Received: 23 January 2024 | Revised: 1 April 2024 | Accepted: 27 May 2024 | Published online: 10 June 2024

RESEARCH ARTICLE

Journal of Optics and Photonics Research
2024, Vol. 1(4) 202–209
DOI: 10.47852/bonviewJOPR42022509

Exploring the Feasibility of $Eu(TTA)_3TPPO$ as Pigment for Fluorescent Paint

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Abstract: We outline the synthesis of rare earth hybrid organic β-diketonate $Eu(TTA)_3TPPO$ complex [Eu: Europium, TTA: Thenoyltrifluoroacetone, TPPO: Triphenylphosphine oxide] pigment to formulate luminous paints. Epoxy and urea-formaldehyde resins were synthesized by catalytic homo polymerization and solution polymerization technique, respectively. The fluorescent pigment was synthesized at ambient temperature by solution technique, maintaining the stoichiometric ratio at pH 7. Nextly, fluorescent paint was developed on glass substrate with epoxy (E) and urea-formaldehyde (UF) resins and toluene as solvent with thickness 0.1 mm, respectively. Photoluminescence spectra and photometric assessment of the complex were carried out to probe its photophysical parameters. The excitation spectra of the synthesized complex depict a wide-ranging excitation peak at 466 nm with a wide shoulder at 538 nm, while emission spectra disclosed an intense peak, recorded at 617 nm which portrays color in the reddish-orange region of the visible spectrum. The paint compositions were checked for tack-free, hard dry, adhesion, water, salt, alkali, and acid spray test to study their resistance against distinct surroundings. The painted panels also portrayed reddish-orange emission at a unique wavelength of 617 nm. Amid all the paint compositions, the intensity of the painted panel with UF as a binder was found to be maximum, followed by the painted panel with epoxy as the binder. Photometric evaluation revealed reddish-orange emission from pure pigment as well as painted panels, which are portrayed in the visible spectrum. This proposes a way to develop fluorescent paints that find applications in several areas such as flexible displays, glow-in-dark road paints, OLED devices, automotive rare lighting, fluorescent sensors, aircraft cabin and floor lighting, architectural and interior decorations, and many more.

Keywords: β-diketonate, europium, binder, epoxy, urea-formaldehyde, fluorescent paint

1. Introduction

Luminous paints are defined as the paints that reveal the property of luminescence. Luminescence could be either in the form of fluorescence or phosphorescence; accordingly, they are classified as fluorescent and phosphorescent paints, respectively [1]. Fluorescent paints glow when they are exposed to short-wave UV radiation, and the color of emission depends on the energy gap of the pigment used [2]. On the other hand, there are certain paints which emit light for an extended period of time even if the external light source is ceased, maybe for a few seconds, minutes, or even hours, known as phosphorescent paints [3]. Here we propose a brief study on eco-friendly fluorescent paints that sight subtle beneath normal light and flash beneath UV light. Prior state of art reports diverse research activities involving the synthesis of novel Eu^{3+} complexes for light-emitting devices (LEDs) due to their unique features like sharp emission peak in 612–617 nm range [4]. Particularly, among all the rare earth ions, β-diketones (1,3-diketones) are the most favored and thoroughly studied rare earth coordination complexes [5–7]. Multifractionated applications of the proposed fluorescent phosphor include flexible displays, glow-in-dark road paints, OLED devices, automotive rare lighting, fluorescent sensors, aircraft cabin and floor lighting, architectural and interior decorations, and many more. Hence, we report europium-activated β-diketonate complex [8] and formulation of luminous paints with toluene as solvent.

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2. Literature Review

Different studies related to the synthesis and characterization of Eu^{3+}, TTA, and TPPO complex have been carried out. One such complex, $Eu(TTA)_3(TPPO)_2$, exhibited ligand-stipulated red emission with good thermal stability [9]. Energy transfer efficiency from ligands (TTA and TPPO) to the central metal ion (Eu^{3+}) decides the luminescent properties of the rare earth-based emitting materials [10]. Eu^{3+} doped $CaSrSb_2O_7$ phosphor was successfully synthesized by high-temperature solid-state method [11]. It portrayed color-tunable properties when the dopant concentration and temperature were altered. A similar complex, $Sm_{0.5}Eu_{0.5}(TTA)_3$ dpphen, disclosed that the rare earth complex was thermally stable at 273.76°C (melting point = 507.66°C) with tunable emission from 614 nm to 443 nm, when excited at 385 nm [12]. The spectroscopic properties of $Eu(TTA)_3$ bipy revealed that complex possessed maximum optical density at neutral pH (pH = 7) [13]. In case of lowering or increasing the pH (either 6 or 8), the intensity and optical density of the complex were adversely affected. This study helped in exploring the pH-sensitive nature of rare earth complexes. A β-diketonate europium complex that exhibits excellent photophysical properties for application of white LEDs was reported in similar lines [14].

3. Reagents and Solvents

Rajhans et al. [15] reveals that resins like epoxy ($C_{21}H_{25}ClO_5$) and urea-formaldehyde (UF) ($C_5H_{12}N_8O_2$) have indeed good potential as binders and toluene as a solvent to formulate fluorescent paint and hence chosen in the present work.

3.1. Synthesis of pigment

The pigment, $Eu(TTA)_3TPPO$, was synthesized through solution technique at ambient temperature according to the classical method [16], by following the steps illustrated in Figure 1, and the light emission of complex beneath (a) normal daylight and (b) ultraviolet radiation is shown in Figure 2.

3.2. Chemical structure

In the current study, europium – the central metal atom with a +3 oxidation state forms 8 coordinate bonds (6 with TTA and 2 with TPPO). TTA and TPPO. Subsequently, 3 molecules of TTA and 1 molecule of TPPO are complexed with Eu^{3+} ion to yield $Eu(TTA)_3TPPO$ hybrid organic complex as shown in Figure 3.

Though $Eu(TTA)_3$ is a red light-emitting phosphor, it is non-volatile in nature. The reason is its high polarity due to an unsaturated coordination number. One way to decrease the polarity is by introducing the second ligand, and hence, TPPO was chosen as the ancillary ligand [17–19] in this study. Nevertheless, the foregoing research of this composition discloses that its use as a red light-emitting phosphor in LEDs/ OLEDs is delineated and no work is claimed on the formation of luminous paints with $Eu(TTA)_3TPPO$ as a pigment and hence this study.

3.3. Preparation of substrate

For further analysis, the fluorescent paints were coated over glass slides (substrate for paints). To ensure the painted panels were free from contamination, the slides were cleaned 3–4 times thoroughly with acetone.

3.4. Different compositions of paints

Practically, paint is composed of mainly three constituents, specifically pigment, binder/ resin, and solvent in suitable stoichiometry. Accordingly, two compositions of paint were formulated using epoxy (E) and UF resins as binder and toluene as solvent. When pure binder (epoxy/UF) was coated onto the glass slide and exposed to UV radiation, interestingly, both the resins were found to be inert to UV radiation. On the other hand, substrate with pigment-binder combination emitted reddish-orange color in the visible region of EM spectrum as depicted in Figure 4. This proves that binder has no absorption for UV radiation and the emission under UV is solely due to the pigment.

In the current study, the pigment to binder ratio was taken in the order of 1:9 by wt% with suitable amount of solvent added dropwise to adjust the consistency of paint formulation. The viscosity of solvent was 0.53 mPa.s at room temperature (30°C). When binder was added to the pigment, it has been observed that the pigment easily dispersed uniformly within the binder; however, such viscous gel cannot be employed for paint application directly, and hence to bring down the viscosity to a value which is suitable for coating purpose, solvent was added appropriately. The formulated paint was casted onto the glass slide manually by ensuring uniform thickness over the coated area.

Figure 1
Synthesis technique of $Eu(TTA)_3TPPO$ pigment

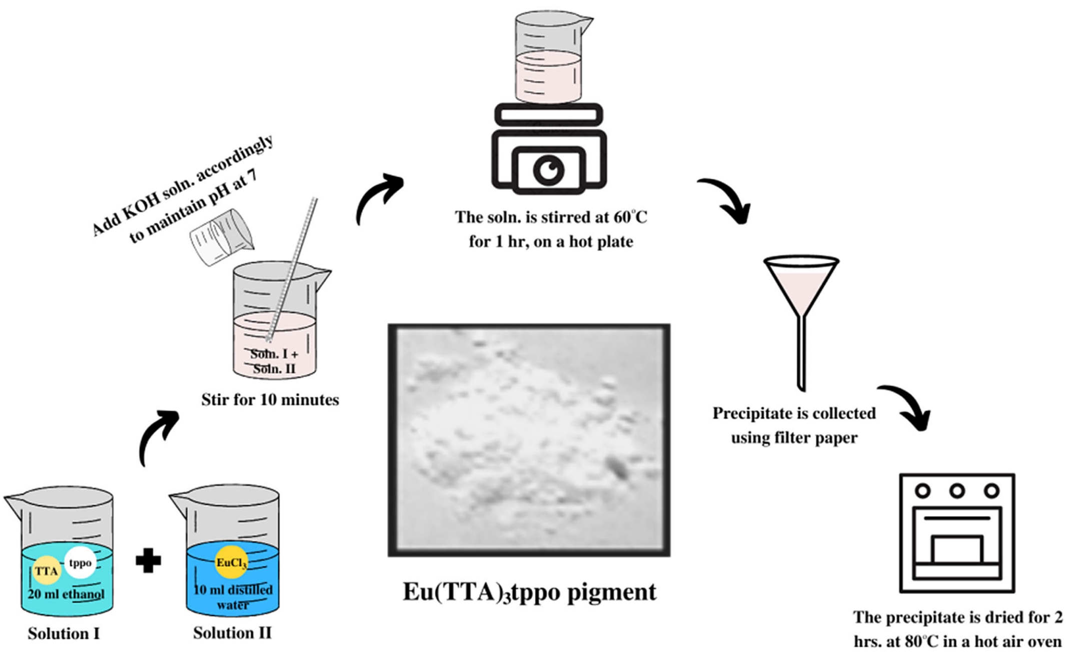


Figure 2
Pigment beneath normal daylight and ultraviolet radiation (280–320 nm)

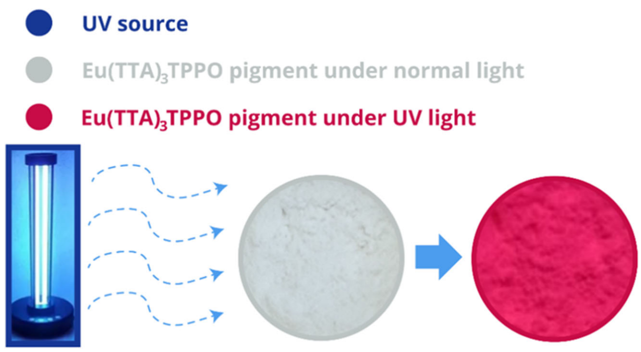


Figure 3
Chemical structure of $Eu(TTA)_3TPPO$ molecule

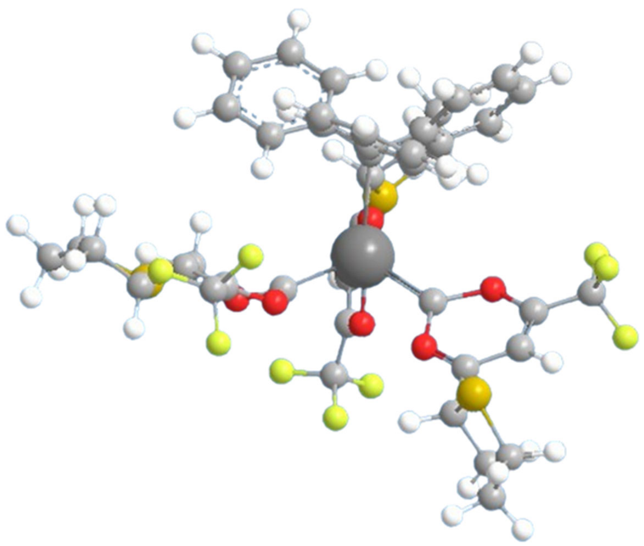
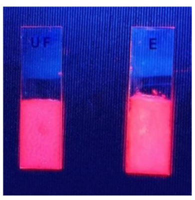


Figure 4
$Eu(TTA)_3TPPO$ painted panels (UF and E) under normal light and ultraviolet light

Painted panels under normal light	Painted panels under UV light
	

Thickness of the epoxy and UF-based painted panels was determined with the help of wedge shape thin film experiment and was found to be 0.112 and 0.110 cm, respectively. Our previous reports reveal that the pigment, when solvated in toluene for 10^{-3}M, peaked at 258 to 259 nm and 356 to 358 nm that can be allocated to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ optical transitions, and the energy band gap was found to be 3.13 eV [20] and hence not reported here.

4. Results and Discussion

PL spectra of $Eu(TTA)_3TPPO$ complex and the paint formulations were interpreted on Hamamatsu F-4500 spectrofluorometer. The program, 1931 Commission Internationale de l'Elcairage (CIE) Radiant Imaging color calculator software, was used to calibrate the chromaticity coordinates (x, y). Further, the paint formulations were assessed for the tack-free, hard dry, adhesion, and water and chemical resistance test.

4.1. Pigment characterization

At first, the synthesized pigment was featured for PL spectra to delve into its excitation and emission wavelengths.

4.1.1. Photoluminescence (PL) spectra of pigment

PL spectroscopy deals with the emission of photons when light of a particular wavelength is incident on light-emitting phosphor (pigment in the case of luminous paint). This is a tool to check the excitation and emission wavelength of the phosphor under investigation. It also helps to probe the suitability of the phosphor for optoelectronic or photocatalytic applications. In the current study, excitation spectra of the pigment under investigation revealed two peaks, registered at 466 and 538 nm with greater separation as depicted in Figure 5(a). Emission spectra of the

Figure 5
(a) Excitation spectra of $Eu(TTA)_3TPPO$ pigment.
(b) Emission spectra of $Eu(TTA)_3TPPO$ pigment

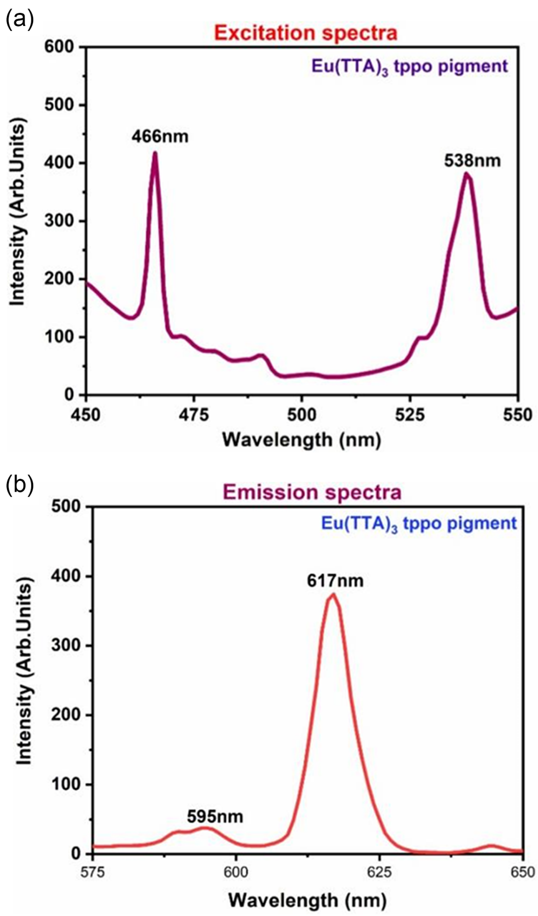
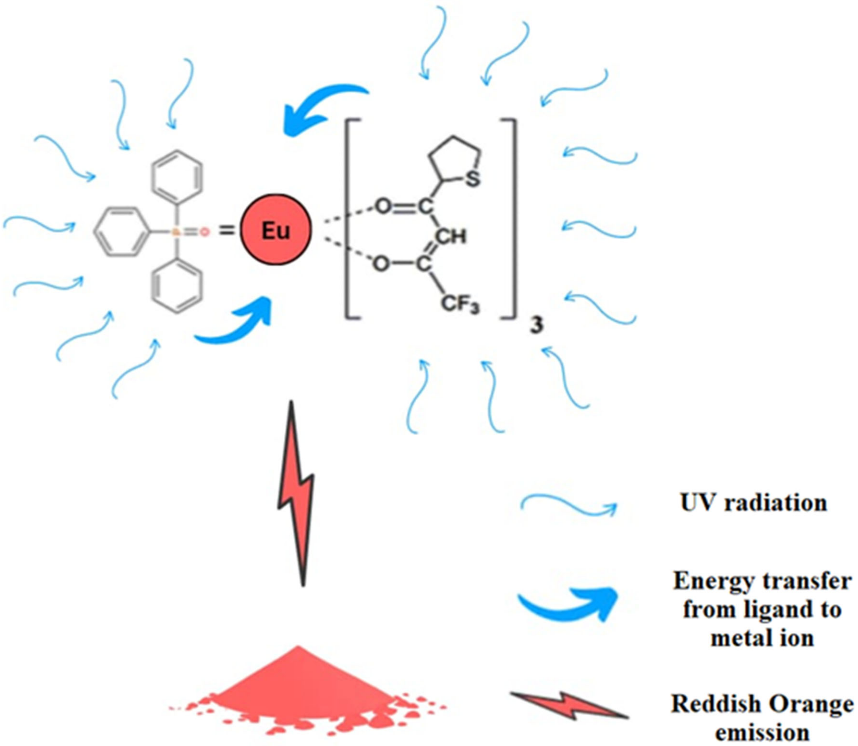


Figure 6
Illustration: Antenna effect in $Eu(TTA)_3TPPO$



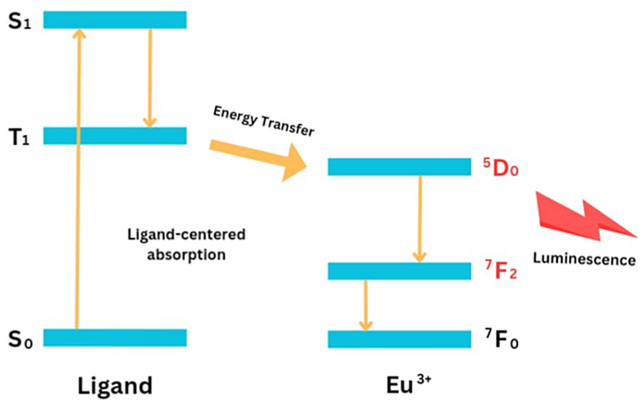
pigment revealed intense emission peak due to their electrons in the 4f shell, one registered at 617 nm, allocated to $^5D_0 \rightarrow {}^7F_2$ transition and a shoulder at 595 nm due to $^5D_0 \rightarrow {}^7F_1$ transition (Figure 5(b)) [21], which portrayed color in the red region of the visible spectrum. The energy gap connecting the triplet state of ligands and the emissive state of Eu^{3+} is responsible for the sensitization of Eu^{3+} ion by chromophore ligands.

The transformation of an excitation energy from ligand TTA to the Eu^{3+} ion in the excited state resulted in the emission from $Eu(TTA)_3TPPO$. Here both the ligands (TTA and TPPO) take part in the energy transfer mechanism. The incident energy is captured by the ligands and then transmitted to the central metal ion, i.e., Eu ion. The synthesized Eu complex possessed a synergistic effect in which the ligand could entrap ultraviolet radiation and transfer energy from Eu (III), causing antenna effect as shown in Figure 6. Compared to the magnetic dipole of $^5D_0 \rightarrow {}^7F_1$ transition, the intensity of the electric dipole of $^5D_0 \rightarrow {}^7F_2$ transition is much stronger; thus, very low symmetric sites are occupied by Eu^{3+} ions in these systems [22]. As a result, the intermediate surroundings around Eu^{3+} ions get highly reactive (due to hyper-sensitive $^5D_0 \rightarrow {}^7F_2$ transition). Figure 7 shows the schematic representation of the mechanism involved in shifting of energy between triplet states of the ligand and the Eu^{3+} ion in the process of PL [23].

4.2. PL spectra of paint formulations

PL spectra of both the paint formulations have been evaluated to study their excitation and emission spectra. The outcomes are indexed in Table 1. This reveals that the complex exhibits its true nature with emission peaking at 617 nm irrespective of the binder employed. However, variation in intensity was observed due to variation in the refractive indices of the pigment and the binders.

Figure 7
Illustration: Energy transfer mechanism in $Eu(TTA)_3TPPO$ from ligand to central and metal ion



4.2.1. PL spectra of epoxy-based $Eu(TTA)_3TPPO$ paint formulation

Luminous paint was formulated with the synthesized phosphor as pigment, epoxy as binder (with 1:9 ratio, respectively), and toluene as solvent. The so-formed paint was coated on a glass slide manually to obtain painted panels of uniform thickness to yield uniform intensity throughout the coated area. When subjected to PL spectroscopy, its excitation spectrum revealed two peaks, registered at 308 and 391 nm as portrayed in Figure 8(a). Emission spectra of the paint formulations revealed an emission peak at 617 nm and a shoulder at 595 nm, attributed to $^5D_0 \rightarrow {}^7F_2$ and $^5D_0 \rightarrow {}^7F_1$

Table 1
An extensive comparison between photophysical properties of pure pigment and paint formulations is under investigation

Sr. No.	Photophysical properties	$Eu(TTA)_3TPPO$ pigment	$Eu(TTA)_3TPPO$ (E)	$Eu(TTA)_3TPPO$ (UF)
1.	λ_{exc} (nm)	538	391	466
2.		466	308	392
3.	Intensity (a.u)	381.652	57.449	1.433
4.		417.404	40.559	75.414
5.	λ_{emi} (nm)	617	617	617
6.		595	595	595
7.	Intensity (a.u)	374.095	52.194	70.542
8.		37.544	−0.556	2.398

* λ_{exc} = Excitation wavelength
λ_{emi} = Emission wavelength

Figure 8
(a) Excitation spectra of epoxy-based paint panel.
(b) Emission spectra of epoxy-based paint panel

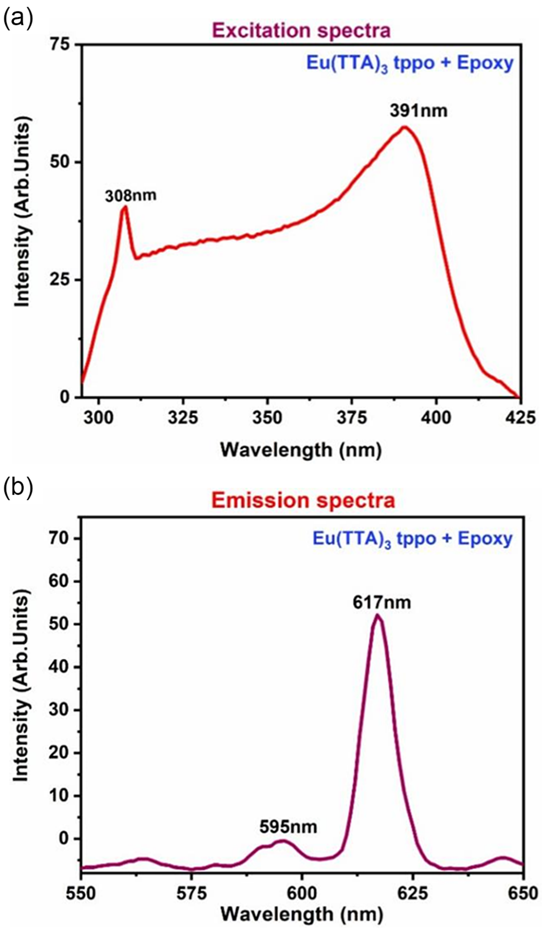
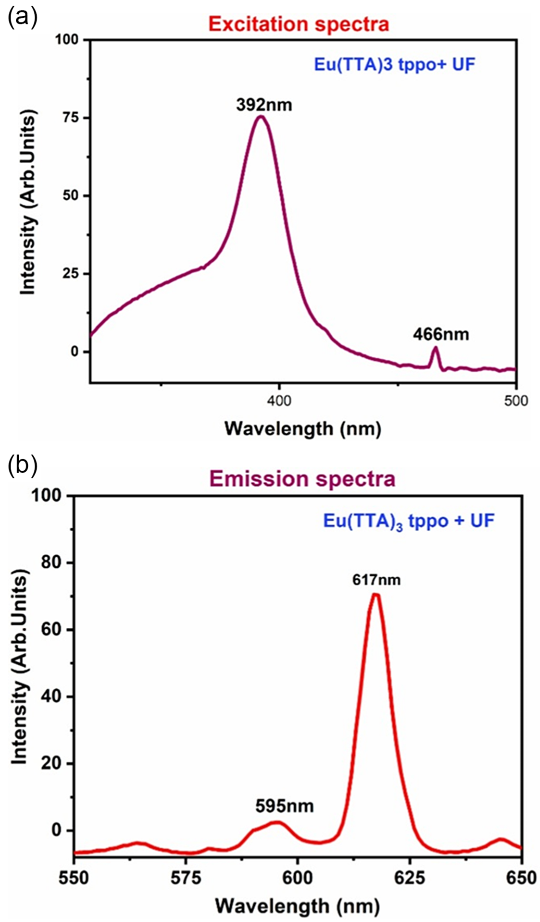


Figure 9
(a) Excitation spectra of UF-based paint panel.
(b) Excitation spectra of UF-based paint panel



transition, respectively, when excited at 391 nm (Figure 8(b)) [24, 25]. A noteworthy point here is its unique lasing wavelengths as that of the pure pigment. However, the decrease in the emission intensity is loud and vivid.

Epoxy possesses an aromatic group [26] and is prone to photochemical reactions and hence degradation in its intensity. However, the panels even then emit intense light that is reasonably sufficient for paint applications.

4.2.2. PL spectra of UF-based $Eu(TTA)_3TPPO$ paint formulation

Luminous paint formulated using $Eu(TTA)_3TPPO$ as pigment, UF as binder (with 1:9 ratio, respectively), and toluene as solvent was manually coated on a glass slide; it revealed excitation spectrum at 392 and 466 nm as portrayed in Figure 9(a). Emission spectra of the painted panel again revealed an emission peak at 617 nm and a shoulder at 595 nm when excited at 392 nm (Figure 9(b)). Interestingly, the intensity of the painted panel was found to be

Table 2
Photometric parameters of Eu(TTA)$_3$TPPO pure pigment and painted panels

Sr. No.	Photometric parameters	Eu(TTA)$_3$TPPO pigment	Eu(TTA)$_3$TPPO E	Eu(TTA)$_3$TPPO UF
1.	λ_{dom} (nm)	610	586	616
2.	X	0.9072	0.9072	0.9072
3.	Y	0.4170	0.4170	0.4170
4.	Z	0.0002	0.0002	0.0002
5.	x	0.6646	0.5503	0.6820
6.	y	0.3352	0.4486	0.3178
7.	u′	0.4669	0.3023	0.5006
8.	v′	0.5299	0.5544	0.5248
9.	u	0.4669	0.3023	0.5006
10.	v	0.3533	0.3696	0.3499
11.	ΔE	0.1766	0.1848	0.1749
12.	CCT (K)	3268	2052	4496

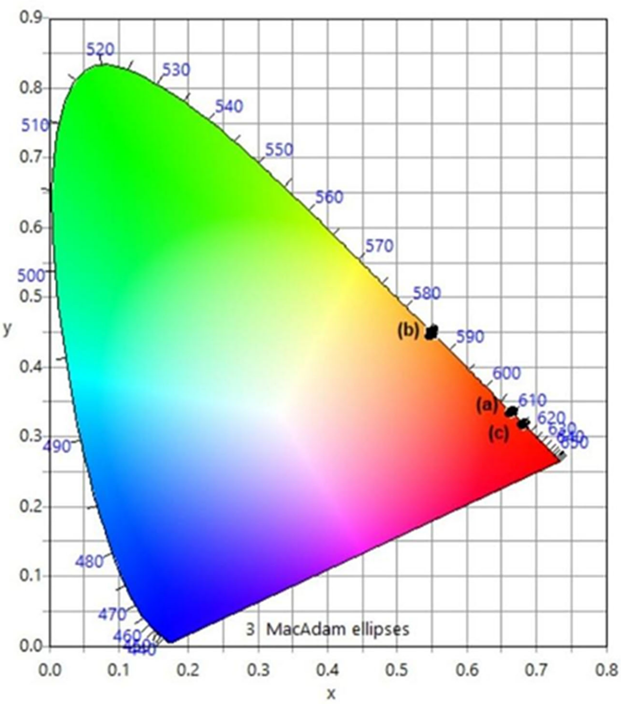
* λ_{dom} = Dominant wavelength

more than that of the epoxy-based painted panel, but less than the intensity of pure pigment. This may be due to its poor absorption ability relative to the absorption of epoxy resin.

4.3. Photometric evaluation of the pigment and the paint panels

Photophysical properties such as Color Correlated Temperature (CCT), CIE chromaticity coordinates, tristimulus values, and dominant wavelength of the Eu complex and the paint formulations were investigated by CIE software. Normally, the color stimulus for any visible color is denoted with regard to chromaticity coordinates as (x, y) in 2D. In the case of three-dimensional, the same are specified with regard to tristimulus values (X, Y, Z). The chromaticity defines the shade and impregnation, and not the color lightness. Nevertheless, a color's lightness (dark or light) and shade (red, blue, green etc.,) are defined by Tristimulus values X, Y, and Z. In a chromaticity coordinate system, the distance of a color point from the black body curve is denoted by D_{uv} (difference in u v values). CCT is termed as the perfect temperature at which a black body radiator should be handled to obtain chromaticity same as that of the light source. These properties were investigated through classical methods and online software [27] and are indexed in Table 2, and the CIE diagram is portrayed in Figure 10. Point (a) depicts chromaticity coordinates of Eu(TTA)$_3$TPPO pure pigment, point (b) depicts chromaticity coordinates of epoxy-based painted panel, and point (c) depicts chromaticity coordinates of UF-based painted panel, respectively.

Figure 10
CIE diagram of Eu(TTA)$_3$TPPO pure pigment and painted panels



4.4. Testing of paint panels

The paint formulations were subjected to the following tests to check different properties that define the longevity and congruity of the paints, and the outcomes are tabulated in respect of pass or fail in Table 3.

4.4.1. Drying test

A paint film transforms into a dry film (from a wet state) due to the volatility of solvent and reaction between its various components. During this transformation, various changes are observed in its chemical structure. To analyze the changes in the configurational properties of paints, the drying test is performed in two stages [28]:

1) Tack-free test: A 0.5 g weight is placed (for 5 s) over a piece of paper (with the paper placed above the painted panel). Further,

Table 3
Outcomes of testing of paint samples

Physical appearance and tests	Eu(TTA)$_3$TPPO (E)	Eu(TTA)$_3$TPPO (UF)
Finish	Smooth	Smooth
Color	Colorless	Colorless
Tack-free test	Pass	Pass
Hard dry test	Pass	Pass
Adhesion test	Fail	Pass
Water resistance test	Pass	Pass
Salt spray test	Pass	Pass
Alkali spray test	Pass	Pass
Acid spray test	Pass	Pass

the weight is taken off, and the panel is turned down. Falling of paper proves the coating to be tack-free and is considered to be passed in this test.

2) Hard dry test: Utmost force is applied over the painted panel by gripping it in between the forefinger and the thumb. The coating is rubbed with cloth in case of visibility of fingerprint, proving the paint as hard dry.

4.4.2. Adhesion test

Adherence (sticking nature) of the coating with the substrate is checked during investigation. This is done by applying an adhesion tape over the painted panel and pulled off swiftly with steady speed. Failure of paint (in this test) is proven if traces of paint are found sticking to the tape (after removing it).

4.4.3. Water resistance test

In a beaker filled with water, the suitably dried painted panels are thoroughly immersed for 24 h. Later, the panels are taken out from the beaker and cleansed under gushing tap water. Occurrence of blisters, loss in adherence, luster, etc., proves the coatings to have poor resistance toward water.

4.4.4. Chemical resistance test

Salt, alkali, and acid spray tests are carried out to examine the withstanding property of paint compositions against different commercial chemicals.

1) Salt spray test: In a beaker filled with 5% NaCl solution, dried painted panels are immersed for 24 h. Occurrence of blisters, loss in adherence or luster, etc., proves the coatings to have poor resistance toward salts.
2) Alkali spray test: In a beaker filled with 5% NaOH solution, dried painted panels are immersed for 15 min and cleansed with water. Occurrence of blisters, loss in adherence or luster, etc., proves the coatings to have poor resistance toward alkalis.
3) Acid spray test: In a beaker filled with 5% HCOOH solution, dried painted panels are immersed for 15 min and cleansed with water. Occurrence of blisters, loss in adherence or luster, etc., proves the coatings to have poor resistance toward acids.

To determine the durability and compatibility of the paints, drying test (hard dry and tack-free), adhesion test, water resistance test, and chemical (salt, alkali, and acid) spray test were probed under adverse surrounding conditions. The outcomes of these tests are tabulated in Table 3. These outcomes unveiled that the UF-based paint formulation passed in all the tests and the epoxy-based paint formulation too passed in all the tests except the adhesion test. The reason for this evaluated that the epoxy-based $Eu(TTA)_3TPPO$ paint was highly sticky in nature which resulted in paint marks over the adhesion tape.

5. Conclusion

We outline the synthesis of $Eu(TTA)_3TPPO$ pigment with aim to formulate fluorescent paints on glass substrates. Epoxy resin and UF resin were chosen as binders, and toluene as solvent. PL spectra were explored to examine the photophysical and photometric parameters of the pigment ($Eu(TTA)_3TPPO$) and the paint formulations. The excitation spectra of the complex portrayed a wide excitation peak at 466 nm with a wide shoulder at 538 nm, while emission spectra disclosed an intense peak, registered at 617 nm. It portrayed CIE coordinates as (0.6646,0.3352) which portrayed reddish-orange color of the visible spectrum. The painted panels also revealed reddish-orange emission at a distinct wavelength of 617 nm with CIE coordinates as (0.5503,0.4486) for epoxy-based paint and (0.6820,0.3178) for UF-based paint. Further, the painted panels were examined for drying, adhesion, water resistance, and chemical spray test. The outcome of these tests disclosed that the paints were durable and compatible under various environments. Among both the paint compositions, the intensity of UF-based paint was found to be maximum, followed by epoxy-based paint. The photometric evaluation revealed that both paints emitted reddish-orange color in the visible spectrum. These results manifest that the $Eu(TTA)_3TPPO$ complex confirms its fluorescent behavior irrespective of amalgamating with different binders extending a way to standardize the emission wavelength. This offers a way to come up with fluorescent paints that find multifaceted applications in several areas such as in flexible displays, glow-in-dark road paints, OLED devices, automotive rare lighting, fluorescent sensors, aircraft cabin and floor lighting, architectural and interior decorations, and many more.

Ethical Statement

This study does not contain any studies with human or animal subjects performed by any of the authors.

Conflicts of Interest

The authors declare that they have no conflicts of interest to this work.

Data Availability Statement

Data are available on request from the corresponding author upon reasonable request.

Author Contribution Statement

Vyankatesh Rajhans: Formal analysis, Investigation, Data curation, Writing – original draft, Visualization. **N. Thejo Kalyani:** Conceptualization, Methodology, Validation, Resources, Writing – review & editing, Supervision, Project administration. **Akhilesh Ugale:** Software, Writing – review & editing. **S. J. Dhoble:** Supervision, Project administration.

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How to Cite: Rajhans, V., Kalyani, N. T., Ugale, A., & Dhoble, S. J. (2024). Exploring the Feasibility of $Eu(TTA)_3TPPO$ as Pigment for Fluorescent Paint. *Journal of Optics and Photonics Research*, *1*(4), 202–209. https://doi.org/10.47852/bonviewJOPR42022509