

Oligomer-containing Aqueous Quench Media Based on Polyethylene Glycol Esters of Oleic Acid

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Abstract: The results of studies of the cooling capacity, rheological and surface-active properties of aqueous solutions of oligomers: mono- and dipolyethylene glycol esters of oleic acid are presented. The availability of these oligomeric surfactants and the complex of their physicochemical properties make it possible to obtain, by varying the concentration of components, the compositions of hardening media (QM) with the required indicators. The process of cooling in oligomeric solutions depends on the concentration and stereochemistry of surfactant molecules. An increase in the content of oligomers is accompanied by a decrease in the rate and a shift in the maxima of the cooling rate curves towards low temperatures. As a result of comprehensive studies, a substantiated version of the heat transfer mechanism during cooling of a metal sample in aqueous micellar solutions of oligomeric surfactants was proposed. The stereochemistry of surfactant molecules, the shape of micelles, their strength, and ability to rearrange determine the complex mechanism of adsorption interaction with the metal surface, thus affecting the stages of shock boiling, vapor film, and nucleate boiling, and, ultimately, the intensity of heat transfer and the magnitude of the heat flux.

Keywords: Quenching Medium, Surfactants, Polyethylene Glycol Oleates, Micellar Solutions, Heat Flux, Heat Transfer Coefficient

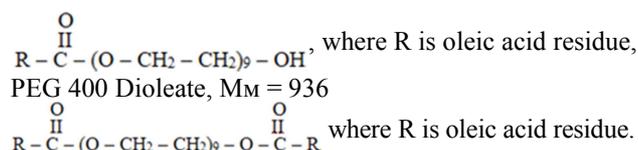
1. Introduction

The task of improving the quality of hardened parts remains relevant at the present time. One of the options for solving it is the development of new compositions of quenching media (QM), which, in addition to the intended purpose, provide manufacturability, environmental friendliness of the heat treatment process and are in an affordable price range. For example, aqueous QMs based on polyalkylene glycols [1–5] have been used for quenching for more than 30 years and ensure product quality, but the price of the polymer and high concentration in the working solution reduce the availability of the product on the world market. Studies of QM based on other water-soluble polymers have shown [6–12] the possibility of their practical use, however, this complicates the technological maintenance, because due to thermomechanical destruction of

macromolecules and a decrease in molecular weight (Mm), the viscosity of the working fluid decreases, which negatively affects the main technological indicators of QM [11–12]. Taking this into account, the aim of the present work is to study QM based on aqueous solutions of ethoxylated oligomers: mono- and disubstituted polyethylene glycol esters of oleic acid.

2. Materials, Devices and Experimental Technique

2.1. PEG 400 Monooleate, $M_m = 668$



2.2. Installation and Technique

A description of the laboratory thermo-acoustic system for determining the cooling properties of quenching media is given in [13].

2.3. Investigation of the Rheological Properties of Water-Polymer Quenching Media

The dynamic viscosity of the solutions was determined by the falling ball method (Lovis 2000M viscometer, Anton

Paar, Austria) and a Reotest-2 rotational viscometer (Germany) with digital indication of the results.

3. Experimental Results

3.1. Rheological Properties of Polymer Solutions

The results of studies of the dynamic viscosity of aqueous solutions of oligomers are presented in Figure 1 (a, b) and Figure 2 (a, b).

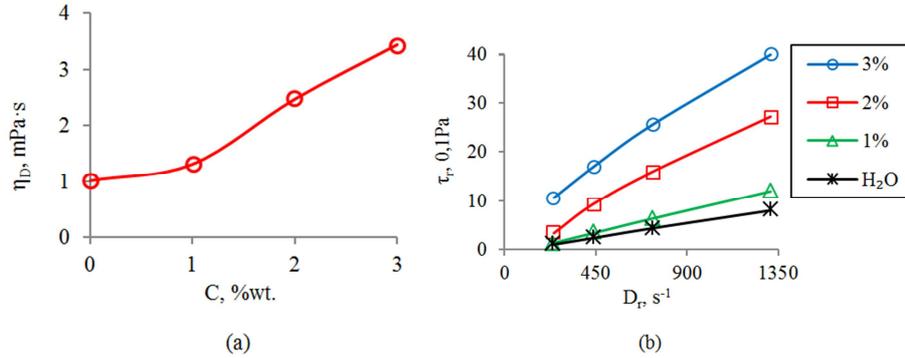


Figure 1. a) Dependence of dynamic viscosity η_D of PEG 400 Monooleate solutions on concentration at $T=20^\circ\text{C}$, $D_r=1312 \text{ s}^{-1}$; b) Dependence of shear stress τ_r on shear rate D_r and concentration of PEG 400 Monooleate, $T=20^\circ\text{C}$.

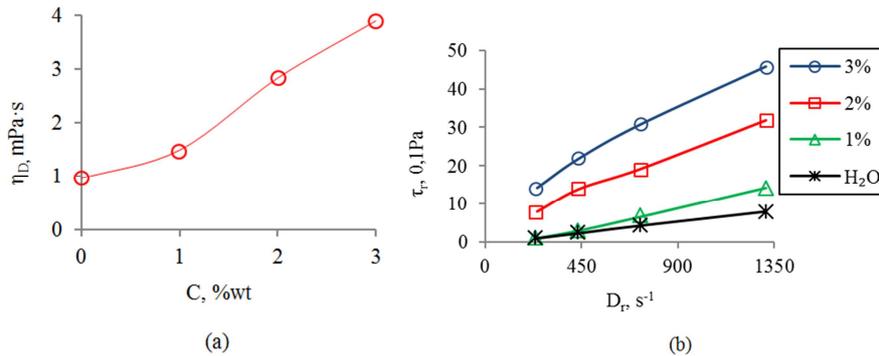


Figure 2. a) Dependence of dynamic viscosity η_D of PEG 400 Dioleate solutions on concentration at $T=20^\circ\text{C}$, $D_r=1312 \text{ s}^{-1}$; b) Dependence of shear stress τ_r on shear rate D_r and concentration of PEG 400 Dioleate, $T=20^\circ\text{C}$.

In the region of low concentrations of oligomers, a close to linear dependence of the shear stress τ_r on the shear rate D_r is observed (Figure 1 and Figure 2), which makes it possible to classify PEG 400 Monooleate and PEG 400 Dioleate solutions as Newtonian liquids [14]. The Newtonian nature of the flow of solutions indicates the absence of concentration and density fluctuations in the volume, which contributes to their hydrodynamic stability at high temperatures. This significantly affects the values of the critical heat flux density q_{cr}^1 , which determines the possibility of the formation of a vapor shell separating QM from the cooled metal surface [15].

3.2. Surface Tension of Polymer Solutions

One of the indicators of QM, which significantly affects the processes of nucleation, growth, and detachment of bubbles at the stage of nucleate boiling, is wettability, i.e. surface tension (γ) [16]. To assess the wettability of

oligomeric solutions, the dependence of γ values on the surfactant concentration was studied using a modified Wilhelmy plate (platinum) method [17] (Figure 3).

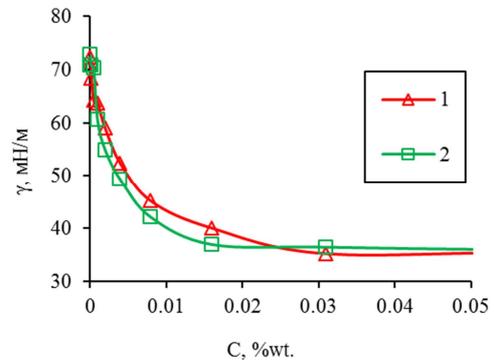


Figure 3. Dependence of surface tension γ of aqueous solutions of PEG 400 Monooleate (1) and PEG 400 Dioleate (2) on the concentration of oligomers, $T=20^\circ\text{C}$.

As can be seen from the results shown in Figure 3, the values of γ corresponding to the critical micelle concentration (CMC) [18] are in the region of 0.016% wt. for PEG 400 Dioleate and 0.032% wt. for PEG 400 Monooleate, which is consistent with the results [19] on the higher surface activity of bianker oligomers, which include PEG 400 Dioleate.

3.3. Effect of Oligomer Concentration on Cooling Curves (T , °C) and Cooling Rate of the Thermal Probe (dT , °C/s)

3.3.1. Cooling in Aqueous Solutions PEG 400 Monooleate

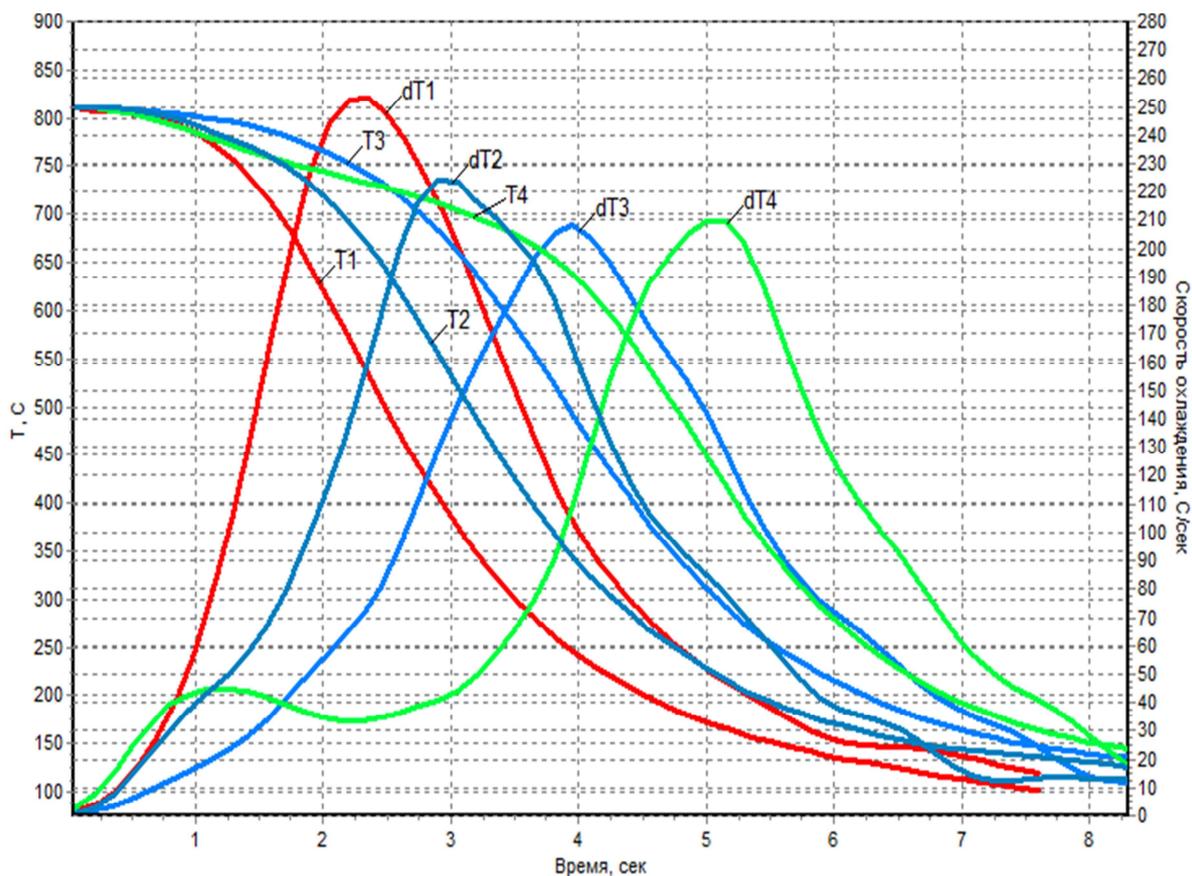
On figure 4(a, b) shows the cooling curves T and rate dT of cooling the thermoprobe in PEG 400 Monooleate solutions and distilled water, table 1 shows the parameters of the process of cooling the thermoprobe.

It is known that, during a film boiling crisis, the stability of a

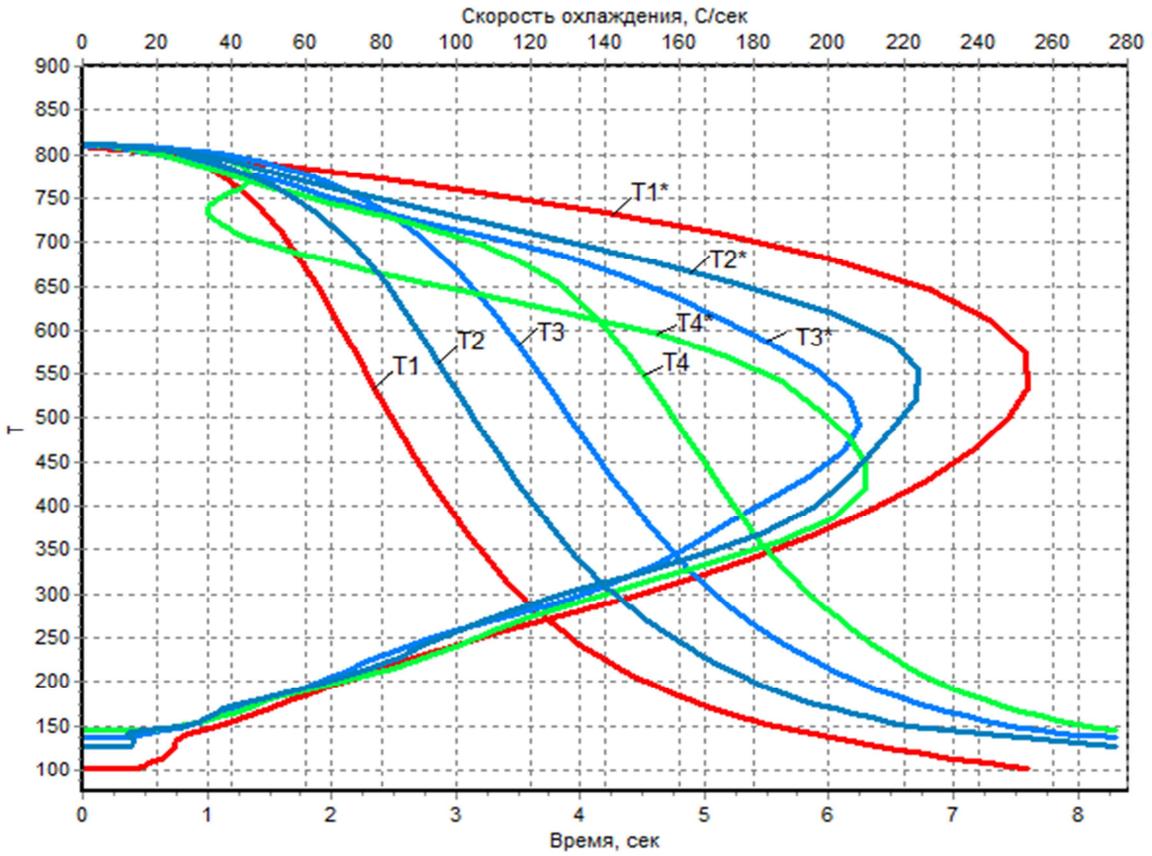
vapor film is determined by both the dynamic action and the surface tension [15, 16, 20]. Since the γ values of PEG 400 Monooleate solutions in the CMC region $\geq 0.03\%$ wt. reach a "shelf" (Figure 3), therefore, the positions of the peaks of the cooling curves on the time scale shift in proportion to the surfactant concentration (Figure 4,a), which determines the thickness and strength of the oligomeric film. The presence of a clear peak in the region $\tau = 1.2$ s at a concentration of 3%wt. should be attributed to the boiling of the PEG 400 Monooleate film formed at the moment of impact boiling [21]. An analysis of the dependence of the cooling rate T^* on temperature (Figure 4,b) shows that with an increase in the concentration of the PEG 400 Monooleate oligomer, both a decrease in the values of T^* and a noticeable shift of the curve maxima towards low temperatures are observed. In this case, it becomes possible to influence the QM composition on the temperature range of phase transformations [22].

Table 1. Cooling parameters obtained by ISO9950 at 20°C bath temperature and with no agitation.

No.	Indicator (unit)	Cond. Design	Concentration of PEG 400 Monooleate, %wt.			
			0,0	1,0	2,0	3,0
1	Probe Cooling Time from 850°C to 600°C, s	τ_{600}	2,1	2,7	3,4	4,2
2	Probe Cooling Time from 850°C to 400°C, s	τ_{400}	2,9	3,6	4,4	5,2
3	Probe Cooling Time from 850°C to 200°C, s	τ_{200}	4,5	5,4	6,2	6,9
4	Maximum cooling rate, °C/s	V_1^{\max}	253,0	224	208,1	210,0
5	Temperature TP at max. cooling rate, °C	T_{TP}^{\max}	534	555,0	493,0	418,0
6	Cooling rate, at temperature TP=300°C, °C/s	V_1^{TP300}	144,4	118,0	128,2	135,0



(a)

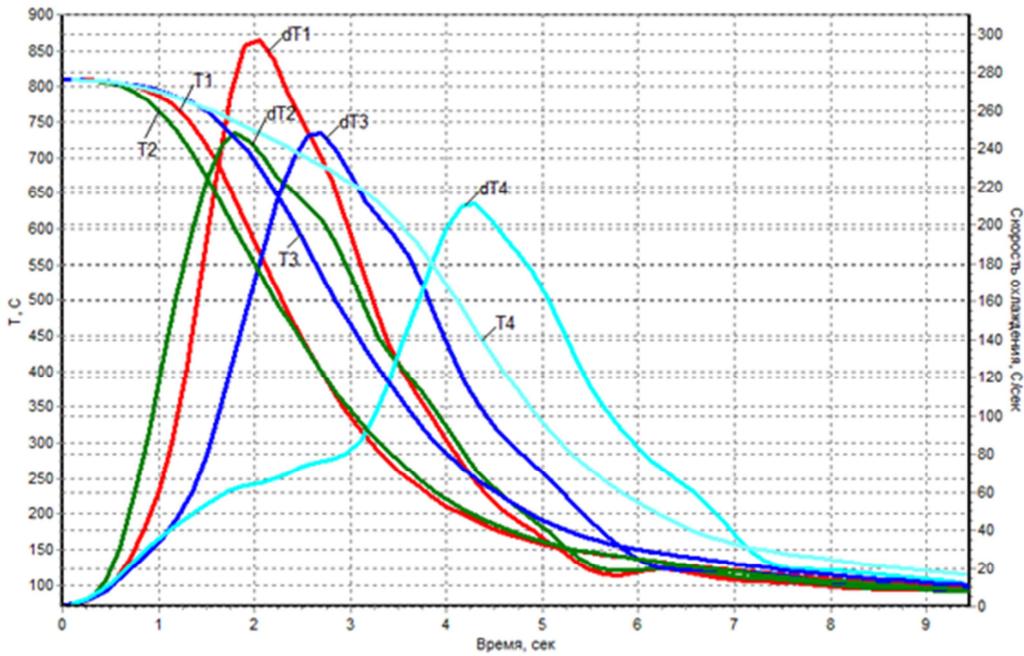


(b)

Figure 4. a) Dependence of cooling temperature T , °C and cooling rate dT , °C/s in PEG 400 Monooleate solutions at 20°C on time and concentration C , %wt.: 1 - 0.0; 2 - 1.0; 3 - 2.0; 4 - 3.0; b) Curves of cooling T , °C and cooling rate T^* (°C/s) in solutions of PEG 400 Monooleate at 20°C and concentration C , %wt.: 1 - 0.0; 2 - 1.0; 3 - 2.0; 4 - 3.0.

3.3.2. Cooling in Aqueous Solutions PEG 400 Dioleate

Figures 5(a,b) show the cooling curves T and cooling rates dT in PEG 400 Dioleate solutions and distilled water, Table 2 shows the parameters of the thermal probe cooling process.



(a)

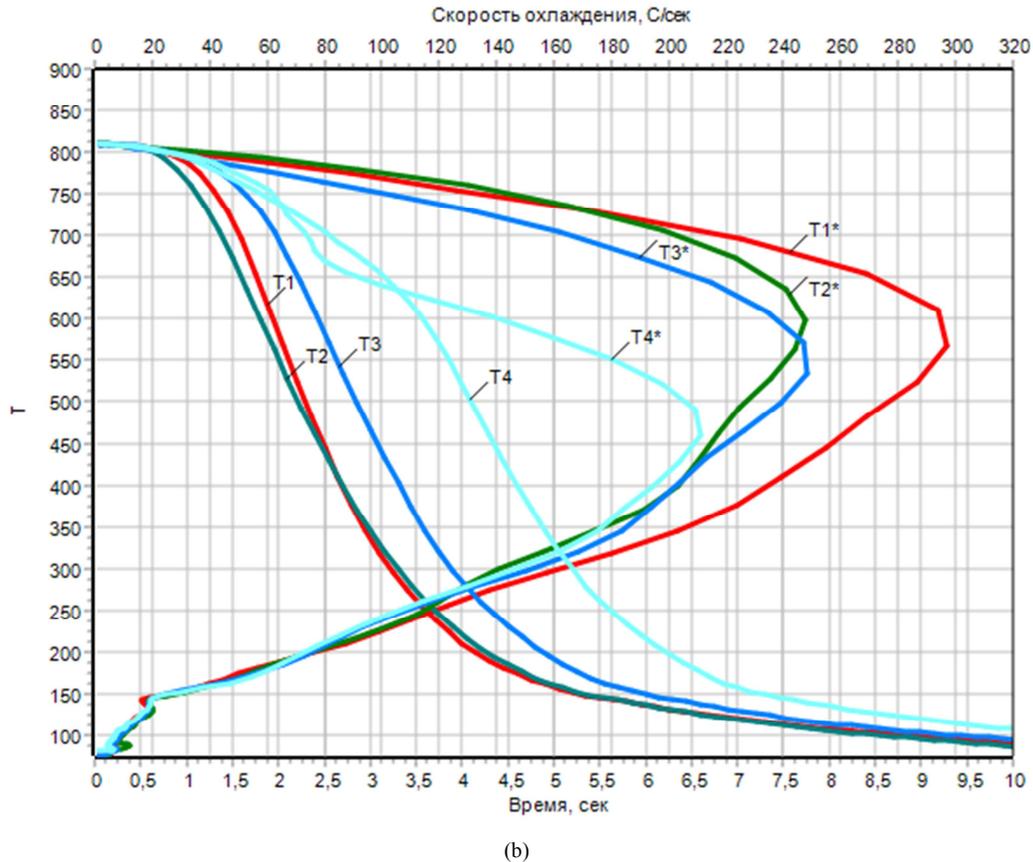


Figure 5. a) Dependence of cooling temperature T , °C and cooling rate dT , °C/s in PEG 400 Diolate solutions at 20°C on time and concentration, C , %wt.: 1 – 0.0; 2 – 1.0; 3 – 2.0; 4 – 3.0; b) Cooling curves T , °C and cooling rates T^* , °C/s in PEG 400 Diolate solutions at 20°C and concentration C , %wt.: 1 – 0.0; 2 – 1.0; 3 – 2.0; 4 – 3.0.

Table 2. Cooling parameters obtained by ISO9950 at 20°C bath temperature and with no agitation.

No.	Indicator (unit)	Cond. Design	Concentration of PEG 400 Diolate, %wt.			
			0,0	1,0	2,0	3,0
1	Probe Cooling Time from 850°C to 600°C, s	τ_{600}	1,9	1,8	2,4	3,6
2	Probe Cooling Time from 850°C to 400°C, s	τ_{400}	2,7	2,7	3,3	4,6
3	Probe Cooling Time from 850°C to 200°C, s	τ_{200}	4,3	4,3	4,9	6,2
4	Maximum cooling rate, °C/s	V_t^{\max}	296,7	247,9	248,2	210,9
5	Temperature TP at max. cooling rate, °C	$T_{TP}^{V_t^{\max}}$	567	599	535	459
6	Cooling rate, at temperature TP=300°C, °C/s	V_t^{TP300}	157,3	140,4	150,4	145,7

A comparative analysis of the cooling curves (Figure 4 and Figure 5) and the standard parameters of the thermal probe cooling process in PEG 400 Monooleate and PEG 400 Diolate solutions shows that the effect of the concentration of these oligomers differs significantly. Thus, in a 1% PEG 400 Monooleate solution, the maximum on the cooling curve is observed at ~3.0 s, and in a PEG 400 Diolate solution, at ~1.8 s. This can be explained by differences in M_m , stereochemistry of surfactant molecules, structure and shape of micelles [18]. At the moment of shock boiling, spherical micelles of PEG 400 Monooleate, which are resistant to hydrodynamic action, retain their surface activity in proportion to the concentration in the solution (Figure 4,a, curves 2-4).

The presence of a flexible polyester chain in the PEG 400 Diolate molecule, which connects oleic acid residues,

allows it, passing through the stage of destruction of micelles at the moment of shock boiling, to realize a horizontal orientation of the surfactant solution - vapor film with respect to the phase boundary [19]. As a result, an oligomeric film is formed on the interface between the solution and the vapor film [21], which determines the appearance of a peak at ~1.8 s on the dT_2 curve, i.e. 0.3s earlier than the peak on the dT_1 curve corresponding to distilled water (Figure 4,a). With an increase in the PEG 400 Diolate concentration to 2% and 3%, the “peak” time is not proportional to the oligomer concentration, which can be explained by the rearrangement of the micelle shape at the moment of shock effervescence.

It is known that each steel grade has its own critical cooling rate in the range of martensitic transformations, [15, 22] beyond which crack formation decreases and strength properties increase. An analysis of the families of cooling

curves in aqueous solutions of PEG 400 Monooleate and PEG 400 Dioleate suggests that it is possible to choose QM compositions that will meet the basic requirements for high-quality hardening of steels: high cooling capacity in the pearlitic range and lower in the martensitic transformation range.

3.4. Heat Fluxes Density and Heat Transfer Coefficients

3.4.1. Heat Fluxes Density and Heat Transfer Coefficients in Solutions PEG 400 Monooleate

Using the parameters of the cooling curves (Figures. 4 (a,b)) presented in Table 1, the dependences of heat fluxes $q(T_s)$ (Figure 6) and the heat transfer coefficient $\alpha(T_s)$ (Figure 7) were calculated using the INC solution method [23]. In the pearlite transformation temperature range of

650–400°C, the $q(T_s)$ values rapidly increase and reach a maximum in the bainitic transformation region of 500–300°C, while the peak values of $q(T_s)^{\max}$ noticeably decrease at an oligomer concentration of 1%wt. (Figure 6, curve 1) and 2%wt. (Figure 6, curve 2) by 8.5% and 25%, respectively, relative to the values of $q(T_s)^{\max}$ in water (Figure 6, curve d). With an increase in the concentration of the oligomer to 3%wt. the $q(T_s)$ curve becomes asymmetric, its maximum at ~300°C is in the region of martensitic transformations, in this case, the peak values of $q(T_s)^{\max}$ are close to the values of $q(T_s)$ at ~400°C, in the field of bainitic transformations. Small peaks in $q(T_s)$ at 780°C and 740°C correspond to heat fluxes accompanying the stages of shock boiling [24] and formation of an oligomeric film [21].

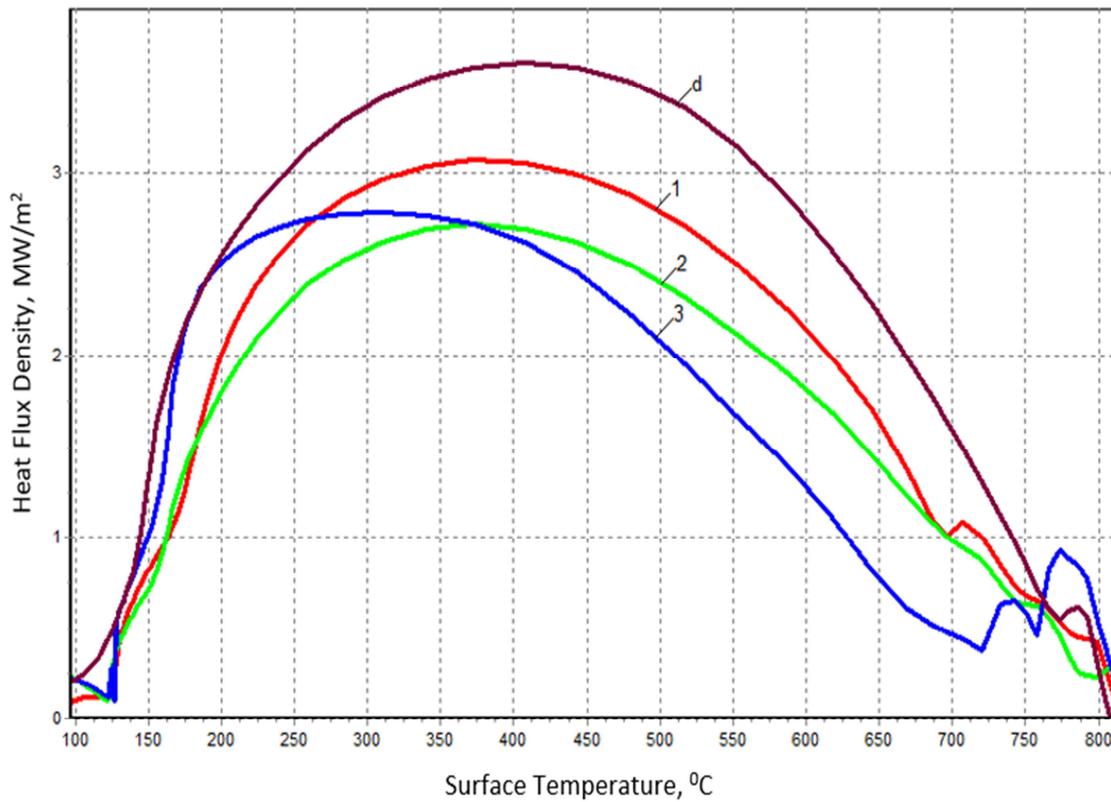


Figure 6. Heat flux density $q(T_s)$ in solutions of PEG-400 Monooleate at distilled water with concentration: d - 0%wt.; 1 - 1%wt.; 2 - 2%wt.; 3 - 3%wt. at $T=20^\circ\text{C}$.

The dependence of the heat transfer coefficient $\alpha(T_s)$ on the concentration of PEG 400 Monooleate and the surface temperature of the thermal probe is shown in Figure 7. Addition of 1%wt. of the oligomer reduces the values of $\alpha(T_s)^{\max}$ by 2000 $\text{W/m}^2\cdot\text{K}$ (Figure 7, curve 1), while the position of the peak shifts by 25°C to the region of high temperatures, which can be explained by an increase in the duration of the vapor film phase and a decrease in the heat transfer rate [25]. With an increase in the concentration of the oligomer to 2% wt. (Figure 7, curve 2) the position of the maximum of the $\alpha(T_s)$ curve does not change, however, the peak value of $\alpha(T_s)^{\max}$ decreases by 1500

$\text{W/m}^2\cdot\text{K}$, which indicates a noticeable decrease (by 500 $\text{W/m}^2\cdot\text{K}$) in the effect Surfactants for the heat transfer process. With an increase in the concentration of the oligomer to 3%wt. (Figure 7, curve 3) there is an inversion of the influence of the oligomer: the peak value of $\alpha(T_s)^{\max}$ increases by 500 $\text{W/m}^2\cdot\text{K}$ relative to water, while the position of $\alpha(T_s)^{\max}$ is preserved.

Thus, the PEG 400 Monooleate oligomer is an additive that makes it possible to control the cooling capacity of QM in a fairly wide range of $\alpha(T_s)$ values in the martensitic transformation temperature range (150–300°C).

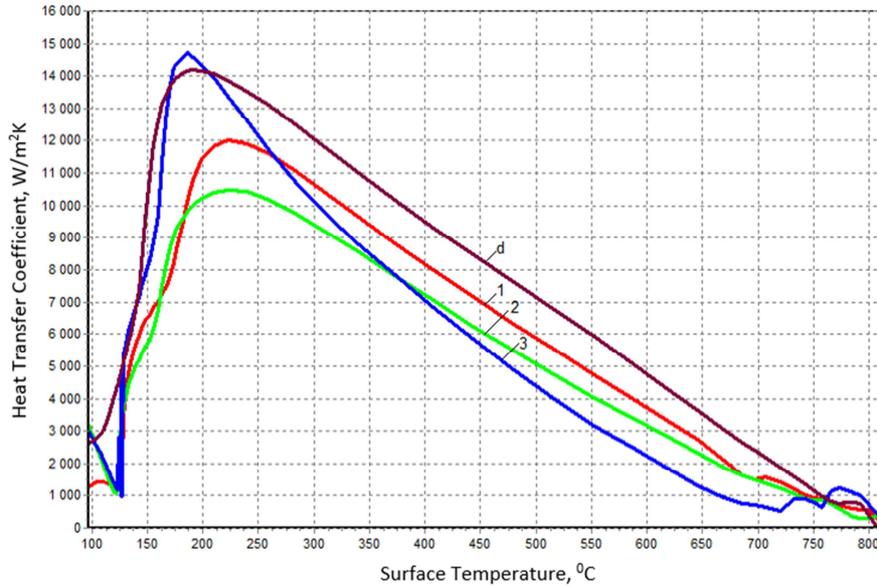


Figure 7. Heat transfer coefficient $\alpha(T_s)$ in solutions of PEG-400 Monooleate at distilled water with concentration: d - 0%wt.; 1 - 1%wt.; 2 - 2%wt.; 3 - 3%wt. at $T=20^\circ\text{C}$.

3.4.2. Heat Fluxes Density and Heat Transfer Coefficients in Solutions PEG 400 Dioleate

Figure 8 shows the results of calculations of heat fluxes $q(T_s)$ in PEG 400 Dioleate solutions versus surface temperature and oligomer concentration. A fast and high value of $q(T_s)$ is achieved upon cooling in water, in 1% and 2% oligomer solutions (Figure 8, curves d, 1 and 2) in the range of 750–650°C, i.e. in the temperature range of austenitic transformations. In the temperature range of 650–400°C for pearlite transformations, the $q(T_s)$ curves for 1% and 2% solutions reach a maximum, while the $q(T_s)^{\text{max}}$

values decrease by 700 MW/m² and 1000 MW/m², respectively, with respect to $q(T_s)^{\text{max}}$ values in water. With an increase in the concentration of the oligomer to 3% wt. the $q(T_s)$ curve is symmetric with respect to the $q(T_s)^{\text{max}}$ position at $\sim 330^\circ\text{C}$, i.e. practically in the temperature range of martensitic transformations (Figure 8, curve 3). Small peaks in the region of 780°C on curves 1 and 2 should be attributed to heat fluxes corresponding to the impact boiling stage [24, 21], and a wide maximum $q(T_s)^{\text{max}} \sim 700^\circ\text{C}$ (Figure 8, curve 3) corresponds to the stage film boiling of a 3% solution of PEG 400 Dioleate.

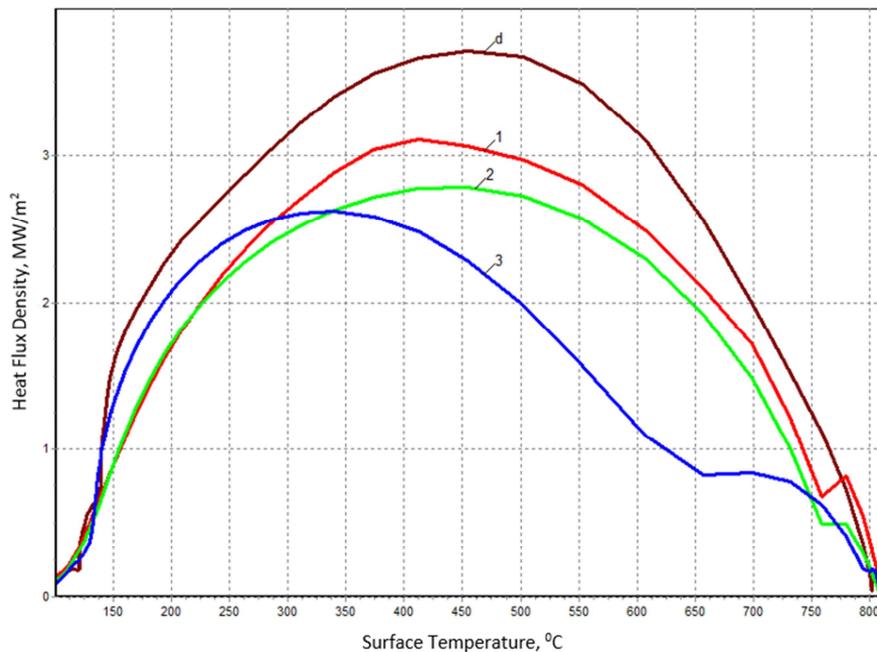


Figure 8. Heat flux density $q(T_s)$ in solutions of PEG 400 Dioleate at distilled water with concentration: d - 0%wt.; 1 - 1%wt.; 2 - 2%wt.; 3 - 3%wt. at $T=20^\circ\text{C}$.

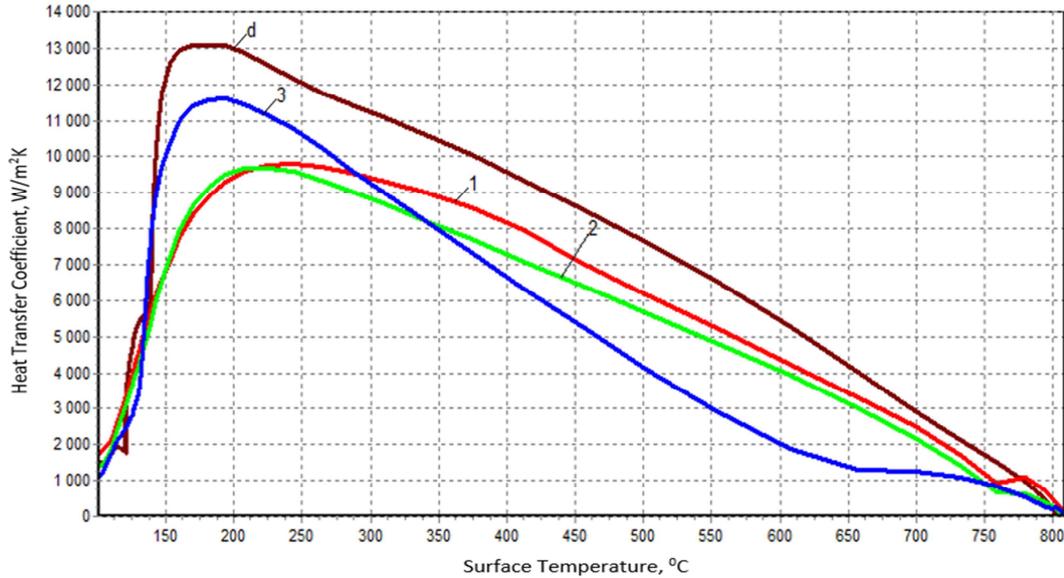


Figure 9. Heat transfer coefficient $\alpha(T_s)$ in solutions of PEG 400 Dioleate at distilled water with concentration: d - 0%wt.; 1 - 1%wt.; 2 - 2%wt.; 3 - 3%wt. at $T=20^\circ\text{C}$.

The dependence of the heat transfer coefficient $\alpha(T_s)$ on the concentration of PEG 400 Dioleate and the surface temperature of the thermal probe is shown in Figure 9. Addition of 1% wt. oligomer into water is accompanied by a noticeable shift in $\alpha(T_s)^{\max}$ from 185°C to 245°C (Figure 9, curves d and 1), while the values of $\alpha(T_s)^{\max}$ decrease from $13000\text{ W/m}^2\cdot\text{K}$ to $9800\text{ W/m}^2\cdot\text{K}$, i.e. by 24.8%. An increase in the concentration of the oligomer to 2% (Figure 9, curve 2) is accompanied by a slight shift in the position of $\alpha(T_s)^{\max}$ up to 225°C , while the value of $\alpha(T_s)^{\max}$ decreases by $120\text{ W/m}^2\cdot\text{K}$ with respect to 1%- solution (Figure 9, curve 1).

With an increase in the concentration of PEG 400 Dioleate to 3% wt. observed as in the case of PEG 400 Monooleate (Figure 7, curve 3) inversion of the influence of the oligomer: the peak value of $\alpha(T_s)^{\max}$ increases relative to the values of $\alpha(T_s)^{\max}$ in 1% and 2% solutions by $1500\text{ W/m}^2\cdot\text{K}$, and the position of the maximum of the $\alpha(T_s)^{\max}$ curve shifts to 185°C , i.e. 40°C towards lower temperatures. Thus, the maximum heat transfer in a 3% solution of the PEG 400 Dioleate oligomer is realized in the temperature range of $300\text{--}150^\circ\text{C}$ for martensitic transformations, which is a positive result, because in this case, the steel hardens and the probability of crack formation decreases [15, 26].

4. Discussion

The use of surfactants as additives in water-based QMs is explained by their ability to significantly affect the surface tension of the solution, which largely determines the process of bubble nucleation, the stage of their growth and detachment from the surface. As a result, it becomes possible to influence the stage of nucleate boiling, including the position of the maximum of the cooling curve and its intensity. The range of surfactants that are used as additives to QM is quite large, but almost all of them belong to classical surfactants: anionic and nonionic. In the presented work, nonionic surfactants were

chosen as additives: mono- and disubstituted polyethylene glycol esters of oleic acid, which differ in the presence of one (mono-) or two (di-) oleic acid residues in the surfactant molecule. In this case, the physicochemical interaction of the surfactant "head" with the metal surface changes insignificantly, but their stereochemistry obviously differs. This mainly affects the stage of formation of micelles, the shape, size, and strength of which differ significantly for mono- and bianker surfactants [19]. In this case, it can be assumed that the metal cooling process is largely determined in the range of low concentrations by the micellar structure of the surfactant solution; QM. This option is supported by the location of the maximum of the cooling curves in PEG 400 Mono- and Dioleate solutions at a concentration of 1% wt. In Figure 4,a (curve 2), the maximum of the $dT2^{\max}$ curve in a PEG 400 Monooleate solution is observed at $\tau = 3.0\text{ s}$, i.e. shifts relative to $dT1^{\max}$ in distilled water by $\Delta\tau = +0.7\text{ s}$, while $dT2^{\max}$ in PEG 400 Dioleate solution (Figure 5,a, curve 2) is observed at $\tau = 1.8\text{ s}$, i.e. the shift with respect to $dT1^{\max}$ in water is $\Delta\tau = -0.3\text{ s}$. Such a diametrically opposite effect of two oligomers can be explained by differences in the shape of micelles: spherical for PEG 400 Monooleate and spherical for PEG 400 Dioleate [27], which a priori significantly differ in resistance to destruction under conditions of strong hydrodynamic action at the moment of shock effervescence. Apparently, the PEG 400 Dioleate solution in the range of low concentrations is a liquid nanostructured by longitudinally elongated micelles, the thermal conductivity (λ) of which exceeds the λ values of distilled water, as, for example, in nanofluids based on carbon nanotubes (CNT) [25].

5. Conclusion

The mechanism of cooling in oligomeric solutions depends on the concentration and stereochemistry of surfactant molecules. An increase in the content of oligomers is

accompanied by a decrease in the rate and a shift in the maxima of the cooling rate curves towards low temperatures. Heat fluxes increase rapidly and reach a maximum in the region of bainitic transformations upon cooling to 1%-2%wt. solutions, and when the concentration is increased to 3% wt. $q(T_s)^{\max}$ values are shifted to the temperature range of 350-200°C. The heat transfer coefficient $\alpha(T_s)$ is significantly affected by both the concentration and the stereochemistry of oligomers, and the maximum $q(T_s)^{\max}$ values are observed in the temperature range of 300–150°C, i.e. in the range of martensitic transformations. Taking into account the results of the conducted studies, we can assume the possibility of implementing cooling in oligomeric solutions with a controlled duration of the vapor phase, which makes it possible to carry out heat treatment of high-alloy steels [10, 15, 22, 26].

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