



Section 1. Chemistry

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SYNTHESIS OF IONIC LIQUIDS BASED ON ORTHO-PHENYLENEDIAMINE AND PHOSPHORIC ACID

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Abstract

Ionic liquids have attracted the attention of researchers due to their properties such as high solubility and stability, high ionic conductivity, and low toxicity. Compounds based on ionic liquids exhibit high water solubility and high inhibition efficiency at various temperatures and low concentrations. These compounds are highly effective in preventing metal corrosion in cooling and heating systems and consist of water-soluble, inexpensive components for corrosion protection. These properties make them a new generation of corrosion inhibitors and increase their potential for widespread use in the future. The influence of the pH value of the medium, time, and temperature on the synthesis of ionic liquids was studied. The efficiency of ionic liquids at different temperatures and concentrations was determined using results obtained by gravimetric analysis and analyzed in detail by atomic force microscopy (AFM).

Keywords: Ionic liquid, 1,2-diammoniumbenzene hydrophosphate, physical sorption, chemical sorption, gravimetric analysis, and AFM (atomic force microscopy)

Introduction

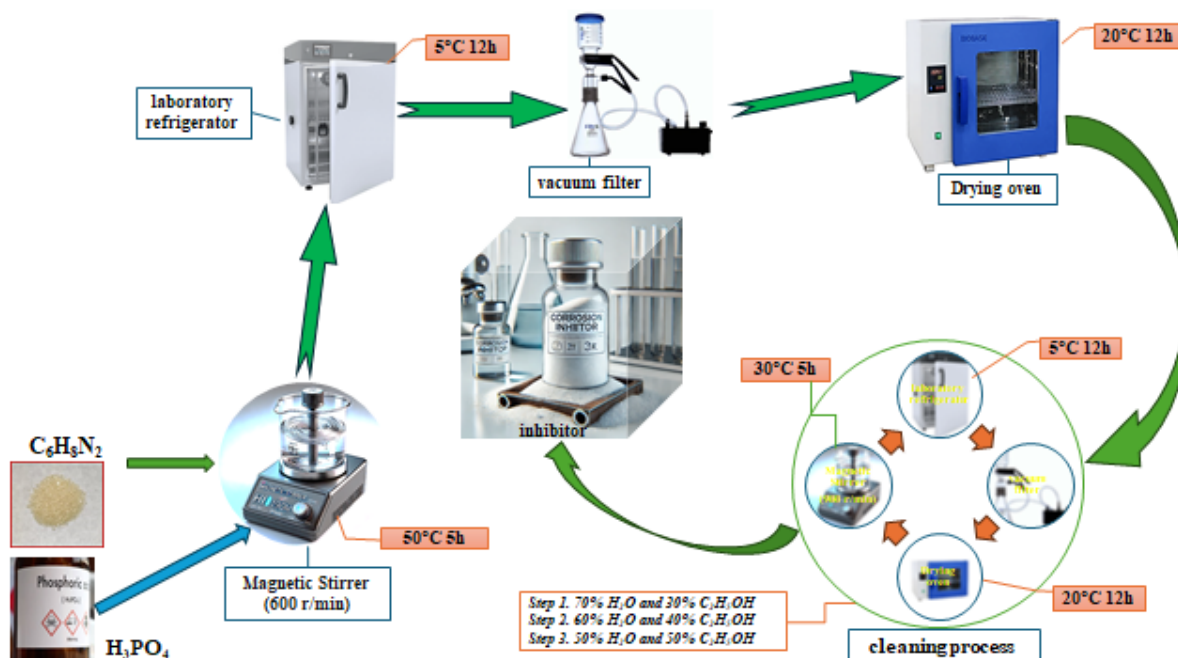
Ionic liquids are salts of organic compounds, consisting of organic cations and organic or inorganic anions. Ionic liquids typically possess high thermal stability, low vapor pressure, non-flammability, moderate polarity, high ionic conductivity, and non-toxic properties (Gobara M., Saleh A., Naeem I., 2019). Inhibitors based on ionic

liquids form a strong protective thin film and adsorb onto the metal surface. Chemical and physical adsorption mechanisms are the most suitable for describing the adsorption of inhibitors and the formation of a protective layer. Effective corrosion protection can be achieved by forming a stable protective layer at the boundary between the metal surface and the solution (Tawfik S. M.,

2016; Lgaz H., Anejjar A., Salghi R., Jodeh S., Zougagh M., Warad I., Larouj M., Sims P., 2016). As a result, the diffusion or penetration of ions, water molecules, and oxygen to the surface is significantly reduced. Derivatives of ionic liquids contain many nitrogen atoms; therefore, their nitrogen atoms are protonated. The results obtained in this research work showed that physical adsorption occurs due to electrostatic interaction between the positively charged regions of the inhibitor and the negatively charged metal surface (Cao S., Liu D., Zhang P., Yang L., Yang P., Lu H., Gui J., 2017). On the other hand, they adsorb onto the metal surface by forming strong covalent bonds. Almost all ionic liquids have low toxicity and high efficiency, which has attracted many researchers. As a result of these investigations, the anti-corrosion properties and mechanisms of ionic liquids on steel in saline water were studied. Ionic liquids serve as a protective layer on the metal surface. This, in turn, depends on the inhibition efficiency, which in-

creases with increasing concentration and decreases with increasing temperature. Currently, most of the studied research is based on solid ionic liquid inhibitors (Gerengi H., Solomon M. M., Umoren S. A., Ugras H. I., Yildiz M., Slepiski P., 2018; Qi-bo Z., Yi-Xin H., 2011). The use of ionic liquids such as 1,2-diammoniumbenzene hydrophosphate, N, N-diphenylammonium dihydrogenphosphate, 2-methylbenzylammonium dihydrogenphosphate, and 4-(diazophenyl) benzylammonium dihydrogenphosphate, as well as the application of their anti-corrosion activity, is also motivating scientists' interest. Our research group synthesized an ionic liquid based on a compound of ortho-phenylenediamine and phosphoric acid, and then, using several experimental and computational methods, studied the anti-corrosion effect of the ionic liquid on a steel sample in 0.5 M HCl and 0.5 M NaOH media (Nanthini R., Mutalib M. I., 2019; Hasib-ur-Rahman M., Larachi F., 2013).

Figure 1. Synthesis process of the 1,2-diammoniumbenzene hydrophosphate ionic liquid derived from ortho-phenylenediamine and phosphoric acid



In this study, the 1,2-diammoniumbenzene hydrophosphate ionic liquid inhibitor demonstrated excellent corrosion inhibition properties for steel samples in an acidic solution. A 0.5 M solution of ortho-phenylenediamine in 50 ml of alcohol was

mixed with a 0.5 M solution of phosphoric acid in a 1:1 ratio in a flask. Both solutions were initially stirred separately for 5 minutes, and then the ortho-phenylenediamine solution in alcohol was slowly poured into the phosphoric acid solution. This mix-

ture was attached to a reflux condenser and reacted using a magnetic stirrer at 50 °C ($t = 5$ h) and 600 rpm. After a homogeneous mixture was formed, the obtained solution was cooled to room temperature and placed in a refrigerator at 5 °C for 12 hours to allow the ionic liquid to form. The resulting ionic liquid solution was filtered. The obtained ionic liquid was dried at 20 °C for 12 hours to obtain the dry ionic liquid. The effect of 1,2-diammoniumbenzene hydrophosphate ionic liquids on corrosion inhibition indicators is studied in various aggressive media on different metals or alloys.

Results obtained via gravimetric analysis and AFM (atomic force microscopy) and their analysis: Effect of the concentration of 1,2-diammoniumbenzene hydrophosphate ionic liquid. The corrosion rate (CR_{Grav.}), the protection degree (η _{Grav.}), and the surface coverage (θ _{Grav.}) for steel No. 20 in 0.5 M HCl with the 1,2-diammoniumbenzene hydrophosphate inhibitor are presented in Table 1. It is evident that with increasing concentration, η _{Grav.} and θ _{Grav.} increase, but CR_{Grav.} decreases. This is because the increase in inhibitor concentration coats the metal surface with a thin protective film. At 303 K, for steel No. 20, a protection degree of 97.54% was observed at 100 mg/L and 92.75% at 75 mg/L. However, it was confirmed that the 1,2-diammoniumbenzene hydrophosphate inhibitor is a good inhibitor for the corrosion of steel No. 20 even at low concentrations in a 0.5 M HCl medium. The magnitude of the inhibitory efficiency of 1,2-diammoniumbenzene hydrophosphate depends on the molecular volume of the inhibitor. This is because even at low concentrations, the inhibitor covers a large part of the metal surface. The experimental results show that the selected inhibitor is more effective at low concentrations. This is related to the adsorption of the 1,2-diammoniumbenzene hydrophosphate inhibitor in a parallel adsorption position. As a result, it covers a large portion of the metal surface. The increase in concentration is related to a change in the adsorption state. Thus, 1,2-diammoniumbenzene hydrophosphate is an inhibitor that is effective even at low concentrations. In the temperature range of 303–333 K, η _{Grav.} at 100 mg/L is 97.54% at 303 K and

83.06% at 333 K. Therefore, this 1,2-diammoniumbenzene hydrophosphate ionic liquid is considered a good corrosion inhibitor at high temperatures as well. Thus, the 1,2-diammoniumbenzene hydrophosphate inhibitor forms an effective protective film on the surface of steel No. 20 and effectively isolates it from aggressive Cl^- ion attack (Kamali Ardakani E., Kowsari E., Ehsani A., 2019).

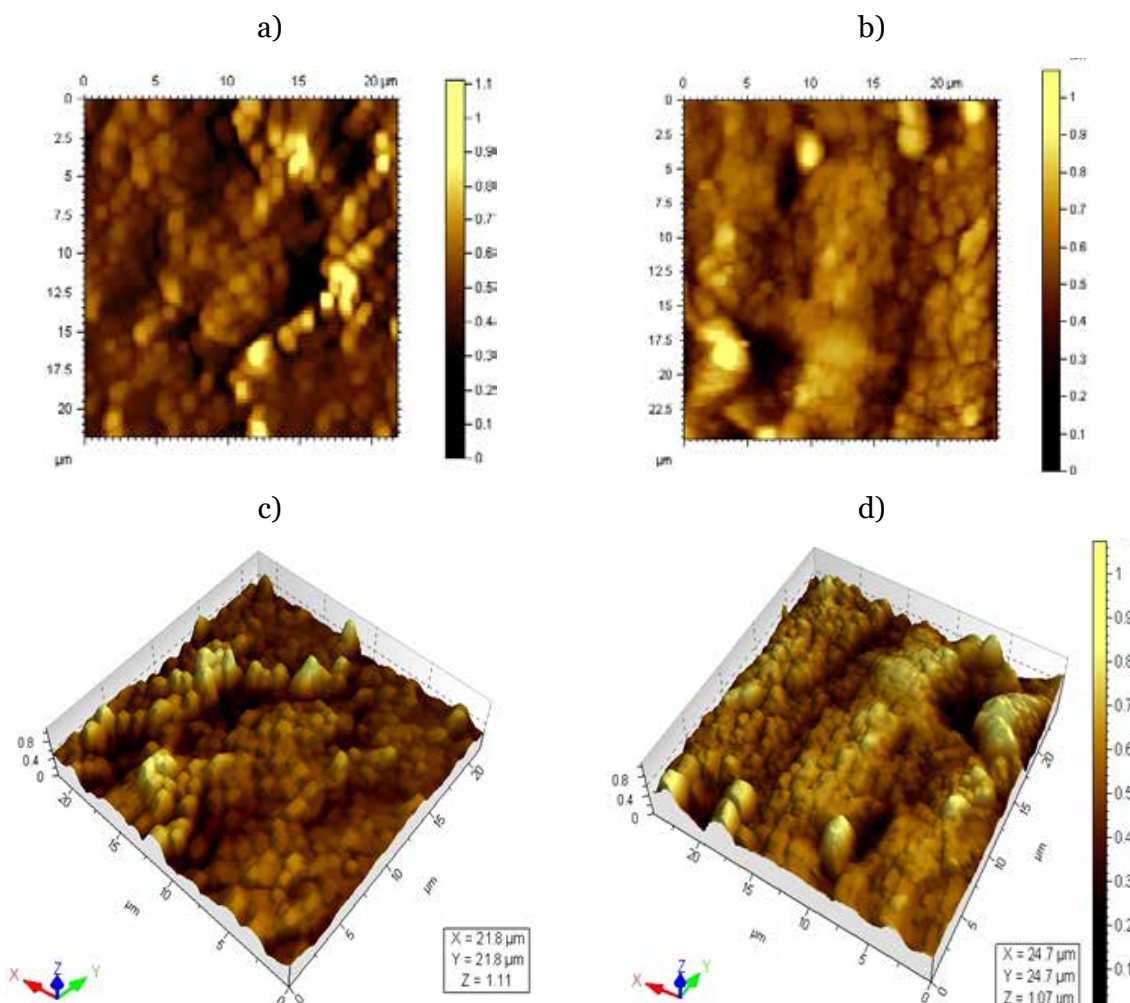
Effect of temperature

The effect of temperature was analyzed with various concentrations of the 1,2-diammoniumbenzene hydrophosphate inhibitor in 0.5 M HCl solution. The data observed in Table 1 show that the corrosion rate increases with rising temperature. This indicates that steel No. 20 has lower corrosion resistance at higher temperatures. The inhibitor efficiency decreases from 97.54% at 303 K to 83.06% at 333 K. This confirms that the 1,2-diammoniumbenzene hydrophosphate inhibitor excellently protects the surface of steel No. 20 from aggressive chloride ion solutions in the range of 303 to 333 K. It should be noted that corrosion protection is lower at high temperatures because the increase in kinetic energy causes the desorption of the inhibitor from the metal surface, which affects its inhibition performance. Furthermore, the electrostatic interaction between the inhibitor and the metal surface is lost at high temperatures. These factors also contribute to the decrease in inhibition indicators at elevated temperatures. The electrostatic interaction between the inhibitor and the metal surface is dependent on the temperature rise. The 1,2-diammoniumbenzene hydrophosphate inhibitor adsorbs onto the metal surface via chemical and physical adsorption mechanisms. Physical adsorption occurs through electrostatic interaction between the inhibitor and the metal surface. The positively charged nitrogen atoms of the corrosion inhibitor electrostatically interact with the negatively charged metal surface. Chloride and hydroxyl ions are adsorbed onto the metal surface, resulting in a negatively charged metal surface (El aoufir Y., Zehra S., Lgaz H., Chaouiki A., Serrar H., Kaya S., Salghi R., AbdelRaheem S. K., Boukhris S., Guenbour A., Chung I.-M., 2020). The corrosion inhibitor neutralizes the metal surface through electrostatic interaction. When the temperature rises,

chloride and hydroxyl ions become more active, causing the electrostatic interaction force to gradually decrease. AFM (Atomic Force Microscopy). The surface morphology of the plate-shaped steel No. 20 samples before and after inhibition was studied using atomic force microscopy. The surface of the clean steel No.

20 sample is almost smooth, with no signs of corrosive attack. The surface structure of steel No. 20 inhibited with the 1,2-diammoniumbenzene hydrophosphate inhibitor in Background-1 solution was studied using atomic force microscopy (Figure 2).

Figure 2. Microphotography of the surface of steel No. 20 uninhibited and inhibited with 1,2-diammoniumbenzene hydrophosphate using atomic force microscopy



Using atomic force microscopy, the state of the crystalline protective layer formed as a result of the inhibitor covering the steel surface and natural pits was studied and analyzed. The surface of the clean steel No. 20 sample is almost smooth, with no signs of corrosive attack. The 3D AFM images show the corresponding height parameters for the corroded and inhibited steel No. 20 surfaces. The obtained results revealed that numerous corroded areas were identified. In contrast, when the steel surface was treated with the selected corrosion inhibitor, 1,2-diammoniumbenzene

hydrophosphate, the metal surface became smoother, indicating a significant improvement in the surface condition. The improvement in surface morphology can be attributed to the protective layer formed by 1,2-diammoniumbenzene hydrophosphate, which effectively shields the metal surface from the acidic medium and reduces surface porosity.

Conclusion:

The dependence of the synthesis of 1,2-diammoniumbenzene hydrophosphate ionic liquid on temperature and time was

studied. It was observed that 50 °C, 5 hours, and 600 rpm are favorable conditions and ensure stability. The 1,2-diammonium-benzene hydrophosphate ionic liquid was synthesized via a reaction involving ortho-phenylenediamine and phosphoric acid. The

gravimetric analysis and AFM (atomic force microscopy) results for the 1,2-diammonium-benzene hydrophosphate inhibitor were obtained. The obtained 1,2-diammonium-benzene hydrophosphate ionic liquid is stable, inexpensive, and readily soluble in water.

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