

TECHNOLOGY FOR OBTAINING DETERGENT-DISPERSANT ADDITIVES BASED ON CARBONIC ACID AND AMIDE DERIVATIVES FOR DIESEL FUELS

Karimova Sadokat Amrilloevna
sadoqat_karimova37@bsmi.uz

Abstract: This article presents the technology for obtaining detergent-dispersant additives for diesel fuels based on carboxylic acids and amines.

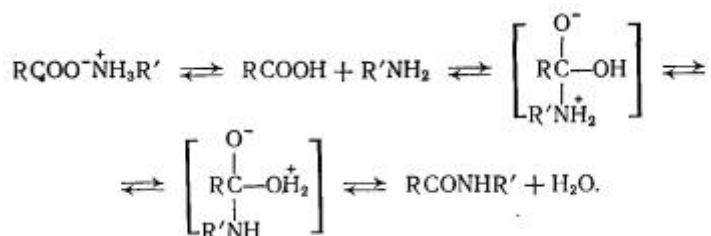
Keywords: carboxylic acids, amines, amidation, diesel fuel, detergent-dispersant additives.

In the development of detergent-dispersant additives, experimental data obtained by special methods are of great importance. A complete description of these methods can be studied by the method of a set of qualified methods for evaluating diesel fuels. The existing methods for assessing the activity of detergent-dispersant additives can be divided into two groups: 1. The method of studying the dispersing and stabilizing properties of additives, their effect on the stability of model fuels (individual hydrocarbons, binary mixtures of hydrocarbons) or artificially prepared suspensions in real fuels of known composition (composition, deposits in real fuels). The dispersing properties of additives, the change in the dispersion of the compositions in the presence of the additive are evaluated by optical or sedimentation methods, and the stabilizing properties are determined by the change in the stability of the obtained dispersion over time. The solubility properties of additives are evaluated by their ability to transfer additives from model fuels into a colloidal solution.

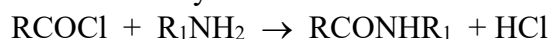
2. A method for assessing the effect of additives on the formation of precipitates when heating real fuels to a temperature of 220-225 ° C. Dynamic methods include a method based on the initial temperature of the resinification process of diesel injector nozzles, in which real fuel with and without additives, heated by electric current at a temperature of 120-220 ° C for 5 hours, passes through the nozzles.

When synthesizing detergent additives, caprylic, lauric, myristic, palmitic and stearic acids, as well as benzoic and n-toluic acids, were obtained as carboxylic acids.

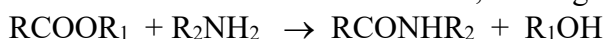
The amidation process is of great importance in the organic and petrochemical industries for the production of a number of valuable products. Amidation is similar to the esterification reaction. The structure of the carboxylic acid affects the rate and thermodynamics of amidation, with the elongation and branching of the carboxylic acid carbon chain increasing the equilibrium constant of the reaction, which reduces the rate of the process. Compared to alcohols, ammonia and amines are strong nucleophilic reagents, therefore, amidation is carried out in the liquid phase without the participation of a catalyst, by heating the reagents at 200-300 ° C. The removal of water from the reaction phase, the removal of excess ammonia or amine, allows for a high degree of conversion. In some cases, acidic catalysts (Al_2O_3) are used. It is known that when carboxylic acids react with amines, an ammonium salt is first formed, which is inactive for amidation, and its formation reduces the activity of both reactants. Therefore, the salt is dissociated into free carboxylic acid and amine, and then amidation is carried out. The amidation reaction can be written as follows:



Here R is C₉-C₁₇. Morpholine, diethylamine, and acrylamide were used as amines. The rate of the amidation reaction is directly proportional to the concentration of free carboxylic acid and amine, and is determined by the salt dissociation equilibrium. This equilibrium constant increases with increasing temperature and depends on the choice of amidation temperature. Amidation, like esterification, can be carried out at very mild temperatures under the influence of carboxylic acid chloride:



Esters also react with amines, shifting the equilibrium to the right.



These reactions proceed much faster than the amidation of carboxylic acids. Therefore, these reactions are considered convenient as a method of synthesizing some amides at temperatures of 50-100 ° C without the participation of a catalyst.

The continuous technology for obtaining amides was implemented as follows. From the measuring device 1, a mixture of reagents - carboxylic acid, amine, solvent and catalyst - is continuously fed to the heat exchanger 2, the reaction mixture is heated by steam and enters the upper plate of the reaction column 4. During the formation of amide, as a result of the intense steam heating of the cube column, amine and water vapor are expelled from the column, the liquid moves down along the plates and becomes enriched with water. The time of residence of the reaction mass in the reactor and the ratio of the initial reagents are adjusted so that the cube liquid contains a small amount of unreacted carboxylic acid. This liquid is removed from the cube and, after neutralization, is discharged into the sewer. The steam entering the reactor from above contains 70.0% amine and 20.0% amide. It is sent for cooling and condensation occurs first in heat exchanger 2, where the initial reagents are heated, and then in condenser 3. Condensate from device 2 and part of the condensate from device 3 are returned to the upper plate of reactor 4. The rest of it passes into rectification column 5, where an azeotropic mixture is separated from the aqueous amine.

The technological scheme for obtaining detergent-dispersant additives based on amides in a continuous method is shown in Figure 1.

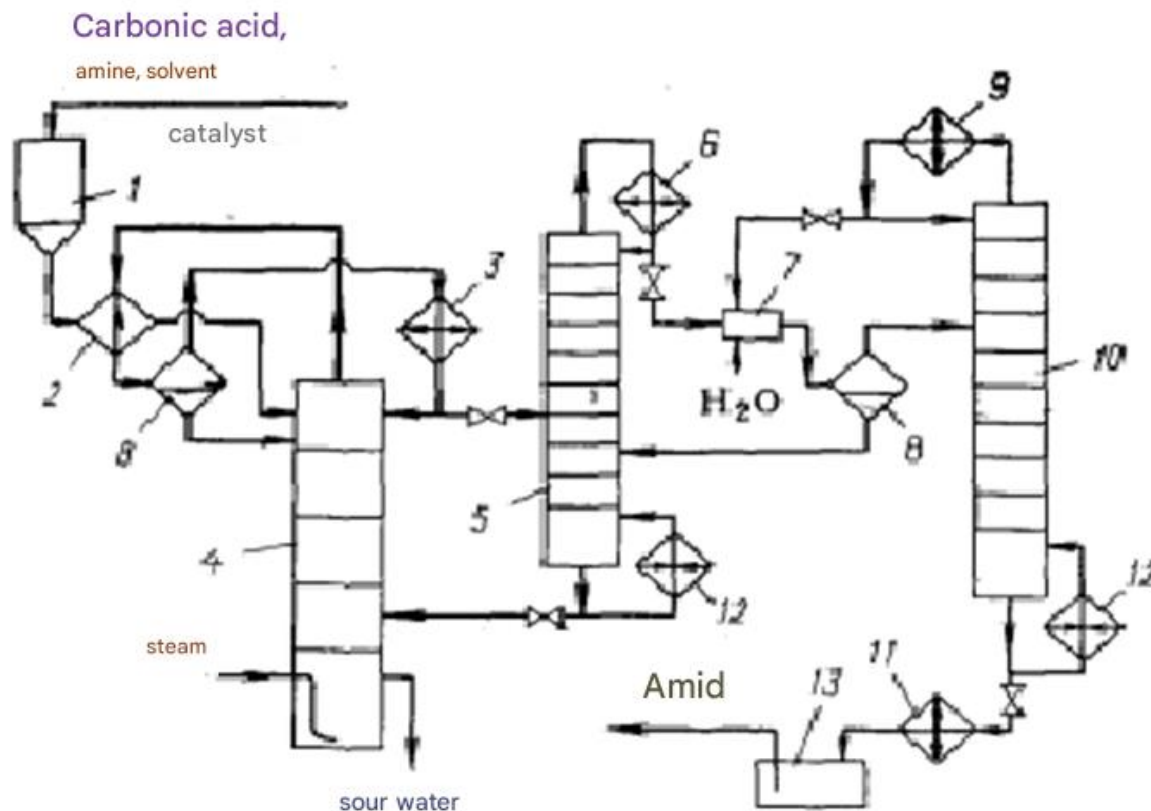


Fig. 68. Technological scheme for the continuous production of amides:

1- measuring device; 2- heat exchanger; 3- condenser; 4- amidizer-reactor; 5,10- rectification columns; 6,9- condenser-dephlegmators; 7- mixer; 8- separator; 11- cooler; 12-boiler; 13- collector.

The cube column 5 is heated by a boiler 12, a flash is formed in the apparatus 6, part of the condensate is diluted with water. The liquid in the cube column 5 consists of amine (most of it) and water. It leaves the column and enters the reactor 4 through the lower plate. This ensures that a sufficient amount of amine is present in the lower part of the column, and complete conversion of carboxylic acid.

The steam leaving the column 5 is condensed in the apparatus 6, from where part of the condensate is diluted, and the remaining amount is diluted with an equal volume of water in the mixer 7. The resulting emulsion is separated into two layers in a continuously operating separator 8, the upper layer contains the amine and amide dissolved in water, the lower layer consists of an aqueous solution of amine and amide. The lower layer is returned to the middle plate of the column 5. Amide is directed to the separator 8 to separate water and amine. It is sent to the distillation column 10 and separated into a tertiary azeotropic mixture of amide, amine and water by distillation. Part of this mixture is diluted in the condenser 9 and then in the column 10, and the remaining amount is returned to the mixer 7. The amide cube passes through the column 10 and, after cooling in the cooler 10, is directed to the collector 13.



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