ТЕХНИЧЕСКИЙ СЕРВИС В АПК

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Technology for producing anticorrosive materials from fat-containing waste

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Abstract. The processing of fat-containing waste from meat-packing enterprises that cannot be landfilled is an urgent problem. The authors developed the technology of processing fat-containing waste in a chemical reactor in one step, without dividing the technology into two steps: triglyceride hydrolysis and fatty acid amide production. The technology for processing fat-containing waste was tested in a laboratory plant. The target product obtained was analyzed using IR spectroscopy. As a result, the structural formula was clarified, functional groups were determined, and chemical intermediates in the synthesis process were identified. The proportions of the reagents were determined: technical fat in the amount of 65.3-72.4 wt%, monoethanolamine – 14.5-17.0 wt% and boric acid – up to 100 wt% of the mixture. The reaction time was 1.5 h. The protective effect was selected as a quality control indicator of the surfactant obtained. Solvent selection was carried out for producing anticorrosive materials. I-20 industrial oil, diesel fuel and SN-150 oil were selected as solvents. Electrochemical studies carried out on the AVTOLAB PGSTAT302N potentiostat-galvanostat made it possible to determine the mechanism of action of the surfactant as a corrosion inhibitor. Its optimum concentration was found to be 15%, with a protective efficiency of 99.33%. To increase the protective anticorrosive efficiency of the surfactant, the tests were carried out in a humidity chamber for 60 days on St3 steel plates. The test results showed high efficiency of the surfactant used as a corrosion inhibitor, with the Z value of more than 90%.

Keywords: technology for processing fat-containing waste, organic synthesis, hydrolysis, surfactants, environmental safety, fatty acids, triglycerides, corrosion inhibitors, protective efficiency

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ОРИГИНАЛЬНАЯ СТАТЬЯ

Технология производства защитных антикоррозионных материалов из жиросодержащих отходов

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Аннотация. Переработка жировых отходов мясоперерабатывающих предприятий, не подлежащих захоронению, является актуальной проблемой. В работе предложена технология переработки жировых отходов, позволяющая в химическом реакторе осуществить синтез продукта в одну стадию, без разделения технологии на два этапа: гидролиза триглицеридов и получения амидов жирных кислот. Технология переработки жирсодержащих отходов была реализована на лабораторной установке. Полученный целевой продукт был проанализирован с использованием ИК-спектроскопии. В результате была уточнена

структурная формула, определены функциональные группы, а также установлены промежуточные химические соединения в процессе синтеза. Установлены соотношения реагентов: технический жир в количестве 65,3…72,4 мас.%, моноэтаноламин – 14,5…17,0 масс.% и борная кислота – до 100 масс.% смеси. Время протекания реакции 1,5 ч. В качестве показателя контроля качества, полученного ПАВ, выбран защитный эффект. Для изготовления защитных материалов от коррозии проведен подбор растворителя. В качестве растворителей выбраны индустриальное масло И-20, дизельное топливо и масло SN-150. Электрохимические исследования, проведенные на потенциостате-гальваностате AVTOLABPGSTAT302N, позволили определить механизм действия ПАВ в качестве ингибитора коррозии. Установлена его оптимальная концентрация в растворе 15% и защитный эффект (защитная эффективность) – 99,33%. Для повышения достоверности результатов защитной антикоррозионной эффективности ПАВ, были проведены испытания в камере влажности в течении 60 суток на металлических пластинах из Ст3. Результаты испытаний показали высокую эффективность ПАВ, используемого в качестве ингибитора коррозии (Z более 90%).

Ключевые слова: технология переработки жиросодержащих отходов, органический синтез, гидролиз, поверхностно-активные вещества, экологическая безопасность, жирные кислоты, триглицериды, ингибиторы коррозии, защитная эффективность

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Introduction

The efficient processing and utilization of fat- containing waste is an unsolved problem. A significant proportion of the secondary resources generated by the industrial processing of agricultural raw materials are used inefficiently and often end up in landfills or waterways, causing significant environmental damage. Waste water from many meat processing enterprises contains fat emulsions, which are class 4 waste. Flotation treatment of this waste water results in the formation of by-products (fatty foam masses). The decontamination and utilization of this waste is a pressing issue[1,2,3]. Organic fat-containing waste cannot be landfilled and its incineration is problematic[4] [1].

From an environmental and economic point of view, it is preferable to use this waste as raw materials for manufacturing secondary products. Thus, the annual processing of 110-115 million tons of agricultural raw materials generates more than 50 million tons of by-products, which represent a significant reserve for obtaining various types of products[4] [2, 3].

At present, only large companies collect and process by-products. According to the standards of E.I. Sizenko[3] about 1888 thousand tons of by-products can be obtained, but only about 30% of this volume is actually collected. The profitability of industrial enterprises does not exceed 3-4%. With full collection and utilization of by-products the profitability can be tripled.

The transition to low- and zero-waste production cycles will help solve the problems of rational use of natural resources and environmental protection. Scientific developments in the processing of fat-containing waste into biogas, liquid and solid biofuels will solve the problem of environmental safety for companies in the industry[5].

For example, waste from the meat industry is an inexpensive renewable raw material for the production of surfactants. Surfactants are mainly synthesized from fatty acids derived from vegetable and animal fats (triglycerides)[6]. Surfactants are used to produce detergents, corrosion inhibitors, emulsifiers, mineral dispersants, lubricating oil additives[7], etc.

The aim of the research is to develop a technology for the synthesis of surfactants from fat-containing waste from meat processing enterprises, to carry out

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physicochemical analysis of the surfactant obtained and to evaluate its potential use as a corrosion inhibitor.

Materials and methods

Animal fats are esters of the triatomic alcohol glycerol and of higher fatty acids:

$$\begin{array}{l} CH_2-OCOR' \\ | \\ CH-OCOR'' \\ | \\ CH_2-OCOR''' \end{array}$$

Chicken fat obtained from non-food raw materials with quality indicators that are in compliance with GOST 17483-73 is used for producing surfactants.

Triglycerides of animal fats contain saturated and unsaturated fatty acids.

Fatty acids with an even number of carbon atoms, as well as palmitic (C_{16}), stearic and oleic (C_{18}) acids predominate in animal fats[8].

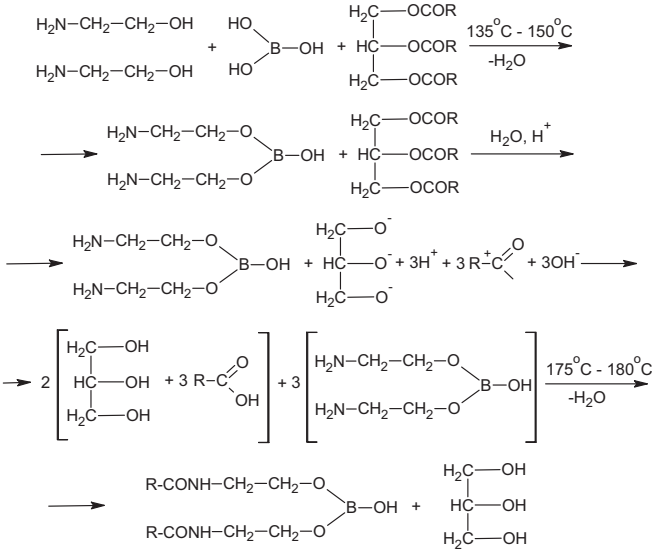
Ethanolamines (EAs) are products of petrochemical synthesis from ethylene oxide and ammonia and are widely used for producing surfactants[9]. Monoethanolamine (MEA) is used in organic synthesis.

Boric acid (H_3BO_3) was used for producing fatty acid amides[10]. The outer shell of boron has three electrons, so in trivalent compounds it does not have a full electron octet and has a great affinity for electron-donor reagents, forming molecular bonds with them, causing B to become tetracoordinated and adopt a tetrahedral configuration. It is known that borates of amino alcohols can act as inhibitors of steel corrosion[3]. The increase in the protective ability of mono-, di- and triethanolamine borates in comparison with aminoalcohol is apparently due to the formation of a thinner protective film on the metal surface, formed simultaneously by a donor-acceptor mechanism through the OH-group of the aminoalcohol with homosorption of borate ions.

The processing of fat-containing waste from the meat industry consists of two stages. In the first stage it is necessary to solve the problem of separation of fat from water (waste water from catering, meat and dairy industries contains a significant amount of fat components in the emulsified state. In the second stage after hydrolysis of fats (triglycerides), ethanolamides of fatty acids are obtained as a product of condensation of fatty acids with ethanolamine borate.

A process of the production of fatty acid amides includes a stage of heating to a temperature of 180ºC for 1.5 h of technical fat (TF), monoethanolamine (MEA) and boric acid (BA) at a ratio of technical fat, monoethanolamine and boric acid taken in the ratios: 65.3-72.4:14.5-170:100 wt% [4].

The reaction of hydrolytic cleavage of fat and obtaining of fatty acid amide is carried out according to the algorithm:



First, amino alcohol and boric acid are added to the reactor. The reaction mass is stirred and heated up to 110ºC until a homogeneous mixture is formed. Then at the temperature of 135 to 150°C the condensation reaction takes place with the formation of amino alcohol borate and release of water. When triglycerides are introduced into the reactor, steam and boric acid catalyst act simultaneously on the fat. Boric acid provides an active proton which promotes the attachment of water molecules in the process of triglyceride hydrolysis. This results in the hydrolytic cleavage of the triglyceride to form fatty acids and glycerol.

When triglycerides are hydrolyzed, it is the acyl-oxygen bonds that are broken, not the akyl-oxygen bonds. As a result, H^+ is bound to the alcoholic residue and OH^- to the acyl group [5].

When the temperature of the reaction mass is raised to a temperature of 180ºC, a condensation reaction takes place to form fatty acid amides. The final product is a mixture of fatty acid amides and glycerol.

A sample of surfactant was prepared in a laboratory plant. Chemical analysis of the surfactant obtained was carried out by IR spectroscopy[11] using a Spoctrun Two instrument (PerkinElmer, USA).

The amine number was determined according to the following procedure: 1.0 to 1.2 g of the surfactant

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obtained was poured into a 250 ml conical flask, then 35 ml of benzyl alcohol and 5 ml of isopropanol were added. The mixture was stirred until a clear homogeneous mixture was obtained. It was then titrated with a 0.1H solution of HCl in isopropanol to a pH value of 4.0.

The amine number (A, mg HCl/g) was calculated using the following formula:

$$A = 3.65 \cdot V / m,$$

where V is the volume of HCl solution used for the titration, ml; m is the mass of the surfactant, g.

To determine surfactant solubility in hydrocarbons, the aniline point was determined using the AST-MD611-2016 method. Surfactant solubility was determined as follows: from 5 to 25% of the surfactant was added to 80.0 to 85.0 grams of I-20A mineral oil, SN-150 oil or to diesel fuel (DT). The mixture was heated to 60-70° C with stirring for 30 min, then cooled to room temperature and kept at this temperature for 10 days. The stratification and/or precipitation of the mixture was observed.

The mechanism and efficiency of the anticorrosion protection were determined by electrochemical studies using the AVTOLAB PGSTAT302N potentiostat-galvanostat [6, 7].

The tests in the humidity chamber were carried out continuously for 60 days at a temperature of about 50°C and a relative humidity of 100%. At the same time, the specimens were periodically checked for the presence of corrosion centers on their surfaces.

The effectiveness of the obtained surfactant as a corrosion inhibitor was evaluated according to GOST 9.054-75 (method 1) [8, 9, 10].

Removal of corrosion products was carried out according to GOST 9.907-2007.

The rate of corrosion processes was determined by weighing the plates before and after the tests. The rate of corrosion processes was determined according to the formula:

$$K_c = \frac{\pm \Delta m}{S \cdot \tau},$$

where ± Δm is the change in the mass of the plates during the test, g; S is the square of the plate, m^2; τ is the test time, h.

The specimens were weighed after the tests before the removal of corrosion products $(+\Delta m)$, and after their removal $(-\Delta m)$.

The protective efficiency (Z) was determined according to the formula.

$$Z = \frac{(K_c - K_I)}{K_c} \cdot 100\%,$$

where K_c, K_I are corrosion rates of the control specimen and the specimen preserved with the composition.

Results and discussion

The results of IR spectroscopy of the surfactant are presented in Figure 1.

The analysis of the IR spectroscopy results (Fig. 1) shows that during the amidation process, the intensity of the absorption band at 1619 cm^{-1} is observed, which corresponds to the carbonyl group of tertiary amides in the spectrum of the reaction products.

Simultaneously, absorption intensities of ester groups (1739 and 1074 cm^{-1}) are observed in the spectrum, which can be explained by the parallel formation of amino

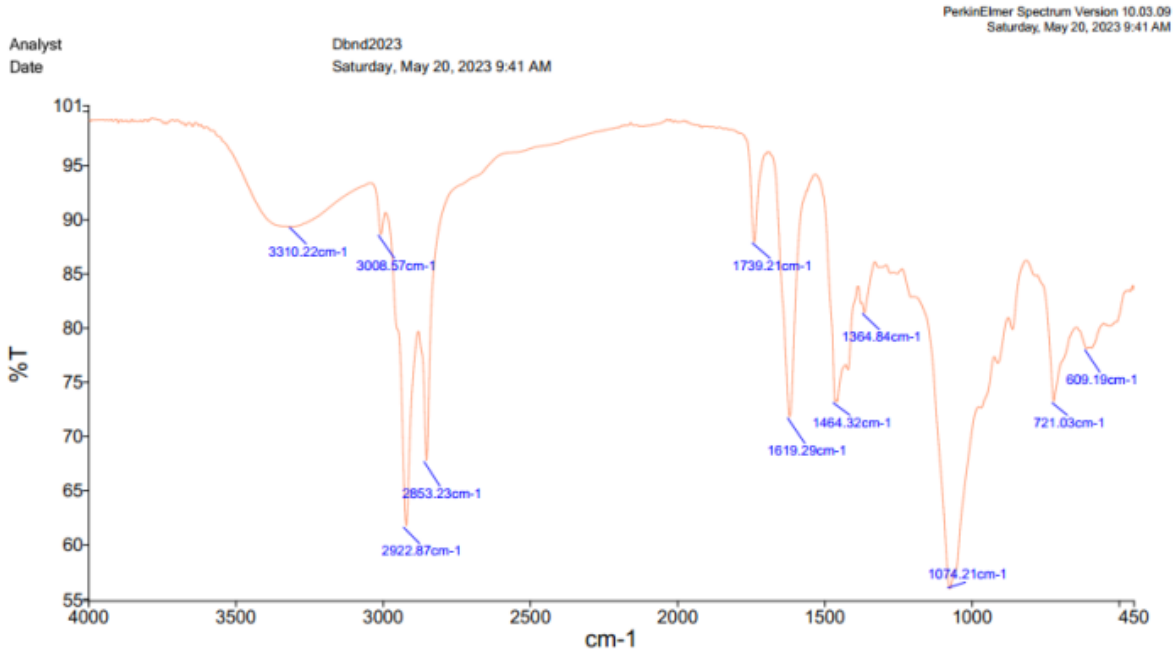


Fig. 1. IR spectroscopy of surfactants

Рис. 1. ИК-спектроскопия ПАВ

esters in equilibrium with amides. Increasing the reaction time at 180 to 200°C does not affect this equilibrium.

It is also noteworthy that the decrease in the absorption intensity of the hydroxyl group band (valence vibrations: -OH = 3274 cm^{-1}, C–OH = 1053 cm^{-1}) occurs practically up to 2.5 h of synthesis.

To determine surfactant solubility in hydrocarbons, the aniline point was determined (Table 1). One of the qualitative indicators of surfactants is the amine number. The results of the determined amine number and surfactant solubility are given in Table 2.

The analysis of Table 2 shows the following:

- increasing the ratio of boric acid to ethanolamine raises the amine number of the resulting surfactants;
- increasing the reaction time reduces the amine number of the surfactants obtained (when the reaction is carried out at 180 to 200ºC for one hour, the amine number decreases to 49.2 mg HCl/g).

The process of surfactant dissolution in hydrocarbons shows the following:

- the lower the aniline point of the solvent, the higher the solubility of the surfactant obtained. The solubility of the surfactant decreases in the order: DT>I-20A>SN-150;
- the longer the reaction time, the higher the solubility of the surfactants obtained;
- as the ratio of ethanolamine to triglyceride in the initial reaction mixture increases, the solubility of the obtained surfactants in the solvent decreases;
- increasing the ratio of boric acid to ethanolamine in the initial reaction mixture has no significant effect on the solubility of the obtained surfactants in the solvent.

It is found that the surfactant at a concentration of 5 to 25 wt% in I-20A mineral oil is an inhibitor of anodic action and ennobles the stationary potential (Fig. 2). The greatest protective effect is observed at the 15% surfactant content in mineral oil (Table 3).

The preservation oil containing surface-active molecules is easily permeable to electrolyte solutions, which enables polarization measurements on metals coated with them. The results are presented in Table 3 and Figure 2.

The effectiveness of the obtained surfactant as a corrosion inhibitor was evaluated in the humidity chamber. The preservation oil was prepared in the following ratio: surfactant – 15%, I-20A oil – 85%. St3 steel plates were used as specimens, three specimens for each test.

The first corrosion centers on the control specimens appeared after 12 hours of testing. The onset of pitting on the specimens preserved with the preservative oil (15% surfactant solution in I-20A oil) was observed after 816 hours (34 days).

After 60 days of testing, the control specimens had 100% corrosion products on the surfaces.

The test results are presented in Figure 3 and Table 4.

Removal of corrosion products was carried out according to GOST 9.907-2007 with a solution (1000 cm^3) consisting of 100 g of citric acid, 50 cm^3 of sulfuric acid, 2 g of thiourea (distilled water was used as a solvent).

The protective effect of the anticorrosion coating was $Z^+ = 98.78\%$; $Z^- = 99.33\%$.

Table 1

Aniline point value of solvents according to the ASTMD611-2016 method

Таблица1

Значение анилиновой точки растворителей по методу ASTMD611-2016

Oil solvent	Diesel fuel	Oil I-20A	Oil SN-150
Aniline point	72.1°C	90.3°C	113.8°C

Table 2

Determination of the amine number and surfactant solubility in hydrocarbons

Таблица 2

Определение аминного числа и растворимости ПАВ в углеводородах

No.	Reagent ratio (*g*/*g*)			Reaction time at the temperature of 180 to 200°C		Mass of the surfactant obtained, *g*	Mass of extracted water, *g*	Amine number, mg *HCl*/*g*	Surfactant solubility		
	Technical fat (*TF*)	Monoethanolamine (*EA*)	Boric acid (*BA*)	Obtaining of the clear solution	Reaction time				DT	Oil I-20A	Oil SN-150
1	130	65	6.5	20 min.	1 *h*	192.3	7.8	47.9	+	+	-
2	130	65	9.75	40 min.	1 *h*	195.1	9.2	48.1	+	+	+
3	130	65	13	1 *h*	1 *h*	194.0	11.2	49.2	+	+	+
4	130	65	16.25	1*h*	1 *h*	196.4	12.4	50.8	+	+	+
5	130	65	19.5	1 *h*	1 *h*	195.2	17.9	52.7	+	+	+

Table 3

Results of electrochemical measurements on St3 steel coated with films of I-20A oil with the surfactant in 0.5% solution of NaCl

Таблица 3

Результаты электрохимических измерений на стали Ст. 3, покрытой пленками масла И-20А с ПАВ в 0,5 М растворе NaCl

Studied mixture	Electrode potential ($-E_{cor}$), *V*	Corrosion current density (i_{cor}), A/m^2	Tafel's cathodic constant (b_k), *mV*	Tafel's anodic constant (b_a), *mV*	Corrosion rate ($K_{e/c} \cdot 10^{-4}$), kg/m^2h	Protective effect (*Z*), %
Control specimen	0.47	0.063	100	60	0.670	–
I-20A	0.01	0.018	100	66	0.185	72
I-20A +5% of the surfactant	0.05	0.008	100	100	0.0832	88
I-20A +10% of the surfactant	0.09	0.006	75	85	0.065	90
I-20A +15% of the surfactant	0.08	0.0058	100	100	0.061	91
I-20A +20% of the surfactant	0.04	0.008	75	70	0.083	88
I-20A +25% of the surfactant	0.05	0.025	100	100	0.26	61

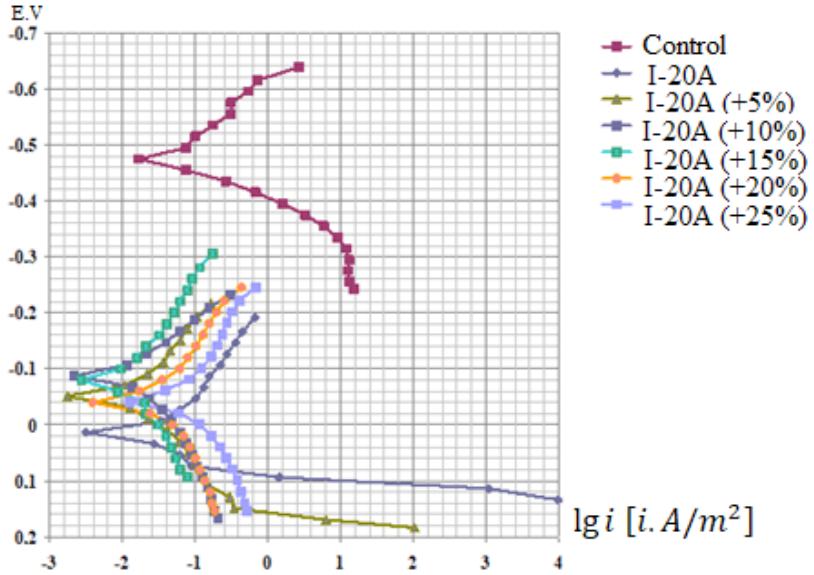
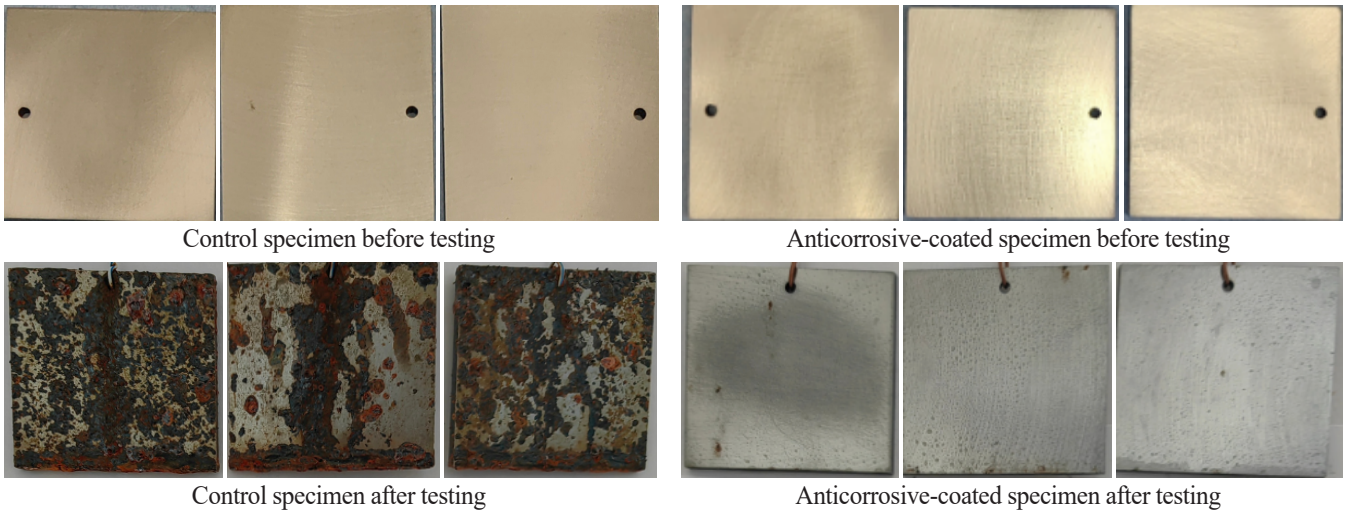


Fig. 2. Polarization curves of St3 steel at surfactant concentration in I-20A:
▲ – 5%; ■ – 10%; ■ – 15%; ● – 20%; ■ –25%

Рис. 2. Поляризационные кривые на стали Ст3 при концентрации ПАВ в И-20А:
▲ – 5%; ■ – 10%; ■ – 15%; ● – 20%; ■ –25%



Control specimen before testing — Anticorrosive-coated specimen before testing

Control specimen after testing — Anticorrosive-coated specimen after testing

Fig. 3. Test results in the humidity chamber

Рис. 3. Результаты испытаний в камере влажности

Table 4

Results of corrosion tests in the humidity chamber

Таблица 4

Результаты коррозионных испытаний в камере влажности

No. of the specimen*	Mass of the specimen before testing, *g*	Mass of the specimen after testing, *g*	Mass of the specimen after removal of corrosion products, *g*	Change in mass of the specimen after testing, *g*, $\pm\Delta m$	Protective effect, %
1*C*	57.1742	57.4382	56.3203	+0.2640 –0.8539	-
2*C*	57.3145	57.5501	56.4630	+0.2356 –0.8515	
3*C*	57.3601	57.5741	56.6975	+0.2140 –0.6626	
1*I*	57.0112	57.0143	57.0057	+0.0031 –0.0055	$Z^+ = 98.78$ $Z^- = 99.33$
2*I*	57.3274	57.3317	57.3196	+0.0043 –0.0078	
3*I*	57.3448	57.3474	57.3389	+0.0026 –0.0059	

** C – control specimen; I – anticorrosive-coated specimen*

Conclusions

The production of surfactants from non-food fatty raw materials solves the environmental problem associated with the use of fat-containing waste. The technology developed makes it possible to synthesize the product in one stage in a chemical reactor without dividing the technology into two stages: hydrolysis of triglycerides and production of fatty acid amides. The obtained non-ionogenic surfactant having good solubility in the I-20A industrial oil is a highly effective anodic corrosion inhibitor. At its optimum concentration in 15% industrial oil, its protective effect is 99.33%.

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