

CORROSION INHIBITOR AND OXIDANTS EFFECT ON IRON LEACHING FROM CARBON STEEL DURING MERCURY REMOVAL

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Abstract

The Iodine/potassium iodide (I₂/KI) lixiviant chemical possesses efficient treatment capabilities for elemental mercury (Hg⁰) removal on carbon steel's porous surface. However, the mercury removal process on the contaminated carbon steel was observed to cause iron (Fe) to leach on the material's surface. The results showed that the addition of imidazole as the corrosion inhibitor reduces 78.9% of Fe leaching with only 55% of Hg⁰ removal. Compared to the oxidants of sodium hypochlorite (NaOCl) and tert-Butyl hydroperoxide (TBHP), only peroxyacetic acid (PAA) possesses a positive influence on the I₂/KI reaction. Interestingly, PAA oxidant does not only enhance the Hg⁰ removal up to 99% and reduce 73% of the Fe leaching but also reduces half of the treatment time from 16 to 8 h. While in the addition of NaOCl and TBHP oxidants, the efficiency of the decontamination process was only obtained at 54% and 59% for Hg⁰ removal with 28.4% and 35.8% of Fe leaching, respectively.

Keywords: Carbon steel; Iron leaching; Lixiviant; Mercury; Peroxyacetic acid

Introduction

Mercury (Hg) is of great concern to human health due to its high toxicity, volatility, and persistence in the environment. Most of the Hg in water, soil, sediments, plants, and animals is found in the form of inorganic Hg or organic Hg (e.g., methylmercury) (Keating *et al.*, 1997), while Hg in the atmosphere is mainly found in the form of vapor (Chalkidis *et al.*, 2020). Crude oil and unprocessed gas from petroleum products can contain a significant amount of Hg.

The Hg from petroleum industries and power plants is emitted primarily as Hg vapor, consisting mainly of elemental Hg (Hg⁰) and dimethylmercury. Hg⁰ is mobilized to the atmosphere where it is subjected to atmospheric oxidation processes to yield water-soluble forms, and it is subsequently scavenged by wet or dry deposition, while dimethylmercury degrades relatively faster, compared to Hg⁰ in the air (Pacyna *et al.*, 2006). In gas processing, pipelines that carry

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Hg-contaminated fluids can become contaminated over time and damage equipment (Nengkoda *et al.*, 2009). Moreover, the interaction of Hg with pipe surfaces affects worker health.

Foust (1993) has developed a process for the mobilization/removal of Hg from solid wastes by using a lixiviant consisting of an aqueous solution of potassium iodide/iodine (KI/I₂). The oxidizer increases Hg solubility that transforms the insoluble Hg⁰ to very soluble Hg²⁺. Research by Ebadian *et al.* (2001) has also proved that using iodine/iodide lixiviant successfully removes more than 90% of Hg⁰ from the porous surface of the steel. Following the observations of Mattigod *et al.* (1999), the KI functions as a complexing agent or solubilizing agent, which reacts with the oxidized Hg to form a water-soluble compound with the formula K₂HgI₄ as Eq. (1) and Eq. (2). Chaiyasit *et al.* (2010) used the same technique on gas-processing pipeline carbon steel coupons of type API 5L-X52 after adsorption of Hg⁰ at 25°C in the presence of air and obtained a relative percentage of about 99% Hg⁰ removal. However, they only mentioned the surface corrosion of carbon steel at a higher concentration of I₂ without further investigation towards Fe leaching. Fe leaching from carbon steel surface during the removal process is crucial to be determined as its occurrence will affect the mechanical properties of the carbon steel leading to cracking and brittleness of the surface material (Siddiqui *et al.*, 2007; Jafari *et al.*, 2019). Fe contains more than 95% of carbon steel compositions while the rest may comprise of carbon, manganese, silica, phosphorus, sulfur, chromium, and nickel (Jafarzadegan *et al.*, 2012).



The use of a corrosion inhibitor is one of the most practical methods of protecting metals against Fe leaching that contributes to corrosion, and it is widely being applied to inhibit the dissolution of metal and acid consumption (Jafari *et al.*, 2019). As stated by Döner *et al.* (2011), the chemical inhibitors will be adsorbed on the metal surface while forming a protective film layer as a barrier. The use of imidazoline on X65 carbon steel as the corrosion inhibitor has been studied by Cai *et al.* (2018). They observed that the corrosion rate of X65 carbon steel in an oxygenated environment dropped when 200 ppm of imidazoline was applied.

Carbon steel is a material commonly used for the construction of many types of industrial equipment, such as tanks and pipes, because of its low cost and excellent mechanical properties

(Winnik, 2008). This paper presents the work of using a corrosion inhibitor and 3 types of oxidants towards minimizing Fe leaching during I₂/KI lixiviant treatment on carbon steel of SAE J429 after adsorption with Hg⁰ without deteriorating the removal performance. To the best of our knowledge, and as emphasized by previous studies on the removal of Hg vapor, no study has been conducted while considering the minimization of Fe leaching.

Materials and Methods

Materials

The Iodine and KI were purchased from Sigma and Merck, respectively, and were used for the preparation of the I₂/KI lixiviant. Three other types of oxidants used with the I₂/KI lixiviant for Hg⁰ removal were sodium hypochlorite (NaOCl, Merck), tert-Butyl hydroperoxide (TBHP, (CH₃)₃COOH, Merck), and peroxyacetic acid (PAA, CH₃CO₃H, Merck). Hydrogen peroxide (H₂O₂ 30%, Merck) and glacial acetic acid (GAA, CH₃COOH, Merck) were used in PAA preparation. Liquid Hg⁰ was supplied by Merck. Imidazole (C₃H₄N₂, Merck) was used as a corrosion inhibitor along with the I₂/KI lixiviant for Hg⁰ removal. All chemicals were used as received, without further purification. The SAE J429 carbon steel was purchased from a hardware shop. The chemical composition of the SAE J429 carbon steel is presented in Table 1.

Table 1. Chemical composition of the SAE J429 carbon steel

Chemical composition (wt.%)			
Fe	C	Si	Mn
99.22	0.25	0.06	0.40
P	S	Cr	Ni
0.007	0.005	0.05	0.01

Mercury Adsorption

Carbon steel was applied and contaminated with Hg⁰ using a physisorption method. During the preparation, the carbon steel was immersed directly into 0.3 kg of liquid Hg⁰ and left for 30 d for the physisorption of liquid Hg⁰ on the carbon steel surface to occur (Chaiyasit *et al.*, 2010).

Preparation of I₂/KI lixiviant

During the preparation of the 0.1 M I₂/0.5M KI solution, 12.69 g of I₂ and 41.5 g of KI were mixed, stirred, and diluted with deionized water in a volumetric flask (500 mL). The prepared I₂/KI lixiviant was stored in a glass bottle covered with aluminum foil until it was used. The same procedure

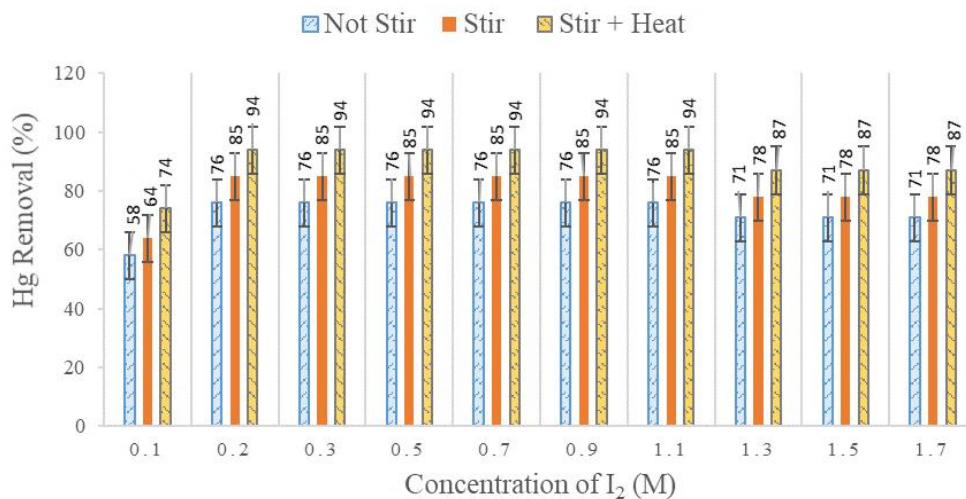


Figure 1. Removal of Hg⁰ from carbon steel at different concentrations of I₂ (0.1 M-1.7 M) and 0.5 M KI after 16 h of reaction

with different I₂ and KI mass was applied to prepare other concentrations of the I₂/KI lixiviant (varying from 0.1 M to 1.7 M).

Analytical Method

For the quantitative analytical study, the sample solutions were collected before the treatment reaction (as blanks) and after the treatment reaction. The amount of oxidized Hg⁰ (HgO) and Fe concentration present in the solution after the treatment reaction was examined using the mercury-hydride atomic absorption spectrometer (MHS-AAS, Perkin Elmer Analyst 400) and Flame-ASS, Perkin Elmer AA400, respectively.

The Removal of Hg⁰ from Carbon Steel Using the I₂/KI Lixiviant

For the Hg⁰ removal treatment, the Hg⁰-contaminated carbon steel was fully immersed in the I₂/KI lixiviant in a sealed bottle sample (250 mL) for 16 h. The experiment was carried out in a dark and closed box to prevent the loss of iodine due to sunlight (Chaiyasit *et al.*, 2010). Various concentrations of I₂ and KI (ranging between 0.1 M and 1.7 M) were taken and the study was conducted under different conditions, such as being left to soak at room temperature with no stirring (No Stir), stirring at room temperature (Stir) and stirring at 35°C-40°C (Stir + Heat). After the reaction ended, the I₂/KI solution was collected for the analysis of Hg⁰ removal and Fe leaching. The initial I₂/KI solution without Hg⁰ contaminated carbon steel was used as a blank. The carbon steel sample was removed and rinsed with distilled water for subsequent testing.

Corrosion Inhibition and Influence of Other Oxidants

These studies were conducted using the best Hg⁰ decontamination condition obtained, as discussed in Section 2.4 (at 0.2 M I₂, 0.5 M KI, stirred at 35°C-40°C). For the corrosion inhibitor study, 100 ppm of imidazole was added to the I₂/KI lixiviant during the Hg decontamination treatment which continued for 16 h. As for the influence of other oxidants, 500 ppm and 1000 ppm each of NaOCl, TBHP, and PAA oxidants were separately added. In this study, PAA (with an H₂O₂ to GAA mole ratio of 1:1) was applied. The 500 ppm /1000 ppm of PAA (250 mL) was prepared by mixing 0.38 mL/0.75 mL of H₂O₂ with 0.67 mL/1.34 mL of GAA, respectively, in a volumetric flask (50 mL) and diluted with distilled water. The reactions were allowed to continue.

Results and Discussions

The Removal of Hg⁰ and the I₂/KI Lixiviant

In this study, the experiments of Hg⁰ removal were carried out stepwise by (i) varying the I₂ concentration at constant KI, and (ii) varying the KI concentration with constant I₂.

Figure 1 shows the percentage of Hg⁰ removal by various concentrations of I₂, ranging between 0.1 M and 1.7 M, at a constant concentration of 0.5 M KI and under different experimental conditions. Our findings revealed that the percentage of removal of Hg⁰ was dependent on the concentration of I₂ and the experimental conditions. The results showed that the percentage of Hg⁰ removal was better under

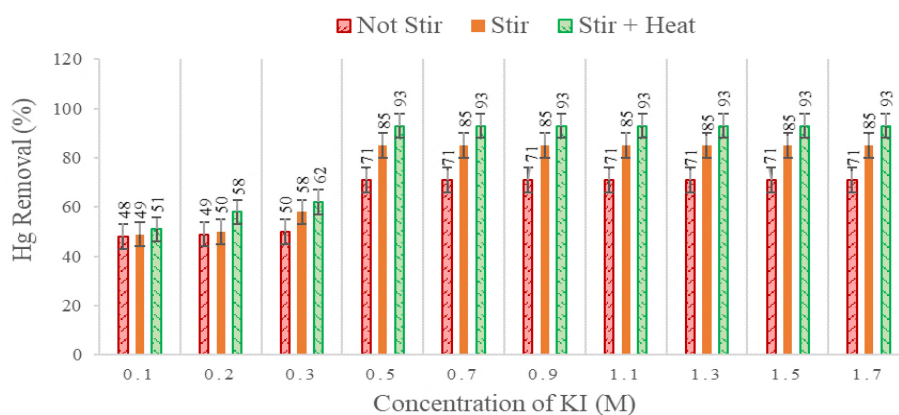


Figure 2. Removal of Hg° from carbon steel at different concentrations of KI (0.1 M–1.7 M) and 0.2 M I_2 after 16 h of reaction

Table 2. Leached Fe at Stir + Heat after 16 h of reaction

The concentration of I_2 or KI (M)	The concentration of leached Fe (ppm)	
	Varied I_2 at 0.5 M KI	Varied KI at 0.2 M I_2
0.1	341	411
0.2	360	406
0.3	375	400
0.5	365	360
0.7	371	360
0.9	369	360
1.1	372	360
1.3	400	360
1.5	402	360
1.7	411	360

As the Hg° mass was fixed during the carbon steel contamination, the lixiviant was found to achieve chemical equilibrium at 0.2 M–1.1 M I_2 with constant 0.5 M KI, as shown by a constant percentage of removal of Hg° . After exceeding this optimal dosing, the percentage of removal of Hg° decreased accordingly. This event could be explained by the equilibrium law, also called Le Chatelier's principle, whereby the increase in the concentration of I_2 will enhance the reversible reaction towards the formation of the starting reactant (I_2) and thus decrease the chemical reaction on oxidization of the Hg° (see Eq.1). This agreed with the study by Khaing *et al.* (2019), which discussed the aspects of gold leaching using iodide/iodine solutions.

stirred conditions, compared to non-stirred conditions, and it was best when the stirring was conducted at a high temperature of 35°C - 40°C.

All treatment conditions showed a similar trend of Hg° removal; the removal increased when the I_2 concentration was increased to 0.2 M and became consistent at I_2 concentrations between 0.2 M and 1.1 M. The removal decreased when the I_2 concentration was increased to 1.3 M and became consistent until the I_2 concentration was increased to 1.7 M. The percentage of removal of Hg° ranged from 58% to 94%. The lowest removal was obtained at 0.1 M I_2 under non-stirred conditions, while the highest removal was achieved at 0.2–1.1 M I_2 under the Stir + Heat condition. Stirring increases the dissolution rate of Hg, as Marek (1997) observed when studying the dissolution rate of Hg vapor in H_2O_2 , and the increased temperature increases the extraction efficiency of the I_2/KI lixiviant (Foust, 1993). The removal of the Hg° reached the optimum level at the I_2 dosing concentration of 0.2 M–1.1 M.

The percentage of Hg° removal at different concentrations of KI (between 0.1 to 1.7 M), 0.2 M I_2 , and under different experimental conditions are shown in Figure 2. The best concentration of iodine (0.2 M) used in this set of experiments was chosen from the previous set of experiments. A similar trend of increased removal was observed when the treatment was conducted in a stirred environment and at a high temperature of 35°C - 40°C. The removal of Hg° increased slightly (average of 48–51% to 50–62%) when the KI concentration was increased from 0.1 M to 0.3 M, and it increased significantly when the KI concentration was increased to 0.5 M. Nevertheless, when the KI concentration was further increased, no further improvement in Hg° removal was observed. The lowest removal (48%) was obtained at 0.1 M KI and a non-stirred condition, while the highest (93%) was obtained at 0.5 M KI and a stir + heat condition. Similar to the optimum concentration of I_2 , the

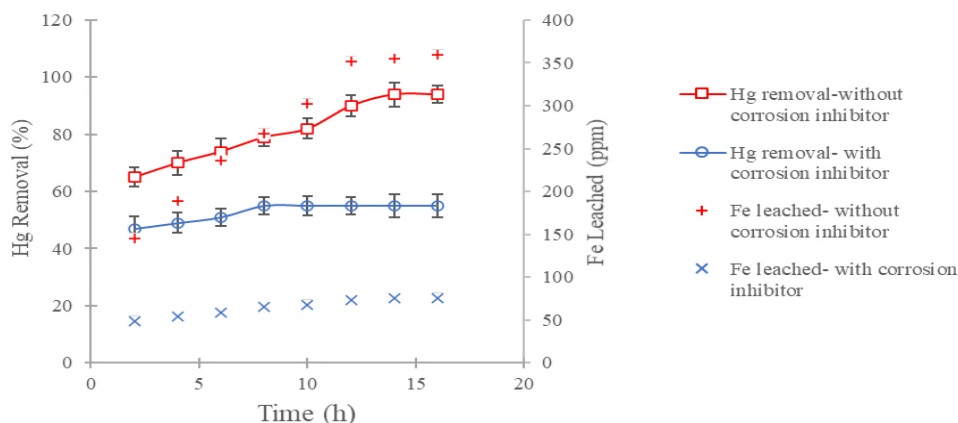


Figure 3. The effect of the corrosion inhibitor towards the removal of Hg° and the leaching of Fe under the Stir + Heat condition in 0.2 M I_2 /0.5 M KI

lixiviant was found to achieve chemical equilibrium at 0.5 M KI with a constant of 0.2 M I_2 , as shown by the constant percentage of Hg° removal. In this case, the KI sufficiently solubilized the I_2 and complexed all Hg_2^+ in the system. This result is also in agreement with that of Foust (1993).

The concentrations of Fe leached from carbon steel, determined at different concentrations of I_2 and KI, after 16 h of decontamination in the Stir + Heat condition are given in Table 2. At a constant 0.5 M KI, the leaching of Fe was observed to increase, as a higher concentration of I_2 was applied. A slight increase of Fe from 341 ppm to 360 ppm was detected when the I_2 concentration was increased from 0.1 M to 0.2 M. As the I_2 concentration was increased from 0.2 M to 1.1 M, more Fe was leached but in a fluctuating pattern, ranging from 360 ppm to 375 ppm. However, at a higher I_2 concentration (1.3 M to 1.7 M), Fe leaching was observed to even though the Hg° removal was observed to have decreased (from 94% to 87%) (refer to Figure 1). This trend is in agreement with the fact that I_2 is an oxidizing agent and will simultaneously oxidize Hg° and corrode carbon steel (indicated by Fe leaching) (Chaiyasit *et al.*, 2010). Increasing the oxidizing agent will tend to increase the leaching of Fe, even though the Hg° oxidation (Hg° decontamination) at a high I_2 concentration has achieved chemical equilibrium.

A different result was observed when the concentration of KI was varied at a constant of 0.2 M I_2 . At 0.1 M to 0.3 M KI, Fe leaching was observed to have slightly decreased, ranging from 411 ppm to 400 ppm. The leaching of Fe was observed to be constant at 360 ppm when the concentration of KI was varied between 0.5 M and 1.7 M. It was observed that at 0.1 M to 0.5 M KI,

the amount of KI was sufficient to dissolve the available I_2 that had undergone the oxidation process until the concentration of leached Fe became linear after 0.5 M KI (Foust, 1993).

Addition of Corrosion Inhibitor

The corrosion inhibitor is particularly useful for minimizing the leaching phenomenon. This is due to the involvement of inhibitor molecules in the interaction of the steel surface with the reaction sites, which reduces the contact between iron and the aggressive medium, thereby reducing the leaching of iron (Jafari *et al.*, 2019).

Figure 3 illustrates the effect of the corrosion inhibitor on Hg° removal and Fe leaching. It was found that the addition of a corrosion inhibitor reduced the corrosion of the carbon steel, as only 76 ppm of leached Fe was found in the aqueous solution, compared to 360 ppm of leached Fe without the corrosion inhibitor, after 16 h of treatment.

However, the corrosion inhibitor also reduced the effectiveness of the I_2 /KI lixiviant removal of Hg° . The percentage of removal of Hg° with the corrosion inhibitor was reduced to 55%, as compared to 94% of Hg° removal without the corrosion inhibitor. The reduction of Hg° removal was possible as the corrosion inhibitor formed an adsorption film on the carbon steel surface, which may have also inhibited the oxidation of Hg° by the I_2 /KI lixiviant.

Influence of Other Oxidants

Figure 4 shows the removal of Hg° under the influence of three different oxidants at the end of 10 h of reaction. All oxidants showed an increase in the removal of Hg° when their concentration was increased from 500 ppm to 1000 ppm.

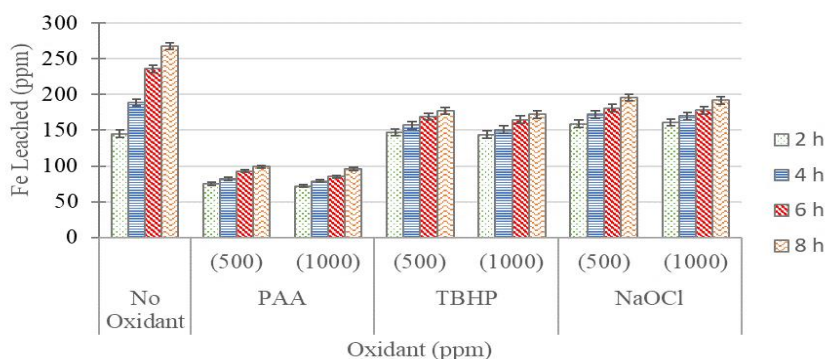


Figure 4. The influence of different types and concentrations of oxidants towards the removal of Hg° in the I_2/KI lixiviant

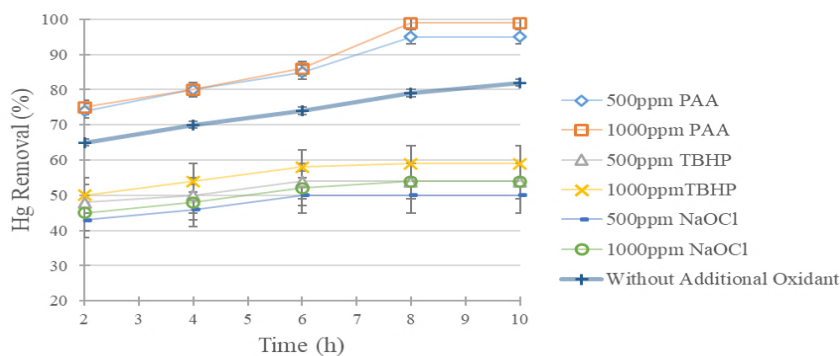


Figure 5. The effect of additional oxidants towards the leaching of Fe in the 0.2 M $\text{I}_2/0.5$ M KI lixiviant system

The highest percentage of removal of Hg° was observed with the addition of 1000 ppm of PAA, which resulted in achieving an average of 99% of Hg° removal. Both the addition of TBHP and NaOCl gave low percentage removal of Hg° at under 60%. Further, 500 ppm of NaOCl resulted in the lowest percentage removal of Hg° at 50%. Wigfield and Perkins (1985) studied the compounds bearing the -O-O-H functionality towards the oxidation of Hg° in aqueous solutions. The findings proved that PAA is a far more effective oxidant than TBHP, and it appears that the carbonyl group of PAA may be playing a role in the oxidation process. Additionally, without the carbonyl group, TBHP showed little oxidation that involved the diffusion-controlled formation of an aqueous solution of Hg° , coupled with a small amount of oxidation. While TBHP and NaOCl have less power to enhance the I_2/KI lixiviant for the oxidization of Hg° , both TBHP and NaOCl have a high possibility of reacting with the I_2/KI lixiviant (Lister and Rosenblum, 1963; Wu *et al.*, 2014; Wang *et al.*, 2017). Based on the study by

Khaing *et al.* (2019), I_2 and KI in the I_2/KI lixiviant produce triiodide (I_3^-), which later oxidizes the Hg° and forms complexes with KI. As the I_2/KI lixiviant is reduced by the TBHP and NaOCl oxidants, the removal of Hg° is also reduced.

The total Fe leached during the oxidation of Hg° in the presence of additional oxidants is shown in Figure 5. It was observed that the addition of the oxidants decreased the concentration of Fe. The leaching of Fe increased with an increase in the total time allotted for the reaction and was lower at a higher oxidant dosage. Interestingly, the addition of PAA led to the lowest concentration of Fe leached (below 100 ppm). As the addition of PAA enhanced the decontamination reaction of the I_2/KI lixiviant (99% of Hg° removal after 8 hours), the I_2/KI lixiviant achieved chemical equilibrium within a short amount of time, and therefore, the leaching of Fe was reduced. For both of the oxidants TBHP and NaOCl, the leaching of Fe was also reduced due to the decreased oxidation power of the I_2/KI lixiviant, as it was affected by the oxidants.

Conclusions

0.2 M I₂/0.5 M KI lixiviant ratio with stirring and heating at 16 h of treatment time demonstrated a potential for Hg^o removal from the carbon steel surface, with a concentration of 360 ppm of leached Fe. It was found that the addition of 1000 ppm PAA enhanced the removal of Hg^o to 99% within 8 h of reaction time, with the reduction of Fe leaching to 96 ppm. The study shows that PAA is a good additive for enhancing the performance of the I₂/KI lixiviant towards Hg^o removal from the carbon steel surface.

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