

Encapsulation-Driven Modulation of Fluorescence and Förster Resonance Energy Transfer of Cationic Dyes Confined in Zeolite KLTL Nanochannels

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Abstract

The encapsulation of organic dye onto zeolite KLTL was prepared using cation exchange and a gas-phase method. Because cation exchange limits dye inclusion, we used gas-phase methods to improve the process. In this work, cationic dyes such as acridine hydrochloride (AC) and hemicyanine iodide (Hemi) were encapsulated onto KLTL zeolite by the gas-phase method. The amounts of dye with different occupation probabilities (ρ) of 0.1, 0.25, 0.5 and 1.0 were used. It was found that an occupation probability of 0.25 is suitable for the preparation of the dye-zeolite, as it prevents dye aggregation in the zeolite and quenching of the dye emission. To synthesize the antenna system, a donor-acceptor Acridine-Hemicyanine iodide (AC-Hemi) was prepared by cation exchange-gas phase (i-g), gas phase-cation exchange (g-i), gas phase-gas phase (g-g), and/or cation exchange-gas phase (i-g). The results show that all systems show strong quenching in KLTL, and AC(i)-Hemi(g) has the highest quenching efficiency (65.9%). This high efficiency can be applied to antenna materials. Additionally, this study shows that confining dyes in zeolite KLTL plays a key role in enhancing fluorescence and developing optical materials.

Keywords: Encapsulation, Fluorescence, Förster resonance energy transfer

Introduction

Traditionally, inorganic nanopore-structured materials, such as zeolites, metal-organic frameworks, and clay, have produced inorganic and organic photoluminescent materials (Calzaferri et al., 2006; Sola-Llano et al., 2019; Gutiérrez et al., 2022). Host-guest materials, such as dye-LTL, have gained increasing attention over the years (Calzaferri, 2012; Minkowski et al., 2006; Insuwan et al., 2015). More recently, this concept has been extended to related nanoporous and zeolite hosts, including dye-loaded pore zeolites exploited for FRET (Oliden-Sánchez et al., 2024), and rhodamine B confined within zeolitic frameworks for enhanced energy transfer, as reported by Hu and coworkers (Hu et al., 2023). LTL has a pore structure of one-dimensional channels running across the crystal axis. The performance of zeolite LTL in applications relies upon its physical properties, such as crystal size and morphology (Insuwan & Kunwadee, 2012). In this work, cationic dye molecules such as acridine hydrochloride (AC) and hemicyanine iodide (Hemi) (Fig. 1) were prepared by ion exchange. Because some dyes, such as hemicyanine iodide, are large, ion exchange makes insertion difficult. At the same time, the high-temperature gas-phase method causes the pores of zeolite L to expand. The fluorescence intensity of dyes is significantly enhanced, attributed to the isolation and confinement of the dye in the nanochannels of zeolite L.

Förster resonance energy transfer (FRET) is a non-radiative energy-transfer process between an excited donor and a nearby acceptor via dipole-dipole coupling, typically effective at distances of 1–10 nm (Lakowicz, 2006). Energy transfer also depends on the inverse sixth power of the distance between the donor and acceptor

chromophores. FRET between an organic or laser dye in zeolite LTL channels is an efficient process for antenna applications such as dye-loaded zeolite LTL antennas in solar cells and Luminescent solar concentrators (LSC) (Calzaferri, 2012; Insuwan et al., 2015). Monzón-Benosojo (2023) recently synthesized natural pigment-zeolite composites and suggested applications in environmentally friendly antenna designs. Moreover, researchers constructed discrete supramolecular assemblies (DSAs) by incorporating fluorophores as donors and amphiphiles as acceptors (Chen et al., 2024). Chen et al. (2023) reviewed the construction, modulation, and applications of artificial light-harvesting systems. This work focuses on how the encapsulation sequence affects FRET efficiency to elucidate the role of spatial organization and confinement in energy transfer processes. This work provides insight into confinement-controlled photophysics and establishes design principles for fluorescence-based artificial antenna systems.

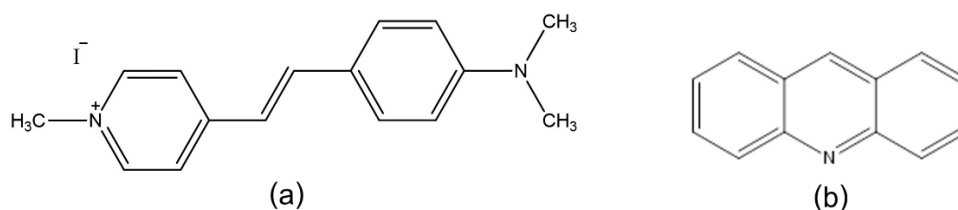


Figure 1 The structure of the donor-acceptor molecule (a) trans-4-[4-(Dimethylamino)styryl]-1-methylpyridinium (Hemi) and (b) Acridine hydrochloride (AC)

Materials and Methods

Synthesis of Zeolite KLTL

Zeolite KLTL crystals were synthesized following a reported hydrothermal method with minor modifications (Ban et al., 2007; Insuwan & Kunwadee, 2012). Aluminum hydroxide (0.59 g) was dissolved in a boiling potassium hydroxide solution prepared by dissolving 1.28 g of potassium hydroxide in 2.51 g of double-distilled water until a clear solution was obtained. This solution was subsequently added to a mixture of 5.52 g of Ludox HS-40 and 4.82 g of double-distilled water under continuous stirring. The resulting gel was stirred for 3 min until it formed a homogeneous mixture with appropriate viscosity and turbidity. The precursor gel was transferred into a Teflon-lined autoclave and crystallized at 180 °C for 48 h without stirring. After crystallization, the autoclave was rapidly cooled in cold water prior to opening. The solid product was recovered by washing repeatedly with distilled water until the supernatant pH was approximately neutral. The resulting zeolite KLTL crystals were dried overnight at 80 °C in a hot air oven. The morphology of the synthesized zeolite KLTL was examined by scanning electron microscopy (SEM; JEOL JSM-6010LV) at an acceleration voltage of 10–20 kV.

Dye Encapsulation by Ion Exchange (i)

Cationic dyes such as AC and hemi were incorporated into zeolite KLTL at 30 °C using 0.5 g of accurately weighed, dehydrated zeolite KLTL in a 125 mL conical flask. Then, 50 mL of the dye solution (0.1 mM) was added to the flask. The flask was sealed and transferred to a shaker bath for 24 hours to equilibrate the system. The dyes loaded with zeolite KLTL were collected by separating the solid from the solution by centrifugation and dried overnight at 100 °C. The following washing procedure was used to remove dye molecules from the zeolite surface. The suspension was transferred into a 125 mL conical flask with 50 mL n-butanol, sonicated

for 10 minutes, then stirred at room temperature for 24 hours. The n-butanol washing was repeated until the supernatant no longer showed dye luminescence.

Dye Encapsulation by Gas-Phase Diffusion (g)

In this experiment, 100 mg of zeolite KLTL was mixed with neutral or cationic dye at various occupation probabilities ($\rho = 0.1, 0.25, 0.5, \text{ and } 1.0$). Then 1.0 mL of ethanol was added to the mixture. The mixture was sonicated for 10 min to dissolve the dye and ensure good dispersion of the zeolite KLTL. The solvent was removed in a vacuum oven at 50 °C for 2 h, yielding a homogeneous dye coating on the zeolite particles. The dried material was ground into a fine powder using an agate mortar and further dehydrated under vacuum at 1×10^{-2} mbar for 2 h. Gas-phase insertion was then carried out by heating the sample at the respective dye's melting temperature for 24 h under vacuum. After cooling, the colored powder was thoroughly washed with an appropriate solvent until the wash solution became colorless, indicating removal of excess dye from the external surface.

Determination of Dye Loading

The loading level, expressed as occupation probability (ρ), was defined according to previously reported methods. The occupation probability represents the fraction of available zeolite unit cells occupied by dye molecules and was calculated using equations 1 and 2 (Hennessy, 1999).

$$\rho = \frac{\text{number of occupied sites}}{\text{total amount of sites}} \quad (\text{Eq. 1})$$

From (Eq. 1), one gets the following expression to calculate the loading or occupation probability (Devaux et al., 2013).

$$\rho = \frac{m_D}{M_D} \left(\frac{M_Z n_s}{m_Z} \right) \quad (\text{Eq. 2})$$

where M_D is the molar mass of the dye molecule; M_Z is the molar mass of zeolite KLTL per one unit cell (2883 g/mol); m_D and m_Z are the weights of dye and zeolite KLTL in grams, respectively; n_s represents the number of unit cells occupied by one dye, which is determined by the length of the dye molecule.

Determination of Effective Loading by HF Dissolution

The effective dye loading was determined by dissolving the zeolite framework using hydrofluoric acid. A representative sample (10 mg) of dye-loaded zeolite KLTL was suspended in 3 mL of ethanol in a polystyrene cuvette. Subsequently, 300 μL of a 4% aqueous HF solution was added to dissolve the zeolite matrix completely. After 20 min, the solution became transparent. The dye concentration was determined from the corresponding UV-visible absorption spectrum, and the effective loading level was calculated accordingly.

Spectroscopic Measurements

UV-visible absorption spectra were recorded using a UV-2550 UV-visible spectrophotometer. Dye solutions were measured in quartz cuvettes using various solvents. Diffuse reflectance spectra of dye-loaded zeolite KLTL were obtained using the same spectrophotometer equipped with an integrating sphere, with a slit width of 5 nm, a sampling interval of 0.1 nm, and a wavelength range of 200–800 nm. Fluorescence spectra were recorded on a PerkinElmer LS 50B luminescence spectrometer. Measurements were performed either on

dye solutions in quartz cuvettes or on dye-loaded zeolite samples suspended in chloroform at approximately 1 mg per 100 mL.

FRET Analysis

The efficiency of Förster resonance energy transfer (FRET) was evaluated by comparing donor dye fluorescence intensity in the absence and presence of the acceptor. The quenching efficiency (%Q) was calculated from the ratio of donor fluorescence intensities (Eq.3). The Förster distance (R_{DA}) was determined using Förster's theory, assuming a random orientation of transition dipoles ($K^2 = 2/3$) (Eq.4). The spectral overlap integral was calculated from the normalized donor emission and acceptor absorption spectra. Energy-transfer rate constants were estimated using established photophysical relationships (Cucinotta et al., 2014).

$$\%Q = \left(1 - \frac{F_{DA}}{F_D}\right) \times 100\% \quad (\text{Eq. 3})$$

where F_{DA} is the donor fluorescence intensity in the presence of the acceptor, and F_D is the donor fluorescence intensity in the absence of the acceptor (Lakowicz, 2006; Minkowski et al., 2006).

$$R_{DA}^6 = \frac{9000(\ln 10) \kappa_p^2 \Omega_{DA} \Theta_D}{128\pi^5 N_A n^4} \quad (\text{Eq. 4})$$

When K_p^2 is the orientation factor set to $2/3$, this assumes a random orientation of the emission and absorption dipole moments of the donor and acceptor, respectively. Ω_{DA} is the overlap integral between the donor emission spectrum and the acceptor absorption spectrum, Θ_D is the donor quantum yield, N_A is the Avogadro constant, and n is the refractive index of the solvent (1.33 for H₂O and 1.4 for zeolite). The overlap integral, Ω_{DA} , between the donor emission spectrum and the acceptor excitation spectrum is measured at the donor excitation energy (Insuwan & Rangsiwatananon, 2015; Saha et al., 2016; Viani et al., 2016). Although the dyes are confined within the one-dimensional KLTL channels, $K^2 = 2/3$ remains a reasonable approximation. This is because rapid rotational motion of the dyes about the channel axis, together with the distribution of tilt angles among occupied sites, dynamically averages the donor-acceptor dipole orientation over the donor excited-state lifetime, consistent with the convention adopted in previous dye-zeolite L FRET studies.

Results

Morphology of Zeolite KLTL

The morphology of the synthesized zeolite KLTL crystals was examined by scanning electron microscopy (SEM). As shown in Fig. 2, the crystals are round, with an average size of approximately $1 \times 2 \mu\text{m}$, consistent with previously reported zeolite LTL structures (Ban et al., 2007; Insuwan & Kunwadee, 2012).



Figure 2 Scanning electron microscopy images of Zeolite KLTL at 8,000 $\times$ magnification

Fluorescence Behavior of Acridine Confined in Zeolite KLTL

The absorption spectra of acridine hydrochloride (AC) in water and acidic solution are revealed in Fig. 3a. The acridine molecule exists as a protonated form when it is dissolved in water and acidic solution (0.1 M HCl), which shows the absorption peak at 355 nm, suggesting that the band has π - π^* character (Kubota & Steiner, 1977). It shows a broad band around 400–450 nm. Furthermore, the absorption maxima of AC in neutral, hydrogen-bonded, and protonated species appeared at 355 nm (Ryder et al., 2003). Only the spectrum of the protonated species (ACH^+) has a broad shoulder within the 370–430 nm range. The protonated form of AC (ACH^+) appeared in both water and 0.01 M HCl solution, but ACH^+ in 0.01 M HCl shows a broader shoulder with higher intensity than in water, indicating AC's preference for the protonated form. Because only a small amount of cationic AC was encapsulated in the KLTL channel via cation exchange, we used a gas-phase method to insert cationic dye, in addition to neutral dye. Similarly, neutral-dye AC was prepared with occupation probabilities of 0.10, 0.25, 0.50, and 1.00 and heated at 120 °C (the melting point of AC) under vacuum. The samples were prepared successfully and characterized for their luminescence properties. The samples of AC-KLTL zeolite with different occupation probabilities ($\rho = 0.10, 0.25, 0.50, \text{ and } 1.0$) were prepared by the gas-phase method. The maximum emission spectra are shown at 470–500 nm when excited at 355 nm. A superimposed image in Fig. 3b shows the normalized emission positions of all samples, and the emission shifts slightly to longer wavelengths (red shift) from 475 to 500 nm as the occupation probability increases ($\rho \geq 0.50$). This indicates dye aggregation in the zeolite KLTL channel, leading to self-absorption and non-radiative energy dissipation (Insuwan et al., 2015). Whereas occupation probabilities of 0.10 and 0.25 show enhanced emission intensity, which can be attributed to effective isolation of dye molecules within the zeolite nanochannels and suppression of aggregation-induced quenching (Minkowski et al., 2006).

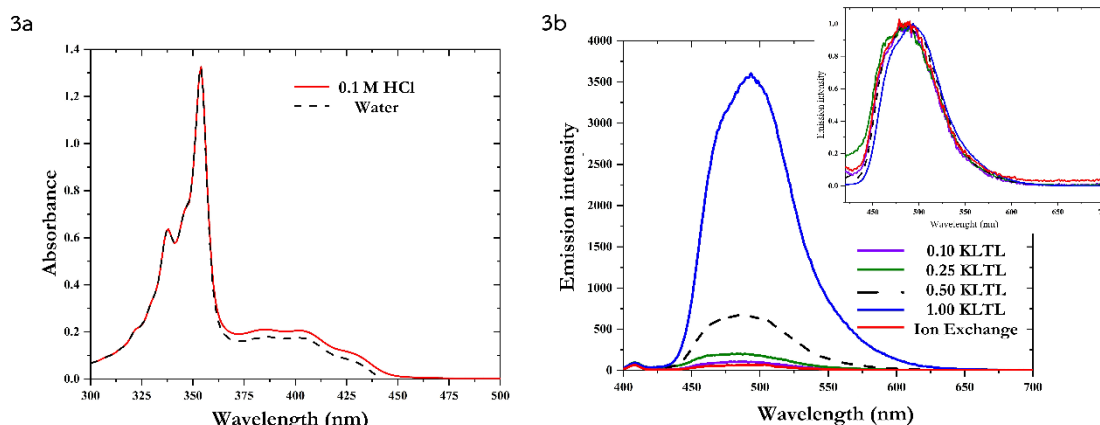


Figure 3 (a) The absorption spectra of AC in water and 0.1 M HCl solution and (b) Emission spectra of AC with different loadings onto KLTL zeolite by gas phase method, excited at 355 nm. Insert; normalizes spectra AC with different loadings onto KLTL zeolite

Photophysical Properties of hemicyanine iodide in Solution and Confined in KLTL

Normally, hemicyanine iodide dye has a low emission quantum yield in polar solvents ($\Phi = 0.07$) (Cerdán et al., 2012; Abdel-Halim, 2011). It is well-known that Hemi has a strong absorption band around 500 nm, which comes from the charge-transfer transition, and its derivatives in the past two decades show that the Hemi chromophore has negative solvatochromic behavior (absorption peak position blue-shifts through the increase of the solvent polarity), indicating that the ground state has a larger dipole moment than the excited state. The absorption and fluorescence spectra of Hemi in four different common solvents: Acetone, chloroform, ethanol and water. The absorption spectra clearly show negative solvatochromic behavior of Hemi (Fig. 4a). The red-shifted absorption spectra were observed in more polar solvents, indicating progressively greater stabilization of the excited state in a polar environment. However, in water, the ground state is stabilized more than the excited state of the hydrophobic Hemi molecule due to the quinonoid-to-benzenoid transformation (Huang et al., 2002; Sahoo et al., 2011), and the absorption band shifts blue compared with other polar solvents. Whereas Fig. 4b shows the fluorescence spectra of the probe molecule in different solvents. A significant shift in the peak position from 430 nm in a nonpolar solvent (like acetone) to 588 nm in a more polar solvent (like water) was observed. The fluorescence excitation spectra of Hemi in other solvents match well with the absorption spectra in the same solvents, indicating that each emission band arises from excitation of the same ground-state species. Consistent with this solvent dependence, recent studies on related interactions of hemicyanine dyes show that their fluorescence is controlled not only by solvent polarity but also by the surrounding host environment. When these dyes are confined within, or attached to the surface of, an inorganic host, their fluorescence can be either increased or decreased, depending on the distance between neighboring dye molecules, unlike their behavior in free solution (Assaf et al., 2022; Chen et al., 2025; Planetová et al., 2025).

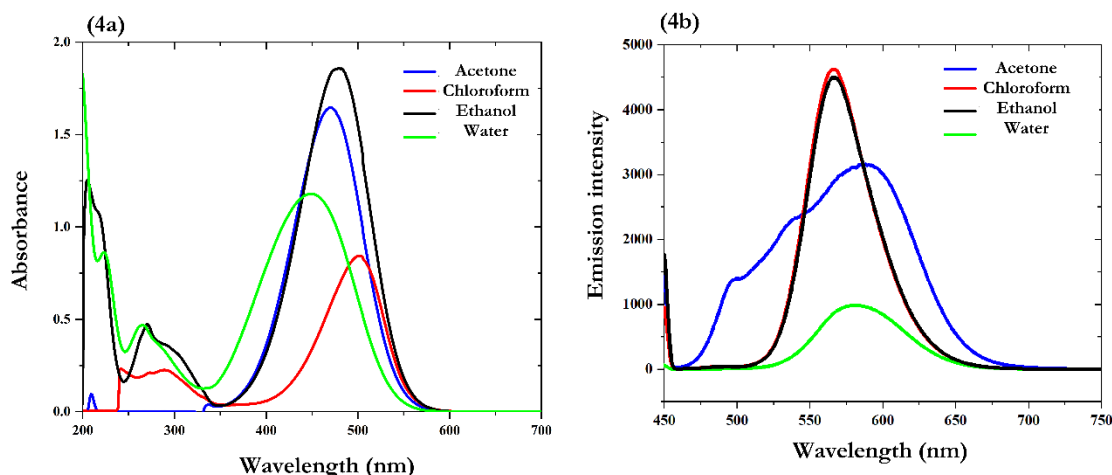


Figure 4 (a) The absorption spectra of hemicyanine iodide in various solvents and (b) Emission spectra of Hemi in different solvents, excited at 450 nm

Encapsulating Hemi within KLTL zeolite via gas-phase diffusion modifies its photophysical behavior. Recent studies show that including cyanine and hemicyanine dyes within methylated cyclodextrin hosts can restrict intramolecular motions and suppress non-radiative relaxation pathways, resulting in enhanced fluorescence emission compared with the free dye in solution (Chen et al., 2025). These findings support the concept that spatial confinement can modulate the excited-state dynamics of hemicyanine chromophores. The diffuse reflectance spectra of Hemi-loaded KLTL (Fig. 5a) confirm successful incorporation of the dye within the zeolite framework. As shown in Fig. 5b, the fluorescence intensity increases with occupation probability up to $\rho = 0.25$, followed by a pronounced decrease at higher loadings. This decrease can be attributed to aggregation-induced quenching facilitated by steric constraints and strong $K^+ - \pi$ interactions between the zeolite framework and the aromatic moieties of Hemi (Escudero et al., 2008; Ruan & Rodgers, 2004). Additionally, intramolecular twisting of the aniline ring within the confined environment further promotes non-radiative decay (see Fig. 6) (Huang et al., 2002; Sahoo et al., 2011).

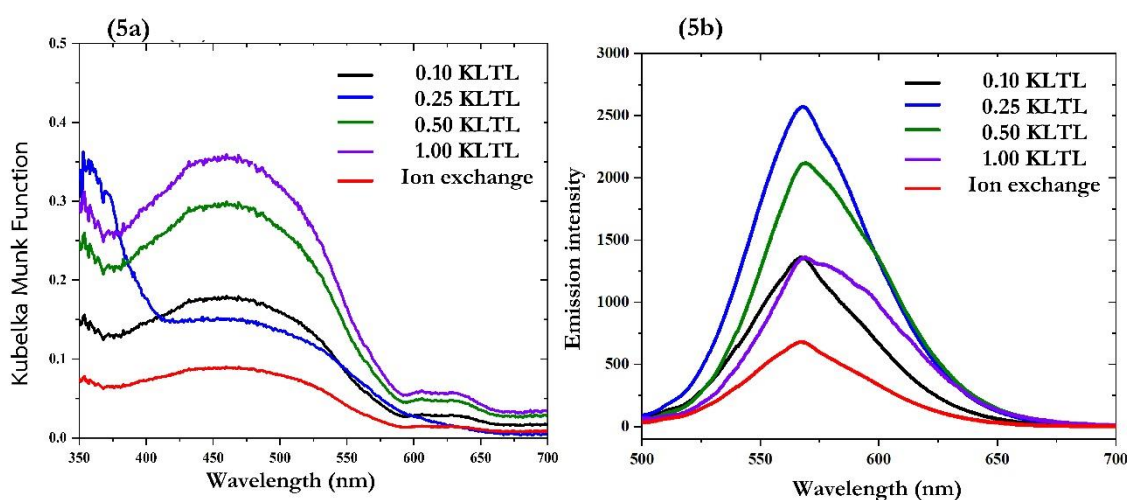


Figure 5 (a) Diffuse reflectance spectra of hemicyanine iodide in KLTL zeolite with various occupation probabilities and (b) the emission spectra of Hemi_KLTL zeolite with various occupation probabilities, excited at 470 nm

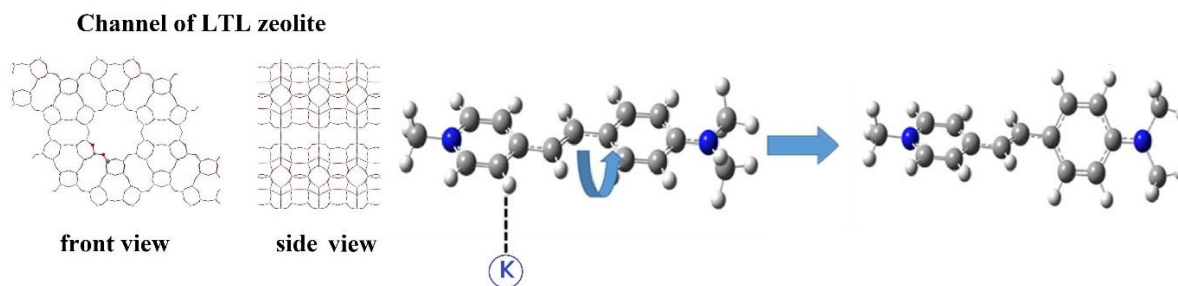


Figure 6 Twisting motion of Hemi chromophore through the torsion of the C–C bond (the aniline ring)

Dye Loading Efficiency

Table 1 summarizes the effective loading levels of AC and Hemi prepared by ion exchange and gas-phase diffusion.

Table 1 The amount of dyes in the KLTL after loading

ρ	AC		Hemi	
	Dye offer (mol g ⁻¹)	Dye uptake (mol g ⁻¹)	Dye offer (mol g ⁻¹)	Dye uptake (mol g ⁻¹)
0.10	1.73×10^{-5}	8.15×10^{-6}	1.15×10^{-5}	2.91×10^{-7}
0.25	8.76×10^{-5}	1.87×10^{-5}	2.86×10^{-5}	8.78×10^{-7}
0.50	4.33×10^{-5}	2.85×10^{-5}	5.76×10^{-5}	1.83×10^{-6}
1.00	1.51×10^{-4}	1.73×10^{-5}	1.15×10^{-4}	4.46×10^{-6}
Ion Exchange	1.00×10^{-4}	1.18×10^{-7}	1.00×10^{-4}	2.0×10^{-7}

As expected, gas-phase diffusion yields significantly higher dye uptake than ion exchange, particularly for the larger hemicyanine iodide molecules, which experience steric hindrance during ion exchange. However, excessive dye loading leads to aggregation and fluorescence quenching, highlighting the importance of optimizing the occupation probability for photophysical applications.

Spectral Overlap and Förster Resonance Energy Transfer

Energy transfer in this system is governed by long-range dipole–dipole interactions known as the Förster mechanism due to the absence of any orbital overlap between the dye molecules. Energy transfer processes occur when the separation distance between donor and acceptor species is at least 10 Å. Fig. 7 illustrates the absorption and emission spectra of both dyes (AC and hemi) in aqueous solution.

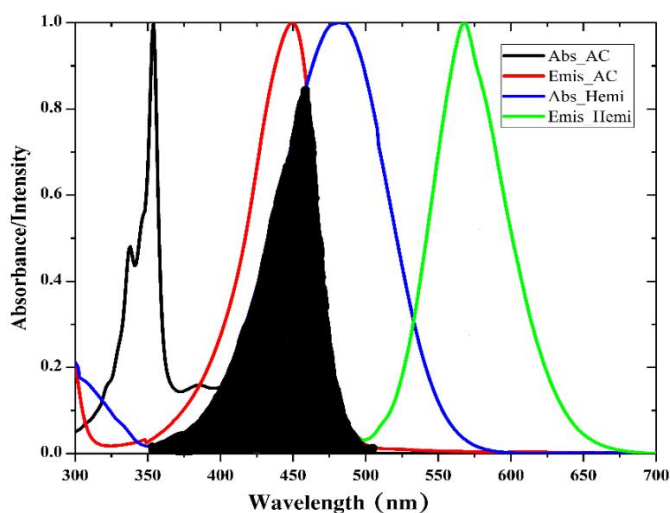


Figure 7 Energy transfer experiment: absorption and emission spectra of AC and Hemi dissolved in aqueous solution

Effect of Encapsulation Sequence on FRET Efficiency

The AC_Hemi KLTL samples prepared by different sequences using the method were loaded with a dye concentration of 0.1 mM by ion exchange and by gas phase with an occupation probability of 0.25 ($p=0.25$). Fig. 8 shows the emission spectra of these samples.

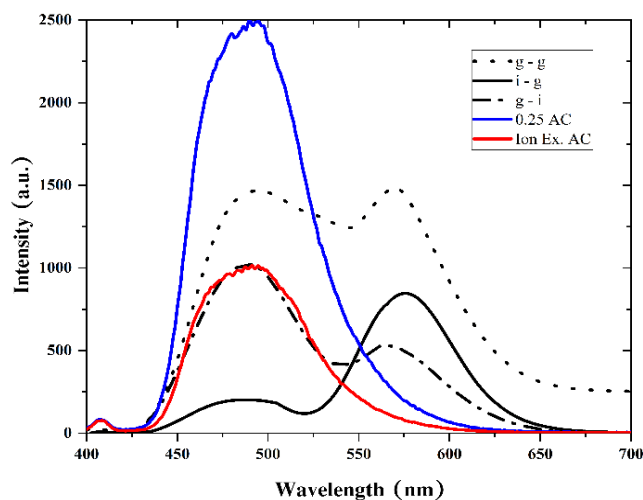


Figure 8 The emission spectra of AC and Hemi in the system of AC_Hemi KLTL with different sequences of the method for inclusion and that excited at 355 nm

The excitation wavelength of the dyes loaded on KLTL was fixed at 355 nm, which only AC (donor) can absorb. The emission spectra of AC loaded on zeolite KLTL were measured by ion exchange (i) and the gas-phase method (g). The emission spectra observed at 485 nm overlap with the excitation spectra of Hemi on KLTL (at 485 nm), implying that energy transfer might happen at an appropriate distance. The sequence of inserting AC and Hemi onto zeolite KLTL is important for energy-transfer efficiency. Table 2 summarises the calculated FRET parameters. In this case, AC_Hemi-KLTL prepared by ion exchange (i) – gas phase (g) shows higher quenching efficiency (65.9%) and the largest Förster distance (26.5 Å).

Table 2 Calculated overlap integral, quantum yield, Förster distance, and quenching efficiency for the donor–acceptor pairs of AC_Hemi KLTL

Sequence of the loading method	Overlap integral (Ω_{DA}) $10^{12} \text{ cm}^{-1} \text{ M}^{-1}$ $^1 (\text{nm})^4$	Quantum yield (Φ_D)	Forster distance, $R_{DA} (\text{\AA})$	Quenching efficiency (%Q)
AC_Hemi -KLTL				
i – g	15.58	0.66	26.5	65.9
g – i	10.75	0.66	24.3	46.5
g – g	12.56	0.66	24.7	58.7

Discussion

Encapsulating the photophysical properties of dyes (AC and Hemi) in zeolite KLTL channels can improve them. The occupation probabilities at 0.10 and 0.25 show stronger fluorescence emission, which can be attributed to the complete isolation of dye molecules within the zeolite nanochannels. In addition, interactions between extra-framework potassium ions in the zeolite and the dye molecules' aromatic rings can quench fluorescence via cation– π interactions. The antenna system (AC_Hemi-KLTL) was designed by the ion-exchange (i)–gas-phase (g) method. AC molecules absorb energy and then transfer it to Hemi molecules, resulting in visible-light emission. The quenching efficiency and Förster distance were 65.9% and 26.5 Å, respectively. This behavior suggests that encapsulating donor and acceptor dyes within zeolite channels reduces average separation distances and strengthens dipole–dipole coupling (Viani et al., 2016; Dounmanee et al., 2018). In contrast, gas-phase diffusion of both dyes leads to aggregation, decreasing the effective Förster distance and energy-transfer efficiency.

Conclusion and Suggestions

A variety of fluorescent dyes, such as acridine (AC) and hemicyanin (Hemi) were used in the dye-zeolite KLTL composite. The appropriate cationic and neutral dyes can be encapsulated onto KLTL zeolite. Not only the cation exchange but also the gas-phase method were used at different occupation probabilities ($p=0.10$, 0.25, 0.50, and 1.00). The KLTL zeolite samples prepared by the gas-phase method show a high amount of dye intercalated in the zeolite channels. High loading ($p = 0.50$ and 1.00) causes dye aggregation in the KLTL zeolite channels, leading to self-absorption and reduced luminescence. In the gas-phase method, we chose an occupation probability of 0.25 for the FRET study because it achieved high luminescence and avoided dye aggregation. The ion-exchange/gas-phase configuration exhibited the highest quenching efficiency (65.9%) and the largest Förster distance (26.5 Å), indicating favorable donor–acceptor spatial organization and strong dipole–dipole coupling. These results demonstrate the potential of confinement-controlled photophysics for designing artificial antenna systems and fluorescence-based photonic materials. The results provide valuable insight into how nanochannel environments and encapsulation strategies can tune fluorescence quenching and energy-transfer efficiency in host–guest systems.

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Author Contributions

Wilaiporn Insuwan: Conceptualization, Methodology, Formal Analysis, Writing – Original Draft.

Suriya Duangmanee: Investigation, Data Curation, Visualization.

Conflict of Interests

All authors declare that they have no conflicts of interest.

Declaration of Generative AI and AI-assisted Technologies

During the preparation of this manuscript, the authors used ChatGPT (OpenAI) for minor language editing and grammar checking. After using this tool, the authors thoroughly reviewed and revised the manuscript and take full responsibility for the final content.

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