

Assessing bifunctional benzyldodecyldimethylammonium bromide in mitigating abiotic and biotic corrosion using a new test kit

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ABSTRACT

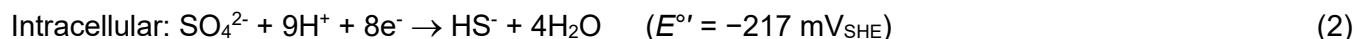
Benzyldodecyldimethylammonium bromide (BDA-C12) is a corrosion inhibitor which forms a protective film layer on metal surfaces. It can also function as a biocide. This work assessed the performances of BDA-C12 in mitigating abiotic corrosion caused by CO₂ and microbiologically influenced corrosion (MIC) caused by highly corrosive sulfate reducing bacterium (SRB) *Desulfovibrio ferrophilus* IS5 against X65 carbon steel. The initial efficacy screening was performed conveniently using a 10 mL disposable test kit with two solid-state electrodes for electrochemical measurements. In the corrosion prevention tests in which BDA-C12 was added into both the abiotic and biotic systems upon coupon immersions, 200 ppm (w/w) BDA-C12 was found to reduce the abiotic corrosion considerably (by 85%), and it successfully prevented the SRB biofilm formation, reflected by the linear polarization resistance (LPR) and potentiodynamic polarization scans. The loose coupon weight loss tests in 125 mL anaerobic vials containing 50 mL enriched artificial seawater confirmed the 10 mL test kit data. In the injection tests, the 500 ppm BDA-C12 injections in the abiotic and biotic systems achieved 14% and 34% polarization resistance (R_p) increases (corrosion rate decreases) from LPR measurements, respectively. Sessile cell counts and biofilm images supported the MIC inhibition results.

Keywords: *Microbiologically influenced corrosion, bifunctional inhibitor, inhibition efficiency, Biofilm test kit, electrochemical test*

INTRODUCTION

In the oil and gas industry, corrosion is a major threat as it causes equipment failures and substantial economic losses¹⁻³. Both abiotic corrosion and microbiologically influenced corrosion (MIC) are important contributors. Carbon steel pipelines and equipment are known to be vulnerable to abiotic corrosion such as CO₂ and H₂S attacks^{4,5}. Among them, CO₂ corrosion is considered a primary concern as most gas and crude oil reservoirs contain dissolved CO₂ which aggressively attacks carbon steel at moderate temperatures⁶⁻⁸. Several mitigation approaches have been adopted to combat CO₂ corrosion such as corrosion inhibitor addition, material optimization, protective coating application and effective monitoring⁹⁻¹¹.

MIC is said to account for 20% of the total corrosion costs and has become a serious menace in oilfields¹². MIC is typically caused by the activities of corrosive biofilms or their metabolic products on metal surfaces¹³. Various microorganisms including bacteria, archaea, fungi, and microalgae have been documented to cause MIC under different conditions¹⁴⁻¹⁶. Among them, sulfate reducing bacteria (SRB) have been extensively studied as they are widely believed to cause the most serious MIC¹⁷⁻²⁰. The corrosion mechanisms of SRB are mainly classified into extracellular electron transfer MIC (EET-MIC) and metabolite-MIC (M-MIC)²¹. The former occurs to energetic metals such as iron which can serve as the electron donor for SRB biofilms to harvest extracellular electrons and perform sulfate reduction to produce energy²². This process can be elucidated in the biocatalytic cathodic sulfate reduction (BCSR) theory²¹ using the following two half reactions,



In Eq. (2), the apostrophe in equilibrium potential $E^{\circ'}$ indicates pH 7 physiological condition in bioenergetics to replace the standard 1 M [H⁺]²³. Another type of MIC, M-MIC can be explained using Cu corrosion as an example. Cu is rather noble, which is not energetic enough to donate electrons for sulfate reduction directly²¹. In this case, corrosive metabolites such as H₂S secreted by SRB can corrode Cu with H⁺ serving as the electron acceptor. The extremely insoluble Cu₂S results in a rather negative Gibbs free energy change in the following corrosion reaction²¹,



The precipitation of sulfide detoxifies the SRB broth which promotes SRB growth^{24,25}. H₂ from the corrosion provides energy for hydrogenase-positive SRB²⁶.

These two different types of MIC can be distinguished by using EET-promoters such as riboflavin and magnetite nanoparticles (MNPs)^{27,28}. Riboflavin and MNPs were found to accelerate EET-MIC through facilitating the electron transfer process²⁹. In comparison, the corrosion rates of M-MIC were not affected^{28,30}.

In addition to physical pigging, biocide treatment is a traditional method targeting corrosive biofilms to mitigate MIC³¹⁻³⁵. Compared to planktonic cells, sessile cells in biofilms are more difficult to treat due to

comprehensive defense mechanisms possessed by biofilms which provide protections from external antimicrobial agents³⁶. Thus, a much higher dosage is often needed to eradicate a pre-established biofilm than preventing its growth and formation³⁷. Tetrakis hydroxymethyl phosphonium sulfate (THPS) is one of the most prevalent green biocides in oilfields because it has a broad-spectrum efficacy and is readily biodegradable³⁸. Glutaraldehyde is another popular biocide due to its excellent efficacy and biodegradability³⁹.

Quaternary ammonium compounds (QACs) are effective cationic biocides and commonly used in the oil and gas industry to control MIC⁴⁰. The positive charge of QACs allows them to electrostatically bind to the negatively charged bacterial cell wall. After penetrating the cell wall, QACs disrupt the cytoplasmic membrane which leads to the leakage of intracellular contents and cell death^{41,42}. They were documented to effectively inhibit oilfield biofilm formation and MIC⁴³. Alkyldimethylbenzylammonium chloride (ADBAC) has been reported to be effective in mitigating carbon steel MIC caused by *Desulfovibrio vulgaris* (a widely studied SRB) biofilm. Benzyl dodecyl dimethyl ammonium bromide (BDA-C12) has emerged as a promising bifunctional agent capable of simultaneously suppressing microbial growth as a biocide and forming protective adsorption layers on steel surfaces as a corrosion inhibitor, suggesting its potential of mitigating both MIC and abiotic corrosion^{44–47}. However, the dual performances of BDA-C12 need validation data.

Traditional corrosion tests rely heavily on coupon tests such as weight loss measurements. They are reliable in studying direct corrosion evidence, but the one-shot cumulative results limit their applications for continuous monitoring or real-time corrosion rate interpretation. In contrast, electrochemical tests are more convenient to perform repeatedly and can provide near-real time corrosion rate results in minutes. Several electrochemical techniques such as linear polarization resistance (LPR), electrochemical impedance spectroscopy (EIS), and potentiodynamic polarization (PDP) or Tafel scan have been consistently confirmed to corroborate coupon test results^{48–51}.

For the purpose of conveniently conducting electrochemical tests on site, a disposable test kit was newly developed³⁰. Compared to the traditional 3-electrode (3E) setup consisting of a working electrode (WE), a counter electrode (CE) and a dedicated reference electrode (RE), the new test kit skips the fragile independent RE and uses the CE as both a CE and a pseudo-reference electrode (p-RE), forming a 2-electrode (2E) setup. This test kit has been shown to be reliable in monitoring biofilm growth and corrosion rate variations²⁹.

This study investigated the effectiveness of bifunctional BDA-C12 in mitigating SRB MIC and abiotic CO₂ corrosion. The new test kit was employed in initial testing to measure corrosion rates in both systems with and without the addition of BDA-C12. The coupon tests were subsequently performed to confirm the electrochemical test findings from the test kit. The results provide insights into BDA-C12 mechanisms and its potential applications in corrosion mitigation. They also illustrate a new system to test a corrosion inhibitor more conveniently.

EXPERIMENTAL PROCEDURE

Chemicals, bacterial strain and metal

For CO₂ corrosion tests, a 5% (w/w) NaCl solution was deoxygenated with N₂ gas and then sparged with CO₂ gas for 10 min to reach a final pH of 4.0. A super-corrosive SRB *Desulfovibrio ferrophilus* (IS5 strain) was used for SRB MIC testing. Enriched artificial seawater (EASW) culture medium (composition in Table 1) was prepared to grow *D. ferrophilus* at 28°C (optimal)⁵². The pH of EASW culture medium was adjusted to 7.0 with a 5% (w/w) NaOH solution. After autoclave sterilization, EASW was sparged with filter-sterilized N₂ gas for 1 h to remove dissolved oxygen. L-cysteine was finally added as an oxygen scavenger at a concentration of 20 ppm (w/w) in an N₂-filled anaerobic chamber. BDA-C12 was supplied by Dr. David Young with its synthesis documented elsewhere⁴⁶.

Table 1. Composition of EASW culture medium⁵².

Chemicals	Amount
NaCl	23.476 g
Na ₂ SO ₄	3.917 g
NaHCO ₃	0.192 g
KCl	0.664 g
KBr	0.096 g
H ₃ BO ₃	0.026 g
MgCl ₂	4.965 g
SrCl ₂ ·6H ₂ O	0.04 g
CaCl ₂ ·2H ₂ O	1.469 g
Sodium lactate	3.5 g
Yeast extract	1.0 g
Sodium citrate	0.5 g
MgSO ₄ ·7H ₂ O	0.71 g
CaSO ₄ ·2H ₂ O	0.1 g
NH ₄ Cl	0.1 g
K ₂ HPO ₄	0.05 g
Fe(NH ₄) ₂ (SO ₄) ₂ ·6H ₂ O	1.37 g
DI water	1 L

X65 pipeline carbon steel was used for testing in this work. Its elemental composition is listed in Table 2. All the X65 coupons were coated with inert liquid Epoxy coating (3M Product 323) with only the top surface (1 cm²) exposed, which was polished to 600 grit finish before coupon incubation testing. Pure isopropanol was employed for coupon cleaning and sanitation.

Table 2. Elemental composition (wt. %) of X65 carbon steel (Fe balance)⁵³.

C	Si	Mn	P	S	V	Nb	Ti
0.16	0.45	1.60	0.015	0.006	0.06	0.04	0.04

Electrochemical tests

The test kit vial used for electrochemical measurements consists of an X65 WE (1 cm² working surface) and a graphite CE/p-RE in a 10 mL serum vial. The vials were sealed with rubber septa and aluminum caps in an N₂-filled anaerobic chamber. Liquid samples were either added into the test kit vial before sealing or injected with a syringe needle in the anaerobic chamber. Electrochemical tests including LPR and Tafel measurements were performed daily during a 7-d incubation period using a PCI4/750 potentiostat (Gamry Instruments, Warminster, PA, USA). The LPR scan rate was 0.167 mV/s with a voltage range from -10 to 10 mV vs. open circuit potential (OCP). Each scan took 2 min. Tafel scans were conducted with a scan rate of 0.167 mV/s with a voltage range from -200 to 200 mV vs. OCP. Dual-half scans were adopted so multiple Tafel scans could be performed on the same WE⁵⁴.

Coupon incubation tests

The electrochemical test results from the test kit were validated using the traditional coupon incubation tests in 125 mL anaerobic vials. In SRB MIC tests, each vial contained three replicate X65 coupons which were incubated in 50 mL EASW inoculated with 0.5 mL *D. ferrophilus* seed culture. Weight loss was calculated after a 7-d incubation. Sessile cells on each coupon were dislodged and re-suspended before being enumerated on a hemocytometer under a 400× microscope since SRB cells are motile and can be easily distinguished from FeS precipitates⁵⁵. Biofilms on coupon surfaces were observed under a confocal laser scanning microscope (CLSM) (Model LSM 510, Carl Zeiss, Jena, Germany)⁵⁶. A Live/Dead® BacLight™ Bacterial Viability Kit L7012 (Life Technologies, Grand Island, NY, USA) was first used to stain the biofilms so that live cells and dead cells could be detected as green and red dots under CLSM, respectively⁵⁷.

RESULTS AND DISCUSSION

Electrochemical tests of SRB MIC prevention

Fig. 1 shows three test kit vials each containing 5 mL EASW before and after 7-d incubation with *D. ferrophilus* at 28°C with different BDA-C12 concentrations. Healthy *D. ferrophilus* growth was observed in test kit vials after 7-d incubation with 0 ppm (not treatment) and 50 ppm (inadequate treatment) BDA-C12 indicated by the black broth color due to SRB growth and black FeS precipitates. In comparison, with 200 ppm BDA-C12 the culture medium did not turn black, suggesting no significant SRB growth. Thus, 200 ppm BDA-C12 was preliminarily considered effective in preventing *D. ferrophilus* biofilm growth.

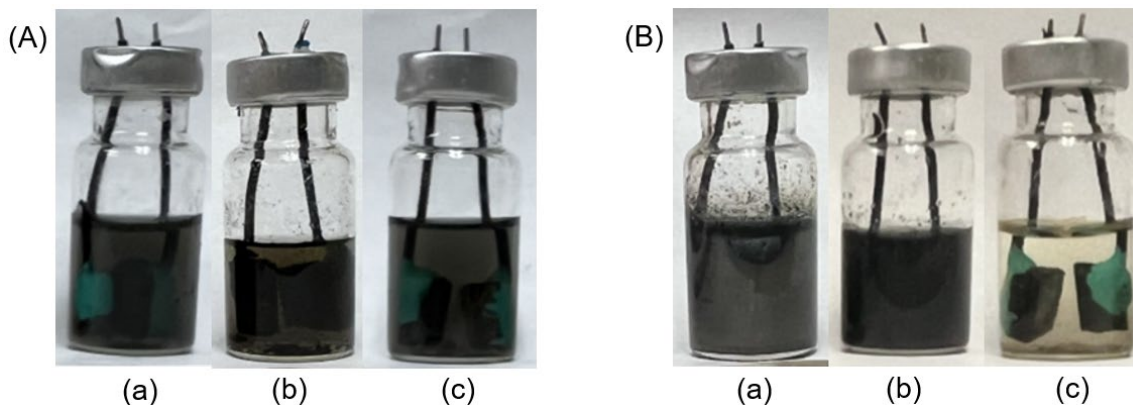


Fig. 1. Ten-mL test kit vials containing 5 mL EASW inoculated with *D. ferrophilus* before (A) and after (B) 7-d incubation with (a) no treatment, (b) 50 ppm BDA-C12, and (c) 200 ppm BDA-C12 at 28°C.

Electrochemical test results in the test kit vials are shown in Fig. 2. The polarization resistance (R_p) curve of the no treatment control in Fig. 2A exhibited a typical slide-shape MIC trend³⁰. With SRB biofilm growth, R_p gradually declined, suggesting corrosion rate increase due to biofilm buildup. At around 3-4 d, R_p reached its lowest level and remained stable for the following days, which means biofilm became mature around 3-4 d with the highest corrosivity achieved. With 50 ppm BDA-C12, the overall R_p trend was similar despite relatively higher R_p values indicating lower corrosion rates due to the biocidal effect of BDA-C12. However, BDA-C12 at 50 ppm was insufficient to prevent *D. ferrophilus* biofilm formation and its MIC. With 200 ppm BDA-C12, there was no obvious R_p decreasing trend suggesting no SRB growth, which was consistent with the test kit vial color in Fig. 1B. Thus, 200 ppm was considered effective in inhibiting *D. ferrophilus* biofilm growth in the biofilm prevention test.

Fig. 2B presents 7-d corrosion current density (i_{corr}) curves from Tafel scans. The results corroborated LPR scans results that the corrosion rate sequence was no treatment > 50 ppm BDA-C12 > 200 ppm BDA-C12. The 50 ppm BDA-C12 curve was close to the no treatment case, suggesting it was insufficient to prevent SRB biofilm growth. In comparison, with 200 ppm BDA-C12, the corrosion rate did not increase, suggesting 200 ppm BDA-C12 was very effective in inhibiting *D. ferrophilus* growth.

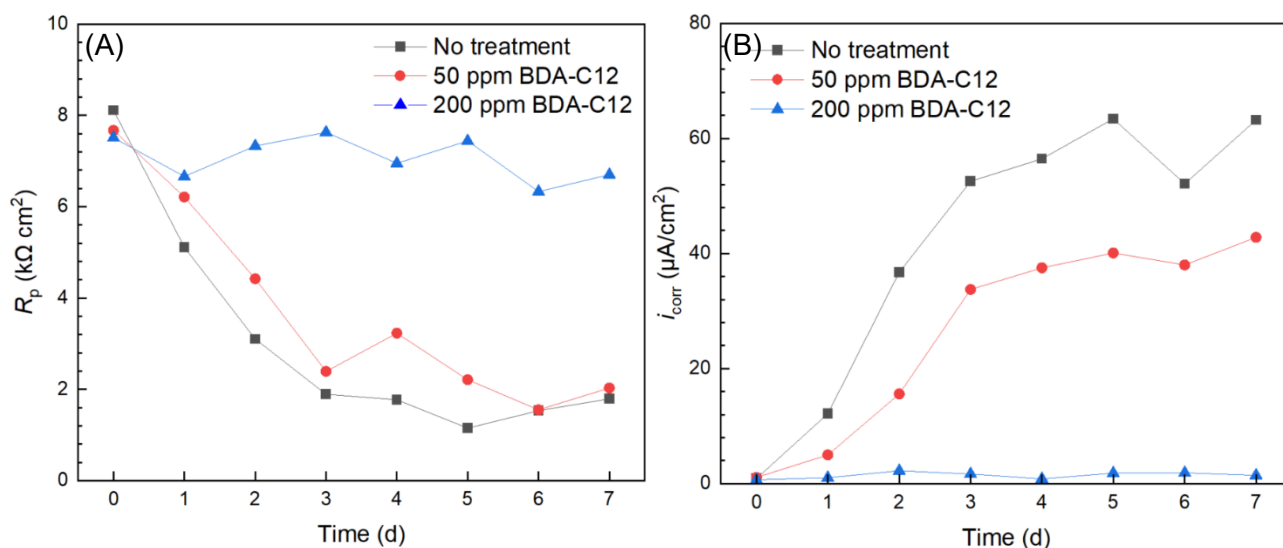


Fig. 2. R_p curves from LPR (A), and i_{corr} curves from Tafel (B) of X65 WEs in 10 mL test kit vials each containing 5 mL EASW inoculated with *D. ferrophilus* during 7-d incubation at 28°C.

Tandem chemical injection tests were conducted in a separate test kit vial at 3 d of incubation with *D. ferrophilus*. In Fig. 3, a 1 h stable R_p curve was first obtained before injections. A 20 ppm riboflavin injection was performed followed by continuous LPR scans every 10 min until R_p stabilized. The 500 ppm BDA-C12 was subsequently injected with R_p recorded every 10 min. The 20 ppm riboflavin injection resulted in 23% R_p decrease or 23% corrosion rate increase in 1 h because riboflavin as an EET promoter accelerated SRB MIC against X65 carbon steel. The subsequent 500 ppm BDA-C12 caused 34% R_p increase or corrosion rate decrease, suggesting its biofilm kill effect. Thus, the biocidal effect of BDA-C12 was confirmed in both biofilm prevention and biofilm eradication effects.

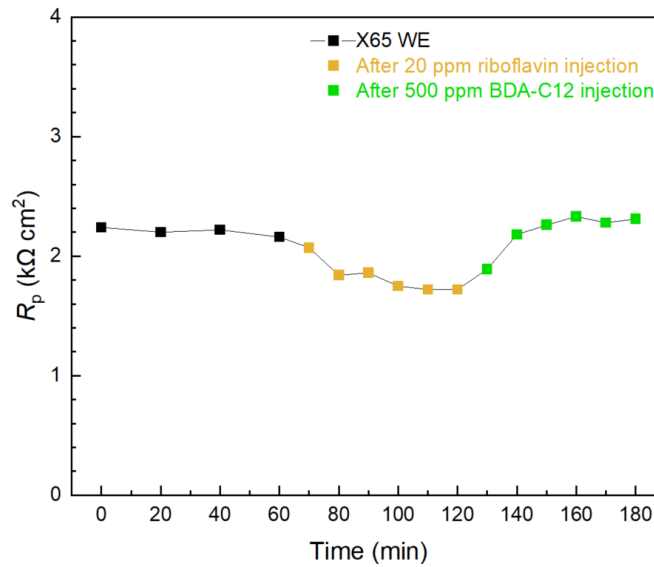


Fig. 3. X65 WE R_p response to tandem injections of riboflavin and BDA-C12 in 10 mL test kit vial containing 5 mL EASW at 3 d of incubation with *D. ferrophilus* at 28°C.

Coupon tests of SRB MIC mitigation with BDA-C12

Cell count tests were further conducted to support electrochemical test results in the 125 mL anaerobic vials as shown in Fig. 4. Consistent with the results in the 10 mL test kit vials, *D. ferrophilus* growth was seen in 125 mL vials with no treatment and 50 ppm BDA-C12 while no black broth color was observed in the 200 BDA-C12 vial. Planktonic and sessile cell count results are summarized in Fig. 5. Compared to the no treatment control, 50 ppm BDA-C12 achieved 0.7-log and 0.4-log reductions in sessile cells and planktonic cells, respectively. With 200 ppm BDA-C12, the two cell counts went below the detection limits of 5×10^4 cells/cm² and 5×10^4 cell/mL, corresponding to at least 3.1-log and 3.2-log reductions, respectively. The biofilms on X65 coupons surfaces after the 7-d incubation were examined under CLSM. The results in Fig. 6 indicate robust *D. ferrophilus* biofilm growth with no treatment as abundant live cells (green dots) were detected. With 50 ppm BDA-C12, fewer live cells were observed and some dead cells (green dots) appeared in the biofilm, suggesting insufficient inhibition effect of the 50 ppm BDA-C12. The 200 ppm BDA-C12 resulted in rarely detected cells with dead cells in the majority. Therefore, the CLSM biofilm observation findings are consistent with the sessile cell count results. Because sessile cells in biofilms are mainly responsible for MIC⁵⁸, the corrosion rate sequence from electrochemical tests that no

treatment > 50 ppm BDA-C12 > 200 ppm BDA-C12 was well supported and explained by the sessile cell count and biofilm visualization results.

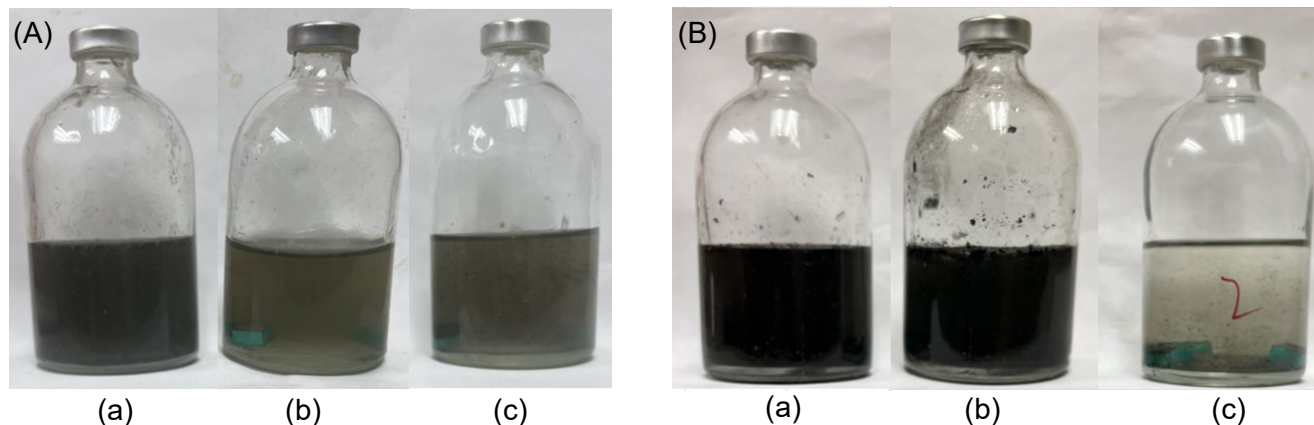


Fig. 4. X65 coupons in 125 mL anaerobic vials containing 50 mL EASW before (A) and after (B) 7-d incubation with *D. ferrophilus* with (a) no treatment, (b) 50 ppm BDA-C12, and (c) 200 ppm BDA-C12 at 28°C.

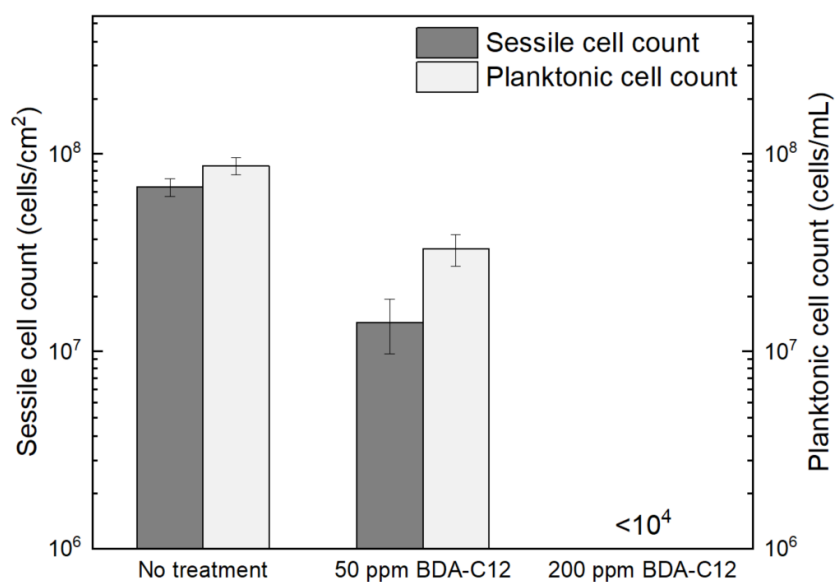


Fig. 5. Average planktonic and sessile cell counts on X65 coupons after 7-d incubation with *D. ferrophilus* in EASW at 28°C. (n = 3, ± s.d.)

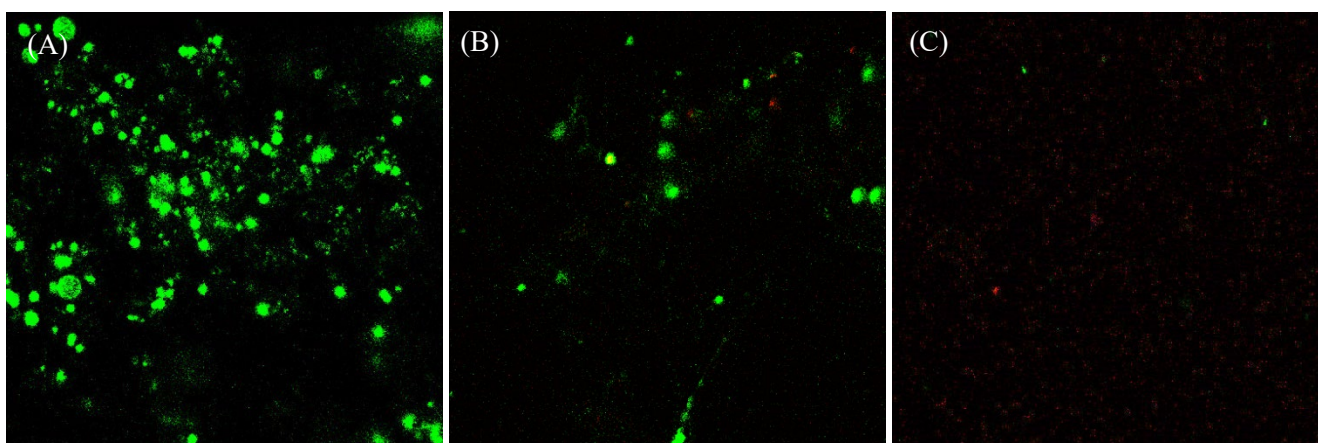


Fig. 6. CLSM images of biofilms on X65 coupons after 7-d incubation in 125 mL vials containing 50 mL EASW with *D. ferrophilus* at 28°C with (A) no treatment, (B) 50 ppm BDA-C12, and (C) 200 ppm BDA-C12.

Weight loss is direct evidence of MIC and an industry standard to evaluate MIC severity. Fig. 7 shows weight loss results of X65 coupons after 7-d incubation with *D. ferrophilus*. Without treatment, a 13.2 ± 2.0 mg/cm² weight loss was detected, corresponding to 0.88 mm/a uniform corrosion rate. The 50 ppm BDA-C12 reduced the weight loss to 7.6 ± 0.5 mg/cm² (0.50 mm/a uniform corrosion rate). The 50 ppm BDA-C12 achieved 42% weight loss reduction, suggesting biocidal effect of BDA-C12 but the dosage of 50 ppm was insufficient to completely inhibit *D. ferrophilus* MIC against X65 carbon steel. In comparison, with 200 ppm BDA-C12, the weight loss became negligible. Therefore, 200 ppm BDA-C12 was considered as highly effective in preventing *D. ferrophilus* MIC, which was consistent with the sessile cell count results.

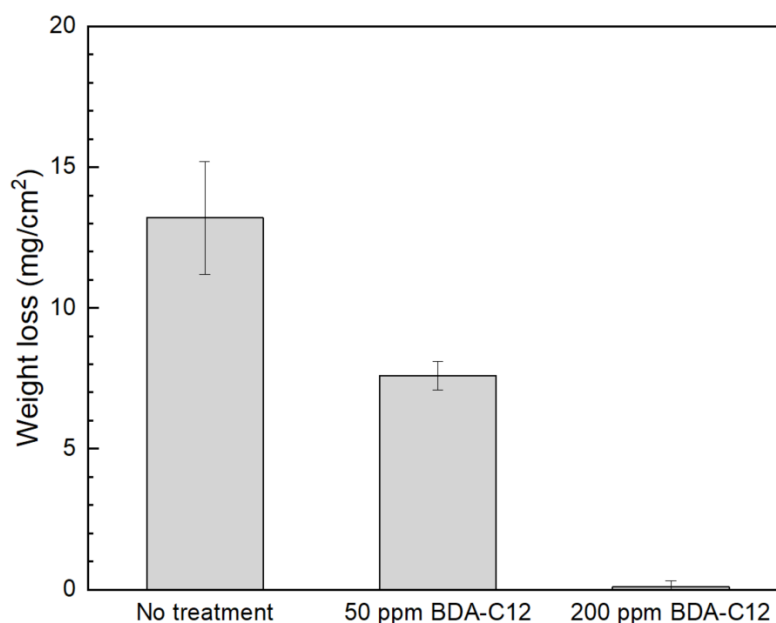


Fig. 7. Average weight losses of X65 coupons after 7-d incubation in 125 mL vials containing 50 mL EASW with *D. ferrophilus* at 28°C. (n = 3, $\pm$ s.d.)

Assessment of BDA-C12 in mitigating CO₂ corrosion

The efficacy of BDA-C12 in mitigating corrosion was first evaluated in 10 mL test kit vials each containing 5 mL 5% NaCl solution sparged with CO₂ (pH 4). Fig. 8A shows LPR scan results from test kit vials. Without BDA-C12 addition, the maximum corrosion rate was achieved within 1 d. With 50 ppm BDA-C12, the overall R_p values were greater than those with no treatment, suggesting decreased corrosion rates due to the corrosion inhibitory of BDA-C12 which can form a shielding layer on metal surfaces⁵⁹. The R_p values further increased with 200 ppm BDA-C12, but the decreasing trend in the initial days still suggesting the existence of mild corrosion, indicating that 200 ppm BDA-C12 was still not sufficient to inhibit CO₂ corrosion against X65 carbon steels completely. Tafel scan results in Fig. 8B show similar results as the i_{corr} values of 50 ppm BDA-C12 were slightly lower than the no treatment ones, corresponding to lower corrosion rates. The 200 ppm BDA-C12 further reduced the i_{corr} values or corrosion rates, but there was still a corrosion rate increasing trend suggesting CO₂ corrosion was not completely inhibited. The electrochemical test results of LPR and Tafel scans both confirmed the effectiveness of BDA-C12 in mitigation CO₂ corrosion. However, the CO₂ corrosion was not fully prevented even with 200 ppm BDA-C12.

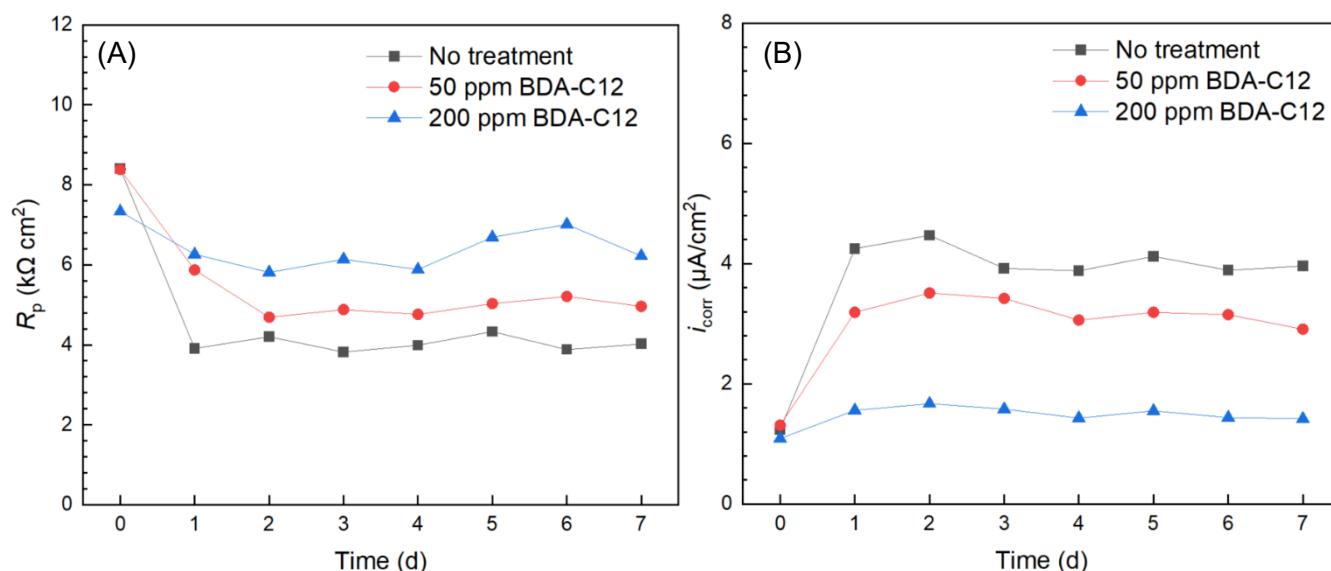


Fig. 8. R_p curves from LPR (A), and i_{corr} curves from Tafel (B) of X65 WEs in 10 mL test kit vials with 5 mL 5% NaCl solution pre-sparged with CO₂ (pH 4).

The tandem injection test was conducted for the abiotic CO₂ corrosion system similar to the SRB corrosion case at 3 d as shown in Fig. 9. Unlike the SRB MIC case, 20 ppm riboflavin injection in CO₂ corrosion did not yield significant R_p changes which means the corrosion rate was not affected because riboflavin is an EET promoter which only accelerates EET-MIC. This was also found to be an effective way to distinguish abiotic corrosion from EET-MIC³⁰. The subsequent 500 ppm BDA-C12 injection resulted in a 14% increase in R_p , indicating 14% corrosion rate decrease. Compared to the 34% corrosion rate decrease in the SRB MIC system, the BDA-C12 was not as effective in mitigating CO₂ corrosion.

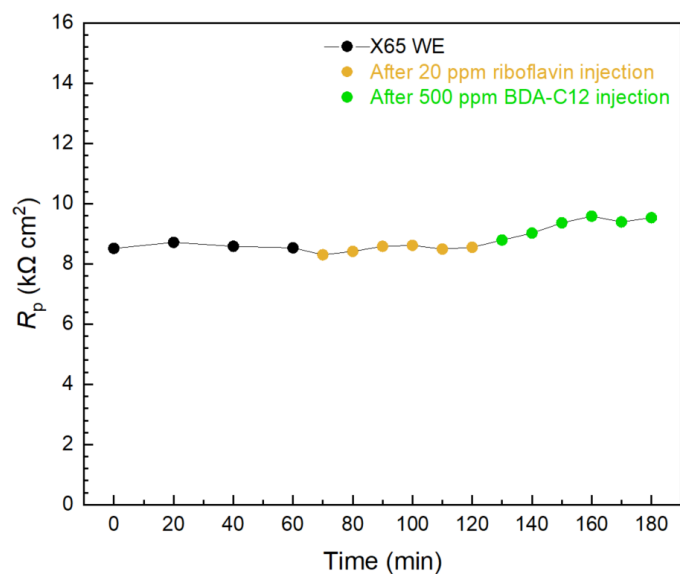


Fig. 9. R_p response of X65 WE to tandem injections of riboflavin and BDA-C12 in 10 mL test kit vial containing 5 mL 5% NaCl solution pre-sparged with CO_2 (pH 4) at 3 d.

The weight loss results of X65 carbon steel coupons in the CO_2 corrosion test are shown in Fig. 10. The weight losses were found to be $3.4 \pm 0.3 \text{ mg/cm}^2$, $2.1 \pm 0.4 \text{ mg/cm}^2$, and $0.5 \pm 0.2 \text{ mg/cm}^2$ for coupons with no treatment, 50 ppm BDA-C12, and 200 ppm BDA-C12, respectively. The 50 ppm reduced the weight loss by 38% while 200 ppm BDA-C12 achieved an 85% weight loss reduction. The results proved the very good effectiveness of BDA-C12 in mitigating CO_2 corrosion against X65 carbon steel. However, 200 ppm BDA-C12 was still insufficient in completely inhibiting CO_2 corrosion. These findings were consistent with the electrochemical test results from the test kit vials.

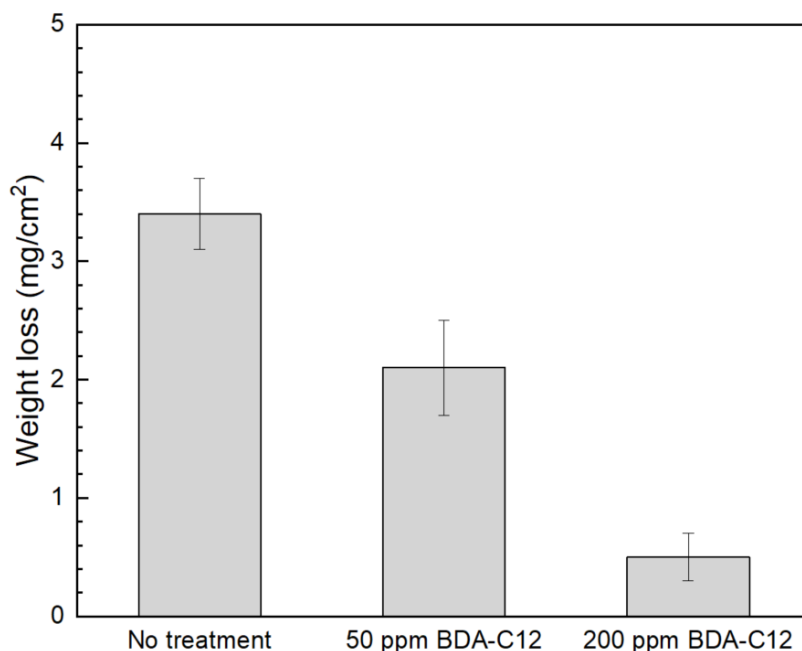


Fig. 10. Average weight losses of X65 coupons after 7-d immersion in 125 mL vials with 50 mL 5% NaCl solution pre-sparged with CO_2 (pH 4). ($n = 3$, $\pm$ s.d.)

CONCLUSIONS

This work demonstrated the applications of an electrochemical test kit in evaluating the effectiveness of a bifunctional chemical BDA-C12 in mitigating both SRB *D. ferrophilus* MIC and abiotic CO₂ corrosion. The following conclusions can be drawn from the experimental data in this work:

- BDA-C12 at 200 ppm effectively inhibited the biofilm growth of *D. ferrophilus* and its MIC.
- BDA-C12 at 200 ppm achieved 85% weight loss reduction in mitigating CO₂ corrosion.
- The test kit was able to interpret the corrosivity of abiotic and biotic samples with each LPR scan taking only 2 min, and each Tafel scan set 60 min.
- The electrochemical test results in the test kit were confirmed by the traditional coupon test results for weight loss and cell counts.
- The test kit can be conveniently used as the preliminary tool to evaluate the performances of corrosion inhibitors in both abiotic systems and MIC systems.

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