

ВЛИЯНИЕ КАТАЛИЗАТОРА ФАЗОВОГО ПЕРЕНОСА НА НАКОПЛЕНИЕ И РАСПАД ГИДРОПЕРОКСИДА ЭТИЛБЕНЗОЛА

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Исследованы закономерности окисления этилбензола и разложения гидропероксида в микрогетерогенных системах, образующихся при добавлении додецилсульфата натрия к углеводородным средам. Установлено выраженное влияние природы ПАВ на механизм превращения. Анионные мицеллярные ПАВ, включающие линейный углеводородный фрагмент, каталитически ускоряют разложение ROOH, тогда как углеводород-растворимые неионогенные ПАВ и твердые оксиды практически не влияют на скорость реакции. Детальное исследование кинетики разложения в присутствии анионных алкилсульфатов показало, что ускоряющее действие ПАВ связано с их коллоидными свойствами, а именно с образованием совместных агрегатов типа обратных мицелл, в которых облегчается разрушение пероксидной связи. Впервые установлено синергическое ингибирующее действие смесей алкилсульфата натрия с фенольными антиоксидантами при окислении различных углеводородов. При окислении этилбензола с небольшими добавками (<1%) додецилсульфата натрия (DDS) установлен уникальный случай эффективного самоторможения, механизм которого включает два положения: гидропероксид, основной источник свободных радикалов, разлагается гетеролитически в совместных микроагрегатах с DDS, разложение происходит избирательно с образованием ацетальдегида и фенола; фенол, являясь акцептором свободных радикалов, ингибирует окисление этилбензола. Основными продуктами окисления RH в этой системе является фенол и ацетальдегид. Следует отметить, что образование фенолов при разложении алкилароматических ROOH обычно связано с сильными кислотами. В случае DDS движущей силой разложения ROOH, по-видимому, является успешная ориентация молекул ROOH в микроагрегатах, образованных анионными поверхностно-активными веществами. Однако ряд экспериментальных факторов, а именно влияние длины углеводородной цепи в алкилсульфатах натрия на скорость окисления додекана и инертность дисперсии Na₂SO₄ в реакциях разложения гидропероксидов, позволяют предположить, что каталитическое действие DDS на разложение гидропероксидов связано с его коллоидными свойствами, а разложение происходит в совместных микроагрегатах, образованных DDS и ROOH. Эффективная энергия активации разложения ROOH при концентрации 10 мм составляет E_{ef}=66,3 кДж/моль. Окисление этилбензола полностью ингибируется при небольших добавках DDS (1 мм), а скорость разложения в атмосфере N₂ значительно возрастает. DDS увеличивает эффективную константу разложения ROOH. Это поверхностно-активное вещество можно рассматривать как эффективный катализатор разложения ROOH.

Ключевые слова: поверхностно-активные вещества, энергия активации, додецилсульфат натрия, этилбензол, гидропероксид этилбензола, фенол, ацетальдегид

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INFLUENCE OF THE PHASE TRANSFER CATALYST ON THE ACCUMULATION AND DECOMPOSITION OF ETHYLBENZENEHYDROPEROXIDE

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The regularities of ethylbenzene oxidation and hydroperoxide decomposition in microheterogeneous systems formed by adding sodium dodecyl sulphate to hydrocarbon media have been studied. A pronounced influence of surfactant nature on the transformation mechanism has been revealed. Anionic micellar surfactants including a linear hydrocarbon fragment catalytically accelerate ROOH decomposition, whereas hydrocarbon soluble non-ionic surfactants and solid oxides practically do not influence the reaction rate. Detailed study of the degradation kinetics in the presence of anionic alkyl sulphates has shown that the accelerating effect of surfactants is connected with their colloidal properties, namely, with the formation of joint aggregates of inverse micelles type, which facilitate the destruction of the peroxide bond. For the first time, a synergistic inhibitory effect of sodium alkyl sulphate mixtures with phenolic antioxidants in the oxidation of various hydrocarbons has been found. During the oxidation of ethylbenzene with minor additives (<1%) of sodium dodecyl sulphate (DDS) a unique case of effective self-inhibition has been determined, the mechanism of which includes two steps. Hydroperoxide, the main source of free radicals, decomposes heterolytically in joint microaggregates with DDS. The decomposition occurs selectively with the formation of acetaldehyde and phenol. Phenol being an acceptor of free radicals inhibits the oxidation of ethyl benzene. The main oxidation product of RH in this system is phenol and acetaldehyde. It should be noted that phenols formation at decomposition of alkylaromatic ROOH is usually connected with strong acids. In the case of DDS decomposition of ROOH seems to be driven by the successful orientation of ROOH molecules in the microaggregates formed by anionic surfactants. But a number of experimental factors, namely the influence of the carbon chain length in sodium alkyl sulphates on the dodecane oxidation rate and inertness of the Na₂SO₄ dispersion in the hydroperoxide decomposition reactions, make it possible to suggest the catalytic reaction of ROOH. Suggest that the catalytic effect of DDS on hydroperoxide decomposition is connected with its colloidal properties and decomposition occurs in common microaggregates formed by DDS and ROOH. The effective activation energy of ROOH decomposition at concentration of 10 mm is $E_{ef} = 66.3$ kJ/mol. The oxidation of ethyl benzene is completely inhibited at low additions of DDS (1 mM), and the decomposition rate in an atmosphere of N₂ significantly increases. DDS increases the effective decomposition constant of ROOH. This surfactant can be considered as an effective catalyst of ROOH decomposition.

Key words: surfactants, activation energy, sodium dodecyl sulphate, ethylbenzene, ethylbenzenehydroperoxide, phenol, acetaldehyde

INTRODUCTION

The oxidation of hydrocarbons by molecular oxygen is one of the most attractive areas of petrochemical synthesis: peroxides, ketones, acids and other oxygen-containing products [1-6]. On the other hand the actual problem is the protection of organic materials such as food and industrial oils, fuels, cosmetics and others from thermal-oxidative degradation under the influence of air oxygen. The scientific basis for the management of oxidation processes is the theory of liq-

uid-phase oxidation, developed as applied to homogeneous and heterogeneous processes [7-10]. However, many real-world systems are microheterogeneous, water-organic or become so during operation. Surfactant additives regulate the microstructures of the medium, prevent the release of water dissolved and formed during the oxidation process. The effect of surfactants on the oxidation behaviour of hydrocarbon substrates is practically unexplored, although apriori one would expect significant effects related to the possibility of association of surfactants and oxidation products con-

taining polar oxygen-containing groups, the involvement of inhibitors including hydrophilic fragments in these associations and consequently changes in their reactivity and even directions of reactions involving these compounds [11-15].

Additives of ionogenic surfactants have a catalytic effect on hydroperoxide decomposition and hydrocarbon oxidation.

In the presence of surfactants, not only the rate of the process can change, but also the composition of the resulting products, i.e. the direction of the reaction. The nature of the effect differs for cationic and anionic surfactants and depends on the nature of the oxidised hydrocarbon and the resulting hydroperoxide, as well as on the structure of the hydrocarbon fragment [11-16].

In recent years, surfactants, often called phase transfer catalysts, are widely used in various catalytic processes. In this regard, studies on the effect of individual surfactants on the process kinetics appeared [17-19].

Sodium dodecylsulfate anionic surfactants including saturated hydrocarbon fragments in molecule composition were used as initiators of microaggregation [13-15], the best known and the least reactive in radical-chain oxidation processes. The nature and extent of the influence of microheterogeneity and kinetic heterogeneity of the medium on the kinetics and mechanism of ethylbenzene oxidation and decomposition of ethylbenzenhydroperoxide were studied.

A large body of information on the influence of surfactants of various structures (cationic, anionic, nonionic) on the rate of oxygen uptake during oxidation of tetralin, cumene and cyclohexene is presented in work [6]. The absorption rate of oxygen increases in the presence of most cationic surfactants. Anionic surfactants in the form of acids increase the rate of oxygen uptake while their sodium salts slightly inhibit oxidation. In the two-phase system, the catalytic activity of cationic surfactants is greatly reduced in the presence of water as a separate phase (15, 17). It is noted that the presence of hydroperoxides is essential for surfactants catalysed oxidation of hydrocarbons. In this case, cationic and anionic surfactants react with more nucleophilic (internal) and more electrophilic (external) hydroperoxide O-atom, respectively, which facilitates homolysis of the O-O bond. Non-ionic surfactants affect the hydroperoxide decomposition rate due to the H-bond formation [20].

Sodium alkyl sulphate (SDS), which includes saturated hydrocarbon fragments in its molecule, is the best known and the least reactive in radical-chain oxidation processes, was used as a microaggregation initiator.

The aim of the present work is investigation of sodium dodecyl sulfate (SDS) action on ethylbenzene-hydroperoxide decomposition.

EXPERIMENTAL PART

Oxidation of ethylbenzene was performed at 120 °C in a barbotage reactor with a reverse cooler in a stream of air or oxygen, decomposition of hydroperoxide – at 90 °C in a stream of ultra-pure nitrogen or air (1.6 l/h). Chlorobenzene (industrial) solvent was purified by the conventional method [19]. The ethylbenzenhydroperoxide was diluted 10 times with chlorobenzene and dehydrated with sodium sulphate before adding it to the reaction mixture. The DDS was recrystallised twice from ethanol. An ultrasonic dispersant was used to dissolve the surfactant in chlorobenzene. Concentration of peroxides was determined by iodometric determination. The rate of oxygen absorption was measured on the gas-metric device at the constant pressure of O₂ equal to 0.1 MPa.

Products of ethylbenzene oxidation and hydroperoxide decomposition were analyzed by chromatomass-spectrometry (GC-MS). Conditions of analysis were similar [3].

Ethylbenzene (JSC Nizhnekamskneftehim) was purified by the conventional method [11]. Sodium dodecyl sulfate (Serva) was used without additional purification and distilled in vacuum at 100 °C to get rid of crystallization water.

RESULTS AND DISCUSSION

The influence of the nature of surfactants on the hydrocarbon oxidation processes is studied in details by the example of ethylbenzene oxidation. The kinetics and mechanism of oxidation of ethylbenzene is sufficiently well studied in a wide range of temperatures in the presence of various inhibitors and catalysts, that allows us to consider this reaction as one of the basic models in the theory of liquid-phase oxidation [13-15]. The auto-oxidation of ethylbenzene at elevated temperatures develops with an auto-acceleration due to an increase in the chain

initiation rate (W_i) at the decomposition of the formed ethylbenzenhydroperoxideto free radicals. In the presence of cationic surfactant cetyltrimethylammonium bromide (CTAB), the initial rate of ROOH accumulation increases and the maximum concentration of hydroperoxideis reached relatively quickly. In the presence of DDS ROOH accumulation is completely inhibited and the oxidation process is inhibited, although in special experiments it is shown that DDS is not an acceptor of peroxyradicals.

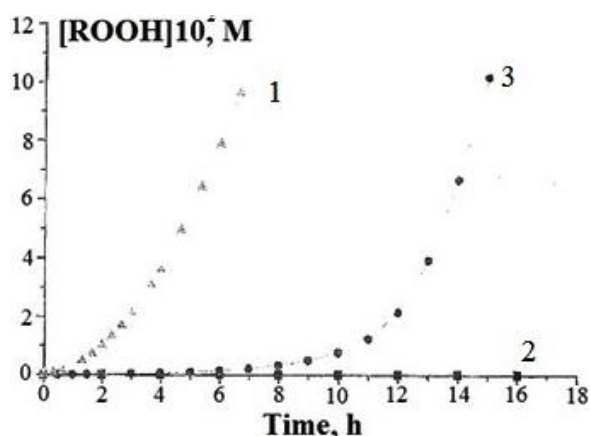


Fig. 1. Auto-oxidation of ethylbenzene without additives (1), in the presence of 1 mM DDS (2) and 0.1 mM ionol (3) at 120 °C
Рис. 1. Автоокисление этилбензола без добавок (1), в присутствии 1 mM ДДС (2) и 0,1 mM ионола (3) при 120 °C

In the presence of DDS the decomposition of ethylbenzenehydroperoxide produces almost equal amounts of phenol and acetone. The high decomposition rate and the set of decomposition products unexpected for ethylbenzenehydroperoxide in the presence of DDS stimulated a more detailed study of the kinetic features of this reaction. The kinetic curves of ethylbenzenehydroperoxide decomposition in the presence of different amounts of DDS (Fig. 2a) have S-shaped form typical for autocatalytic processes. With increasing amounts of DDS at constant initial concentration of ethylbenzenehydroperoxide, the induction period shortens, and maximum rate of decomposition (W_{max}) increases. The dependence of W_{max} on the amount of DDS is nonlinear (Fig. 2b).

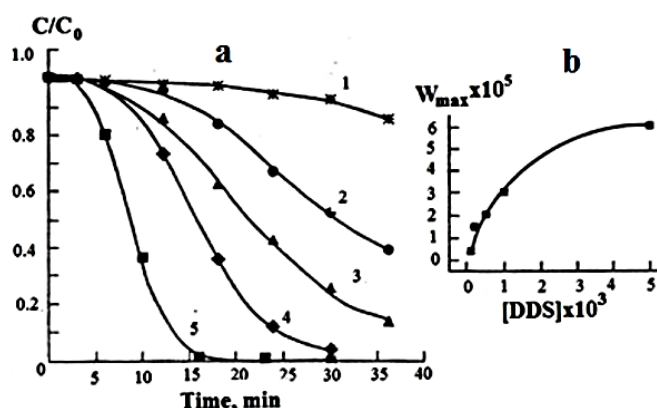


Fig. 2. a-kinetic curves of α -phenethyl consumption (4.5×10^{-2} M) at different contents of DDS. $[DDS] \times 10^{-2}$ M: 0.1-1; 0.2-2; 0.5-3; 1.0-4; 5.0-5; chlorobenzene. б-dependence of maximum rate of hydroperoxide decay on DDS concentration

Рис. 2. а-кинетические кривые расходования α -фенилэтила ($4,5 \times 10^{-2}$ M) при разном содержании ДДС. $[DDS] \times 10^{-2}$ M: 0,1-1; 0,2-2; 0,5-3; 1,0-4; 5,0-5; хлорбензол, б-зависимость максимальной скорости распада гидропероксида от концентрации ДДС

Table 1

Dependence of DDS on the reaction rate

Таблица 1. Зависимость ДДС от скорости реакции

$[DDS] \cdot 10^3$	$W_{max} \cdot 10^5$
0.1	0.3
0.4	1.7
0.5	2
1	3
2	4
2.8	4.5
3	4.6
4	5
4.5	5.1
4.7	5.15
5.3	5.2

It should be noted that DDS is poorly soluble in hydrocarbons and in chlorobenzene. After long-term incubation in a mixture of chlorobenzene and DDS in an ultrasonic dispersant at room temperature, a slightly opalescent stable microdispersion is obtained. We were not able to determine the size of the formed DDS aggregates by quasi-elastic light scattering, which gives good results in the study of microaggregates formed when dissolving *n*-heptane-bis-(2ethylhexyl) sodium sulfosuccinate (AOT) in hydrocarbon. However, a number of experimental factors, namely the influence of hydrocarbon chain length in sodium alkyl sulphates on dodecane oxidation rate and inertness of Na_2SO_4 dispersion in hydroperoxide decomposition reactions, suggest that the catalytic effect of DDS on hydroperoxide decomposition is related to its colloidal properties, and decomposition occurs in joint microaggregates formed by DDS and ROOH [18-20].

The detected features of ethylbenzenehydroperoxide decomposition in the presence of DDS explain the unusual fact. It may be supposed that in joint aggregates of anionic DDS and ROOH due to stabilization of RO^+ cation and further carboxyathion Ph^+ by DDS anions, facilitates heterolytic decomposition of ROOH with phenol formation.

It should be noted that formation of phenols at decomposition of alkylaromatic ROOH is usually connected with strong acids. In the case of DDS, the driving force of ROOH decomposition seems to be the successful orientation of ROOH molecules in the microaggregates formed by anionic surfactants.

The action of anionic surfactant DDS on ethylbenzene oxidation is radically different from the action of cationic surfactant cetyltrimethylammonium bromide (DTAB) [3]. Already with small additions of DDS the oxidation process is completely inhibited. The decomposition rate in a nitrogen atmosphere increases considerably in the presence of DDS (1 mM).

DDS increases the effective decomposition constant of ROOH at 90 °C (Fig. 3). A new portion of ROOH added to the reactor also decomposes at a high rate (Fig. 3, cr. 5). Thus, DDS can be considered as an effective catalyst for the decomposition of ROOH.

The dependence of maximum ROOH decomposition rate on temperature at constant initial concentrations of DDS and ROOH is described by Arrhenius equation with effective activation energy equal to $E_{ef.} = 66.3$ kJ/mol.

In contrast to DTAB in the presence of DDS, the kinetic curves of ROOH decomposition in ethylbenzene medium in an inert atmosphere and in oxygen current practically coincide (Fig. 3).

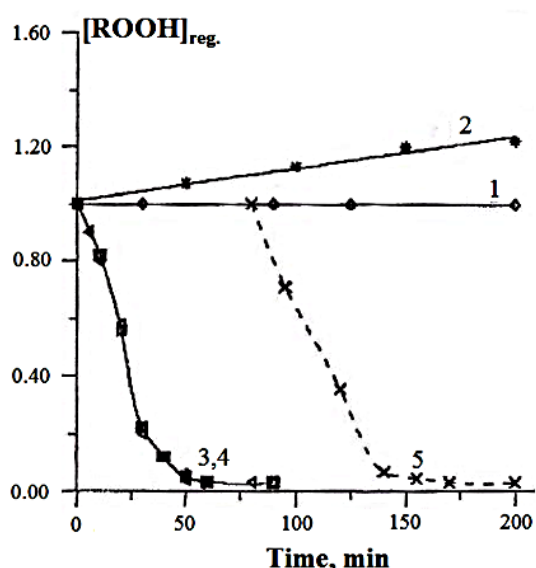
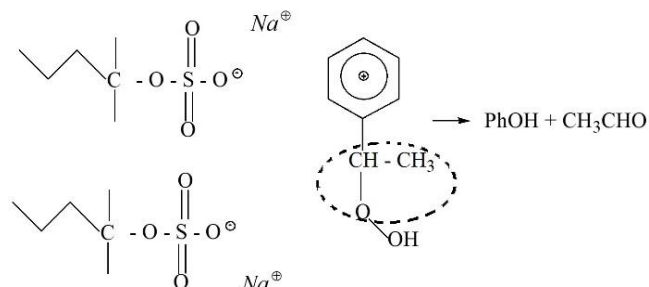


Fig. 3. Effect of DDS on ROOH decomposition in O₂ (2,3) and N₂(1,4,5) atmospheres: 1,2 without dopes; 3,4,5-DDS, 10 mM; 5 addition of new ROOH portion, 60 mM; [ROOH]₀-60 mM; 90 °C

Рис. 3. Влияние ДДС на разложение ROOH в атмосфере O₂ (2, 3) и N₂(1, 4, 5): 1, 2 – без добавок ;3, 4, 5 – ДДС, 10 мМ; 5 -добавка новой порции ROOH, 60 мМ; [ROOH]₀ = 60 мМ; 90 °C

Analysis of ROOH decomposition products in the presence of DDS showed that phenol is the main decomposition product both in N₂ and O₂. The obtained data indicate that the accelerated decomposition of ROOH under the action of DDS occurs heterolytically, without the formation of free radicals. One can suppose that DDS, as cetyltrimethylammoniumbromid, forms in hydrocarbon medium microaggregates of the inverted micelles type, in which ROOH, H₂O and other oxidation products containing hydrophilic groups are concentrated.

According to this scheme, acetaldehyde is formed along with phenol during hydroperoxide decomposition. Acetaldehyde was detected in the exhaust gases entrained by the nitrogen barbotaged through the reaction mixture.



It should be noted that the formation of phenol during decomposition of alkylaromatic hydroperoxides is usually associated with catalysis by strong acids. In case of DDS, apparently, the driving force of ROOH decomposition is successful orientation of hydroperoxide molecules in microaggregates formed by anionic surfactant and stabilization of state with charge transfer in electrostatic fields of polar micelle core.

In case of ethylbenzene and, apparently, other alkylaromatic hydroperoxides in the presence of DDS in the destruction of the respective hydroperoxide phenol is formed which is a free radical acceptor and belongs to the inhibitors of antiradical action [20]. Thus, during the oxidation of alkylaromatic hydrocarbons with DDS addition, a peculiar autosynergism of inhibitory action of surfactant and oxidizing hydrocarbon takes place: DDS destroys hydroperoxides radically with the formation of phenol, which results in the appearance of a complex action antioxidant – peroxiracil acceptor and radical-free hydroperoxide destroyer in the system.

The data obtained show that factors influencing the microstructure of the medium influence the mechanism and kinetics of hydrocarbon oxidation and the effectiveness of inhibitors in particular [23-25]. Possibly many previously known effects, in particular the effect of alcohols and organic acids on ROOH decomposition, inhibitor and catalyst efficiency are largely related to the microheterogeneity of the oxidizing systems, either initially or acquired during oxidation.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

The authors declare the absence a conflict of interest warranting disclosure in this article.

Table 2

Dependence of ROOH on time

Таблица 2. Зависимость ROOH от времени

Time, min.	[ROOH] _{reg.}	Time, min	[ROOH] _{reg.}	Time, min	[ROOH] _{reg.}	Time, min	[ROOH] _{reg.}	Time, min	[ROOH] _{reg.}
0	1	0	1	0	1	0	1	80	1
30	1	50	1.07	4	0.9	4	0.9	100	0.7
90	1	100	1.13	9	0.8	9	0.8	120	0.4
125	1	150	1.18	18	0.58	18	0.58	140	0.1
200	1	200	1.22	31	0.22	31	0.22	155	0.05
				40	0.1	40	0.1	170	0.04
				50	0.03	50	0.03	200	0.02
				60	0.01	60	0.01		
				80	0.01	80	0.01		
				90	0.01	90	0.01		

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