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DEVELOPMENT OF TECHNOLOGY AND INVESTIGATION OF THE PROPERTIES OF CORROSION INHIBITORS BASED ON GOSSYPOL RESIN

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Abstract

The study examines the condensation process of gossypol resin with urea within a temperature range of 50–70 °C. By adding the distillation residue of monoethanolamine to the resulting condensation product in a 1:1 ratio, a composition containing azomethine groups and π-bonds was prepared. These components facilitate adsorption on the metal surface and form stable complexes with various salts. The resulting product was used as a corrosion inhibitor, the protective efficiency of which meets the requirements for hydrogen sulfide and acidic environments. A single-stage technology for the production of the corrosion inhibitor has been proposed.

Keywords**:** *inhibitor, corrosion, mineral salts, gossypol resin, urea, protective effect, hydrogen sulfide environment*

Introduction

In nature, there are so-called abnormal phenomena – erosion, corrosion, mineral salt deposits, etc. – which cause enormous damage to the national economy. Mineral salts, carbonates, calcium, iron and barium sulphates, etc., dissolved in water, form a hard-to-dissolve scale when heated, which leads to a decrease in thermal conductivity and overconsumption of energy resources, requiring additional costs associated with the prevention of salt deposits.

Currently, well-deserved attention is being paid to the introduction of inhibitors to protect equipment from corrosion and salt deposits. The use of such inhibitors can increase the service life of expensive equipment several times over. At the same time, year after year, it has been proven that the most effective inhibitors for neutral and slightly alkaline environments are nitrogen-, sulphur- and oxygen-containing organic compounds, as well as metal-complex organophosphorus compounds – zinc organophosphonic acids, which effectively prevent corrosion and mineral salt deposits on the surface of equipment.

There are many publications devoted to the catalytic condensation of aliphatic aldehydes with ammonia in the vapour phase (Kwong, 2007), which leads to the formation of pyridines.

The production of substituted pyridines by thermal cyclocondensation of aldehydes with ammonia is called the Bayer-Chichibabin reaction (Zaitseva, 2001) and is important in the synthesis of various alkylpyridines with predetermined structures, which can be effective corrosion inhibitors (Yu, 2005). However, due to the low selectivity of the product, this method of obtaining alkylpyridines has not found practical application.

The condensation of aromatic aldehydes with aromatic amines leads to the formation of Schiff bases, which was studied in the work of (Letunov and Kulakova., 2012) using the example of the reaction of benzylideneaniline with antipyrine.

$$Ar-CHO + H_2N-Ar' \longrightarrow Ar-CH=N-Ar' + H_2O$$

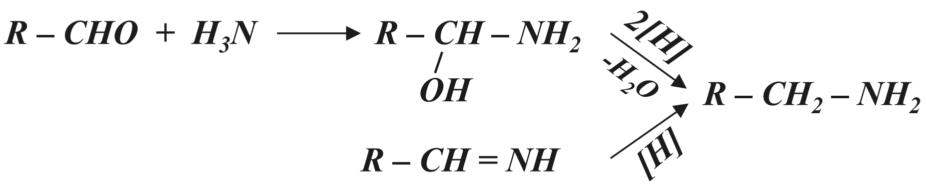
Schiff bases, obtained by the interaction of various aliphatic and aromatic aldehydes and amines, are significantly more active and inhibit metal corrosion than the starting substances. When a mixture of amines and aldehydes is used as corrosion inhibitors, Schiff bases are formed on the surface of catalytically active transition metals, which explains the synergism noted above (Rahimkulov et al., 2005).

The production of primary, secondary and tertiary amines by reduction-alkylation of carbonyl compounds is of great importance.

The reduction-alkylation reaction of ammonia consists in the addition of ammonia to a carbonyl compound and the subsequent reduction of either the addition product itself or the dehydration product of the latter.

Materials and methods

In the case of catalytic reduction, the reaction is carried out in an alcoholic solution. The reaction proceeds according to the following scheme:



The primary amine formed in the presence of aldehyde is also capable of reacting similarly to ammonia: the resulting addition product, Schiff base R – CH = N – CH2 – R, is converted into a secondary amine upon reduction. Similarly, a primary amine can react with an imine to form an addition product, the reduction of which also leads to the formation of a secondary amine:

$$R-CH=NH + R-CH_2-NH_2 \rightleftarrows R-CH(NH_2)-NH-CH_2-R$$

$$R-CH(NH_2)-NH-CH_2-R \xrightarrow{2[H]} R-CH-NH-CH_2-R + NH_3$$

In turn, secondary amines can react with either aldehydes or imines; the resulting products are reduced to tertiary amines:

$$(R-CH_2)_2NH + R-CHO \rightleftarrows (R-CH_2)_2N-CH(NH_2)-R$$

$$(R-CH_2)_2N-CH(NH_2)-R \xrightarrow{2[H]} (R-CH_2)_3N + H_2O$$

The reductive amination reaction of carbonyl compounds is a key reaction for obtaining amines of various structures, but the main disadvantage of this method is the formation of a mixture of products of complex composition and low yield of target products.

Among the products of the processing of aldehydes, amines and amides for the production of corrosion and scale inhibitors, the following are used:

1) imidazolines obtained as a result of chemical reactions in a vacuum reactor (the ratio of imidazoline to amide parts is 30:70);

2) amines of various structures;

3) fatty acids with 17 to 25 carbon atoms (at C < 17, foaming and emulsification may occur; at C > 25, the inhibitor will be insoluble in both water and hydrocarbons);

4) pyridines and their derivatives;

5) various oxygen-containing compounds;

6) compounds with double and triple bonds in the molecule;

7) Schiff bases;

8) additives that reduce surface tension at the water-liquid hydrocarbon interface in order to facilitate the transition of the inhibitor into water.

Diethylenetriamine is used as an amine in the production of imidazoline. If diethylenetriamine contains a large radical – R – in its composition, the inhibitor will lose its homogeneity during storage and form a precipitate in storage containers, clog injection devices, and have low thermal stability.

The three-stage reaction for obtaining imidazoline takes place in reactors under vacuum:

The reaction for obtaining imidazoline never proceeds to completion, resulting in the formation of a mixture of amide (stage 2) and imide (stage 3) in a specific ratio. At the optimum ratio (e.g., imide: amide approximately 30:70), a stable substance with high protective properties is obtained.

The aim of this work is to develop a technology for the production of mineral salt and corrosion inhibitors based on the condensation product of gossypol resin with urea, with the addition of a distillation residue of monoethanolamine.

Experimental part

Place 50 g of gossypol resin in a round-bottom flask, heat to a temperature of 50 °C and add 15 dm^3 of a 40% aqueous solution of carbamide. The mixture is heated while stirring until a homogeneous mass is formed. Then, while continuing to stir intensively, 45 g of vacuum distillation residue of monoethanolamine is added. The resulting product was tentatively named 'SUMONO-extra-M' and is characterised by the following indicators:

The SUMONO-Extra-M corrosion inhibitor is a product of the condensation of gossypol (Kobilov et al.,, 2012) with urea, a mixture of fatty acids and the vacuum distillation residue of monoethanolamine.

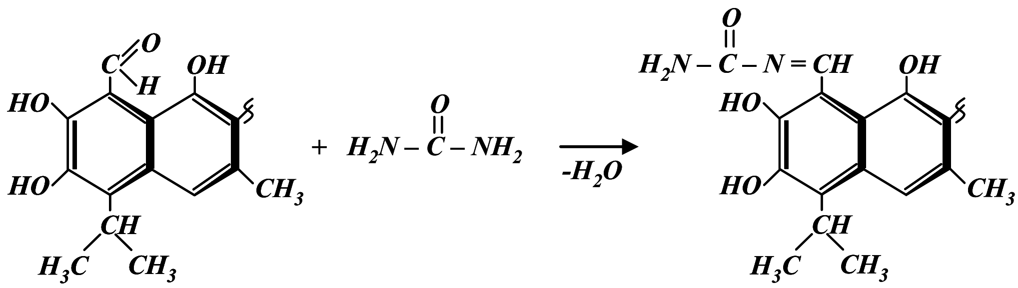
In terms of its physical, chemical and operational characteristics, the SUMONO-Extra-M corrosion inhibitor must meet the requirements and standards.

Name of indicators	Standard
Appearance	Homogeneous liquid ranging from light
Density at 20 °C, g/cm^3	1.0±0.05
Amine number, mg HCl per 1 g of inhibitor, not less than	45
Protective effect in hydrogen sulphide environment, % not less than	90.0
In hydrochloric acid with a mass fraction of... 15%, not less than	96.0
In oil, % not less than	85.0
Solidification temperature, °C, not higher than	–18
Dry residue, % not less than	67
Solubility in gas condensate in water	soluble Dispersible
Kinematic viscosity at 20 °C mm^2/sec	25

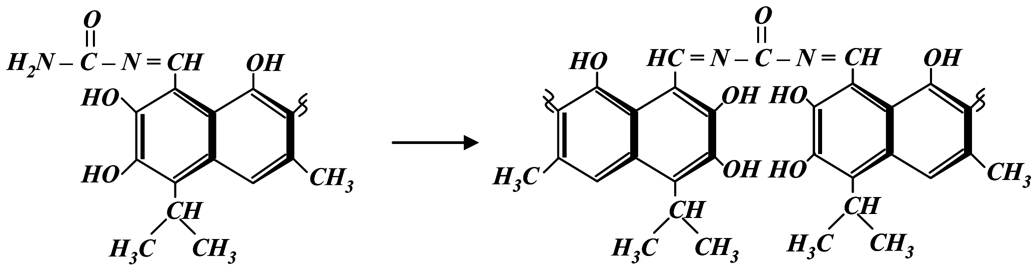
Results and discussion

The corrosion inhibitor SUMONO-Extra-M is obtained by condensing gossypol resin with a urea solution at temperatures of 40–70 °C and shifting the resulting product with the distillation residue of vacuum distillation of monoethanolamine in a ratio of 0.5:0.03:0.47.

When gossypol resin interacts with urea, the aldehyde group of gossypol forms Schiff bases according to the following scheme:



The second NH_2 group of urea can react with a new gossypol molecule and form compounds with the following structure:



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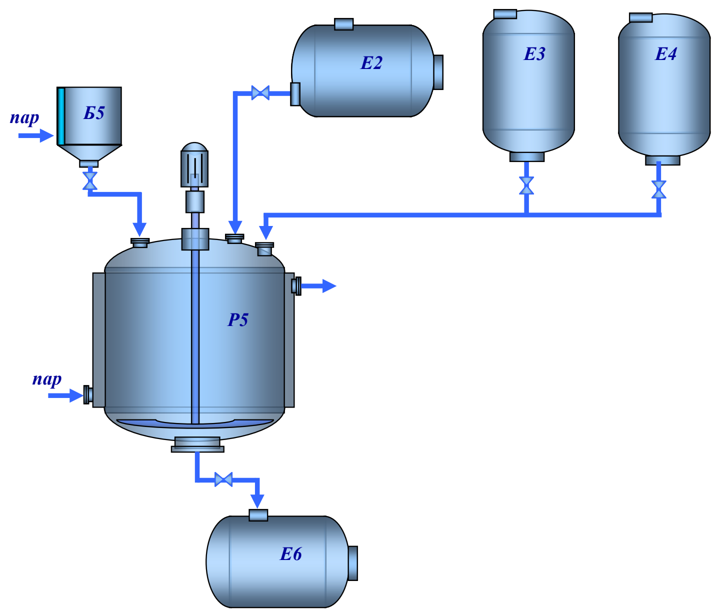
The vacuum distillation residue of monoethanolamine contains a mixture of amines and oligomers. The presence of OH groups, azomethine groups and π bonds in gossypol resin promotes the adsorption of the latter on the metal surface, forming a strong film. In this regard, the mixture of gossypol carbamide resin and the vacuum distillation residue of monoethanolamine has high protective properties.

The technological scheme for the production of the SUMONO-Extra-M corrosion inhibitor consists of a single technological line (Fig. 1):

Figure 1. *Schematic diagram of the production of the SUMONO-Extra-M inhibitor:*



B1 –hopper for gossypol resin;
E2, E3, E4 – containers for the bottom residue of vacuum distillation of monoethanolamine; 40% solution of urea and solvent (petroleum products such as petrol, kerosene, diesel fuel, etc.);
P5 – reactor;
E6 – container for finished products

Gossypol resin is fed from bunker B1 into reactor P5, which is equipped with a mechanical stirrer and a heating jacket. The gossypol resin in bunker B1 is heated to a temperature of 50–60 °C. A 40% aqueous solution of urea is fed into the reactor from the container pos. E2. The mixture is heated while stirring at a temperature of 50 °C until a homogeneous stable mass is formed. Next, an operational amount of the vacuum distillation residue of monoethanolamine is added to the reactor from container E3 and stirred for 1 hour until a homogeneous mass is formed. If phase separation is observed when the sample taken from the reactor is left to stand, stirring is continued. If the mixture is difficult to flow, a solvent (naphtha, petrol, gas condensate, kerosene, diesel fuel, etc.) is added to the mixture. The finished product is stored in container E6.

Corrosion tests were carried out on samples of pipe steel of strength category D. The test medium was a mixture of gas condensate and water in a ratio of 1:2 by volume, saturated with hydrogen sulphide to 2.5–3.0 g/l and carbon dioxide (CO2 pressure up to 1.0 MPa). The inhibitor concentration was 0.1–0.5 g/l, and the medium was stirred (Tables 2 and 3).

Table 2. *Effect of the SUMONO-Extra-M inhibitor on steel corrosion in a hydrogen sulphide environment. Room temperature*

Inhibitor concentration, g/l	Test duration, hours	Corrosion rate, g/m²·hour	Degree of protection, %
Without inhibitor	12	1.15	–
0,1		0.051	95.56
0,2		0.036	96. 86
0,3		0.014	98.86
0,5		0.013	98.9
Without inhibitor	24	0.93	–
0,5		0.018	98.04
Without inhibitor	48	0.28	–
0,5		0.09	96.78

As can be seen from the data obtained, SUMONO-Extra-M demonstrates high protection in all cases.

Table 3. *Effect of the SUMONO-Extra-M inhibitor on steel corrosion in a carbon dioxide environment*

Inhibitor concentration, g/l	Test duration, hours	Corrosion rate, g/m²·hour	Degree of protection, %
Without inhibitor	20	3.7	–
0,2	20	0.141	96.2
Without inhibitor	40	3.8	–
0,2	40	0.170	95.5
Without inhibitor	60	3.5	–
0,2	60	0.138	96.0
Without inhibitor	80	3.4	–
0,2	80	0.138	95.94

As can be seen from the data in the table, SUMONO-Extra-M effectively protects steel from corrosion in hydrochloric acid, with a protective effect of up to 95.94%.

Conclusion

Thus, by condensing gossypol resin with carbamide at a temperature range of 50–70 °C and mixing it with the distillation residue of vacuum distillation of monoethanolamine in a ratio of 0.5:0.03:0.47, the corrosion inhibitor SUMONO-Extra-M was obtained. A single-stage technology for the production of the SUMONO-Extra-M corrosion inhibitor has been proposed. It has also been established that azomethine groups and π-bonds promote the adsorption of molecules on the metal surface, providing high protective efficiency (up to 96%) in hydrogen sulphide and acidic environments.

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