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RESEARCH PAPER

The role of internal intensifier microemulsion for monitoring acid anti-corrosion activity of enhanced cinnamaldehyde contained-PMMA nanocolloid in industrial pipeline simulations

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Abstract

The quest to replace toxic and hazardous chemicals in the future in all industrial pipelines is interested in revolutionizing the anti-corrosion material research world by turning its attention to green formulations based on nanotechnology. Herein, we report a new combination of corrosion inhibiting potential of cinnamaldehyde contained Poly(methyl methacrylate) nanocolloid and KI internal intensifier mixture on the corrosion of C1018 steel in 3 Wt.% HF and 15 Wt.% HCl solution. KI internal intensifier was prepared by water-in-oil microemulsion and was characterized with Dynamic Light Scattering (DLS) analysis. The average particle size of the KI internal intensifier containing surfactant-based microemulsion was 73 ± 2 nm. We also report the effect of the inhibitor concentration, types of steel alloy, intensifier additive concentration, and temperature on the corrosion-inhibiting performance of the corrosion inhibitor + KI microemulsion mixture. corrosion inhibitor + KI microemulsion mixture exhibits inhibiting ability but the extent of inhibition is dependent on concentration, temperature, and intensifiers' concentration. The adsorption of cinnamaldehyde-contained polymer + organic sulfur compound + KI internal intensifier is synergistic. An increase in the temperature study resulted in a slight decline in the inhibition efficiency of corrosion inhibitor + KI with an efficiency of above 95% achieved at 343 K and inhibition efficiency by amine film forming on the surface of alloy was confirmed with Atomic Force Microscopy (AFM) and compared to uninhibited alloy sample.

1. Introduction

In the oil and gas industry for protecting well casings and other tubular goods in a well from the corrosive effects of an acid that is used in suitable acid stimulation technologies useful for dissolving calcium and magnesium-based carbonates there exists a need for green inhibitors with high efficiency [1]. It is clear that a significant portion of the world's oil and gas reserves lie in carbonate reservoirs and these were estimated the values of 60% and 40%, respectively. The mineralogy of these heterogeneous carbonate formations primarily consists of calcite, dolomite, or combinations thereof [2]. Production enhancement methods based on the nanomaterials routinely rely on the use of suitable acid stimulation technologies useful for dissolving calcium and magnesium-based carbonates [3]. Several acid platforms have been proposed and are widely used by oil and gas operators to stimulate rock formations. These include but are not limited to the use of strong mineral acids (for example, hydrochloric acid, HCl), gelled and emulsified acids, organic-based acids such as formic acid and acetic acid, and combinations thereof [4]. While these technologies are effective, strong acids are corrosive to steel and other metal substrates that are present in the wells, for example, as part of the well casings [5]. Corrosion represents a large portion of the total costs for oil and gas production because corrosion occurs at all stages from downhole to surface equipment and processing facilities. For example, leaks in tanks, casings, tubing, pipelines, and other equipment often stem from corrosion. Choudhary et al. [6] synthesized cinnamaldehyde thiosemicarbazone as a corrosion inhibitor on the deterioration of mild steel in 1M and 15% HCl. Recently, Noor Mohammad Beigi [7] prepared e novel type of cinnamaldehyde contained poly(methyl methacrylate) nanocolloid as an efficient acid anti-corrosive for steel in acidic media. He investigated the role of amine film forming on the surface of metal alloys with atomic force microscopy (AFM). This type of inhibitor has highlighted the influence of a unique combination of cinnamaldehyde-contained poly(methyl methacrylate) based on the microemulsion system on the deterioration of alloy surface in 4% HCl and 15% HCl at different temperatures 303 K-443 K.

The present study provides a variety of insights relating to technologies for alleviating corrosion in well casings and other tubular goods in a well via an acid corrosion inhibitor based on the unique combination of contained poly(methyl methacrylate) based on the microemulsion system and internal intensifier-containing surfactant free microemulsion. Traditional microemulsion, as a soft template [8-12] can be used for several nanotechnologies such as nanoparticle preparation [13-16], nanocomposites fabrication for several applications for example wastewater treatment [17-23], anti-corrosion materials [24,25], and so for. To our knowledge, this effect of internal intensifier-containing surfactant-free microemulsion as a stable nanocolloid has not been investigated by researchers to the best of our knowledge. Here, we prepared a cinnamaldehyde-contained poly(methyl methacrylate) nanocolloid and then the effect of the internal intensifier nanocolloid with a viewpoint of the investigation of the concentration of internal inhibitor, internal intensifier concentration, the effect of steel type surface and also temperature on the corrosion performance were studied.

2. Experimental

2-1. Materials

Lecithin as stabilizer agent to produce oil-in-water microemulsion, methyl methacrylate monomer ($CH_2C(CH_3)COOCH_3$) (AR grade), were purchased from Merck Company. Cinnamaldehyde ($C_6H_5CH{=}CHCHO$) as filming agent, thiourea (CH_4N_2S) active corrosion intermediate, and Potassium iodide-starch paper (KI) as internal intensifier were purchased from Sigma-Aldrich. Potassium persulfate as a hydrophilic initiator for the polymerization of MMA was purchased from Haihang Industry Co. LTD. Deionized water was used for all experiments. C1018, P110, and N80 alloy that the chemical compositions of the alloys are summarized in Table 1, was received for acid anti-corrosion study.

Table 1. The chemical compositions of the exemplary alloys

Chemical composition (%) of C1018 alloy									
C	Si	Mn	P	S	Cr	Ni	Cu	Mo	V
0.14-0.20	-	0.60-0.90	≤ 0.040	≤ 0.050	-	-	-	-	-
Chemical composition (%) of P110 alloy									
0.26~0.395	0.17-0.37	0.40-0.7	≤0.020	≤0.010	0.80-1.1	≤0.20	≤0.20	0.15-0.25	≤0.08
Chemical composition (%) of N80 alloy									
0.34-0.38	0.20-0.35	1.45-1.7	≤0.020	≤0.015	≤0.15	-	-	-	0.11-0.16

2-2. Preparation of the acid anti-corrosive

In this section, the procedure of Noor Mohammad Beigi was used for the preparation of the acid anti-corrosive [7,24]. Briefly, PMMA nanoparticles were prepared in amino acid-based oil-in-water microemulsion composed of lecithin as a surfactant, MMA as an apolar phase that adjusted at %2 (Wt.), and water should be obtained. Potassium persulfate was used as an oxidizing agent and initiator to generate the free radicals and to start the polymerization at 60°C for 4 h [7, 26, 27]. Next, to fabricate cinnamaldehyde contained PMMA nanoparticles, the water-in-cinnamaldehyde oil microemulsion system was used, and an aqueous PMMA nanocolloid was replaced with the water that the PMMA nanocolloid was fixed at 5 % (Wt.). Finally, to produce acid anti-corrosive material, thiourea as an organic-sulfur compound with the molar ratio of cinnamaldehyde to thiourea = 3 was mixed with the above nanocolloid.

2-3. Preparation of the internal intensifier-containing surfactant based-microemulsion

The same procedure and formulation that was used to prepare cinnamaldehyde contained PMMA nanocolloid was also employed to fabricate internal intensifier microemulsion but KI solution (0.05 mol/L) was replaced with PMMA nanocolloid.

2-4. Characterization

Zeta-average diameters (Dz), particle Size distributions, polysispersity index, and also zeta potentials to confirm the stability of polymer nanocolloid and particle charge were studied by a dynamic light scattering (DLS) using a Zeta plus−Zeta potential Analyzer (Brookhaven Instruments Corporation).

2-5. Gravimetric analysis to study the corrosion test

To study the impact of acid corrosion inhibitor, inhibitor concentration, and also impact of temperature, weight loss-based C1018, P110, and N80 alloys surface corrosion analysis for all experiments were carried out at different temperatures from 343 K to 373 K in 3% HF and 12% HCl in the absence of inhibitor as control sample and the presence of new green inhibitor at 2-5 Wt.% concentrations, respectively that the total exposure time of 4 h was selected. The experiment conditions are as follows: 50 ml of uninhibited or control sample and working solution (inhibited corrosion solution) was employed, and the size of the metal surface samples was 8.7 cm × 1.4 cm × 0.1 cm. The following equation (Eq. 1) was utilized for the calculation of the corrosion rate of the prepared green inhibitor and control sample [7].

$$\eta\ (\%) = w_o - w / w_o \times 100 \qquad (1)$$

$$\text{Corrosion rate (mm/Y)} = K_2 \Delta m / \rho.A.t \qquad (2)$$

where w_0 and w are the weight loss of C1018, P110, and N80 alloys in the absence and presence of the acid anti-corrosive, respectively. K2 is a conversion constant from cm h1 to mm y1 (87,600), Δm is the mass change, ρ is density (in g/cm3), A is the sample area (in cm^2), and t is the immersion time (in h). In corrosion study, an acid corrosion inhibitor provides sufficient corrosion inhibition so that a pitting index has definitions of the integers. A pitting index estimates pitting (for example, localized corrosion) susceptibility with integer values ranging from 1 through 5, where a value of 1 designates a low susceptibility to pitting and 5 is a high susceptibility [28].

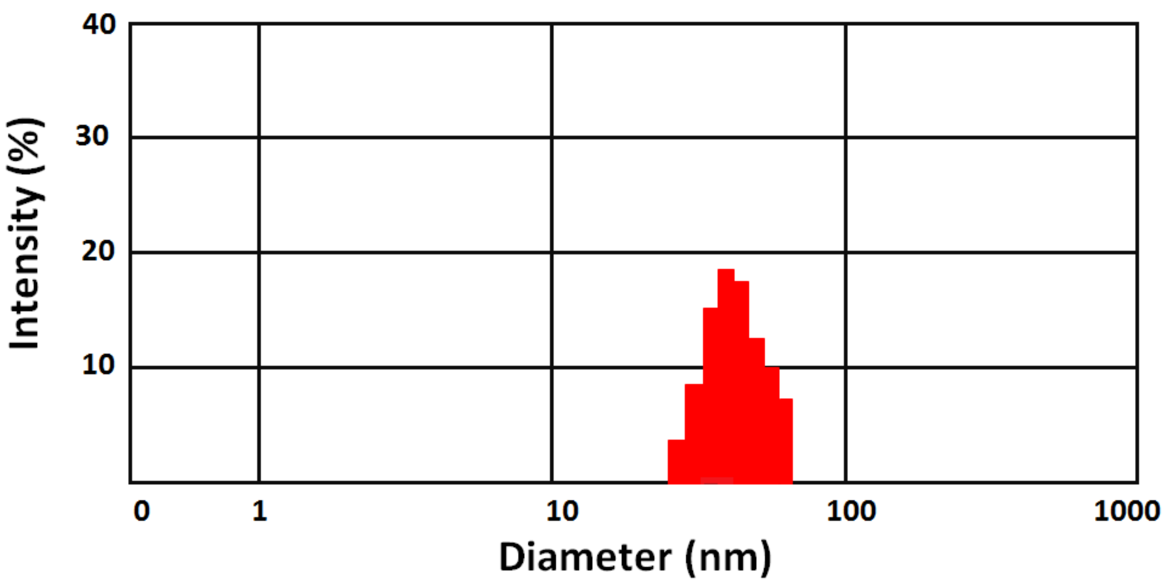

Fig. 1. DLS analysis of KI internal intensifier W/O microemulsion

3. Result and discussion

3-1. DLS analysis

The particle size distribution of KI internal intensifier microemulsion was determined using DLS technique (Fig. 1). Data was recorded without sonication process. The particle size of KI internal intensifier microemulsion prepared in W/O microemulsion was 73 ± 2 nm, PDI=0.25 with Z-average 120 nm according to DLS analysis. Microemulsion system as nanoreactor is useful system to prepare a stable colloid solution with high monodispersity. Based on the composition that Noor Mohammad Beigi introduced for preparation of PMMA nanocolloid, the average PMMA particle size is 86 nm, Z-average diameter (Dz) (<101 nm), low polydispersity index (0.05), high positive zeta-potential (-28.34 mV) were observed for the polymer nanocolloid prepared in O/W system as soft template method [7].

Table 2. Effect of acid anti-corrosion inhibitor concentration with 1 Wt.% KI intensifier

Inhibitor (%)	T (°C)	HCl (%)	HF (%)	KI (%)	Alloy	η (%)	Corrosion rate	Pitting
	303	12	3	1	C1018	79.75	0.0124	2
2	323	12	3	1	C1018	75.25	0.0803	2
	343	12	3	1	C1018	68.3	0.1134	3
	303	12	3	1	C1018	98.78	0.0020	0
3	323	12	3	1	C1018	98.20	0.0025	0
	343	12	3	1	C1018	95.57	0.0028	0
	303	12	3	1	C1018	97.23	0.0030	0
4	323	12	3	1	C1018	94.63	0.0030	0
	343	12	3	1	C1018	91.33	0.0039	0
	303	12	3	1	C1018	95.19	0.0038	0
5	323	12	3	1	C1018	94.32	0.0039	0
	343	12	3	1	C1018	91.30	0.0030	1

3-2. Effect of acid anti-corrosion inhibitor concentration

Corrosion problems can exist at every stage of production, from the initial extraction to refining and storage before use, and the need a corrosion inhibitor is necessary. In this study, different inhibitor concentration of cinnamaldehyde-contained poly(methyl methacrylate) nanocolloid (2-5 Wt.%) in the presence of KI microemulsion system (1 Wt.%) was used to investigate the optimal concentration of acid anti-corrosive to protect of the C1018 alloy surface at 303 K, 323 K, and 343 K. As can be found from Table 2, it is clear that the 3 Wt.% of the chemical substance is an effective concentration in very small amounts of internal intensifier concentration when added to a corrosive environment to decrease the corrosion rate of the exposed C1018 alloy. Minor changes in the inhibition effectiveness (η) were observed at high concentrations of inhibitor. At low concentrations of acid anti-corrosive due to the low amount of amine film forming in the composition of inhibitor and weak inhibition of C1018 alloy surface, a significant change in the inhibition effectiveness happened. Their η increases with increased corrosion inhibitor concentration and they mainly act as mixed with internal intensifier and also amino acid, cinnamaldehyde, and thiourea compound in composition. Cruz et al. [31] tested 2-aminomethylbenzimidazole and bis(benzimidazol-2-ylethyl)sulphide as anti-corrosive materials for alloy surfaces in deaerated 0.5 mol/L HCl. They reported that the former acts as a cathodic-type inhibitor and the latter as a mixed-type inhibitor.

3-3. Effect of internal intensifier concentration on the promoting acid anti-corrosion inhibitor

To formulate a anti corrosion inhibitor cocktail (a mixture containing an active amine compound, surfactant, intensifier, solvent, co-solvent, etc.), an important aspect always investigated is how to achieve high performance or adequate corrosion inhibition at a low cost in all conditions. The use of internal intensifiers such as KI in corrosive inhibition of the prepared cocktail is sacrosanct due to the role of an intensifier that can be enhanced the inhibitive force of the active and as a consequent decrease the amount of active used [32].

Table 3. Effect of KI internal intensifier concentration on the corrosion inhibitory property

Inhibitor (%)	T (°C)	HCl (%)	HF (%)	KI (%)	Alloy	η (%)	Corrosion rate	Pitting
	303	12	3	1.00	C1018	98.78	0.0020	0
3	323	12	3	1.00	C1018	98.20	0.0025	0
	343	12	3	1.00	C1018	95.57	0.0028	0
	303	12	3	1.25	C1018	98.79	0.0013	0
3	323	12	3	1.25	C1018	98.24	0.0021	0
	343	12	3	1.25	C1018	96.03	0.0026	0
	303	12	3	1.50	C1018	97.88	0.0012	0
3	323	12	3	1.50	C1018	97.52	0.0018	0
	343	12	3	1.50	C1018	96.25	0.0023	0
	303	12	3	1.75	C1018	98.32	0.0008	0
3	323	12	3	1.75	C1018	98.46	0.0012	0
	343	12	3	1.75	C1018	97.25	0.0015	0

Table 3 shows the concentration effect of the KI microemulsion system for C1018 steel at different temperature in 12 wt.% HCl, and 3% HF solution. The obtained data from the analysis are also listed in Table 2. These sets of corrosion experiments were carried out to understand the extent to which the selected intensifiers alone can protect from the corrosion or cannot inhibit it. The concentration of the internal intensifier as microemulsion colloid was varied to determine the optimum concentration of the intensifier for all the tested experimental conditions. It is clear from Table 2 that the selected KI intensifier microemulsion exhibits corrosion inhibitory properties to a certain degree and concentration for C1018 alloy. The best concentration for KI for the corrosion inhibition performance is 1.75 Wt.% and this concentration afforded inhibition efficiency in the range of 97% with no pitting. The corrosion inhibition activity of all KI concentrations is very close and should be of benefit to use the low concentration. This seems to suggest that KI microemulsion intensifier is suitable as an intensifier under the studied conditions. For KI nanodroplets in a microemulsion system, the prevailing theory is that, in an acid solution, dissolved oxygen oxidizes iodide ions to triiodide and pentaiodide ions, which are preferentially adsorbed on the metal surface [33]. Table 3, also shows the variation of the corrosion rate and inhibition efficiency with temperature. Temperature promoted the dissolution of the carbon steel in both uninhibited and inhibited with anti-corrosive acid solutions. As can be seen, the corrosion rate increases with temperature rise. This observation is not surprising because an increase in temperature causes a rise in the energy levels of molecules involved in a reaction [34].

3-4. Effect of alloy surface

Fig. 2 shows the inhibition efficiency of the acid anti-corrosive + KI microemulsion mixture for different steels. It is worth pointing out that, despite the slight decline in the (a) inhibition efficiency η (%) and (b) the corrosion rate of C1018, P110, and N80 alloys at 303 K, inhibition efficiency above 98% was still achieved. This again shows the potential of acid anti-corrosive + KI microemulsion mixture in acting as acidizing corrosion inhibitors for all steel surface. The corrosion rate was also varied between 0.0012-0.0013.

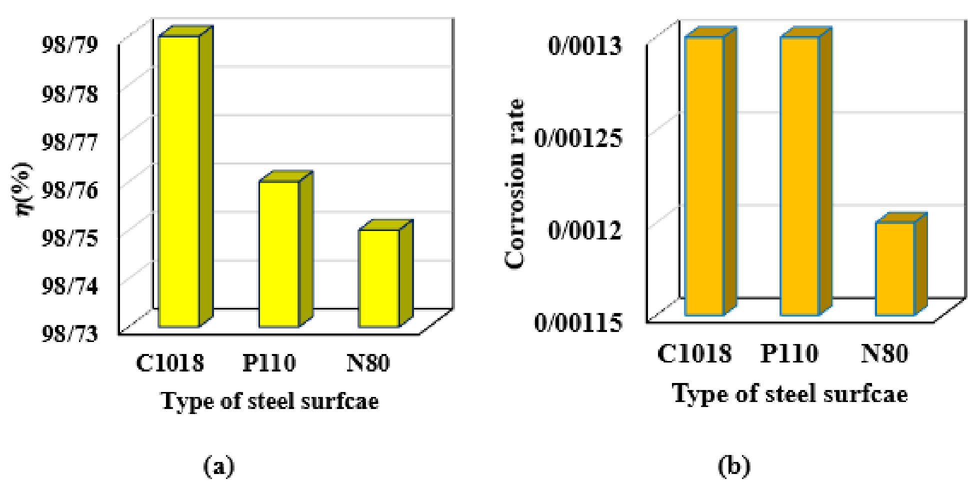

Fig. 2. (a) Inhibition efficiency η (%) and (b) the corrosion rate of C1018, P110, and N80 alloys at 303 K.

3-5. Surface analysis with AFM images

A detailed visualization of a tested C 1018 sample surface can be investigated by an AFM three-dimensional (3-D) image. As can be seen from Fig. 3, the surface with the highest roughness is always adjudged to indicate a severe corrosion attack and in this study, the corrosion attack was also observed for the uninhibited C1018 alloy surface. The C1018 steel surface exposed to the uninhibited acid solution (Fig. 3-a) exhibited the roughest topography (R=393.85 nm) relative to the inhibited C1018 surfaces shown in Fig. 3(b-d) at 303 K (R=94.23 nm), 323 K (R=130.64 nm) and 343 K (R=137.13 nm). In the present case, the lesser roughness value for protected/inhibited C1018 steel surfaces in comparison with the roughness value of the unprotected C1018 surface is most probably due to C1018 surface protection occasioned by the adsorption of the inhibitors molecules and amine film forming.

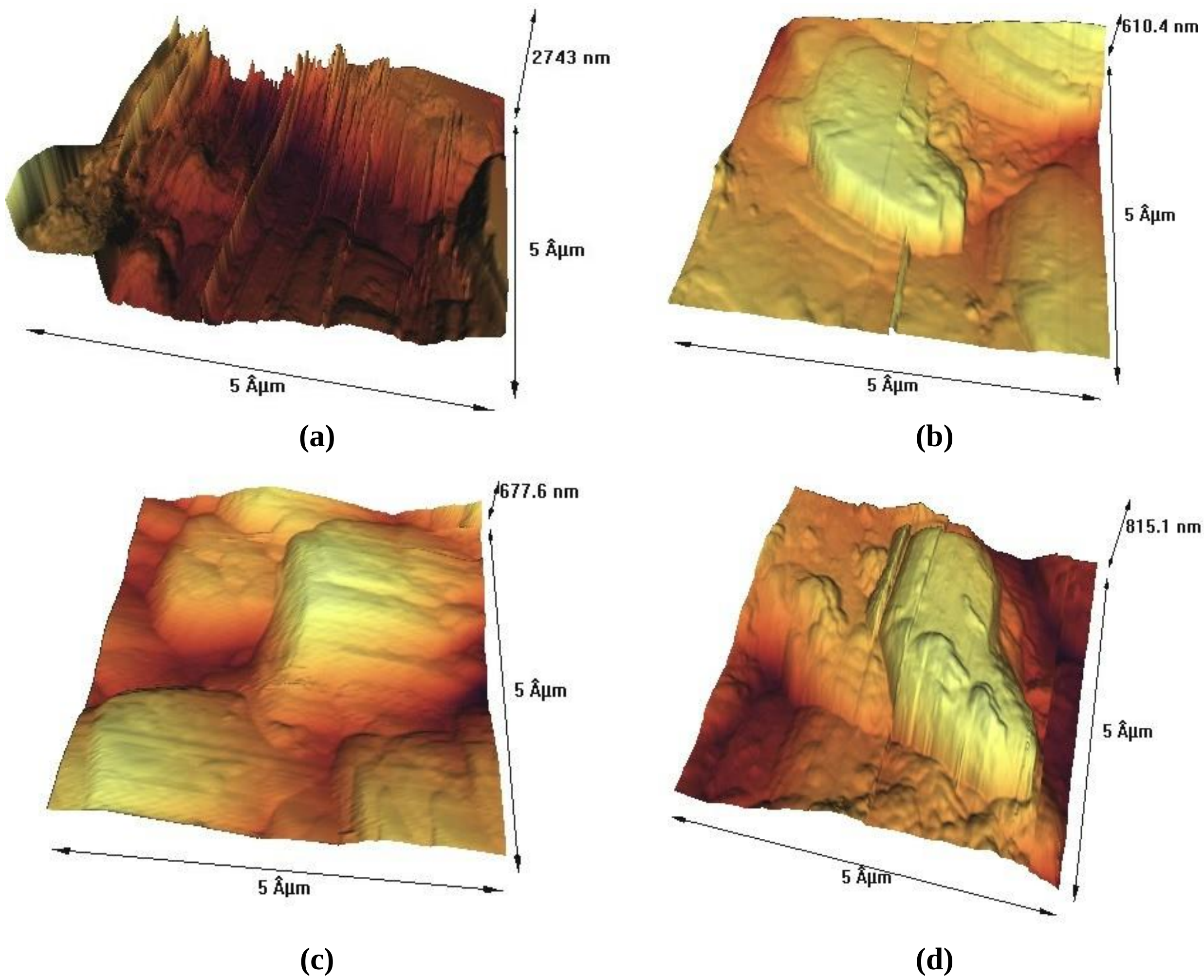

Fig. 3. AFM surface analysis of (a) uninhibited of C1018 alloy, and inhibited of C1018 surface at optimum condition at (b) 303 K, (C) 323 K, and (d) 343 K.

4. Conclusion

The corrosion inhibition performance of acid anti-corrosive inhibitor based on the cinnamaldehyde-contained poly(methyl methacrylate) nanocolloid on C1018 steel dissolution in 15 wt.% HCl and 3% HF solution was studied. The effect of internal intensifiers, namely potassium iodide (KI) microemulsion, as well as temperature on the corrosion inhibiting performance of cinnamaldehyde contained poly(methyl methacrylate) nanocolloid+ KI microemulsion, the effect of internal intensifiers concentration, types of alloy surfaces were also investigated. From the studies, the cinnamaldehyde-contained Poly(methyl methacrylate) nanocolloid+ KI microemulsion has the potential to serve as an active corrosion inhibitor package developed for different steel protection in an acidic media and KI at appropriate concentrations can be utilized as an intensifier for this inhibitor. With 3 Wt.% of inhibitors + 1 Wt.% KI, inhibition efficiency above of 95% can be realized at 343 K. The adsorption of inhibitor + KI is synergistic. An increase in the temperature of the acid solution would cause a slight decline in the inhibition efficiency inhibitor + 1Wt.% KI and inhibition efficiency of above 95% is achievable at 343 K. Amine film forming for inhibited samples at all temperatures was confirmed with AFM-3D image analysis. The obtained data of roughest topography revealed that after film formation on the surface of C1018 alloy, the roughest is less than the uninhibited C1018 sample. The use of inhibitor + KI mixture as corrosion inhibitors is advantageous because of certain properties such as eco-friendliness, biodegradability, renewability, and low cost when compared to synthetic organic and inorganic corrosion inhibitors. However, there are still some unanswered questions regarding the use of the mechanism of corrosion inhibition.

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