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Corrosion Morphological Characteristics of the Blackening Process on Plate and Cylindrical Workpieces

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ABSTRACT

Corrosion is a prevalent issue in metallic materials, leading to significant damage and potential hazards for individuals. Take, for instance, AISI 1015 steel, belonging to the low carbon steel category commonly employed in crafting machine tools. Hence, there is a requirement for a coating solution capable of application without adding thickness to the surface of the workpiece while effectively mitigating corrosion. One effective method for mitigating corrosion is through the application of a blackening coating. The objective of this study is to investigate how varying heating time and temperature during the blackening process of AISI 1015 steel impacts both thickness and corrosion rate. The experimental approach involves subjecting test specimens of AISI 1015 steel, in both plate and cylinder forms, to a salt solution comprising 30% Sodium Hydroxide (NaOH), 10% Sodium Nitrate (NaNO3), 10% Sodium Nitrite (NaNO2), and 50% water (H2O). The specimens will be exposed to different heating durations of 30, 60, and 90 minutes at a constant temperature of 100°C. Next, the blackened material will undergo immersion in hydrochloric acid (HCl) solutions with concentrations of 5% and 10%. The evaluation will involve assessing weight loss for each material and utilizing scanning electron microscopy (SEM) for further analysis. The research show that the thickest surface layer was observed after a 90-minute coating duration, measuring 20.27µm for plate specimens and 19.24µm for cylindrical ones. Regarding corrosion rates, for cylindrical AISI 1015 steel, the lowest rate was recorded at 2662.90 mm/year with a 5% HCL solution, and the highest at 3099.79 mm/year. For plate-shaped AISI 1015 steel, the lowest corrosion rate was 520.68 mm/year with a 5% HCL solution and the highest was 2082.72 mm/year with a 10% HCL solution. It can be concluded that the blackening process results in a thicker and more uniform surface layer on plate-shaped materials compared to cylindrical ones. Consequently, this leads to a lower corrosion rate on plate-shaped materials compared to cylindrical ones.

Keywords:

Blackening process; Layer thickness; Corrosion rate

1. Introduction

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Steel is a type of material that has many applications in everyday life. However, one of the worst properties of steel is that it easily experiences corrosion in atmospheric environmental conditions, so a coating is needed that can block and reduce the corrosion process. There are several ways that can be used to prevent and reduce the corrosion process, including metal coating.

Technology is increasingly developing rapidly, forcing us to always innovate and be creative without limits. One of them is the automotive industry which is always required to create a surface coating technology that can be resistant to all types of corrosion. Electroplating is a type of technology that is carried out through a process of deposition of a metal layer on an electrode with the aim of making the surface different from the original [1]. One of these processes is the blackening process, which is a metal coating process that does not use electric current. Metal coating using a chemical process is called conversion plating. This treatment is a coating that uses a chemical reaction to produce a thin layer on the surface [2-4].

The blackening layer occurs when the Fe atoms on the steel surface react to form magnetite (Fe_3O_4). The surface of the blackening product must be clean of dirt by placing it in an alkaline bath and rinsing it, then placing it in a blackening solution. The finish will be covered with a rust preventative, which can result in varying final shapes [5-7]. In the blackening coating service industry in Sidoarjo, Indonesia, there are products that do not meet quality requirements in terms of surface layer thickness. This is because the layer is very thin and easily damaged by the corrosion process. These results can occur due to imperfect processes and less than optimal determination of operational process variables [8]. Improvements are needed, starting from the mechanical and chemical cleaning process [9]. Apart from that, the coating process itself must see the maximum conditions, for example by determining the required process variables [8].

The materials used in a study are also influenced by many factors. The material will experience an increase in layer thickness on its surface, if the heating temperature in the blackening process also increases [10]. This process is caused by good and appropriate operating conditions, namely the longer the heating carried out in the blackening process will make the surface layer of the material thicker [8]. Apart from that, existing operating conditions will influence the increase in material surface thickness [6]. The objective of this research is to investigate the surface layers and corrosion rate induced by the blackening coating on the material.

This study focuses on optimizing the bleaching process for AISI 1015 steel material by varying heating time and temperature to assess their effects on surface thickness, corrosion rate, and morphological properties. The aim is to identify the ideal combination of time and temperature for the bleaching process. The treatment involves immersing the material in hydrochloric acid (HCl) solutions with concentrations of 5% and 10%. The effectiveness of different time-temperature combinations will be evaluated through weight loss tests and analysis using Scanning Electron Microscopy (SEM) to assess morphological changes.

2. Proposed Methodology

The metal is made from AISI 1015 steel. Chemical composition of AISI 1015 steel (5% by weight): 0.15C, 0.082Mn, 0.166 Si, 0.054P, 0.15S, and Bal. Fe. [11,12]. In this experiment, the work objects were AISI 1015 steel plates and cylinders. The test specimens, comprising both plate and cylindrical shapes, had dimensions of 100 mm in length, 20 mm in width (for the plate), 3 mm in thickness (for the plate), and 20 mm in diameter (for the cylindrical specimen). The experimental setup follows a factorial influence model. The focus variables in this study are the thickness of the surface layer and the corrosion rate induced by the blackening process on the workpiece. The independent variable in the blackening process is the length of heating time, namely: 30; 60; and 90 minutes, and the test objects are plate and cylinder shapes. The control variables are the type of workpiece, blackening solution [13], and heating temperature of 100ºC. Meanwhile, in the corrosion resistance test, the independent variables were the concentration of the HCl solution, namely: 5% and 10%, and the test objects were plate and cylindrical. The control variables are the type of workpiece, the amount of HCl solution for the soaking process, and the quality of the chemicals.

The research procedure begins with preparing the workpiece to its specified dimensions and gently cleaning it. Cleaning is achieved by rubbing the surface with abrasives of various grit sizes, including 100, 500, 1000, 1500, and 2000. Subsequently, the workpiece is weighed to establish the initial weight. Then, the specimen is immersed in an ethanol solution to ensure thorough cleaning. Prepare a blackening solution, and heat it at a constant temperature of 100ºC. Prepare the test object to be coated, tie it to the position of the test object floating in the coating bath. A total of 18 test specimens were prepared, followed by the application of the coating process at various time intervals. Upon completion of the coating process, the test objects are rinsed with distilled water and dried. Subsequently, the surface thickness of the coating is measured using an ultrasonic thickness measuring device. Prior to immersion, the initial weight of each test specimen is recorded. Then the test object which has been coated with blackening is soaked using an HCl solution. Preparation of HCl solutions with concentrations of 5% and 10%. Soak plate and cylindrical test objects that have been coated with blackening in the HCl solution with thread so that the position of the test object hangs in the HCl soaking tub. After the soaking process is complete, rinse the test object with distilled water and dry. Then do the final weighing of the test object. And do SEM testing.

3. Results and Discussion

3.1 Coating Thickness Testing

This is a data display from the results of testing the layer thickness of the workpiece, the results of metal coating are as follows.

Table 1
Data on test results for the layer thickness of plate-shaped workpieces

No	Time (minute)	Thickness Test (um)			average (um)
		I	II	III	
1	30	18.12	18,14	18.12	18.13
2	30	18.10	18.16	18.16	18,14
3	30	18.16	18.20	18.18	18.18
4	60	19.10	19.12	19.16	19.13
5	60	19.14	19.16	19.12	19.14
6	60	19.08	19.18	19.18	19.15
7	90	20.27	20.28	20.26	20.27
8	90	20.20	20.26	20.23	20.23
9	90	20.22	20.25	20.27	20.25

Table 2
Data on test results for the layer thickness of plate-cylinder workpieces

No	Time (minute)	Thickness Test (um)			average (um)
		I	II	III	
1	30	17.48	17.42	17.46	17.45
2	30	17.46	17.48	17.44	17.46
3	30	17.47	17.46	17.42	17.45
4	60	18.36	18,32	18.34	18.34
5	60	18.40	18.38	18.38	18.39
6	60	18.38	18.36	18.36	18.37
7	90	19.24	19,20	19.22	19.22
8	90	19.18	19.22	19.24	19.21
9	90	19,20	19.26	19.26	19.24

Then the data obtained is made in graphic form as in Figure 1 below.

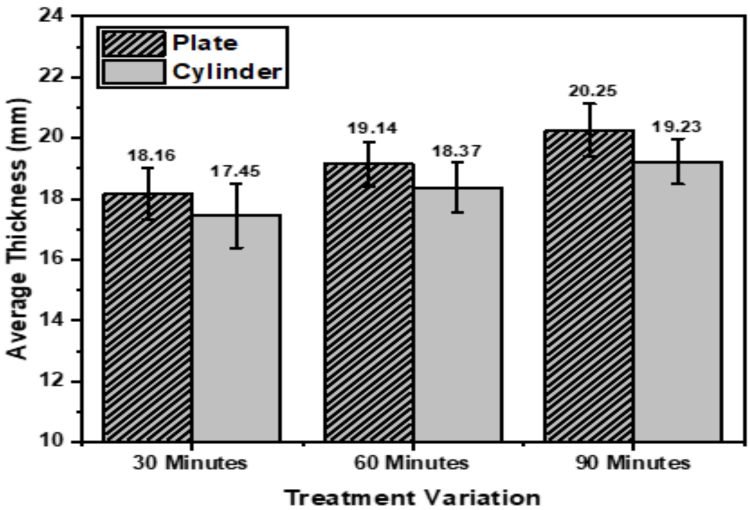

Fig. 1. Thickness graph of plate and cylindrical workpieces resulting from blackening coating with a time of 30, 60 and 90 minutes

The coating process using the blackening method affects the surface thickness value as the blackening process progresses. In Figure 1, it can be seen that the heating times observed at 30, 60, 90 minutes resulted in the best thickness being 17.45 um to 20.27 um. This heating time creates the most obvious interaction with the specimen, this is due to the layer sticking to the metal surface until the magnetite (Fe_3O_4) is completely formed and the outer colour of the metal becomes blacker (Fe atoms on the surface react with carbon to become magnetite (Fe_3O_4)). In this process, OH- ions cause diffusion interactions in the layer, then new atomic bonds are created with negative Fe ions which then become uniform in thickness on the surface of the workpiece. This process causes a layer to accumulate on the surface of the workpiece throughout the heating time[14-16].

Likewise, the heating temperature will affect the size of the metal surface, because the increasing heating temperature causes OH- ions to rise and shift towards the metal surface. Sufficient OH- ions on the surface of the workpiece cause the surface condition to become alkaline. The chemical reaction increases gradually until it forms a magnetite (Fe_3O_4) film on the surface [17,18]

3.2 Corrosion Testing

Before the metal coating process is carried out, the workpiece must be cleaned from the chipped chips that are still attached due to residue from the machining process. Plate and cylinder specimens are weighed first for weight testing before and after the workpiece cleaning process. After the weight weighing process was carried out, the plate and cylinder specimens were also carried out by calculating the corrosion rate on twelve plate specimens and twelve-cylinder specimens. Below you can see in Tables 3 and 4 the results of the corrosion rate after the blackening process, the results of which can be seen below.

Table 3
Data on the corrosion rate of plate-shaped workpieces

No	Composition HCL (%)	Weight before Work (gr)	Heavy After Work (gr)	Weight Difference (gr)	Corrosion Rate (mm/year)
1	5	0,43	0,21	0,22	2622,90
2	5	0,45	0,24	0,21	2503,67
3	5	0,44	0,22	0,22	2622,90
4	5	0,46	0,20	0,26	3099,79
5	5	0,45	0,20	0,25	2980,57
6	5	0,47	0,21	0,26	3099,79
7	10	0,45	0,19	0,26	3099,79
8	10	0,47	0,23	0,24	2861,34
9	10	0,44	0,17	0,27	3219,01
10	10	0,46	0,13	0,33	3934,35
11	10	0,45	0,13	0,32	3815,12
12	10	0,47	0,11	0,36	4292,01

Table 4
Data on the corrosion rate of plate-cylinder workpieces

No	Composition HCL (%)	Weight before work (gr)	Heavy After Work (gr)	Weight Difference (gr)	Corrosion Rate (mm/year)
1	5	0,31	0,26	0,05	520,68
2	5	0,33	0,28	0,05	520,68
3	5	0,34	0,29	0,05	520,68
4	5	0,34	0,25	0,09	937,23
5	5	0,32	0,24	0,08	833,09
6	5	0,35	0,25	0,10	1041,36
7	10	0,31	0,19	0,12	1249,63
8	10	0,33	0,17	0,16	1666,18
9	10	0,34	0,15	0,19	1978,59
10	10	0,34	0,20	0,14	1457,91
11	10	0,32	0,15	0,17	1770,32
12	10	0,35	0,15	0,20	2082,72

After testing the corrosion rate, the data is displayed in the form of a graph in Figure 2. The graph that has been created will allow us to know the difference between the corrosion rate on plate and cylindrical workpieces.

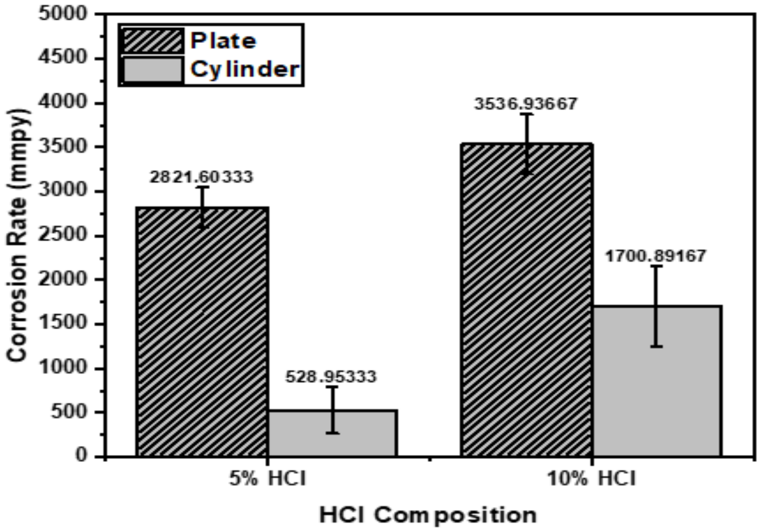

Fig. 2. Corrosion Rate Graph for Plate and Cylindrical Materials with 5% and 10% HCL Composition

In the process of dipping a workpiece using an acid solution (HCL), a chemical reaction occurs, namely an erosion process on the surface of the plate and cylinder workpiece. This occurs due to the reactive nature of the HCL solution which reacts with the workpiece. From Tables 3 and 4 above, it can be seen that the composition of the HCL solution and the shape of the workpiece greatly influence the corrosion rate of the workpiece. The greater the composition of the HCL solution, the less the weight of the workpiece, namely in plate material the concentration of the HCL solution is 5% from the initial weight of 0.46 grams to 0.20 grams after dipping, so there is a weight difference of 0.26 grams. Meanwhile, with plate-shaped material with a 10% HCL solution concentration, from an initial weight of 0.47 grams to 0.11 grams after dipping, so there is a weight difference of 0.36 grams. In a cylindrical workpiece with a HCL solution concentration of 5%, that is, from an initial weight of 0.35 grams to 0.25 grams after immersion, so there is a weight difference of 0.10 grams. Meanwhile, with cylindrical material with a HCL solution concentration of 10%, that is, from an initial weight of 0.35 grams to 0.15 grams after dipping, so there is a weight difference of 0.20 grams.

In plate-shaped material, the concentration of 5% HCL solution was the smallest corrosion rate was 2662.90 mm/year and the largest was 3099.79 mm/year. Meanwhile, the plate-shaped material with a concentration of 10% HCL solution showed the smallest corrosion rate of 2861.34 mm/year and the largest 4292.01 mm/year. In cylindrical material, the HCL solution concentration of 5% shows the smallest corrosion rate of 520.68 mm/year and the largest 1041.36 mm/year. Meanwhile, the cylindrical material with a concentration of 10% HCL solution showed the smallest corrosion rate of 1249.63 mm/year and the largest 2082.72 mm/year.

The difference in weight that occurs in plate and cylindrical workpieces is due to the fact that cylindrical workpieces are more easily corroded/eroded by acid solutions compared to plate workpieces. This is caused by the coating results on cylindrical workpieces being thinner and more uneven than those on plate shapes. so that the ions and electrons in the cylindrical material become unstable due to the meeting between the work object and the HCL solution which indirectly causes the atomic bonds in the material to become loose [17,18]. By releasing atomic bonds, the material will erode and become porous. Meanwhile, the greater the percentage composition of the HCL solution, the greater the material will be eroded/porous. This is influenced by the greater the percentage of HCL solution, the solution inside the hole will become more concentrated and more acidic. The increasingly acidic pH of the solution will cause the material to become easily eroded/porous.

3.3 Testing SEM

The surface morphology of plate and cylindrical workpieces that were not treated or treated with varying HCL concentrations was observed through SEM images. The results of the SEM images show that workpieces in the form of plates and cylinders that have not received treatment, have experienced slight erosion on the surface. This occurs due to friction between the surface of the workpiece and the grinding stone during the cleaning and polishing process. In Figures 3 and 4 it can be seen that the surface of the plate-shaped workpiece is experiencing erosion on the surface. With the greater concentration of HCL applied to the surface of the workpiece, at an HCL concentration of 5% erosion or pitting corrosion begins to occur in the form of small holes spread over a small area. At an HCL concentration of 10%, more pitting corrosion can be seen on the surface area of the workpiece, the distribution of which is wider.

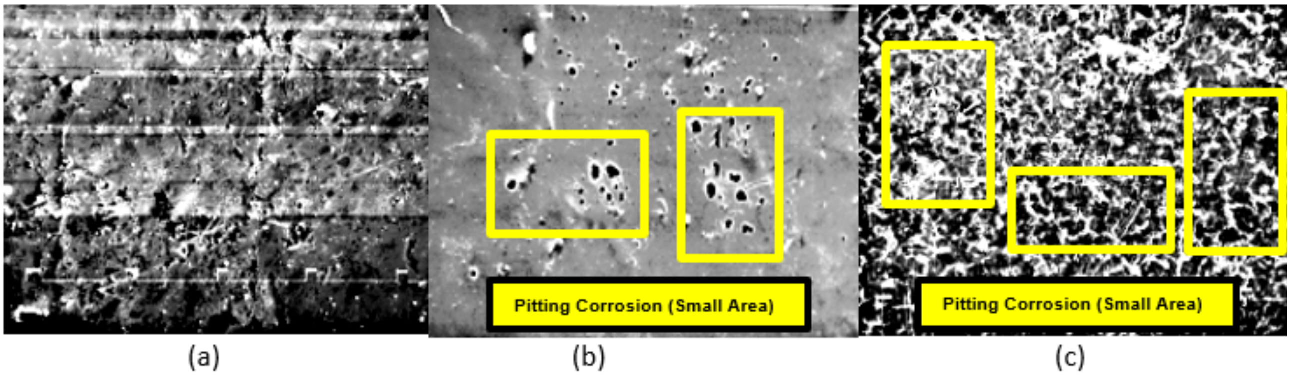

Fig. 3. Plate shape (a) without treatment, (b) 30 minutes blackening coating and 5% concentration HCL immersion, (c) 90 minutes blackening coating and 5% concentration HCL immersion

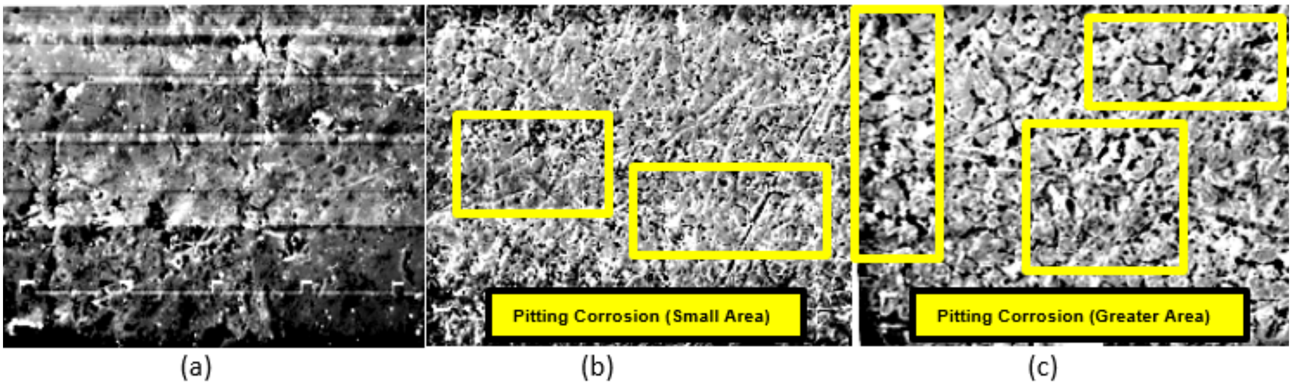

Fig. 4. Plate shape (a) without treatment, (b) 30 minutes blackening coating and 10% concentration HCL immersion, (c) 90 minutes blackening coating and 10% concentration HCL immersion

The cylindrical workpiece can be seen in Figures 5 and 6. The untreated workpiece also shows the surface of the workpiece being damaged as a result of the cleaning and polishing process. With an HCL concentration of 5%, damage to the surface of the workpiece can be seen from pitting corrosion which has formed large holes in a small area. Meanwhile, at an HCL concentration of 10%, the surface of the workpiece already shows a lot of major damage caused by pitting corrosion on a large surface area [19-21].

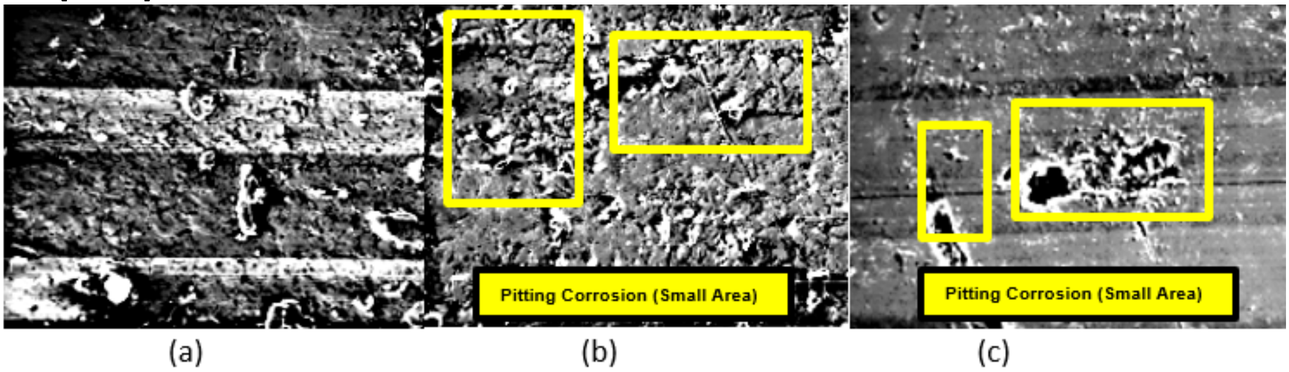

Fig. 5. Cylindrical shape (a) without treatment, (b) 30 minutes blackening coating and 5% HCL concentration immersion, (c) 90 minutes blackening coating and 5% HCL concentration immersion

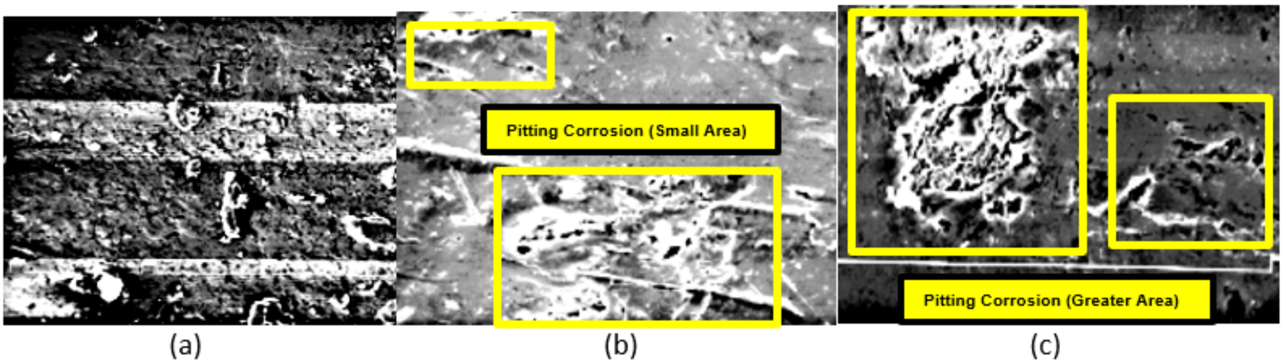

Fig. 6. Cylindrical shape (a) without treatment, (b) 30 minutes blackening coating and 10% concentration HCL immersion, (c) 90 minutes blackening coating and 10% concentration HCL immersion

5. Conclusion

From the research that has been carried out, several conclusions have been drawn, namely:

i. Increasing heating time will affect the thickness distribution and flatness of the surface layer of the workpiece.
ii. The greater the concentration of the HCL solution, the greater the rate of corrosion that occurs and the larger the pitting holes and area of corrosion distribution.

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