

Plasmonic-Upconversion NaYF₄:Eu³⁺,Ga³⁺,Te⁴⁺@Au Nanocomposites for pH/NIR-Responsive Cisplatin Delivery and Combined Chemo-Photothermal Treatment of Anaplastic Thyroid Cancer Cells

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Received: 22 May 2026
Accepted: 09 June 2026
Published online: 19 June 2026

Abstract Multifunctional nanoplatforms that combine imaging, controlled drug delivery, and photothermal conversion may provide complementary treatment functions for aggressive cancers. Here, plasmonic-upconversion NaYF₄:Eu³⁺,Ga³⁺,Te⁴⁺@Au nanocomposites were prepared by in situ deposition of approximately 10 nm Au nanoparticles on approximately 40 nm doped NaYF₄ cores and subsequently conjugated with an SH-PEG-Cis-Pt prodrug. The design integrates an acid-labile hydrazone-containing linker with Te-Pt coordination to provide pH- and NIR-responsive cisplatin release. TEM/HRTEM, XRD, FTIR, XPS, DLS/zeta potential, UV-Vis-NIR spectroscopy, and upconversion luminescence (UCL) measurements were used for physicochemical characterization. The maximum reported drug loading was 51.62%. Cis-Pt release was limited at pH 7.4 (approximately 6% at 72 h) but increased under acidic conditions (approximately 45% at pH 4.5) and under NIR irradiation (approximately 75%). Under 808 nm irradiation (1.5 W cm⁻²), a 400 µg mL⁻¹ dispersion reached approximately 56.7 °C after 10 min. In SW1736 anaplastic thyroid carcinoma cells, the unloaded carrier showed no loss of viability without irradiation over the tested concentration range, whereas the Cis-Pt-loaded formulation under irradiation reduced normalized viability to approximately 3%, indicating a strong combined chemo-photothermal effect. UCL imaging further demonstrated time-dependent cellular association of the nanocomposite. These results establish an in vitro proof of concept; normal-cell safety, pharmacokinetics, and in vivo therapeutic performance remain to be determined.

Keywords: Up-conversion nanomaterial; Plasmonic; Anaplastic Thyroid Cancer; Photothermal therapy; Chemotherapy.

1.Introduction

Anaplastic thyroid carcinoma (ATC) is highly aggressive and constitutes less than 2% of thyroid tumors but causes 14-39% of thyroid cancer deaths (National Cancer Institute) [1]. Patients typically undergo surgery, radiotherapy, and chemotherapy, though these offer limited prognosis improvements. Treatment failure often stems from therapeutic resistance, with unclear, precise molecular mechanisms [2]. Conventional chemotherapy's non-targeted approach leads to low efficacy, significant toxicity, and drug resistance [3]. Using nanocarriers to encapsulate drugs targeted to tumor tissue may reduce the adverse effects of traditional chemotherapy [4, 5].

Nanostructures that release medicines in response to stimuli, particularly pH-responsive prodrugs, have gained attention in

biological research because they exploit the acidic environment of cancer cells [6, 7]. However, their rapid drug release rates and low loading capacities limit effectiveness.

Photothermal therapy (PTT) is a non-invasive, highly selective technique with lower toxicity than chemotherapy [8]. PTT uses nanomaterials to target cancer cells with near-infrared (NIR) light and combines physical and chemical approaches, each with unique pros and cons. Merging chemotherapy with physical therapy may enhance cancer treatment efficacy [9].

Nanostructures like gold nanoparticles (AuNPs), bismuth derivatives, and carbon nanomaterials have been used as PTT agents [10, 11]. Gold nanocrystals significantly advanced nanomaterial studies *in vitro* and *in vivo*. AuNPs have been validated for diagnostics [12-14] and show promise in therapies, especially PTT, due to their photothermal conversion effect [15]. Nanocomposites for synergistic treatment must be monitored with a visual tracking agent. A multiphoton pathway called upconversion allows luminescent lanthanide (Ln³⁺) doped nanoparticles to convert lower-energy NIR photons into higher-energy ones (UV, Vis, and IR). This multiphoton excitation absorbs NIR photons in a ladder pattern. These optical characteristics are utilized in PTT [16, 17]. For example, due to the synergistic effect of chemo-photothermal therapy, Er³⁺/Gd³⁺-doped UCNPs@Au-DOX nanocomposites, which act as upconversion luminescence (UCL) and magnetic resonance imaging agents, yield effective *in vitro* treatment outcomes [18].

In this present study, a facile procedure was developed to synthesize a hybrid nanocomposite based on the in-situ formation of AuNPs on the NaYF₄:Eu³⁺, Ga³⁺, and Te⁴⁺ (NaYF₄:Eu,Ga,Te@Au), which can function as a PTT agent. The blue up-conversion phosphor sodium yttrium fluoride (NaYF₄) was doped with Eu³⁺ (Europium) (as a UCL agent), and to enhance the luminescence intensity of Eu³⁺ in NaYF₄ systems, other trivalent ions (Ga³⁺- Gallium and other non-toxic ions) partially substitute yttrium in the host lattice [19]. According to some studies, Tellurium (Te) is utilized as a third component for three reasons. Natural semiconductor Te possesses a small band gap and is the fourth most common trace element in the human body, making it biocompatible [20-22]. Moreover, it shows significant promise as a novel biomaterial due to its ultra-sensitive

reactive oxygen species responsiveness, which stems from the low electronegativity of Te [23]. Lastly, the coordination chemistry of platinum (Pt) and Te opens up new possibilities for delivering Pt-based medicines such as cisplatin (Cis-Pt) [24].

The NaYF₄:Eu,Ga,Te@Au nanocomposite exhibits excellent photostability and biocompatibility. An innovative chemotherapy prodrug, SH-PEG-Cis-Pt, was synthesized and combined with the NaYF₄:Eu,Ga,Te@Au nanocomposite to enhance the efficacy of chemo-photothermal therapy. In CCK-8 cytotoxicity experiments, the NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites showed low cell viability but favorable synergistic effects during treatment on SW1736 (anaplastic thyroid cancer) cells under 808 nm irradiation. The effective use of NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites in intracellular UCL indicates their potential as valuable tools for multimodal bioimaging-guided therapy.

2. Experimental

2.1. Chemicals and materials

All chemicals were applied upon receiving with no further refinement. EuCl₃·6H₂O (99.99%), GaCl₃·6H₂O (99.9%), TeCl₄ (99.99%), YCl₃·6H₂O (99.99%), and DOX·HCl were provided from Sigma Aldrich Company. Moreover, sodium hydroxide (NaOH, 96%), methanol (MeOH, 99.5%), ammonium fluoride (NH₄F, 98%), cetyltrimethylammonium bromide (CTAB, 99%), N,N-Dimethylformamide (DMF), isopropyl alcohol, oleic acid (OA, 90%), and tert-butyl alcohol (*t*-BuOH) were purchased from Merck Company. Additionally, other prepared materials from the company mentioned above included the 1-octadecene (ODE, 90%), acetone, cyclohexane, and phosphoric acid. Roswell Park Memorial Institute (RPMI) 1640, Fetal Bovine Serum (FBS), and Cis (Pt(NH₃)₂CL₂) were provided from Sigma Aldrich Company Ltd. Eventually, SH-PEG-CONHNH₂ (SH-PEG-HZ, MW 5000, 90%) and WST-8 cell counting kit (CCK-8) were purchased from Shanghai Ponsure Biotech, Inc. and Dojindo Molecular Technologies Inc, respectively.

2.2. Synthesis of NaYF₄:Eu,Ga,Te nanocrystals

A reported method [25] was used to synthesize the upconversion nanostructure. An aqueous solution of YCl₃ (1.56 mmol, 78%), EuCl₃ (0.36 mmol, 18%), GaCl₃ (0.06 mmol, 3%), and TeCl₄ (0.01 mmol, 1%) in deionized water was added to a 100 mL flask with 1-ODE (30 mL) and OA (12 mL). The solution was blended at 25 °C for 1 hour, then heated to 165 °C for a clear solution. MeOH (20 mL), containing NH₄F (8 mmol) and NaOH (1.6 mmol), was added and stirred until MeOH evaporated. The solution was heated to 300 °C under argon for 1 hour, then cooled to 25 °C. The product was washed with acetone and cyclohexane, then diffused in cyclohexane (20 mL) [26, 27].

2.3. Synthesis of NaYF₄:Eu,Ga,Te@AuNPs

1 mL NaYF₄:Eu,Ga,Te and 250 μL HAuCl₄·3H₂O (0.01 M) were added to 10 mL CTAB (0.1M) in a round-bottom flask and mixed vigorously at 25 °C for 10 min. Then, 15 μL of ice-water NaBH₄ (0.01 M) was added, and the mixture was stirred for 2 h until the solution turned blood red. The mixture was centrifuged at 10,000 rpm for 15 min to collect the product and remove excess CTAB. The product was washed with ultra-pure water several times, and NaYF₄:Eu,Ga,Te@AuNPs were suspended in 2 mL ultra-pure water and stored at 4 °C for later use [28].

2.4. Prodrug preparation

The SH-PEG-Cis-Pt prodrug was synthesized with modifications per [1]. In a stirring DMF solution, 0.05 mmol of Pt(NH₃)₂CL₂ and 0.15 mmol of SH-PEG-HZ were added along with 10 μL of H₃PO₄ as a catalyst. The reaction continued for 48 h at 25 °C while stirring. The

mixture was dialyzed until colorless using a 3500 MWCO dialysis membrane into a PBS buffer (pH = 8.0). Finally, the SH-PEG-Cis-Pt powder was vacuum freeze-dried for 48 h at 25 °C.

2.5. Drug loading

To load the prodrug into the nanocomposite, disperse 2 mg of the SH-PEG-Cis-Pt prodrug in 5 mL of ultra-pure water. Then, 2 mL of the NaYF₄:Eu,Ga,Te@Au nanocomposite solution was gradually added to the prodrug solution, followed by stirring for 12 h at 25 °C (200 rpm). The final nanocomposite (NaYF₄:Eu,Ga,Te@Au-Cis-Pt) was obtained by centrifuging the solution (10000 rpm, 15 min) and washing it with ultra-pure water. ¹H NMR (500 MHz, D₂O) δ (ppm): 4.15 (4H, b, NHCOOCH₂), 3.66 (8H, b, OCH₂CH₂ of PEG), 2.62 (4H, b, TeCH₂), and 2.18 (4H, b, TeCH₂CH₂).

2.6. Instrumentation

The Rigaku D/max 2500V diffractometer with monochromator graphite was used for X-ray diffraction (XRD) analysis, detecting patterns with co-filtered CoKα radiation (λ=1.79Å) at 10 mA and 45 kV. Experiments were conducted at 25 °C within the 2θ range of 10–90 °. XRD phases were compared to ASTM powder data. FT-IR spectra were recorded from 400–4000 cm⁻¹ after pressing a few mg of sample with KBr powder using a hydraulic press. A Perkin-Elmer device assessed transmission versus wavenumber (cm⁻¹), followed by optical absorption analysis with a Shimadzu UVmini-1240 UV-Vis Spectrophotometer in quartz cells. Zeta potential and size were measured using a Malvern Zetasizer Nano ZS90 and a Zeiss Transmission Electron Microscope (EM10C, 80 kV). Surface chemical compositions were measured by X-ray photoelectron spectroscopy (XPS) analysis using the Thermo Fisher Scientific K-Alpha system. Perkin-Elmer spectroscopy (200–800 nm range with Lambda-900 Spectrophotometer) computed the UV-Vis. The M Series Laser (808 nm and 980 nm) featured a 5-mm diameter laser module (Dragon Lasers, China) with adaptable power (0–2 W/cm²). In the cell viability test, the optical density (OD) at 450 nm was recorded using a microplate reader (Bio Tek, USA).

2.7. Drug release profiles

To investigate the release profile of Cis-Pt from the NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposite, 1 mg/mL was placed in a dialysis bag (3500 MW) with 4 mL PBS (pH = 4.5, 6, and 7.4) at 37 °C. Every 2, 4, 6, 8, 10, 12, 24, and 48 h, 2 mL of the solution was removed and replaced with fresh PBS. The absorbance at 510 nm (λ_{max}= 510 nm) was measured for the removed solution using a UV-Vis spectrometer, allowing calculation of the released Cis-Pt concentration at each interval. Additionally, Cis-Pt release profiles at different pH values were measured under laser irradiation (λ>980 nm) due to interactions with Te elements.

2.8. Photothermal effects

To evaluate NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites as PPT agents, 500 mL NaYF₄:Eu,Ga,Te@Au-Cis-Pt solution in various concentrations (50, 100, 200, 400 μg/mL) underwent laser (808 nm-NIR, 5 mm module) irradiation. The output power was measured with an optical power meter calibrated to 1.5 W/cm². A thermocouple with 0.1 °C precision was placed perpendicular to the laser path in the dispersion. Temperature was recorded every 20 s, and data was collected from three experiments for reliability.

2.9. Cell viability

The Iranian Biological Resource Center (Tehran, Iran) provided human SW1736 (ATC) cells. A humidified environment with 5% CO₂ sustained SW1736 cells at 37 °C in RPMI with 1% penicillin/streptomycin and 10% FBS. The WST-8 kit (CCK-8,

Dojindo, Japan) evaluated the *in vitro* toxicity of Cis-Pt, NaYF₄:Eu,Ga,Te@Au, and NaYF₄:Eu,Ga,Te@Au-Cis-Pt. The SW1736 cells (5000 per well) were seeded in 96-well plates and cultured overnight in 100 μ L media. Next, the medium was removed, and 200 μ L of fresh medium with NaYF₄:Eu,Ga,Te@Au-Cis-Pt or NaYF₄:Eu,Ga,Te@Au nanocomposites at concentrations (0, 50, 100, 200 μ g/mL) or free Cis-Pt (0, 3.75, 7.5, 15 μ g/mL) was added to cells at 37 °C with 5% CO₂ for 4 h. The identical sample was placed in two widely spaced wells to prevent temperature impacts. One well was illuminated using an 808 nm laser for 24 min (with 8-minute pauses after each 8-minute irradiation), while the other was not. The control group contained only SW1736 cells. Incubation continued for another 24 h in all wells. The medium was removed and 100 μ L of CCK8 solution (10% CCK8) was added to each well, incubating for 1.5 h. A microplate reader at 450 nm tested the viability of SW1736 cells, with three independent tests conducted for each exam.

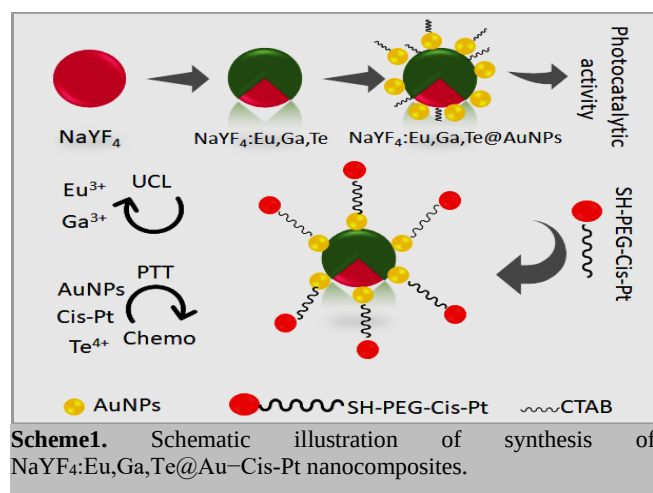
2.10. Imaging of tumor cells *in vitro*

SW1736 cells were seeded at 2×10^5 cells/well in petri plates and grown for 12 h. The medium was then replaced with new media of varying compositions. The SW1736 cells were photographed with an LSM 710 confocal microscope after 2 and 4 h incubation at 37 °C.

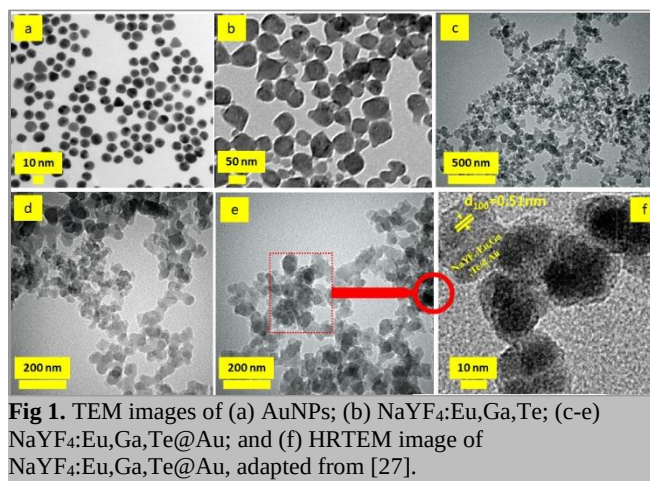
3. Results and discussion

3.1. Synthesis and characterization of NaYF₄:Eu,Ga,Te@Au and NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites

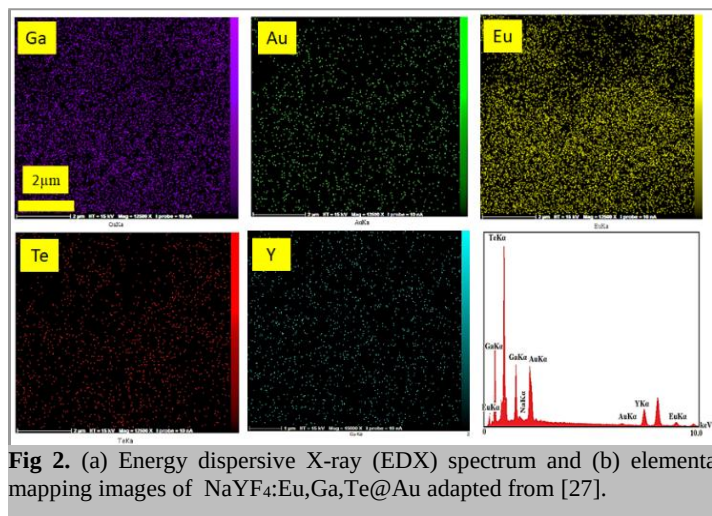
As shown in Scheme 1, composite upconversion nanoparticles (NaYF₄:Eu,Ga,Te) were produced, with an Au shell providing potential PTT and photocatalytic activity. CTAB is a common stabilizing and reducing agent in gold nanocrystal production [29, 30]. It acts as a stabilizer for NaYF₄:Eu,Ga,Te and a reducing agent for HAuCl₄ in the *in-situ* growth of AuNPs on NaYF₄:Eu,Ga,Te, where NaBH₄ is the co-reducer. This demonstrates that PTT agents can be synthesized by directly reducing HAuCl₄ on the NaYF₄:Eu,Ga,Te surface. Additionally, a chemotherapy prodrug, SH-PEG-Cis-Pt, was combined with NaYF₄:Eu,Ga,Te@AuNPs to enhance the efficacy of chemotherapy and PTT. Most AuNPs feature a robust Au-S bond, facilitating easy interaction with -SH groups [26]. Hydrazine bonds were integrated into the SH-PEG-Cis-Pt structure to enable pH-responsive drug release in the acidic microenvironment of cancer cells. Furthermore, the bond between Te and Cis-Pt can increase the drug load on the nanostructure, improving drug release efficiency [31].



The AuNPs and NaYF₄:Eu,Ga,Te (Fig. 1a-b) are spherical, averaging 10 ± 1.1 and 40 ± 2.6 nm, respectively. AuNPs evenly distribute over NaYF₄:Eu,Ga,Te's surface (Fig. 1c). AuNP crystallization does not significantly affect NaYF₄:Eu,Ga,Te morphology (Fig. 1d-e). In HRTEM images of NaYF₄:Eu,Ga,Te@Au, uniformly spherical AuNPs around ~ 10 nm are visible (Fig. 1f), matching synthesized AuNPs. Both AuNPs and NaYF₄:Eu,Ga,Te maintain excellent crystal structures with significant lattice fringes (Fig. 1f). The stacking of these fringes indicates AuNPs formed on NaYF₄:Eu,Ga,Te's surface, not merely as a physical mixture. The energy-dispersive X-ray spectrum and elemental mapping of NaYF₄:Eu,Ga,Te@Au in Fig. 2 confirm the successful synthesis of NaYF₄:Eu,Ga,Te@AuNPs.



Dynamic light scattering analyses (Fig. 3) showed the average diameters of NaYF₄:Eu,Ga,Te, NaYF₄:Eu,Ga,Te@Au, and NaYF₄:Eu,Ga,Te@AuCis-Pt were 51.9, 65.8, and 137.4 nm, respectively. The zeta potentials of NaYF₄:Eu,Ga,Te, NaYF₄:Eu,Ga,Te@Au, SH-PEG-Cis-Pt, and NaYF₄:Eu,Ga,Te@Au-Cis-Pt were -33.9 , $+1.5$, $+6.5$, and $+11.0$ mV, indicating SH-PEG-Cis-Pt effectively associated with NaYF₄:Eu,Ga,Te@Au, forming NaYF₄:Eu,Ga,Te@Au-Cis-Pt (Fig. 4). The NaYF₄:Eu,Ga,Te@AuCis-Pt nanocomposites are better suited for cellular uptake due to their electropositive nature.



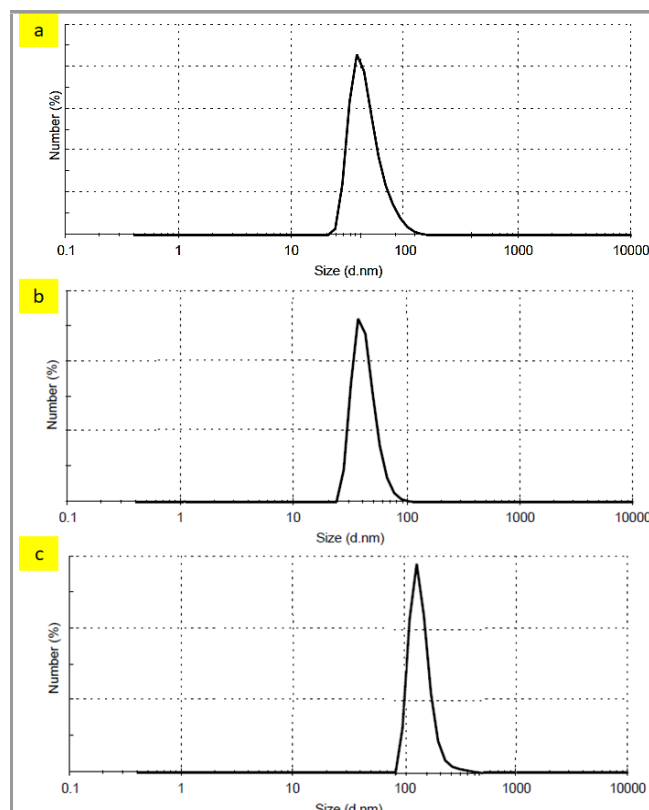


Fig 3. Dynamic light scattering (DLS) of (a) NaYF₄:Eu,Ga,Te; (b) NaYF₄:Eu,Ga,Te@Au and (c) NaYF₄:Eu,Ga,Te@Au-Cis-Pt in water (200 µg/mL).

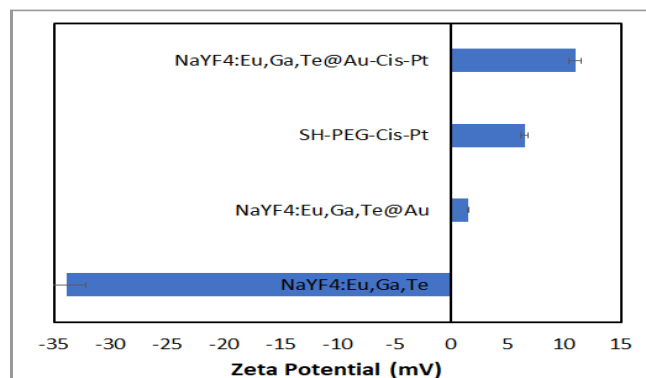


Fig 4. Zeta potentials of NaYF₄:Eu,Ga,Te; NaYF₄:Eu,Ga,Te@Au and NaYF₄:Eu,Ga,Te@Au-Cis-Pt in water (200 µg/mL).

Gold nanocrystals on NaYF₄:Eu,Ga, and Te were grown in situ, unlike prior nanocomposites with NaYF₄ and AuNPs. This straightforward technique achieves a tight bond between AuNPs and NaYF₄:Eu,Ga, and Te, making them inseparable and enhancing synergistic effects in biomedical applications [18].

¹H NMR and XPS examined the coordination between Te and Pt. The synthesis of NaYF₄:Eu,Ga,Te@Au-SH-PEG-Cis-Pt was verified by ¹H NMR (Fig. 5a). The 2.6 ppm peaks from the α protons of Te shifted to 2.9 ppm after compound formation (Fig. 5b). Te binding energy increased from 569.1 and 568.6 eV to 579.9 and 579.5 eV, according to XPS of Te-3d (Fig. 5c). Pt values shifted downward from 76.3 and 72.9 eV to 76.1 and 72.7 eV, consistent with ¹H NMR results [31].

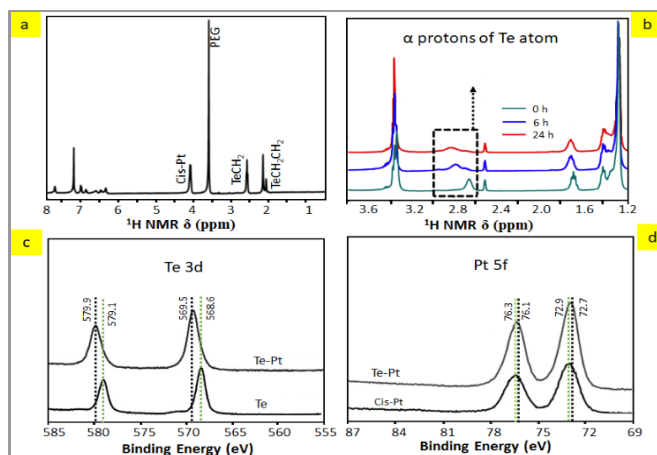


Fig 5. (a) ¹H NMR (500 MHz, D₂O) δ (ppm): 4.15 (4H, b, NHCOOCH₂), 3.66 (8H, b, OCH₂CH₂ of PEG), 2.62 (4H, b, TeCH₂), 2.18 (4H, b, TeCH₂CH₂); (b) α protons of Te atom for different time; (c) XPS spectra of Te-3d in coordination with Cis-Pt and (d) XPS spectra of Pt-5f in Cis-Pt and coordination with Te.

All the crystal diffraction peaks of AuNPs are observable in the XRD patterns of NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites (Fig. 6a). The planes (111), (200), (220), (311), and (222) of AuNPs correspond to the Bragg diffraction peaks at 2θ values of 38.24°, 44.94°, 64.74°, 77.58°, and 81.94° [32]. The additional diffraction peaks are appropriately associated with distinctive peaks of the typical hexagonal step of β-NaYF₄ (JCPDS no.16-0334). The results also demonstrate that the simple technique is successful for producing NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites.

FT-IR spectra helped study the attachment of SH-PEG-Cis-Pt and NaYF₄:Eu,Ga,Te@Au. The FT-IR spectra of Cis-Pt, SH-PEG-HZ, AuNPs, NaYF₄:Eu,Ga,Te@AuNPs, and NaYF₄:Eu,Ga,Te@Au-Cis-Pt are shown in Fig. 6b. Peaks at 2926 and 2857 cm⁻¹ in the NaYF₄:Eu,Ga,Te spectra correspond to the methylene (-CH₂) stretch in the alkyl chain of OA. Additionally, the band at 1567 cm⁻¹ indicates the presence of the C=O group, suggesting OA covers the NaYF₄:Eu,Ga,Te surface[33]. Furthermore, Fig. 2b reveals 1578 and 1715 cm⁻¹ peaks in the SH-PEG-Cis-Pt spectra, linked to C=N and -CONH- in the hydrazone bond, confirming effective SH-PEG-Cis-Pt production[34]. The new peak at 1711 cm⁻¹ in NaYF₄:Eu,Ga,Te@Au-Cis-Pt spectra signals successful conjugation of SH-PEG-Cis-Pt and NaYF₄:Eu,Ga,Te@Au, due to the strong Au-S bond [35].

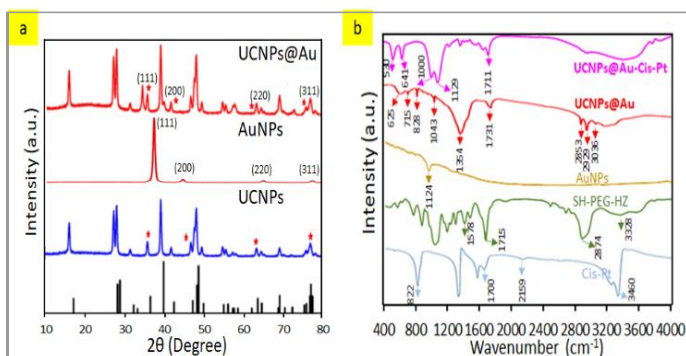


Fig 6. (a) XRD patterns of NaYF₄:Eu,Ga,Te, NaYF₄:Eu,Ga,Te@Au and AuNPs; and (b) FTIR spectra of Cis-Pt, SH-PEG-HZ, AuNPs, NaYF₄:Eu,Ga,Te and NaYF₄:Eu,Ga,Te@Au.

XPS determined the component elements and chemical states, along with the integration of ions (Eu^{3+} , Ga^{3+} , Te^{4+}) into the NaYF_4 host material. Nanocomposite XPS spectra were examined for electronic interactions (Fig. 7). As shown in Fig. 7a, the survey scan spectrum includes Na-1s, Y-3p, F-1s, Eu-3d, Ga-2p, Te-3d, and Au-4f, indicating the presence of AuNPs. Fig. 7b shows Na-1s spectra, with binding energy peaks at 1081 and 1080 eV for $\text{NaYF}_4:\text{Eu,Ga,Te}$ and $\text{NaYF}_4:\text{Eu,Ga,Te@AuNPs}$, respectively. After AuNP incorporation, a 1 eV negative shift in Na-1s binding energy is observed, indicating intimate contact. This peak confirms the Na^{1+} state in both nanocomposites. Fig. 7c shows high-resolution Y-3p spectra with peaks at 309 and 298 eV for $\text{Y-3p}_{3/2}$ and $\text{Y-3p}_{1/2}$ states, indicating the Y^{3+} state. The $\text{NaYF}_4:\text{Eu,Ga,Te@AuNP}$ nanocomposite shows Y-3p peaks (308 and 297 eV) slightly lower than $\text{NaYF}_4:\text{Eu,Ga,Te}$. The F-1s XPS spectra (Fig. 7d) display 693 and 694 eV peaks, confirming F's -1 oxidation state. Peaks at 1119 and 1137 eV ($\text{NaYF}_4:\text{Eu, Ga, Te}$) and 1118 and 1136 eV ($\text{NaYF}_4:\text{Eu, Ga, Te@AuNPs}$) correspond to $\text{Eu-3d}_{5/2}$ and $\text{Eu-3d}_{3/2}$, respectively (Fig. 7e). The XPS spectrum for Ga-2p (Fig. 3f) shows a peak at 1164 eV for $\text{NaYF}_4:\text{Eu,Ga,Te}$ and slightly lower at 1165 eV for $\text{NaYF}_4:\text{Eu, Ga, Te@AuNP}$. Te-3d spectra (Fig. 7g) reveal 580 and 542 eV peaks for $\text{Te-3d}_{3/2}$ and $\text{Te-3d}_{5/2}$ states. For $\text{NaYF}_4:\text{Eu, Ga, Te@AuNP}$, peaks appear at 579 and 540 eV, slightly lower than $\text{NaYF}_4:\text{Eu, Ga, Te}$. The high-resolution Au-4f spectrum (Fig. 7h) displays peaks at 70.9 eV ($\text{Au-4f}_{7/2}$) and 86.6 eV ($\text{Au-4f}_{5/2}$), confirming the presence of metallic Au in nanocomposites [36]. $\text{NaYF}_4:\text{Eu, Ga, Te}$ shows lower binding energy in the nanocomposite formation compared to AuNPs, suggesting electron flow from AuNPs to $\text{NaYF}_4:\text{Eu,Ga,Te}$. Fig. 8a depicts the UCL spectra of $\text{NaYF}_4:\text{Eu,Ga,Te}$, $\text{NaYF}_4:\text{Eu,Ga,Te@Au}$, and $\text{NaYF}_4:\text{Eu,Ga,Te@AuCis-Pt}$ nanocomposites. All nanomaterials exhibit three distinct emission bands at 611 nm ($^5\text{D}_0\text{-}^7\text{F}_2$ transition of Eu^{3+}), which are complemented by other faint emission bands at 580, 593, 654, and 687 nm contributing to the $^5\text{D}_0\text{-}^7\text{F}_0$ transition of Eu^{3+} , $^5\text{D}_0\text{-}^7\text{F}_1$ transition of Eu^{3+} , and $^5\text{D}_0\text{-}^7\text{F}_4$ transition of Eu^{3+} under continuous wavelength excitation of 980 nm, respectively [37]. However, the UCL intensity of $\text{NaYF}_4:\text{Eu,Ga,Te@AuNPs}$ diminished in both green and red regions after the crystal formation of AuNPs on the surface of $\text{NaYF}_4:\text{Eu,Ga,Te}$ (Fig. 8a). Luminescence resonance energy transfer (LRET) occurs as a result of the overlap between the $\text{NaYF}_4:\text{Eu,Ga,Te}$ UCL spectrum and the $\text{NaYF}_4:\text{Eu,Ga,Te@AuNPs}$ absorption spectrum [18]. The reduction in UCL in the green region is stronger compared to the red one due to a greater overlay between $\text{NaYF}_4:\text{Eu,Ga,Te}$ in the green area UCL spectrum and $\text{NaYF}_4:\text{Eu,Ga,Te@AuNPs}$ absorption spectrum. The UCL intensity of $\text{NaYF}_4:\text{Eu,Ga,Te@AuCis-Pt}$ nanocomposites relatively decreased after connecting with SH-PEG-Cis-Pt. Given that the ionic radii of Te^{4+} (0.97 Å) and Ga^{3+} (0.62 Å) were essentially lower than those of Y^{3+} (1.02 Å), the crystal field around Eu^{3+} (1.07 Å) ions were predicted to represent a significant change, leading to a shift in PL characteristics. The intensity of the primary Eu^{3+} emission peak at 611 nm is compared for one sample $\text{NaYF}_4:\text{Eu}^{3+}$, Te^{4+} , Ga^{3+} with $\text{YNaF}_4:\text{Eu}^{3+}$ phosphor in Fig. 8. All other Te^{4+} and Ga^{3+} co-doped samples demonstrate greater intensity, comparatively undoped materials. The primary Eu^{3+} emission peak, which corresponds to the $^5\text{D}_0\text{-}^7\text{F}_2$ transition, does not move after co-doping (Fig. 8), meaning that all Eu^{3+} ions are located on the noncentral symmetric C_2 site of Y^{3+} in the M-type monoclinic fergusonite crystal structure. Some previous studies indicated that $I_{2/1}$ is the space group of the M-type structure [19, 38]. It should be noted that no symmetric center exists in this construction.

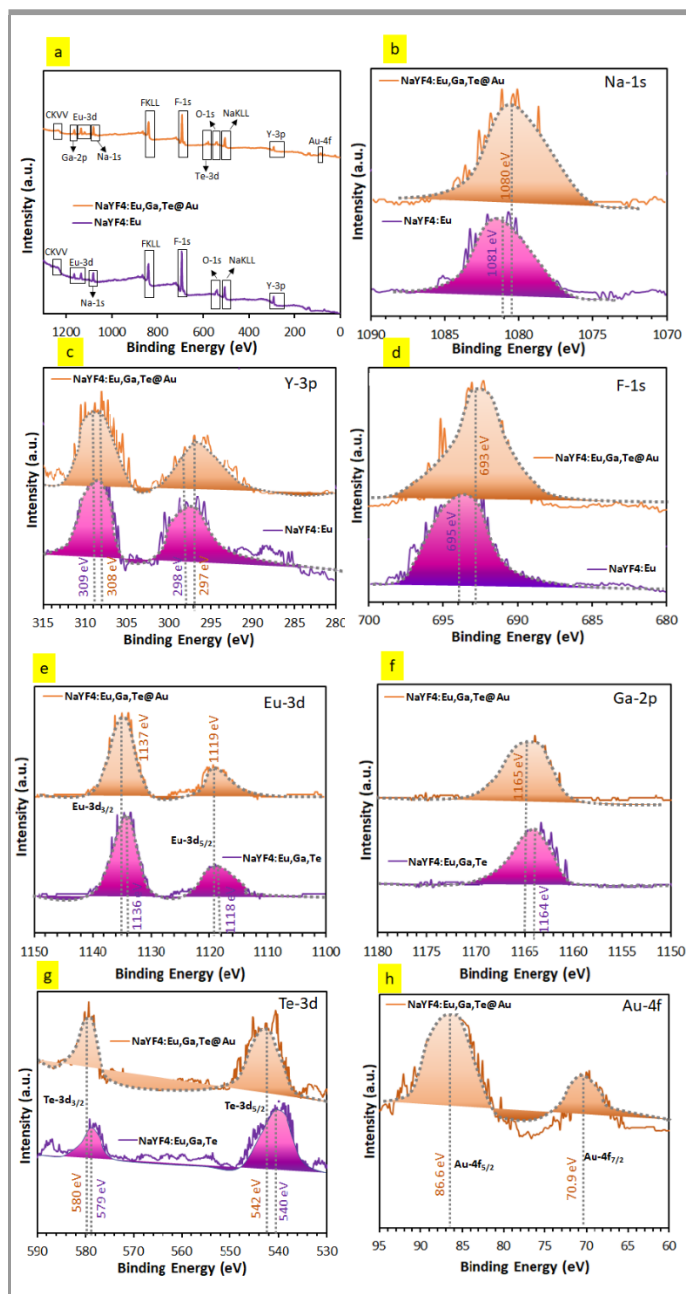


Fig 7. XPS for $\text{NaYF}_4:\text{Eu,Ga,Te@AuNPs}$ (a) survey spectrum; (b) Na-1s; (c) Y-3p; (d) F-1s; (e) Eu-3d; (f) Ga-2p; (g) Te-3d and (h) Au-4f.

Specifically, the transition emission is the greatest peak in low-symmetry lattices, namely, $\text{Eu}^{3+} \text{ } ^5\text{D}_0\text{-}^7\text{F}_2$ [39]. The magnetic dipole transition $^5\text{D}_0\text{-}^7\text{F}_1$ may be employed as an internal standard to compensate for ligand discrepancies since it is normally unaffected by local symmetry. The symmetry of the Eu^{3+} environment is measured by the intensity ratio of the magnetic-dipole transition to the electric dipole transition, which is considered to be hypersensitive to a local symmetry [39]. Based on the results, the intensities of Ga and Te doped materials increased approximately 1.5 times in all emission lines encompassing the most essential $^5\text{D}_0\text{-}^7\text{F}_2$ (611 nm) emission [19].

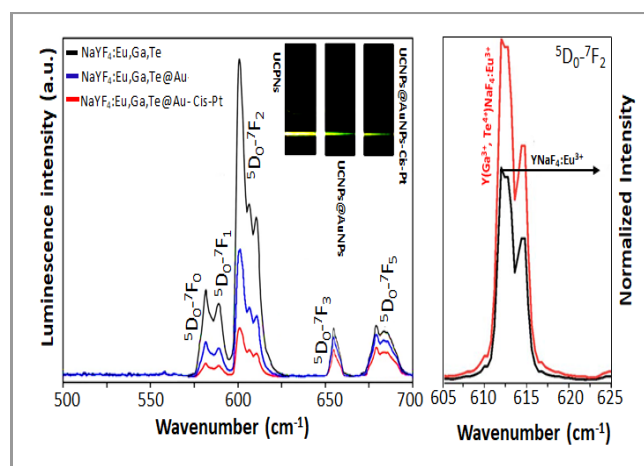


Fig 8. Upconversion luminescence (UCL) spectra of NaYF₄:Eu,Ga,Te, NaYF₄:Eu,Ga,Te@Au and NaYF₄:Eu,Ga,Te@Au-Cis-Pt in water (200 µg/mL); insets are the photographs of NaYF₄:Eu,Ga,Te, NaYF₄:Eu,Ga,Te@Au and NaYF₄:Eu,Ga,Te@Au-Cis-Pt (from left to right) under excitation of a 980 nm laser, 1.5 W/cm².

3.2. PTT

3.2.1. In vitro Cis-Pt release profiles of NaYF₄:Eu,Ga,Te@Au-Cis-Pt

Drug-loaded nanostructures offer advantages like high drug loading and regulated release, enhancing bioavailability while reducing toxicity. NaYF₄:Eu,Ga,Te@Au effectively delivers Cis-Pt with a maximum loading of 51.62% (Fig. 9a). Its loading efficiency surpasses similar composites by attaching Cis to Te and SH-PEG[18]. In acidic conditions, hydrazone bond instability leads to pH-dependent Cis-Pt release from NaYF₄:Eu,Ga,Te@AuCis-Pt[40]. In physiological PBS buffer, release profiles were measured at pH 4.5, 6.0, and 7.4, using Cis-Pt absorption at 510 nm to calculate release. At pH 7.4, the nanocomposites were stable (Fig. 9b), releasing 6% Cis-Pt in 72 h. At pH 6, release was 16.9% in the same period. Fast hydrolysis at pH 4.5 mirrored cancer cell intracellular pH, reaching a plateau with 45.0% total release after 48 h. NaYF₄:Eu,Ga,Te@AuCis-Pt nanocomposites exhibited this due to the reversible hydrazone bond and equilibrium. Most Cis-Pt could be released within 10 h with 808 nm laser irradiation (Fig. 9c). Laser exposure oxidizes water, producing radicals that break the Te-Cis-Pt bond, facilitating significantly faster (vs. 72 h) and higher (vs. 74.9%) release. These findings highlight the potential of NaYF₄:Eu,Ga,Te@AuCis-Pt nanocomposites for intracellular Cis-Pt release in chemotherapy.

3.2.2. Photothermal conversion performance

The Vis-NIR absorbance of NaYF₄:Eu,Ga,Te@AuCis-Pt nanocomposites from 400-850 nm is shown in Fig. 10, indicating it may be suitable for PTT use [41]. Initially, NaYF₄:Eu,Ga,Te@AuCis-Pt was tested in 5 mL bottles for photothermal properties. The temperature of NaYF₄:Eu,Ga,Te@AuCis-Pt at different concentrations was measured over time under constant 808 nm laser radiation (1.5 W cm²) for 10 min (Fig. 11a). The control group, ultrapure water, had a temperature rise of less than 10.4 °C from a starting point of 23.2 °C in 10 min. As concentrations increased (50, 100, 200, and 400 g/mL), the solution's temperature quickly rose in the first 6 min, followed by a slower increase until plateauing. The final temperatures recorded were 10.4, 17.6, 23.3, and 33.5 °C,

respectively (Figs. 11a-b). NaYF₄:Eu,Ga,Te@AuCis-Pt effectively converts light energy (808 nm) to thermal energy.

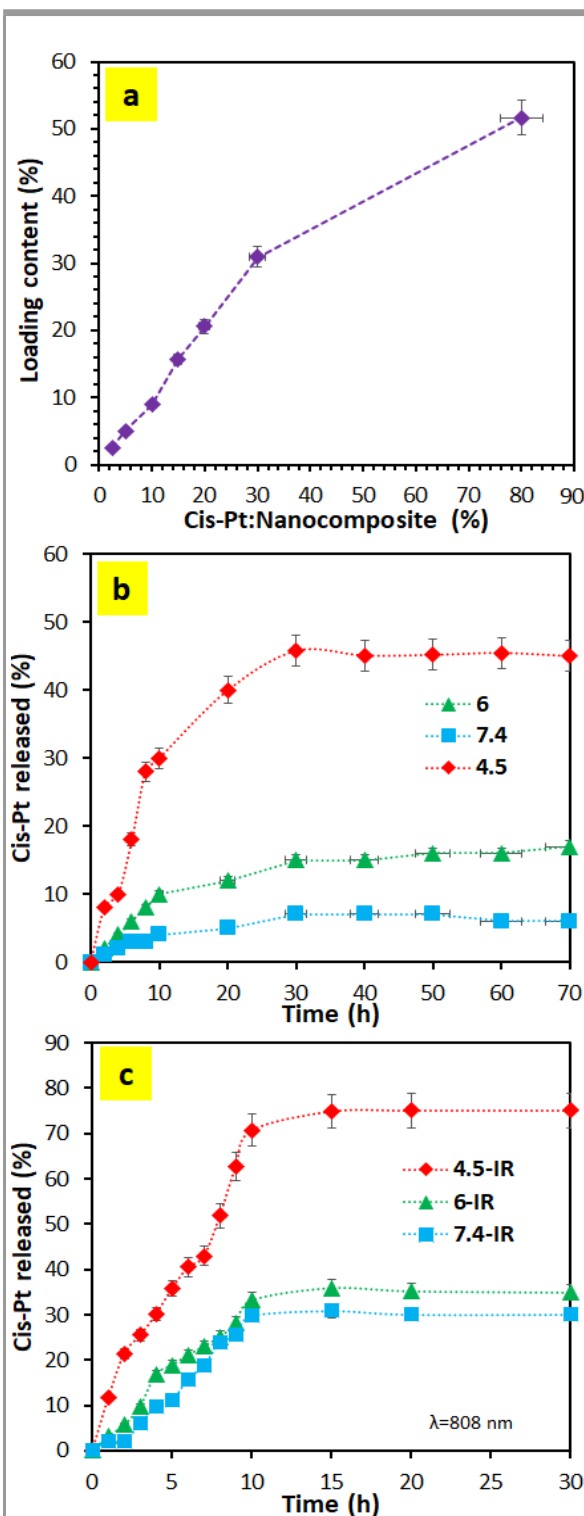


Fig 9. (a) Efficiency of Cis-Pt loading content into NaYF₄:Eu,Ga,Te@Au; Release behavior of Cis-Pt from NaYF₄:Eu,Ga,Te@Au-Cis-Pt in PBS buffer (b) in absence and (c) in presence of laser irradiation.

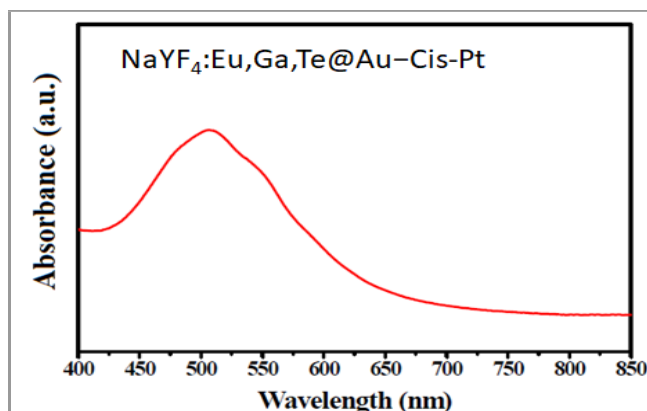


Fig 10. Visible absorption spectrum of NaYF₄:Eu,Ga,Te@Au-Cis-Pt (400 µg/mL) in water.

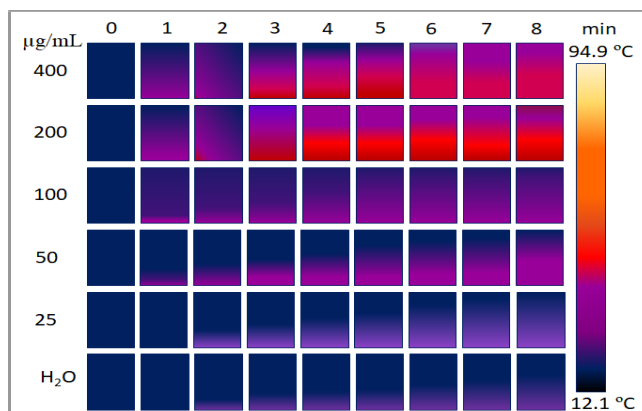


Fig 12. Infrared thermal images at the different time and different concentrations of NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites under 808 nm laser irradiation (1.5 W/cm²).

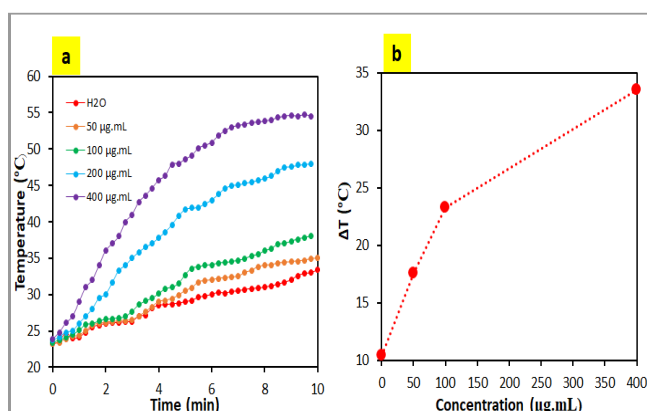


Fig 11. (a) Temperature profiles of pure water and water solution of NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites at different concentrations as a function of irradiation time (0–10 min) under 808 nm laser (1.5 W/cm²) and (b) Plot of temperature change (ΔT) over 10 min versus different concentration of NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites.

Fig. 12 shows infrared thermal images of various concentrations of NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites over time (0 to 8 min) under continuous 808 nm laser (1.5 W/cm²) irradiation. The temperature increased rapidly with concentration, indicated by the intensity bar from 12.1 °C to 94.9 °C. Cancer cells are more heat-sensitive than normal cells and can be destroyed at <42 °C. NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposite is a potential photothermal treatment candidate. After 8 min of irradiation, the nanocomposites reached a stable temperature, making this duration optimal for cytotoxicity evaluation.

3.2.3. Cytotoxicity assessment

Due to the effective release of Cis-Pt in PBS buffer and the robust energy transfer from the 808 nm laser of NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites, we investigated the synergistic impact of photothermal therapy (PTT) and chemotherapy on cancer cells using a constant wavelength laser. The cytotoxicity was evaluated in SW1736 cells treated with NaYF₄:Eu,Ga,Te@Au and NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites at concentrations ranging from 50 to 200 µg/mL, with free Cis-Pt serving as a control.

The toxicity of NaYF₄:Eu,Ga,Te@Au was assessed (Fig. 13), revealing nearly 100% viability in SW1736 cells after 24 hours at 200 µg/mL, demonstrating excellent biocompatibility. Importantly, NaYF₄:Eu,Ga,Te@AuNPs at 50 µg/mL exhibited no toxicity to SW1736 cells when exposed to the 808 nm laser, consistent with temperature data showing no harm (Fig. 11a). Conversely, NaYF₄:Eu,Ga,Te@Au-Cis-Pt exhibited reduced viability, underscoring the synergy between chemotherapy and photothermal treatment. PTT substantially influenced the effectiveness of NaYF₄:Eu,Ga,Te@Au and NaYF₄:Eu,Ga,Te@Au-Cis-Pt at higher concentrations (100 and 200 µg/mL). Under 808 nm laser exposure, NaYF₄:Eu,Ga,Te@AuNPs (200 µg/mL) caused notable phototoxicity, reducing cell survival to 18.6%. Laser treatment decreased viability to 3.0%, significantly lower than the 65.0% viability of NaYF₄:Eu, Ga, Te@Au-Cis-Pt without laser. When NaYF₄:Eu, Ga, Te@AuNPs are above 100 µg/mL, PTT notably affects cancer cells. The NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites (200 µg/mL) also demonstrated acceptable cell viability and treatment efficiency under 808 nm laser irradiation, optimizing the combined effects of chemotherapy and PTT.

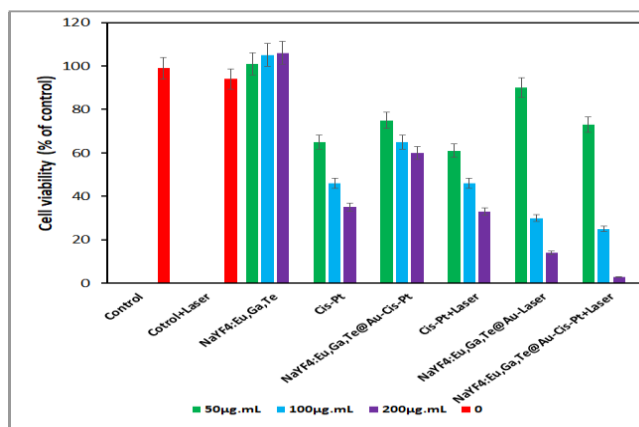


Fig 13. Cytotoxicity assays of SW1736 cells incubated with various concentrations of free Cis-, cit, NaYF₄:Eu,Ga,Te@Au, and NaYF₄:Eu,Ga,Te@Au-Cis-Pt without and with 808 nm laser irradiation for 24 min (1.5 W/cm², 8 min break after each 8 min irradiation).

3.2.4. Cellular uptake and internalization

Under continuous 980 nm excitation, confocal laser scanning microscopy was used to determine the internalization location of NaYF₄:Eu,Ga,Te@Au-Cis-Pt in cells. Nanocomposites may serve as visual tracking agents for anti-cancer treatments. After treating SW1736 cells with NaYF₄:Eu,Ga,Te@Au-Cis-Pt for 2–4 h, green and red UCL signals indicated increased uptake by these cells (Fig. 14). NaYF₄:Eu,Ga,Te@Au-Cis-Pt may enhance UCL signals by releasing Cis-Pt intracellularly. UCL intensities recovered as Cis-Pt was released, reducing the luminescence resonance energy transfer effect between NaYF₄:Eu,Ga,Te@Au and Cis-Pt [42]. The rose-red emission from Cis-Pt significantly increased with incubation time, indicating progressive release from the nanocomposites in SW1736 cells. Various nuclear membrane permeabilities of Cis-Pt and NaYF₄:Eu,Ga,Te@Au influence uptake- Cis-Pt is absorbed more easily via the nuclear pore [43]. Released Cis-Pt from NaYF₄:Eu,Ga,Te@Au-Cis-Pt tends to penetrate the cell nucleus longer, resulting in some Cis-Pt signals (rose red) not fully overlapping with UCL signals (yellow). Thus, NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites are promising for bioimaging applications.

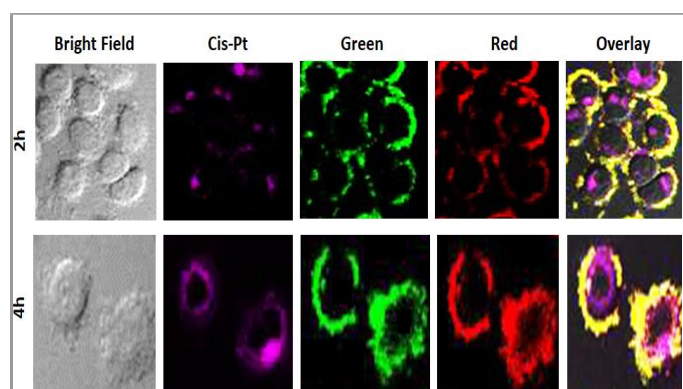


Fig 14. Confocal laser scanning microscopy (CLSM) images of SW1736 cells after incubation with NaYF₄:Eu, Ga, Te@Au-Cis-Pt for 2 and 4 h; the rose red emission from Cis-Pt was collected at 550–700 nm under excitation of 510 nm; a green UCL channel collected upconversion luminescence (UCL) emission at 500–600 nm and a red channel at 600–700 nm, $\lambda_{\text{ex}} = 980 \text{ nm}$, 500 mW.

4. Conclusion

A hybrid nanostructure consisting of a NaYF₄:Eu,Ga,Te core and an AuNPs shell was developed as a photothermal therapy (PTT) agent. In situ production of spherical AuNPs occurred on the surface of NaYF₄:Eu,Ga,Te. The SH-PEG-Cis-Pt prodrug, acting as a chemotherapy agent, was attached to NaYF₄:Eu,Ga,Te@AuNPs, resulting in the NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites, which leverage the effective photothermal properties of NaYF₄:Eu,Ga,Te@AuNPs. The prodrug features a hydrazone bond that allows for pH-responsive drug release in the acidic environment of cancer cells, and Te within NaYF₄:Eu,Ga,Te readily reacts with Cis-Pt, making drug release dependent on irradiation, significantly increasing the release rate to approximately 74.9% at pH 4.5. In vitro studies of cell viability indicated that NaYF₄:Eu,Ga,Te@Au-Cis-Pt nanocomposites exhibit a synergistic effect from both chemotherapy and photothermal therapy, reducing cell viability.

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How to cite this manuscript: Somayeh Mirsadeghi^a, Hamed Zandavar^b, Mahmoud Roushani^a, Seied Mahdi Pourmortazavi^b, Tailoring the Surface Plasmon-Upconversion Interface via Eu³⁺, Ga³⁺, and Te⁴⁺ Doping for Chemo-Photothermal Therapy Treatment against Anaplastic Thyroid Cancer, *Frontiers in Chemical Research*, 2026, 3, 23-31, doi: 10.22034/fcr.2026.2091111.1031.