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ORIGINAL ARTICLE

Copper Dissolution Rate of Modified Self-Polishing Antifouling Paint with Cerium Oxide

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ABSTRACT – The main objective of the work is to substitute SeaNine 211 in self-polishing antifouling (SPA) paint formulations for marine coating using cerium oxide as a co-biocide. The development of SPA paint with cerium oxide as a co-biocide is expected to significantly improve the environmental friendliness and efficacy of antifouling paints. The preparation of modified SPA paint was undertaken by incorporating the cerium oxide with various concentrations (0.1, 0.3, and 0.5 wt.%) in the SPA paint formulation without SeaNine 211. The copper dissolution rate determines the efficacy of antifouling paint, where the greater the copper release rate, the greater the efficacy of the antifouling paint. The leached-out test results obtained suggest that with an increase of 0.1% wt in the concentration of cerium oxide, there was an improvement in the copper dissolution rate. It was also observed that the mild steel specimens were coated with modified SPA paint when immersed in the seawater for 7 days for testing. The properties were observed by utilizing scanning electron microscopy (SEM). An improvement in the copper dissolution rate was found, as shown by cerium oxide SPA paint when contrasted with bare paint. The contact angle test finding also suggests that adding 0.1% wt of cerium oxide to the SPA paint will increase the dissolution rate of copper. This work could provide insight into developing an environmentally friendly antifouling paint co-biocide.

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INTRODUCTION

Biofouling refers to the growth of undesirable organisms on artificial structures immersed in water [1], [2]. Biofouling is a worldwide concern in the maritime shipping industry because it has a significant impact on ship performance, power demands, fuel consumption, and total shipping costs [3], [4]. For instance, a fouled hull grows rougher during the biofouling process. The resulting roughness causes increased drag, which results in either increased fuel usage or decreased speed [5]. According to some data, biofouling increases fuel usage by about 10%. It is estimated that the shipping industry spends approximately USD $30 billion annually to prevent and control biofouling problems. Besides technical and economic challenges, ship biofouling has an environmental impact since it increases carbon emissions and the potential spread of invasive foreign species worldwide [6]. Further, biofouling can degrade material performance, causing corrosion on metallic surfaces [7].

Antifouling is the technique of preventing or reducing the accumulation of biofouling on the substrate. Commercial antifouling methods include mechanical cleaning, biocides, toxic antifouling paints, and foul-release coatings. Among the foregoing, antifouling paints with hazardous compounds are the main methods employed against biofouling [1]. Antifouling paints containing tributyl tins (TBT) have significantly improved the shipping industry's economy [8]. Unfortunately, the use of TBT has been banned since 2003 by the International Maritime Organization due to its toxic effects when used for antifouling paints [9]–[11]. Therefore, copper-based antifouling paint such as cuprous oxide (Cu2O), copper thiocyanate (CuSCN), or metallic copper is now utilized as the principal biocide [12].

Copper has antifouling properties against barnacles, tube worms, and a wide range of algae fouling species. However, certain algae species, such as Enteromorpha spp., Ectocarpus spp., and Achnanthes spp., demonstrate high physiological tolerance to copper. To defend against these tolerant species, a number of co-biocides have been utilized in concert with copper to prevent copper-resistant fouling organisms [9], [13], [14]. Several co-biocides have been registered to be used as active ingredients in antifouling paint (e.g., Diuron, Irgarol 1051, zinc pyrithione, copper pyrithione, chlorothalonil, and SeaNine 211).

SeaNine 211 is a commercial antifouling co-biocide that is now in use globally. SeaNine 211 contains the biocidal component 4,5-dichloro-2-n-octyl-4-isothiazolin-3-one (DCOIT), a member of the isothiazolone group, which has antifouling activity. DCOIT is an efficient and broad-spectrum antifouling biocide that targets bacterial slime, diatoms, tubeworms, algae, and barnacles [15]. Nevertheless, the environmental stability and toxicity of SeaNine 211 on non-target creatures have sparked public concern and faced strict regulations or

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prohibitions [16]. SeaNine 211 is very harmful to a non-target marine species [17]–[21]. As a result, an environmentally acceptable antifouling paint co-biocide is required. In this present work, cerium oxide is used as an environmentally friendly co-biocide in SeaNine 211-free SPA paints.

Cerium oxide was chosen as a substitute for SeaNine 211 in this study because cerium oxide nanoparticles have antibacterial properties. However, the antibacterial properties of cerium oxide are unlike SeaNine 211; cerium oxide is not harmful to non-target organisms [22]. Antibacterial properties must be possessed by materials used as co-biocides in antifouling paints because the initial stage of biofouling formation begins with bacterial attachment. In addition, previous studies conducted by Ronggen Wu et al. [23] and N. Wang et al. [24] stated that cerium oxide nanoparticles can catalyze haloperoxidase reactions like vanadium oxide. Haloperoxidase reactions are reactions between hydrogen peroxide and halide ions contained in seawater, producing hypohalide acids that can disrupt communication between bacterial cells so that they can prevent biofouling attachment [25]. Cerium oxide is not like vanadium oxide, which is mutagenic, so it has the potential to be applied to antifouling paints. From previous studies, this becomes the reason this study used cerium oxide as a co-biocide to replace SeaNine 211. The novelty in this study is the use of micron-sized cerium oxide instead of nanoparticles, where micron-sized cerium oxide is cheaper, so it is more economical to apply in the paint industry. In addition, the effect of cerium oxide on SeaNine 211-free SPA paint on the Cu dissolution rate using the leached-out test method is another novelty that has never been studied.

EXPERIMENTAL METHOD

Preparation of Modified Paint and Specimen

SeaNine 211-free self-polishing antifouling paint and epoxy anticorrosion paint were obtained from a local antifouling paint factory. Table 1 lists the specifications of the paint used in this work. Micron-sized cerium oxide was purchased from e-commerce and analyzed using a particle size analyzer (PSA) to confirm the particle size. The average particle size of cerium oxide used in this research, according to PSA, was 1.24 µm. Low-carbon steel with the composition (Fe: 99%, Si: 0.14%, C: 0.042%, Mn: 0.247%, P: 0.023%, and S: 0.0098%) was employed as a specimen in this work.

SeaNine 211-free self-polishing antifouling (SPA) paint contains cuprous oxide (±30 wt% Cu_2O) and zinc oxide (±10 wt% ZnO). The modified SPA paint was prepared by incorporating cerium oxide with various concentrations (0.1, 0.3, and 0.5 wt%) into SeaNine 211-free SPA paint using a high-speed mixer. The modified paint is thoroughly mixed at 1000 rpm for 1 hour at room temperature. To prepare the specimens, low-carbon steel with size 2 × 2 cm was sanded and painted using a brush with epoxy anticorrosion paint as primer coating and modified SPA paint as a top coating. The dry thickness of the primer and the top coating was 125 and 150 microns, respectively. The steel specimens will undergo scanning electron microscopy (SEM) and electrochemical impedance spectroscopy (EIS) testing.

Table 1. Specification of SeaNine 211-free SPA and epoxy anticorrosion paint

Specification	SeaNine 211-free SPA Paint	Epoxy Anticorrosion Paint
Description	Long-life antifouling with synthetic plasticizer polymer resin system	Durable rust-inhibitive epoxy primer paint
Colors	Red	Red
Volume solids	60%	66%
Typical thickness	150 micron	75–150 micron
Application temperature	Max. 40ºC	Max. 40ºC
Flash point	Greater than 23ºC	Greater than 29ºC
Pot life	Single pack (not applicable)	4 hours
Shelf life	12 months	12 months

Leached-Out Test

A leached-out test was conducted to calculate the copper dissolution rate of modified SPA paint in accordance with

ASTM D6442-99. The leached-out test samples were made of a polycarbonate cylinder of 12 cm in height and approximately 6.4 cm in diameter. The polycarbonate samples were immersed in 10% hydrochloric acid for 12 hours, then rinsed with distilled water and sanded with 200-grit sandpaper before being covered with antifouling paint. The surface area of the antifouling paint film is around 200 cm^2 on the polycarbonate samples. Each painted polycarbonate was immersed and rotated in the individual measuring glass container for 1 hour. Prior to assay, samples were immersed in artificial seawater for 24 hours in the holding tank. After the dissolution rate test, the samples were placed back in a holding tank filled with artificial seawater at a controlled pH 8.2. After a certain period (1, 3, 7, 10, 14, 21, 24, 28, 31, 35, 42, 45) of the holding interval, the samples were moved from the holding tank to individual measured glass containers. In the container, the solution temperature and rotation speed are 25°C and 60 ± 5 rpm, respectively. This certain rotation speed is determined to simulate a laminar flow speed of a ship or boat around 0.2 m/s (<0.4 knots). The copper and zinc dissolution rates were obtained by calculating the concentration of copper in the test solution using the DR 1900 Spectrophotometer instrument [26]. The key steps of the leached-out test are illustrated in Figure 1.

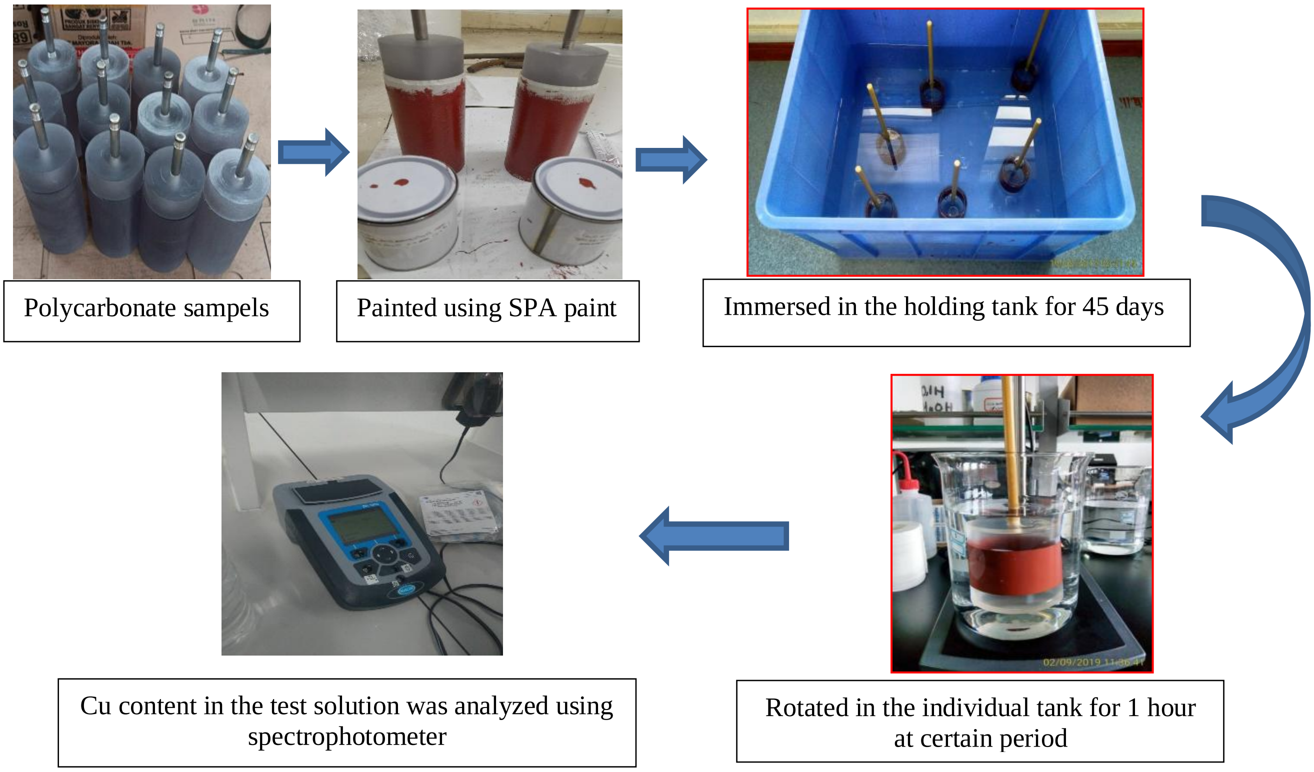

Figure 1. The key steps of the leached-out test

Characterization

The characterization of this work uses SEM to observe the layer of paint and copper (Cu) mapping before and after the immersion in the artificial seawater. The SEM specimens were immersed for 7 days in artificial seawater and rotated for 1 hour each day. The cross-sections of steel specimens after immersion were observed using SEM JEOL to characterize the paint film. Further, the wettability of the steel after being painted with various cerium oxide concentrations was characterized by the OSSILA Goniometer contact angle measuring tool. To perform the contact angle test, a droplet of artificial seawater was dropped on a steel specimen and photographed with a camera. The angle was then determined based on the image. The contact angle test was conducted on days 0 and 45.

RESULT AND DISCUSSION

Copper (Cu) Dissolution Rate

The copper dissolution rate in the artificial seawater solution was measured using a DR 1900 Spectrophotometer, according to Standard Method 8026 (Cu). The dissolution rate of Cu for modified self-polishing antifouling (SPA) paint with or without cerium oxide addition at a rotation speed of 60 rpm is shown in Figure 2. This figure depicts the variance in the copper dissolution rate as a function of immersion time for up to 45 days. In all modified SPA paints, the maximum magnitude of copper dissolution rate was found at the first stage of the immersion period (the 1st day). Copper dissolution rates in the final stage (45 days of exposure) are two times lower than in the first stage. An exception is paint with 0.5 wt.% cerium oxide concentration, which, unlike the other paints, has the maximum Cu dissolution rate on the 3rd day of immersion time. On the first day, SPA paint without cerium oxide had the greatest Cu dissolution rate compared to SPA paint with cerium oxide. Otherwise, on the final day, SPA paint in the absence of cerium oxide has the lowest Cu dissolution rate compared to SPA paint in the presence of cerium oxide. It assumes that on the first day, cerium oxide does not directly release into the seawater, which prevents Cu from dissolving. However, the following day, the release of cerium oxide into the seawater creates more pores in the paint matrix, increasing the rate of Cu release.

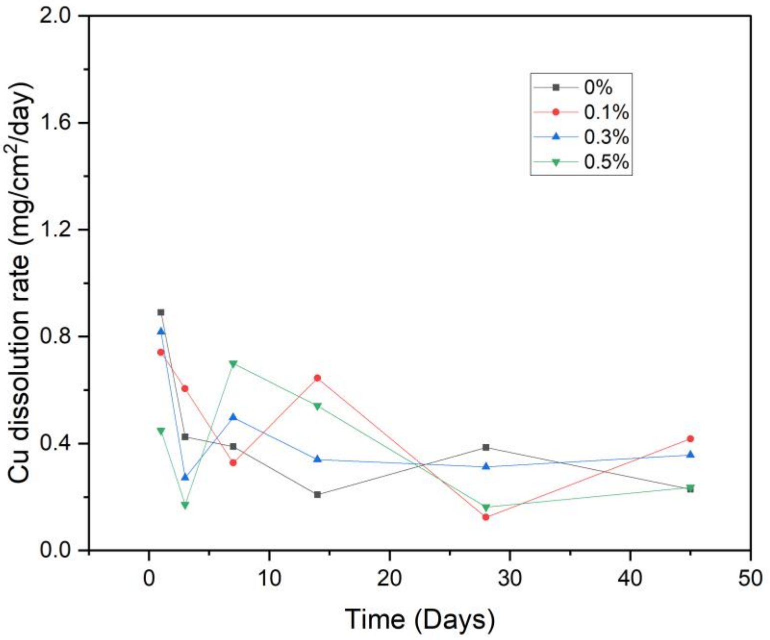

Figure 2. The cu dissolution rate of modified SPA paint with various cerium oxide concentrations

To obtain the Cu dissolution rate in Figure 2, the Cu concentration in seawater (mg/l) as a result of the spectrophotometer measurement was calculated using Equation (1). The total Cu dissolution rate over 45 days can be calculated using Equation (2). As long as the Cu dissolution rate is greater than 0.01 mµg /cm^2.day, the efficacy of SPA paint still keeps to mitigate attached biofouling on the subtract [27], [28]. Based on Figure 1, the Cu dissolution rate for all paints is more than 0.01mµg /cm^2.day. Moreover, Ytreberg and co-workers reported that, in artificial seawater, the Cu dissolution rate was higher than in natural for most SPA paints [29]. It also implies that the Cu dissolution rate in artificial seawater is not exactly represented in the field.

$$R_a = (C_{cu} \times V \times D) / (T \times A) \quad (1)$$

$$R_b = R_1 + 2R_3 + 4R_7 + 3R_{10} + 4R_{14} + 7R_{21} + 3R_{24} + 4R_{28} + 3R_{31} + 4R_{35} + 3R_{38} + 4R_{42} + 3R_{45} \quad (2)$$

where
R_a : Cu dissolution rate (mg/cm^2/day)
C_{cu} : Concentration of copper in seawater (mg/l)
V : seawater volume in individual container, (l)
D : hours/day
T : spin time (hours)
R_b : cumulative dissolution rate (mg/cm^2), and
$R_{1,}$ $R_{3,}$ $R_{7,}$ etc : dissolution rate for sampling days 1, 3, 7, etc., respectively (mg/cm^2/day).

The total cumulative Cu dissolution rate up to final immersion in artificial seawater is shown in Figure 3. From Figure 3, it is clear that the addition with 0.1 wt.% cerium oxide concentration exhibits the greatest cumulative Cu dissolution rate than the other samples. The concentration of cerium oxide affects the cumulative dissolution rate of copper, according to Figure 3. Generally, the addition of cerium oxide in the modified SPA paint will increase the cumulative Cu dissolution rate. This event occurs because the release of cerium oxide into artificial seawater from the SPA paint matrix leaves behind small pores and a wide area in that matrix, which increases the total wetted area that could initiate the further dissolution of copper particles. On the other hand, the cumulative Cu dissolution observed for the SPA paint containing 0.5 wt.% of cerium oxide was different. Cerium oxide, at these weight concentrations, has not proven effective in increasing the Cu dissolution rate. The reason for such different behavior at 0.5 wt.% of cerium oxide is unclear to the authors. It is possible to remark a partial aggregation of cerium oxide at a concentration of 0.5 wt.% that prevents the dissolution of copper [30]. Furthermore, the cumulative Cu dissolution rate of 0.3 wt.% of cerium oxide was slightly higher than SPA paint without cerium oxide but lower than 0.1 wt.% of cerium oxide. This is because adding more than 0.1 wt. % cerium oxide makes the cerium oxide dispersion inhomogenous and lumpy in the SPA paint matrix. Thus, the addition of a cerium oxide concentration of more than 0.1 wt.% reduces the Cu dissolution rate. This result is similar to the result of L.G. Ecco et al. research [30] that 1 wt.% of cerium oxide in the paint has better performance than the higher weight percent (2 wt.% and 3 wt.%).

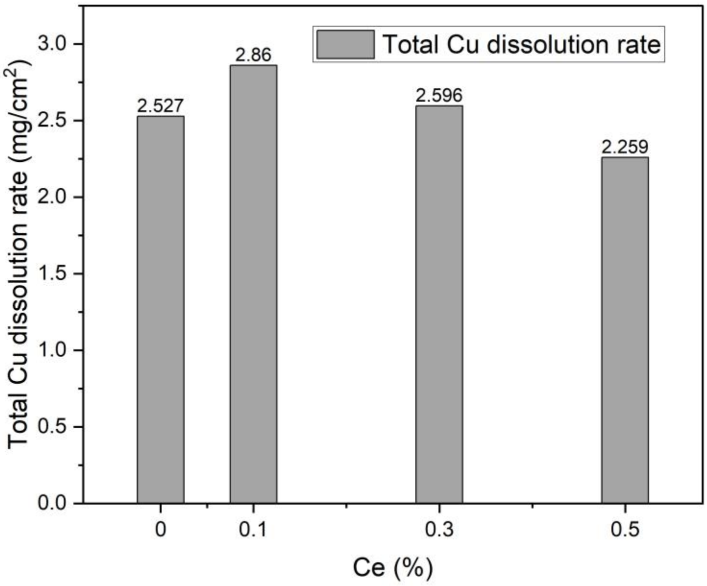

Figure 3. Cumulative Cu dissolution rate of modified SPA paint with various cerium oxide concentrations

Paint Layer Analysis

Paint layers pre and after exposure to artificial seawater for 7 days were investigated using scanning electron microscope (SEM). Figure 4 shows SEM images of cross-section specimens after 7 days of exposure to artificial seawater. It can be seen from Figure 3 that there are three layers above the bare steel: primer paint, antifouling paint, and resin. The application of resin on the surface of antifouling paint separates the unexposed layer from the solution-exposed layer. Therefore, the paint layer before exposure to artificial seawater can be observed under the resin. There is a difference in the layer on the surface of the antifouling paint prior to and post-exposure on the surface of the antifouling paint layer under the resin (prior to exposure) has a good surface, while the surface of the antifouling paint layer exposed to the solution, the layer is damaged and eroded. After exposure to artificial seawater, samples will experience shafts or eroded due to the dissolution of biocides or substances contained in antifouling paint. The copper oxide (Cu_2O) biocide contained in the SPA paint will react with Cl- ions contained in the artificial seawater, resulting in copper complex ions ($CuCl_2^-$) and ($CuCl_3^{2-}$) which will continue to be dissolved into the artificial seawater continuously. This causes the formation of pores in the outer layer or antifouling paint layer [31]. Direct contact with artificial seawater causes a reduction in the thickness of antifouling paint. The thickness reduction of SPA paint with 0.3% cerium oxide (200 µm) was greater than SPA paint without cerium oxide (94 µm). It can be assumed that cerium oxide accelerates the erosion of antifouling paint into seawater. In addition to the antifouling paint layer, the primer paint layer is also the focus of observation in the paint layer analysis. There is no change in the primer paint layer before and after exposure to artificial seawater. This demonstrates that antifouling paint provides effective protection. Thus, steel samples will not directly contact seawater, contaminants, or other fouling organisms.

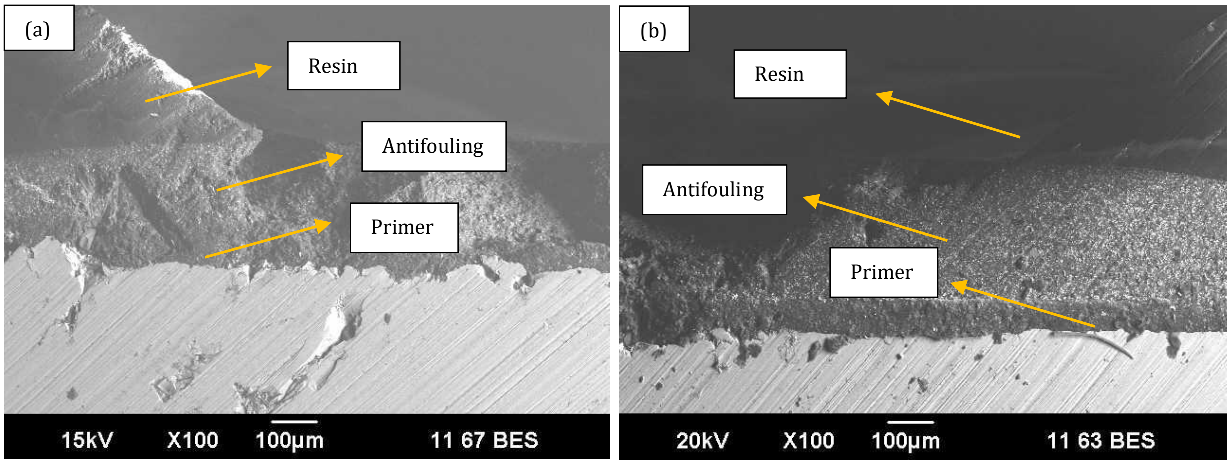

Figure 4. SEM images of paint after exposure 7 days (a) 0 wt.% cerium oxide and (b) 0.3 wt.% cerium oxide

SEM mapping for the Cu element was carried out to analyze the antifouling paint layers further. Cu mapping post-exposure to artificial seawater for 7 days in the absence and 0.3 wt.% of cerium oxide of modified SPA paint are illustrated in Figure 5 (a) and Figure 5 (b), respectively. Based on Figure 5, Cu mapping of antifouling paint with 0.3 wt.% cerium oxide is denser than without cerium oxide, as indicated by the green color in the image. The correlation between the color parameters on each image shows that the denser the colored dots are, the higher the content of Cu. The copper released into seawater is marked with a red line in Figure 5. Copper released into seawater is slightly visible in Figure 5 (a) and visible in Figure 5(b). The higher Cu release for SPA paint with 0.3 wt.% cerium oxide is due to the influence of cerium oxide. Cerium oxide is assumed to be an erosion facilitator for Cu because, when released into seawater, it will form pores, which makes copper easier to dissolve into seawater.

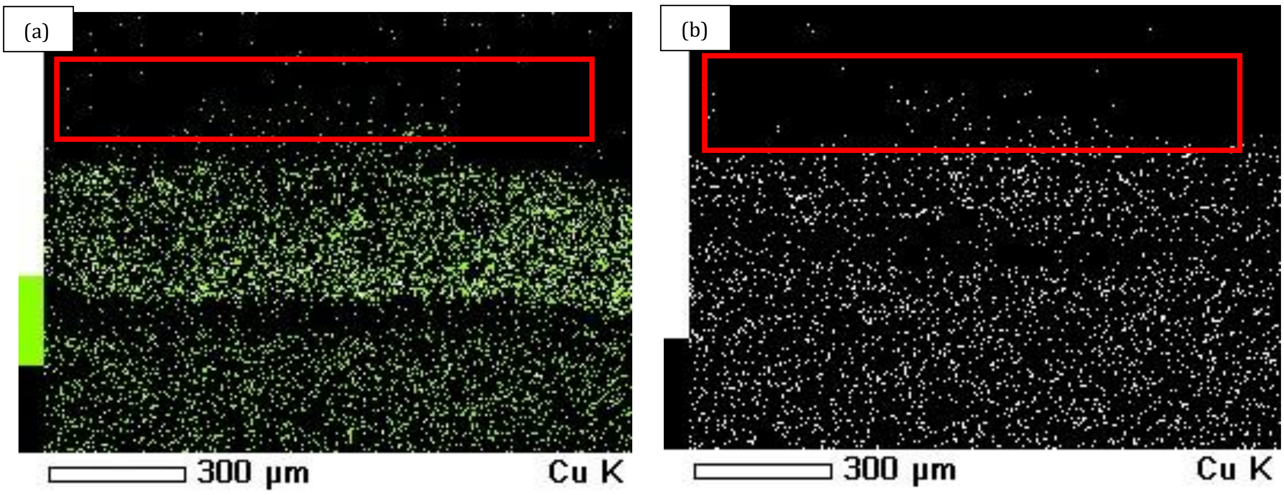

Figure 5. Cu mapping of paint after exposure 7 days (a) 0 wt.% cerium oxide and (b) 0.3 wt.% cerium oxide

Contact Angle Test

A contact angle test was employed to determine the surface properties of modified SPA paint, whether it is hydrophilic or hydrophobic. A surface is considered hydrophilic if its contact angle is less than 90° and hydrophobic if it is larger than 90° [32]. Contact angle test data is acquired by calculating the inclination of liquid or fluid droplets on antifouling paint samples in different variations. The contact angle is measured by examining the picture of a liquid droplet and connecting each point on its perimeter. Figure 6 shows a graph illustrating the relationship between cerium oxide concentration and contact angle prior to and post-45 days of immersion.

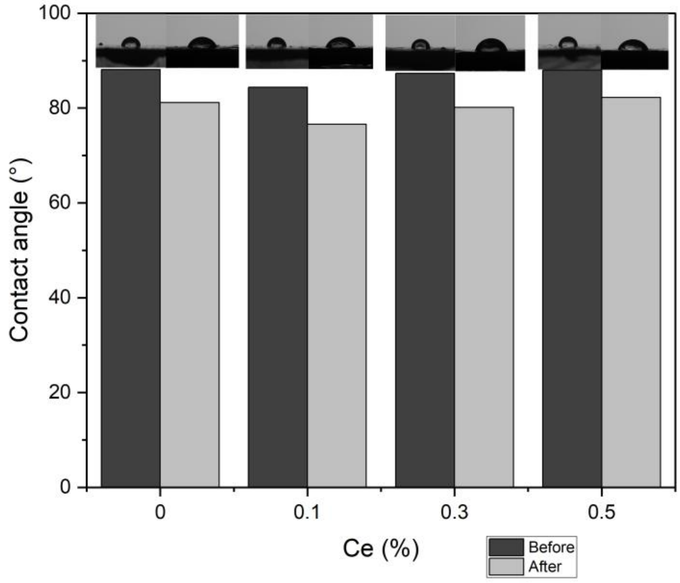

Figure 6. The contact angle of modified SPA paint before and after exposing to artificial seawater for 45 days

In Figure 6, all contact angles decrease after 45 days of immersion. The contact angle before exposure in artificial seawater approaches 90º (hydrophobic). This is because SPA paint, based on an acrylic matrix with various pendant groups, is attached to the polymeric backbone, which prevents seawater from penetrating the paint film. However, the contact angle becomes more hydrophilic subsequent to immersion in artificial seawater for 45 days. The change in contact angle alongside exposure in artificial seawater is due to the presence of soluble polymer on top of SPA paint. The leaching process begins when the fresh surface is immersed in seawater. The soluble toxic particles (Cu_2O, ZnO, and CeO_2) begin to dissolve where seawater can fill the pores from the dissolution of pigments. Seawater slowly reacts with silyl acrylate hydrolytically unstable under slightly alkaline conditions, which alter to soluble acidic polymer [33]. Therefore, the presence of soluble polymer on top of the SPA paint surface can be identified by the lower magnitude of the contact angle. Based on the results, there is no difference in the wettability behavior of SPA paints when adding cerium oxide.

It can also be seen in Figure 6 that modified SPA paint with 0.1 wt.% cerium oxide has the lowest contact angle than the other concentrations. This result is in accordance with the leached-out test results. The SPA paint with 0.1 wt.% cerium oxide has the highest Cu dissolution rate. This means more pores are formed due to the large amount of copper biocide released. The more pores on the surface of the SPA paint, the greater the water penetration into the surface, making it more hydrophilic.

CONCLUSION

This work reported the influence of micron-size cerium oxide in self-polishing antifouling paint without SeaNine 211 to copper (Cu) dissolution rate by means of leached-out test, scanning electron microscopy (SEM), and contact angle test. Generally, an enhanced Cu dissolution rate has been obtained due to the incorporation of cerium oxide into the modified self-polishing antifouling (SPA) paint. The addition of 0.1wt.% cerium oxide improves the copper dissolution rate and the total amount of released copper. The higher concentration of cerium oxide up to 0.1wt.% could not increase the Cu dissolution rate due to the partial aggregation. The improvement of copper dissolution rate with the cerium oxide addition was also confirmed by the SEM test results through Cu mapping and the increase in the wettability from the contact angle test results. The increasing copper dissolution rate leads to an improvement in the efficacy of SPA paint in combating biofouling. Therefore, micron-sized cerium oxide has the potential to be used as an environmentally friendly co-biocide in SPA paint to replace SeaNine 211 because it can increase the dissolution rate of Cu.

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