Theoretical approach to lyoluminescence of organic materials

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Considering the mechanism of lyoluminescence (LL) in organic materials, an expression is derived for LL intensity, which indicates that, when the organic material is dissolved into the solution, then, initially, the LL intensity should increase linearly with time, attain a peak value and then it should decrease exponentially with time. Expressions are also derived for the time t0 at which, the LL corresponding to the time t0, and for the total LL intensity \( I_T \). From the decay of LL intensity, the value of the rate constant \( \sigma \) for the decay of alkyl radical concentration is determined and it is found to be 0.49 and 1.72 sec\(^{-1}\) for saccharide and mannose. These values give that, the decay time of alkyl radical concentration should be 2.04 and 0.87 sec for saccharide and mannose. From the values of \( t_0 \) and \( \sigma \), the value of the rate constant \( (\alpha+\lambda) \) for the decay of peroxy radical concentration is determined and it is found to be 1.0 and 2.3 sec\(^{-1}\) for saccharide and mannose respectively. These values of rate was constant which indicates that, decay-time of peroxy radical concentration should be 0.62 and 0.43 sec for saccharide and mannose respectively. A good agreement is found between the theoretical and experimental results.

[Key words: Aqualuminescence, Lyoluminescence, Organic materials, Radicals]

1 Introduction

Lyoluminescence (LL), the emission of light during dissolution of previously irradiated solids, has found many applications in the field of radiation research. It is quite a general phenomenon occurring with many materials, but most of the systematic studies turned to the dosimetry application have been concentrated on alkali halides and on two classes of organic compounds regarded as tissue equivalents, namely, saccharides and amino acids\(^1\). Realizing its possible application as a dosimeter, considerable activities on lyoluminescence are noted in the 1970s. Most of the experiments, however, were carried out, using organic substances due to their similar composition with human tissues and with a hope to develop a tissue-equivalent dosimeter.

Chandra et al.\(^3\) have reported the theory of the LL of alkali halides. Chatterjee et al.\(^4\) have previously reported the theory of the LL of organic substances, whereby, they have discussed the dependence of total LL intensity on the radiation doses given to the samples. The present paper reports the theory of the dependence of LL intensity of organic materials on different parameters and makes a comparison between the theoretical and experimental results.

2 Mechanism of LL in Organic Materials

On \( \gamma \)-rays irradiation, energetic electrons deposit energy in the sample and free radicals are formed on breakage of chemical bonds of the sample. Although the exact process of the light emission has not yet been positively identified, it has been known that, free radicals thus formed, play a key role\(^*\) in LL. Of all possible schemes to explain LL, the Russell-Vassil'ev (RV) scheme\(^7\) seems to be capable of explaining many of the features of LL. According to the R-V scheme, alkyl radicals are produced in the sample during irradiation, which are then oxidized to form peroxy radicals on dissolution. These then combine to form excited carbonyls (triplet), which during transition to ground state, emit light in the wavelength range 397-510 nm. De-excitation is, however, one of the several mechanisms by which, the peroxy radicals take part in the emission of light, the details of which have been discussed by Ettinger & Puite\(^6\). According to them, the oxygen present in the sample is responsible for oxidation of the alkyl radicals.
In LL, the entire process takes place in two distinct stages, one in the solid phase of the sample during irradiation and storage and the other in the liquid phase during dissolution. The entire process can be represented by a rate kinetic model schematically shown below:

Here, $A'$ is the excited molecule of the sample (A) produced with a frequency $\Gamma$ on the irradiation, which then may break with a frequency $\mu$ to form alkali radicals ($B'$) or may quench back with a frequency $\delta$ ($B'$ may also decay at a rate $\alpha$). Also, during irradiation, oxygen present in the sample is dissociated at a frequency $\Gamma$, to form reactive nascent oxygen ($O'$), which is lost in subsequent reactions. Both $\Gamma$ and $\tau$ are proportional to the irradiation dose rate. The solid sample after irradiation for a time $T$ is usually stored for time (say $\tau$) before dissolution.

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**3 Theory**

**3.1 Process during irradiation and storage (in solid phase)**

In this stage, ionizing radiations produce excited molecules ($A'$) directly from the ground state...
molecule (A) which may either revert back to the ground state, without undergoing a chemical change or dissociate creating alkyl radicals (B') in the following way:

\[
\begin{array}{c}
A \xrightarrow{\gamma} A^* \xrightarrow{\mu} B' \\
(\text{decay product})
\end{array}
\]

A portion of these alkyl radicals may decay by recombination reaction indicated by the bimolecular loss frequency \(\alpha\). Along with this, some of the oxygen present in the sample is dissociated forming reactive nascent oxygen, which takes part in subsequent reactions, thereby, removing it from the scene as shown below: \(O_2 \rightarrow O^*\).

These reactions may be described by the following rate equations:

\[
\begin{align*}
\frac{d}{dt}[A^-] &= \Gamma[A] - (\delta + \mu)[A^*] \\
\frac{d}{dt}[B'] &= \mu[A^*] - \alpha[B'] \\
\frac{d}{dt}[O_2] &= -r[O_2]
\end{align*}
\]

where \(\delta, \mu\) and \(r\) are rate frequencies of quenching the excited molecule \(A^*\), dissociation of \(A^*\) producing alkyl radicals, and the dissociation of oxygen molecules present in the sample due to irradiation. Since, even at the highest dosage commonly used for irradiation, a negligible fraction of the parent molecule would be affected by the irradiation, it has been approximated to be a constant. The solution of Eq. (1) can be obtained in the following way.

From Eq. (1), we get:

\[
\frac{d}{dt}[A^-] + (\delta + \mu)[A^-] = \Gamma[A]
\]

Integrating Eq. (4) and taking \([A]=0\) at \(t=0\), we get:

\[
[A^*] = \frac{r[A]}{\delta + \mu}[1 - \exp[-(\delta + \mu)t]] \quad \ldots(5)
\]

Integrating Eq. (2) and taking \([Br]=0\) at \(t=0\), we get:

\[
[B'] = \frac{\mu r[A]}{(\delta + \mu)\alpha}[1 - \exp(-\alpha t)] - \\
\frac{\mu r[A]}{(\delta + \mu)(\delta + \mu + \alpha)}[\exp(-\alpha t) - \exp[-(\delta + \mu)t]]
\]

Integrating Eq. (3) and taking \([C]=0\) at \(t=0\), we get:

\[
[O_2] = [O_2] \exp(-YT)
\]

where \([O_2]_0\) is the oxygen concentration, initially present in the sample.

If \(T\) is the irradiation time, then Eq. (14) may be written as:

\[
[O_2] = [O_2]_0 \exp(-YT)
\]

It is to be noted that \(\Gamma\) and \(Y\) are proportional to the dose rate.

![Fig. 3 — Comparison of experimental results of mannose obtained from lyoluminescence studies with fitted values of Eq. 21 (after Ref. 5)](image-url)
Usually, the sample after irradiation is stored for a considerable period \( \tau \) (about a month), before dissolution. During this post-irradiation storage, the reaction proceeds in the following way:

\[
\begin{align*}
A & \xrightarrow[\gamma]{} A^* + \mu \xrightarrow[]{} B' \\
& \text{(decay product)}
\end{align*}
\]

The above reactions may be represented by the following rate equations:

\[
\frac{d}{dt}[A^*] = -[\mu + \delta] [A^*] 
\]

and

\[
\frac{d}{dt}[B'] = \mu[A^*] - \alpha[B']
\]

The solutions of Eqs (9) and (10) are given by:

\[
[A^*] = [A^*]_0 \exp[-(\mu + \delta) \tau]
\]

and

\[
[B'] = [B']_0 \exp[-\alpha \tau] + \frac{\mu[A^*]_0 \exp[-\alpha \tau]}{[\mu + \delta - \alpha]} \left[\exp[-\alpha \tau] - \exp[-(\mu + \delta) \tau]\right]
\]

Using Eqs (5) and (6), Eq. (12) gives the alkyl radical concentration, just prior to dissolution, and it may be given by:

\[
[B'] = \frac{\mu [A] \exp[-\alpha \tau]}{\alpha [\delta + \mu - \alpha]} \left[1 - \exp[-\alpha \tau]\right] - \frac{\mu [A] \exp[-(\delta + \mu) \tau]}{[\delta + \mu - \alpha] [\delta + \mu]} \left[1 - \exp[-(\mu + \delta) \tau]\right]
\]

The unit of time has been conveniently chosen in the units of the dose received (Gy).

3.2 Process during dissolution (liquid phase)

During dissolution, the alkyl radicals \( B' \) combine with the oxygen present in the sample, forming peroxy radicals \( BO' \), which, either subsequently, recombine forming the excited carbonyls (triplet) which emit light \( L \) during transition to ground state, or take part in other light emitting processes, as represented by the following reaction scheme:

\[
B' \xrightarrow{k} BO' \xrightarrow[\lambda \nu]{} L \xrightarrow[\zeta \omega]{} \text{(decay Products)}
\]

where \( \zeta \) and \( \omega \) are the two and three body-loss rates, \( k \) the oxidation reaction rate constant, \( \xi \) the loss rate for the peroxy radicals, \( \lambda \) and \( \nu \) are the two and three body photo-chemical reaction frequencies. Rate equation associated with the above reactions may be represented as:

\[
\frac{d}{dt}[B^*] = -[\xi + k [O_2]] [B^*] - [\omega [B^*]]^2
\]
and
\[ \frac{d}{dt}[BO^*] = k \cdot [O_2] \cdot [B^*] - (\xi + \lambda)[BO^*] - \nu [BO^*] \]

...(15)

Generally, the three body-loss rate is small and Eq. (14) may be written as:
\[ \frac{d}{dt}[B^*] = -\sigma [B^*] \]

...(16)

where \( \sigma = \xi + k[O_2] \) is the rate constant for the decay of alkyl radical concentration.

Solution of Eq. (16) may be expressed as:
\[ [B^*] = [B^*]_0 \exp(-\sigma t) \]

...(17)

where \([B^*]_0\) is the radical concentration after a storage period \( \tau \), i.e., just prior to the dissolution, and given by Eq. (13).

\[ \frac{d}{dt}[BO^*] = k [O_2]_0 \exp(-Y \tau) [B^*]_0 \exp(-\sigma t) - (\xi + \lambda)[BO^*] \]

...(19)

Integrating Eq. (19) and taking \([BO^*]=0\) at \( t=0 \), we get:
\[ [BO^*] = \frac{k [O_2]_0 \exp(-Y \tau) [B^*]_0 \exp(-\sigma t) - \exp(-\xi t) - \lambda \exp(-\theta t)}{(\xi + \lambda - \sigma)} \]

...(20)

Fig. 5 — Effect of water temperature on LL for three saccharides (after Ref. 11)

Generally, the probability of three body photochemical reaction is small and the loss of peroxyl radical in non-emissive process is much larger than that in three body emissive process. Thus, Eq. (15) may be expressed as:
\[ \frac{d}{dt}[BO^*] = k [O_2] [B^*] - (\xi + \lambda)[BO^*] \]

...(18)

Substituting the values of \([O_2]\) from Eq. (8) and \([B^*]\) from Eq. (17) in Eq. (18), we get:
\[ I = \lambda [BO^*] \]

Fig. 6 — Dependence of LL of glutamine on the temperature of irradiation normalized at \( T = 22^\circ C \); 100 GY; temperature from 14.6 to 38.4 \( ^\circ C \); 100 GY; 14.6 to 39.8 \( ^\circ C \); 250 GY; 14.6 to 38.4 \( ^\circ C \); 250 GY; 14.6 to 38.4 \( ^\circ C \) (repeat); 250 GY; 14.6 to 39.8 \( ^\circ C \) (after Ref. 12)

Thus, the time dependence of LL intensity may be expressed as:
\[ I = \lambda [BO^*] \]
or 
\[ I = \frac{\lambda k [O_2]_0 \exp(-Y \tau) [B^*]_1 [\exp(-\sigma t) - \exp(-[(\xi + \lambda) \tau])]}{[(\xi + \lambda - \sigma)]} \]  
...(21)

(iii) Estimation of \( t_m \)

Eq. (21) indicates that, \( t=0 \), for \( t=\infty \), as well as for \( t=0 \) and for \( t=\infty \). Thus, the value of \( I \) should be maximum for a particular value of time \( t=t_m \), after the dissolution of the irradiated specimen. \( I \) will be maximum when \( dI/dt = 0 \). Thus, Eq. (21) gives:

\[ \sigma \exp(-\sigma t_m) = (\xi + \lambda) \exp(-[(\xi + \lambda) \tau]) \]

or \( t_m = \frac{1}{(\xi + \lambda - \sigma)} \ln \left( \frac{\xi + \lambda}{\sigma} \right) \)  
...(24)

(iv) Estimation of \( I_m \)

Substituting \( \exp(-\sigma t_m) = (\xi + \lambda) \exp(-[(\xi + \lambda) \tau]) \) in Eq. (21), we get:

\[ I_m = \frac{\lambda k [O_2]_0 \exp(-Y \tau) [B^*]_1}{[(\xi + \lambda - \sigma)]} \]  
...(25)

(v) Total intensity \( I_T \) of LL

From Eq. (21), the total LL intensity \( I_T \), i.e., the integrated area below the LL intensity versus time curve may be expressed as:

\[ I_T = \int_0^{t_m} \frac{\lambda k [O_2]_0 \exp(-Y \tau) [B^*]_1 [\exp(-\sigma t) - \exp(-[(\xi + \lambda) \tau])]}{[(\xi + \lambda - \sigma)]} \]  
... (26)
(vi) Dose dependence of LL intensity

Substituting the value of \([B^r]\), from Eq. (13) in Eq. (26), we get:

\[
I_1 = \frac{\lambda [O_2]_0}{\sigma(\xi + \lambda)} \times \frac{\mu [A] \exp (-\Delta \tau) \alpha(\delta + \mu - \alpha)}{[1 - \exp(-\alpha T)]} \\
\text{and} \quad \frac{\mu [A] \exp (-(\mu + \alpha) \tau)}{(\delta + \mu - \alpha)(\mu + \alpha)} \left[1 - \exp(-(\mu + \alpha) T)\right],
\]

...(27)

\[
\text{Fig. 9 — Quantum efficiency of chemiluminescence as a function of absorbed dose for irradiated glucose dissolved in the media at different pH (after Ref. 9).}
\]

As \(T\) is the time for the irradiation of material, it is evident from Eq. (27) that, initially, the total LL intensity \(I_1\) should increase linearly, with \(T\), and then it should attain a saturation value for longer duration of the exposure time \(T\).

3.4 Comparison between the theoretical and experimental results

Alkyl radicals \(B^r\) plays a key role in causing LL in organic materials. Eq. (13) indicates that, initially, the concentration of \(B\) radicals should increase linearly, with time and then it should attain a saturation value for longer duration of time. Chatterjee et al.\(^5\) have measured concentration of \([B^r]\) radicals from the ESR experiment. Fig. 1 shows the dependence of ESR data for lactose (a dose rate employed is 20 Gy min\(^{-1}\)), whereby, ESR data have been fitted with Eq. (13). The value of the parameters obtained by fitting Eq. (13) are as follows:

\[
\mu [A] = 6.3 \times 10^9 \text{ Gy}^{-2} \text{ g}^{-1} \\
\alpha = 2.78 \times 10^{-3} \text{ Gy}^{-1} \quad \text{and} \quad \tau = 195.6 \text{ Gy}
\]

Although, it is difficult to obtain the values of all the parameters, one still gets knowledge of the self-life \((\alpha^{-1})\) of alkyl radicals, life-time \([(\mu + \delta)^{-1}]\) of excited states \(A^*\) and the storage time \(\tau\). The knowledge of any one of the parameters is sufficient to obtain the values of the remaining parameters.

\[
\text{Fig. 10 — Changes in the LL yield with pH in various buffers (after Ref. 19).}
\]

Chatterjee et al.\(^5\) have fitted the ESR data for mannose (dose rate employed 330, 28 and 2.6 Gy min\(^{-1}\)) and sucrose (dose rate employed 12 Gy min\(^{-1}\)) available only in the linear region as in Figs 2 and 3 and they have shown that, the dependence of \(B^r\) on \(T\) can be expressed by the relation:

\[
\log ([B^r]) = \log(a) - b \log (T) \quad \ldots(28)
\]

where \([B^r]\) is the concentration of alkyl radicals, \(T\) is the dose time measured in units of dose received, \(a\) and \(b\) are constants. Fitting the data of mannose and sucrose, gives the values of the parameters, for mannose, as \(a=5.590 \times 10^4, b=0.9409\) and for sucrose, as \(a=7.603 \times 10^4, b=0.6264\). As the
experiment for mannose has been done at different
dose rates, mannose gives an excellent chance to
verify one of the interesting features of the proposed
model. Since the dose time is measured in units of
dose received, the fitting parameters would, in
general, is expected to be dose-rate dependent,
unlike what is observed for mannose. For a small
enough value of $\alpha T$ and $(\mu+\delta T)$, the exponential
terms in Eq. (13) may be expanded, retaining only
the first-order times in $T$. This on combining with
the rate frequency $\Gamma$, which is dose-rate dependent,
provides a linear dependence of $[B^r]$ on the dosage.
Hence, by the proposed model, the dose-rate
independence of the data for mannose demands that,
the parameter $b$ be unity, which is in fact observed.

Fig. 1 shows the time dependence of LL
intensity of mannose reported by Arnikar & Kalkar$^{10}$
and Puite & Ettinger$^{11}$. It is seen that, initially, the
LL intensity increases linearly, with time, attains a
peak value and then decreases with time. Fig. 2 shows
that, the plot of log of LL intensity versus
time is a straight line within the slope. From
the slope of this curve, decay-time of LL is estimated
and it is found to be 0.87 s for mannose. The values
of rate constants $\sigma$ and $\xi+\lambda$ have been calculated
from Fig. 1, using Eqs (23) and (24) and they are
found to be 1.25 and 2.3 s$^{-1}$, respectively for
mannose. As shown in Fig. 1, a good agreement is
found between the experimental and theoretical
results.

Figs 3 and 4 show the dependence of total LL
intensity on the radiation dose. It is seen that,
initially, the LL intensity increases with radiation
dose given to the samples, attains a peak value and then
decreases with further increase in radiation
doses given to the samples. The fitting of the curve
with Eq. (21) provides the value of parameters as
shown in Table 1.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\sigma$ ($Oe/m^2$)</th>
<th>$\beta b/\lambda$ $^2$</th>
<th>$\gamma$</th>
<th>$\sigma/\alpha$ ($g^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lactose</td>
<td>$1.695 \times 10^9$</td>
<td>$1.136 \times 10^7$</td>
<td>$6.729 \times 10^3$</td>
<td>$1.30 \times 10^3$</td>
</tr>
<tr>
<td>Sucrose</td>
<td>$5.664 \times 10^{10}$</td>
<td>$7.218 \times 10^{10}$</td>
<td>$1.50 \times 10^3$</td>
<td>$1.56 \times 10^3$</td>
</tr>
<tr>
<td>Mannose</td>
<td>$4.403 \times 10^9$</td>
<td>$5.882 \times 10^{10}$</td>
<td>$1.130 \times 10^3$</td>
<td>$2.43 \times 10^3$</td>
</tr>
</tbody>
</table>

Fig. 5 shows the temperature dependence of LL
intensity for three saccharides$^{11}$. It is seen that, the
LL intensity decreases with increase in temperature
of the solution. This may primarily be attributed to
the decrease of concentration of alkyl radicals and
decrease in the probability of radiative transition.

A study has also been made on the effect of the
temperature at the irradiation occurred$^{20}$. Fig. 6
shows LL intensity of glutamine increases with the
temperature sample, at the time of irradiation.

Ahnstron & Ehrenstein$^{13}$ have already observed
a 20-fold increase in the lyoluminescence of
irradiated glucose, when the solvent is made basic
with 0.1 N solution of NaOH. A similar effect was
then noticed in a number of sugars. Fig. 7 shows the
pH dependence of the LL intensity of glucose in
H$_2$O solution$^{14}$. This effect appears to be due to two
different chemical mechanisms responsible for the
emission of light, one of which is to prevent, in
acidic, and the other in alkaline solutions$^{15,16}$. The
emission spectra of saccharides show clearly that,
with an increase in alkalinity, the emission
maximum shifts to the blue region of the spectrum$^{17}$.
This has been illustrated with glucose-
monohydrate$^{18}$ in Fig. 8. The actual increase in the
LL quantum efficiency is rather modest, and most of
the apparent intensity increase can be attributed to the
S-11 and bi-alkali spectral characteristics of
photo-multipliers used in the earlier investigations.
These characteristics show a rapid decrease in the
quantum efficiency with increasing wavelength.

According to the measurement of Matyushkov
et al.$^{15}$, the LL quantum efficiency of glucose (and
by implication, other saccharides), in acidic
solution, is essentially constant for doses up to about
1 kGy, but for neutral solutions there is some drop in
the efficiency at above about 20 Gy. For strongly
alkaline solutions, the quantum efficiency is an
almost linear function of dose up to about 300 Gy
(Fig. 9). In an alkaline medium the dose-yield
relationship for sugars is non-linear$^{15}$. With the pH
of solution close to neutral, the spectrum of the
emission, and thus the LL mechanism, are affected
by the local changes in acidity, caused by the
radiolytic products of glucose$^{15,17}$. In practice, only
neutral or slightly acidic solutions have been used in
lyoluminescent dosimetry, because of the lack of
reproducibility of alkaline solvents. Takavar$^{13}$ has
reported that, non-reducing sugars fare much better
in that respect. The acidity of distilled water can be
maintained and accurately controlled, only in closed
systems with known gas atmosphere. The use of
buffers has been attempted by Anmuso\textsuperscript{9} for
mannose (Fig. 10) which is much less sensitive to
the changes in pH than glucose. It is notable that,
buffers introduce foreign ions and molecules into
the solvent and thus, in addition to their stabilizing
influence on pH, may quench the lyoluminescence.

Ettinger & Buschan\textsuperscript{9} have reported the LL
spectra of amino acids, even in neutral or slightly
acidic solutions, resemble the alkaline spectra of
sugars, the effect of pH being negligible.

5 Conclusions

Following are the important conclusions drawn
from the theoretical studies of the LL from the
organic materials:

Considering the mechanism of LL in organic
materials - an expression is derived for LL intensity,
which is as given below:

\[
l = \frac{2k_0}{(\xi + \lambda - \sigma)} \cdot \exp(-Yr) \cdot [B_r] \cdot \exp(-\sigma) - \exp(-\xi) \cdot \exp(\sigma) \cdot \exp(-\lambda) \cdot \exp(-\xi - \lambda - \sigma)
\]

The above equation indicates that, when the
organic material is dissolved into the solution then,
initially, the LL intensity should increase linearly
with time, attain a peak value and then it should
decrease exponentially with time.

(ii) Expressions are derived for \( t_m \), \( I_m \) and \( I_t \)
which are as given below:

\[
t_m = \frac{1}{(\xi + \lambda - \sigma)} \ln \left( \frac{\xi + \lambda}{\sigma} \right)
\]

\[
I_m = \frac{k_0 [O_2] \cdot \exp(-Yr) [B_r]}{(\xi + \lambda - \sigma)}
\]

\[
I_t = \frac{2k_0 [O_2] \cdot \exp(-Yr) [B_r]}{\sigma(\xi + \lambda + \sigma)}
\]

(iii) It has been found that, the slope of ln(\( I_t \))
versus \( t \) plot decreases with increasing time. This
may primarily be due to the fact that, the rate of
dissolution decreases with increasing concentration
of the solute in the solvent.

(iv) From the decay of LL intensity, the value of
\( \sigma \) is determined and it is found to be 0.49 and 1.25
sec\(^{-1}\) for saccharide and mannose. These values give
that, the decay-time of alkyl radical concentration
should be 2.04 and 0.87 sec for saccharide and
mannose. From the values of \( t_m \) and \( \sigma \), the value of
the rate constant (\( \xi + \lambda \)) for the decay of peroxy
radical concentration is determined and it is found to
be 1.6 and 2.3 sec\(^{-1}\) for saccharide and mannose
respectively. These values gives that, the decay-time
of peroxy radical concentration should be 0.62 and
0.43 sec for saccharide and mannose respectively.

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