V.S. Dutka [a], Ya.P. Kovalskyi [a], G.G. Midyana [b]

ADSORPTION OF PEROXIDES ON AEROSILS AND MAGNESIUM OXIDE

[a] Ivan Franko National University of Lviv, Lviv, Ukraine
[b] Department of Physical-Chemistry of Fossil Fuels of the Institute of Physical-Organic Chemistry and Coal Chemistry named after L.M. Lytvynenko of the National Academy of Science of Ukraine, Lviv, Ukraine

The adsorption of oligomeric peroxide of sebacic acid and benzoyl peroxide on aerosils and magnesium oxide is studied. The parameters of the adsorption processes are determined. It is shown that the adsorption occurs via formation of hydrogen bonds between surface OH-groups of adsorbents and peroxide groups. The adsorption of benzoyl peroxide on the surface of aerosils and magnesium oxide is low. The thermal decomposition of oligomeric peroxide of sebacic acid and benzoyl peroxide in the presence of aerosil is investigated. The thermal decomposition of oligomeric peroxide of sebacic acid in the presence of aerosils is faster than in a homogeneous system. The corresponding activation energy increases as the aerosil content in the system decreases. In contrast to oligomeric peroxide of sebacic acid, aerosils has no effect on the thermal decomposition rate of the benzoyl peroxide. The polymerization of styrene in the presence of aerosil leads to the formation of a mineral-polymer composite. An increase in aerosil content in the polymerization system leads to a decrease in the rate of polymerization of styrene.

Keywords: oligomeric peroxide, adsorption, reactivity, thermal decomposition, polymerization.

Introduction

The polymerization of vinyl monomers in the presence of highly dispersed minerals is an effective method of producing composite polymer-mineral composites. The presence of a highly developed active surface due to selective adsorption can significantly alter the nature of the polymerization process and affect the molecular weights and molecular weight distribution of the polymers formed under these conditions. Oligomeric peroxide compounds are effective initiators of radical polymerization [1,2]. Initiators of this type can be used to obtain polymer-mineral composites, treat the surfaces of various natures, and modify polymeric materials. The adsorption of both monomers and initiators on the surface will affect the processes occurring with oligomeric peroxides in the presence of dispersed minerals.

The introduction of dispersed oxides Fe_2O_3, Cr_2O_3 and V_2O_5 into the polymerization mixture affects the rate of thermal release of initiators of radical polymerization [3]. The destruction of

peroxide occurs both in a homogeneous solution and on the surface of the investigated disperse oxides. The oxidative-donor reactions, peroxide−surface or initiator heterolysis, are possible because of the nature of the surface. The rate of polymerization decreases in the heterogeneous decomposition of peroxide on the surface of a dispersed mineral in comparison with the homogeneous system. The oxidation-reduction interaction between peroxide and the surface of a dispersed mineral leads to an increase in the rate of the polymerization process. Apart from the effect on the rate of thermal destruction of the peroxide initiator, the nature of a dispersed oxide, affects the growth and breakdown of the polymer chains. It is likely to indicate the features that will be manifest for aerosol and magnesium oxide. Aerosil and magnesium oxide are often used to produce polymer-mineral composites having unique properties [4]. When the mineral is added to the polymerization system, the adsorption of both the monomer and the peroxide initiator is observed. This feature affects both the basic parameters of polymerization and the

molecular characteristics of polymers, which are formed in the filled systems. Aerosil and dispersed oxides Fe_2O_3, Cr_2O_3, V_2O_5, PbO and natural mineral clinoptilolite can affect both the rate of the polymerization process and the thermal decomposition of the peroxide initiator used in the filled systems [3−5]. The dispersed minerals influence molecular weights and molecular weight distribution of polymers, which are formed in their presence [6]. In the presence of dispersed minerals, a linear breakdown of the growing polymeric radicals with the participation of surface groups of the filler is observed. Modification of the surface of the dispersed oxide TiO_2 changes the rate of polymerization and affects the rate constant of the thermal decomposition of peroxide. In the course of polymerization in the presence of dispersing minerals, grafting of macromolecules of polymer to the surface of the filler is observed. The degree of grafting of macromolecules to the surface of the minerals depends on the nature of the filler and the initiator of radical polymerization. Consequently, the nature of the surface of dispersing minerals affects the course of the polymerization process and the properties of polymer-mineral composites, in which the adsorption of components will play an important role.

Thus, this work was aimed at studying the adsorption of oligomeric peroxide of sebacic acid and benzoyl peroxide on aerosils and magnesium oxide, characterizing the thermal decomposition of these peroxides in the presence of aerosil and determining the effect of aerosil on polymerization of styrene.

Experimental

In our work, different samples of aerosil were studied as adsorbents: Aerosil A-175; aerosil modified with alumina (AlA); aerosil modified with titanium oxide (TiA) and aerosil in which a part of the surface OH-groups is replaced by methoxyl groups (MeA). The specific surface of the studied samples was determined by low-temperature adsorption of nitrogen, it was equal to 190 m²/g, 170 m²/g, 120 m²/g and 190 m²/g for A-175, AlA, TiA and MeA, respectively. The size of the filler particles was in the range of 10−40 nm and the bulk density was 40−60 g/l. The content of aluminum oxide in AlA was 3.5%. The content of titanium oxide in TiA samples was 40.3%. Methyl aerosil contained 4.0% methoxyl groups. Magnesium oxide (MERCS) (with a specific surface area of 40 m²/g) was used in adsorption experiments. Measurements of the adsorption of sebacic acid (OSA) on aerosils and magnesium oxide (MgO) were carried out in a solution of styrene. Some of the adsorption experiments on OSA were performed in solutions of dioxane, benzene, dimethylformamide (DMFA) and chloroform. Adsorption measurements were carried out in the temperature range of 298−318 K. In an ampoule containing a certain amount of aerosil, a solution of OSA of known concentration was added, purged with argon and sealed. During the experiment, the ampoules were continuously rotated. After that, the ampoules were removed from the thermostat, opened, and centrifuged. The content of peroxide groups in styrene was determined by the iodometric method [7].

The oligomeric peroxide of sebacic acid was synthesized by the reaction of polycondensation on the phase's interface of the corresponding sebacic acid dichloride and sodium peroxide [8]. The degree of the polycondensation of oligomeric peroxide was within the range of 9−20. Benzoyl peroxide (BP) was purified by a standard procedure. The adsorption experiments were carried out in a solution of styrene, which was released from the inhibitor and was fractionated before adsorption experiments. All organic solvents that were used in the work were thoroughly cleaned and fractionated in an argon atmosphere [9,10].

The thermal decomposition OSA and BP in organic solvents were investigated using an ampoule method at the temperatures of 338−353 K. A solution of peroxide in the solvent of a certain concentration was placed in a clean and dry ampoule. The ampoule was purged with argon and sealed. The ampoules were placed in a thermostat and the beginning of the reaction was fixed. The experiments on the thermal decomposition of peroxides were conducted in the temperature range of 338−353 K. At certain intervals, the ampoules were removed from the thermostat and abruptly cooled, and then the concentration of peroxide, which was not decomposed, was determined by the iodometric method [7]. The error in determining the constants of the thermal decomposition of peroxides did not exceed 3%. The polymerization rate of vinyl monomers in a homogeneous system and in the presence of aerosils was determined dilatometrically at 348 K. The error in determining the polymerization rate did not exceed 3%.

Results and discussion

The adsorption of oligomeric peroxide of sebacic acid on aerosil and magnesium oxide was studied. Adsorption of OSA on aerosils and MgO is well described by the well-known Langmuir equation. Figures 1 and 2 show the adsorption isotherms of OSA on aerosil A-175 and magnesium oxide at various temperatures.
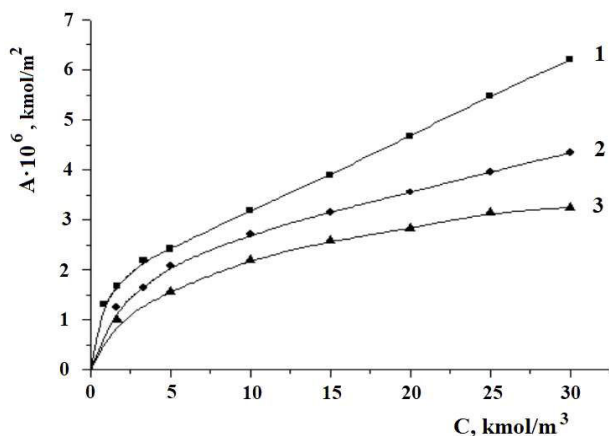
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Fig. 1. Adsorption isotherms of OSA on aerosil A-175. Solvent − styrene. Temperature, K: 1 − 308; 2 − 313; and 3 − 318

Fig. 3. The dependence 1/A vs. 1/C for the adsorption of OSA on aerosil A-175. Solvent − styrene. Temperature, K: 1 − 318; 2 − 313; and 3 − 308

Fig. 2. Adsorption isotherms of OSA on MgO. Solvent − styrene. Temperature, K: 1 − 308; 2 − 313; and 3 − 318
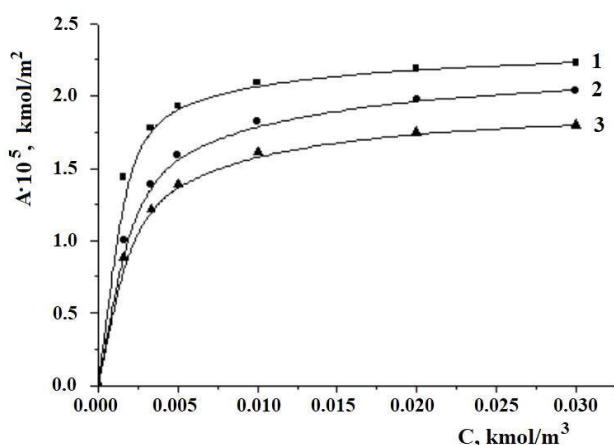

Fig. 4. The dependence 1/A vs. 1/C for the adsorption of OSA on MgO. Solvent − styrene. Temperature, K: 1 − 318; 2 − 313; and 3 − 308

Thus, adsorption of OSA on aerosil and MgO can be described by the following equation:

$$A = A_{\infty}KC/(1+KC), \qquad (1)$$

where K is the constant of the adsorption-desorption equilibrium, A is the adsorption, A_{∞} is the maximum adsorption, and C is the equilibrium concentration.

The Langmuir equation (1) can be transformed into a linear form in the following way:

$$1/A = 1/A_{\infty} + 1/A_{\infty}KC. \qquad (2)$$

Thus, by plotting 1/A vs. 1/C dependences (Figs. 3 and 4), it is possible to determine the maximum adsorption and the adsorption-desorption equilibrium constant.
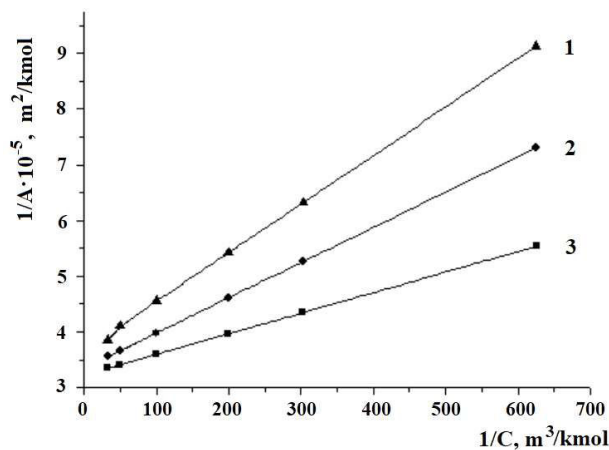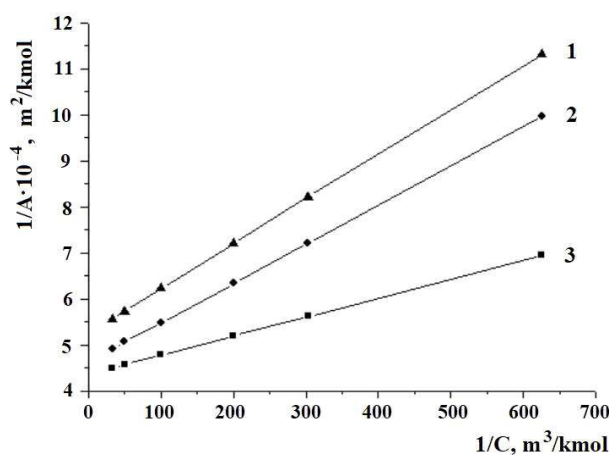
The dependences shown in Figs. 1 and 2 are well aligned in 1/A vs. 1/C coordinates (Figs. 3 and 4), which may be an argument for the conclusion that OSA adsorption on aerosil and magnesium oxide is described by the Langmuir equation and is monomolecular. The adsorption isotherms for modified aerosils (AlA, TiA and MeA) are of a similar nature. The replacement of styrene with other organic solvents also does not change the nature of the adsorption isotherms.

The obtained results show that the OSA adsorption decreases as the temperature increases. Using Eq. (2) and the adsorption results, we calculated the maximum adsorption A_{∞} and the adsorption-desorption equilibrium constants K (Table 1). In addition, the surface area occupied by one elementary element of OSA on the surface of

Parameters of OSA adsorption on the surfaces of aerosils and MgO

Solvent	Adsorbent	T, K	$A_\infty \cdot 10^6$, mol/m²	$S_a \cdot 10^{20}$, m²	K_p, m³/kmol	$-\Delta G$, kJ/mol	$-\Delta H$, kJ/mol	$-\Delta S$, J/mol·K
Styrene	A-175	308	3.10	53	871	17.3		
Styrene	A-175	313	2.99	56	529	16.3	50.2	107
Styrene	A-175	318	2.74	61	421	16.0		
Styrene	AlA	313	1.99	84	576	16.5	–	–
Styrene	TiA	313	1.14	146	221	14.0	–	–
Styrene	McA	313	0.29	572	28	8.76	–	–
Benzene	A-175	298	3.86	43	99.4	11.2	–	–
Chloroform	A-175	298	3.71	45	37.1	8.95	–	–
DMFA	A-175	298	1.28	130	27.9	8.25	–	–
Dioxane	A-175	298	1.07	155	24.9	7.96	–	–
Styrene	MgO	308	22.9	13.7	1059	17.8		
Styrene	MgO	313	21.6	13.0	543	17.2	55.1	121
Styrene	MgO	318	19.1	11.5	533	17.0		

the adsorbent (S_a) was calculated by the formula:

$$S = 1/A_\infty N_A, \tag{3}$$

where N_A is the Avogadro number.

It is worth noting that the patterns of OSA adsorption on aerosils and magnesium oxide are similar, but the surface areas occupied by the elemental element of the peroxide on the surface are very different. Under the same conditions, the adsorption of BP on the surface of aerosil and magnesium oxide is smaller.

The physicochemical parameters (ΔG, ΔH and ΔS) of the adsorption process were calculated by the temperature dependence of the adsorption constants (Table 1). The thermodynamic parameters of the adsorption processes on the surface of aerosil and magnesium oxide are similar. The area of the peroxide layer on the surface of adsorbents is different $(61-53) \cdot 10^{-20}$ m² and $(13.7-11.5) \cdot 10^{-20}$ m² for aerosil and MgO, respectively. The different values of S_a on the surface of aerosil and magnesium oxide may indicate the different orientation of macromolecules OSA in the adsorption layer. Adsorption of OSA occurs due to the formation of hydrogen bonds between peroxide groups and OH-groups on the surface of the aerosil and MgO. For methyl-aerosil, the number of OH-groups on their surface is low and the OSA adsorption is insignificant (Table 1).

The adsorption of peroxide on the surface of aerosils and magnesium oxide are similar. The adsorption of peroxide groups occurs due to the formation of hydrogen bonds with superficial OH-groups of aerosil or magnesium oxide.

The replacement of styrene by other organic solvents leads to a change in the parameters of the adsorption process (Table 1). For benzene and chloroform, the values of A_∞ and S_a are close to the corresponding values observed for styrene solutions. For dioxane and DMFA, these parameters are different (Table 1). A significant decrease in A_∞ and an increase in S_a for OSA indicate competitive adsorption of peroxide solvents on the aerosil surface. Dioxane and DMFA can form hydrogen bonds with OH-groups of aerosil.

The results of the study of thermal decomposition of OSA and BP in styrene are shown in Table 2. In a homogeneous system, changing the concentration of OSA within $0.01-0.05$ mol/l does not change the rate constants of the thermolysis and the activation energy of the process (E_a). A value $E_a=131.4$ kJ/mol indicates a radical mechanism of peroxide decomposition. The study of the rate of thermal decomposition in the presence of 2 wt.% of A-175 reveals an increase in effective rate constants (k) in the presence of an adsorbent. At low OSA concentrations in the reaction medium, the values of k are higher than the corresponding values at larger OSA concentrations. The activation energy of the decomposition process is equal to 104.6 kJ/mol for the concentration of OSA 0.01 mol/l, while it increases to 118.0 kJ/mol for the concentration of 0.05 mol/l. Increasing A-175 content to 4% results in an increase in k of more than 50% for an OSA concentration of 0.01 mol/l. The E_a value increases from 95.0 kJ/mol for OSA concentration of 0.01 mol/l to 109.6 kJ/mol for 0.05 mol/l.

The results obtained indicate that the adsorbed OSA molecules on the surface of the aerosil are decomposed via a heterolytic mechanism.

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Table 2

Effective rate constants of thermal decomposition of OSA and BP in the presence of A-175 and MgO

Adsorbent	Content, wt%	C_o, mol/l	$k \cdot 10^5$, s^{-1}				E_a, kJ/mol
			338 K	343 K	348 K	353 K	
OSA							
–	0	0.010	2.75	5.70	10.30	19.6	131.4
–	0	0.025	2.78	5.12	10.00	19.7	131.4
–	0	0.050	2.75	5.20	10.00	19.7	131.4
A-175	2	0.010	7.30	12.7	19.40	36.7	104.6
A-175	2	0.025	4.80	8.80	15.30	27.0	113.0
A-175	2	0.050	4.00	7.27	12.80	23.7	118.0
A-175	4	0.010	11.6	19.3	30.80	49.8	95.0
A-175	4	0.025	7.00	12.5	20.40	34.8	104.6
A-175	4	0.050	5.40	9.00	15.80	28.0	109.6
BP							
–	0	0.025	–	1.35	2.46	5.39	137.0
A-175	2	0.025	–	1.34	2.48	5.38	137.0
A-175	4	0.025	–	1.35	2.47	5.39	137.0
MgO	2	0.025	–	1.34	2.47	5.37	137.0
MgO	4	0.025	–	1.35	2.46	5.39	137.0

Table 3

The rate of styrene polymerization (W) in the presence of A-175 at different OSA concentrations at 348 K

Content, wt.%	$W \cdot 10^4$, (mol/l·s)							n
	[OSA]$\cdot 10^2$, mol/l							
	1.25	1.50	2.00	2.50	3.00	4.00	5.00	
0	2.33	2.60	3.21	3.39	3.66	4.52	4.68	0.50
4	1.96	2.47	2.71	3.05	3.56	4.02	–	0.52

The rate of benzoyl peroxide decomposition is the same for a homogeneous and heterogeneous system (Table 2). The activation energy of the BP decomposition process in the homogeneous system and in the presence of aerosil and magnesium oxide is the same and equal to 137.0 kJ/mol. This fact confirms the conclusion about poor adsorption of BP on the surface of the adsorbents under study. Tokareva et al. [11] observed the influence of silica surface on the thermostability of peroxide groups. Voronov and Samaryk [12] noted the important role of oligomeric peroxides in the reaction at the phase boundary.

The results of the study of the polymerization rate of styrene in the presence of aerosil at different concentrations are shown in Table 3. The reaction order of the polymerization reaction with respect to the initiator (n) was calculated using the obtained data. In a homogeneous solution, the value of n corresponds to its theoretical value (0.5). In the presence of aerosil, the reaction order with respect to the initiator increases, which indicates an increase in the proportion of linear break in the total reaction. Introduction A-175 in the polymerization system

leads to a decrease in the rate of polymerization.

A decrease in the rate of styrene polymerization when introducing aerosil into the polymerization system can be explained by a decrease in the OSA concentration in the styrene solution due to adsorption. Along with a decrease in the rate of polymerization of styrene in the presence of A-175, the reaction order of the polymerization with respect to the initiator increases. A small increase in the value of n can be explained by the involvement of the A-175 surface in the reaction of linear breakage of the polymer chains.

Conclusions

The process of adsorption of oligomeric peroxides on the surface of aerosil and magnesium oxide is well described by the Langmuir equation for monomolecular adsorption. The surface areas that fall on the peroxide link in the adsorption layer are 61−53 Å^2 and 13.7−11.5 Å^2 for the aerosil and magnesium oxide, respectively. This feature indicates the different orientation of the oligomer peroxide in the monolayer. The thermodynamic parameters of the adsorption process for aerosil and magnesium oxide are similar, indicating the similarity of both

processes. The thermodynamic parameters ΔG, ΔH and ΔS of the adsorption process are calculated based on the temperature dependence of adsorption-desorption equilibrium constants. Adsorption of oligomeric peroxides on aerosil and magnesium oxide occurs due to the formation of hydrogen bonds between the surface OH-groups of the adsorbent and peroxide groups. The OSA decomposition on the SiO_2 surface occurs with a higher rate and less activation energy than in homogeneous systems. The SiO_2 surface inhibits the styrene polymerization as compared to unfilled systems. In addition to the effect on the rate of peroxides destruction, the surface affects the interface of the polymer chains.

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АДСОРБЦІЯ ПЕРОКСИДІВ НА АЕРОСИЛАХ І МАГНІЙ ОКСИДІ

В.С. Дутка, Я.П. Ковальський, Г.Г. Мідяна

Вивчено адсорбцію олігомерного пероксиду себацинової кислоти та пероксиду бензоїлу на аеросилах і магній оксиді. Знайдено параметри адсорбційних процесів. Показано, що адсорбція перебігає за рахунок формування водневих зв'язків між поверхневими ОН-групами адсорбентів та пероксидними групами. Адсорбція пероксиду бензоїлу на поверхні аеросилів та магній оксиді є невеликою. Досліджено термічне розкладання олігомерного пероксиду себацинової кислоти та пероксиду бензоїлу за присутності аеросилу. Термічне розкладання олігомерного пероксиду себацинової кислоти за наявності аеросилу перебігає швидше, ніж в гомогенній системі. Відповідні енергії активації зростають при зменшенні вмісту аеросилу в системі. На противагу олігомерного пероксиду себацинової кислоти, аеросил не впливає на швидкість термічного розкладу пероксиду бензоїлу. При полімеризації стирену за наявності аеросилу формуються полімер-мінеральні композити. Збільшення вмісту аеросилу у полімеризаційній системі призводить до зменшення швидкості полімеризації стирену.

Ключові слова: олігомерні пероксиди, адсорбція, реакційна здатність, термічний розклад, полімеризація.

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*V.S. Dutka [a, *], Ya.P. Kovalskyi [a], G.G. Midyana [b]*

[a] Ivan Franko National University of Lviv, Lviv, Ukraine

[b] Department of Physical Chemistry of Fossil Fuels of the Institute of Physical-Organic Chemistry and Coal Chemistry named after L.M. Lytvynenko of the National Academy of Science of Ukraine, Lviv, Ukraine

[*] e-mail: vdutka@ukr.net

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