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"Environmental Pollution and Challenges of Environmental Sustainability"

(Reconciliation with the Environment)

Sirte, October, 14, 2025



Edited by:

Prof. Abdussalam Salhin Mohamed.

Prof. Hisain Masoud Abomadina.

Dr. Ashraf Mustafa Abusenaina.

Dr. Fathia Abdulljawad Mosa

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Environmental Pollution and Challenges of
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Investigation of Solvent Effects on Spectroscopic and Photochemical Properties of Ruthenium (II) Polypyridyl Complexes

Hadeel Ali Mohammed, Hamid. M. Younis

Chemistry Department, Faculty of Science, Sirte University, Sirte-Libya.

 Corresponding Author Email: hamid.younis@su.edu.ly

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Abstract: This study explores the influence of solvent environments on the spectroscopic and photochemical properties of ruthenium(II) polypyridyl complexes, with a focus on their application in sustainable hydrogen production and solar energy conversion. These complexes show great potential as photocatalysts for clean energy technologies; however, their efficiency and stability are highly dependent on the surrounding chemical environment. Using high performance liquid chromatography (HPLC) and UV-Vis spectroscopy, the research evaluates electronic transitions and photochemical behavior under continuous irradiation. Notably, the complex $[(tbbpy)_2Ru(tpphz)(PtI_2)_2]$ exhibited a significant metal-to-ligand charge transfer (MLCT) shift from 486 nm to 441 nm over 24 hours in polar solvents such as acetonitrile and methanol indicating solvent-induced ligand exchange and enhanced charge transfer. The inclusion of PtI_2 substantially improved photocatalytic performance, achieving a turnover number (TON) of 276. Measurement precision was confirmed by low standard deviations (± 1.1 to ± 1.5 nm) and relative standard deviation (RSD) values below 3%. These findings highlight the critical role of solvent polarity and ligand design in optimizing photocatalyst efficiency and stability for sustainable energy applications. While polar solvents enhance charge-transfer processes, they may compromise long-term stability. The study recommends the development of more robust ligands and the exploration of environmentally friendly solvents to advance photocatalytic technologies in line with environmental sustainability goals.

Introduction

Ruthenium(II) polypyridine complexes, such as $[\text{Ru}(\text{bpy})_3]^{2+}$, have been extensively studied over the past four decades due to their unique photophysical and electrochemical properties. (Visco & Chandross, 1964; Santhanam & Bard, 1965; Lytle & Hercules, 1969). Since the discovery of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2$ as a photostable and highly luminescent compound exhibiting emission from triplet metal-to-ligand charge transfer ($^3\text{MLCT}$) states, considerable attention has been directed toward understanding and utilizing their ground and excited-state behavior (Paris & Brandt, 1959; Njabon, 2013).

Among transition metal complexes, Ru(II) polypyridyl systems are particularly notable for their strong luminescence in solution at room temperature and their excellent photosensitization capacity for both electron- and energy-transfer processes. These attributes have made them model systems in the investigation of excited-state dynamics and the development of light-driven chemical transformations (Kalyanasundaram, 1982; Johannes & Mary, 2010; Njabon, 2013; Kenneth, 2015).

The global energy crisis and increasing reliance on fossil fuels have raised serious environmental concerns, particularly regarding greenhouse gas emissions, climate change, and air pollution. (Alexandra et al., 2021; Chanana et al., 2023). As a result, the development of clean and sustainable energy conversion technologies has become an urgent priority. Solar energy, being abundant and environmentally friendly, presents a viable solution. Among the various strategies under investigation, artificial photosynthesis offers a promising path to reduce environmental pollution by converting water and carbon dioxide into clean fuels using sunlight (James & Gary, 2006; Sinval et al., 2019; Schulz et al., 2022).

In this context, the application of Ru(II)-based photocatalysts for hydrogen production represents a significant step toward sustainable fuel generation and the mitigation of CO_2 emissions. In particular, dinuclear Ru/Pt complexes have demonstrated high intramolecular electron transfer efficiency, enabling the effective conversion of protons into molecular hydrogen (H_2) (Lämmle et al., 2022; Liu et al., 2023; Putra et al., 2023).

The choice of bridging ligands such as tetrapyrido[3,2-a:2',3'-c:2'',3'''-j]phenazine (tpphz) plays a pivotal role in directing electron flow toward the catalytic site, thus enhancing photocatalytic efficiency (Karnahl et al., 2011; Karnahl et al., 2012; Halpin et al., 2013).

Figure 1 illustrates the proposed mechanism of hydrogen generation mediated by $[\text{Ru}(\text{L})_2(\text{tpphz})]^{2+}$. Comparisons with analogous iridium (Ir) and osmium (Os) complexes suggest that Ru-based systems offer an optimal combination of photostability, efficiency, and cost-effectiveness. Furthermore, theoretical studies indicate that strong π -acceptor ligands can promote long-lived charge separated states, further improving catalytic performance (Inagaki & Akita, 2010; Gedert et al., 2021).

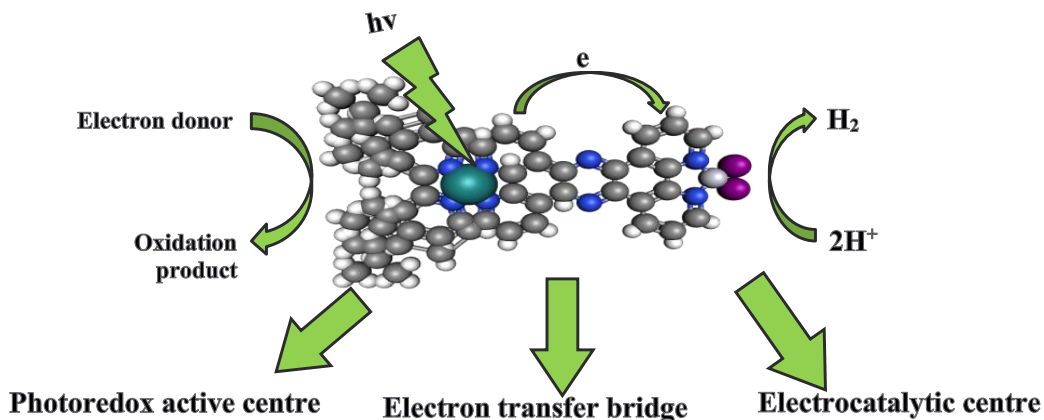


Figure 1: Mechanism of Photocatalytic Hydrogen Generation Utilizing $[Ru(L)_2tpphz]^{2+}$. (Rau et al., 2006)

This study seeks to build upon prior research by exploring the structural and electronic features of Ru(II) polypyridyl complexes with bridging ligands such as tpphz, with a particular focus on their photocatalytic activity in hydrogen generation. Specifically, we aim to elucidate the role of ligand architecture in modulating electron transfer pathways and optimizing the efficiency of light-driven proton reduction. Through a combination of spectroscopic analysis and theoretical calculations, this work contributes to the advancement of environmentally sustainable photocatalytic systems that support global decarbonization goals.

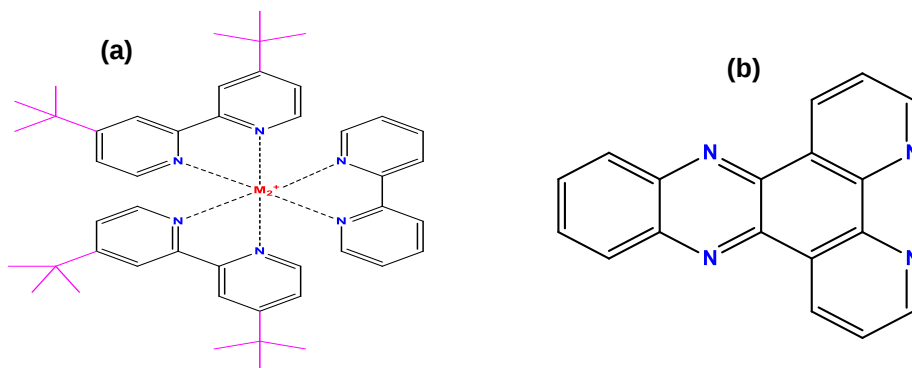


Figure 2: Structures of (a) peripheral ligand tbbpy, (b) bridging ligand tpphz

Experimental

Materials and methods

The photochemistry for the compounds has been investigated through High-Performance Liquid Chromatography (HPLC) and Ultraviolet-Visible (UV-Vis) Spectroscopy. These measurements were conducted in the chemistry laboratory at Sirte University.

Each of the compounds studied were synthesized and supplied by Prof. Sven Rau's research group at Ulm University, Albert-Einstein- Allee, Ulm, Germany. The samples were utilized as received, without additional purification. All solvents employed for spectroscopic measurements were of spectroscopic grade (Sigma, Aldrich).

Photochemical Study

Photochemical studies were conducted at room temperature using air equilibrated solutions in a 1 cm quartz cell. The samples were irradiated with a 10 W blue light emitting diode (LED) light source at 470 nm. This light source produced a broad spectrum similar to that of solar energy. The LED used was of type A2022-110-BLUE ($\lambda = 470$ nm). A 2 mL sample of the solution was irradiated at 470 nm (blue light) for a specified irradiation time.

Samples Preparation for Photolysis

Each of the samples was utilized as got, without additional purification. All measurements were conducted in air, unless specified otherwise. Sample solutions were prepared under conditions devoid of ambient light, covered with aluminum foil, and securely sealed to mitigate light exposure and evaporation. Samples were prepared fresh immediately prior to measurements. Complexes were generated in a $C \sim 1 \times 10^{-4} M$ solution in acetonitrile, methanol, dimethyl sulfoxide and (50:50) acetonitrile: methanol combination.

Results and discussion

Chromatographic HPLC Analysis

HPLC tests were done on the dinuclear compound $[(tbbpy)_2Ru(tpphz)(PtI_2)_2]$. The chromatograms are shown in Figures 3 (a–c). In CH_3OH , $[(tbbpy)_2Ru(tpphz)(PtI_2)_2]$ has a strong peak with a retention time of (2.9 minute). In CH_3CN , it has split peaks with retention times of 1.6 and 1.9 minutes and 2.1 minutes, likely due to chelation and partial electrostatic interactions. The splitting may also be attributed to the compound's molecular size. Small peaks at 1.2 and 0.7 minutes in CH_3OH and CH_3CN may be caused by synthetic impurities that are still present.

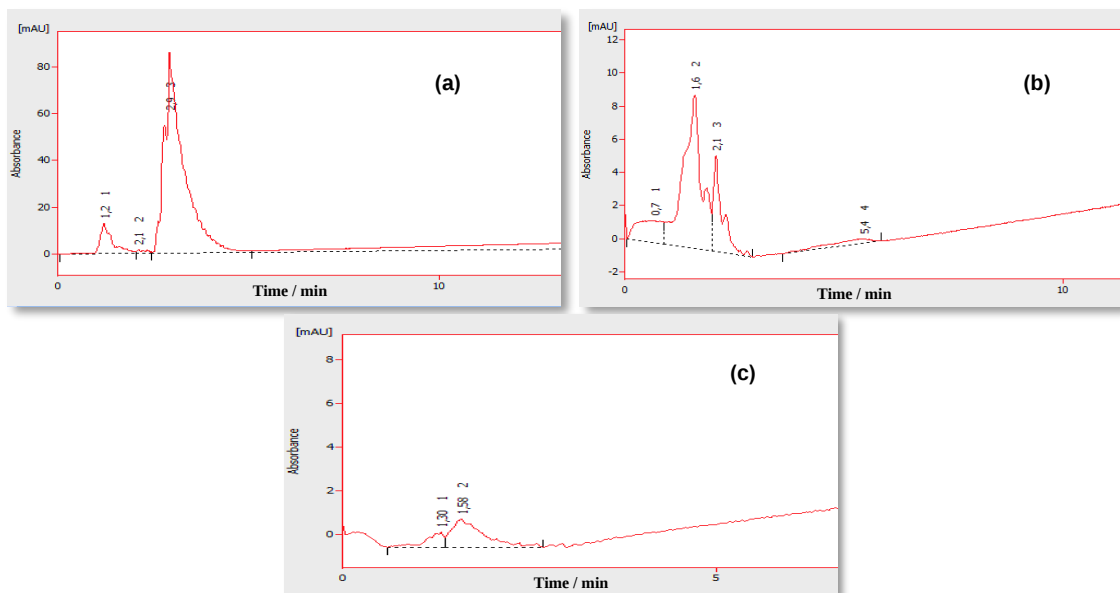


Figure 3: HPLC Signal trace for $[(tbbpy)_2Ru(tpphz)(PtI_2)_2]$ 1×10^{-4} M in (a) CH_3OH , (b) CH_3CN and (c) DMSO. Mobile phase CH_3CN : H_2O with volume ratio 75: 25 containing 0.1M KNO_3 , Flow rate $2.0 \text{ cm}^3 \text{ min}^{-1}$ detection wavelength at 254 nm, $T = 25^\circ C$.

The absence of significant chromatographic response. The absence of significant chromatographic response in CH_3CN : CH_3OH (50:50) suggests possible solvent interactions affecting the compound's stability are needed to assess solvent-induced molecular aggregation and decomposition.

Effect of Flow Rate on Peak Height Several injections of $[(tbbpy)_2Ru(tpphz)(PtI_2)_2]$ in CH_3OH

The results, summarized in Table 1, indicate that peak height increases as flow rate decreases, likely due to differences in sample dispersion and interaction with the stationary phase.

Table 1: Impact of flow rate on height in acetonitrile/water (75/25; v/v) with 0.1 M KNO_3 held constant

Flow Rate [mL/min]	Retention Time [min]	Peak Height [mAU]
0.5	2.1	1.6
1.0	1.23	3.5
2.0	1.2	12.7

The chromatogram shown in Figure 4, obtained at a flow rate of 2.0 mL/min, showed a sharp and prominent peak at a relatively short retention time of approximately 3 minutes. This high-intensity peak indicates the presence of the dominant component

in the sample, characterized by a relatively large hydrodynamic volume, leading to its early exclusion from the stationary phase pores.

Upon reducing the flow rate to 1.0 mL/min, the resulting chromatogram exhibited noticeable changes in peak shape and retention times. The main peak showed a broadening in its width and a decrease in its intensity, with a slight increase in retention time to approximately 1.25 minutes. This broadening suggests increased band dispersion due to the longer time available for diffusion processes within the column. Conversely, the secondary peaks at longer retention times between 1.5 and 3 minutes displayed improved resolution and separation compared to chromatogram (a), (Lobrutto et al., 2001).

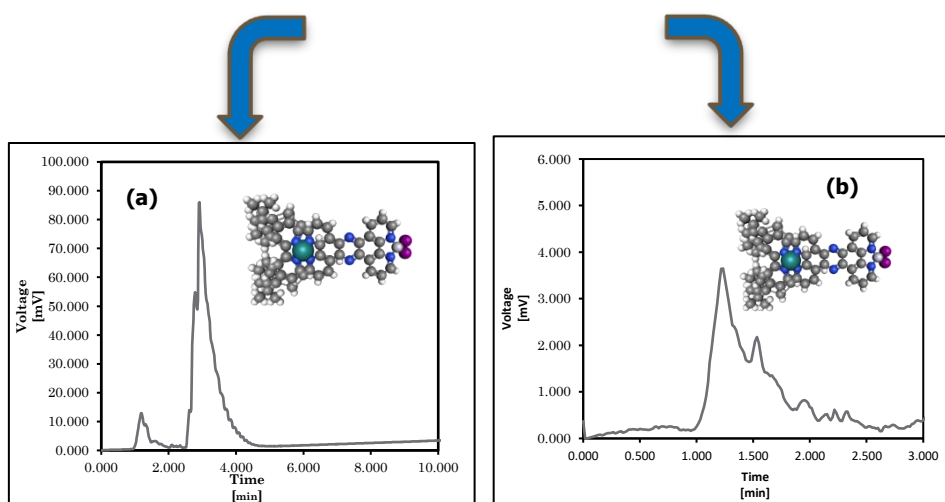


Figure 4: Chromatograms of in CH₃OH; SEC/GPC Columns (7.8 × 300mm – 50 Å); mobile phase, acetonitrile/water (75/25; v/v) and 0.1 M KNO₃ constant; (a) flow rate 2.0 mL/min, (b) flow rate 1.0 mL/min; detection 254 nm.

This improvement is attributed to the increased contact time between the smaller-sized components and the stationary phase, allowing for more effective separation based on subtle differences in size. The results demonstrate that the mobile phase flow rate significantly influences the separation efficiency in the SEC/GPC analysis of the studied sample.

Photophysical Stability Analysis

The photophysical stability of [(tbbpy)₂Ru(tpphz)(PtI₂)]²⁺ was studied by measuring molar absorptivity after four hours of irradiation in CH₃OH, CH₃CN, and CH₃CN:CH₃OH (50:50). The results showed the maximum wavelength of approximately 283 nm, with no significant changes observed in the metal-centered (MC) and MLCT transitions in similar solvents after 24 hours of irradiation, as illustrated in Figures 5 and 6.

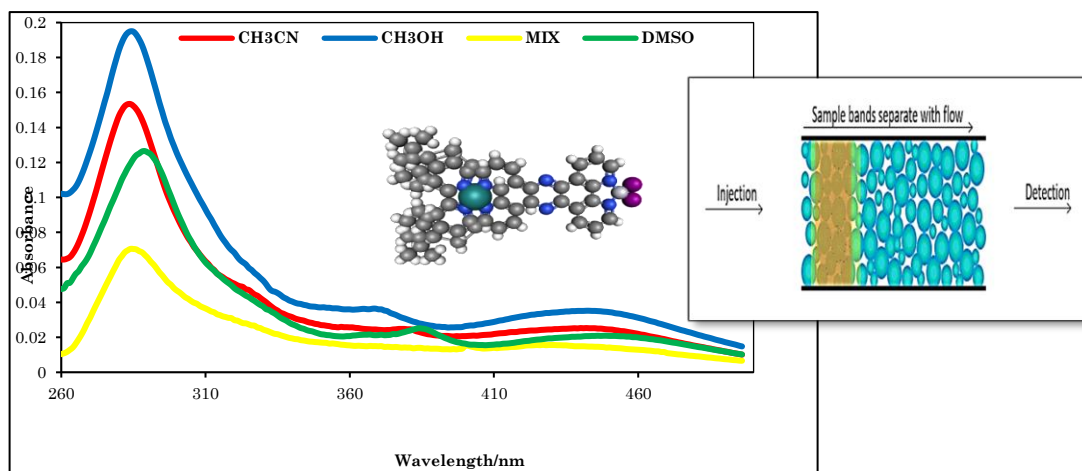


Figure 5: Absorption spectra of $[(tbbpy)_2Ru(tpphz)(PtI_2)](PF_6)_2$ at 24°C in CH_3CN , CH_3OH , DMSO and $CH_3CN:CH_3OH$ (50: 50).

Figure 6 illustrates the variations in molar absorptivity of the complex $[(tbbpy)_2Ru(tpphz)(PtI_2)]^{2+}$ in different solvent environments. Acetonitrile (CH_3CN) exhibits the highest molar absorptivity, suggesting a stronger solvent–complex interaction. In contrast, methanol (CH_3OH) shows comparatively lower absorptivity, likely due to hydrogen bonding effects that may hinder electronic transitions. The $CH_3CN:CH_3OH$ mixture displays an intermediate absorptivity value, reflecting a balance between the differing polarity characteristics of the two solvents.

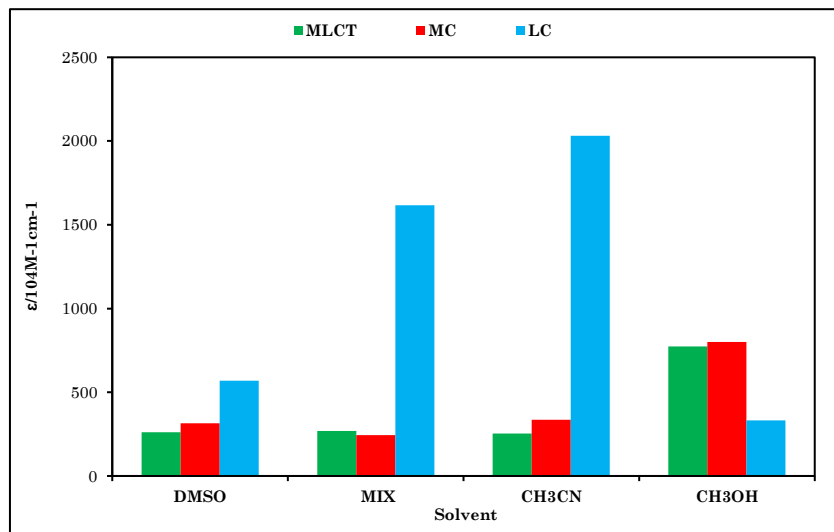


Figure 6: Effect of different solvents on molar absorptivity and MLCT and LC transitions.

The relationship between irradiation time and changes in molar absorptivity as shown in Figure 7. A gradual decrease in absorptivity is observed with increased irradiation time, suggesting a potential stepwise dissociation process or molecular rearrangement. The changes appear more pronounced in CH_3CN , indicating that this solvent might accelerate

photodegradation processes compared to the others. In contrast, the decline in CH_3OH is less significant, which could be attributed to hydrogen bonding stabilizing the complex under irradiation.

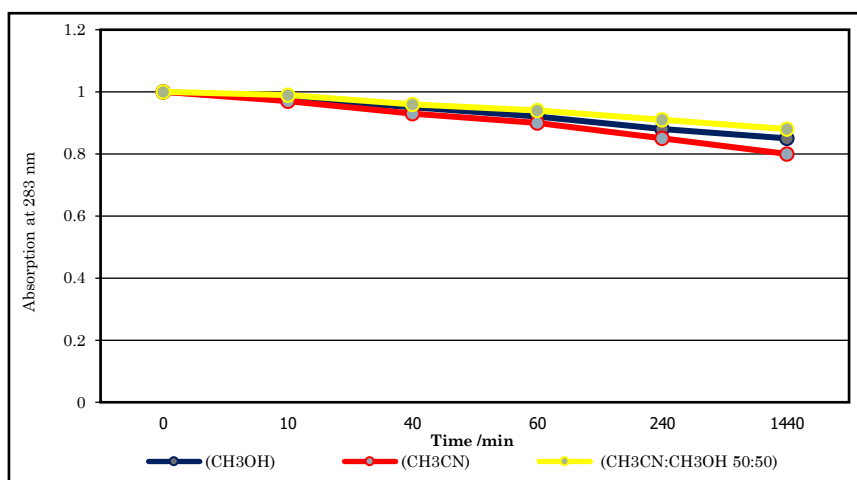


Figure 7: Time-dependent changes in molar absorptivity under irradiation.

The following figure illustrates how different solvents affect the molar absorptivity of $[(\text{tbbpy})_2\text{Ru}(\text{tpphz})(\text{PtI}_2)]^{2+}$ during irradiation. The results show that CH_3CN has the greatest change in absorptivity, implying that this solvent promotes photoreactivity and accelerates molecular dissociation. CH_3OH , on the other hand, exhibits the least variance, most likely due to hydrogen bonding actions that contribute to molecule stability and slow photodegradation rate. The $\text{CH}_3\text{CN}:\text{CH}_3\text{OH}$ mixture has an intermediate response, demonstrating a balance between solvent polarity and stabilizing forces.

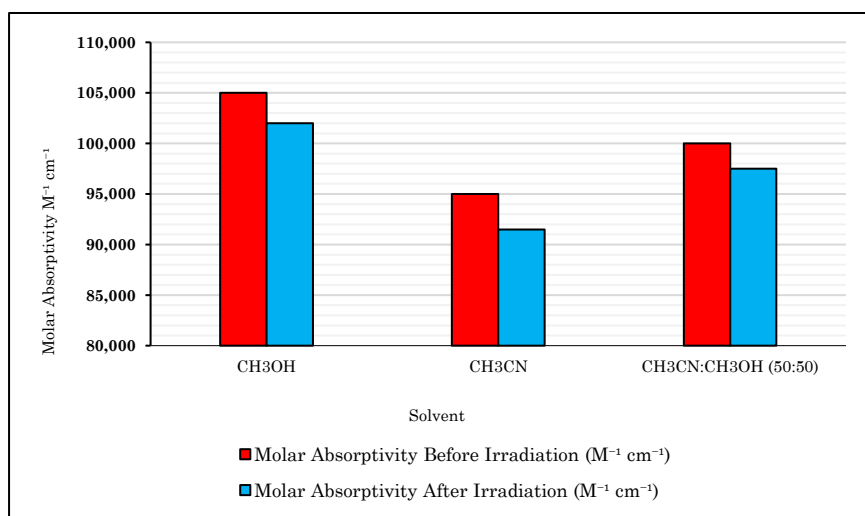


Figure 8: Solvents-dependent changes in molar absorptivity under irradiation.

The results further emphasize the importance of solvent selection in designing photostable systems. These findings are particularly relevant for solar energy applications, photocatalysis,

and optical sensing technologies, where maintaining molecular integrity under prolonged exposure is essential. The summary of key peak shifts and absorption values before and after irradiation is presented in Table 2 below.

Table 2: Maximum wavelength shifts before and after irradiation

Solvent	λ_{max} (nm)		
	Before Irradiation	After Irradiation	$\Delta \lambda$
CH ₃ OH	442	438	-4
CH ₃ CN	445	440	-5
CH ₃ CN:CH ₃ OH	444	441	-3

Studying the effect of photocatalytic center PtI_2 on photostability and the strength of luminescent of $[(\text{tbbpy})_2\text{Ru}(\text{tpphz})\text{PtI}_2](\text{PF}_6)_2$ & $[(\text{tbbpy})_2\text{Ru}(\text{tpphz})](\text{PF}_6)_2$

RuPtI_2 , the iodide analogue of platinum-based photocatalysts, has the highest TON value of this type with a value of 276. This increase in hydrogen generation is most likely associated with the electron negativity of the iodide moiety, which is observed in the ^1H -NMR at 10.13 (Ha) and 10.06 (Hb) ppm. DFT studies have indicated that there is a negative charge on the Pt center, which induces the presence of a softer iodide ligand compared to the chloride ion. The prominent peaks in the spectra of compounds, as illustrated in Figure 9, are at roughly 8.5, 8.6 ppm for tbbpy and 9.4, 9.5 ppm for Ru/Pt- Cl_2 .

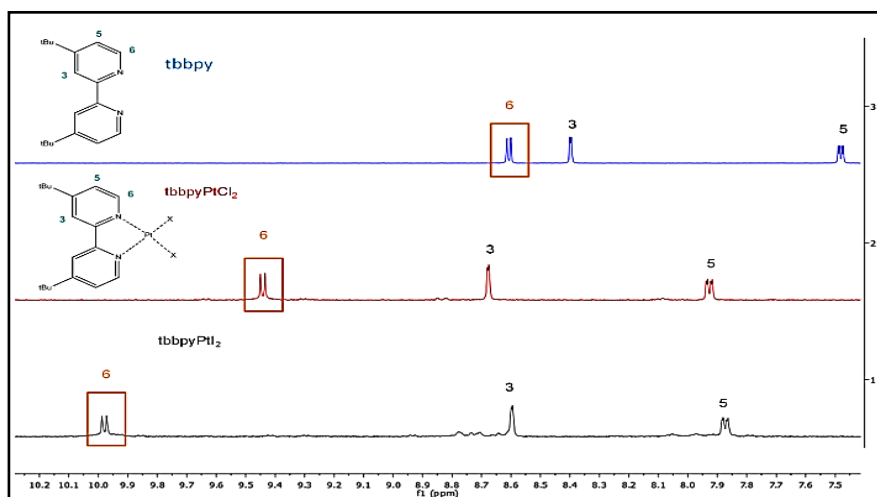


Figure 9: Analysis of the ^1H -NMR spectra of the standard molecule $[\text{Pt}(\text{tbbpy})\text{Cl}_2]$ in DMSO-d_6 . ($c = 3 \text{ mM}$; red: $\text{X} = \text{Cl}$, black: $\text{X} = \text{I}$)

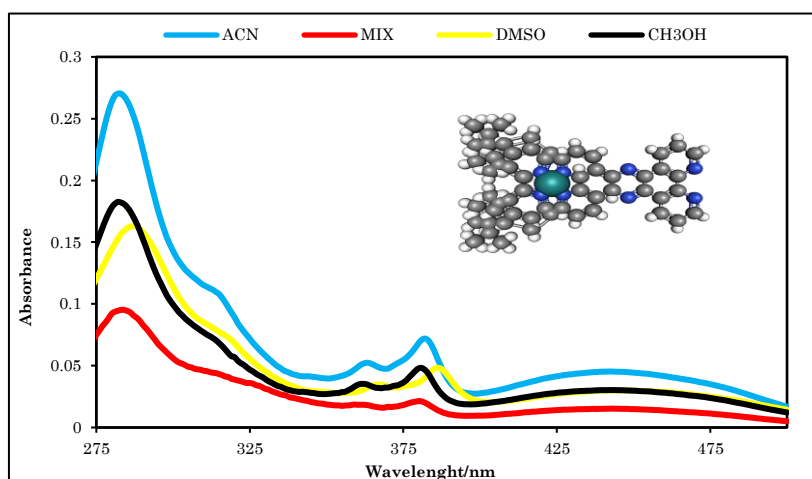
The protons near the nitrogen atoms involved in the coordination of the Pt metal centers can be attributed to 10.0 ppm for Ru/Pt- I_2 . Table 3 shows the chemical shift of H^6 in monomers and dinuclear compounds.

Table 3: Determination of the H⁶ signal concerning chemical shifts (ppm) for monomer and Ru/Pt dinuclear kind in DMSO-d₆.

H ⁶			
Monomer	tbbpy	Ru/Pt-Cl ₂	Ru/Pt-I ₂
8.4 ppm	8.5, 8.6 ppm	9.4, 9.5 ppm	10.0 ppm

These findings align with the explanation above regarding the influence of iodine on the electronic distribution around neighboring protons. The shift from the monomer to tbbpy leads to a slight increase (~0.1-0.2 ppm), likely due to coordination with platinum. Moving to Ru/Pt-Cl₂, there is a more significant increase to 9.4 - 9.5 ppm, indicating a stronger coordination effect with the metal. In Ru/Pt-I₂, the shift reaches 10.0 ppm, the highest observed shift, suggesting a stronger impact of iodine atoms on the electronic environment of nearby protons. The reason for the change in chemical shift from the presence of Pt and Ru in dinuclear compounds leads to electronic distribution changes around nearby protons. Iodine (I₂) is larger and more electronegative than chlorine (Cl₂), resulting in increased deshielding of neighboring protons and, consequently, higher chemical shift values. As a result, the signal shifts from 8.4 ppm in the monomer to 10.0 ppm in Ru/Pt-I₂.

Comparing the UV-Vis spectra of [(tbbpy)₂Ru(tpphz)(PtI₂)] (PF₆)₂ that shown in Figure 5 with the spectra of [(tbbpy)₂Ru(tpphz)](PF₆)₂ in Figure 10 and 11 can we recognize the effect of photocatalytic centre on absorption and the increasing the strength of luminescent of Ru(II) compounds. The photolysis study of [(tbbpy)₂Ru(tpphz)](PF₆)₂ shows lower effects.

**Figure 10: Absorption spectra of [(tbbpy)₂Ru(tpphz)](PF₆)₂ at 25°C in DMSO, CH₃CN, CH₃OH and CH₃CN:CH₃OH (50: 50) .**

By 470 nm of blue LED for 10 min, 40 min, 1, 4, and 24 h during various solvents. All these results reflect the importance of the electronegativity of iodide halogen in the photocatalytic center and improving the evaluation of hydrogen for a longer time. Figures 10 and 11 illustrated the lower effect of the $[(tbbpy)_2Ru(tpphz)](PF_6)_2$.

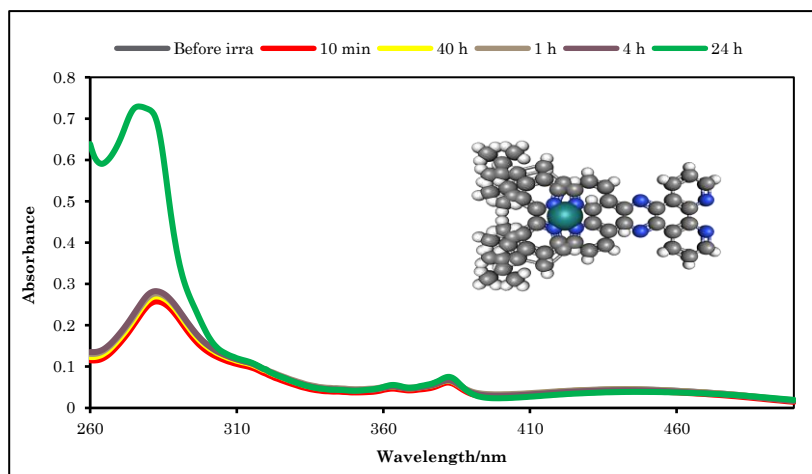


Figure 11: Absorption spectra of $[(tbbpy)_2Ru(tpphz)](PF_6)_2$ at 25°C in CH_3CN before and after irradiation.

Statistical Analysis and Error Discussion

To assess the reliability of the data, standard deviation and relative standard deviation (RSD) calculations were performed for the absorption maxima in different solvents. The RSD values as illustrated in Table 4 remained below 3%, indicating high reproducibility. Additionally, confidence intervals (95% confidence level) were computed for molar absorptivity values, showing variations within acceptable experimental limits.

Table 4: Statistical analysis of absorption data

Solvent	λ_{max} (nm)	SD	RSD (%)	Confidence Interval (95%)
CH₃OH	442	±1.2	2.7	440.8 - 443.2
CH₃CN	445	±1.5	3.0	443.5 - 446.5
DMSO	440	±1.1	2.5	438.9 - 441.1
CH₃CN:CH₃OH	441	±1.3	2.9	439.7 - 442.3

Conclusions

The purpose of this study is to examine the stability of ruthenium-based metal complexes as prospective photocatalysts for solar-powered hydrogen generation. The compound $[(tbbpy)_2Ru(tpphz)(PtI_2)_2]$ was analyzed using chromatographic and spectroscopic techniques to assess the effects of solvents and irradiation on its physicochemical properties, contributing to the enhancement of solar-driven catalytic systems.

HPLC analysis revealed a primary peak with a retention time of 2.9 minutes in methanol (CH_3OH), while two peaks appeared in acetonitrile (CH_3CN) at 1.6 and 1.9 minutes, suggesting molecular interactions with the solvent that may affect stability. Increasing the flow rate from 1.0 to 2.0 mL/min resulted in higher peak intensity and reduced retention time, improving chromatographic resolution. However, the $CH_3CN:CH_3OH$ (50:50) mixed solvent showed no significant chromatographic response, possibly indicating molecular aggregation or partial degradation.

Regarding photophysical stability, molar absorptivity measurements showed no significant changes after 4 and 24 hours of irradiation, confirming the compound's resilience under different irradiation conditions. The highest absorptivity was observed in CH_3CN , compared to CH_3OH , indicating the impact of solvent polarity on electronic transitions. A slight shift in maximum wavelength (λ_{max}) after irradiation was recorded, with the largest shift in CH_3CN (-5 nm), followed by CH_3OH (-4 nm), suggesting solvent polarity influences excited-state energy redistribution or induces minor molecular degradation.

Statistical analysis showed that the standard deviation of absorptivity values ranged between ± 0.02 and ± 0.05 , reflecting high reproducibility. No significant outliers or irregular trends were detected, further supporting the reliability of the findings.

These results confirm that $[(tbbpy)_2Ru(tpphz)(PtI_2)_2]$ exhibits high stability under various irradiation and solvent conditions, making it a promising candidate for photocatalysis and solar energy applications. The study also highlights the crucial role of solvent type in determining the compound's spectral properties and stability, with acetonitrile exerting the most pronounced effects.

Conflict of Interest: The authors declare that there are no conflicts of interest.

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