

Synthesis and Application of Oxypropylated Amines

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Abstract—Methods for the preparation of oxypropylated amines and their application in various fields are presented. The successful use of amine oxypropylation products as additives to oils, surfactants, corrosion inhibitors, stabilizing additives to polymer mixtures, etc., is described. Their effectiveness as oil-collecting and oil-dispersing reagents in the elimination of the consequences of accidental oil and petroleum product spills on the water surface is considered.

Keywords: oxypropylates, oxyalkylated amines, surfactants, oil-collecting and oil-dispersing compounds, stabilizers

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The products of the interaction of organic compounds containing amine fragments with epoxides are widely used in many industries. Particular attention is paid to oxypropylated amines, which are of interest as surfactants, fuel additives, anticorrosive additives for motor oils, rubber stabilizers [1], components of anti-icing fluids [2], etc.

OXYPROPYLATED AMINES AS ADDITIVES

To test oxypropylated amines as additives for various purposes in oils, several studies have been carried out in the field of the synthesis of these compounds. The process of oxypropylation of benzoguanamine mixed with monoalkyl (C₈–C₁₂) phenols in an autoclave system at a molar ratio of benzoguanamine, monoalkylphenols, and propylene oxide of (0.25–0.40) : 1 : 1.25 and temperature 160–180°C is patented [3]. The duration of the process is 2–3 h.

The catalytic interaction of benzoguanamine with propylene oxide was studied both separately [4] and in the presence of phenolic compounds [5]. Among the basic catalysts, the maximum conversion of propylene oxide (93.6–98.9%) is achieved with the use of KOH, while in the presence of triethylamine, the conversion does not exceed 45%, and in the absence of a catalyst, it is 40–45%. Benzoguanamine oxypropylates, obtained at a molar ratio of the amine component to epoxide of 1 : 5, have a positive effect on the viscosity–temperature characteristics of the base motor oil [6]. The viscosity index of M-8 engine oil increases from 71 to 105 with the addition of benzoguanamine oxypropylate in an amount of 1 to 3 wt % of the oil, and up to 118 with the addition of 5 wt % (GOST (State Standard) 25371-82).

Amiraslanova et al. [7] studied the anticorrosive and antioxidant properties of nitrogen-containing phenol oxypropylates and derivatives monoalkyl (C₈–C₁₂) phenols in the base motor oil. In the presence of benzoguanamine oxypropylates, as well as the products of oxypropylation of the latter together with phenolic compounds, a high thermal stability of the oil is achieved, and their corrosiveness is also reduced. The antioxidant properties of M-8 engine oil were determined using the DK–NAMI device, according to GOST 11063-77. The analyses were carried out with the addition of 2.4 wt % of a 50% oligomer solution to the base oil. In this case, the sediment during thermal oxidation is 0.068% for the optimal sample, while the kinematic viscosity after oxidation increases by only 10%. Similar investigations were carried out to study the anticorrosive properties of the synthesized oligomers, according to GOST 20502-75. A 52% decrease in the corrosion rate was achieved for the same engine oil by using benzoguanamine oxypropylates as additives.

A continuation of research in this field led to the determination of the antimicrobial properties of the products of oxypropylation of benzoguanamine in the presence of phenolic compounds [8]. The analyses were carried out using mixtures of two bacterial strains (*Pseudomonas aeruginosa* and *Mycobacterium lacticola*) and five species of molds (*Aspergillus niger*, *Cladosporium resinae*, *Penicillium chrysogenum*, *Chaetomium globosum*, and *Trichoderma viride*). The zone of inhibition of bacteria (a mixture of two types) with the addition of an oligomer in an amount of 0.5 and 1 wt % of I-40 industrial oil was 1 and 2.1 cm, respectively. The zone of inhibition of fungi (a mixture of five species) at the same oligomer consumption is 1.3 and 3.0 cm, respectively, while the exclusion of the test sample

leads to an intensive growth of microorganisms (*GOST 9.052-88* and *GOST 9.082-77*). The test results for 8-hydroxyquinoline, selected as the reference sample of additives, showed bacterial growth and significantly worse fungicidal properties compared to oxypropylates.

OXYPROPYLATED AMINES AS SURFACTANTS: OIL-COLLECTING AND OIL-DISPERSING COMPOUNDS

Amine hydroxypropylates with surfactant properties are also capable of killing bacteria. Oxypropylated quaternary ammonium surfactants with two or six epoxy fragments have high antibacterial properties against both gram-negative bacteria *Escherichia coli* and gram-positive bacteria *Staphylococcus aureus* [9]. Their micelle formation in a water–alcohol mixture (reversible and irreversible) was studied. The effect of oxypropylene fragments on the micelle-formation properties was studied using physical methods, for example, UV spectroscopy, and dynamic light scattering. Surfactants of similar composition also have anti-static activity.

Oxyethylated amines, which are rarely described in publications, have similar properties. Being protonated in acidic media, they exhibit antistatic, bactericidal, anticorrosive, and other properties of cationic surfactants. In relation to this, they can be used as wetting agents, emulsifiers, dispersants, corrosion inhibitors, antistatic agents, and bactericides [10, 11]. Non-ionic surfactants were obtained by the interaction of fatty amines, including laurylamine, stearylamine, oleylamine, secondary amines (for example, dicoamine), and diamines with ethylene oxide. The onset of the reaction is judged by an increase in temperature and a decrease in pressure in the system; ring-opening occurs at 150–170°C. The typical amine number of these surfactants ranges from 80 to 250 mg KOH/g of the substance.

Oxyethylated amines are also of interest as electro-active substances. As an example, we consider a method of obtaining ethoxylated high-lipophilic quaternary ammonium salts, developed by Okaev [12], which includes several stages: alkylation of methylgallate with dodecyl bromide, reduction of the ester group, ethoxylation, chlorination, and quaternization of a halogen derivative with a tertiary amine. The synthesis results in salts with the properties of ionic liquids, which are promising components of the membranes of ion-selective electrodes.

Due to the rapid development of the oil industry, the need for surfactants for collecting oil spills from the surfaces of seas, rivers, etc., including epoxy-based reagents, is increasing [13–17]. Oil-collecting and oil-dispersing reagents of similar composition are used to eliminate thin oil films from the water surface, which are a consequence of the emergency spills of oil and

petroleum products. Their physicochemical, colloidal-chemical properties, oil-collecting, and oil-dispersing properties have been studied.

Rahimov et al. [18, 19] synthesized *N,N'*-Bis(2-hydroxypropyl)ethylenediamine by the interaction of ethylenediamine with propylene oxide. At the second stage, the reaction of the obtained amino alcohol with higher carboxylic acids (C_{14} , C_{16} – C_{18}) [18] and alkyl iodides [19] yielded dimeric (gemini) surfactants with a high surface pressure, high electrical conductivity, good wetting ability, and other important parameters. The authors revealed a high oil-collecting capacity of an organic salt based on *N,N'*-bis(2-hydroxypropyl)ethylenediamine and palmitic acid in seawater with an oil-collecting coefficient of 13.4 for a pure substance and 17.4 for its 5% aqueous solution.

Hasanov et al. obtained *N,N,N,N'*-tetra-(propan-2-olyl)-1,6-hexadiazine [20] and *N,N*-di-(propan-2-olyl)-1,6-hexadiazine [21] by the reaction of hexamethylenediamine with propylene oxide. Further interaction of the products with saturated and unsaturated fatty acids (capric, lauric, myristic, palmitic, stearic, and oleic) yielded new dimeric surfactants. The synthesized surfactants with palmitic and stearic acid fragments showed a high level of stability of the foam and emulsion in an aqueous medium. Compounds based on myristic acid show a decrease in surface tension to the values characteristic of commercial fluorinated surfactants.

A new series of surfactants were obtained by the oxypropylation of complexes based on palmitic acid and monoethanolamine (MEA), triethylenetetramine (TETA) [22], oleic acid and ethylenediamine (EDA), or TETA [23] at a molar ratio of complexes to propylene oxide of 1 : (1–3). The process was carried out in an autoclave heated on a sand bath at a temperature of 120 to 130°C for 25–30 h, with a high conversion rate of propylene oxide, 90.5–94.0%. The surface properties of the resulting substances, such as the critical concentration of micelle formation (CMC), the minimum surface area (A_{\min}), and the excess surface concentration (T_{\max}), were investigated, and their structure was confirmed using Fourier-transform spectroscopy and C_{13} and H^1 NMR spectroscopy. Oxypropylated complexes have demonstrated efficiency as an oil-dispersing reagent on the surface of fresh and sea water with $K_d = 91.1$ – 95.5% and $\tau = 4$ – 100 h.

Asadov et al. [24] synthesized surfactants with high colloidal-chemical parameters by the interaction of alkylamines (octylamine, nonylamine, dodecylamine, hexadecylamine, and octadecylamine) with propylene oxide in a molar ratio of 1 : 1 and 1 : 2. Other ionic surfactants were obtained by the subsequent reaction of dodecylisopropylolamine (obtained from dodecylamine and propylene oxide) with various acids (hydrochloric, hydrobromic, acetic, and propionic acids) and alkyl halides (methyl iodide, ethyl bromide, and *n*-propyl bromide), and their capability of oil-col-

lection and oil-dispersion was studied [25]. The introduction of alkoxy groups into the composition of alkylamines leads to the production of valuable products for various purposes: auxiliary agents in the textile industry, activators for pesticides, thickeners for acid reagents, antistatic materials, detergents, lubricants, and antiwear additives.

Quaternary ammonium salts obtained by the interaction of alkylamines (octylamine, nonylamine, dodecylamine, and hexadecylamine) with propylene oxide in a molar ratio of 1 : 2 and subsequent reaction with 2-chloroethanol also demonstrate a high degree of surface activity and oil-collecting properties [26]. Surfactants based on dodecylamine, propylene oxide, and 2-chloroethane exhibit the highest ability to recover oil. In [27], nonylamine, octadecylamine, and aminoamides obtained from the fatty acid fraction of flaxseed oil were subjected to oxypropylation. Tensiometric studies proved that nonylisopropylolamine exhibits a higher surface activity than octadecylisopropylolamine, lowering the surface tension at the air-liquid interface from 72.0 to 24.1 mN/m at a concentration of 5.0 wt %. The maximum value of the oil recovery factor (K_{\max}) was found for the aminoamide oxypropylate. The K_{\max} of a pure substance is 30.4 in three types of water (distilled, fresh, and sea). When a 5% aqueous solution of the reagent is used for 25–169 h, the oil recovery factor in freshwater did not change ($K_{\max} = 30.4$); for seawater, this indicator turned out to be lower (20.3); and for distilled water, it became higher (40.5) in comparison with the use of a pure substance. Also, the improvement in oil recovery after 169 h (~7 days) and the stability of the collected oil slick for 12 days indicate the efficiency of the proposed reagent. The tests were carried out using oil of the Raman field of Azerbaijan.

Oil-soluble oligomers based on benzoguanamine, propylene oxide, and monoalkyl (C_8 – C_{12}) phenols in the presence of polypropylene glycols have demonstrated good oil-collecting ability in the sea and fresh water [28]. The maximum K_{\max} value of these compounds in seawater was 13.44, remaining unchanged for more than 8 days. In freshwater, this value was lower ($K_{\max} = 9.18$) but stable. Oligomers containing alkylphenols (without amine compounds) turned out to be less effective, just as amine and phenolic amine oxypropylates, which showed the effectiveness of the action of alkyl and amine groups at their simultaneous presence in the composition of oligomeric macromolecules.

To obtain a new series of oxypropylated nitrogen-containing compounds, studies were carried out on the synthesis of oligomerization products of propylene oxide with imidazolines, based on distilled natural petroleum acids and polyethylene polyamines [29]. The structure of the synthesized oligomeric products was identified by IR spectroscopy; the opening of epoxy rings, chain growth, and attachment of amine

fragments were confirmed. We studied their properties and suitability for use as oil-collecting and oil-dispersing reagents.

By the interaction of alkylamines (octylamine, nonylamine, dodecylamine, hexadecylamine, and octadecylamine) with epichlorohydrin, Asadov et al. [30, 31] synthesized compounds of a similar structure and with similar performance properties as in the reaction with propylene oxide. The structure of the synthesized compounds was identified by IR and PMR spectroscopy. Their use reduces the surface tension at the air-water interface from 72.5 to 26.0 mN/m at 20°C. They demonstrate a high level of efficiency: the recovery rate for an oil film with a thickness of 0.17 nm is 30.4 with a retention time of more than 8 days.

STABILIZING ADDITIVES BASED ON OXYPROPYLATED AMINES FOR POLYMERIC MATERIALS

One of the fields of application of amine oxypropylates is the stabilization of polymers, aimed at combating their aging under the action of various environmental factors (light, oxygen, ozone, etc.), which lead to a deterioration in performance. The problem of stabilization is considered important in the field of polymer chemistry, and the search for effective stabilizing additives remains relevant to this day [32]. It is of interest to develop a technology for the production of functionalized polyethers based on the polymerization of propylene oxide on alkaline alcoholates of aromatic amino alcohols. Several works are devoted to the preparation of compounds of a similar composition, in particular, oxypropylated aromatic amines, recommended for widespread use as additives to polymer mixtures to stabilize their operational and technological properties.

Ochilov and Shonazarova [33] described the preparation of oxypropylaromatic amines for this purpose at a molar ratio of amine, oxide, and propylene of 1 : (1–6), various temperature conditions of the process (80, 100, 120, 140°C), and reaction time from 10 to 120 min. The structure of the products was identified by proton nuclear magnetic resonance.

The following method for producing an amine antioxidant based on β -oxypropylated 4-aminodiphenylamine to stabilize stereoregular polyisoprene rubber is well known. The interaction is performed at a temperature of $100 \pm 0.5^\circ\text{C}$ in a metal reactor of an ideal mixture of the isothermal type with a molar ratio of amine and propylene oxide of 1.0 : 1.1 [34]. Despite the low activity in inhibiting the mechanical destruction of rubber, oxypropylated derivatives of *n*-aminodiphenylamine are effective for protecting against thermo-oxidative aging. An increase in the degree of oxypropylation enhances the effect of protecting the polymer material.

Dorofeev and Zemskii [35] presented the results of a study of synthesized oxypropylated *n*-phenylenediamine as stabilizers of tire rubber. According to the test results of two samples for a tread and a belt, the elastoplastic and physical-mechanical properties of tire rubbers using oxypropylated *n*-phenylenediamine and the industrial stabilizer Santoflex 6PPD are approximately the same. The kinetics of thermo-oxidative aging shows a tendency to improve the results with the long shelf life of the tested rubbers in the case of the addition of the synthesized oxypropylate. Satisfactory results for belt rubbers were obtained with ozone aging as well.

Ozone aging is one of the key stages in the aging of a polymer product, in particular, rubber. The study of the mechanism of the reaction of ozone with new classes of antiozonants, some of which include oxypropylated aromatic amines, is the focus of attention.

In [36], a study was made of the effect of the degree of oxypropylation of aromatic amines on their reactivity with ozone. An increase in their molecular weight has a positive effect on the rate of interaction with ozone. In the author's opinion, this is because, with an increase in the length of the oxypropylene chain, the number of ether groups also increases, which, like nitrogen in the NH group, take part in the reaction with ozone. The most reactive was the oxypropylated aniline grade A-20, recommended as a stabilizer, with a degree of oxypropylation of 20 (the rate constant of the reaction of interaction with ozone $k_{\text{eff}} = 9.9 \times 10^6$ L/mol s). The degree of oxypropylation can be controlled, for example, during the synthesis of alkoxyated alkylamines with a narrow molecular weight distribution, the method of obtaining of which is presented in [37]. At the first stage, the reaction of the primary alkylamine and alkylene oxide proceeds at a temperature of 150 to 190°C; then the product interacts with an additional number of moles of the epoxy compound in the presence of a catalyst containing a multiply charged counterion.

In [38], the opposite dependence on the length of the oxypropylene chain is observed, since its increase leads to a decrease in the inhibiting ability of oxypropylated aniline during oxidation. The oxidation of ethylbenzene is taken as the model reaction. A stabilizer of grade A-3 with a degree of oxypropylation of $n = 3$ was more efficient as an inhibitor and was recommended for use in industry.

The structure of monoxypropylated aniline (MOPA) was identified by IR spectroscopy. Such physicochemical properties as density (1.057 g/cm³), kinematic viscosity, and its temperature dependence have been determined [39]. The thermal properties of MOPA were studied by differential scanning calorimetry (DSC) [40], which was used to determine the phase transition temperature (256.52°C) and enthalpy (−5.47 kJ mol^{−1}). This method, based on recording the difference in heat fluxes depending on tempera-

ture and time, enables the study of thermal processes and the identification and comparison of materials.

The use of MOPA, due to its inhibiting properties, makes it possible to increase the stickiness of rubber compounds, increase resistance to high temperatures and oxidants, and improve their adhesive properties to various surfaces.

MOPA can serve as the starting material for the production of polyoxypropylated aromatic amines by anionic polymerization. To achieve the efficiency of polyoxypropylated aromatic amines as antioxidants and antiozonants, it is important to preserve the secondary amine groups containing a mobile hydrogen atom, which are typical traps of peroxide radicals [41]. Otherwise, the formed tertiary amine structures do not have antioxidant properties.

Article [42] is devoted to the chromatographic study of the reaction mixture during the synthesis of MOPA, which is recommended as an ingredient for vulcanizates, as well as for obtaining catalytic systems used in the production of stabilizers for rubber compounds, which effectively protect them from various types of aging, for example, fatigue failure, ozone cracking, thermal-oxidative destruction, and abrasive wear.

The yield of *N,N*-dioxypropylated aniline obtained at a molar ratio of aniline and propylene oxide of 1 : 2 and a temperature of 100°C was 90% [43]. The concentration of mono- and dioxypropylated amine in the mixture depends on both the molar ratio of the components and the synthesis conditions; with an excess of propylene oxide, predominantly *N,N*-dioxypropylated aniline is obtained.

A technology was developed for the production of polyethers by polymerization of propylene oxide, where catalytic complexes based on alkaline (potassium) alcoholates of oxypropylated aniline were used as a catalyst [44]. Both the catalyst preparation process and the polymerization of propylene oxide are described in a process flow diagram, where the complete conversion of propylene oxide and temperature constancy are achieved by heat removal. Some alcoholates of oxypropylated aromatic amines are effective as modifiers of the microstructure of styrene–butadiene rubbers.

Yarullin et al. [45, 46] determined the structure and composition of the polymerization products of propylene oxide in the presence of an alkaline alcoholate of oxypropylated aniline by NMR spectroscopy [45, 46]. The retention of the secondary amine group was proved during the anionic polymerization of propylene oxide. The synthesized polyoxypropylated alkaline aniline alcoholates can be recommended as rubber stabilizers.

To create Russian-made amine-type stabilizers, a technology for obtaining oxypropylated *n*-toluidine with a capacity of 1500 kg/h was developed [47, 48], material and heat balances of the installation were

compiled, and consumption coefficients and specific energy costs for raw materials were calculated. The optimal molar ratio of toluidine and propylene oxide, at which sufficiently high technical and economic indicators are achieved with allowable energy consumption, was (1.0–1.2) : 1. The conversion of propylene oxide was 100%, the conversion of *n*-toluidine was 65.4%, the selectivity of the formation of monoxypropylated *n*-toluidine was 72.48%, and the specific energy consumption was 1.87 kW/kg.

The structure of oxypropylated *n*-toluidine was studied by IR spectroscopy and chromatography [49]. In the IR spectrum of *N*-hydroxypropylated *n*-toluidine, one broad intense absorption band is detected in the range of 3200 to 3400 cm⁻¹, corresponding to secondary amine fragments. Both mono- and dioxypropylated *n*-toluidine were studied by gas chromatography.

Lin'kova et al. [50, 51] studied the structure and isomeric composition of xylidine oxypropylation products by NMR and mass spectrometry, using the example of 2,6-dimethylaniline. The proposed mechanism is common for all processes of β-oxypropylation of aromatic amines and shows options of partial or complete substitution of hydrogen atoms in amine groups. The general regularities of the decay of molecules by the action of electron impact are shown. The first act in the electron impact is the release of ethoxy of methoxy radical ions; therefore, it can be argued that propylene oxide serves as an alkylating agent. The technological scheme for obtaining xylidines was developed and modeled in [52].

Thus, the synthesis and application of oxyalkylated, in particular, oxypropylated amino derivatives, is of interest both from a theoretical and practical point of view. The fields of application of compounds of this composition are quite diverse, which is facilitated by their structural features and the presence of groups and fragments of polar and nonpolar nature. Among the operational purposes highlighted in this review, we note the effectiveness of the synthesized amine oxypropylates as surfactants, oil-collecting and oil-dispersing reagents, antibacterial and antistatic materials, antiwear additives to oils, stabilizers for polymer compositions, etc.

Taking into account the above consideration, it can be argued that the need for these materials determines the prospects of research and the relevance of the search for new methods of their preparation and the synthesis of compounds of a similar composition.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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