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Valorization of BaCO₃ with 8-HQS to produce the optical chemo-probe for highly selective sensing of Cd²⁺ in aqueous media

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Valorization of chemicals is considered a method to prevent loss of valuable substances. Barium carbonate, as an initial precursor in chemical industries, is a suitable candidate for valorization to prevent the accumulation of waste BaCO₃ and the loss of these valuable resources. In this study, BaCO₃ was valorized with 8-hydroxyquinoline-5-sulfonate (8-HQS) to produce the optical chemo-probe for highly selective sensing of Cd²⁺ in aqueous media. The emission intensity of BaCO₃-8-HQS was enhanced in the presence of Cd²⁺ at 520 nm ($\lambda_{\text{ex}} = 392$ nm). The electrochemical experimental investigation, including cyclic voltammetry and Mott–Schottky techniques, revealed the ICT-enhancing mechanism of Cd²⁺ and showed Cd²⁺ interacts with the sulfonate head of BaCO₃-8-HQS. Furthermore, the as-prepared BaCO₃-8-HQS exhibited a good affinity and selectivity for sensing Cd²⁺, with a limit of detection of 2.2×10^{-7} M.

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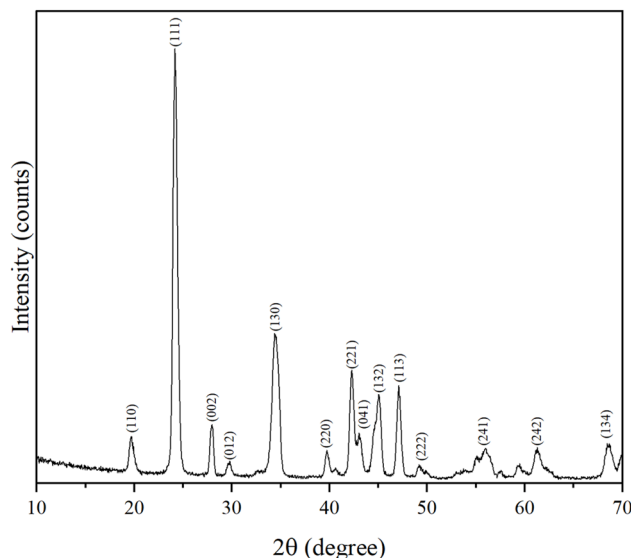
Introduction

Valorization refers to converting waste into high-value-added materials. One of the main challenges in recycling is the intricate process of separating waste into usable secondary raw materials. To tackle this important topic, tight controls on waste management and substantial investments in waste materials, valorization are being introduced. In this context, the idea of recycling and reusing waste to produce valuable products has inspired researchers to use waste for various applications.^{1–3} Nowadays, the industrial revolution has entered a fast-paced stage, leading to numerous developments. Consequently, through the industry's process, large amounts of precursors as industry raw materials can remain unusable or wasted. In this regard, the valorization of raw materials has been considered a sustainable method to obtain various high-value-added materials.^{4–6} Among these materials, barium carbonate stands out as a promising candidate for valorization due to its low cost, availability, and abundance as an inorganic compound.

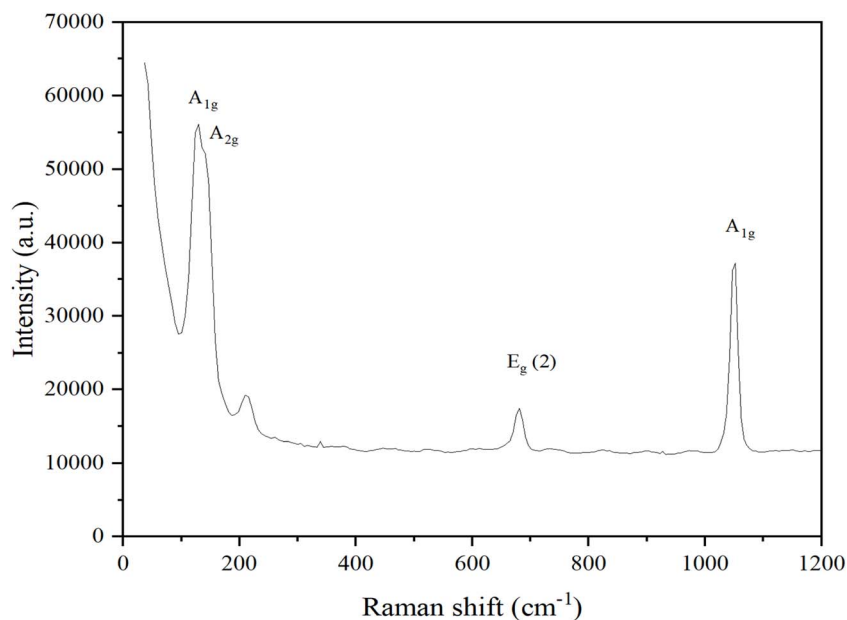
Barium is a white alkaline metal that exists in two forms: sulfate and carbonate. BaCO₃, also known as witherite, offers several significant advantages. In industry, barium carbonate is normally produced from barite and is enormously favorable for many applications in glass manufacturing, oil drilling, photography, ceramics, paint, brick production, and chemical

processes.^{7,8} However, in some industries and university laboratories, large quantities of BaCO₃ have gone to waste over the years.⁹ By valorization of this waste, BaCO₃ can be reused, reducing waste and contributing to sustainable development.

In recent years, drinking water pollution has become a significant issue that poses a threat to human society.^{10–12} As a common heavy metal pollutant, cadmium ions (Cd²⁺) are well-known for their detrimental effects on food safety, ecosystems,


 Fig. 1 XRD pattern of BaCO₃.

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Fig. 2 Raman spectrum of BaCO₃.

and human health. High concentrations of Cd²⁺ are highly toxic and can cause serious disasters.^{13,14} Traditional detection methods require skilled technicians, expensive instruments, and complex sample pretreatment, which limits their wide applications in routine testing.^{15–18} In this context, the development of optical chemo probes has garnered significant scientific attention. Optical chemo probes have successfully addressed these drawbacks, offering cost-effectiveness, high selectivity and sensitivity, instantaneous response, and operational simplicity.^{19–24} The notable fluorescence characteristics of 8-hydroxyquinoline (8-HQ) and its derivatives highlight their potential for developing selective and highly sensitive fluorescent chemo-sensors. Known as a chelating agent, 8-HQ exhibits weak fluorescence mainly due to a non-radiative relaxation

mechanism linked to intramolecular proton transfer (from the oxygen to the nitrogen of 8-HQ) in its excited state. When 8-HQ interacts with metal ions, the metal ion can replace the hydroxyl proton, thereby inhibiting the intramolecular proton transfer. Consequently, the binding of metal ions to 8-HQ can lead to a significant alteration in fluorescence intensity.²⁵ 8-Hydroxyquinoline-5-sulfonate (8-HQS) is a derivative of 8-HQ, a widespread moiety due to excellent properties for the formation of complexes with different metal ions and anions. Also, it

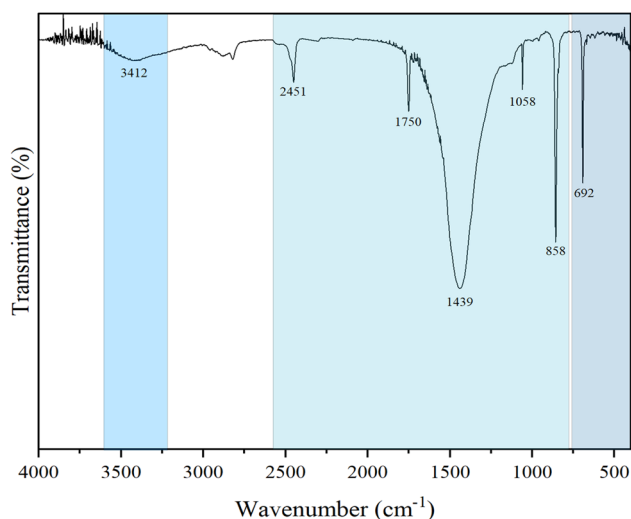
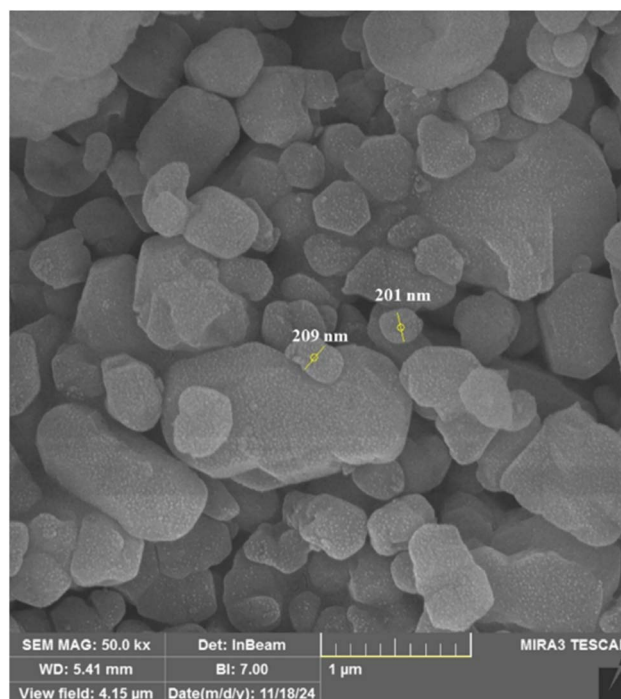
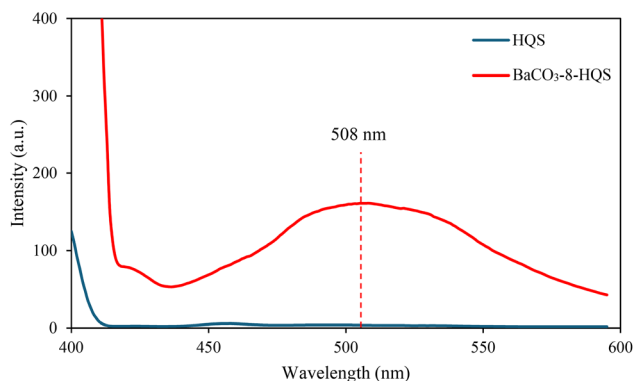
Fig. 3 FTIR spectrum of BaCO₃.Fig. 4 SEM image of BaCO₃.

Table 1 Zeta potential of BaCO₃ and BaCO₃-8-HQS

Sample	Zeta potential (mV)
BaCO ₃	-0.3
BaCO ₃ -8-HQS	-22.4

Fig. 5 Fluorescence spectra of HQS and BaCO₃-8-HQS.

can act as a fluorophore ligand in complex formations with transition metal ions.²⁶ The interaction between barium carbonate and 8-HQS will lead to an optical chemo probe.

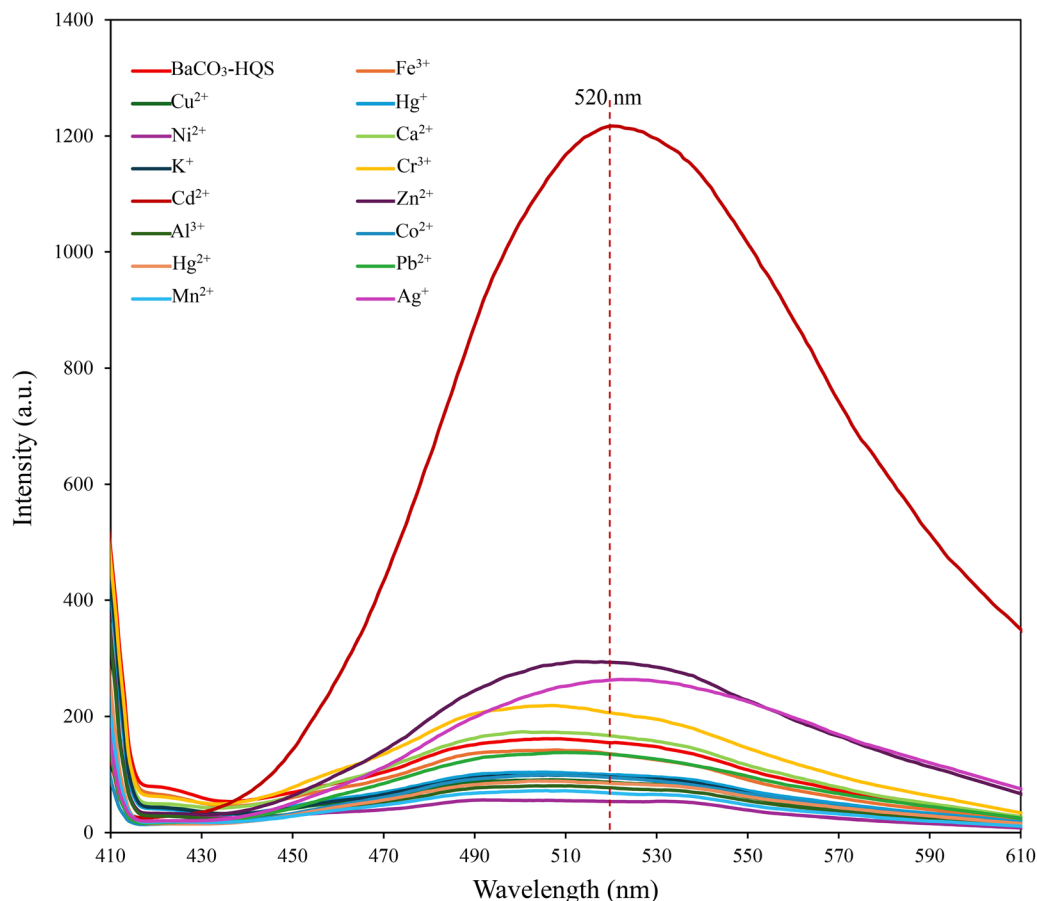
Herein, to the best of our knowledge, BaCO₃ has been valorized with 8-HQS to produce the optical chemo probe for the first time. The as-prepared optical probe shows high affinity to probe Cd²⁺ with high sensitivity (limit of detection (LoD) = 2.2 × 10⁻⁷ M) in aqueous media. The result showed a remarkable enhancement of emission intensity for sensing Cd²⁺. The BaCO₃-8-HQS was characterized by photoluminescence (PL), Mott-Schottky analysis, cyclic voltammetry, and zeta potential. These techniques were used to evaluate PL properties, electronic characteristics, and surface charge, respectively.

Chemicals

8-Hydroxyquinoline sulfonic acid (C₉H₇NO₄, ≥98% purity) was obtained from Sigma-Aldrich. The following ions were investigated in aqueous solutions: K⁺, Ag⁺, Hg⁺, Cu²⁺, Ni²⁺, Cr³⁺, Zn²⁺, Co²⁺, Pb²⁺, Ca²⁺, Cd²⁺, Hg²⁺, Mn²⁺, Fe³⁺, and Al³⁺. Analytical-grade metal salts, either chlorides or nitrates, were used to prepare the stock solutions. All materials were used as analytical grade, without any further purification.

Synthesis of BaCO₃-8-HQS

In an undergraduate inorganic chemistry laboratory class, BaCO₃ was synthesized through the aqueous titration of barium chloride with sodium carbonate. Unfortunately, it was

Fig. 6 Fluorescence spectra of BaCO₃-8-HQS in the presence of various metal ions in aqueous solution ($\lambda_{\text{ex}} = 392$ nm).

discarded at the end of the experiment. This study utilized the wasted BaCO₃ to illustrate the concept of waste valorization. For the preparation of BaCO₃-8-HQS, 0.01 g of BaCO₃ was dispersed in 50 mL of deionized water and stirred for 1 h. Then, 8-HQS (0.01 g) was introduced to it and sonicated for 20 min.

Characterization of BaCO₃

The crystal phase pattern of BaCO₃ was investigated by powder X-ray diffraction (XRD) with Cu K α irradiation in the 2θ range of 10–80° (Rigaku Ultima IV). Fourier transform infrared spectroscopy (FT-IR), Rayleigh WQF-510A, was utilized to examine the structure of the BaCO₃ in the range of 400–4000 cm⁻¹. Raman spectroscopy was utilized to investigate crystalline structure stability with the Teskan N1-541 instrument. The fluorescence spectra were used at ambient temperature on an Agilent-G980A spectrofluorometer. A Raleigh UV-1600 spectrophotometer was used for UV-vis spectroscopy studies. The morphology of BaCO₃ was investigated by scanning electron microscopy (MIRA3-Tescan). Zeta potential data were collected by Horiba SZ-100 to examine the surface charge. The Mott-Schottky (M-S) analysis and cyclic voltammetry (CV) were performed using an Ivium Vertex with a 0.1 M Na₂SO₄ electrolyte

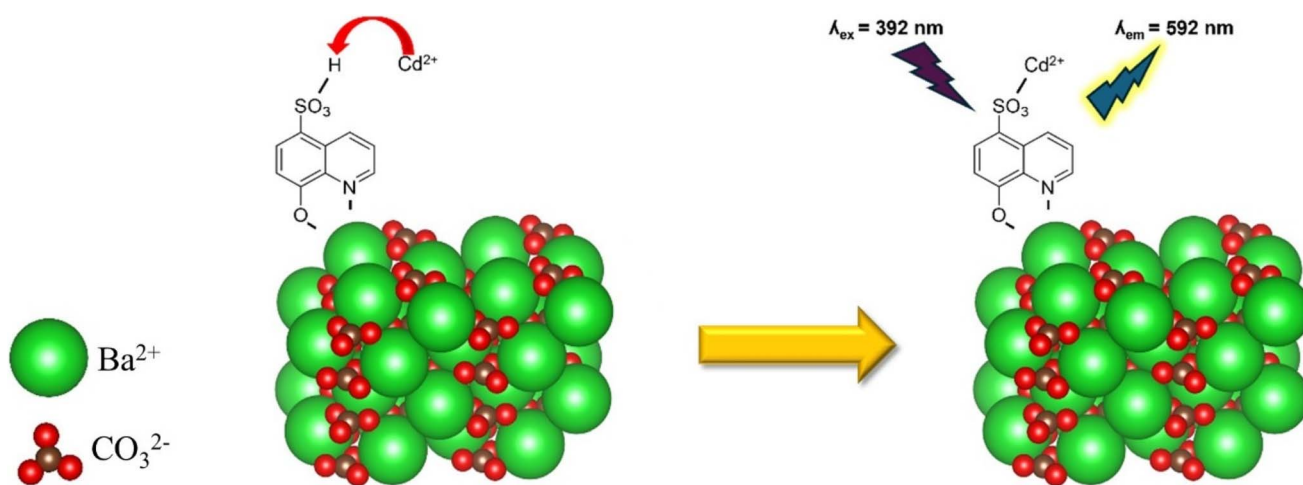
solution. The M-S analysis was conducted at a frequency of 1467.8 Hz.

Results and discussion

The powder X-ray diffractometry has been utilized to analyze the crystallographic phases. Fig. 1 depicts the obtained XRD pattern of the prepared sample. In the XRD pattern of BaCO₃, the diffractions located at 19.72°, 24.16°, 27.94°, 29.79°, 34.43°, 39.74°, 42.26°, 43.03°, 45.04°, 47.09°, 49.16°, 55.93°, 61.24°, and 68.55° can be respectively assigned to the (110), (111), (002), (012), (130), (220), (221), (041), (132), (113), (222), (241), (242), and (134) planes of the BaCO₃ crystalline structure.²⁷ According to the Scherrer equation, which is given in eqn (1), the average crystallite size of BaCO₃ was calculated and obtained as 21.54 nm.

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

Raman spectroscopic analysis has been utilized to get a deep understanding of the crystalline structural stability of as-prepared BaCO₃. Fig. 2 illustrates that the as-prepared BaCO₃



Scheme 1 The feasible interaction pathway between BaCO₃-8-HQS and Cd²⁺.

Table 2 Performance comparison of reported studies for sensing Cd²⁺

Sensor	LoD (μ M)	Measured signal	Ref.
(E)-4-((2-(2,4-Dinitrophenyl)hydrazineylidene)methyl)-N,N dimethyl-aniline	0.0756	Fluorescence	31
5-(4-(Dimethylamino)phenyl)-3-(2-hydroxyphenyl)-4,5-dihydro-1H-pyrazole-1-carbothioamide	0.8158	Fluorescence	32
Hydroxy-naphthaldehyde and tris(2-aminoethyl)amine (TREN)	0.0678	Fluorescence	33
5-(4'-([2,2':6',2''-Terpyridin]-4'-yl)-[1,1'-biphenyl]4-yl)7,8,13,14-tetrahydrodibenzo [a, i] phenanthridine (TBTP)	0.01181	Fluorescence	34
GTaunPs	0.025	Fluorescence	35
GSH-AunPs	0.0188	Fluorescence	36
N, B-CQDs	0.45	Fluorescence	37
AgInZnS QDs	0.0378	Fluorescence	38
Eu-MOF	0.00991	Fluorescence	39
BaCO ₃ -8-HQS	0.224	Fluorescence	This study



has the signature of internal CO_3 Raman bands at 1054 cm^{-1} (A_{1g}) and 686 cm^{-1} ($E_g(2)$), which belong to symmetric stretching (ν_1) and in-plane bending (ν_2), respectively. Furthermore, the prepared sample has a couple of strong lattice Raman bands at 149 and 130 cm^{-1} that belong to A_{2g} and A_{1g} Raman bands, respectively.²⁸

Fig. 3 illustrates the FT-IR spectrum of the as-prepared sample. The peaks at 1058 cm^{-1} and 1439 cm^{-1} might be due to the symmetric vibration of $\nu_s[\text{COO}^-]$ and the asymmetric vibration of $\nu_{as}[\text{COO}^-]$, respectively. In the fingerprint region, the BaCO_3 peak could be observed at 692 cm^{-1} ; the out-of-plane bending appears at 858 cm^{-1} and the overtone/combination band is observed at 1750 cm^{-1} .^{29,30} The peak at 3412 cm^{-1} is characteristic of the O-H bond.

Fig. 4 shows the SEM images depicting the morphology of BaCO_3 . The particle size of BaCO_3 was calculated by the digimizer software and obtained as approximately 205 nm . The zeta potential of the BaCO_3 -8-HQS and BaCO_3 samples was investigated. The zeta potential also confirms the interaction between 8-HQS and BaCO_3 (Fig. S1). According to the result, the zeta potential was -0.3 and -22.4 mV for BaCO_3 and BaCO_3 -8-HQS, respectively (Table 1). Based on the zeta potential results, 8-HQS interacts with BaCO_3 through its nitrogen and oxygen atoms, while the sulfonic acid head remains free. Consequently, the zeta potential increases after the interaction of BaCO_3 with 8-HQS.

The photoluminescence properties of 8-HQS and BaCO_3 -8-HQS were investigated to confirm the interaction of 8-HQS and

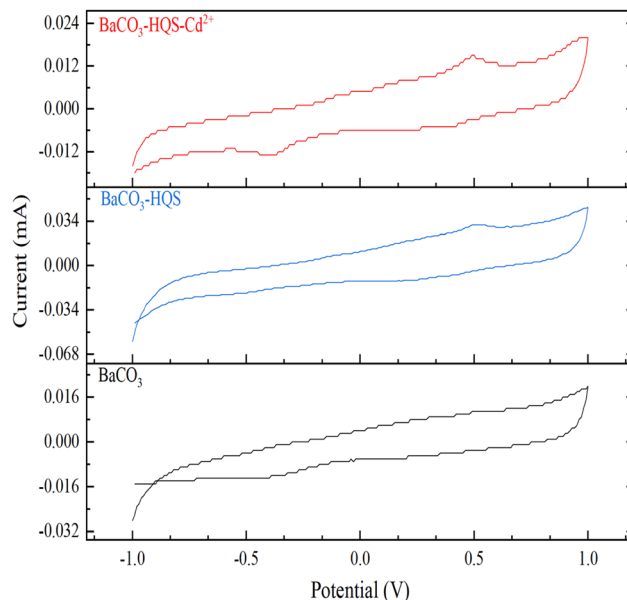


Fig. 8 The cyclic voltammetry (CV) plots for three conditions: the BaCO_3 , BaCO_3 -8-HQS, and BaCO_3 -8-HQS in the presence of Cd^{2+} .

BaCO_3 (Fig. 5). The result clearly demonstrates the successful interaction of BaCO_3 with 8-HQS. The excitation wavelength (λ_{ex}) was set to 392 nm for PL analysis. 8-HQS did not illustrate any emission intensity, but after introducing BaCO_3 , its emission intensity was enhanced at 508 nm .

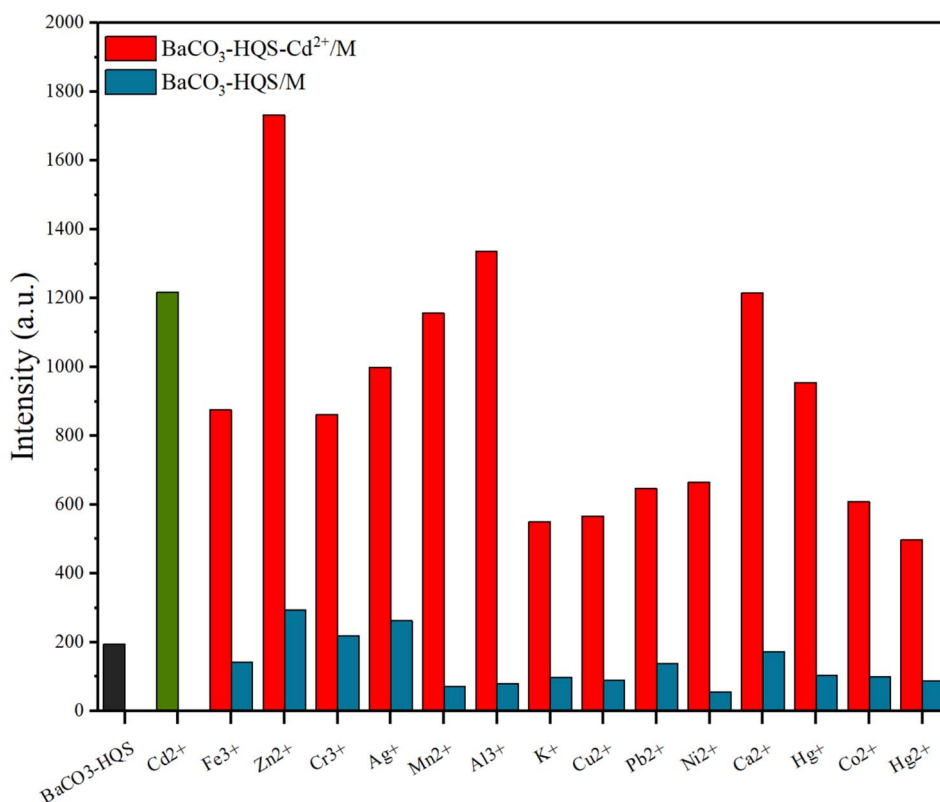


Fig. 7 The emission intensity of BaCO_3 -8-HQS in the presence of Cd^{2+} and interfering species.



Photoluminescence examination

The PL behavior of BaCO₃-8-HQS was investigated in the presence of a broad range of metal ions, including K⁺, Ag⁺, Hg⁺, Cu²⁺, Ni²⁺, Cr³⁺, Zn²⁺, Co²⁺, Pb²⁺, Ca²⁺, Cd²⁺, Hg²⁺, Mn²⁺, Fe³⁺, and Al³⁺. For the PL investigation, 5 μL of a desired concentration of metal ion (0.01 M) was added to 2 mL of BaCO₃-8-HQS, and then the entire sample was transferred into a cuvette. Fig. 6 illustrates the sensing results of the BaCO₃-8-HQS with these metal ions. According to the results, adjusting the excitation wavelength to 392 nm slightly enhanced the emission intensity of BaCO₃-8-HQS in the presence of Ag⁺, Ca²⁺, Cr³⁺, and

Zn²⁺. However, the emission intensity was quenched in the presence of other metal ions. Notably, upon the addition of Cd²⁺, emission intensity significantly increased, accompanied by a red shift to 520 nm. Scheme 1 illustrates the proposed interaction pathway between BaCO₃-8-HQS and Cd²⁺ through optical chemo sensing. For LoD determination of Cd²⁺, the as-prepared BaCO₃-8-HQS was titrated with a design concentration of Cd²⁺ (Fig. S2). The LoD for sensing Cd²⁺ was obtained as 2.2×10^{-7} M. Table 2 shows the comparison of different optical chemo-sensors to probe Cd²⁺.

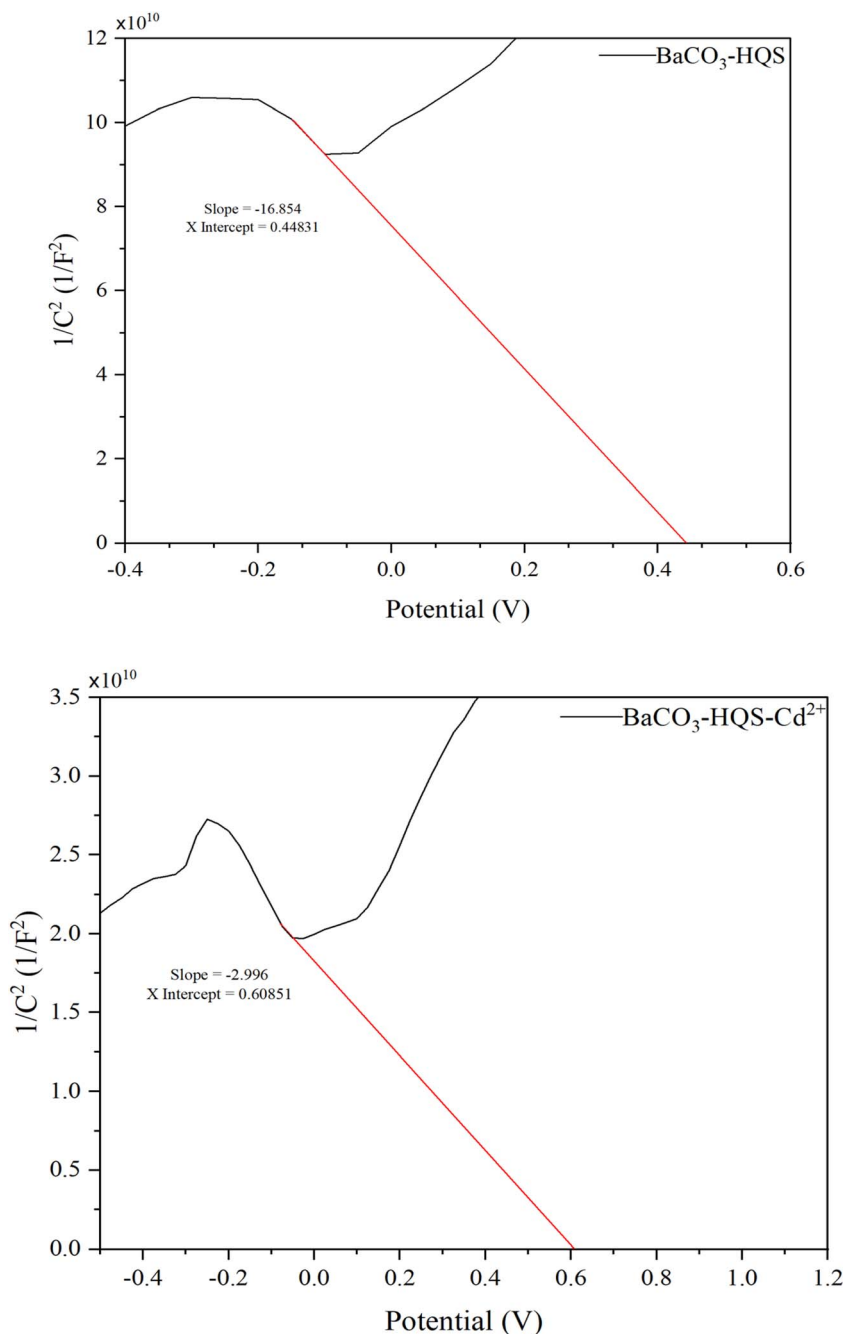


Fig. 9 Mott-Schottky plots for BaCO₃-8-HQS and BaCO₃-8-HQS+Cd²⁺ in a 0.1 m Na₂SO₄.



For the investigation of the selectivity and affinity of the as-prepared BaCO₃-8-HQS, the emission intensity was measured for detecting Cd²⁺ in the presence of interfering species ($\lambda_{\text{ex}} = 392 \text{ nm}$). The results showed that the emission intensity of the BaCO₃-8-HQS + Cd²⁺ complex was not completely quenched by these interfering species (Fig. 7). Therefore, the BaCO₃-8-HQS demonstrated a high selectivity and stability for sensing Cd²⁺.

Enhancing mechanism

According to the principles of intramolecular charge transfer (ICT) in view of receptors, within the same molecular framework, an electron-acceptor fragment is interconnected with an electron-donor fragment, leading to the establishment of a "push-pull" electronic condition in the excited state. The interaction of the electron-donating part with the analyte (such as a cation) leads to a reduction in the electron-donor capability of the prepared probe. This change results in a blue shift in the absorption spectrum of the sensor. Conversely, the interaction of the electron-accepting part with the analyte, leading to a hyperchromic shift, occurs in the absorption spectrum due to the predominant ICT. Furthermore, the ICT phenomenon may either be enhanced or constrained, leading to either a hyperchromic or hypsochromic shift in the PL properties of the probe.⁴⁰ To investigate the enhancing mechanism, UV-Vis spectroscopy and M-S analysis were utilized. M-S is used by a standard three-electrode electrochemistry method, including the working electrode (glassy carbon), counter electrode (the platinum wire), and reference electrode (Ag/AgCl). M-S analysis of BaCO₃, BaCO₃-8-HQS, and BaCO₃-8-HQS+Cd²⁺ samples was carried out in a 0.1 M Na₂SO₄ aqueous solution. To ensure results from the M-S analysis, CV analysis was performed to investigate the electrical properties of BaCO₃-8-HQS. According to Fig. 8, BaCO₃ shows no anodic or cathodic peaks in the CV. However, after the interaction with 8-HQS, anodic peak

potentials appeared, and following the introduction of Cd²⁺, both anodic and cathodic peaks were observed. According to the results, the bond gap potential of BaCO₃-8-HQS decreased after interaction of BaCO₃-8-HQS with Cd²⁺. In the ICT-enhancing mechanism reviewed by Paul and co-authors,⁴¹ it is noted that the analyte can interact with acceptor and donor heads, yielding different results in PL and UV-Vis spectroscopies. Therefore, determining the highest occupied molecular orbital (HOMO) energy level of the optical chemo probe before and after interaction with the analyte can show which part of the optical chemo probe interacts with the analyte. When the analyte interacts with the acceptor part, the HOMO energy level of the optical chemoprobe decreases. When the analyte interacts with the donor part, the HOMO energy level of the optical chemo probe increases. In this regard, M-S analysis was utilized to estimate this mechanism. For M-S measurements, the frequencies adjusted at 1467.8 Hz reveal the electronic characteristics of BaCO₃-8-HQS and BaCO₃-8-HQS+Cd²⁺, with negative slopes illustrating the p-type nature of the electronic band

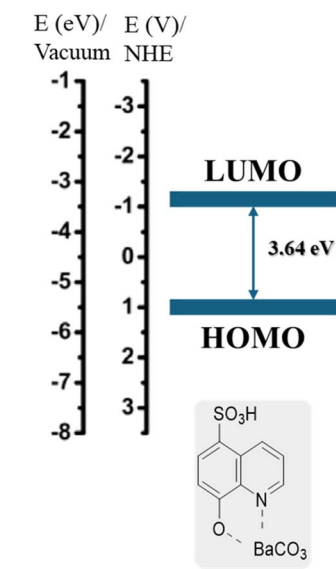
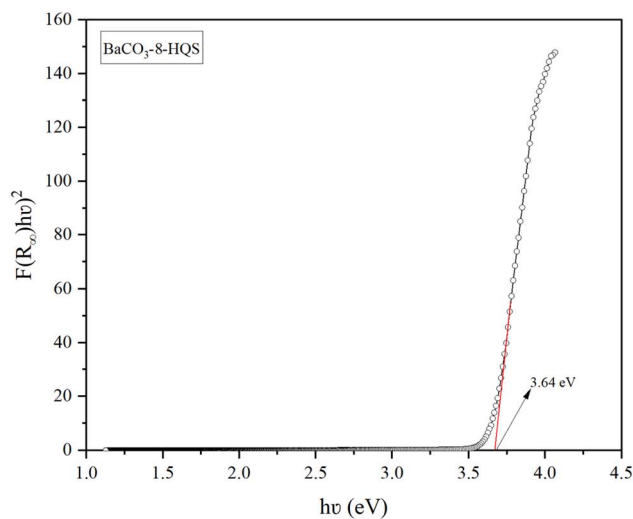


Fig. 10 Illustration of HOMO and LUMO energy levels of BaCO₃-8-HQS and BaCO₃-8-HQS + Cd²⁺.

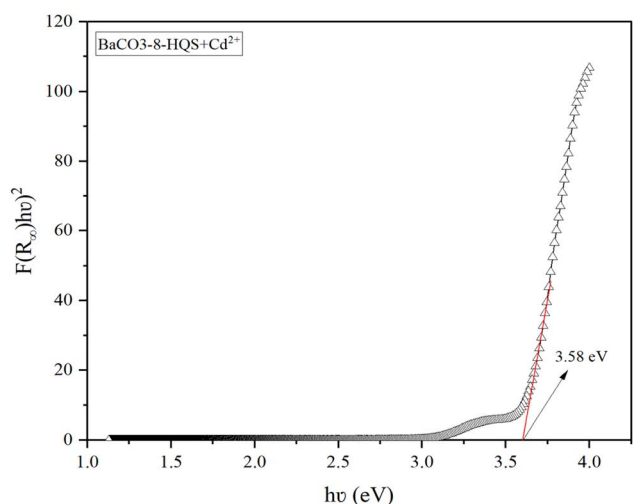


Fig. 11 Bond gap analysis of BaCO₃-8-HQS and BaCO₃-8-HQS + Cd²⁺ using Tauc equation.



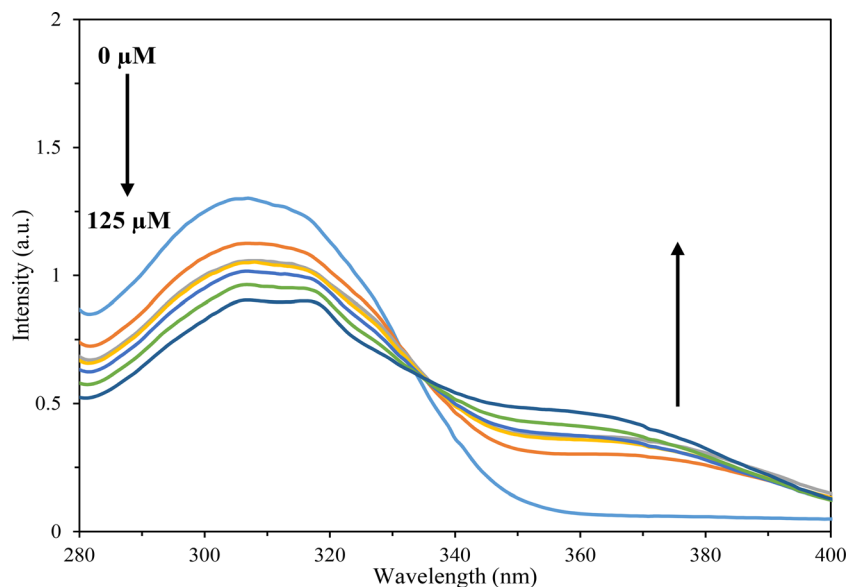


Fig. 12 UV-Vis absorption spectral changes through adding Cd²⁺ with varying concentrations.

structure (Fig. 9). The intersection point is the flat band position, examined from the intersection, which is 1.06 V (−5.56 eV) and 1.22 V (−5.72 eV) *versus* RHE for BaCO₃-8-HQS and BaCO₃-8-HQS+Cd²⁺, respectively (Fig. 10), approximately corresponding to the HOMO. According to results, Cd²⁺ interacts with the acceptor part of BaCO₃-8-HQS, specifically with the sulfonate head of 8-HQS. For determining bond gap of BaCO₃-8-HQS and BaCO₃-8-HQS+Cd²⁺, Tauc equation⁴² was utilized to obtain bond gap and lowest unoccupied molecular orbital (LUMO). According to Fig. 11, the bond gaps of BaCO₃-8-HQS and BaCO₃-8-HQS+Cd²⁺ were determined to be 3.64 eV and 3.58 eV, respectively.

The UV-Vis spectroscopy was employed to investigate the effect of introducing Cd²⁺ ion into the as-prepared chemo probe suspension. The results showed that a little red shift was observed as the concentration of Cd²⁺ increased from 0 μM to 125 μM. Furthermore, this complexation of BaCO₃-8-HQS with Cd²⁺ resulted in the noted isosbestic point approximately at 335 nm (Fig. 12). According to the result, the enhancing mechanism is examined as an ICT.

pH effect examination

The environmental factors affecting optical chemo probes are crucial for the detection of analytes. Among these factors, the pH of the environment plays a significant role in sensory performance. In this context, the performance of the BaCO₃-8-HQS was investigated across a wide pH range. Fig. S3 clearly shows that the probe remains stable within a narrow pH range of 5 to 6. In harsh acidic conditions, the emission intensity is completely quenched. In the pH range of 7 to 10, the emission intensity decreases but stabilizes at a certain level. Based on the results obtained, the optimal pH range for the as-prepared optical probe for detecting Cd²⁺ is thus identified as 5 to 6.

Conclusion

In summary, this work represented the valorization of barium carbonate to a high-value-added compound based on an optical chemosensor as a low-cost and widely available material that did not require complex functionalization or energy-intensive synthesis. It is noteworthy that the BaCO₃-8-HQS demonstrates remarkable selectivity and good sensitivity for Cd²⁺ ions in aqueous media (LoD = 2.2 × 10^{−7} M). The enhancing mechanism of Cd²⁺ was experimentally determined as ICT and revealed that Cd²⁺ interacts with the sulfonate head of 8-HQS, which was investigated using the electrochemical techniques such as CV and M-S. According to the results, the HOMO energy level of BaCO₃-8-HQS and BaCO₃-8-HQS+Cd²⁺ was obtained as −5.56 eV and −5.72 eV *versus* RHE, respectively. Furthermore, the bond gap of BaCO₃-8-HQS and BaCO₃-8-HQS+Cd²⁺ were calculated using Tauc equation, yielding 3.64 eV and 3.58 eV, respectively. The development of this simple method can open a new path for the development of optical chemo probes, specifically with metal oxides, to selectively probe heavy metal ions in aqueous media.

Conflicts of interest

All authors declare that they have no conflicts of interest.

Data availability

The data which support the findings of this study are available from the corresponding author, upon request and also the data supporting this article have been included as part of the supplementary information (SI). Supplementary information is available. See DOI: <https://doi.org/10.1039/d6ra01012a>.



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