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INFLUENCE OF HYDROCHLORIC ACID AGAINST FOOD-GRADE STEEL SUS304 AND ITS CORROSION IMPACT

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Abstract

This study aims to investigate the progress of corrosion rate on stainless steel (SUS304) in hydrochloric acid (HCl) as exposed for 8 days. The HCl as a corrosive medium was used at pH 2 and pH 5. The evaluation of corrosion rate was done through weight loss measurement. Morphology and structure of metal specimens were characterized by optical microscope before and after immersion test. The results showed that the corrosion rates were increased significantly within 4 days of immersion. The corrosion rate in pH 2 reveals the highest values at 0.020 mm/y. While in pH 5, the rate of corrosion exhibits moderate value ranging from 0.006-0.011 mm/y. The corrosion behavior of the stainless steel coupon was influenced by the duration of exposure in the HCl medium. The concentration of hydrogen cations and aggressiveness of chloride anion in the corrosive medium has been associated with corrosion.

Keywords: Corrosion, Immersion test, Weight loss, Stainless steel, Thin oxide

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Introduction

Austenitic stainless steel (SS) is one of the most widely used metallic materials due to its excellent corrosion resistance, good strength, hygienic and aesthetic features. Stainless steel shows a good formability, weldability and so relatively low cost (Dalipi et al., 2016; Oldring & Nehring, 2007; Zaffora et al., 2021). Stainless steel was highly used in the food industry for food containers or food cans (Deshwal & Panjagari, 2020). Unfortunately, local breakdown and pit nucleation inside the food container restrict the corrosion resistance of passive stainless steel in the presence of certain aggressive ions such as chlorides. This affected the organoleptic properties of food and cause toxicological problems. The contaminated canned food may release a sort of acidic chemicals that may harm the human body. In addition, the presence of an imbalance acidic chloride solution although due to a little failure on the food container may generate organism growth at pH 4.9–9.3 (Bintsis, 2017).

Grain boundary corrosion (intergranular corrosion), hole corrosion (crevice corrosion), pitting corrosion, and stress corrosion cracking are all examples of corrosion that are always found on kitchen tools-base stainless steel surfaces (Saefuloh et al., 2020). Stainless steel corrosion arises on two sides; (i) inside of a container, characterized by the metal dissolution of an electrochemical reaction and (ii) outside of a container, due to metal and environmental conditions. Sunlight exposure has been reported to be the effect of corrosion for tinplate cans of various types of beverages as revealed by the amount of Fe metal content (Rahayu & Asmorowati, 2019). A corrosion study for the same metal of green corn shows that the increase of an aqueous chlorides concentration and the pH medium directly influence the increase of the corrosion rate (Reis et al., 2018). Figure 1(a) and (b) shows the minor corrosion on the inner wall of the food container which is affected by an electrochemical reaction of a liquid containing salt. Figure 1(c) represents the outer wall of the food container which shows modest corrosion caused by improper storage perhaps exposed to the debris of salts grain, moisture or acidic liquid in the pantry rack. The design of food containers may be one of the factors for the corrosion attack (Partington, 2006) especially to prevent crevice corrosion. Though, a study on the susceptibility of 304 stainless steel in chloride medium has reported that crevice corrosion was tend to occur at the chloride pH≤5, even the medium tested was below nominal dilutions in beverage industries (Kamaraj et al., 2019).

Figure 1. A sign of corrosion and rusty product occupied on (a) inner wall of food container bases; (b) inner wall on the bending site of the food container (c) the outer wall of food container

Another factor contributing to corrosion inside the food container associated with food packaging is the imbalance pH of food. Acidic food such as fruits and vegetables might cause tin containers to form a layer of corrosion products (Abdel-rahman, 2015). This promotes acute toxicity on the food which has been identified as the cause of gastric pain, nausea, vomiting, diarrhea, damage to the digestive tract and abdominal distress after ingesting the canned food. A review by Zaffora et al. (2021) stated that the presence of chloride ions in fatty food like milk or dairy product may induce the initiation of pitting corrosion. However it is dependent on the composition of the stainless steel itself and the exposed temperature during food production (Zaffora et al., 2021). A comparative gravimetric studies on carbon steel corrosion in selected fruit juices and HCl at different pH has reported that the estimated pH range for the steel to corrode was around pH 2.6-4.24 (Ofoegbu, 2021). The report also found that the presence of a hollow microsphere was observed on the carbon steel even though the juice has capability to inhibit the corrosion process. A study on corrosion resistance of stainless steel 304 types in simulated food contact in an acid medium showed that the iron (Fe) release in citric acid at pH 2.2 was lower than in the combination of chloride anions. The presence of chloride ions cause the pre-passivation of the stainless steel coupon (Mazinanian et al., 2016).

The stainless-steel corrosion was not only affected by the food itself but also impacted by the storage and handling method which depends on the consumer awareness and culture. Figure 2 (a)-(e) shows the typical kitchen appliance that easily suffered corrosion and produces rusty products whenever intended with improper storage. The rusty product on the kitchen tools would reduce the quality of food nutrition and hygiene eventually affecting human health. In this way, the consumer should know how to avoid the corrosive environment to keep the tools clean and free from corrosion.

It is well known that instead of stainless steel as a food container, tin and aluminium are the materials available for food packaging (Ansari et al., 2019). Tin and aluminium are cheap but prone to corrosion. Meanwhile, stainless steel is more resistant to corrosion, however costly as compared to tin and aluminium, therefore it is mainly used for returnable containers in food packaging. Conflicts in managing the cost of materials and health issues are often debated nowadays. Therefore, this paper is significant for a reader to have the fundamental parts of the evolution of stainless steels corrosion in various pH acidic environments. Knowledge from this paper will provide a general idea of the capabilities of stainless steel as an alternative to tin and aluminium. This could help industries related to food-grade stainless steel manufacturers in overcoming the crisis of the cost and health of bringing up stainless steel as preferable food packaging. Other than that, the consumer may learn and notice the impact of the corrosive environment on stainless steel surfaces. Therefore, this knowledge will benefit them to manage stainless steel stuff.

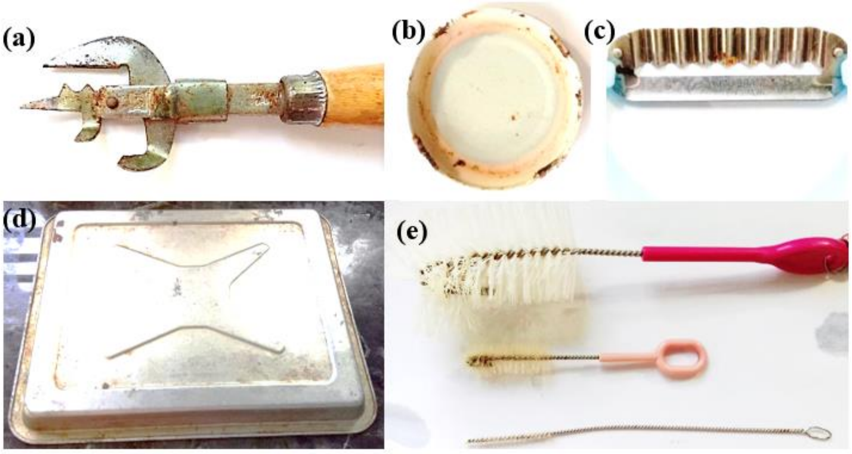


Figure 2. Corrosion and rusty kitchen tools (a) can opener; (b) glass bottle's lid; (c) slice knife; (d) food tray; and (e) milk bottles brush

In this study, the corrosion behavior of stainless steel at different pH of acid solutions was implemented through a method of immersion test and weight loss measurement. Observation of the corrosion effect was carried out to identify the surface morphology effect.

Methods

Metal specimen or coupon preparation

The metal specimen used in this work was stainless steel SUS304. The composition of SUS304 was shown in Table 1. The specimen was cut into a small coupon with an average size of 20 mm x 20 mm and a thickness of 1 mm. Stainless steel coupon was polished through successive grades of abrasive paper. This procedure was performed by using a polishing machine (PLATO E-F Series Grinder/Polishers). The purpose of this process is to clean and remove any oxide layer on the coupon surfaces before immersion in HCl. The samples were then subjected to a cleaning process with ethyl alcohol to remove dirt and any grease or oil followed by a dried-up process. Then, the initial weight was recorded for each sample. To avoid atmospheric corrosion, the prepared specimens were stored in desiccators or dry cabinets. A triplicate coupons samples were prepared for each test and subjected to different pH of corrosive medium.

Table 1. Chemical composition of stainless-steel coupon SUS304

Chemical components	Content (%)
C	0.042
Si	0.470
Mn	0.970
P	0.029
S	0.004
Ni	8.090
Cr	18.060
N	0.058
Cu	0.020
Fe	72.260

Preparation of corrosive medium

In a corrosive medium, 0.1 M HCl solution was prepared at different acidity; pH 2 and pH 5. The adjustment of pH was carried out by using a 0.1 M NaOH solution. In this experiment, a corrosive medium at pH 2 and 5 were represented as the pH of acidic food or juice in a food can.

Total-immersion tests

For total-immersion tests, 250 mL different pH of the corrosive medium was filled in the 500 mL beakers. The samples were exposed to corrosive medium throughout different immersion times 2, 4, 6, and 8 days at room temperature (25±2°C). During the tests, the concentration was kept constant throughout the immersion phase. The immersion test was performed as standard (ASTM G 31-72, 1985) with a slight modification of the parameter.

Weight loss measurement

At the end of each test cycle, samples were withdrawn and washed with tap water, distilled water and ethanol. After drying, the samples were weight out to measure the weight loss. The method of cleaning, drying, and measuring weight loss was repeated subsequently as typically done by other researchers (ASTM G1-90, 1999). To obtain the weight loss value, the difference between initial weight and final weight was calculated as shown in Eq. (1).

$$\text{Weight Loss (W)} = W_1 - W_2 \qquad \text{Eq. (1)}$$

Where:
W = metal weight loss (milligrams)
W_1 = initial weight (milligrams)
W_2 = final weight (milligrams)

The corrosion rate was calculated as in Eq. (2).

$$\text{Corrosion rate (mm/y)} = 87.6 \text{ x } (W/DAT) \qquad \text{Eq. (2)}$$

Where:
W = weight loss in milligrams
D = metal density in g /cm^3
A = area of the sample in cm^2
T = time of exposure of the metal sample in hours
87.6 = corrosion constant factor

Morphology

The surface morphology of the specimens was performed by using an optical microscope (Olympus CX22 Microscope Series) before and after the removal of the corrosion product. Observation of the morphology was done to investigate the evolution of corrosion as exposed to HCl within 8 days. The coupon image was captured at 4x magnification.

Result and Discussion

Corrosion rate analysis

Table 2 shows the weight loss and corrosion rate values for a coupon after immersion in 0.1 M HCl of pH 2 and pH 5 at various times. The immersion test in pH 2 shows a significant increase in corrosion rate from 0.009 mm/y to 0.02 mm/y within 2 to 4 days respectively. It is noticeable that the corrosion rate was then decreased to 0.013 mm/y after 6 days of immersion. However, immersion time up to 8 days caused the coupon to lost its weight and the corrosion rate become more reduced to 0.006 mm/y.

The high corrosion rate at 4 and 6 days immersion compared to 2 days, was due to the increased in the chloride concentration in the solution as immersion time was extended. The decrease in corrosion rate for 8 days of immersion was probably due to the formation of the thin layer of oxide film that may prevent further surface dissolution.

The immersion test in pH 5 shows moderate values of corrosion rate, 0.011 mm/y at 2 days immersion. Further immersion for up to 4 days shows an increase in weight loss, however the corrosion rate remains constant. An increase in immersion time up to 6 and 8 days caused a decrease in the corrosion rate value. The formation of the passive film and thin oxide layers was the possible reason for the decrease in corrosion rate. In some cases, however, a precipitation of oxide from the electrolyte can contribute to passivation (Bard & Stratmann, 2003).

Table 2. Weight loss and corrosion rate for pH 2 and pH 5

Immersion Time, days	**Corrosive medium**			
	pH 2		**pH 5**	
	Weight loss (g)	**CR (mm/y)**	**Weight loss (g)**	**CR (mm/y)**
2	0.0003	0.009	0.0004	0.011
4	0.0014	0.020	0.0008	0.011
6	0.0014	0.013	0.0006	0.006
8	0.0009	0.006	0.0009	0.006

The oxidation and reduction processes were the mechanism of stainless steel corrosion. The two half-cell reactions below show the oxidation (Eq. 3) and reduction (Eq. 4) process as typically happened in an acid environment (Sastri et al., 2007):

$\mathrm{Fe} \rightarrow \mathrm{Fe}^{2+} + 2e^{-}$ Eq. (3)
$2\mathrm{H}^{+} + 2e^{-} \rightarrow \mathrm{H}_2$ Eq. (4)

The overall chemical reaction when stainless steel was exposed to HCl is shown in Eq. (5):

$\mathrm{Fe} + 2\mathrm{H}^{+} \rightarrow \mathrm{Fe}^{2+} + \mathrm{H}_2$ Eq. (5)

The general mechanism of corrosion attack in hydrochloric acid also can be summarized in Eqs. (6)-(9) (Singh et al., 2010)

$\mathrm{Fe} + \mathrm{Cl}^{-} \leftrightarrow (\mathrm{FeCl}-)_{\mathrm{ads}}$ Eq. (6)

$(\mathrm{FeCl})_{\mathrm{ads}} \leftrightarrow (\mathrm{FeCl})_{\mathrm{ads}} + e-$ Eq. (7)

$(\mathrm{FeCl})_{\mathrm{ads}} \rightarrow (\mathrm{FeCl}^{+}) + e-$ Eq. (8)

$(\mathrm{FeCl}^{+}) \leftrightarrow \mathrm{Fe}^{2+} + \mathrm{Cl}-$ Eq. (9)

In the electrochemical process, chloride ions simply serve as a medium or catalyst. The presence of chloride anions (Cl^{-}) in the solution may aid in the removal of metal cations (Fe^{2+}) deposited and adsorb (ads) on the anode by creating soluble compounds, resulting in a rapid anodic reaction and, as a result, speeding rusting of the metals (May, 2016).

Figure 3 shows a graph of the corrosion rate against immersion time measured at different pH of HCl. The results indicate that the corrosion rate of coupon in pH 2 has increased drastically until day-4. The values were then decreased dramatically after immersion 6 to 8 days. Immersion in pH 5 shows a moderate corrosion rate with a fluctuated value. It is noticeable that at immersion for 6 and 8 days, the corrosion rates were at the lowest value for all pH tested. Generally, the longer the immersion time in acid solution, the higher the corrosion rate of the coupon. However, in this study, the results show different. The longer the immersion time, the rate of corrosion was reduced after 4 days and remained constant until 8 days of exposure. The corrosion products produced from the steel dissolution were believed to form a thin passive layer, thus reducing the rate of corrosion.

In this study also the unsteady data obtained may due to the limitation of the weight loss method, uncertainty in measurement and human error as generally reported by others (Anthony et al., 2016; Satpati et al., 2021; Wade & Lizama, 2015).

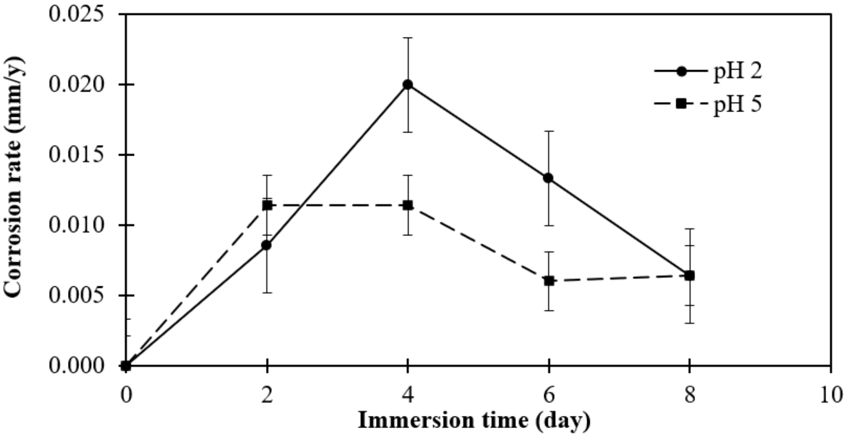


Figure 3. The variation of corrosion rate against immersion time for stainless steel exposed to acid solution in different pH

Morphology analysis

Figure 4 represents the morphology surface of stainless steel before and after immersion in HCl for 8 days at pH 2. A coupon before immersion shows a smooth surface without any major degradation (Figure 4(a)). Even though a little scratch formed during coupon preparation (polishing), however in general, there was no major corrosion sign observed.

Meanwhile a coupon in pH 2, shows the effect of pitting corrosion as marked in the Figure 4(b). The pits were localized and distributed unevenly on the entire specimen. The large pits were developed along with the scratch. It is believed that the pits were initiated from the scratch, then propagated due to the action of Cl^- ions (Ait Albrimi et al., 2015; Osman, 2001; Talebian et al., 2019). The corrosion sign observed indicates the high corrosion rates. Furthermore, the high chloride content and as a result, the low oxygen concentration may accelerate corrosion and cause the pit to form. The presence of chloride ions in the bulk solution causes an increase in acidity inside the pit formation, which promotes corrosion.

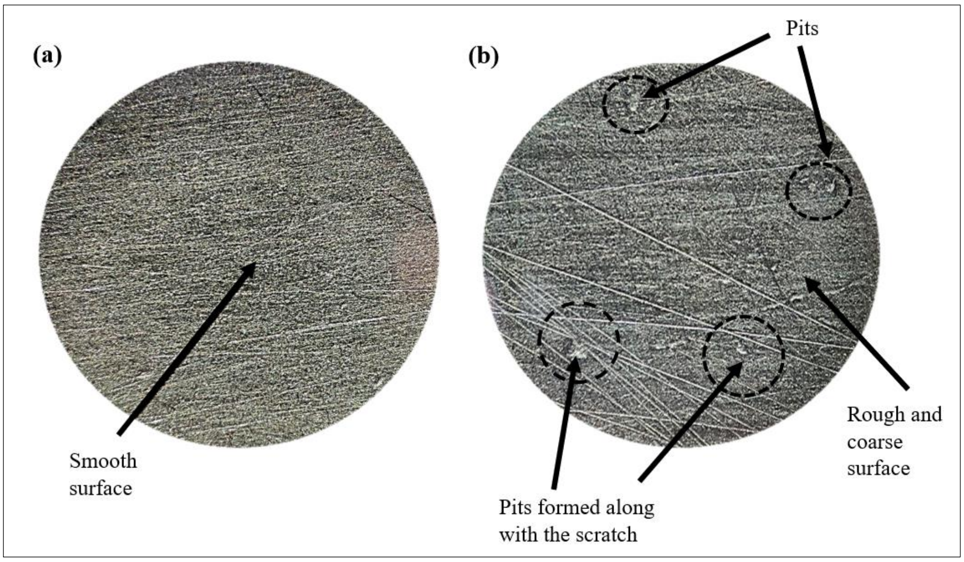
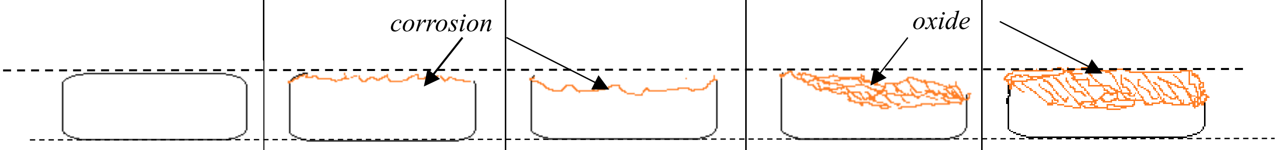


Figure 4. The surface morphology of (a) specimen before immersion test and (b) corroded specimens after immersion test in pH 2 at day-8

Figure 5 summarize the corrosion behavior of stainless steel in pH 2 at different immersion time. The evolution of coupon size has been illustrated through quantitative weight loss data and this explains the corrosion was influenced by immersion time. The size, as well as weight of the coupon might be changed due to degradation of surface (either corrode or growth of oxide) as it contacts with the corrosive medium. As long duration of the immersion, the acidic medium may have a greater quantity of hydrogen ions than hydroxyl ions. Hydroxyl ions were presents due to the hydrolysis of metal cations with water. Meanwhile, the excess hydrogen ions caused in balancing the cathodic reaction, are inconsequence to the evolution of hydrogen. Therefore, the steel coupon can corrode more rapidly as both the hydrogen ions and the hydrogen gas can diffuse very quickly (Bard & Stratmann, 2003).

Instead of pH, oxygen, solution chemistry, temperature, fluid flow rate, immersion time and metallurgy of the steel also influence the physical changes and formation of passive films (Eyu et al., 2016). Typically, iron(III) and chromium(III) oxides were the most oxide presence on the stainless steels which exposed to an acidic environment at ambient temperature (Loto, 2017). Loto 2017 also reported that the formation of visible micro and macro-pits on the SS304 steel type in the study was due to the electrolytic action of Cl^- ions. The stainless steel surface was slightly deteriorated due to the autocatalytic process.



0 day	2 days	4 days	6 days	8 days
Stainless steel coupon with no corrosion	Moderate change in weight. Modest corrosion rate value. Little pitting corrosion	Significant weight change. High corrosion rate. Pitting corrosion	Significant weight change. Modest corrosion rate. Oxide layer has formed ($Fe(OH)_3$)	Moderate weight change. Low corrosion rate value. Stable oxide with pitting corrosion has formed (Fe_2O_3, $FeCl_2.4H_2O$)

Figure 5. Schematic representation of the corrosion behaviour of stainless steel in pH 2 at different immersion times.

Conclusion

In conclusion, the corrosion behaviour of stainless steel can be influenced by all the parameters studied especially time exposure. The stainless steel exhibited corrosive behaviour in pH 2 compared to pH 5 due to the aggressiveness of hydrogen cations and chloride anions in more acidic acid. Besides, the longer the immersion or exposure to the acid solution, the higher the corrosion rate. However, in this study, the stainless steel shows active-passive phenomena along with the 8 days immersion. Both pHs of the corrosive medium have reduced the corrosion process ont day 6 and continue to corrode the stainless steel surface after 6 days of exposure. Pitting corrosion was formed on the stainless steel surface due to the presence of Cl^- ions in the acid solution. It is necessary for a consumer to increase an awareness of the proper way to manage a stainless steel-based tools. For instance, avoid any contact with stainless steel stuff from an acidic environment, keep away from chloride agent (for example salt), prevent moisture and avoid remaining food in an opened stainless steel container for long periods. This is important to prevent food from being contaminated perhaps affecting health.

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Author Contribution

Nur Dhiya Syariah Mazelam – Investigation, Writing Original Draft; Solhan Yahya – Supervision, Funding Acquisition, Writing - Review & Editing; Zaidi Ab. Ghani – Conceptualization, Validation, Zuliahani Ahmad – Visual, Writing - Review & Editing; Dalina Samsudin – Visualization, Writing - Review & Editing; Nabilah Akemal Muhd Zailani – Visualization, Writing - Review & Editing.

Conflict of Interest

Author declares no conflict of interest.

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