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OBTAINING OF OLIGOMERIC CORROSION INHIBITORS BASED ON POLYCAPROLACTAM WITH MELAMINE ADDUCT (PUM-1)

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Abstract

In this article, we synthesized oligomer corrosion inhibitors from polycaprolactam and acetic acid, in addition, melamine adducts was used for PUM-1 inhibitor. The structure of the obtained corrosion inhibitors was confirmed by spectroscopic techniques, as well as fundamental analysis. The inhibition efficiency of this PUM-1 brand corrosion inhibitor was determined by electrochemical methods in a 1 M HCl environment. Specially, the factors affecting the inhibition efficiency, such as the pH of the solution, the duration of time, and the concentration of the inhibitor, were studied.

Keywords: corrosion inhibitor, polycaprolactam, urea, melamine, orthophosphoric acid.

1. Introduction

Corrosion inhibitors are widely used to protect metals against various corrosive environments [1–3]. A corrosion inhibitor is a compound that is added in low concentrations to a corrosive solution to reduce and/or minimize the corrosion rate [4-6]. If we care about the economic damage of this corrosion process, then the figure is really devastating in modern word. According to the results of international research conducted by NACE (IMPACT 2016), the annual economic damage of the corrosion process worldwide is 2.5 trillion US dollars. We can conclude that the percentage for individual countries is about 3.4% of the average gross domestic product (GDP) [7-9]. According to the results of many scientific studies, the environment should be taken into account when selecting corrosion inhibitors, and the use of nitrogen- and sulfur-containing compounds based on them is more effective in acidic environments[10].

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Experimental part

Obtaining of oligomeric corrosion inhibitor with melamine Adduct (PUM-1)

The optimal conditions for the synthesis of the condensation product of polycaprolactam with the melamine adduct (PUM-1) were carried out at a temperature of 118°C, i.e., at the boiling point of acetic acid. First, 1.13 kg of polycaprolactam, and 5.00 l of acetic acid are loaded into a reactor with a volume of 0.01 m^3, equipped with a refrigerator and a stirrer, and 2.52 kg of melamine adduct with phosphoric acid is gradually poured and boiled with stirring at the boiling point of acetic acid 391 K for 2.0 hours. Then the excess acetic acid is distilled off and 3.56 kg of an oily light yellow product, highly soluble in water, is obtained. Density 1.3 g/cm^3[17,18].

Preparation of melamine adduct with phosphoric acid

1.26 kg of melamine and 1.60 kg of phosphoric acid are loaded into the reaction chamber. The reaction mixture is stirred for 15-30 minutes, and a clear syrupy liquid is obtained. The resulting substance has the following characteristics: viscous liquid of a light yellow or brown color and non-volatile.

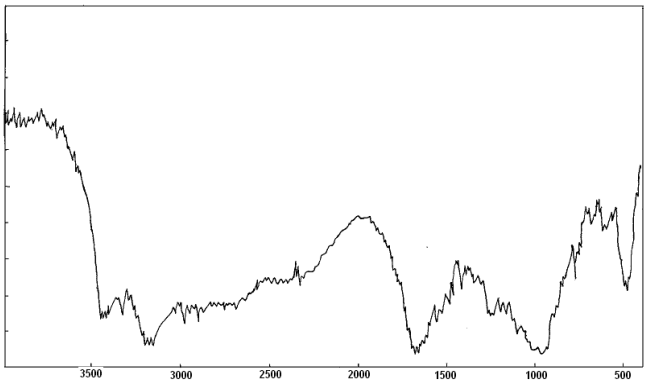


Fig 1. IR spectra of an oligomeric corrosion inhibitor based on polycaprolactam and melamine adduct.

The IR spectrum of the resulting compound has absorption bands at 3450 and 3330 cm^{-1}, which correspond to the free hydroxyl group, and accordingly, there is –NH bands in the regions of 3150, 3200 and 1550 cm^{-1}, C=O in the regions of 1650 and 1680 cm^{-1}, P = O and P-O-C in the regions of 960 and 970 cm^{-1}. The data obtained showed that if the reagent ratio of 2: 1 is violated, the yield of the oligomer decreases, and, in relation to this, a decrease in molecular weight (viscosity) from 0.1 to 0.08 dL/g is observed,

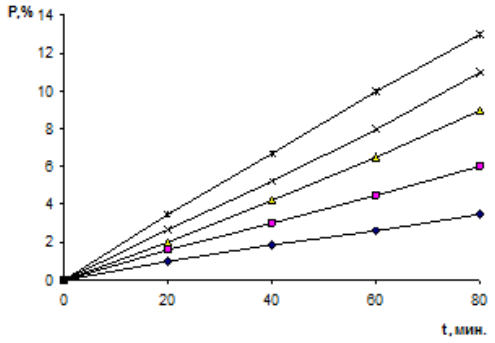
We also studied the effect of the solvent on the product yield. When using a solvent in small quantities, a decrease in the yield of the product is observed, and, in connection with this,

the molecular weight decreases. The identified features are probably associated with the shielding of the active groups of biradicals during the reaction.

Table 1. The influence of the ratio of reagents on the composition of the product (T = 391 K, Time = 2 hours)

PC+Ad Melamin	Yield, %	$\eta_{пр}$ 0.5 aq. solution. dl/g	Elemental analysis			
			Nitrogen		Nitrogen	
			Calculated	Calculated	Calculated	Calculated
1:3	63.6	0.08	28,8	28.4	8.91	8.87
1:2	67.2	0.07		28.3		9.08
1:1	73.5	0.1		27.6		9.1
2:1	77.9	0.09		28.91		8.89
3:1	71.4	0.08		28.5		8.65

Table 1 shows the results of the interaction of polycaprolactam with the melamine adduct in different ratios. At a molar ratio = 2:1, the yield of the oligomer increases and at the same time the molecular weight of the product increases.



1—2:1; 2—1:1; 3—3:1; 4—1:2; 5—1:3

Figure 2. Kinetic dependence of polycondensation in the system polycaprolactam: melamine adduct in acetic acid. (T =391 K).

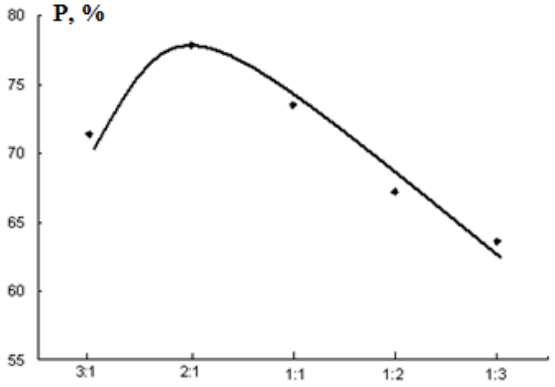


Figure 3. Dependence of oligomer yield on ratio starting materials. T= 391 K. Time 1.5 hours.

A study of the kinetics of the dependence of the reaction rate, determined by the rate of consumption of the melamine adduct on the ratio of the initial reagents, showed that the

kinetic dependence of the formation of the oligomeric product is characterized by a decrease in the slope in the case of a violation of the 2:1 equimolarity (Figures 2 and 3).

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