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DISSOLVED SUGAR LUMINESCENCE

We report on experiments conducted to investigate dissolved sugar luminescence in order to clarify the photophysics of the process. The conclusions we have drawn provide evidence of a quadratic increase in the luminescence response at small concentrations (<100 g/l) and a linear increase at relatively large concentrations (>200 g/l). We consider the possible effect of excimer formation and luminescence. The apparently existing intermolecular interaction existence is also supported by observed variations in the absorption spectra. A temporal response to a short light pulse was measured, revealing a time delay of about 15 ns.

K e y w o r d s: sugar luminescence, intermolecular interaction, absorption.

1. Introduction

Organic materials possess quite specific luminescence properties in comparison with solid-state inorganic luminescence [1]. Normally, excited electronic states spread photons during the radiative relaxation to a ground state, producing a set of emission bands [2], or manifesting as Raman scattering [3, 4]. Apart from this general type of molecular luminescence other effects of radiative energy release take place such as exciton luminescence [constitutes M.T. Shpak, 5, 6]. In addition luminescence of solutions constitutes a special chapter [7].

The main idea of this study is an attempt to clarify a photophysics of this sort of emission with the use of a disaccharide molecular crystal (sugar). In general, bright luminescence of sugar, stimulated by ultraviolet (UV) radiation, is a well known effect, especially a persistent afterglow observed upon cooling the sample to the liquid nitrogen temperature. Nevertheless, the nature of this emission is still quite unclear. Spectral measurements of nominally pure sugar (refined) show the presence of luminescence emission in the visible range from 350 nm to 750 nm.

Such a broad luminescence contour can hardly be attributed to a single sugar molecule. To find an explanation for this broadband emission, we decided to investigate the luminescence of sugar in aqueous solutions to follow its gradual transformations from the emission of isolated molecules in an aqueous environment to that of condensed syrup.

2. Experiment

A set of samples was prepared with concentrations from 5 g to 600 g of a refined sugar per a liter of distilled water. A silica cell $10 \times 10 \times 50$ mm was filled with the corresponding solution and placed in a spectrofluorimeter to detect the luminescence spectrum.

First, we checked the UV-excited luminescence of a saturated solution, the results being shown in Fig. 1 for three different excitation wavelengths. Very similar to bulk sugar samples, the emission appears at the Stokes side in a shape of broad non-structured band. Moreover, the luminescence spectrum follows the excitation wavelength in the Stokes side throughout this region.

With dilute solutions, we have observed the luminescence band for different concentrations. Figure 2 shows the spectra registered with the concentrations 20, 40, 80, 160 and 320 g/l with EDINBURGH Instruments FS5 spectrofluorimeter. We detected that a decrease in concentration did not influence the spectrum contour shape but the signal intensity decreased in a specific manner. The inset in Fig. 2 shows the nonlinear dependence in the region of small concentrations, and we have paid attention to inspect the behavior in more detail.

The concentration dependence was measured with the choice of the excitation wavelengths 337 nm and

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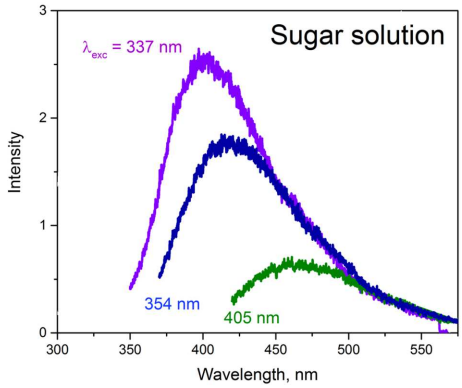

Fig. 1. Examples of the emission spectra of sugar dissolved in water (saturated solution). Three excitation wavelengths were used, as indicated

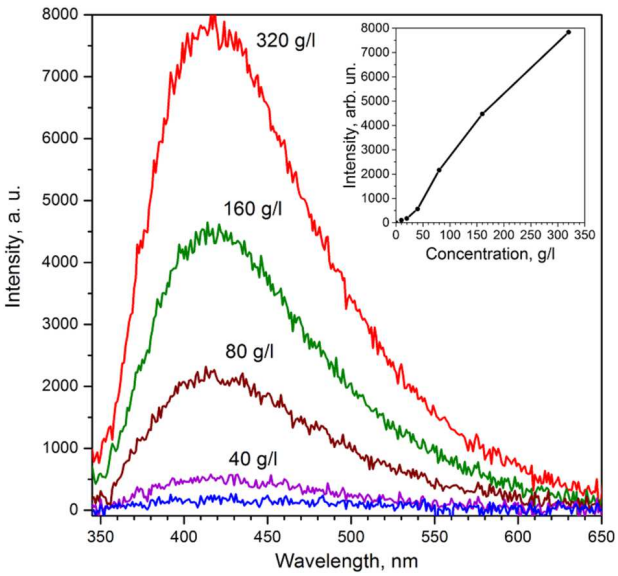

Fig. 2. Spectra registered at the concentrations as indicated. (The spectrum for 20 g/l is not marked.) Inset: the growth of the contour intensity with increasing concentration

405 nm conditioned by a maximum peak value of the emission band at 400 nm and 440 nm, and the possibility of comparing the results with bulk samples luminescence excited by N_2 pulse laser radiation and diode laser emitting at 405 nm.

Experimental results are presented in Fig. 3 for the excitation wavelength 337 nm (*a*) and for 405 nm (*b*). The measurements were performed with a fluorescence spectrophotometer Hitachi MPF-4.

The use of log-log scale in Fig. 3 permits easy to find the power of the dependence function. The

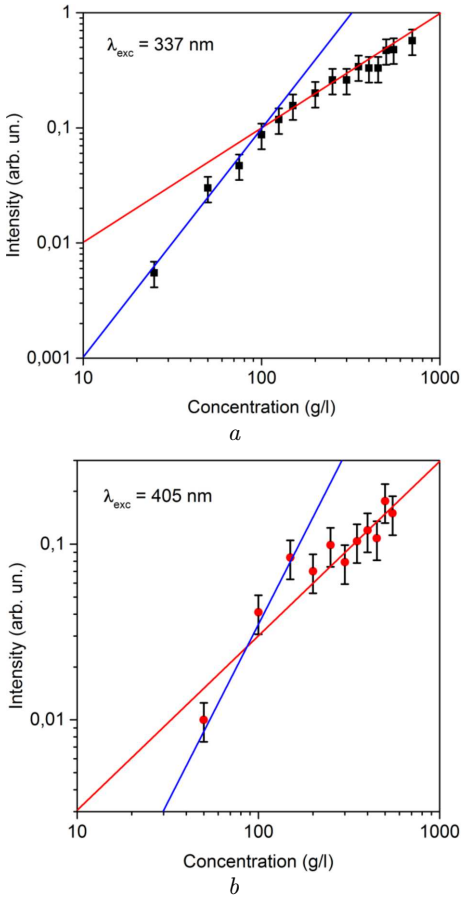

Fig. 3. Experimental results for the dependence of the maximum luminescence peak on concentration, presented in log-log scale for the excitation wavelength of 337 nm (*a*) and 405 nm (*b*). The reference lines of linear dependence ($y \sim x$) and quadratic dependence ($y \sim x^2$) are not data fits, but indicate the corresponding slope

reference lines indicate the inclination corresponding to the linear dependence and quadratic dependence. Both plots in Fig. 3 show the same qualitative behavior, namely quadratic signal growth with respect to the concentration rise in the region of relatively small concentrations. Then, over the concentration about 150 g/l the dependence gradually transforms to a linear one.

We note one more peculiarity which is seen in Fig. 3, *b* but not well resolved in Fig. 3, *a*: the transfer from quadratic dependence to linear one (crossover) occurs with some signal decrease at the concentration range around 250 g/l. To inspect the detected feature, we have measured the luminescence response

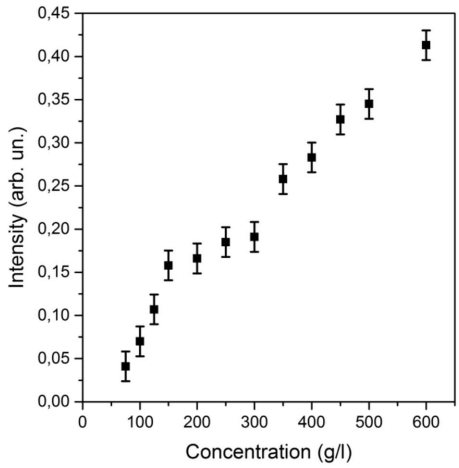

Fig. 4. Concentration dependence of the luminescence response in linear scale measured at the crossover region

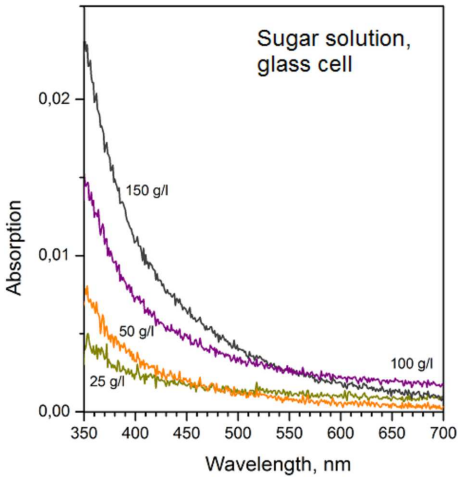

Fig. 5. Absorption spectra recorded in visible for the concentrations 25, 50, 100 and 150 g/l

in this interval with maximum accuracy. The results are presented in Fig. 4 in linear scale.

Indeed a deceleration of the signal growth was observed in the interval between the quadratic and linear parts, as seen in Fig. 4 in between concentrations of 150 g/l and 300 g/l. The reason for the broken dependence could be related to the variation in the absorption of the incident excitation light, and on the next step we have checked the absorption of the samples. The measurements were performed with a Shimadzu UV-2450, UV-Vis spectrophotometer. The cell used had dimensions of 10×10 mm. The peculiarity of low absorption measurements is the account

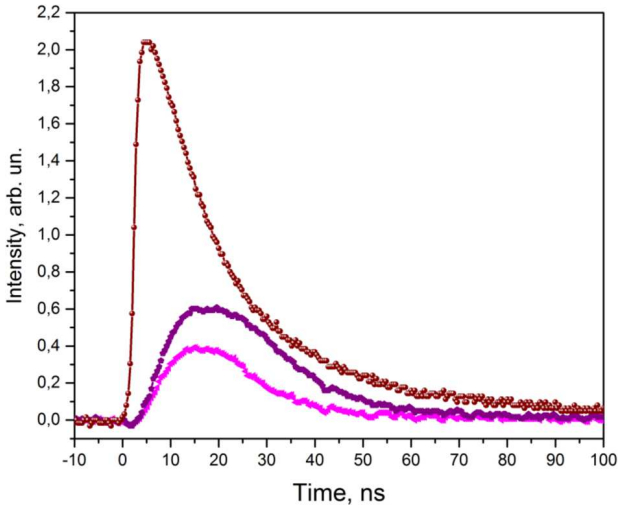

Fig. 6. Temporal luminescence response of dissolved sugar for two concentrations (50 and 100 g/l) and the reference laser pulse

of Fresnel reflection variation in the case of index matching between the cell material ($n = 1.45$ for silica cell) and the solution (in accordance with literature data [8], the refractive index for the concentration range up to 500 g/l can reach 1.43). To avoid errors, we duplicated the experiment with a glass cell ($n = 1.51$). A set of recorded spectra is shown in Fig. 5. In the visible range we observed decreasing absorption without pronounced maxima (probably located in UV region). At low concentrations, the linear increase in absorption was detected. With the concentration rise, a surprising behavior of the spectra was observed, namely, a decrease in absorption. These variations of the absorption spectra most likely evidence the transformation of the molecular absorption bands with the concentration rise; an important qualitative conclusion is that the absorption growth in the low-concentration region exhibits a sublinear dependence, i.e. the relative number of the absorption centers diminishes. Again, apparently, intermolecular interaction can be expected.

3. A Temporal Check of the Response

The scope of the results obtained with the dissolved sugar permits us to conclude that some kind of intermolecular aggregation does take place. A simple model assumes that an excited molecule creates with a non-excited one an excimer, i.e. a molecular dimer that is instable in the ground state. That is, af-

ter the relaxation an excimer dissociates into individual molecules. The following breakup of an excimer is accompanied by photon emission, i.e. the observable luminescence.

As the model predicts, the absorption of the excitation photons occurs by an isolated molecule, but the radiation centers (excimers) are created by the association of two sugar molecules, one being in the excited state. Hence, some time is required for the excimer formation, after starting the external illumination in the non-stationary case. (This assumption concerns only the small concentration stage).

In this view we applied pulse radiation from an N_2 laser (337 nm wavelength, 10 ns duration and 5 ns rise time (checked with an avalanche photodiode). To register rather weak luminescence of the samples, a photomultiplier was used. The pump UV light was blocked by a color glass filter placed at the photomultiplier window. The signal was developed with the oscilloscope Rigol DS1102E. The temporal resolution of our setup permits proper registration of the front of the pulse but gives some protraction of the rear front. Within these conditions we observed a retardation of the luminescence response to the excitation pulse on about 15 ns (see Fig. 6).

4. Discussion and Conclusions

The performed research permit us to conclude that the observable emission occurs most likely in the following way:

(i) The concentration dependence of the luminescence shows a square function, thus indicating an association of two molecules for photon release.

(ii) The excitation light absorption acts on an individual molecule in the solution, as evidenced by the absorption measurements (linear rise at small concentrations).

(iii) To release the excited energy via photon emission, some time is required (15 ns measured); this delay can be associated with excimer formation.

We emphasize that intermolecular interaction and dimer formation in sucrose solution have been studied in detail in several researches [9–12]. The effect of sucrose molecules hydration is usually measured in a number of water molecules per one sucrose molecule, and the onset of dimerisation is expected when this number is about 10. That is, in striking contrast to our results, the dimerisation processes become detectable in the concentration range over 300 g/l using the measurement approach adopted in this work. We can expect that the excitation of molecules with external illumination induces the aggregation effect in the lower concentration range.

In summary, this set of experiments revealed, in general, rather complicated photophysics of the dissolved sugar luminescence, associated with intermolecular interaction and possible excimer formation and luminescence. We note the actuality of the study, in view of applications in food industry and medicine [14, 15].

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ЛЮМІНЕСЦЕНЦІЯ ВОДНИХ РОЗЧИНІВ ЦУКРУ

Повідомляється про результати дослідження люмінесценції розчиненого цукру з метою уточнення фотофізики цього процесу. Результати свідчать про квадратичне збільшення потужності люмінесцентної реакції за малих концентрацій (менше ніж 100 г/л) і лінійне для відносно великих концентрацій (понад 200 г/л). Розглянуто можливий вплив утворення ексимерів на люмінесценцію. Очевидне існування міжмолекулярної взаємодії також підтверджується зазначеними змінами спектрів поглинання. Виміри часової реакції на короткий світловий імпульс виявили часову затримку відгуку близько 15 нс.

Ключові слова: люмінесценція цукру, міжмолекулярна взаємодія, поглинання.