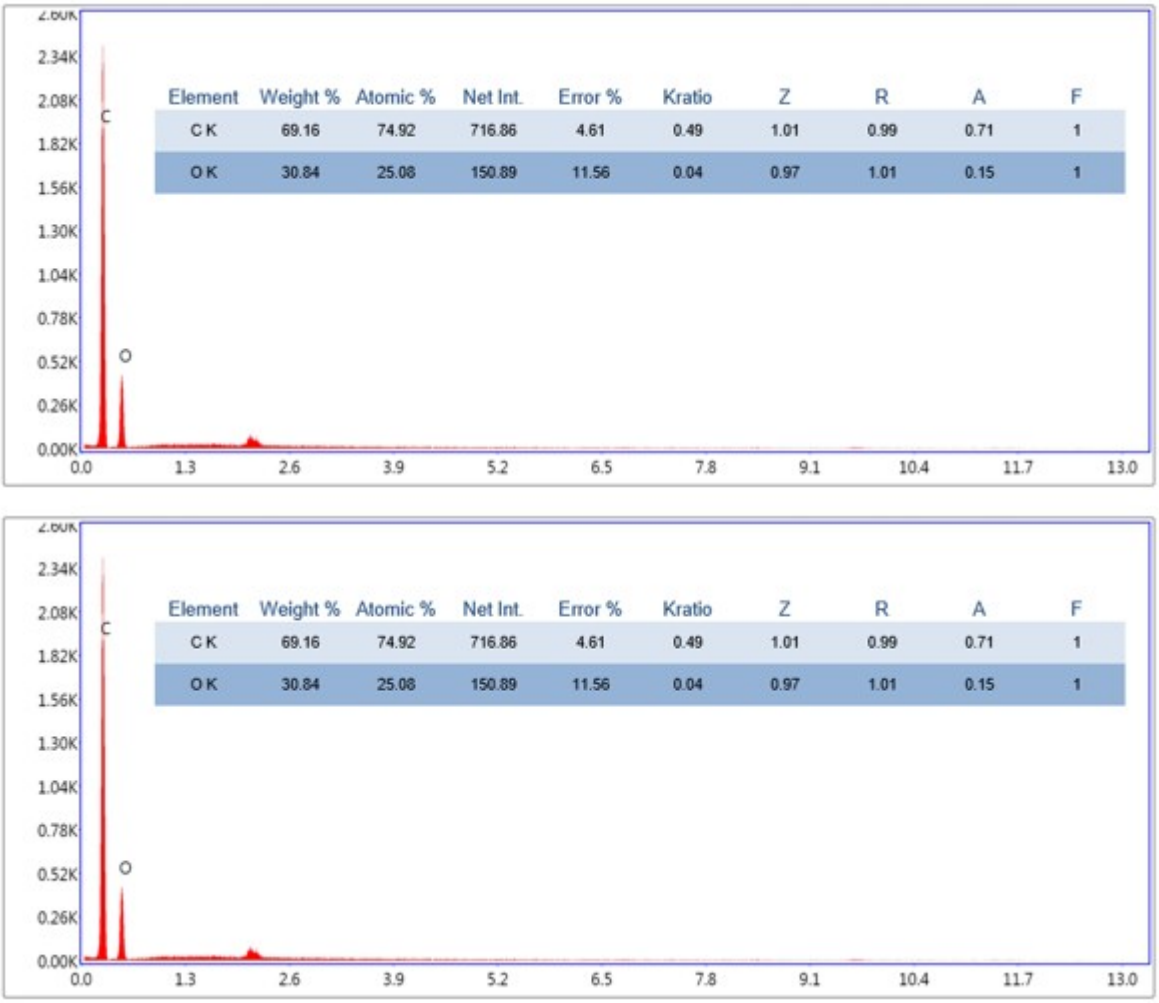


Figure 6: EDX results for sample 2 before corrosion test.

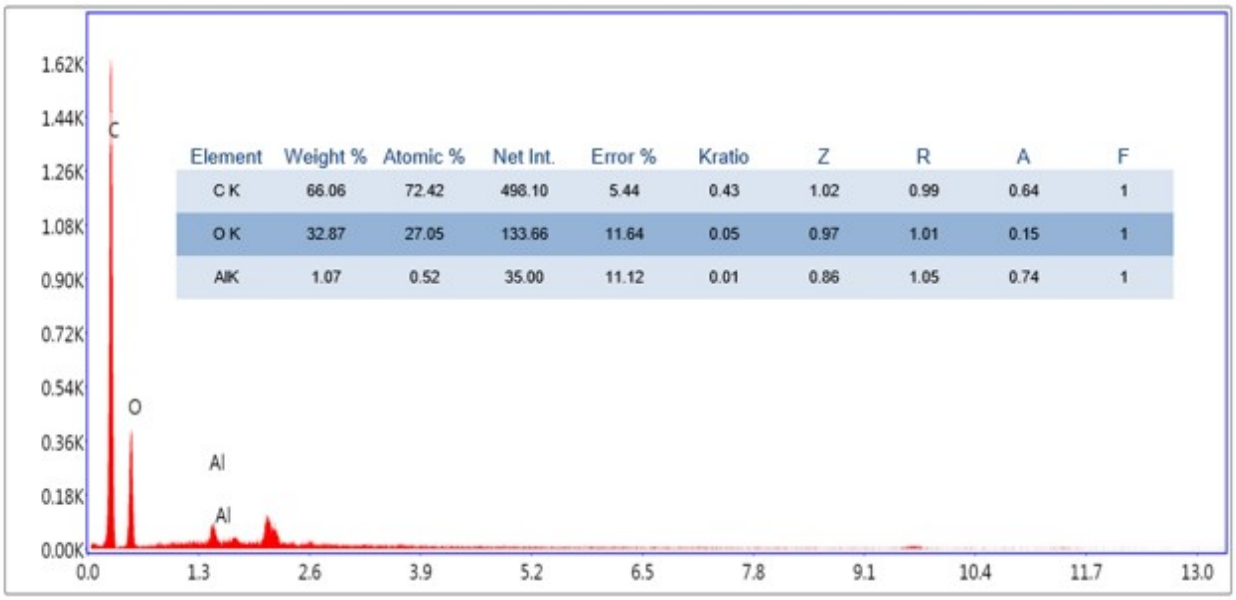


Figure 7: EDX results for sample 2 after corrosion test.

5. CONFLICT OF INTEREST

There is no conflict of interest among the authors.

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