

Effects of Carbon Film and Chloride Ions on the Pitting Corrosion of Copper in Anion Exchange-Treated Water Containing Water Treatment Chemicals

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Abstract

Copper tubes are widely used in heat exchangers for air conditioning units and refrigerators. Carbon film formed on the inner surface of copper tubes during manufacturing have been reported to cause carbon-film-dependent pitting corrosion interaction with the quality of the water flowing in the tube. In field applications phosphonic acid (HEDP) and benzotriazole (BTA) are used to suppress this pitting corrosion. Anion exchange-treated water, produced by passing tap water through anion exchange resin to replace corrosive ions (Cl^- and SO_4^{2-}) with corrosion inhibiting ions (HCO_3^-), it has also been reported to improve corrosion resistance when combined with these water treatment chemicals. In this study, the effects of chloride ion concentration and carbon film on the corrosion resistance of copper in anion exchange-treated water containing water treatment chemicals were investigated. Corrosion potential and anodic polarization curves were measured in anion-exchange treated water containing 10 mg/L of HEDP and 5 mg/L of BTA, at various chloride ion concentrations. Copper tube specimens with residual carbon amounts of 0 mg/m² and 5 mg/m² were used. Corrosion potential measurements show that the corrosion potential decreased and corrosion was occurred in the presence of carbon film at a chloride ion concentration of 100 mg/L. Anodic polarization curve measurement showed that the breakdown potential shifted to the less noble potential side as the chloride ion concentration increased. These results indicate that, in anion-exchange-treated water

containing water treatment chemicals, corrosion is more likely to occur as the Cl^- concentration increases. Furthermore, the presence of a carbon film is considered to enhance this tendency.

Keywords

Copper, Carbon Film, Pitting Corrosion, Anion Exchange-Treated Water, Chloride Ion, Benzotriazole, Phosphonic Acid

1. Introduction

Copper tubes are widely used in heat exchangers for air conditioning units and refrigerators because of their high thermal conductivity, excellent workability and high corrosion resistance. However, pitting corrosion associated with carbon films on the inner surface of the copper tubes has become a problem [1]-[3]. The carbon film forms on the inner surface of copper tubes during the manufacturing process. Pitting corrosion caused by the interaction between the carbon film and the quality of water flowing water inside copper tubes. is known as carbon film-dependent pitting corrosion. Corrosion inhibitors such as phosphonic acid (HEDP) and benzotriazole (BTA) have been reported to be effective in suppressing this type of pitting corrosion [4] [5]. In addition, calcium ions (Ca^{2+}), silicate ions (SiO_4^{4-}), and bicarbonate ions (HCO_3^-) in solutions containing these corrosion inhibitors have been reported to prevent pitting corrosion [6] [7]. Tap water is commonly used as makeup-water for circulating water in air conditioning systems. It contains various ions, including Ca^{2+} , SiO_4^{4-} , chloride ions (Cl^-), sulfate ions (SO_4^{2-}), and bicarbonate ion (HCO_3^-). Previous research has shown that Cl^- and SO_4^{2-} are corrosive whereas HCO_3^- inhibits corrosion.

This study focused on the corrosivity of water obtained by removing corrosive ions as Cl^- and in tap water using an anion exchange resin. This process uses an anion exchange resin regenerated with solutions containing sodium bicarbonate (NaHCO_3) and sodium carbonate (Na_2CO_3) [8]-[10]. Anion exchange treated water (treated water) was obtained by passing tap water through the anion exchange resin whereby SO_4^{2-} and Cl^- ions in tap water are replaced with HCO_3^- ions. We previously reported that treated water containing HEDP and BTA inhibits pitting corrosion of copper tubes [11]. However, these corrosion prevent effects are considered to depend on both the water quality of the treated water and the surface conditions of the copper tube and these influencing factors have not been sufficiently clarified. In particular, chloride ions are known to promote pitting corrosion of copper, and may interact with carbon film on the copper tube surface, thereby influencing pitting corrosion behavior [12]-[14].

In this study, the effect of Cl^- in treated water containing HEDP and BTA on copper corrosion was investigated. The solutions with different Cl^- concentrations were copper tube specimens with and without residual carbon film was im-

mersed in the test water, and the corrosion potential was measured over time. After the corrosion potential measurements, the surface appearance of the specimens was observed. In addition, anodic polarization curves of the test specimens in each test solution were measured.

2. Methods

2.1. Materials

Table 1 shows the dimensions and residual carbon amount of test specimens. Phosphorus-deoxidized copper tubes (JIS H 3300 C1220, OL) were used in this study. Copper tubes with and without carbon film were prepared. The amount of residual carbon was measured using the rapid filling test [15]. As a result of the measurement, the residual carbon amount was 5.5 mg/m². The copper tubes were cut parallel to the tube axis and used as test specimens. The copper tube with the carbon film was designated as Copper tube A. Copper tube A was cleaned with 25 mass % nitric acid to remove the film, and the resulting specimen was designated as Copper tube B. during cleaning with 25 mass % nitric acid, black material was observed to detach from the surface, suggesting that the carbon film had been removed. Therefore, the amount of residual carbon on Copper tube B was considered to be 0 mg/m². Before the testing, Copper tube A and Copper tube B were acid-cleaned with 5 mass % sulfuric acid to remove the oxide film formed on their surface.

Table 1. Sample size and residual carbon.

Sample	Size	Residual carbon
Copper tube A	Length: 100 mm ×Inner diameter: 15.88 mm	5.5 mg/m ²
Copper tube B	Length: 100 mm ×Inner diameter: 15.88 mm	0 mg/m ²

2.2. Test Solutions

The test solutions used Tsukuba City water treated with anion exchange treatment (treated water). **Table 2** shows the water quality of the test solutions. The pH and electrical conductivity of all test solutions were measured before the test using a glass electrode and two platinum electrodes coated with platinum black, respectively. The treated water corresponded to the 0 mg/L Cl⁻ solution, and its alkalinity (pH 4.8), SiO₂, calcium hardness, SO₄²⁻, Cl⁻, and NO₃⁻ concentrations were measured. The alkalinity (pH 4.8) was determined by titration using a bromocresol green-methyl red ethanol solution and sulfuric acid. The concentration of SiO₂ was quantitatively determined by the molybdenum yellow spectrophotometric method. The concentrations of calcium hardness, SO₄²⁻, Cl⁻, and NO₃⁻ were determined by ion chromatography. The test solutions containing 10, 30, 50, and 100 mg/L Cl⁻ were prepared by adding NaCl, 10 mg/L HEDP and 5 mg/L BTA to the treated water. Hereafter, Cl⁻ concentrations of 0, 10, 30, 50, and 100 mg/L are

referred to as Cl^- 0 mg/L, Cl^- 10 mg/L, Cl^- 30 mg/L, Cl^- 50 mg/L, and Cl^- 100 mg/L, respectively.

Table 2. Water quality of the test solutions.

Species	Unit	Test solutions				
		Cl^- 0 mg/L	Cl^- 10 mg/L	Cl^- 30 mg/L	Cl^- 50 mg/L	Cl^- 100 mg/L
pH (25°C)	-	8.4	8.8	8.6	8.4	8.7
Electrical conductivity	mS/m	24	26	22	34	54
Ca hardness	mg CaCO_3/L	49	49	49	49	49
Alkalinity (pH 4.8)	mg CaCO_3/L	147	147	147	147	147
Cl^-	mg/L	<0.3	10	30	50	100
SO_4^{2-}	mg/L	<0.2	<0.2	<0.2	<0.2	<0.2
NO_3^-	mg/L	<0.1	<0.1	<0.1	<0.1	<0.1
SiO_2	mg/L	3	3	3	3	3
HEDP	mg/L	10	10	10	10	10
BTA	mg/L	5	5	5	5	5

2.3. Corrosion Potential Measurement

A schematic drawing of the test apparatus is illustrated in **Figure 1**. A 500 mL beaker was used as the test vessel. The test solution was added to a volume of 500 mL and stirred at 300 rpm under room temperature. Furthermore, this test was conducted under open-air conditions to evaluate changes in the corrosion potential of copper in the test environment. The immersion period was 5 days, and the corrosion potential was measured over time. The corrosion potential was measured using a digital multimeter (Keysight: 34070A). As the reference electrode used saturated Ag/AgCl (SSE). The SSE was immersed only during measurement to avoid the effects of Cl^- leaching from the SSE. The test area was set to 3 cm². The measurements were performed triplicate. After testing, the specimens surface were observed using a digital microscope (KEYENCE: VHX-5000), and their an composition was analyzed using SEM equipped with EDX (Hitachi High-Tech-nologies: S-4300).

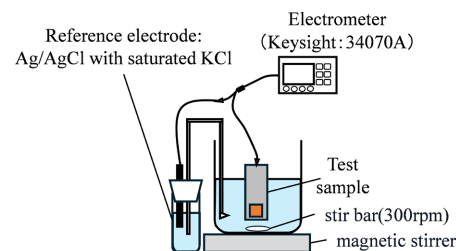


Figure 1. Schematic of corrosion potential measurement apparatus.

2.4. Anodic Polarization Curve Measurement

A schematic drawing of the test apparatus is shown in **Figure 2**. A 500 mL beaker was used as the test vessel. The test solution was added to a volume of 500 mL and stirred at 300 rpm and under room temperature. The measurement was conducted by deoxidizing the test solution using nitrogen gas. The purpose of this was to remove the effects of oxygen and evaluate the reaction of copper under the test environment. The polarization curve was measured using a potentiostat (HZ-7000; Hokuto Denko, Tokyo, Japan). The measurement was conducted by deoxidizing the test solution using nitrogen gas. The sweep speed was set to 20 mV/min. The specimens were used as the working electrode, a saturated Ag/AgCl electrode (SSE) as the reference electrode, and platinum as the counter electrode. The test area was set to 1 cm². Measurements were performed in triplicate.

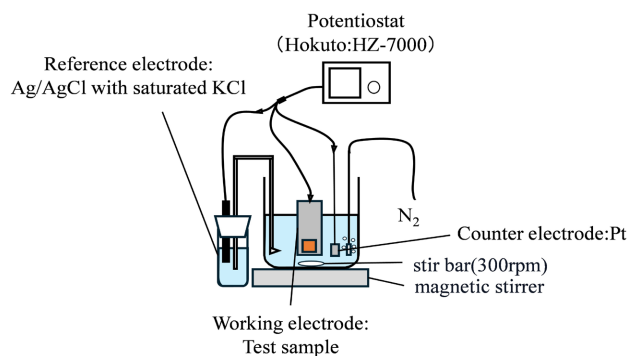


Figure 2. Schematic of anodic polarization curve measurement apparatus.

3. Results and Discussion

3.1. Results of Corrosion Potential Measurements

Results of the corrosion potential measurement are shown in **Figure 3** and **Figure 4**. This shows the average of three measurements. About the Copper tube A (residual carbon: 5.5 mg/m²), the corrosion potential increased to +0.07 V vs. SSE after immersion and then tended to stabilize. However, in the Cl⁻ 100 mg/L solution, the potential continued to decrease even after one day of immersion. The final measurement potential increased in the following order: Cl⁻ 100 mg/L < Cl⁻ 10 mg/L < Cl⁻ 30 mg/L < Cl⁻ 0 mg/L < Cl⁻ 50 mg/L. About the Copper tube B (residual carbon: 0 mg/m²) the corrosion potential increased to +0.07 V vs. SSE after immersion and continued to increase thereafter. After two days of immersion, the potential tended to stabilize for Cl⁻ 0 mg/L and 10 mg/L, whereas it tended to decrease slightly for Cl⁻ 30 mg/L, 50 mg/L, and 100 mg/L. The final measurement potential increased in the following order: Cl⁻ 30 mg/L < Cl⁻ 50 mg/L < Cl⁻ 100 mg/L < Cl⁻ 10 mg/L < Cl⁻ 0 mg/L. At each Cl⁻ concentration, the corrosion potential of Copper tube B tended to be higher than that of Copper tube A.

Copper tube A had a carbon film on its inner surface. Yamada *et al.* reported

that carbon film form unevenly on the inner surface of copper tubes [1]. It has also been reported that the carbon film-covered areas act as cathodic sites, whereas exposed copper substrate areas act as the anodic sites [3]. Based on these findings, the initial increases in corrosion potential observed for Copper tube A is attributed to the promotion of the cathodic reaction by the carbon film. As the cathodic reaction proceed, an oxide film forms on the surface, causing the change in corrosion potential became gradual. In the Cl^- 100 mg/L, Cl^- is considered to damage the film, leading to corrosion and a decrease in the corrosion potential. In contrast, because copper tube B, has no carbon film, the corrosion reaction proceeds slowly and the change in potential was also more gradual.

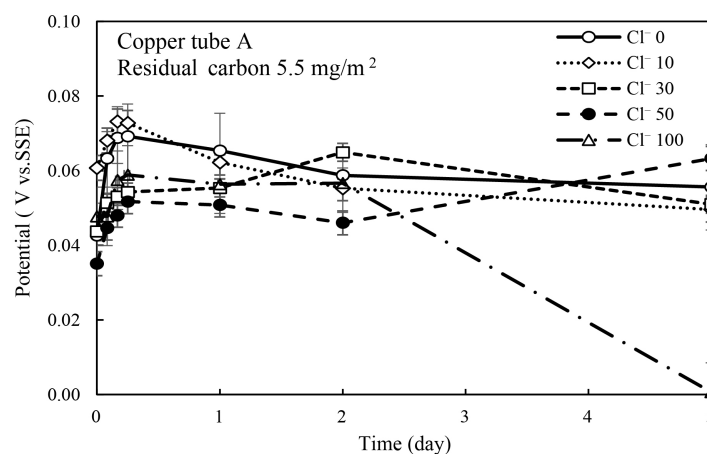


Figure 3. Time dependent changes in the corrosion potential of Copper tube A in test solutions. (stirred at 300 rpm, under room temperature, open-air conditions).

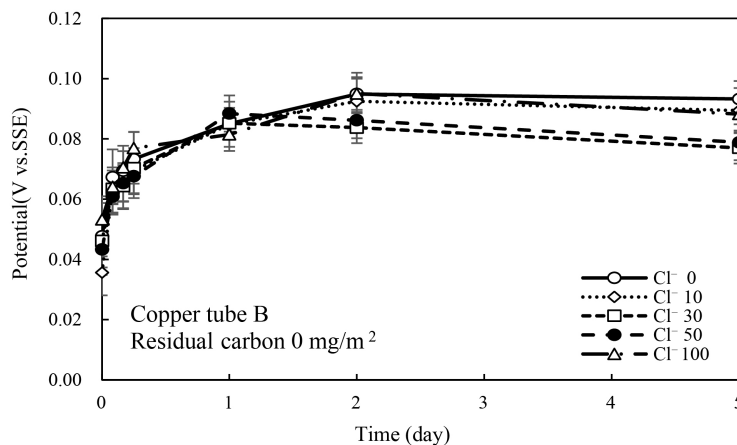


Figure 4. Time dependent changes in the corrosion potential of Copper tube B in test solutions. (stirred at 300rpm, under room temperature, open-air conditions).

3.2. Surface Observation and Analysis

Figure 5 shows the results of the observation and analysis of the test surface after corrosion potential measurement. Corrosion was only observed on Copper tube A at a Cl^- 100 mg/L. **Figure 6** shows SEM images and the EDX analysis results for

areas with and without corrosion products. In the corrosion products, 2.8 mass % Cl was detected, Cl is known to promote pitting corrosion of copper and Yamada *et al.* and Kano *et al.* reported that Cl^- was detected in pitted areas [12] [13]. Based on these results, the corrosion observed on copper tube A in the Cl^- 100 mg/L solution is considered to be the first stage of pitting corrosion.

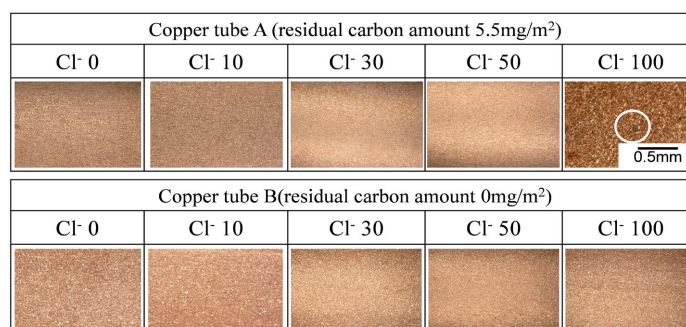


Figure 5. The surface images of Copper tubes A and B after measurement the corrosion potential. ○: The area where corrosion was observed.

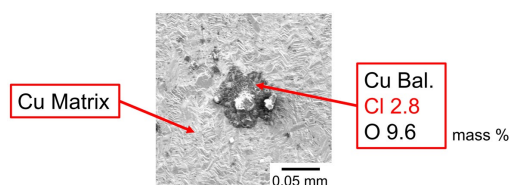


Figure 6. SEM image and the EDX analysis of Copper tube A at a Cl^- 100 mg/L after measurement of corrosion potential with test water.

3.3. Results of Anodic Polarization Curve Measurement

The anodic polarization curves are shown in **Figure 7** and **Figure 8**. For Copper tube A, the current density increased sharply from -0.1 V vs. SSE and then changed gradually under all test conditions. Subsequently, the current density increased sharply again at approximately $+0.1$ to $+0.2$ V vs. SSE. The potential at which the second sharp increase was observed was defined as the break down potential (BDP). The BDP was defined as the potential at the intersection of the tangent to the increasing section where the current density began to increase again and the line extended from the immediately preceding section of gradual current density increase [7]. **Figure 9** shows the BDP under each test condition. The BDP tended to shift to the cathodic side as the Cl^- concentration increased. For Copper tube B, the current density increased sharply from around 0 V vs. SSE under all test conditions, following by gradual change. The current density then increased sharply again approximately $+0.1$ to $+0.3$ V vs. SSE. Similar Copper tube A, the BDP tended to shift to less noble potential with increasing the Cl^- concentration. Comparison of the BDP values of Copper tube A and Copper tube B shows that, Copper tube B tended to exhibit higher values than Copper tube A, irrespective of the Cl^- concentration. These results suggest that ‘in environments with high

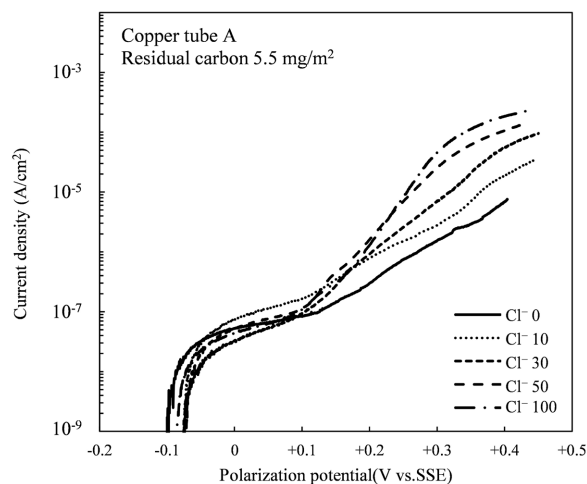


Figure 7. Anodic polarization curves of copper tube A in test solutions (sweep speed 20 mV/min, stirred at 300 rpm, under room temperature, deoxidizing by nitrogen gas).

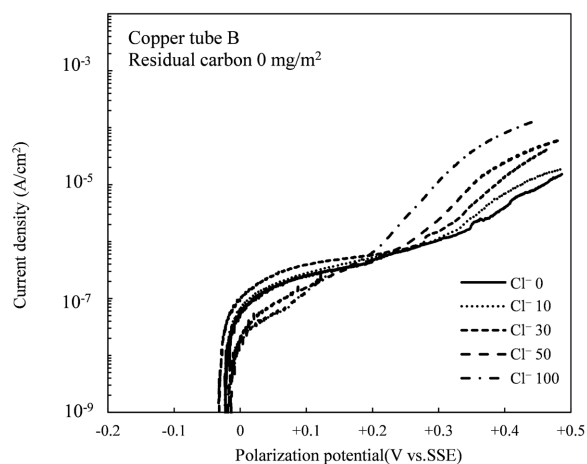


Figure 8. Anodic polarization curves of Copper tube B in test solutions (sweep speed 20 mV/min, stirred at 300 rpm, under room temperature, deoxidizing by nitrogen gas).

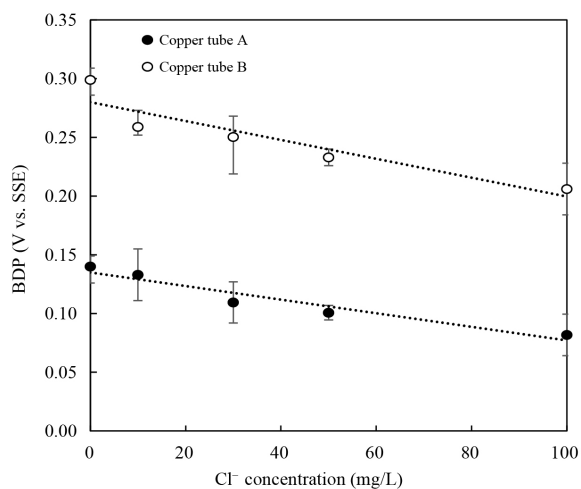


Figure 9. Breakdown potential of copper tubes with different amounts of residual carbon at various chloride ion concentrations.

Cl^- concentrations and the presence of a carbon film, the oxide film is prone to degradation’.

3.4. Effect of Cl^- in Anion Exchanged-Treated Water

In the corrosion potential measurements, the corrosion potential of Copper tube A increased sharply immediately after immersion, after which the change in potential became gradually. However, at a Cl^- concentration 100 mg/L, the potential continued to decrease. After testing, corrosion was observed on the surface of Copper tube A at a Cl^- concentration 100 mg/L. In the anodic polarization curve measurements, the BDPs of both Copper tubes A and B shifted to less noble potential as the Cl^- concentration increased. These results suggest that, in treated water containing water treatment chemicals, the higher the Cl^- concentration, the more likely copper corrosion is to occur. It is thought that the presence of a carbon film makes corrosion even more likely to occur.

Takahashi *et al.* reported effect of Cl^- on pitting corrosion of copper in artificial water [16]. Table 3 summarizes their results and the findings obtained in this study. In artificial water, pitting corrosion was observed at a Cl^- concentrations 10 mg/L. In contrast, in treated water with high concentrations of bicarbonate ions, no pitting corrosion was observed up to a Cl^- concentration of 100 mg/L for copper tubes without carbon film. Even in the presence of a carbon film, pitting corrosion was suppressed up to a Cl^- concentrations of 50 mg/L. Based on these results, it is considered that in treated water containing water treatment chemicals, corrosion is suppressed in the presence of carbon film until the Cl^- concentration reaches to 50 mg/L. In the absence of carbon film, corrosion is suppressed at a Cl^- concentration of 100 mg/L. Furthermore, the mechanism of pitting corrosion in treated water containing chloride ions and water treatment chemicals was investigated. In the presence of a carbon film, cathodic reactions are promoted in the area surrounding the carbon film. This reaction forms copper oxide films and composite films of HEDP and BTA [5]. The carbon film exists unevenly, resulting in uneven areas where cathodic reactions occur. As a result, the copper oxide films and composite films may not form uniformly, leading to an unstable film. This unstable film is considered to be more easily broken down by Cl^- . In contrast, on copper tubes without a carbon film, uniform copper oxide and HEDP/BTA

Table 3. Effect of Cl^- concentration on copper corrosion resistance in the previous study by Takahashi *et al.* and in the present study.

Water type	Test water			Carbon film	Cl^- concentration				
	HCO_3^-	HEDP	BTA		0	10	30	50	100
Artificial water	0	10	5	Present	×	×	-	-	×
Artificial water	0	10	5	Absent	○	×	-	-	×
Treated water	179	10	5	Present	○	○	○	○	×
Treated water	179	10	5	Absent	○	○	○	○	○

○ indicates that pitting corrosion was not observed; × indicates that pitting corrosion was observed.

composite films are likely to form. In addition, treated water contains a high concentration of HCO_3^- , which provides pH buffering capacity. These conditions are considered to promote the formation and maintenance of a stable protective film. Consequently, the film is not easily damaged and corrosion is suppressed, even in environments containing Cl^- . In addition to differences in residual carbon, changes in surface condition resulting from nitric acid pretreatment might also have influenced these results. The effects of nitric-acid pretreatment on the surface condition remain to be investigated.

4. Conclusions

In this study, the effect of different Cl^- concentrations and carbon film on pitting corrosion of copper in anion exchange treated water containing HEDP 10 mg/L and BTA 5 mg/L were investigated. The results are summarized as follows.

(1) In the corrosion potential measurements, the corrosion potential of Copper tube A (residual carbon amount: 5.5 mg/m^2) increased sharply immediately after immersion, after which the change in potential became gradual. At a Cl^- concentration of 100 mg/L, however, the potential continued to decrease. Copper tube B (residual carbon amount: 0 mg/m^2), showed a gradual change in corrosion potential, and it showed higher potential than Copper tube A. After corrosion potential measurements, corrosion was observed on Copper tube A (residual carbon amount: 5.5 mg/m^2) at a Cl^- concentration of 100 mg/L.

(2) In the anodic polarization curve measurements, the break down potential (BDP) tended to shift toward the less noble potential as the Cl^- concentration increased, regardless of the presence or absence of a carbon film. Furthermore, the comparison of BDP values at various Cl^- concentrations showed that at each concentration, Copper Tube A tended to be lower BDP than Copper Tube B.

(3) These results suggest that, in anion-exchange treated water containing water treatment chemicals, higher the Cl^- concentrations, the more likely copper corrosion is to occur. It is thought that the presence of a carbon film makes corrosion even more likely to occur. These results were obtained under the HEDP and BTA concentration and water quality conditions in this study. If these conditions differ, the effects on corrosion could be different. Therefore, further investigations under various water quality conditions are required.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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