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Kinetic Analysis of Aniline Polymerization Considering the Formation of a Charge-Transfer Complex

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Abstract—Kinetic features of oxidative polymerization of aniline hydrochloride in aqueous media have been analyzed under the assumption of formation of a charge-transfer complex between aniline and quinonediimine fragments of aniline *n*-mers. The total reaction rate order of the catalytic stage can be between unity and two. The reaction rate order of the catalytic stage in the presence of polyvinyl alcohol is changed from unity to two at heating. Rate constants of the single-electron transfer and the complex formation are independent of the concentration and molecular mass of polyvinyl alcohol.

Keywords: aniline, polymerization, mechanism, kinetics, autocatalysis, heterogeneous reaction

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Pursuit of the methods of controlled synthesis and processing of polyaniline and its derivatives has constituted a significant part of studies in the field of conducting polymers over recent three decades. The importance of these problems is due to a set of special features of polyanilines allowing development of amperometric sensors, gas-separating membranes, and antistatic and corrosion-inhibiting coatings based on these polymers; moreover, they are promising for molecular electronics and radio engineering applications [1–3]. Despite the evident progress in practical application of the conjugated conducting polymeric aromatic amines, the development of theoretical ideas on the synthesis of the mentioned materials is still well behind the requirements of experimentalists, thus limiting the possibilities of targeted control of the products properties. Indeed, a commonly accepted concept of mechanism and kinetics of oxidative polymerization has been missing so far, whereas the models discussed in the available literature [4–11] are contradictory. On top of that, study of mechanism and kinetics of oxidative polymerization of anilines is of fundamental interest in view of generalization of the knowledge on synthesis of high-molecular compounds.

Polyaniline and its derivatives are commonly prepared via oxidative polymerization of aromatic amines in an aqueous hydrochloric acid solution in the presence of ammonium persulfate (Scheme 1) [1, 3]. Polymers and oligomers of aniline (starting from the tetramer) are insoluble in the reaction mixture and thus form a separate phase in the course of polymerization [4]. Despite the heterogeneous character, the oxidative polymerization of aniline yields the products with molecular mass of up to 500000 Da [3].

Evidently, quantitative description of aniline polymerization should account for its heterogeneous character. Nevertheless, the related studies (for example, [4–7]) have been limited to the formal fitting of the experimental data with one of the general empirical equations (1) and (2):

$$-d[\mathrm{M}]/dt = k_1[\mathrm{m}][\mathrm{Ox}] + k_2([\mathrm{M}]_0 - [\mathrm{M}])[\mathrm{M}], \quad (1)$$

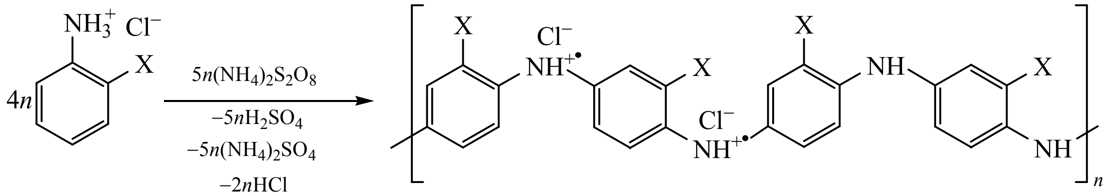
$$-d[\mathrm{M}]/dt = k_1[\mathrm{m}][\mathrm{Ox}] + k_2([\mathrm{M}]_0 - [\mathrm{M}]), \quad (2)$$

where k_1 and k_2 are the rate constants; [M] and [m] are concentrations of the salt and the basic forms of the aromatic amine (monomer), respectively; [Ox] is the oxidizer concentration; and *t* is time.

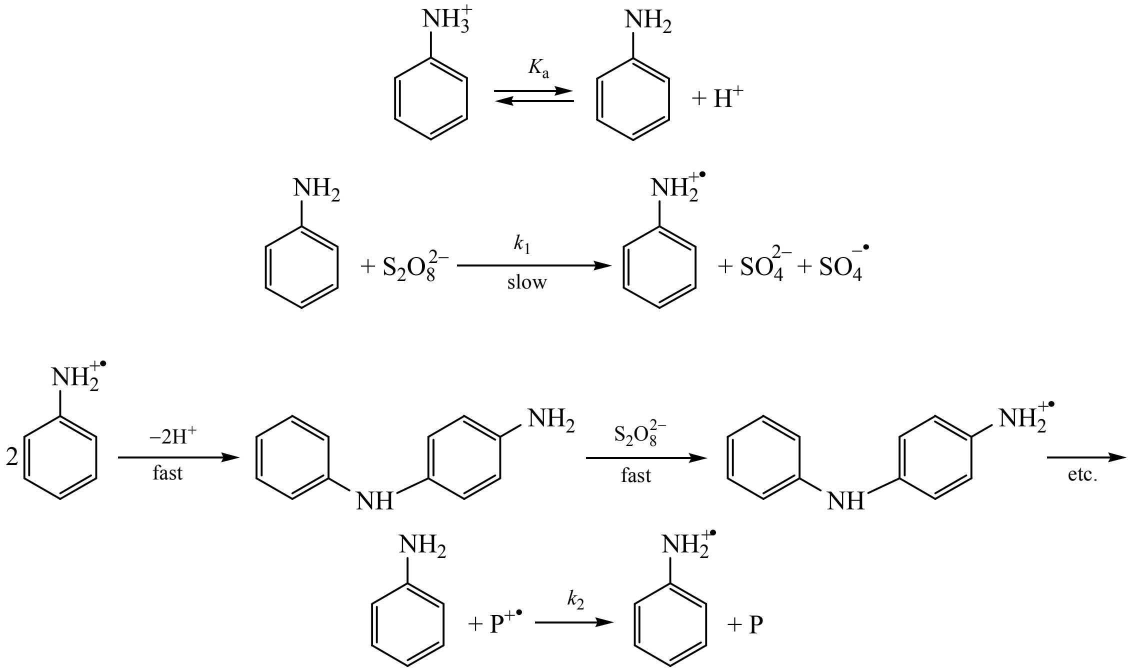
Equations (1), (2) presume the autocatalytic character of oxidative polymerization of aniline; this has been reliably confirmed by the experiments [1–7].

According to the published data, the oxidative polymerization of aniline (Scheme 2) starts with a slow stage of single-electron oxidation of aniline [reaction (2.2)]; only the basic form existing in the equilib-

Scheme 1.



Scheme 2.



(2.1)

(2.2)

(2.3)

(2.4)

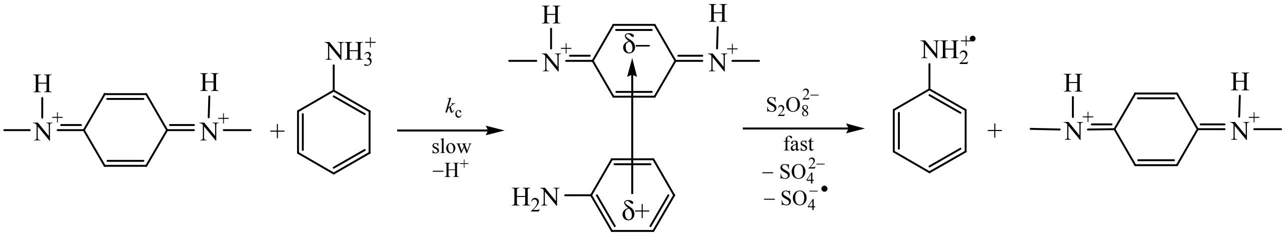
rium with phenylammonium cation (2.1) takes part in the reaction (2.2) [8]. Aniline cation-radicals formed in the stage (2.2) further undergo fast recombination via reaction (2.3) yielding the dimer [6, 7, 9]. The reducing activity of aniline oligomers enhances with the increase in the molecular mass; hence, further processes of their single-electron oxidation can be considered kinetically fast stages [4–7]. It has been suggested that the autocatalytic effect is due to the fast electron transfer from the cation-radicals of aniline oligomers ($P^{+\cdot}$) to the monomer, forming the monomer (aniline) cation-radicals and neutral aniline oligomers (P) [reaction (2.4)] [6, 7].

It is known that the stability of the cation-radicals of aniline *n*-mers is enhances with the increase of the molecular mass due to higher diversity of the resonance structures. In particular, this has been confirmed experimentally by the decrease in the standard electrode potentials of aniline oligomers in the monomer >> dimer > trimer series [4, 5]. Therefore, even though the above-described autocatalysis mechanism formally corresponds to kinetic equation (1), it includes the unfavorable process of formation of cation-radicals of aniline monomer from the more stable cation-radicals of aniline *n*-mers in the fast stage (2.4).

This work considers the general features of mechanism and kinetics of aniline oxidative polymerization in water and in aqueous solutions of polyvinyl alcohol (PVA), the latter acting as stabilizer of the formed polyaniline particles.

Derivation of kinetic equation of aniline oxidative polymerization. It has been demonstrated [10] that qualitative features of oxidative polymerization of aromatic amines can be satisfactorily explained under assumption of sequential stages of slow formation of charge-transfer complex (CTC) between the monomer and quinonediimine fragments of the formed aniline *n*-mers followed by fast oxidation of the CTC with a

Scheme 3.



persulfate anion (Scheme 3). That route is an alternative channel of the active centers formation (in addition to the single-electron transfer) catalyzed by the reaction products.

The described mechanism is different from the one represented by Scheme 2 in that it presumes only the prior polarization of the monomer with its oxidation products, whereas the persulfate ion acts as oxidizer.

Even the lower oligomers of aromatic amines are insoluble in the reaction medium; therefore, the CTC can be formed exclusively in the adsorption layer of aggregates of the oligomer products forming a new phase and acting as adsorbent. Hence, the formation of the CTC at the interphase boundary should be preceded by physical adsorption of the monomer at the surface of aggregates of its *n*-mers. Physical adsorption is generally faster than chemical transformations [11], and we hereafter assume that the adsorption equilibrium between the monomers and aggregates of its *n*-mers is established instantly.

Assuming validity of Scheme 3 and assuming the fast establishment of the adsorption equilibrium following the Langmiur isotherm, we further present the quantitative description of kinetic features of oxidative polymerization of aromatic amines and check the agreement between the derived model and the experimental data.

Let the rate of single-electron transfer from the monomer molecule to the oxidizer ion be w_s, and the rate of complex formation be w_c. As the monomer cation-radicals are formed due to the monomer consumption in the parallel processes of the single-electron transfer and the complex formation, the overall rate of the oxidative polymerization w is a sum of the processes rates (3).

$$w = -d[M]/dt = w_s + w_c. \tag{3}$$

As the basic monomer form is exclusively active in the single-electron transfer, and the process is bimolecular [8], Eq. (4) is held:

$$w_s = k_s[m][Ox], \tag{4}$$

with k_s being the rate constant of the single-electron transfer.

The monomer basic form concentration can be expressed via the salt form concentration according to Eq. (5); its substitution into Eq. (4) gives the following Eq. (6) for the rate of the single-electron transfer:

$$[m] = K_a[M][Ox]/[H^+], \tag{5}$$

$$w_s = K_a k_s[M][Ox]/[H^+], \tag{6}$$

where K_a being the dissociation constant of the acid conjugate to the monomer.

Accounting for the law of surface action, the complex formation rate in a unit volume of the reaction mixtures follows Eq. (7)

$$w_c = k\theta_{eq}S/V = (k\theta_{eq}\cdot 4\pi r^2 N)/V, \tag{7}$$

where k is the rate constant of interaction of the adsorbed monomer with the electron-lacking fragments of the chains; θ_{eq} is the equilibrium degree of the coverage of aggregates of aniline *n*-mers surface with the monomer; S is the total surface area of the interface boundaries in the system; N is the number of aggregates of aniline *n*-mers; r is the average radius of the aggregates (hereinafter the aggregates particles are represented by equivalent spheres); and V is the system volume.

Equation (7) is held true if the complex formation stage is almost irreversible; it is so, for instance, if the CTC oxidation stage is considered to be kinetically fast (Scheme 2).

The total number of the aggregates of aniline *n*-mers in the system follows Eq. (8)

$$N = g/g_a, \tag{8}$$

where g and g_a being the current mass of the formed *n*-mers aggregates and the average mass of a single aggregate, respectively.

Then, taking into consideration the meaning of r defined in Eq. (7), we immediately get Eq. (9):

$$N = \{3([\mathrm{M}]_0 - [\mathrm{M}])VM\}/4\pi dr^3, \tag{9}$$

where d being polyaniline density; M being the monomer molar mass; and $[\mathrm{M}]_0$ being the initial concentration of the monomer (the monomer is in the salt form at $t = 0$).

If the monomer adsorption at the aggregates surface follows the Langmuir isotherm, Eq. (10) is true

$$\theta_{\mathrm{eq}} = K[\mathrm{M}]/(1 + K[\mathrm{M}]), \tag{10}$$

where K being the adsorption equilibrium constant.

Substituting Eq. (7) with Eqs. (9) and (10), we get Eq. (11).

$$w_{\mathrm{c}} = \{3KkM([\mathrm{M}]_0 - [\mathrm{M}])[\mathrm{M}]\}/dr(1 + K[\mathrm{M}]). \tag{11}$$

Let us denote the composite rate constant of the complex formation as $k_{\mathrm{c}} = 3KkM/dr$, then accounting for Eq. (11), the Eq. (3) for the overall rate of the oxidation polymerization can be rewritten in the form (12).

$$w = w_{\mathrm{s}} + \{k_{\mathrm{c}}([\mathrm{M}]_0 - [\mathrm{M}])[\mathrm{M}]\}/(1 + K[\mathrm{M}]). \tag{12}$$

Equation (12) has been derived under assumption that in the course of polymerization the number of disperse phase particles grows, their average size being constant. We assume that Eq. (12) universally describes kinetics of oxidative polymerization of aromatic amines.

The differential Eq. (12) cannot be resolved analytically; however, it can be integrated under additional approximations. In particular, the oxidative polymerization of aromatic amines is accompanied with release of protons steeply slowing down the single-electron transfer at the increased monomer conversion [Eq. (6)]; hence, Eq. (13) follows for the rate of that stage.

$$w_{\mathrm{s}}(t = 0) = w;\ w_{\mathrm{s}}(t > 0) = 0. \tag{13}$$

On top of that, the absolute value of the adsorption equilibrium constant K can hardly be determined experimentally. Let us consider two particular cases.

Case 1. If the constant of adsorption equilibrium of the monomer at the surface of its n-mers aggregates is high ($1 \ll K[\mathrm{M}]$), Eq. (12) is transformed into Eq. (14) with the autocatalytic term of the overall first order.

$$-d[\mathrm{M}]/dt = w_{\mathrm{s0}} + k_{\mathrm{c1}}([\mathrm{M}]_0 - [\mathrm{M}]), \tag{14}$$

where $k_{\mathrm{c1}} = k_{\mathrm{c}}/K$ being first-order rate constant of the complex formation.

Then the first-order activation energy of the complex formation (E_{a1}) can be expressed via the adsorption enthalpy (ΔH_{ads}) and the true activation energy of the complex formation (E_{a}) is as follows [Eq. (15)]:

$$E_{\mathrm{a1}} = E_{\mathrm{a}} - \Delta H_{\mathrm{ads}}. \tag{15}$$

The integration of Eq. (14) accounting for Eq. (13) leads to Eq. (16).

$$\ln([\mathrm{M}]_0 - [\mathrm{M}]) = \ln(w_{\mathrm{so}}/k_{\mathrm{c1}}) + k_{\mathrm{c1}}t. \tag{16}$$

According to Eq. (16), $\ln([\mathrm{M}]_0 - [\mathrm{M}])$ is linear with time, the slope of the corresponding plot being equal to the rate constant of the complex formation k_{c1}, and the rate constant of the single-electron transfer can be calculated from the Y-intercept L via Eq. (17).

$$k_{\mathrm{s}} = k_{\mathrm{c1}}e^{\mathrm{L}}([\mathrm{H}^+]_0/K_{\mathrm{a}}[\mathrm{M}]_0[\mathrm{Ox}]_0). \tag{17}$$

Case 2. If the constant of adsorption equilibrium of the monomer at the surface of its n-mers aggregates is low ($1 \gg K[\mathrm{M}]$), Eq. (12) is transformed into Eq. (18) with the autocatalytic term of the overall second order.

$$-d[\mathrm{M}]/dt = w_{\mathrm{s0}} + k_{\mathrm{c2}}([\mathrm{M}]_0 - [\mathrm{M}])[\mathrm{M}], \tag{18}$$

where $k_{\mathrm{c2}} = k_{\mathrm{c}}$ being the second-order rate constant of the complex formation.

In this case, the second-order activation energy of the complex formation (E_{a2}) equals the true activation energy of the complex formation (E_{a}). Hence, considering Eq. (15) it is possible to estimate the adsorption enthalpy of the monomer on the surface of its n-mers aggregates from the difference between the activation energies of the first and the second order, provided that the reaction temperature increase is accompanied with the change in the rate order of the catalytic stage. The change of the rate order of the catalytic (complex formation) stage is possible because physical adsorption is exothermic, and the constant of adsorption equilibrium K decreases with heating.

Integration of Eq. (18) accounting for Eq. (13) leads to Eqs. (19), (20).

$$\ln\{([\mathrm{M}]_0 - [\mathrm{M}])/[\mathrm{M}]\} = a + k_{\mathrm{c2}}[\mathrm{M}]_0 t, \tag{19}$$

$$a = \ln\left(\frac{\sqrt{[\mathrm{M}]_0^2 - (4ws0/k_{\mathrm{c2}})} - [\mathrm{M}]_0}{\sqrt{[\mathrm{M}]_0{}^2 + (4ws0/k_{\mathrm{c2}})} + [\mathrm{M}]_0}\right). \tag{20}$$

According to Eq. (19), $\ln\{([\mathrm{M}]_0 - [\mathrm{M}])/[\mathrm{M}]\}$ is linear with time, the slope of the corresponding plot being equal to the rate constant of the complex formation k_{c2}, and the rate constant of the single-

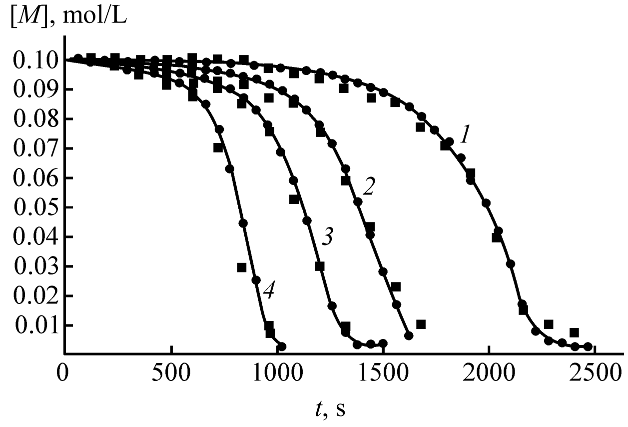


Fig. 1. Kinetic curves of aniline hydrochloride consumption as determined via potentiometry (circles) and absorbance measurements (squares) at (*1*) 15, (*2*) 25, (*3*) 30, and (*4*) 35°C. Initial concentration of aniline hydrochloride was 0.1 mol/L, and that of ammonium persulfate was 0.125 mol/L.

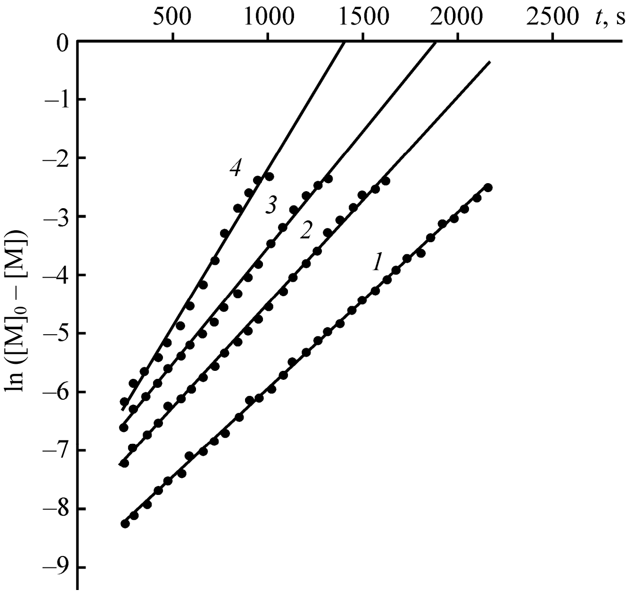


Fig. 2. Kinetic curves of oxidative polymerization of aniline hydrochloride in the coordinates of ln $([M]_0 - [M])$ as a function of time.

electron transfer can be calculated from the Y-intercept *a* via Eq. (21).

$$k_s = \frac{k_{c2}[M]_0[H^+]_0}{K_a[Ox]_0}\left[\frac{e^a}{(1-e^a)^2}\right]. \qquad (21)$$

The kinetic equations derived in this work prove the validity of the commonly used empirical equations (1), (2) [4–7] to describe kinetics of oxidative polymerization of aromatic amines and show the clear physical meaning of the rate constants k_1, and k_2. Moreover, the above-given theoretical analysis may be applied to describe kinetics of other heterogeneous autocatalytic reactions as well.

In particular, from the presented analysis it follows that the kinetic curves of polymerization are linear in the coordinates of either ln $([M]_0 - [M])$ or ln $\{([M]_0 - [M])/[M]\}$ vs. time depending on the value of constant of adsorption equilibrium of the monomer at the surface of its *n*-mers aggregates..

Experimental kinetic curves of aniline hydrochloride polymerization in aqueous medium without PVA added (Fig. 1) were linear in the coordinates of ln $([M]_0 - [M])$ vs. time (Fig. 2). Hence, $1 \ll K[M]$ was true under those conditions up to high monomer conversion, and the kinetic features of the process could be described with Eq. (16) (the overall first rate order of the catalytic stage).

Kinetic parameters of the single stages determined from data in Fig. 2 are shown in Table 1. The tabulated data revealed that the activation energy of the non-catalytic stage of single-electron transfer E_a was noticeably higher than the activation energy of the complex formation E_{a1}, the latter one being responsible for the autocatalytic character of the polymerization.

Oxidation polymerization of aniline and its derivatives is known to yield stable aqueous dispersion of the product in the presence of certain water-soluble polymers (poly-*N*-vinylpyrrolidone, polyethylene glycol, and polyvinyl alcohol) [12–14]. The *n*-mers aggregates are stabilized due to concentrating of the water-soluble polymers segments at the interphase boundary; therefore, the presence of high-molecular stabilizers can affect the kinetic features of the complex formation stage occurring in the adsorption layer.

In the presence of 0.1 mol/L of PVA (50000 Da), kinetic curves of aniline hydrochloride polymerization were linear either in the coordinates of ln $([M]_0 - [M])$ vs. time (*T* 10–25°C, Fig. 3a), or ln $\{([M]_0 - [M])/[M]\}$ vs. time (35–45°C, Fig. 3b), depending of temperature. The kinetic parameters of aniline hydrochloride polymerization in the presence of PVA extracted from the data in Fig. 3 are collected in Table 2.

Change in the reaction rate order of the complex formation stage from unity to two at heating could be ascribed to the decreasing constant of the monomer

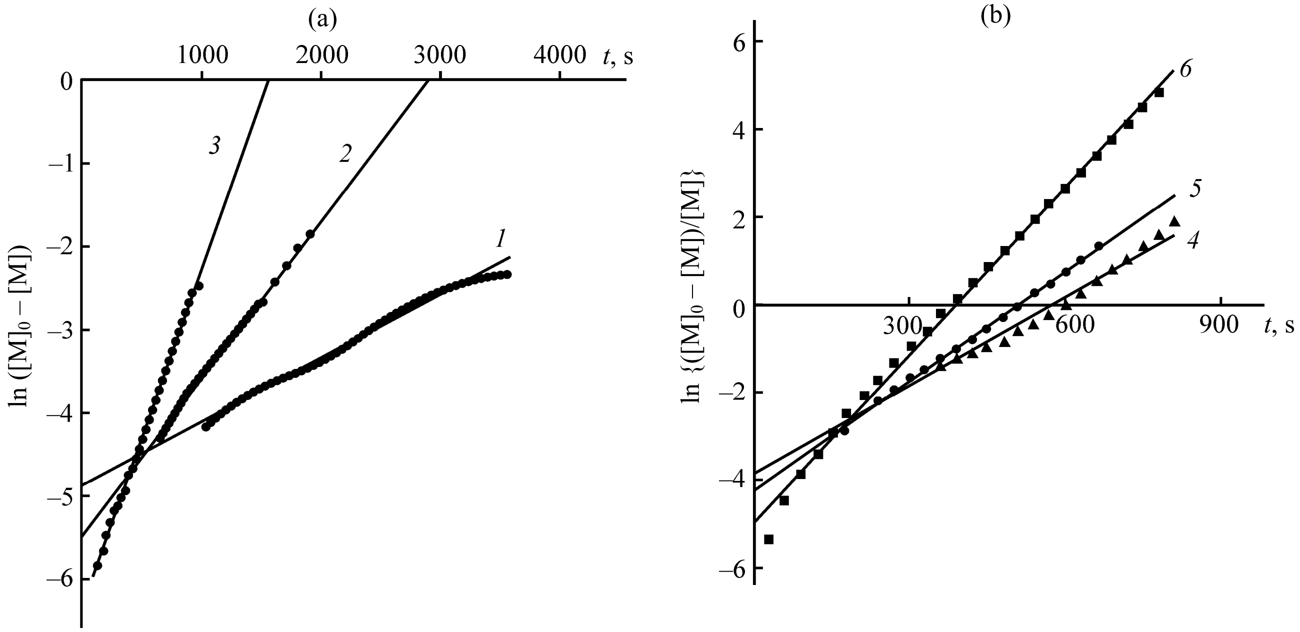


Fig. 3. Kinetic curves of oxidative polymerization of aniline hydrochloride in the presence of PVA (50000 Da, 0.1 mol/L) in the coordinates (a) ln ($[M]_0$ – [M]) and (b) ln {($[M]_0$ – [M])/[M]} as functions of time. Initial concentration of aniline hydrochloride was 0.1 mol/L, and that of ammonium persulfate was 0.125 mol/L. Temperature, °C: (*1*) 10, (*2*) 15, (*3*) 25, (*4*) 30, (*5*) 35, and (*6*) 45.

adsorption equilibrium due to the exothermic character of physical adsorption [11]. The enthaply of adsorption of aniline at the surface of its *n*-mers aggregates stabilized with PVA was 42 kJ/mol [Eq. (15)].

Comparison of the data in Tables 1 and 2 revealed the significant increase in the activation energy of the complex formation stage in the presence of PVA. The aggregates surface (bearing the quinonediimine fragments) was likely screened with PVA chains, thus suppressing the CTC formation in the adsorption layer.

Table 1. Rate constants and activation energies of single-electron transfer and the complex formation stages in the course of aniline hydrochloride oxidative polymerization ($[M]_0$ = 0.1 mol/L) in the presence of ammonium persulfate ($[Ox]_0$ = 0.125 mol/L)

T, °C	15	25	30	35
$k_s \times 10^2$, L mol^{-1} s^{-1}	0.43±0.02	1.30±0.06	2.27±0.11	3.76±0.21
E_a, kJ/mol	81±7			
$k_{c1} \times 10^3$, s^{-1}	2.90±0.14	3.75±0.19	4.15±0.21	4.90±0.27
E_{a1}, kJ/mol	19±3			

On the contrary, activation energy of the single-electron transfer was almost indifferent to the presence of PVA.

The screening of the aggregates surface with PVA chains could reasonably explain the change in the reaction rate order of the complex formation stage as function of PVA concentration at a constant temperature (Fig. 4). Indeed, the first-order kinetics of this stage in the absence of PVA changes to the second-order one at the two-fold molar excess of PVA repeating units with respect to the aniline monomer, and does not change at the further increase of PVA concentration.

The second-order rate constant of the complex formation was practically independent of PVA concentration (0.2–0.4 mol/L) in the reaction mixture, being 0.067±0.005 to 0.085±0.007 L mol^{-1} s^{-1}.

Kinetic studies of oxidative polymerization of aniline hydrochloride in the presence of 0.1 mol/L of PVA of varied molecular mass (8000–150000 Da) demonstrated that the polymer stabilizer molecular mass had no significant effect on the reaction course. In all cases the complex formation stage was of the first order, and the corresponding reaction rate constant was (3.6±0.2) × 10^{-3} to (4.1±0.3) × 10^{-3} s^{-1}, whereas

Table 2. Kinetic parameters of oxidative polymerization of aniline hydrochloride ($[M]_0$ = 0.1 mol/L) in the presence of ammonium persulfate ($[Ox]_0$ = 0.125 mol/L), and PVA (50000 Da, 0.1 mol/L)

T, °C	10	15	25	30	35	45
$k_s \times 10^2$, L mol^{-1} s^{-1}	0.27±0.02	0.43±0.03	1.20±0.07	2.10±0.15	3.30±0.23	8.10±0.61
E_a, kJ/mol	73±5					
$k_{c1} \times 10^3$, s^{-1}	0.85±0.06	1.49±0.10	3.80±0.27	–	–	–
E_{a1}, kJ/mol	70±6					
$k_{c2} \times 10^2$, L mol^{-1} s^{-1}	–	–	–	6.7±0.3	8.3±0.4	12±0.6
E_{a2}, kJ/mol	31±2					

the rate constant of the single-electron transfer was 1.1×10^{-2} to 1.3×10^{-2} L mol^{-1} s^{-1} (25°C).

If the adsorption equilibrium was established slowly, the change of the reaction medium viscosity due to variation of PVA molecular mass and concentration would influence the rate constant of the complex formation. Since no effect of the mentioned parameters was observed experimentally, it confirmed the validity of the assumption of fast establishment of the adsorption equilibrium between the monomer and the aggregates of its *n*-mers. This fact was especially important, because it was used to derive the kinetic model discussed in this contribution.

To conclude, we suggested a model to quantify the kinetics of oxidative polymerization of aniline accounting for its heterogeneous character and pointing to the physical meaning of rate constants in the earlier applied empirical equations. Processing of the experimental data in the frame of the derived model demonstrated the crucial importance of accounting for the monomer adsorption at the surface of its *n*-mers aggregates in order to rationalize the mechanism and features of the oxidative polymerization. The influence of macromolecular stabilizer (PVA) on the process kinetics was as well explained by the suggested model.

EXPERIMENTAL

Aniline hydrochloride (“chemical pure” grade) and ammonium persulfate (“analytical pure” grade) were used without further purification. Degree of hydrolysis of the PVA samples (mass fractions of 8000, 13000, 50000, and 150000) was no less than 98%.

For kinetic experiments on oxidative polymerization, solutions of aniline hydrochloride (50 mL in the 250 mL reactor vessel) and of ammonium persulfate (50 mL) in distilled water were prepared separately. The solutions were kept at the selected temperature during 30 min, the aniline solution being flushed with argon. The oxidizer solution was then added to the aniline solution, the mixing moment was regarded as the reaction start. The reaction was carried out with stirring with a magnetic bar. Each experiment was replicated five times.

The following parameters were monitored in the course of the reaction: temperature (constant within ±0.3°C) and pH (an IPL-311 potentiometer, the accuracy of ±0.005 pH).

Assuming that the oxidative polymerization of aniline was quantitative, the current monomer concentration was calculated using Eq. (22):

$$[M] = [M]_0 \left(1 - \frac{10^{-pH} - [H^+]_0}{[H^+]_\infty - [H^+]_0}\right), \qquad (22)$$

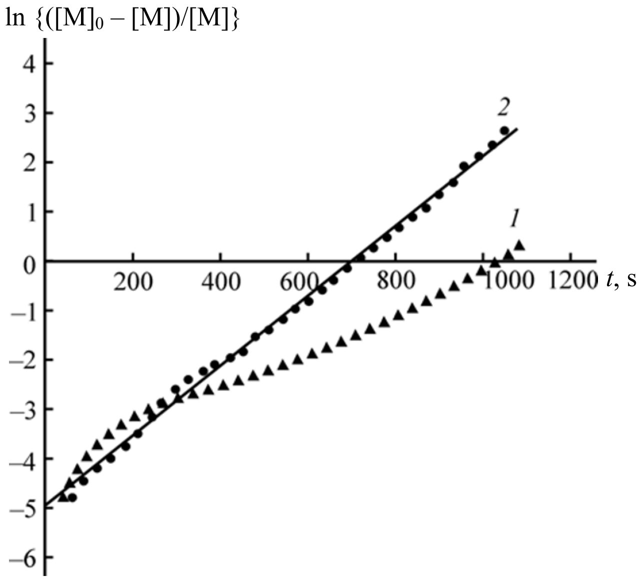


Fig. 4. Kinetic curves of oxidative polymerization of aniline hydrochloride in the presence of PVA [50000 Da; (*1*) 0.1 and (*2*) 0.2 mol/L] at 25°C. Initial concentration of aniline hydrochloride was 0.1 mol/L, and that of ammonium persulfate was 0.125 mol/L.

where $[H^+]_0$ and $[H^+]_\infty$ being the initial and the final concentrations of protons in the reaction mixture, respectively.

Aniline concentration was simultaneously determined by UV photometry. To do so, the reaction mixture was sampled each 120 s, the sample was quickly diluted with 1000× volume of 0.01 mol/L hydrochloric acid, and centrifuged (the reaction was quenched upon the dilution due to the decrease in both the reactants concentration and slowing down the single-electron oxidation of aniline in the acidic medium). Aniline concentration was calculated from the absorbance at λ 204 nm (a Beckman DU-65 spectrometer; ε_{204} of 7493 L mol^{-1} cm^{-1}).

Kinetic studies in the presence of PVA were performed similarly. PVA was dissolved in water prior to the experiment, and then aniline was dissolved in the prepared solution (PVA concentration was expressed in mol/L of the repeating units). Persulfate solution was prepared separately. The solutions were kept at the selected temperature, and then mixed; the reaction course was monitored by means of potentiomentry.

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