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Chemiluminescence from the decomposition of alkyl hyponitrites in poly(methyl methacrylate) and polystyrene

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Abstract

The kinetics of decay of chemiluminescence (CL) from the decomposition of 1-phenylethyl hyponitrite (PEH) and isopropyl hyponitrite (IPH) in poly(methyl methacrylate) (PMMA) and polystyrene (PS) films was investigated in nonisothermal and isothermal experiments. The measured rate constants for decay varied by about 20% with changes in initial concentration, and were about 50% slower than rate constants calculated from solution-based activation parameters. The luminescence yields from the hyponitrites in polymer matrices were lowered with increasing hyponitrite concentration, and also by the presence of low-molecular-weight residues or, most spectacularly, by dissolved oxygen. The photosensitized destruction of IPH by 9,10-dibromoanthracene (DBA) in PMMA was demonstrated. © 1997 Published by Elsevier Science S.A.

Keywords: Alkyl hyponitrites; Kinetics of chemiluminescence; Polymer matrix

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1. Introduction

In papers on the origin of chemiluminescence (CL) from oxidized polymers, the following light-emitting reactions have been suggested:
(a) Disproportionation of secondary or primary peroxyl radicals [1–3]

$$R_1R_2CHOO\cdot + R'OO\cdot \rightarrow \rightarrow R'OH + O_2 \rightarrow R_1R_2C{=}O + h\nu$$

(b) Disproportionation of alkoxyl radicals [4]

$$R_1R_2CHO\cdot + R'O\cdot \rightarrow \rightarrow R'OH + R_1R_2C{=}O + h\nu$$

Electronically excited carbonyl compounds formed in these reactions are usually assumed to be emitters of visible light.

In the autoxidizing system, the above reactions are termination steps of a free radical chain reaction. The radical concentrations in an autoxidizing system are locally at approximately steady-state values. The rate of luminescence for such a system undergoing reaction (a) above is proportional to the rate of termination, which at steady-state is kinetically equivalent to the unknown rate of initiation.

To simulate this process in a system with known rates of initiation, we have studied chemiluminescence from polymers containing relatively simple low-molecular-weight initiators, for which the correspondence between the kinetics of the decomposition in solution and light emission has already been elucidated [4]. In this paper, we used isopropyl (IPH) and 1-phenylethyl (PEH) hyponitrites as precursors of a geminate alkoxy radical pair in a solvent cage providing a chemiluminescent self-reaction.

$$R_1R_2CHO{-}N{=}N{-}OCHR_1R_2 \rightarrow \rightarrow [2R_1R_2CHO\cdot]_{cage} \rightarrow R_1R_2C{=}O^* + R_1R_2CHOH \quad (1)$$

In a previous paper [5], the chemiluminescence from alkyl hyponitrites in *tert*-butylbenzene and also in increasingly viscous solution was studied. In the present paper, the kinetics of chemiluminescence from IPH and PEH was investigated in poly(methyl methacrylate) (PMMA) and polystyrene (PS) matrices in order to obtain a better understanding of the phenomena under the transition from liquid to solid media. The kinetic measurements were performed either under nonisothermal conditions of linearly programmed increase of temperature, or isothermally at temperatures from 37 to 60 °C, with or without 9,10-dibromoanthracene (DBA) as a fluorescent triplet energy acceptor.

2. Experimental

Poly(methyl methacrylate) (Röhm und Haas Co.) and polystyrene (Krasten 120, MW 1.4×10^5) were purified by

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precipitation from benzene solution with methanol. 9,10-Dibromoanthracene (Aldrich) was purified by recrystallization from ethanol. The synthesis of the hyponitrites was described previously [6]. IR measurements were made with a Specord M-80 instrument.

2.1. Film preparation

The hyponitrite was dissolved in dichloromethane solution of polymer (100 mg polymer per ml of solvent) with or without DBA activator and spread over a Petri dish ($d = 3.5$ cm). After standing for 2 h, the solvent was largely evaporated and residual solvent was removed at 0.01 torr for 6 h at room temperature. The film thickness was 100 and 150 μm for nonisothermal and isothermal experiments, respectively. The initial concentration of hyponitrite in some films was determined independently by extracting it with n-heptane, followed by UV measurements at 224 nm. It was found that with ±5% error the initial concentration corresponded to the initial weight of hyponitrite. Control experiments revealed that the CL intensity decreased about 3% during film preparation. Since IPH is somewhat volatile, evaporative losses may play a role for that hyponitrite but not for PEH.

2.2. Chemiluminescence measurements

Isothermal experiments were performed on the photon counting device Biolumat LB 9500 (Berthold). Films of polymer were kept in a special holder placed directly into the reaction cuvette. The measurements were carried out in a given atmosphere, at a gas flow of 30 l h^{-1}. Dynamic measurements with a linearly increasing temperature of the sample were carried out with films cast on a Petri dish, which was inserted into a special heating chamber of the equipment. The emitted light was transmitted to the photomultiplier of the chemiluminescence device Lumat LB 9501 via 1.5 m optical cable. The light intensity expressed in counts per second vs. time or temperature was stored and processed with a computer.

3. Results and discussion

3.1. Chemiluminescence–time isothermal curves for decomposition of alkyl hyponitrites in a polymeric matrix

A particularly distinct maximum can be observed when investigating the chemiluminescence–time runs at temperatures of 45 °C and lower (Fig. 1). The maximum for isopropyl hyponitrite, observable even in a nitrogen atmosphere, occurred at much longer times than that for 1-phenylethyl hyponitrite, presumably because the decomposition rate of the latter was faster. The presence of the activator (DBA) in PMMA shifted this maximum to shorter times, which may be accounted for by rapid triplet energy transfer to the fluorescer, rendering the system less sensitive to variation in quenching. In PS films the maximum appeared at early times, comparable with the PMMA sample containing DBA.

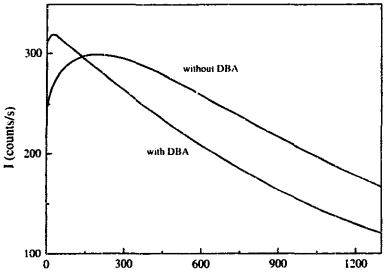

Fig. 1. Chemiluminescence curves for isopropyl hyponitrite decomposition in PMMA with DBA (IPH = 0.13 mol kg^{-1}; DBA = 4.3 mmol kg^{-1}) and without DBA (IPH = 0.18 mol kg^{-1}) at 45 °C in air.

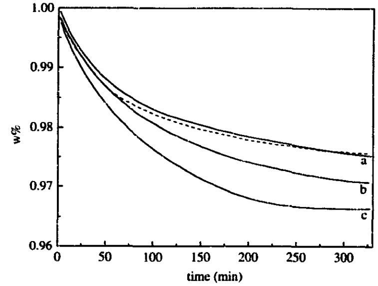

Fig. 2. Thermogravimetric curves for isopropyl hyponitrite in PMMA at 50 °C. Concentration (mol kg^{-1}): (a) 0.1; (b) 0.27; (c) 0.39. Dashed line corresponds to the PMMA film without hyponitrite.

This phenomenon was also seen in the chemiluminescence from the decomposition of tetramethyl-1,2-dioxetane in PMMA and PS [7], which like IPH gives excited acetone. The maximum was ascribed to the evaporation of residual amounts of solvent present in the polymer film, which gradually changes the conditions for photophysical processes involving competitive quenching of excited carbonyls. The loss of weight was revealed by thermogravimetric analysis of PMMA films containing various amount of IPH at 50 °C (Fig. 2). Apparently, part of the weight loss is due to volatilization of IPH major decomposition products (acetone, 2-propanol, nitrogen).

Thus, only the decaying part of the chemiluminescence curve was used for evaluation of first-order rate constants, summarized in Tables 1–5.

Although the emission was too weak to measure the spectral distribution in a conventional fashion, we showed with cutoff filters that the rate constant for decay of CL from IPH in PMMA was independent of the spectral window

Table 1
Measurement of chemiluminescence with colour filters from isopropyl hyponitrite in PMMA film at 80 °C[a]

Transmission λ/nm	$k_d \times 10^3/s^{-1}$
400–480	1.06
480–570	1.11
560–600	1.04

[a] Initial concentration of IPH 1.1 mol kg^{-1}.

Table 2
Rate constants for chemiluminescence decay of isopropyl hyponitrite decomposition in PMMA at 45 °C

$C_{IPH} \times 10$/mol kg^{-1}	$k_d \times 10^5$ (air)/s^{-1}	$k_d \times 10^5$ (N_2)/s^{-1}
0.1	1.28	1.05
1.8	1.24	1.03
3.6	1.06	–
4.9	1.07	0.84

Table 3
Rate constants for chemiluminescence decay of decomposition of 1-phenylethyl hyponitrite in PMMA and PS

T/°C	$C_{PEH} \times 10^2$/mol kg^{-1}	Gas	$k_d \times 10^4$/s^{-1}
PMMA			
37	0.29	N_2	0.33
	0.29	O_2	0.29
	0.63	air	0.31
	0.64	air	0.30
	0.74	air	0.25
	1.4	air	0.29
	1.6	air	0.25
			av. 0.29±0.02
45	0.66[a]	air	1.01
50	0.57	air	1.76
	0.62[a]	N_2	1.44
	0.62[a]	O_2	1.35
60	0.62[a]	N_2	5.67
	0.57	air	7.39
PS			
37	0.62[a]	air	0.42
	0.69	air	0.43
60	0.63[a]	air	5.73

[a] With DBA.

Table 4
Rate constants for chemiluminescence decay of IPH decomposition in PMMA (0.13 mol kg^{-1}) in the presence of activator (DBA) at 45 °C in air

$C_{DBA} \times 10^3$/mol kg^{-1}	I_{max}/counts s^{-1}	$k_d \times 10^{-5}$/s^{-1}
0.5	315	1.24
1.8	628	1.43
2.7	888	1.50
4.5	1636	1.54

(Table 1). This experiment was prompted by the results of Phillips et al. [8], who showed that for CL initiated by dicyclohexyl peroxydicarbonate in PS, two emitters were present with one decaying faster than the other. Unfortunately, the emission from hyponitrites in PS films in this work was too weak to measure with sufficient precision through a cutoff filter.

Table 5
Influence of antioxidant on decay kinetics of hyponitrites in PMMA

Hyponitrite/ mmol kg^{-1}		Antioxidant[a]/ mmol kg^{-1}	$k_d \times 10^5$/s^{-1}	T/°C	I_{max}/ counts s^{-1}
IPH	100	0	1.24	45	252
	100	37	1.09	45	168
	110	0	2.42	50	401
	110	37	2.36	50	249
PEH	10	0	2.81	37	1270
	10	30	2.81	37	1310

[a] Irganox 1076 (octadecyl 3,5-di-*tert*-butyl-4-hydroxycinnamate).

The kinetics from a 20 μm film of PMMA, initially 2.5 mmol kg^{-1} IPH in PMMA at 60 °C, was measured by decay of CL ($7.6 \pm 0.8 \times 10^{-5}$ s^{-1}) and by IR at 990 cm^{-1} ($7.3 \pm 0.8 \times 10^{-5}$ s^{-1}), showing good correspondence between the two independent methods.

3.2. The effect of atmosphere

As demonstrated in Fig. 3, the chemiluminescence emission accompanying the decomposition of the hyponitrites was reduced to about half in the presence of oxygen when compared with experiments in a nitrogen atmosphere. The reduction was less than 20% when air was substituted for oxygen in the experiment. The quenching effect of oxygen on triplet states of ketones is known [9], and similar results have been observed in the CL from dioxetanes in solution [10]. However, in autoxidizing systems the oxygen is necessary for the production of excited carbonyl species from the hydrocarbon substrate and, in general, a dramatic decrease in CL is observed when the oxygen is replaced by an inert gas, and conversely [11,12].

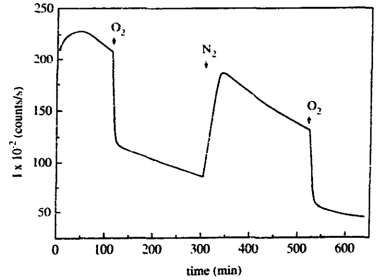

Fig. 3. Effect of the atmosphere on the intensity of chemiluminescence at the decomposition of 1-phenylethyl hyponitrite (2.9 mmol kg^{-1}) in PMMA at 37 °C.

3.3. DBA-photosensitized decomposition of IPH

A PMMA film containing IPH (10 mmol kg^{-1}) and DBA (4.5 mmol kg^{-1}) was irradiated at 20 °C for 280 min at 366 nm with the output from a medium-pressure, 125 W Hg lamp (at a distance of 8 cm). The CL before and after irradiation was 750 ± 17 and 524 ± 14 counts s^{-1}, respectively. The corresponding emission intensities for a second film without DBA, irradiated in the same way, were 92 ± 2 and 98 ± 3 counts s^{-1}, respectively.

3.4. Effect of polymer matrix on decomposition of hyponitrites

The rate constants for the hyponitrites in polymers were lower than values calculated from Arrhenius parameters obtained in solution (*tert*-butylbenzene). For instance, at 45 °C the fastest measured rate constant (Table 2) for IPH in PMMA corresponds to a half-life of 23 h, compared to the half-life [13] in *tert*-butylbenzene of 9.1 h at the same temperature. Similarly, the rate constants for decay of PEH in PMMA at 37 °C (2.9×10^{-5} s^{-1}, Table 3) and in PS (4.3×10^{-5} s^{-1}) were both lower than the calculated value in *tert*-BuPh at that temperature (5.1×10^{-5} s^{-1}).

3.5. Effect of initiator concentration

The effect of the initial hyponitrite concentration on the observed rate constant for chemiluminescence decay was mixed. For IPH in PMMA, rate constants decreased with increasing concentration (Table 2). For PEH in PMMA they stayed about the same (Table 3), while for IPH in the presence of DBA they increased about 50% (Table 4). Experiments carried out in the presence of the phenolic antioxidant Irganox 1076 (Table 5) showed that the antioxidant did not change the rate constant for decay, but lowered the amount of light emitted by up to 40%.

The slower rate of decomposition of hyponitrites in polymers compared with the rate in solution may be caused by an increased cage effect involving the radical pair arising from a single N–O bond scission in the decomposition of the hyponitrites. A higher degree of self-reaction of the alkoxyl pairs in the polymeric media compared with fluid media will also result in a lower extent of induced decomposition of hyponitrite by cage-escaped radicals since the fraction of those cage-escaped radicals will be lower. The fact that Irganox 1076 did not change the observed rate constants in the polymeric system is clear evidence that radical-induced decomposition is unimportant under these particular conditions, and that variations in k_d are due to evaporative losses or other factors.

The plot of the area under the CL vs. IPH concentration curves, however, deviated from linearity (Fig. 4), in a way consistent with self-quenching at higher concentrations. The amount of self-quenching, and hence rate of loss of IPH by this mechanism, will be higher as the hyponitrite concentration increases.

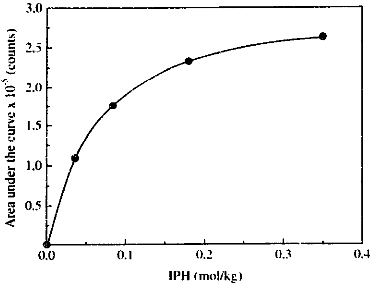

Fig. 4. The dependence of the area under the first peak of the nonisothermal chemiluminescence curve for isopropyl hyponitrite. Data from Fig. 6.

The induced decomposition of IPH via triplet sensitization may also lead to an increased value of the rate constant of HN decomposition (see Table 4) if the excited state products represent a significant fraction of the carbonyl products, most of which appear in the ground state. In *tert*-butylbenzene, the yield of triplet acetone per reacting alkoxyl pair may, in fact, be as high as 20% [14]. In viscous media, the fraction of hyponitrite undergoing cage reactions will be large, and the fraction leading to cage-escaped radicals correspondingly small.

3.6. Nonisothermal chemiluminescence–temperature experiments

The nonisothermal chemiluminescence curves (I vs. T) of PEH in PMMA (Fig. 5) were analyzed by nonlinear regression using an equation valid for nonisothermal chemiluminescence from first-order reactions [7]:

$$I = P\exp(-E_a/RT)\exp\int^{T} -(A/\beta)(\exp(-E_a/RT))\,\mathrm{d}T \tag{2}$$

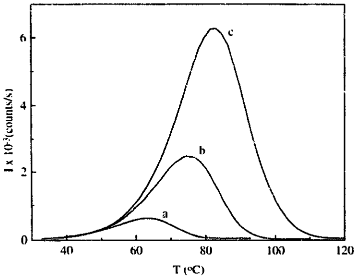

Fig. 5. Nonisothermal chemiluminescence of 1-phenylethyl hyponitrite decomposition in PMMA (6.3 mmol kg^{-1}) in the air, DBA = 4.3 mmol kg^{-1}. Heating rates (°C min^{-1}): (a) 0.5; (b) 2; (c) 5.

Table 6
The activation energy and preexpenontienal factor for isopropyl hyponitrite decomposition obtained from nonisothermal chemiluminescence experiment

	$C_{IPH}/\times 10^2$ mol kg^{-1}	$C_{DBA}/\times 10^2$ mol kg^{-1}		K/min^{-1}	T_{max}/°C	I_{max}/counts s^{-1}	E_a/kJ mol^{-1}	log A/s^{-1}
PMMA								
	0.9	0.43	air	1	84.2	1245	109.3	13.12
	0.9	0.43	air	1	84.6	1306	108.6	13.06
	0.9	0.43	air	2	91.1	2947	112.3	13.63
	0.9	0.43	air	2	92.2	2850	111.2	13.42
	0.9	0.43	air	5	100.7	6940	109.5	13.17
	0.7	0.43	N_2	1	84.4	1396	105.7	12.65
	0.7	0.43	N_2	2	93.2	3007	103.8	12.26
	0.7	0.43	N_2	5	110.3	6319	82.9	9.11
	3.6	0	air	2	92.1	466	111.7	13.50
	8.4	0	air	2	90.0	737	112.8	13.66
	18.0	0	air	2	92.9	1024	121.4	14.87
	35.0	0	air	2	92.5	1205	125.7	15.47
PS								
	0.9	0.43	air	1	82.1	2192	104.5	12.60
	0.9	0.43	air	2	89.0	2915	103.8	12.48
	0.9	0.43	air	2	88.6	3016	103.3	12.42
	0.9	0.43	air	5	96.5	5632	103.0	12.43
	0.9	0.43	air	5	97.7	6915	105.5	12.75
	0.18	0.43	N_2	2	91.3	7367	100.0	11.80
	0.18	0.43	N_2	5	99.0	15382	94.6	11.02
	0.7	0.43	N_2	2	91.2	1532	101.0	11.93
	2.9	0.43	N_2	2	90.8	3621	95.2	11.07
	13.0	0.43	N_2	2	93.5	6620	85.0	9.35

where E_a denotes activation energy, A the preexponential factor and P a parameter proportional to the area under the CL curve. The evaluated activation parameters for decomposition of IPH and PEH in PMMA and PS are given in Tables 6 and 7. As may be seen from these tables, the maximum CL intensity of PEH is situated at lower temperatures than for IPH. This directly indicates the lower thermal stability of PEH compared to IPH.

A second maximum appeared on the nonisothermal CL curves of decomposition of IPH in PMMA at about T_{max} = 124 °C in air (Fig. 6). The peak was attributed to the chemiluminescence accompanying the decomposition of peroxidic compounds formed in the PMMA matrix during the decomposition of the initiator.

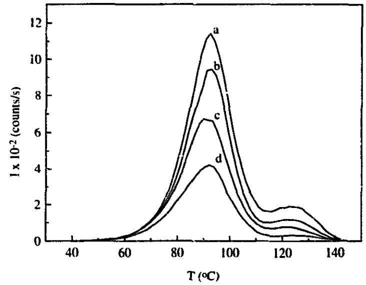

Fig. 6. Nonisothermal chemiluminescence of isopropyl hyponitrite decomposition in PMMA without the presence of an activator in the air; heating rate 2 °C min^{-1}; concentration (mol kg^{-1}): (a) 0.35; (b) 0.18; (c) 0.08; (d) 0.04.

3.7. Activation parameters

Activation parameters obtained by different methods are summarized in Table 8. The data from nonisothermal runs are more extensive, but the values under a nitrogen atmosphere were prone to large variations and have a large standard deviation. The most reproducible results, in air in a PMMA matrix, show E_a and log A that are little different from those in solution.

The activation energies evaluated from nonisothermal experiments are, however, expected to be lower than the conventionally obtained solution value because the former includes a term for the temperature dependence of the yield of photons from the hyponitrite. This term is expected to be negative, since cage-escape of alkoxyls will be more efficient as the temperature rises, so that fewer of them will react to give excited states. Moreover, the apparent activation energy for fluorescence emission from DBA in the temperature range of this study is also negative, and is about −20 kJ mol^{-1} [10].

If the latter value is added to the values of E_a in Table 6 obtained nonisothermally in the presence of DBA, the corrected E_as are higher than the solution values, and the difference is well outside error limits. The three sets of isothermal data also lead to this conclusion when the same correction for DBA fluorescence is made. A higher E_a is, in fact, expected in the more viscous medium because the radical

Table 7
The parameters of nonisothermal chemiluminescence measurements of phenylethyl hyponitrite with 9,10-dibromoanthracene (4.3×10^{-3} mol kg^{-1}) in poly(methyl methacrylate) (PMMA) and in polystyrene (PS)

	$C_{PEH}\times10^2$/mol kg^{-1}		K/min^{-1}	T_{max}/°C	I_{max}/counts s^{-1}	E_a/kJ mol^{-1}	log A/s^{-1}
PMMA							
	0.63	air	0.5	62.9	636	105.9	13.42
	0.63	air	2	74.5	2478	110.7	14.16
	0.63	air	2	74.8	2112	110.2	14.10
	0.63	air	5	83.0	6276	109.4	13.98
	0.63	air	5	84.3	5554	104.9	14.19
	0.63	N_2	1	69.8	1257	109.2	13.90
	0.63	N_2	2	76.5	2345	108.9	13.84
	0.63	N_2	5	87.6	6994	84.3	9.97
	0.47	N_2	2	74.4	2049	109.9	14.03
	1.7	N_2	2	76.1	5647	111.2	14.20
	3.6	N_2	2	75.7	10557	107.9	13.68
PS							
	0.63	air	0.5	63.0	765	104.6	13.25
	0.65	air	1	68.2	866	107.8	13.78
	0.65	air	1	68.1	881	104.8	13.26
	0.65	air	2	73.1	1500	110.0	14.12
	0.65	air	2	75.2	1386	106.3	13.51
	0.65	air	5	82.6	3342	110.2	14.12
	0.65	air	5	81.7	3403	110.2	14.16
	0.71	N_2	1	68.9	1386	110.2	14.11
	0.71	N_2	2	75.2	3884	109.0	13.93
	0.71	N_2	5	84.1	7093	99.1	12.33

Table 8
Activation parameters of chemiluminescence for decomposition of IPH and PEH from various sources

Medium	IPH:log A/s^{-1}	E_a/kJ mol^{-1}	PEH:log A/s^{-1}	E_a/kJ mol^{-1}
Liquid				
PhBu-t[a]	13.71 ± 0.7	112.0 ± 0.4	13.49 ± 0.1	105.6 ± 0.8
Polymer, nonisothermal				
PMMA[b], air	13.8 ± 0.8	114 ± 6	–	–
PMMA[b], air, DBA	14.0 ± 0.3	108 ± 6	14.0 ± 0.3	109 ± 2
PMMA[b], N_2, DBA	11.3 ± 1.6	98 ± 10	12.6 ± 1.8	101 ± 12
PS[b], air, DBA	12.5 ± 0.1	104 ± 1	13.7 ± 0.4	108 ± 2
PS[b], N_2, DBA	11.0 ± 0.9	95 ± 6	13.5 ± 0.8	106 ± 5
Polymer, isothermal				
PMMA[c], air	14.9	121	16.1	123
PS[d], air, DBA	·	–	12.1	98

[a] Data from [13]. Error limits are standard deviations.
[b] Calculated from data in Tables 6 and 7.
[c] Calculated from data in Tables 2 and 3.
[d] Calculated from two data points in Table 3.

fragments from single N–O bond scission in the hyponitrite must diffuse apart sufficiently to avoid re-formation of the initiators, after which the nitrogen-centered radical will decompose to give the second alkoxyl. In somewhat simplified form, we have:

$$HN \underset{k_2}{\overset{k_1}{\rightleftharpoons}} [RON=N\cdot + \cdot OR]_{cage} \xrightarrow{k_3} 2RO\cdot + N_2$$

The observed rate of decomposition for HN will have an activation energy $E_a = E_1 - E_2 + E_3$. If the process corresponding to k_2 occurs within the primary solvent cage, E_2 should be near zero, whereas E_3 will be a substantially positive activation energy for diffusion of a molecular fragment in the polymeric medium. A higher E_a in polymers than in fluid solution is therefore expected, corresponding in this approximation to $E_1 + E_3$.

4. Conclusions

The direct chemiluminescence from disproportionation of alkoxy radicals is observable in PMMA and PS matrices. The first-order decays show significantly lower rates than in solution, which is ascribed to the cage-effect on the primary radical pair in the highly viscous media. The variation in measured rate constants with changes in initiator concentration are probably due to induced modes of decomposition and evaporative losses.

The most striking difference between the system under study here and the polymer undergoing oxidation, which the system was intended to model, is the dramatic decrease of CL from polymer/hyponitrite combination upon changing from an inert to an oxygen-containing atmosphere. The opposite effect commonly observed in oxidizing hydrocarbons is consistent with the requirement of the presence of oxygen for the luminescent peroxyl termination (reaction (a) above).

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