Synthesis and Examination of New PVA-Fe₂O₃-Au Hybrid Composite

Kahtan A. Mohammed^{1*},Alyaa Abdul Hasan Abdul Kareem², Hawraa K. Judi³, Athota Rathan⁴, Sameer Algburi⁵, Mohammed Ayad Alkhafaji⁶, Rahman S. Zabibah⁷, Shubham Sharma^{8,9}

¹Faculty of pharmacy, Jabir Ibn Hayyan Medical University, Najaf, Iraq

²department of chemistry and polymer research, polymer research center, University of Basrah, Basrah, Iraq

³Department of medical physics, Hilla University College, Babylon, Iraq

⁴Department of Aeronautical engineering, Institute of Aeronautical, Hyderabad, Telangana, India

⁵Computer Engineering Techniques Department, College of Engineering Techniques, Al-Kitab University, Iraq

⁶National University of Science and Technology, Dhi Qar, Iraq

⁷Medical Laboratory Technology Department, College of Medical Technology, the Island University, Najaf, Iraq.

Medical Laboratory Technology Department, College of Medical Technology, the Islamic University, Najaf, Iraq.

8Mechanical engineering Department, University Center for research and Development, Chandigarh University, Mohali, Punjab, 140413, India

⁹School of mechanical and Automotive engineering, Qingdao University of Technology, 266520, Qingdao, China

E-mail: Kahtan444@gmail.com

Abstract

In current work, new hybrid composite have been prepared by mixing gold nanocolloid physically with PVA-Fe₂O₃. Morphological, compositional, structure and optical properties of hybrid composites were studied by transmission electron microscope (TEM), scanning electron microscope (SEM), energy dispersive spectroscopy (EDS), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and UV-visible spectroscopy (UV-VIS). XRD results showed that both Au and Fe₂O₃ have peaks in the structure and that confirmed by EDS and FTIR results. Microscopes results indicated the nanosized nature of prepared materials. UV–vis spectrum show absorption edge at 440 nm which relates to PVA-Fe₂O₃ mixed with Au which means there is a red shift after incorporation of Au in the composites. And the energy gap of composite became equal to 2.48 eV after adding Au solution.

Keywords: Au; Fe2O3; nanocomposite; hybrid materials

1. Introduction

are currently undergoing significant growth and offer a diverse range of possible applications in the domains of biology, engineering, and medicine. Nanoparticles are widely recognized as substantial synthetic entities within the field of nanotechnology, owing to their enhanced characteristics. The aforementioned characteristics encompass diminutive dimensions, elevated surface area, consistent size dispersion, and a diverse array of potential functions in biological and catalytic processes [1-4]. Polymer nanocomposites, also known as PNC for short, are considered to be among the most promising classes of materials that are now available in the category of advanced materials. Nano reinforced materials display a remarkable combination of qualities, allowing for a wide variety of design possibilities. These composite materials are in high demand in both the academic world and the commercial world as a result of their tremendous potential and applicability in a wide range of industries [5].

Nanoscience and nanotechnology are two captivating sub

disciplines within the realm of fundamental science that

Inorganic semiconductors as filling materials combined with polymers consider hybrid materials give the both good optical and mechanical properties of metal oxides and flexibility of polymers. Many metal oxides that used in fabrication of hybrid materials like TiO2, CdSe,CdZnS and more [6-9]

In contemporary times, there has been a notable emphasis on the development of magnetic nanoparticles (MNPs), along with a comprehension of their characteristics and the pragmatic implementation of these MNPs in the realm of life science. Magnetic nanoparticles (MNPs) with diverse physicochemical and structural characteristics have been the subject of extensive investigation for a wide range of applications. These applications encompass magnetic drug targeting, hyperthermia, magnetic resonance imaging (MRI), tissue engineering and regeneration, as well as biosensors and bioanalysis [10]. Iron oxide (Fe2O3) is a compound that is inexpensive, favorable to the environment, and resistant to corrosion; as a result, it is a material that is ideal for use in a variety of applications because of its exceptional stability across

a wide range of pH and temperature, a substantial absorption of solar light (band gap between 2.1 and 2.2 eV), and magnetic assembling with a high Curie temperature. Fe2O3 nanoparticles find application in a wide variety of technological fields, including microwave absorption, catalysis, protection of the environment, gas sensing, magnetic storage, clinical diagnostics and treatment, and many more. Hematite is the form of iron oxide that is the most stable, and it is significant in a wide variety of applications [11-13].

Applications in biomedicine have shown great promise for the use of iron oxide nanoparticles, which have distinctive magnetic characteristics. In more recent times, an expanded research with a variety of iron oxides has been examined as part of a wider study of magnetic particles. Because they readily dissociate under the conditions of an acidic environment, the compounds Fe2O3 and Fe3O4 are particularly interesting candidates in terms of biocompatibility applications. This is due to the fact that it has been established that these substances are friendly to the human body [14-19].

Owing to their distinctive physicochemical properties, as well as a widespread spectrum of high-activity catalysts, precious metal nanoparticles, such as Au, Ag, and Pd, have gained considerable attention in recent decades. There have been a variety of applications for precious metal nanoparticles so far, including hydrogenation, hydrolysis, fuel cells and electrocatalysis [20]. Mixing Au nanoparticles with Fe2O3in purpose of enhancing the properties both type of nanoparticles intended from many researchers because of the variety of applications especially in medicine and biology as anticancer and antibacterial [21-27].

Herein, the preparation of novel PVA- Fe2O3/Au nanocomposites, by synthesizing PVA- Fe2O3 nanoparticles through chemical institumethod and mixing with Au nanocolloid by simple solution blending method was attended and the important properties of nanocomposites was tested.

2. EXPERIMENTAL PART:

2.1. Preparing of PVA-Fe2O3 composite

The preparation and characterization of PVA- Fe2O3 nanocomposite had been done according to our published paper [14]. Where the iron chloride (FeCl3) has been used as iron ions sources, PVA as capping material and sodium hydroxide as an alkaline. 0.1 M FeCl3 mixed with 1%PVA solution on hot plate after that drops of 1 M NaOH were added slowly to the FeCl3+PVA solution with continuous stirring at 70 C. brown colloid was presented indicating the formation of PVA-Fe2O3 nanocomposite.

2.2. gold nanoparticles prepartion

The synthesis of gold nanoparticles was conducted by a chemical reduction approach, employing gold chloride as the source of gold ions and trisodium citrate as the reducing agent. Where The gold chloride solution was diluted by dissolving it in non-ionic water to achieve a concentration of 0.005 M. A volume of 5 milliliters of gold solution with a concentration of 0.005 is combined

with 35 milliliters of deionized water. The resulting mixture is placed on a magnetic device and heated until it reaches its boiling point. The mixture is then allowed to boil for a duration of 15 to 20 seconds. One milliliter of Sodium citrate, which was made by dissolving 1 gram of TSC powder in 100 milliliters of deionized water, is into the aforementioned introduced mixture. Subsequently, the heat source is deactivated and the mixture is left on the magnetic stirring apparatus. The solution undergoes a transition from coloration to colorlessness, followed by a subsequent change to a dark red hue (specifically, a violet shade that ultimately transforms into a vibrant red tone). This color transformation serves as an indicator for the attainment of nanoparticles with dimensions that are comparatively diminutive (less than 20 nm in size).

2.3. PVA-Fe2O3/Au nanocomposite preperation

PVA-Fe2O3/Au nanocomposite have been prepared by using solution mixing method which it done by mixing 1ml from PVA-Fe2O3 with 1 ml of Au NPs together and left on magnetic strerr at room temparature for 5 hours after that we get a homogenuos solution.

2.4. Instrumentation

The determination of the crystal structure, surface morphologies, chemical compositions, and optical characteristics of the hybrid nanomaterials was conducted using. The X-ray diffraction (XRD) instrument used in this study was the D2 phaser model manufactured by Bruker, the CuK 1.544 nm target created the X-ray beam, 10 mA at 30 KV. The prepared samples were tested by some of instruments like: TEM Electron Microscopy model a JEOL JEM. 2010 with resolution 0.23 nm and 200kV accelerating voltage(FESEM) model MIRA3 from TE SCAN Company, ALPHA II from Bruker Fourier transformation infrared spectroscopy (FTIR)was used in this investigation, and UV-Visible spectroscopy (UV-Vis) type 1800 from Shemadzu Spectroscopy in the range of wavelength (290-900) nm was employed for the UV-Visible spectroscopy (UV-Vis).

3. Results and discussions

The X-ray diffraction (XRD) procedure has been employed to investigate the crystal structure of the synthesized materials. picture 1 illustrates the diffraction peaks observed in the PVA-Fe2O3-Au composite material, while the inset picture displays the patterns specifically observed in the PVA-Fe2O3 component. The amorphous nature of the PVA was confirmed by the presence of a broad peak corresponding to the polymer matrix at an angle of $2\theta = 210$. The diffraction peaks associated with Fe2O3 are plainly observable. The confirmation of Fe2O3 production was established by the presence of two diffraction peaks at $2\theta = 33.50$ and $2\theta =$ 38.0o. A novel observation was made at a diffraction angle of $2\theta = 270$, indicating the presence of gold nanoparticles (Au NPs). and this results matching ref. [28].

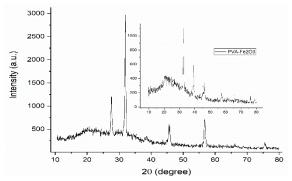


Figure 1. XRD peaks of PVA-Fe2O3-Au composite.

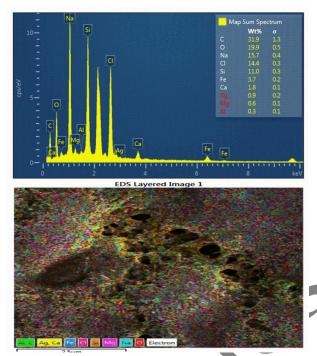


Figure 2. EDS spectrum and mapping of PVA-Fe2O3-Au composite.

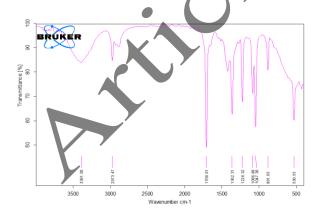


Figure 3. FTIR spectrum of PVA- Fe2O3-Au hybrid composite

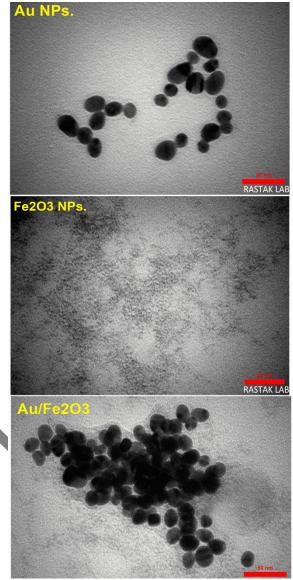


Figure 4. TEM graphs of Au NPs (a), PVA- Fe2O3 (b) and PVA- Fe2O3-Au composites (c).

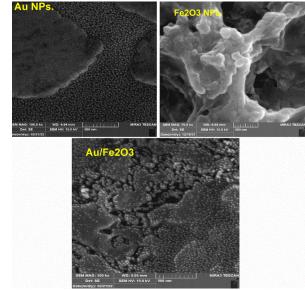


Figure 5. FESEM graphs of Au NPs, PVA- Fe2O3 and PVA-Fe2O3-Au composites

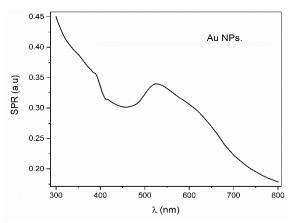


Figure 6. absorption spectrum and corresponding energy gap of Au NPs.

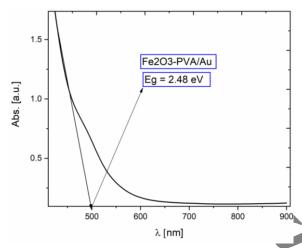


Figure 8. absorption spectrum and corresponding energy gap of PVA-Fe2O3-Au composites.

EDX analysis was performed to ascertain the chemical composition of the prepared samples and identify the components present. Figure 2 presents the EDX spectra and mapping image of the PVA-Fe2O3-Au nanocomposite. The graphic illustrates the presence of numerous ingredients within the remedy. and this results matching ref.[29]

Figure 3 displays the FTIR spectrum of PVA-Fe2O3-Au nanocomposite , where the spectrum have many transmission peaks belong to the PVA, Fe2O3, Au and the interaction between all compounds. All the peaks are assigned to the PVA bonds: The O–H stretching vibration of the hydroxyl group, which is sensitive to the hydrogen bond, is responsible for the broad absorption band that can be found at approximately 3391 cm-1 [30,31].

The peak about 532 cm-1 is related to Fe-O stretch of Fe2O3 and no peaks belong to Au and that confirm there is no chemical interaction between Fe2O3 and Au and the interaction physical only [32,33].

The morphologies and sizes of the Au nanocolloid were reconnoitered using the TEM which is signified by Figure 4a. from the figure, it can be seen that the nanoparticles are disprased in a good way, as there are no agglomerations, and mono particle can be distinguished from another easily, as well as most of the particles are identical in shape and size. The dominant shape of most

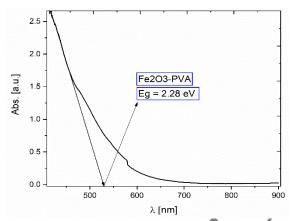


Figure 7. absorption spectrum and corresponding energy gap of PVA-Fe2O3.

of them is spherical, and their diameters are about 10 nm. The form of the PVA-Fe2O3 particles is depicted in Figure 4b. This picture illustrates the encapsulation of iron oxide nanoparticles within a polymer matrix, resulting in the formation of irregularly shaped particles. Figure 4c show the TEM image of mixing both Fe2O3 and Au .

FESEM examination displays the formation of Au, Fe2O3 and their mixing (figure 5). In the case of Au nanoparticles the figure shows formation spherical particles with nanosized distribution. Through this figure, The polymer envelops the iron oxide nanoparticles, resulting in the formation of irregular particle forms. FESEM measurement exposed that the produced nanocomposites have different sizes and shape and the diameters were in nanoscale. There is a possibility that the smaller nanoparticles aggregated together to form the larger ones and this result agreed with reference [30]. UV-Vis spectroscopy is used to study the absorption spectra and corresponding energy gaps nanoprticles, PVA- Fe2O3 and PVA- Fe2O3-Au nanocomposites. UV-Vis spectroscopy is used to study the absorption spectra of gold nanoparticles. Figure 6 shows the UV-Vis absorption spectrum of Au NPs. the figure display the surface plasmon resonance position around 520nm.

Figure 7 illustrates the absorption spectrum of the PVA-Fe2O3 nanocomposite. The absorption edge of the PVA-Fe2O3 composite material was observed at approximately 470 nm. The energy gap was determined to be 2.28 electron volts (eV).

Figure 8. represents the UV–Vis spectrum of absorption for PVA-Fe2O3-Au nanocomposites. The UV-visible spectrum exhibits an absorption edge at a wavelength of 440 nm, indicating the presence of a nanocomposite structure consisting of PVA-Fe2O3 covered with Au. This observation serves as confirmation of the aforementioned materials' composition, which means there is ared shift after incorporation of Au in the composites and this result agreed with results that got from. [33] and [34]. the corresponding energy gap was calculated and equal to 2.48 eV.

Conclusions

Gold (Au) nanoparticle, PVA- Fe2O3 nanoparticle and PVA-Fe2O3-Au nanocomposite were synthesized in this work. These prepared composite materials were characterized by using different analytical methods such as: TEM, FESEM, and UV-Vis spectroscopy. XRD results showed that both Au and Fe2O3 have peaks in the structure and that confirmed by EDS and FTIR results.

The morphology of the prepared nanocomposite was analyzed by TEM and FESEM, which reveals the creation of a tiny spherical shape of PVA- Fe2O3-Au nanocomposites with a diameter that falls between 40 and 60 nm. there is a red shift absorption edge after incorporation of Au in the composites. And the energy gap of composite became equal to 2.48 eV after adding Au solution.

References

- 1. Ahilfi, D. N., Alkabbi, A. S., Mohammed, K. A., & Ziadan, K. M. (2020, November). Fabrication and characterization of polyaniline/CdSe device for applications in nano structured solar cells. In IOP Conference Series: Materials Science and Engineering (Vol. 928, No. 7, p. 072069). IOP Publishing.
- 2. D. De, S.M. Mandal, S.S. Gauri, R. Bhattacharya, S. Ram, S.K. Roy, Antibacterial effect of lanthanum calcium manganate (La 0.67Ca0.33MnO3) nanoparticles against Pseudomonas aeruginosa ATCC 27853, J. Biomed. Nanotechnol. 6 (2010) 138–144.
- 3. Mohammed, K. A., Ziadan, K. M., Al-Kabbi, A. S., Saxena, K. K., Zabibah, R. S., Alrubaie, A. J., & Mohammed, J. H. (2022). Optical, morphological, electrical properties and white light photoresponse of CdSe nanoparticles. Advances in Materials and Processing Technologies, 1-10.
- 4. M. Khatami, H.Q. Alijani, B. Fakheri, M.M. Mobasseri, M. Heydarpour, Z.K. Farahani, A. Ullah, Super-paramagnetic iron oxide nanoparticles (SPIONs): greener synthesis using Stevia plant and evaluation of its antioxidant properties, J. Clean. Prod. 208 (2019) 1171–1177
- 5. Namdev, A., Purohit, R., Telang, A., Kumar, A., Saxena, K. K., Mabuwa, S., ... & Mohammed, K. A. (2022). Optimization of dry sliding wear behavior of epoxy nanocomposites under different conditions. Materials Research Express, 9(6), 065303.
- 6. Talib, R. A., Thbayh, D. K., & Mohammed, K. A. (2022). Study the Optical and Morphological Properties of Prepared PANI/TiO2 Nanocomposites. In Materials Science Forum (Vol. 1065, pp. 101-108). Trans Tech Publications Ltd.
- 7. Talib, R. A., Mohammed, K. A., & Thbayh, D. K. (2022). Study the structural and electrical properties of prepared PANI/TiO 2 nanocomposites for electronic devices. Journal of Ovonic Research, 18(3).
- 8. Kahtan A. Mohammed, Kareema M. Ziadan, Alaa S. AL-Kabbi, Alrubaie, Rahman S. Zbibah and Ali Conducting Polymer/CdSe Hybrid as Bulk Heterojunction Solar Cell, ACTA PHYSICA POLONICA A No. 3 Vol. 141 (2022).
- 9. Husseina, M. A., Mohammedb, K. A., & Talibc, R. A. (2022). Energy band gaps and optical absorption properties of the CdZnS and CdZnS: PEO thin films prepared by chemical bath deposition. Chalcogenide Letters, 19(5), 329-335.
- 10. Mohammed, K. A., Abdulridha, S. A., Alibory, E. H., Alkhayatt, A. H. O., Zabibah, R. S., Alrubaie, A. J., ... & Kadhem, M. J. (2022). Capping agent effect on optical properties of Fe2O3 nanoparticles. Materials Today: Proceedings, 56, 2010-2015.
- 11. Tadic, M., Trpkov, D., Kopanja, L., Vojnovic, S., & Panjan, M. (2019). Hydrothermal synthesis of hematite (α-Fe2O3) nanoparticle forms: synthesis conditions, structure, particle shape analysis, cytotoxicity and magnetic properties. Journal of Alloys and Compounds, 792, 599-609.
- 12. Asadikia, A., Mirjalily, S. A. A., Nasirizadeh, N., & Kargarsharifabad, H. (2020). Characterization of thermal and electrical properties of hybrid nanofluids prepared with multi-walled carbon nanotubes and Fe2O3 nanoparticles. International Communications in Heat and Mass Transfer, 117, 104603.
- 13. Lassoued, A., Lassoued, M. S., Dkhil, B., Ammar, S., & Gadri, A. (2018). Synthesis, photoluminescence and Magnetic properties of iron oxide (α-Fe2O3) nanoparticles through precipitation or hydrothermal methods. Physica E: Low-dimensional Systems and Nanostructures, 101, 212-219.
- 14. Al-Tememe, E. H., Algalal, H. M. A. A., Abodood, A. A. F., Mohammed, K. A., Khamees, E. J., Zabibah, R. S., & Abed, A. S. (2022). Anticancer and Antimicrobial activity of PVA/Fe2O3/TiO2 hybrid nanocomposite. International Journal of Nanoscience, 2250018.
- 15. Theerthagiri, J., Dalavi, S. B., Raja, M. M., & Panda, R. N. (2013). Magnetic properties of nanocrystalline ε-Fe3N and Co4N phases synthesized by newer precursor route. Materials Research Bulletin, 48(11), 4444-4448.
- 16. Chatterjee, J., Haik, Y., & Chen, C. J. (2003). Size dependent magnetic properties of iron oxide nanoparticles. Journal of Magnetism and Magnetic Materials, 257(1), 113-118.
- 17. Basith, N. M., Raj, R. A., AlSalhi, M. S., Devanesan, S., Askar Ali, S. J., Rajasekar, S., ... & Ragupathi, C. (2016). Structural, magnetic, optical, and catalytic properties of Fe3O4 nanoparticles by the sol-gel method. Journal of Superconductivity and Novel Magnetism, 29(8), 2053-2058.
- 18. AlSalhi, M. S., Devanesan, S., Alfuraydi, A. A., Vishnubalaji, R., Munusamy, M. A., Murugan, K., ... & Benelli, G. (2016). Green synthesis of silver nanoparticles using Pimpinella anisum seeds: antimicrobial activity and cytotoxicity on human neonatal skin stromal cells and colon cancer cells. International journal of nanomedicine, 11, 4439.
- 19. AlSalhi, M. S., Devanesan, S., Shanmugam, P., Kim, Y. O., Kwon, J. T., & Kim, H. J. (2020). Synthesis and biocompatible role of hierarchical structured carbon nanoplates incorporated α-Fe2O3 nanocomposites for biomedical

- applications with respect to cancer treatment. Saudi Journal of Biological Sciences, 27(2), 588-593.
- 20. Fu, M., Li, M., Zhao, Y., Bai, Y., Fang, X., Kang, X., ... & Xu, X. (2021). A study on the high efficiency reduction of p-nitrophenol (4-NP) by a Fe (OH) 3/Fe 2 O 3@ Au composite catalyst. RSC advances, 11(43), 26502-26508.
- 21. Shams, S., Khan, A. U., Yuan, Q., Ahmad, W., Wei, Y., Khan, Z. U. H., ... & Ullah, S. (2019). Facile and eco-benign synthesis of Au@ Fe2O3 nanocomposite: Efficient photocatalytic, antibacterial and antioxidant agent. Journal of Photochemistry and Photobiology B: Biology, 199, 111632.
- 22. Zhong, D., Zhao, J., Li, Y., Qiao, Y., Wei, Q., He, J., ... & Zhou, M. (2019). Laser-triggered aggregated cubic α-Fe2O3@ Au nanocomposites for magnetic resonance imaging and photothermal/enhanced radiation synergistic therapy. Biomaterials, 219, 119369.
- 23. Trang, V. T., Tam, L. T., Phan, V. N., Van Quy, N., Huy, T. Q., & Le, A. T. (2017). Two-step hydrothermal synthesis of bifunctional hematite–silver heterodimer nanoparticles for potential antibacterial and anticancer applications. Journal of Electronic Materials, 46(6), 3323-3332.
- 24. Fodjo, E. K., Gabriel, K. M., Serge, B. Y., Li, D., Kong, C., & Trokourey, A. (2017). Selective synthesis of Fe3O4Au x Ag y nanomaterials and their potential applications in catalysis and nanomedicine. Chemistry Central Journal, 11(1), 1-9.
- 25. Zeng, X., Ruan, Y., Chen, Q., Yan, S., & Huang, W. (2022). Biocatalytic cascade in tumor microenvironment with a Fe2O3/Au hybrid nanozyme for synergistic treatment of triple negative breast cancer. Chemical Engineering Journal, 138422.
- 26. Wang, J., Zhu, F., Li, K., Xu, J., Li, P., & Fan, Y. (2022). pH-responsive mesoporous Fe2O3–Au nanomedicine delivery system with magnetic targeting for cancer therapy. Medicine in Novel Technology and Devices, 15, 100127.
- 27. Rajan, D., Francis, F., Thomas, D., Ramankutty, M., & Joseph, S. A. (2022, March). Study of the utility of Fe2O3-Au nanocomposite in photodynamic therapy. In IOP Conference Series: Materials Science and Engineering (Vol. 1233, No. 1, p. 012005). IOP Publishing.
- 28. Song, Y., Tao, L., & Shen, X. (2012). Synthesis of new type of Au-magnetic nanocomposite and application for protein separation thereof. Nanoscale research letters, 7, 1-5.
- 29. Nasri, A., Nezafat, Z., Jaleh, B., Orooji, Y., & Varma, R. S. (2021). Laser-assisted preparation of C 3 N 4/Fe 2 O 3/Au nanocomposite: a magnetic reusable catalyst for pollutant degradation. Clean Technologies and Environmental Policy, 23, 1797-1806.
- 30.Multimodal cancer cell therapy using Au@Fe2O3 core-shell nanoparticles in combination with photo-thermoradiotherapy
- 31. Dai, Y., Tang, Q., Zhang, Z., Yu, C., Li, H., Xu, L., ... & Zou, Z. (2018). Enhanced mechanical, thermal, and UV-shielding properties of poly (vinyl alcohol)/metal organic framework nanocomposites. RSC advances, 8(67), 38681-38688.
- 32. Pariti, A., Desai, P., Maddirala, S. K. Y., Ercal, N., Katti, K. V., Liang, X., & Nath, M. (2014). Superparamagnetic Au-Fe3O4 nanoparticles: one-pot synthesis, biofunctionalization and toxicity evaluation. Materials Research Express, 1(3), 035023.
- 33. Abdillah, P., & Yulizar, Y. (2021, August). Green Synthesis of Porous Fe2O3/Au Nanocomposite with Chitosan Template using Gliricidia Sepium Leaf Aqueous Extract and its Catalytic Activity for the Reduction of 4 Nitrophenols. In Proceedings of The 6th Asia-Pacific Education And Science Conference, AECon 2020, 19-20 December 2020, Purwokerto, Indonesia.
- 34. Nasri, A., Nezafat, Z., Jaleh, B., Orooji, Y., & Varma, R. S. (2021). Laser-assisted preparation of C3N4/Fe2O3/Au nanocomposite: a magnetic reusable catalyst for pollutant degradation. Clean Technologies and Environmental Policy, 23(6), 1797-1806.
- 35. Mirzaei, A., Janghorban, K., Hashemi, B., Bonavita, A., Bonyani, M., Leonardi, S. G., & Neri, G. (2015). Synthesis, characterization and gas sensing properties of Ag@ α-Fe2O3 core–shell nanocomposites. Nanomaterials, 5(2), 737-749.