Sonochemiluminescence of aromatic hydrocarbons

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Multibubble sonoluminescence of water and a series of aromatic hydrocarbons, *viz.*, benzene, toluene, ethylbenzene, and *m*-xylene (at 25 °C), and a naphthalene melt (at 110—120 °C) was studied. An analysis of the influence of oxygen and argon on the sonoluminescence intensity and the luminescence spectra of these liquid compounds, as well as the effect of additives of ionol, ethanol, and 9,10-dibromoanthracene on *m*-xylene sonoluminescence, showed that a considerable contribution to the sonoluminescence of aromatic hydrocarbons is made by chemiluminescence reactions associated with their oxidation. This sonochemiluminescence is observed in both the gas phase of cavitation bubbles and the bulk solution where luminescence is retained for a long time after ultrasonication switching-off (the initial intensity of the residual chemiluminescence is up to 10% of the luminescence intensity during sonolysis). As for thermoinitiated oxidation, the afterglow of *m*-xylene contains the radical and molecular components.

Key words: sonolysis, sonochemical reactions, sonoluminescence, chemiluminescence, sonochemiluminescence, *m*-xylene.

Many works on multibubble sonoluminescence (MBSL) are devoted to revealing details of the mechanism of formation of electron-excited emitters upon inelastic collisions of high-energy particles in the gas phase of cavitation bubbles. These collisions occur during gas heating *via* adiabatic compression of bubbles (thermal theory[1]) or upon their electric breakdown (electrical theory[2]). The mechanism correctly describes the MBSL of water and the most of aqueous solutions.[3] However, in some systems, such as aqueous solutions of luminol[4] and terbium chelate,[5] acetaldehyde, and crotonaldehyde,[6,7] MBSL is caused by physical excitation through collisions of particles in bubbles and also by chemical reactions of sonolysis products occurring in both the bubbles and bulk solution. This chemiluminescence mechanism of MBSL is poorly studied but it can be important for the development of new sonochemical technologies using oxidation processes and nonaqueous solvents. Such processes recently became quite attractive.[8] The present work is devoted to the study of sonochemiluminescence (SCL) appeared during multibubble sonolysis of a series of aromatic hydrocarbons.

Experimental

All solvents were twice purified from admixtures using the known procedure[9] and then were doubly distilled. Sonolysis was carried out using UZDN-A and ACE GLASS standard setups for ultrasonic treatment of solutions with immersed wave guides (20—22 kHz, total acoustic irradiation power 5—30 W). The detection of MBSL from a temperature-maintained quartz cell was carried out on a setup for studying weak glows with a FEU-39 detector equipped with a disk cassette for changeable boundary light filters. which made it possible to determine the spectral composition of radiation. The sonoluminescence spectra of oxygen-saturated *m*-xylene and naphthalene were recorded on an Aminco Bowman J4-8202 spectrofluorimeter with a resolution of 10 nm using a Hamamatsu 1P28 photomultiplier tube as a detector. To detect the long-wavelength glow, a setup was used in which a FEU-83 photomultiplier tube cooled with liquid nitrogen vapor to 230 K and sensitive to IR radiation (to 1300 K) served as the light receiver. A combination of the light filters IKS7 and ZS7 transmitting radiation in the range 1200—1300 nm was mounted between the cell with irradiated xylene and the FEU.

The sonolysis products were analyzed by chromatography coupled with mass spectrometry on a Finigan-4021 instrument (glass capillary column 50000×0.25 mm, stationary phase HP-5, helium as carrier gas, temperature-programmed regime from 50 to 300 °C with a rate of 5 deg $\min^{-1}$, temperature of the evaporator 280 °C, and temperature of the ion source 250 °C, 70 eV).

Results and Discussion

The study of a series of aromatic hydrocarbons showed that the characteristics of MBSL of these compounds and of water differ (Table 1). Note that the purely "physical" mechanism of glow appearance is doubtless for the sonolysis of water.

The MBSL intensities of water saturated with air and with oxygen differ slightly. A 1.5-fold intensity increase

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Table 1. The MBSL intensity (I (arb. units)) of various liquid compounds

Compound	I_{MBSL}	$I_{\mathrm{MBSL}}(\mathrm{Ar})$	$I_{\mathrm{MBSL}}(\mathrm{O_2})$	I_{CL}
Water	100	1000	150	0
Benzene	25	36	68	<1
Toluene	60	110	230	5
Ethylbenzene	75	140	270	6
m-Xylene	310	1240	4650	30
Naphthalene (melt, 110 °C)	250	500	1750	5

Notes. The luminescence intensity of all compounds, except for naphthalene, was measured at ~25 °C; I_{MBSL}, $I_{\mathrm{MBSL}}(\mathrm{Ar})$, and $I_{\mathrm{MBSL}}(\mathrm{O_2})$ are the luminescence intensity of the liquids saturated with air upon argon and oxygen bubbling, respectively; I_{CL} is the initial afterglow intensity upon switching-off ultrasonication after 10 min of sonolysis at a sonication power of 20 W cm^{-2}.

upon oxygen bubbling can be explained by a simple increase in the number of bubbles pulsing in the cavitation region. Saturation with argon enhances the intensity by an order of magnitude. After the end of sonolysis of water, the glow decays rapidly within the time ≤0.1 s.

On the contrary, in aromatic liquids an inert gas slightly affects the MBSL intensity, whereas the effect of oxygen saturation is much more significant (in *m*-xylene the intensity increases by 15 times). In addition, sonolysis of these liquids results in the appearance of afterglow, which lasts for tens of minutes in oxygen-saturated solutions. The initial afterglow intensity is approximately 5—10% of the MBSL intensity during sonolysis. A similar effect of oxygen on the MBSL can reasonably be explained by a considerable role of chemiluminescence oxidation reactions occurring during sonolysis of aromatic compounds in the liquid phase. *m*-Xylene (hereinafter, xylene) is characterized by the highest MBSL and afterglow intensity, which made it possible to study this solvent in detail.

First, the high MBSL intensity of xylene compared to the MBSL intensity of water should be noted. According to the thermal theory, the ratio of these intensities should be reciprocal. According to this theory, the final temperature (T_{f}) achieved in a cavitation bubble upon its collapse can approximately be estimated by the adiabatic process equation[10]

$$T_{\mathrm{f}} = T_{\mathrm{i}}(R_{\mathrm{i}}/R_{\mathrm{f}})^{3(\gamma - 1)}, \quad (1)$$

where T_{i} is the initial temperature; R_{i} and R_{f} are the initial and final radii of a bubble, respectively.

In this equation, the adiabatic exponent (γ) equal to the ratio of heat capacities $C_{\mathrm{p}}/C_{\mathrm{v}}$ for bubbles filled with air and water vapor, respectively, is 1.33. For xylene vapor, it is 1.1 (see Ref. 11). Thus, volatile xylene vapors filling bubbles should lower the final temperature compared to T_{f} in water and, correspondingly, the MBSL intensity. Accepting that $R_{\mathrm{i}}/R_{\mathrm{f}} = 5$ and $T_{\mathrm{i}} = 300$ K, we obtain the temperature value in the final phase of compression of bubbles filled with xylene: $T_{\mathrm{f}} \approx 500$ K. With allowance for incomplete filling of bubbles with xylene, it looks possible to achieve somewhat higher temperatures of an order of 10^3 K. For bubbles in water, we obtain $T_{\mathrm{f}} \approx 1500$ K. Anyway, these temperatures are insufficient for the excitation of glow emitters in the visible spectral range due to inelastic collisions of particles having enhanced kinetic energy.

Note that the ratio $R_{\mathrm{i}}/R_{\mathrm{f}} = 5$ was chosen as average from the following boundary estimates. According to calculations,[12] a bubble filled with a real gas—vapor mixture (not with an ideal gas) cannot be compressed more than 8.8-fold when an infinitely high pressure is created in the liquid. At the same time, it was shown[10,13,14] that the temperature values determined from the emission lines in the MBSL spectra for argon-saturated water and dodecane ($\gamma = 1.67$) coincide with the values (~4000 K) obtained by Eq. (1), if we accept the ratio $R_{\mathrm{i}}/R_{\mathrm{f}} = 3.65$.

Indeed, saturation of a liquid with a monoatomic inert gas (argon) leading to an increase in the adiabatic exponent can increase the temperature in a bubble and the MBSL intensity. Nevertheless, as can be seen from the results obtained, this effect is substantial for water and for saturated hydrocarbons but plays less important role in the case of xylene. This can be due to very high saturated vapor pressure ($P_{\mathrm{s.v}}$) of xylene: 1.12 kPa at 298 K (see Ref. 11), which does not allow one to considerably decrease the fraction of a polyatomic gas in a gas—vapor mixture inside a cavitation bubble during liquid saturation with argon. Indeed, according to the literature data,[14] the maximum temperature for bubbles in argon-saturated dodecane decreases from 3600 K at a saturated vapor pressure of 2.67 Pa (temperature of the solution 276 K) to 3200 K at a saturated vapor pressure of 17.3 Pa (temperature of the solution 298 K). As can be seen, T_{f} decreases substantially in the $P_{\mathrm{s.v}}$ range much smaller than $P_{\mathrm{s.v}}$ for xylene. Therefore, it can be assumed that for saturation of xylene with argon the maximum temperature in cavitation bubbles will increase slightly compared with the value estimated above. Obviously, saturation with oxygen ($\gamma = 1.4$) cannot substantially increase the temperature in bubbles and the MBSL intensity. Thus, rather bright MBSL of xylene and the fact of the significant increase in its intensity with oxygen saturation cannot be explained in the framework of the thermal theory.

It can be assumed that the MBSL of xylene is due mainly to the electric breakdown of cavitation bubbles rather than their thermal warming in the compression phase. Significant translational migrations, various deformations, and electrization of bubbles with the formation of uncompensated charges occur during multibubble sonolysis and, therefore, the probability of bubble breakdown is rather high.[2] In addition, if the liquid contains oxygen, a considerable role in MBSL should belong to the secondary chemiluminescence reactions of fragmentation particles (ions and radicals) appeared upon collisions of xylene molecules with "hot" particles along with the primary processes of physical excitation. Taking into account the high probability of the electric mechanism of MBSL, it can be assumed that electrons act as these particles.

Chemiluminescence (CL) during oxidation processes is associated with the reactions of radical products[15] and, hence, the influence of radical acceptors on the MBSL of xylene was checked to evaluate the validity of the proposed hypothesis. It turned out that ionol is an efficient inhibitor of free-radical reactions in the liquid phase: at the concentration 10^{-2} mol L^{-1} it exerts no effect on the MBSL intensity of xylene but decreases the afterglow intensity. Ionol has low vapor elasticity, and at this concentration its penetration into cavitation bubbles is negligible. Ethanol, being a more volatile acceptor, which is added in higher concentrations than poorly soluble ionol, manifested a noticeable quenching effect (Fig. 1). The addition to xylene of a known activator of the liquid-phase CL, namely, 9,10-dibromoanthracene, exerts no effect on the MBSL intensity but results in the enhancement of the afterglow intensity. These facts indicate a doubtless participation of the radical products of xylene sonolysis in at least the formation of afterglow in the bulk liquid and does not contradict to the possible contribution of free-radical chemiluminescence to the intensity of the MBSL observed during sonolysis and emitted from cavitation bubbles. This assumption is confirmed by the obtained spectral data.

The MBSL spectrum of argon-saturated xylene contains bands at 320 and 500—550 nm (Fig. 2). It is most likely that the emitter of these bands is represented by excited singlet and triplet excimers of xylene, whose emission, as known,[16] is observed in these spectral ranges upon the bombardment of xylene molecules with electrons, which also occurs in bubbles, as it has already been mentioned.

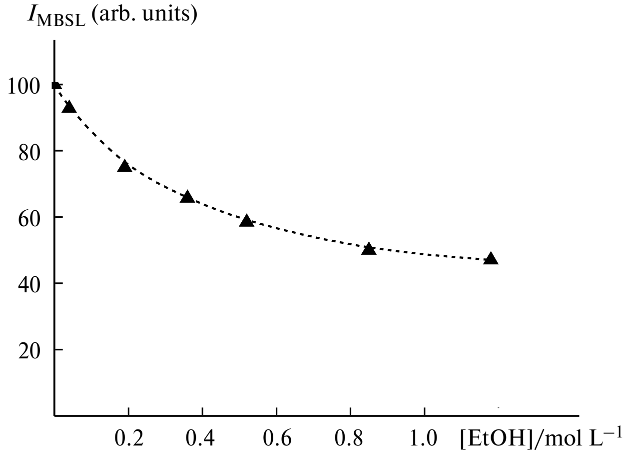

Fig. 1. The MBSL intensity of xylene *vs* ethanol concentration.

Figure 2 shows a band with a broad maximum at 425 nm along with the bands of xylene in the MBSL spectrum. In an air-saturated solution, the intensity of this band becomes predominant over the bands of xylene, whereas for saturation with oxygen this band is a single one. In our opinion, this band is the result of the luminescence of triplet-excited carbonyl products formed by the radical-chain oxidation of xylene.[17] The xylene samples subjected to sonolysis for 1 h contain 3-methylbenzaldehyde (MS (70 eV), m/z ($I_{\rm rel}$ (%): 120 $[M]^+$ (91), 119 $[M - H]^+$ (94), 91 $[M - HCO]^+$ (97), 65 $[M - HCO - C_2H_4]^+$ (50)). Thus, 3-methylbenzaldehyde is the most probable emission emitter responsible for the maximum at λ = 425 nm appeared upon the sonolysis of oxygen-saturated xylene.

It is known that CL emitters of radical-chain oxidation of hydrocarbons can be both the carbonyl compounds and singlet oxygen.[18,19] The luminescence band of $^1O_2$ lies in the IR region with a maximum at λ = 1270 nm.[20] The use of the detector sensitive in the IR region and the light filters transmitting the band of $^1O_2$ showed that about 5% of the total intensity of glow emitted upon oxygen-saturated xylene sonolysis is concentrated in the region of λ > 1200 nm (Fig. 3). Taking into account the low sensitivity of the emission detector in the region (less than 5% of the sensitivity at 425 nm), this result indicates the presence of a considerable amount of IR photons emitted along with UV and visible photons, which are generated by the carbonyl group. An emitter of such glow can be singlet oxygen. The possibility of formation of the lowest excited states of oxygen during ultrasonication of liquids containing dissolved oxygen has earlier been mentioned.[21]

In our opinion, xylene oxidation in cavitation bubbles follows the known mechanism of radical-chain oxidation

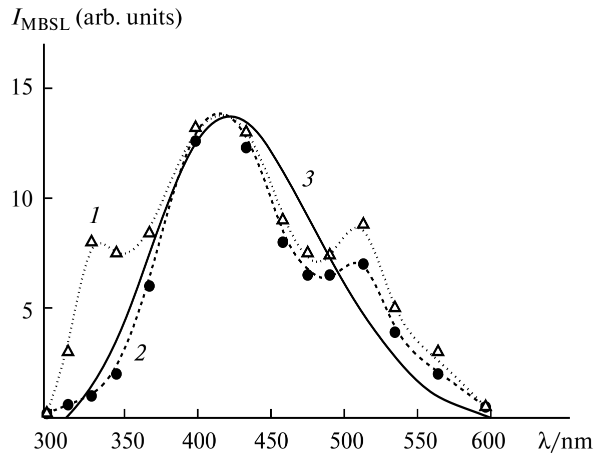

Fig. 2. The sonoluminescence spectra of xylene saturated with argon (*1*) and air (*2*) detected using boundary light filters. The sonoluminescence spectrum of xylene saturated with oxygen detected on an Aminco spectrofluorimeter (*3*). The spectra were normalized by intensity in a maximum at λ = 425 nm.

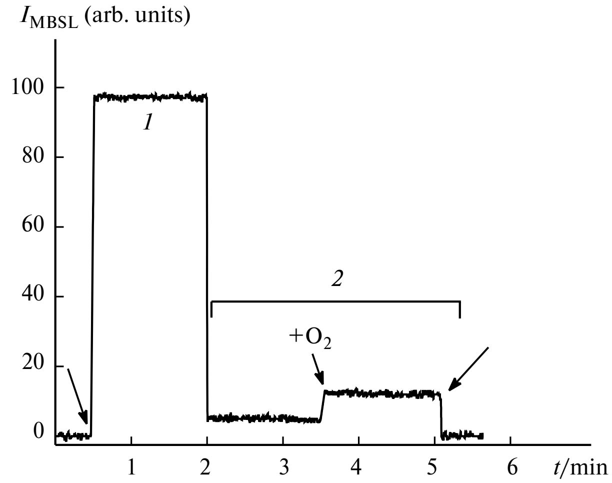

Fig. 3. Detection of the MBSL of xylene using a detector sensitive in the IR spectral range: total luminescence (*1*); and luminescence in the IR range detected through a combination of the light filters IKS7 + ZS7 (*2*). Arrows indicate the moments of ultrasonication switching-on and switching-off and oxygen feeding.

of hydrocarbons in the gas phase with degenerate chain branching.[22—25] The oxidation process includes the following main steps.

Radical generation in chain initiation reactions

$$\mathrm{RH} \xrightarrow{)))} \mathrm{R}^{\bullet} + \mathrm{H},$$

$$\mathrm{RH} + \mathrm{O_2} \longrightarrow \mathrm{R}^{\bullet} + \mathrm{HO_2}.$$

Chain propagation and formation of molecular products

$$\mathrm{R}^{\bullet} + \mathrm{O_2} \longrightarrow \mathrm{RO_2}^{\bullet},$$

$$\mathrm{RO_2}^{\bullet} + \mathrm{RO_2}^{\bullet} \longrightarrow \mathrm{ROOR} + \mathrm{O_2}\,(^1\mathrm{O_2}^*),$$

$$\mathrm{RO_2}^{\bullet} + \mathrm{RO_2}^{\bullet} \longrightarrow \mathrm{RO}^{\bullet} + \mathrm{RO}^{\bullet} + \mathrm{O_2},$$

$$\mathrm{RO}^{\bullet} + \mathrm{RO}^{\bullet} \longrightarrow \mathrm{R'{=}O}\ (\mathrm{R'{=}O^*}) + \mathrm{ROH},$$

$$\mathrm{RO_2}^{\bullet} + \mathrm{RH} \longrightarrow \mathrm{ROOH} + \mathrm{R}^{\bullet},$$

$$\mathrm{RO}^{\bullet} + \mathrm{RH} \longrightarrow \mathrm{ROH} + \mathrm{R}^{\bullet},$$

$$\mathrm{ROOH} \longrightarrow \mathrm{RO}^{\bullet} + {}^{\bullet}\mathrm{OH},$$

$$\mathrm{RH} + {}^{\bullet}\mathrm{OH} \longrightarrow \mathrm{R}^{\bullet} + \mathrm{H_2O},$$

$${}^{\bullet}\mathrm{OH} + {}^{\bullet}\mathrm{OH} \longrightarrow \mathrm{H_2O_2},$$

where RH is *m*-xylene and R′=O is 3-methylbenzaldehyde.

Excited products, *viz.*, singlet oxygen and the carbonyl compound, namely, 3-methylbenzaldehyde, can be formed in the framework of this scheme in the corresponding reaction. These products result in the observed emission

$$\mathrm{R'{=}O^*} \longrightarrow \mathrm{R'{=}O} + h\nu\ (425\ \mathrm{nm}),$$

$$^1\mathrm{O_2}^* \longrightarrow {}^3\mathrm{O_2} + h\nu\ (1270\ \mathrm{nm}).$$

According to the considerable enhancement of the MBSL intensity and a change in the shape of the MBSL spectra, the chemiluminescence glow component at xylene saturation with oxygen becomes predominant. Thus, the MBSL of xylene in oxygen-saturated solutions can be named sonochemiluminescence in cavitation bubbles. Another variety of SCL is CL that continues to occur in the liquid bulk after sonolysis cessation. The CL is caused by the fact that the reaction of the sonolysis products in the bulk liquid do not cease after ultrasonication switching-off and disappearance of cavitation bubbles. These reactions provide rather long glow (about 1 h after 10 min of sonolysis).

The residual CL intensity depends on the sonolysis duration and the oxygen content in xylene during sonolysis. The maximum initial intensity is observed after approximately 10 min of sonolysis. If sonolysis lasts for a longer time (without additional purging with air or oxygen), the initial intensity of residual CL decreases. Probably, this is due to a decrease in the concentration of oxygen dissolved in the liquid. The initial afterglow intensity increases upon solvent saturation with air or oxygen during sonolysis (or before sonolysis).

Several photoreactions contribute to the residual CL of xylene: recombination of short-lived radicals and the CL of rather stable molecular products, for example, the thermal decomposition of 1,2-dioxetanes or polymer peroxides according to the assumption.[17,26] In these studies devoted to the thermoinitiated oxidation of methylbenzenes and olefins, the radical and molecular CL components were distinguished and analyzed. According to these concepts, the reaction of addition of a singlet oxygen to unsaturated compounds can lead to peroxides. The introduction of a radical inhibitor into xylene after sonolysis (ionol, 10^{-2} mol L^{-1}) induces an insignificant decrease in the afterglow intensity and, according to our estimates, the fraction of radical CL in xylene afterglow is about 10%. For thermal oxidation, the fractions of the molecular and radical CL components are 75% and 25%, respectively.[17]

The kinetic curve of xylene afterglow is not rectified in the coordinates of the first- and second-order equations. However, as shown by mathematical processing, this curve can be presented as a sum of several components corresponding to the processes described by the reaction equations of the first (monomolecular decomposition) and second (recombination of radicals) orders. The molecular component with the rate constant $k_{M_1} = (3\pm0.5)\cdot10^{-3}\ s^{-1}$ is the longest one. The second molecular component is characterized by the rate constant $k_{M_2} = (3.6\pm1)\cdot10^{-2}\ s^{-1}$. It is most likely that they can be attributed to the thermal decomposition of two peroxide products. The duration of the fastest third component, which is remained after subtraction of two molecular components from the afterglow curve, is about 10 s, and its maximum intensity is ~10^4 photon s^{-1} mL^{-1}. This components is rectified (Fig. 4) in the coordinates of Eq. (2) for the second-order process

$$\sqrt{I_0/I} - 1 = t\sqrt{k_6 w_i}, \tag{2}$$

where I_0 and I are the initial and current afterglow intensities, respectively; and w_i and k_6 are the rate of formation (initiation) and the rate constant of recombination of peroxy radicals, respectively, in the known[27] scheme of chemiluminescence liquid-phase oxidation of unsaturated hydrocarbons (the CL intensity is proportional to the squared concentration of the radicals)

$$RO_2^{\cdot} + RO_2 \longrightarrow ROOOOR \xrightarrow{k_6}$$

$$\longrightarrow ROH + R'{=}O\ (R'{=}O^*) + O_2\ (^1O_2^*).$$

At $k_6 = 3\cdot10^8$ L mol^{-1} s^{-1} (see Ref. 26), the initiation rate determined from the slope angle of the straight line in Fig. 4, w_i, is (2—6)$\cdot10^{-10}$ mol L^{-1} s^{-1}. At this initiation rate, the quasi-stationary concentration of radicals is achieved during sonolysis and, correspondingly, the initial radical concentration $[RO_2^{\cdot}]_0$ is ~10^{-9} mol L^{-1} after sonolysis cessation.

It follows from the analysis of the obtained data that the MBSL of xylene is caused, to a considerable extent, by its oxidation *via* the radical-chain mechanism. In the gas phase of cavitation bubbles and in the bulk solution, the main SCL emitters are the excited molecules of 3-methylbenzaldehyde and singlet oxygen.

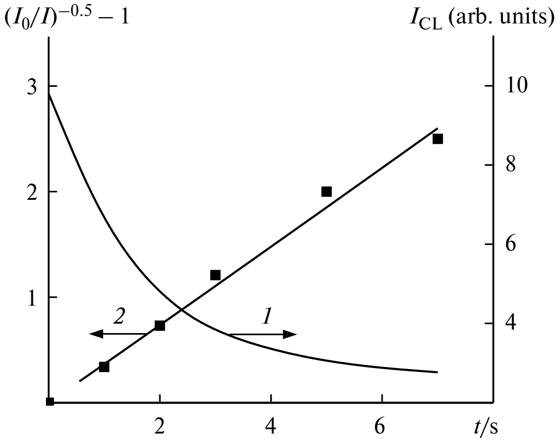

Fig. 4. Kinetics of a decrease in the radical CL intensity of xylene after switching-off untrasonication (*1*) and the corresponding linear anamorphosis (*2*).

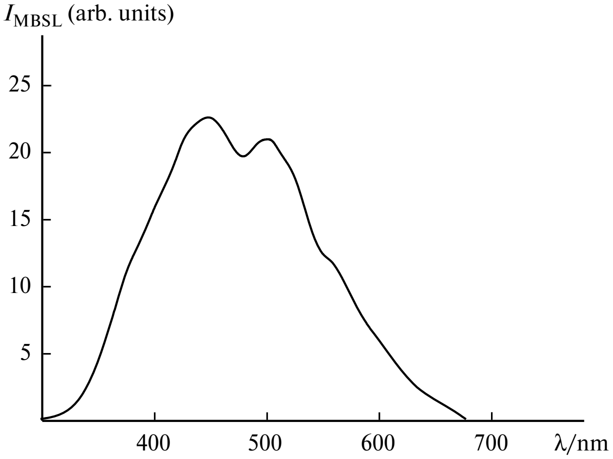

Fig. 5. The MBSL of a naphthalene melt detected on an Aminco spectrofluorimeter.

Another interesting example of SCL of liquid aromatic hydrocarbons is the MBSL of a naphthalene melt observed at rather high (compared to the known MBSL of other liquids) temperatures of 110—120 °C. Evidently, the high temperature increases the efficiency of oxidation processes. The MBSL intensity of naphthalene is more than twofold higher than that of water at ~25 °C (see Table 1). The MBSL spectrum of naphthalene contains two bands at 450 and 500 nm, which can be assigned to triplet-excited naphthalene molecules and naphthalene excimer[16] (Fig. 5).

Passing oxygen through the melt during sonolysis increases the MBSL intensity by 7 times. After cessation of O_2 feeding, the glow intensity decreases. When the melt is purged with argon, after oxygen saturation the MBSL intensity decreases sharply and becomes equal to the glow intensity before gas feeding. Upon the repeated oxygen feeding, the glow intensity increases again by approximately 6—7 times.

The residual CL is observed at a constant temperature of 110 °C after cessation of sonolysis of naphthalene and other aromatic hydrocarbons.

Thus, the SCL related to the oxidation reactions of aromatic hydrocarbons introduces the main contribution to the luminescence intensity during sonolysis, for example, for xylene saturated with oxygen. Sonochemiluminescence can result in a considerable afterglow of these liquids.

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