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SYNTHESIS OF AN INHIBITOR AND ANALYSIS OF ITS PROPERTIES FOR MITIGATING ACID CORROSION IN THE OIL AND GAS INDUSTRY

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Abstract

This article presents the synthesis of a cost-effective, efficient, and environmentally friendly corrosion inhibitor, along with an analysis of its inhibitive properties. The inhibitor was synthesized using linear alkylbenzene sulfonic acid (LABSA), urea, and phosphoric acid. The resulting compound was tested for its ability to protect against acid-induced corrosion, particularly in hydrochloric acid (HCl) solutions, relevant to the oil and gas industry. The molecular weight of the synthesized substance was determined using the cryoscopic method, while its inhibitory performance was evaluated through gravimetric analysis. The inhibitor demonstrated high corrosion protection efficiency, and its synthesis from locally available raw materials makes it economically advantageous.

Keywords: *corrosion, inhibitor, LABSA (dodecylbenzenesulfonic acid), urea, phosphoric acid, weight loss, acidic medium*

Introduction

Corrosion is the degradation of metals resulting from chemical or electrochemical interactions with the surrounding environment, leading to significant economic losses in industrial settings. Equipment, pipelines, storage tanks, and heat exchangers operating in acidic environments are particularly susceptible to corrosion. Therefore, specific agents known as inhibitors are used to prevent or slow down this destructive process (El-Etre, A. Y., 2006; Ebenso, E. E., 2003).

In recent years, considerable attention has been given to the development of environmentally safe and economically efficient corrosion inhibitors. Traditional inhibitors often contain heavy metals, chlorinated organic compounds, and other toxic substances, which restrict their practical applications. This has created a pressing need for the development of biodegradable, inexpensive, and water-soluble inhibitors based on novel components (Quraishi, M.A., et al., 2011; Popova, A., 2007).

The literature reports various substances such as amines, phosphonates, benzotriazole derivatives, imidazoles, and surfactants as corrosion inhibitors. For instance, El-Etre (2006) reported high efficiency of urea and thiourea mixtures in hydrochloric acid media.

Additionally, the passivating properties of phosphoric acids function by forming a protective film on the metal surface. LABSA, containing sulfonic groups, adsorbs onto metal surfaces and contributes to surface coverage and protection (Niyozov, E., Razzakov, K., Nazarov, S., Olimov, B., & Gafurova, G., 2024; Kurbanov F. P., Axmedov V. N., Olimov B. B., Urinov X. X., Gafurova G. A., 2025).

However, there are no reports in the literature on the synthesis of a corrosion inhibitor composed of LABSA, urea, and phosphoric acid. Therefore, this study aims to develop a novel, cost-effective, and efficient inhibitor, investigate its synthesis process, and evaluate its anti-corrosive properties.

Materials and methods

In this study, to synthesize a novel corrosion inhibitor, the following raw materials were selected: LABSA (97%), urea (99%), phosphoric acid (85%), and distilled water.

Synthesis Procedure:

The synthesis of the compound was carried out in four main stages:

Stage 1: A total of 400 g of LABSA was placed into a 1000 mL three-necked round-bottom flask, followed by the addition of 100 mL of distilled water. The mixture was stirred using a magnetic stirrer for 10–15 minutes until a completely homogeneous solution was obtained. During this stage, the sulfonic acid group of LABSA becomes ionized, forming an active anion in the solution.

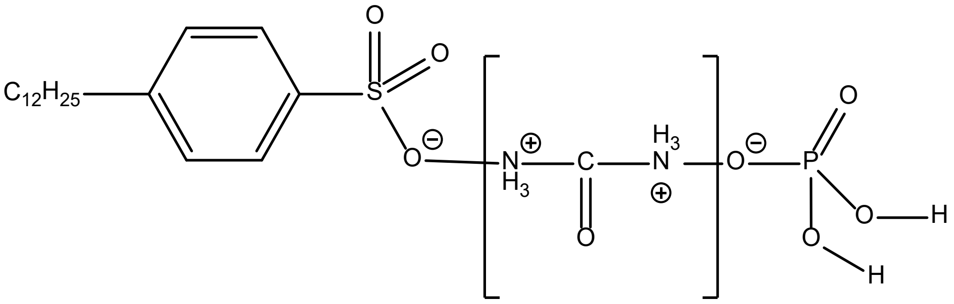
Stage 2: 200 g of urea was added gradually to the solution in 20 g portions. After each addition, the mixture was stirred for 5 minutes. The temperature was maintained at 30 °C using a water bath. Urea forms stable complexes with LABSA molecules through its ionized ammonium group in solution.

Stage 3: 150 g of 85% phosphoric acid was added dropwise to the solution using a pipette or burette. During this stage, the pH of the mixture dropped from 6 to approximately 2.5. Phosphoric acid reacts with urea to form several ionic complexes. Additionally, the phosphate groups act as active centers capable of further interaction with metal surfaces. The resulting mixture was kept at room temperature (20–25 °C) in a sealed container for 2 hours. During this process, ionic bonds are stabilized and macromolecular complexes are formed.

Stage 4: The final mixture was filtered through a 0.45-micron filter. The resulting viscous liquid acts as a surfactant. The substance was stored in an airtight container, protected from light, at 4–8 °C. Based on the isolated product's mass, the reaction yield was calculated to be 92%. The final product is a brown, homogeneous liquid with a characteristic odor and is completely soluble in water. It exhibits high surface activity and polyelectrolyte behavior, enabling efficient adsorption onto metal surfaces.

This step-by-step reaction process yields a multifunctional corrosion inhibitor. The synthesis can be carried out under simple laboratory conditions without the need for complex equipment, making it suitable for potential industrial application.

he structural formula of the synthesized compound is as follows:



Due to the complex ionic structure of the synthesized compound, it was named according to IUPAC nomenclature as: bis(ammonium)urea dodecylbenzenesulfonate dihydrogen phosphate salt (BDDP)

Determination of the Molecular Weight of the Synthesized Compound by the Cryoscopic Method

The determination of the molecular weight of the synthesized compound by the cryoscopic method is based on the change in the freezing point of a pure solvent upon the addition of the compound. In this method, the freezing point of the pure solvent is first measured. In the present study, distilled water was used as the solvent, with a normal freezing point $T_{\text{pure sol.}}=0$ °C and a cryoscopic constant $K_{\text{water}}=1.816$

The measurement of the freezing point is carried out using a calorimeter, as shown in Figure 1. For the determination, 2–3 g of the test compound (accurately weighed to 0.0001 g) is placed into a long test tube (2). The solvent (water) is added in an amount 20–25 times greater than the mass of the test compound. A thermometer (4) and stirrer (3) are then inserted into the tube, which is sealed with a stopper. Care is taken to ensure that the thermometer does not touch the walls of the container and that the bulb of the thermometer is fully immersed in the solution.

Next, the test tube is placed into an ice bath (1). The solution is stirred continuously using the stirrer (3) to ensure uniform cooling. Once crystals begin to form, stirring is stopped. The temperature is recorded at regular intervals (every 8–10 seconds). A correction must be applied to the observed freezing point to account for the exposed mercury column of the thermometer. This correction is calculated using the following equation:

$$\Delta t = k \cdot h \cdot (t_0 - t_1)$$

where:

k – is the volumetric expansion coefficient of mercury in the thermometer tube, taken as 0.000159 °C per 10 °C,

h – is the length of the exposed mercury column, measured in thermometer scale divisions,

t_0 – is the observed freezing point,

t_1 – is the average temperature of the exposed mercury column, determined using an auxiliary thermometer attached to the main thermometer with a rubber band and positioned at the center of the mercury column.

The molecular weight is calculated using the following formula:

$$M = \frac{1{,}816 \cdot 1000 \cdot m}{m_s \cdot \left(t_s - \left(t_m + \Delta t\right)\right)}$$

where:

m – mass of the test sample (in grams);
m_s – mass of the water (solvent);
t_s – freezing point of pure water;
t_m – freezing point of the test solution;
Δt – thermometer correction factor.

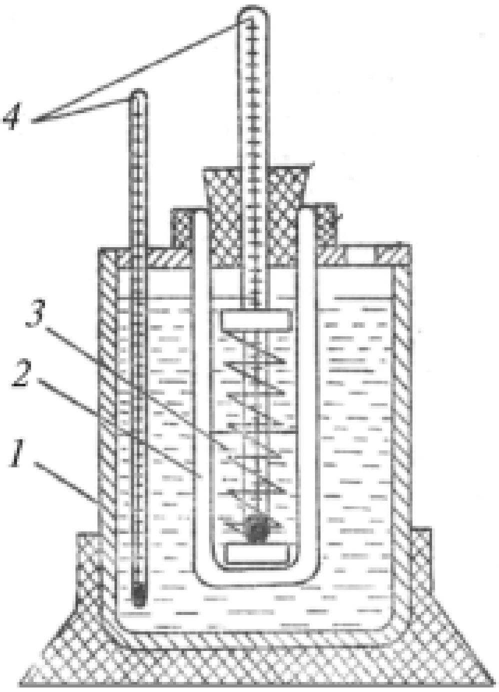


Figure 1. *Apparatus for Molecular Weight Determination by the Cryoscopic Method*

1. *Dewar vessel mounted on a stand*
2. *Thick-walled test tube (150 mL volume)*
3. *Magnetic stirrer*
4. *Thermometers*

To evaluate the inhibitory properties of the synthesized compound, the gravimetric analysis method was employed. In this method, steel samples (coupons) were exposed to various concentrations and ratios of the studied inhibitor in different environments. The corrosion rate and protection efficiency were determined within a specific temperature range.

The samples were immersed for 300 hours in acidic media, both with and without the inhibitor. After exposure, the samples were removed, cleaned, and weighed, and their final masses were compared with the initial masses. Based on these data, the corrosion rate (K) and inhibition efficiency (Z) were calculated using the standard gravimetric formulas:

$$K = \frac{(m_1 - m_2)\cdot 1000}{S\cdot \tau_1}$$

$$Z = 100\% - K$$

where:

m_1 – initial mass of the metal coupon (g);

m_2 – final mass of the metal coupon after the experiment (g);

S – surface area of the metal coupon (m^2);

τ_1 – duration of the experiment (days).

Results and discussion

In the cryoscopic analysis, 12 g of the synthesized compound was dissolved in 100 g of water. At the end of the process, crystal formation and freezing were observed at –0.446 °C. Based on the obtained data and the cryoscopic formula, the molecular weight of the compound was calculated to be 488.1 g/mol. This result is very close to the theoretical molecular weight of 484 g/mol.

The percentage of experimental error was calculated as follows:

$$\chi = \frac{(488.1 - 484)\cdot 100\%}{484} = 0.85\%$$

Thus, the measurement was conducted with an accuracy of:

$$100\% - 0.85\% = 99.15\%$$

This high level of accuracy indicates strong agreement between the theoretical and experimental results.

The corrosion inhibition performance of the synthesized compound in 15% HCl solution at concentrations ranging from 100 mg/L to 300 mg/L is presented in

Table 1. *Dependence of the corrosion inhibition efficiency of the synthesized inhibitor on its concentration*

Blank	S, 10^{-4} m^2	τ, hour	mass m_0, g	mass m, g	Δ m	Concentration of inhibitor, mg/l	Rate of corrosion	Z, %	γ
1	21	300	23,42	22,595	0,825	–	1,31	–	–
2	21	300	23,58	23,383	0,197	100	0,313	76,1	4,18
3	21	300	23,56	23,4	0,160	150	0,255	80,5	5,14
4	21	300	23,61	23,512	0,098	200	0,156	88,1	8,39
5	21	300	23,45	23,365	0,085	250	0,135	89,7	9,7
6	21	300	23,57	23,514	0,056	300	0,085	93,2	14,72

According to the results of the gravimetric tests, the optimum corrosion inhibition efficiency was achieved at a concentration of 300 mg/L. At this concentration, the inhibitor exhibited a protection efficiency of 93.2%, confirming its high inhibitory performance.

From an economic perspective, the cost of raw materials for producing 1 kg of the final product is approximately 11,000 Uzbek so‘m, which is 2–3 times cheaper than global market prices. In addition, local production and environmental safety make this inhibitor highly competitive. Compared to the studies of El-Etre (2006), Ebenso (2003), Quraishi (2011), and others, the efficiency of this inhibitor is comparable or even superior, while also being environmentally friendly and industrially viable.

Conclusion

Within the scope of this study, the efficiency of a newly synthesized urea-based corrosion inhibitor was thoroughly investigated using various methods. The experimental molecular weight of the compound was determined to be 488.1 g/mol, which is very close to the theoretical value of 484 g/mol. Calculations showed a relative error of χ=0.85% indicating that the experiment was conducted with an accuracy of 99.15%. This small discrepancy confirms the strong agreement between theoretical and experimental values.

Corrosion tests using the gravimetric method were conducted in 15% HCl solution with inhibitor concentrations ranging from 100 to 300 mg/L. According to the data, the highest inhibition was observed at 300 mg/L, with a maximum protection efficiency of 93.2%. This result demonstrates that the synthesized compound possesses high efficiency and can effectively protect mild steel in aggressive acidic environments.

In summary, the results of this research indicate that urea-derived corrosion inhibitors combine high efficiency, low cost, environmental safety, and industrial applicability. Therefore, such compounds can be considered promising corrosion protection agents. Future studies will focus on developing even more effective modified derivatives and conducting extended trials under industrial conditions.

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