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Photoluminescence of epoxy resin: explanation of the increase in the flux of photoluminescent radiation during the initial stage of the curing process

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Abstract

When using luminescence spectroscopy to study the curing of epoxy resins, there is an increase in the luminescent radiant flux at the beginning of the curing process. The paper clarifies this phenomenon, which limits the determination of the fundamental kinetic constants of the chemical crosslinking reaction. The reflectance measurement shows that the mentioned phenomenon is of physical origin and is caused by a change in the refractive index of the mixture after the addition of the hardener. This change in refractive index is significant in the context of luminescence spectroscopy as it affects the observed luminescent radiant flux. There is a decrease in the refractive index and, therefore, a decrease in reflectance at the material-atmosphere interface, which causes an increase in the number of photons that leave the material. This assumption was further confirmed by measuring the refractive index for the given wavelengths and by the calculation. The paper provides a method of estimating the time constant of the physical process of increase in the flux of photoluminescent radiation. Therefore, it is possible to determine when this process ends, which is crucial for the subsequent estimation of the kinetic parameters of the chemical curing.

1. Introduction

Epoxy resins are among the most versatile polymers, as their unique properties allow them to be used in various applications. They are widely used as adhesives, coatings, laminates and binders [1], whereas the epoxy adhesives market is still growing [2]. Furthermore, epoxies have a significant role as composite matrices [3], with applications, for example, in the electrical engineering industry [4] and aircraft industry [5]. The uncured resin molecule contains more than one oxirane ring, which consists of two carbon atoms and one oxygen atom. An essential property of resins is that they cure after application. This is provided by the hardener, which is a substance that contains active hydrogen ions in the molecule. These ions react with the oxirane rings and cause them to open. As a result of this reaction, a solid three-dimensional network is formed by crosslinking in a volume of initially liquid material [1, 6]. Specific chemical reactions during the curing of resins are described and explained in detail, for example, in [7–9]. The curing can proceed at room or elevated temperatures, and the resulting material is of great strength and completely insoluble [1, 6]. To improve or modify the properties of the resin, fillers (e.g. fibres and textile structures based on glass [10] or carbon fibres [11]), nanomaterials of various properties (e.g. carbon nanotubes [12, 13] or graphene [14, 15]), dyes [16], or pigments [10] can be added to the mixture.

Epoxy-based materials have good mechanical, electrical and optical properties, are resistant to the environment, and are relatively easy to apply and use. For these reasons, epoxies have a wide range of applications. In the automotive, aerospace and marine industries, epoxies are used as anticorrosion coatings, adhesives and composite matrices [17]. In electrical engineering, because of their excellent insulating properties,

epoxies are used, for example, to produce printed circuit boards and electronic component housings, which protect against short circuits, moisture, dust, and mechanical and physical damage [18]. Resins, where metal is used as filler, are then used for electromagnetic interference shielding [17]. In optoelectronics, epoxies are used primarily as potting compounds for LED encapsulation and as a material for producing optical filters [16]. Demanding applications such as aircraft construction require precisely reproducible composition of the material [19]. Therefore, epoxy prepregs, which are semi-finished products for the manufactory of fibre composites, are used for these applications. Prepregs are made of fibre reinforcements pre-impregnated with partially cured resin [20]. Commercially available prepregs comprise epoxies that require autoclave curing under high-pressure and high-temperature cycles to be fully cured [6].

The final properties of epoxies and materials made from them are significantly influenced by the curing process, which must be performed under conditions optimised for the given material or mixture [3, 21]. The curing kinetics is commonly investigated by contact methods such as differential scanning calorimetry (DSC), which enables analysis under isothermal [22] or non-isothermal conditions [23, 24]. Since non-isothermal analysis has many advantages, it is used more often, as stated in [25]. Moreover, standard contact methods also include dielectric spectroscopy [26, 27] and ultrasound techniques [21]. These methods involve direct physical contact with the sample, allowing for precise heat flow measurements, dielectric properties, or sound wave propagation. However, non-contact methods, which do not require physical contact with a sample, are more suitable for industrial applications as they are less invasive and enable continuous monitoring during production. These methods include FT-IR [28, 29] Raman [12, 13] and luminescence spectroscopy [30].

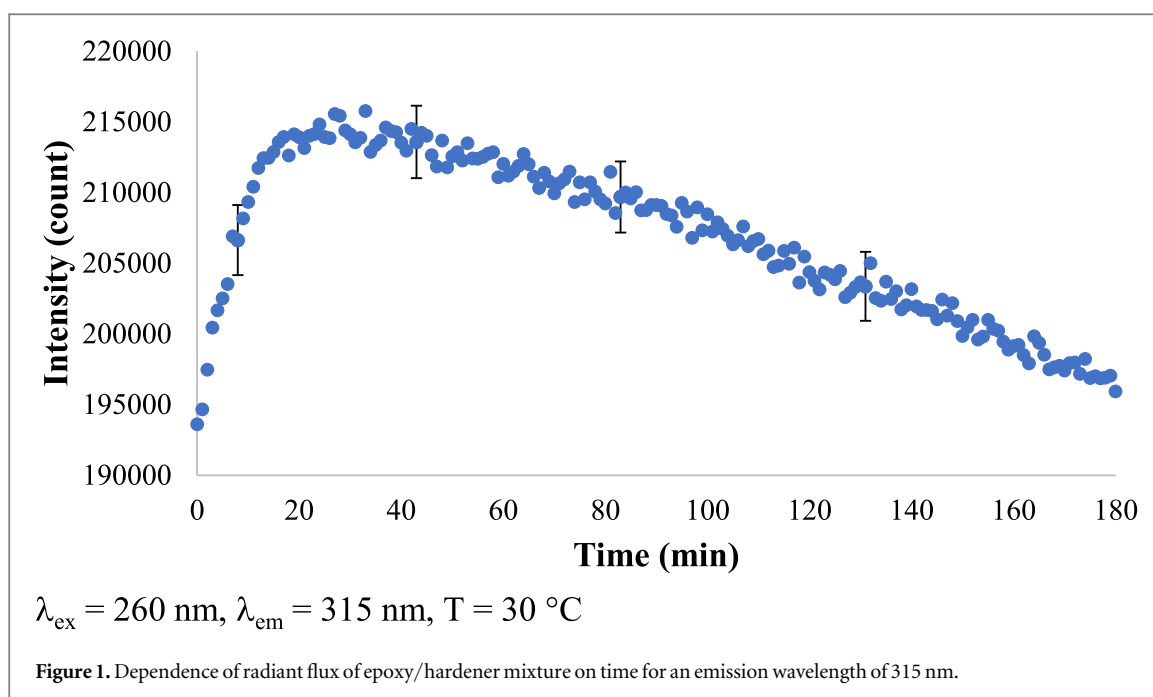
Luminescence spectroscopy is a rapid and very sensitive technique that can record even small changes in the chemical composition of the investigated material. Therefore, it is suitable to monitor the curing process and determine kinetic parameters. As the curing reaction proceeds, the number of photoluminescent centres decreases, which leads to a decrease in the flux of photoluminescent radiation. However, several papers that deal with the study of the luminescent behaviour of epoxy resins have reported an increase in the flux of photoluminescent radiation at the very beginning of the curing reaction after mixing the hardener into the epoxy mixture. For example, in [30], this effect was observed in standard epoxy formulations, while in [12], similar behaviour was documented in a system modified with carbon nanotubes. Furthermore, in [29, 31], the same initial increase was described in glass fibre/epoxy prepregs. This initial increase in signal lasts only a short time compared to the time required for complete curing. In cases when the curing reaction performs quickly, that is, at temperatures above 50 °C, or when the used epoxy system contains a small amount of hardener (based on the authors' experience up to 10% of volume), this process does not appear when monitoring the entire kinetics of the curing reaction. Furthermore, it has no significant influence on determining the kinetic parameters according to the method described in [32]. However, the situation is utterly different for epoxy systems with a large volume of hardener, which are cured at lower temperatures. In this case, the initial increase in radiant flux substantially affects the estimation of kinetic parameters, and it must be said that estimates cannot be made at all in such a situation. It is necessary to wait until this phenomenon ends and the luminescence is controlled by the already ongoing chemical reaction, which forms the molecular network.

In this paper, a transparent epoxy system with a large amount of hardener was studied. For this system, the phenomenon of the increase in photoluminescence appeared to such an extent that the use of the method described in [32] was not possible. Since this phenomenon has occurred in several publications without further explanation, the aim of this paper is to propose a clarification of its cause and to describe the phenomenon mathematically.

2. Description of the phenomenon

When the epoxy resin is mixed with a hardener, a chemical reaction, which causes the opening of the oxirane groups, begins immediately. This reaction results in a decrease in the number of photoluminescent centres and a subsequent reduction in the photoluminescence of the studied material sample, as reported in [12, 29, 32]. However, the unusual phenomenon is the observed initial increase in photoluminescent radiation, which is experimentally evident (figure 1).

Several possibilities are offered; for example, the excitation of luminescence by the transfer of excitation energy from the molecules of the hardener to the epoxy molecules, and thus by the process of chemiluminescence, which would be added to the photoluminescence. If such a phenomenon occurred, it would proceed for a more significant part of the curing process. However, it follows from the measurements that the process of increasing the detected flux of photoluminescent radiation appears only at the beginning of the process and ends quite quickly. This observation, coupled with the fact that an increase in radiant flux is also evident in other types of resins and chemical reactions associated with them, for example, in polyester resins [33], led to the rejection of this hypothesis. Furthermore, as stated in [34, 35], chemiluminescence is strongly



associated with oxidation reactions, which are not present here. If the knowledge that the phenomenon occurs when a relatively large volume of hardener is added is used, a physical explanation for the increase in the flux of photoluminescent radiation is offered. The photoluminescence of the material must decrease as the number of luminescent oxirane rings decreases because they open and, therefore, disappear due to a chemical reaction. However, a higher radiant flux is measured; i.e. a larger flux of radiant energy coming from the material into the detection system is detected since more photons are released from the material. In other words, the reflectance coefficient at the material-atmosphere interface decreases, so more photons are detected. The reflectance coefficient can be described using Fresnel formulas [36]. The hardener has a refractive index lower than the epoxy resin. At the beginning of the curing process, after mechanical mixing, the epoxy/hardener mixture does not become completely homogenised at the molecular level, and homogenisation occurs due to subsequent diffusion. The diffusion of hardener molecules to the surface of the mixture causes a change in the reflectance of the material-atmosphere interface. Hence, more photons leave the examined material.

3. Experimental

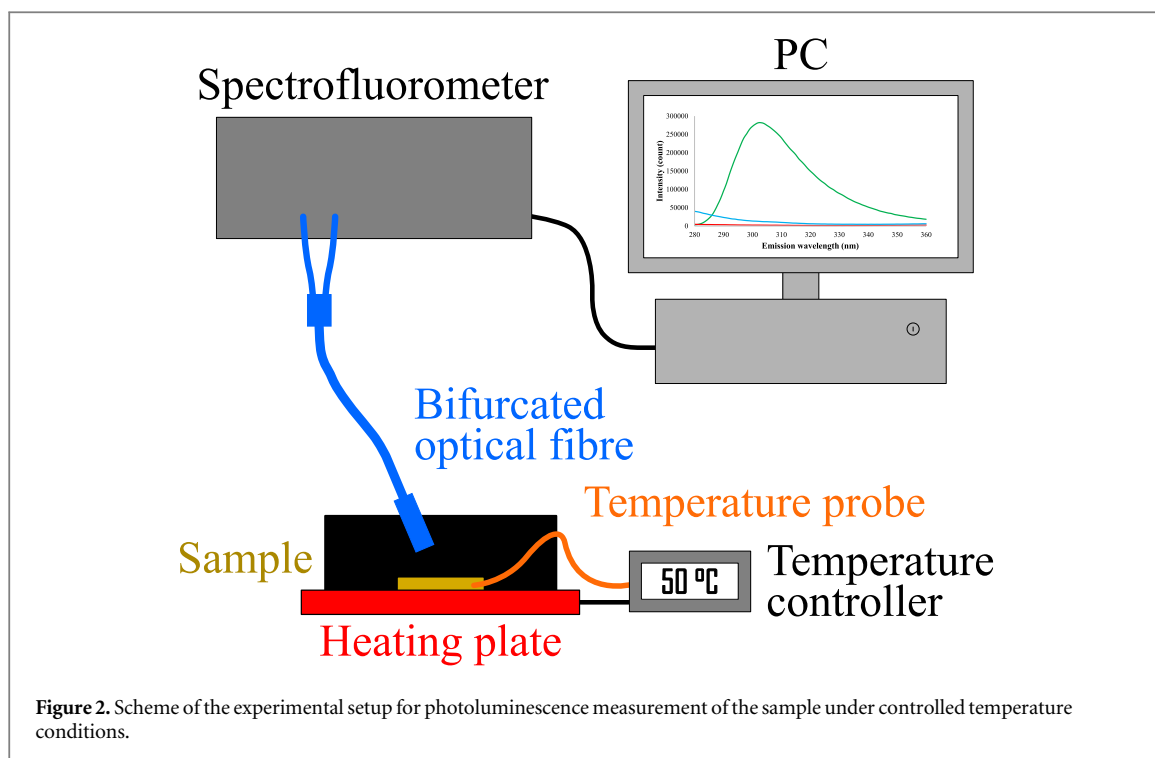
3.1. Materials

Epoxy resin SLIP-LG 100 (based on bisphenol A; density 1.13 g cm^{-3} and viscosity $9\,000\text{--}11\,000 \text{ mPa s}$ for $25 \text{ }^\circ\text{C}$) and polyether hardener S-HG 130 (based on poly(oxypropylene diamine); density $0.96\text{--}0.98 \text{ g cm}^{-3}$ and viscosity $20\text{--}40 \text{ mPa s}$ for $25 \text{ }^\circ\text{C}$) by GRM Systems Ltd were used without further purification. It is a lamination, low-viscosity, and highly transparent epoxy system that allows casting even very thick layers and objects.

3.2. Instrumentation

Photoluminescence spectra were acquired using an ISS PC1 spectrofluorometer featuring a 300 W high-pressure xenon arc lamp as a source of excitation light and the Hamamatsu R928 photomultiplier operating in photon counting mode as a detector. An optical fibre extended from the instrument was used to measure samples in a horizontal position. The excitation wavelength was set to 260 nm, similar to the values reported, for example, in [4, 32, 37]. The resin was mixed with the hardener in a stoichiometric ratio of 100:30. The mixture was stirred for 3 min at room temperature. Subsequently, the layer ($0.23 \text{ mm} \pm 0.04 \text{ mm}$) of the resulting mixture was applied on a pre-heated metal substrate using a micrometric film applicator Gamin Elcometer 3570. The sample was then heated to the curing temperature within 2 min. The measurement time began to be recorded after this preparatory procedure was completed. Individual samples were cured at different temperatures ($30 \text{ }^\circ\text{C}$, $40 \text{ }^\circ\text{C}$, $50 \text{ }^\circ\text{C}$, $60 \text{ }^\circ\text{C}$, $70 \text{ }^\circ\text{C}$ and $80 \text{ }^\circ\text{C}$) for 7 h. Scheme of the experimental setup is shown in figure 2.

UV-vis absorbance spectra were recorded in the wavelength range of 200–1100 nm at room temperature using a Shimadzu UV-1900 double-beam spectrophotometer. The resin sample was placed in a quartz cuvette with an optical path length of 1 mm.



Refractive indices were measured using a UVISEL 2 ellipsometer (Horriba) in standard configuration. Measurement was performed in a spectral range of 200–800 nm. Layers were prepared on microscope slides. Before measurement, the slides were cleaned in an ultrasonic bath (Neodisher surfactant + H₂O in a ratio of 2:5) for 45 min. Subsequently, they were rinsed in isopropyl alcohol and water and dried with compressed air. The hardener and epoxy resin layers were prepared on slides heated to a temperature of 70 °C to prepare a thin layer without bubbles. Horiba's New Amorphous mathematical model was used to evaluate the data.

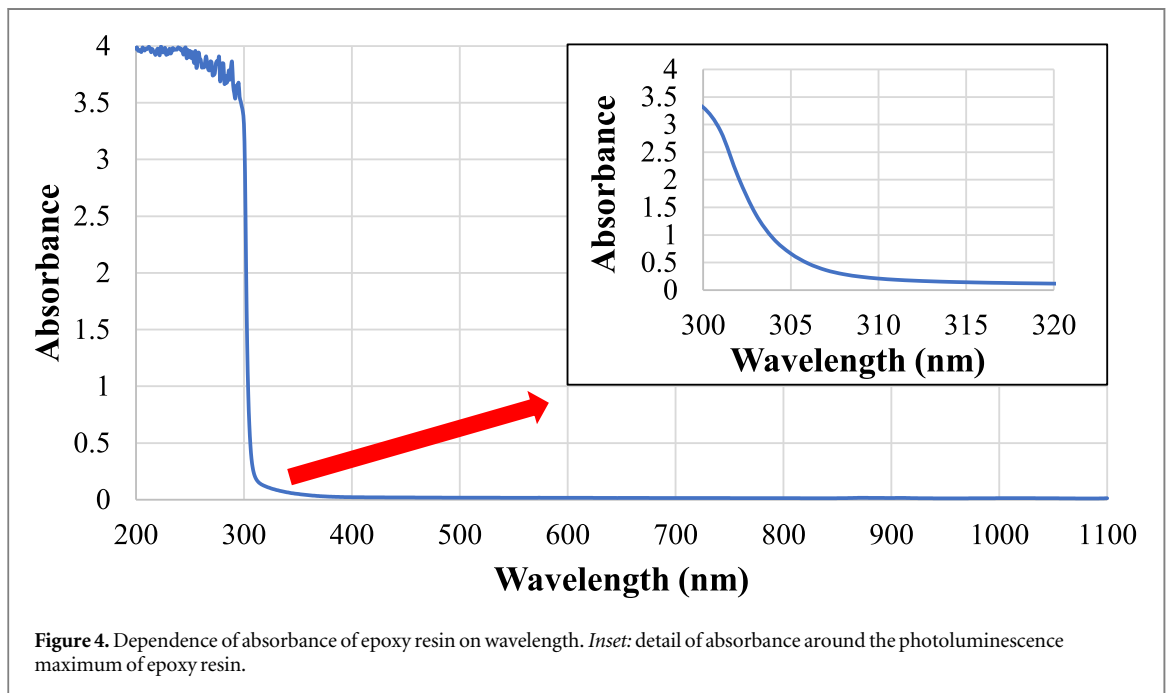
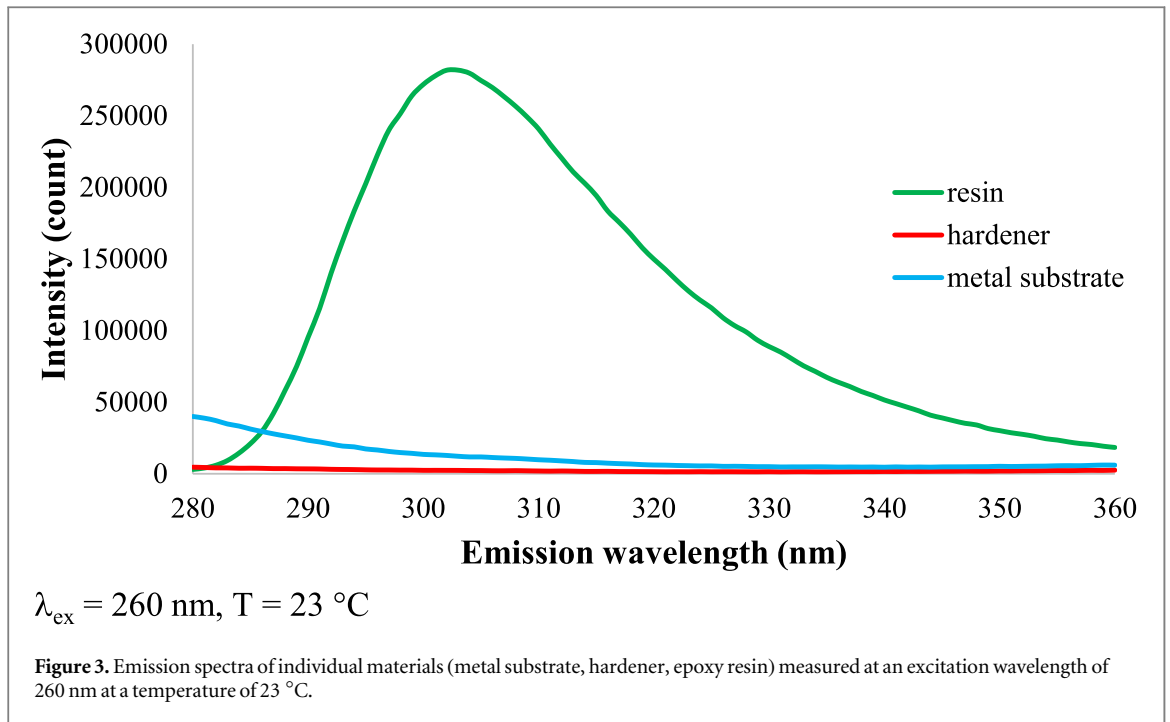
4. Results and discussion

The oxirane ring behaves the usual way when excited by a wavelength of $\lambda_{\text{ex}} = 260$ nm. The emission spectra of the resin, the hardener, and the metal substrate are shown in figure 3. As illustrated, the photoluminescence of the resin, with the emission maximum at $\lambda_{\text{em}} \approx 303$ nm, is dominant. The hardener and the substrate show a negligible contribution to the flux of photoluminescent radiation. However, there is significant absorption of the radiation (figure 4) in this spectrum region. Therefore, a detection wavelength of 315 nm, which is far enough from the absorption edge of the studied epoxy resin, was selected.

Traditional standardisation approaches require the value of the monitored quantity upon completion of the entire reaction, which can theoretically be infinite, but practically a very long time — up to days for room temperature. Since the goal is to predict the curing kinetics of the epoxy system based on measurements at the beginning of the ongoing reaction, the photoluminescence signal is normalised to the initial value at time zero instead. It is also practical to display this quantity in logarithms, as follows from the mathematical model described in [32]. The measurement results for different temperatures of isothermal curing are provided in figure 5.

Figure 5 shows the increase in the flux of photoluminescent radiation at the beginning of the curing process and its disappearance at temperatures above 50 °C. The assumption is that this increase is caused by a change in the reflectance of the material-air interface. If this is the case, the reflectance should also decrease in the opposite direction (from the atmosphere to the material). In other words, more excitation radiation passes into the material, which increases the flux of photoluminescent radiation. In figure 6, the dependence of reflectance on time is measured for the excitation wavelength $\lambda_{\text{ex}} = 260$ nm and the selected emission wavelength $\lambda_{\text{em}} = 315$ nm. The figure shows a decrease in reflectance at the beginning of the curing process and, therefore, a decrease in the refractive index.

The change in the refractive index of epoxy resins during the crosslinking reaction has been studied to determine the degree of curing reaction, for example, in [38–40]. In all the papers, the refractive index increases during curing, enabling optical methods to determine the state of the ongoing reaction. The refractive index significantly depends on the temperature and the wavelength used, which must be considered during



applications. This problem was described and applied excellently in [40], where a clear decrease in the refractive index in the initial stages (approximately the first 30 min) of the reaction was also shown.

The reflection coefficient for perpendicular incidence and unpolarised light can be written from Fresnel formulas [36] as

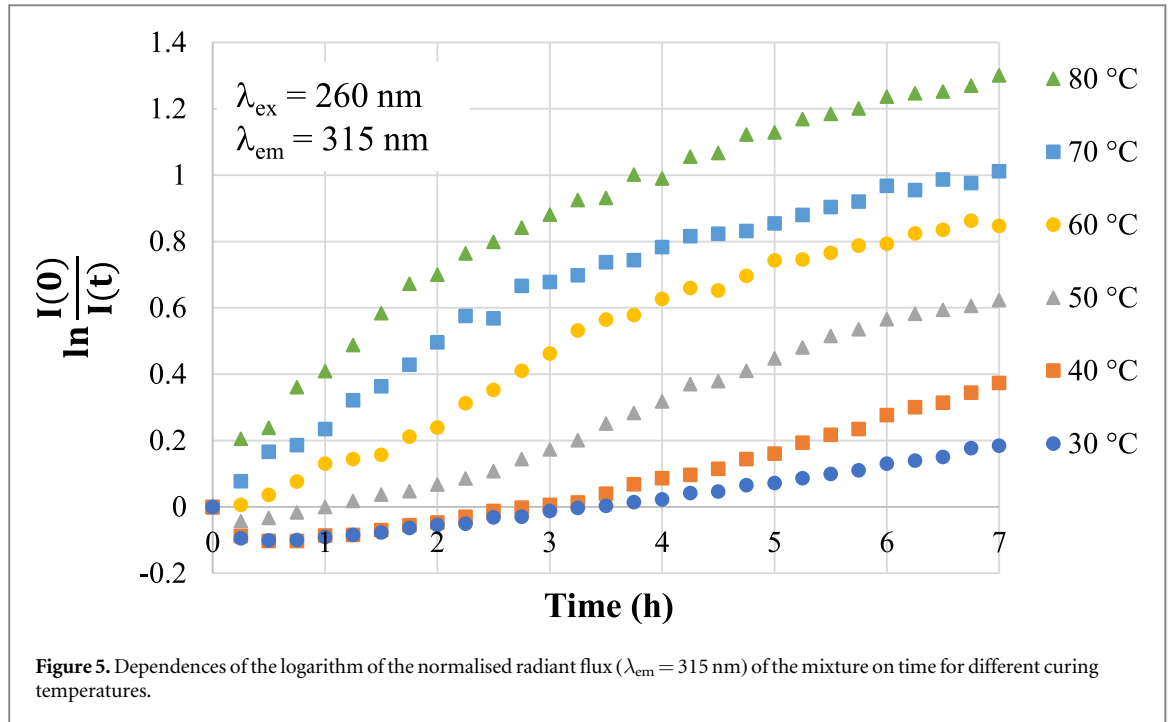
$$R = \left| \frac{n_{\text{material}} - n_{\text{air}}}{n_{\text{material}} + n_{\text{air}}} \right|^2, \quad (1)$$

where n_{air} is the index of refraction of air and n_{material} is the index of refraction of the material, in this case, resin.

The mixing rule can usually be used to calculate the refractive index of a mixture of two substances as

$$n_{\text{material}} = \phi_1 n_1 + \phi_2 n_2, \quad (2)$$

where n_1 , n_2 are the refractive indices and ϕ_1 , ϕ_2 are volume fractions of both mixed substances.



The volume fractions ϕ_1 and ϕ_2 are specified by the manufacturer based on the number of reactive chemical groups on the molecules. For SLIP-LG 100 resin and S-HG 130 hardener, the manufacturer recommends a mass ratio of 100:30 or 100:38 by volume. It is necessary to determine the corresponding refractive indices of the resin and hardener for the wavelength used, which is $\lambda_{em} = 315$ nm.

Using an ellipsometer, refractive indices $n_1 = 1.692 \pm 0.001$ for the resin and $n_2 = 1.506 \pm 0.001$ for the hardener were measured. According to equation (2), it can be calculated that the refractive index for a mixture of resin ($n_1 = 1.692$, $\phi_1 = 0.77$) and hardener ($n_2 = 1.506$, $\phi_2 = 0.23$) is $n_{material} = 0.77 \times 1.692 + 0.23 \times 1.506 = 1.649$. All refractive indices correspond to the wavelength $\lambda_{em} = 315$ nm. After substituting into formula (1), the reflectance drops from $R_{resin} = 0.0658$ for the pure resin to $R_{mixed} = 0.0600$ for the resin/hardener mixture. This change is approximately 10% (R_{resin}/R_{mixed}). According to figure 1, $I(24 \text{ min})/I(0) = \frac{214800}{193600} = 1.11$, which is an 11% increase that corresponds well to the change in reflectance at the material-air interface determined by calculation from the measured refractive indices.

Therefore, the initial increase in the flux of photoluminescent radiation appears to be a physical process and has little to do with the ongoing chemical reaction. However, it prevents the determination of the kinetic parameters of the chemical curing reaction and the subsequent prediction of the duration of the process. Hence, the question is when measurements can be performed to determine these quantities so that they are not influenced by this initial phenomenon. In other words, it is necessary to determine the time parameters of this physical process and when this process can be neglected. Therefore, the aim is to determine the time constants of the processes that occur in the initial phase of the curing reaction. This can be achieved through the phenomenological description of the initial curve according to the dependence shown in figure 7. As can be seen, the entire process can be described by two competitive sub-processes, which can be formulated as

$$dI = -k_{0i} \exp(-t/\tau_0) I dt + k_{1i} \exp(-t/\tau_1) I dt, \quad (3)$$

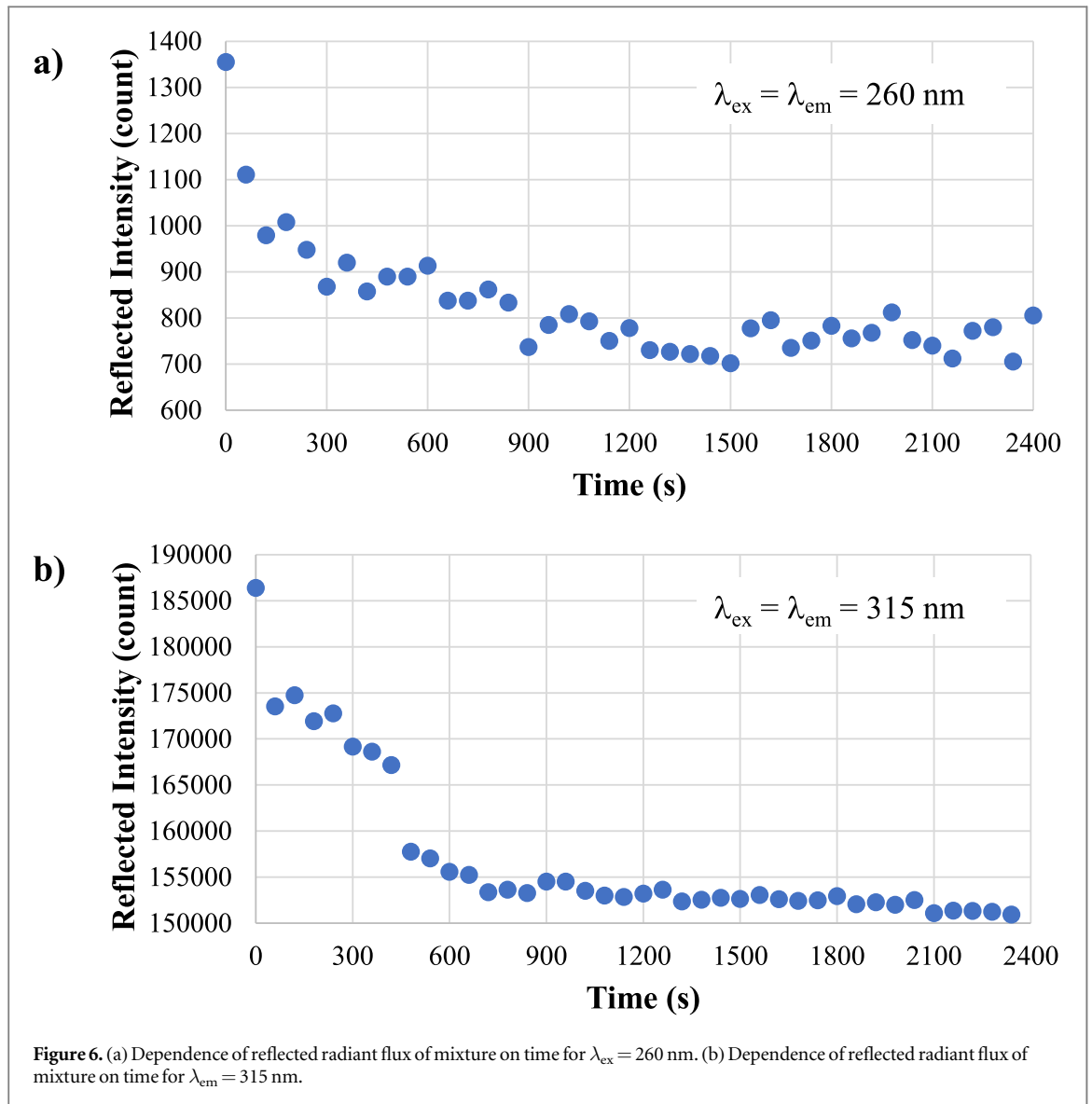
where k_{0i} is the initial value of the curing speed at $t = 0$, and thus the reduction of the number of oxirane rings, and τ_0 is the time constant of the curing process. This term of equation (3) describes the decrease in the flux of photoluminescent radiation. The second term of equation (3) describes the increase in photoluminescent flux, which is related to the decrease in reflectance at the resin-air interface, and k_{1i} is the initial value of the homogenisation speed at $t = 0$, and τ_1 is the time constant of the homogenisation process.

Subsequently, equation (3) was integrated (separation of variables):

$$\ln(I(0)/I(t)) = k_{0i} \tau_0 [1 - \exp(-t/\tau_0)] - k_{1i} \tau_1 [1 - \exp(-t/\tau_1)], \quad (4)$$

whereas the experiment shows that $\tau_1 \ll \tau_0$. Figure 7 shows that τ_1 varies in tens to hundreds of seconds. In contrast, τ_0 varies in the order of thousands to ten thousand seconds.

Formulas for calculating significant time intervals can be determined from the above equations by mathematical modifications. The time t_{min} , at which the quantity $\ln(I(0)/I(t))$ reaches its minimum, can be formulated as



$$t_{\min} = \frac{\ln(k_i/k_{0i})}{1/\tau_1 - 1/\tau_0}, \quad (5)$$

while the time t_0 , at which the quantity $\ln(I(0)/I(t))$ passes through zero, can be described as

$$t_0 \cong \tau_1 k_i/k_{0i}. \quad (6)$$

These formulas are useful primarily for quick orientation as current computer technology allows the required quantities to be estimated using numerical interpolation with measured data. Figure 7 shows the fitting of the measured data by the proposed mathematical model in the Matlab software. After processing the time series of the flux of photoluminescent radiation, the estimate of the time constant for the process of changing the refractive index (reflectance) at the beginning of the curing reaction was obtained, which was $\tau_1 = (17 \pm 2)$ minutes for curing at 30°C . On the contrary, the estimated values of the quantity τ_0 cannot be taken experimentally seriously since a too small part of the time series describing the entire curing process, characterised by this time τ_0 , was used. Nevertheless, this is a mathematical problem that is unimportant from the point of view of characterising the process with the time τ_1 .

Through the processing of the measurements shown in figure 5 in the Matlab software, the following estimates of the time constant τ_1 and the speed constants k_{0i} and k_{1i} were obtained (table 1).

To estimate the time when the process of increasing the photoluminescent flux is finished, and thus the mathematical model described in [32] can be used, it is necessary to start from the uncertainties of the measurement of the luminescent radiation flux. These uncertainties include factors such as instrument precision, environmental conditions, and sample variability. The relative uncertainty of presented measurements is in the interval (2–10) %. When approaching this distance from the predicted value at infinity (a very long time interval), it is no longer possible to determine the state of the ongoing process and its changes.

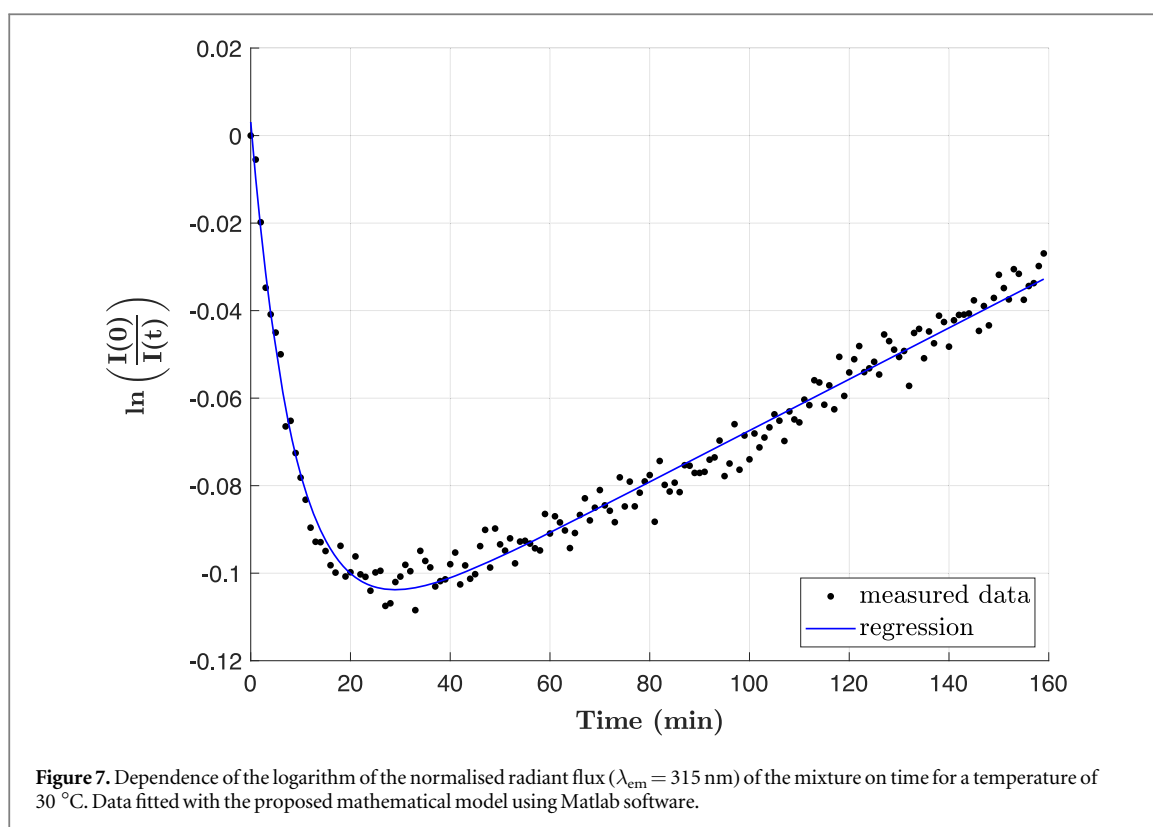


Table 1. Values of the time constant of τ_1 and the speed constants k_{0i} and k_{1i} depending on the temperature.

T (°C)	30	40	50	60	70	80
τ_1 (min)	17 ± 2	15 ± 2	7 ± 2	4 ± 2	—	—
k_{0i} (min^{-1})	$8.0 \cdot 10^{-4}$	$9.0 \cdot 10^{-4}$	$1.1 \cdot 10^{-3}$	$2.7 \cdot 10^{-3}$	—	—
k_{1i} (min^{-1})	$7.7 \cdot 10^{-3}$	$8.6 \cdot 10^{-3}$	$9.9 \cdot 10^{-3}$	$1.1 \cdot 10^{-2}$	—	—

From the simple formula $[1 - \exp(-t/\tau_1)]$, it is possible to estimate that the deviation is 14 % for $t = 2\tau_1$, 5 % for $3\tau_1$ and 2 % for $4\tau_1$, where τ_1 is the time constant of the studied process.

Therefore, for the studied epoxy system, it is possible to estimate the completion of the process of increasing the photoluminescent flux at a temperature of 30 °C for a period of approximately $4 \times 17 \text{ min} = 68 \text{ min}$ (4080 s). After this time, luminescence spectroscopy can be used to study the ongoing reaction solely from the perspective of chemical crosslinking without the disturbing effects of the physical process of changing the refractive index at the material-air interface.

5. Conclusions

In the presented publication, a possible explanation of the phenomenon of increasing the flux of photoluminescent radiation at the beginning of the curing reaction of epoxy resin was offered. After measuring the reflectance of the air-resin interface for both the excitation and the selected emission regions of the spectrum, it was concluded that the observed increase is caused by a decrease in the refractive index of the studied material after mixing. Moreover, this assumption was confirmed by measuring the refractive index of both components using the ellipsometric method and then calculating the change in reflectance. The resulting value was found to agree with the observation of photoluminescent data.

The following conclusions have been drawn from the study:

1. The increase in the luminescence signal at the beginning of the curing reaction of epoxy resins is caused by a decrease in the refractive index of the material when the hardener is mixed in. As the hardener molecules diffuse to the surface of the mixture, the reflectance at the material-air interface decreases, which leads to an increase in the flux of photoluminescent radiation emerging from the material.

2. The change in the reflectance of the interface is a physical process which is significantly faster than the actual crosslinking reaction during the curing of the resin, and it disappears relatively quickly.
3. During curing at higher temperatures, this physical phenomenon proceeds very rapidly and thus can be neglected when observing the kinetics of the crosslinking reaction and its kinetic characteristics.
4. If the time constant of the increase in the flux of the luminescence signal is determined for a given temperature, it is necessary to wait at least four times this time before only the chemical curing process, which is usually the object of interest, becomes the dominant factor in the change of the radiant flux.

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Conflict of interest

There are no conflicts of interest to declare.

Data availability statement

The data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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