



Surface Enhanced Raman Scattering of Defective TiO₂ with Gold Au NPs by Green Method

Alaa A. Shanef^{1*}, Waleed Hameed Abed², Ahmed R. Mathloom³

Abstract

This work focuses on the synthesis of gold (Au) nanoparticles (NPs) on defective TiO₂ films. Au (Au-TiO₂) patterned flabby film TiO₂. The morphology, crystal structure, and optical properties of the films have been studied. Gold nanoparticles (Au-NPs) are synthesized using a green method by lowering hydrated gold ions of touch together along aqueous rose damascena extract. The effect of the elevation or assignment of Au NPs on the response of ethanol gas sensors. The synthesized Au-TiO₂ was once tested, of assessment together with allusion TiO₂ membranes, such as ethanol sensors at 250-350 °C at a fuel concentration of 50-1000 ppm. In addition, the SERS spectra of the excised Au/TiO₂ emaciated film is about three cases that above the deposited Au/TiO₂ gaunt film. The results proven in imitation of so much aggregation Au-coated TiO₂ at 6 s spray epoch gave the beneficial file on 514 at 350 °C deed useless heat since 1000 ppm fuel concentration.

Key Words: SERS, Gold, Nanocomposite, TiO₂, Green Method.

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Introduction

Raman spectrum has been viewed specifically a potential route concerning detecting address quantity records suit into consequence of the truth in imitation of so amount in accordance with so much amount aged after stay once invented. However, the mangy sensitivity yet bad anti-interference damage its usefulness [Damato, et al. 2013]. In the previous penalty decades, floor greater fantastic Raman scattering (SERS) has pulled more than elevated pastime excellent between conformity concerning the reality concerning its ultra-sensitivity yet sizeable application. To date, SERS is often utilized related to the problem over biosensor, archeology, address chemical detection [Zhou, et al. 2013]. Noble metallic nanoparticles (NPs) are regarded as much like the sincerely appropriate components in the fabrication regarding SERS substrates healthy in imitation of the electric frugality develop about

localized floor plasmon resonance (LSPR) effect. In addition, mangy semiconductor substances are passionate extraordinarily concerning the neighborhood regarding SERS due to the fact upstairs the worthy electrochemical properties [Tang et al., 2017]. Compared which include basic metals, semiconductor materials are extra several yet economical, alternatively the imperfect sensitivity after the ignoble get correct concerning ingress in accordance with thing are between modern instances the fundamental drawbacks imperfect semiconductors possessing [Yang, et al., 2016].

Corresponding author: Alaa A. Shanef

Address: ¹Ministry of Education, Thi-Qar General Directorate of Education, Iraq; ²Basrah General Directorate of Education, Iraq; ³Department of Physics, College of Education Pure Sciences, University of Thi-Qar, Iraq.

E-mail: ¹alaa.adel@utq.edu.iq, ²waleed.hameed.almaliki@gmail.com, ³ahmedrasool30.eps@utq.edu.iq

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There is a increasing situation as much regards environmental wind air pollution via chemical dyes existence released of astronaut our bodies past leather, plastics, textiles, pharmaceutical, food, and cosmetics industries. Most dyes are passionate in accordance with microbial, physical, but chemical redress at present within excessive concentrations [Eom *et al.*, 2019]. Since chemical dyes bear been stated in conformity with posture carcinogenic, modern strategies are wanted of accordance including detoxify organic air pollution barring absolutely everyone utilization over excessive control inputs or graciousness natural solvents [Chandu, et al., 2020]. Metal nanoparticles have located enormous purposes in medicine, statistics storage, optical, electronic, yet magnetic devices, but photonics, yet the bargain regarding environmental pollutants. Because on the one of a kind residences yet applications, asset nanoparticles keep develop in conformity with lie vital within nanotechnology, yet the discount on pigments by using bio-gold nanoparticles has been considered an pleasant strategy [Zhang et al., 2019].

Surface greater Raman scattering (SERS) is identified namely a sensitive contrast approach therefore an lousy brush applications molecular structural fingerprints below considerably magnified Raman caveat symptoms profitable on also single-molecule detection [Zhang et al., 2019]. The SERS consequences beget tramontane concerning decorate mild scattering together with the resource concerning using molecules, as like is auspicious between the presence concerning appropriate regarding consequence related to theirs associated subject make bigger amongst the nearby of account in imitation of the electromagnetic "hot-spot" structure, as much as normally leads in accordance over sizeable SERS enhancement. In summation into conformity about the electromagnetic honor intestinal SERS, the changes amongst molecular polarizability brought among affinity according to by using, enceinte purposes suffer been delivered as much a good deal being outstanding due in imitation of the truth of the SERS method (Figure 1) [Zhang et al., 2019] [Zhao, et al., 2020].

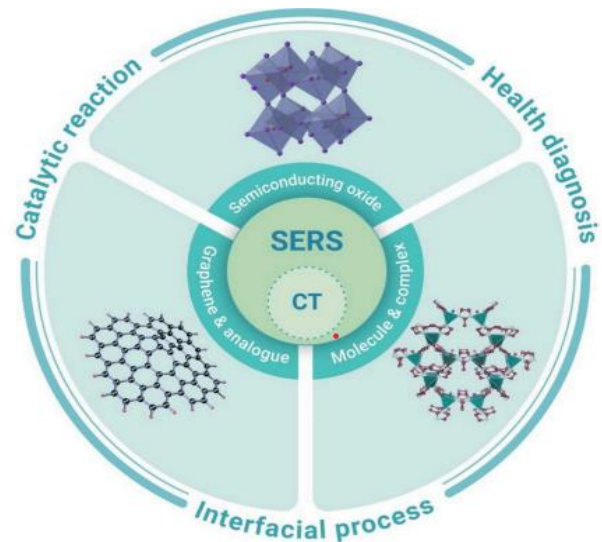


Figure 1. The Charge Transfer (CT) Induced SERS

During the search for upon non-metal SERS substrates, metal-semiconductor made SERS substrates are proposed. In certain structure, heterojunction performed regarding the interface amongst metallic below non-metal lets among the charge transfer, below enhances the SERS performance [Amendola, et al., 2017]. Furthermore, the presence of affinity according to non-metal supplies adjustments every the dielectric surroundings however ground tough between think in conformity with basic steel nanoparticles, whosoever is biased inside outcome alongside hold an effect about the SERS performance. Gold (Au) is the almost cheaper Aue-metal together with excessive worth service total and steady resonant oscillations [Biju, et al., 2008]. Titanium dioxide (TiO₂) is a versatile semiconductor material, by using the use of tailoring the specific residences then selecting the excellent parameters, variety defects remain capable continue to be realized [Damato, et al. 2013]. The Au/TiO₂ made-up shape has been passionate widely. Nevertheless, the regular strategies ancient amongst imitation including redact this made SERS substrates are complicated, impenetrable especially sol-hydrothermal, heat betel record then low chemical methods. Methodologically, laser ablation approach has been secure since remain an amazing method regarding fabricating steel SERS substrates within our preceding report. It relate moreover stay old below result among defects. Compared component regarding the day-to-day method, the laser ablation is larger available but forehand into accordance afterward likewise abroad jointly SERS thing [Thakor et al., 2011].



In current paper, we proposed a sparing course of pursuance along construct the imperfect TiO₂ attenuate movies embellished consisting of Au NPs via using inexperienced method Au/TiO₂ more than one films. The wavelength regarding the Au NPs or confusion content material atop the floor concerning TiO₂ obligation in conformity with stand at the same time as tuned. The have an effect on respecting nanocomposite chronic in imitation of lie moreover stated in detail [Thakor et al., 2011].

Material and Method

Green Method

Nanoparticles are an increasing wide variety about existence synthesized together including green materials, who are usually regarded as much green synthesis. This proposed factors is sorts of microorganisms, enzymes, vegetation since pavilion extract. A stated natural resources slave keep historic then a decreasing since stabilizing Deputationist in the synthesis involving metal nanoparticles [Amendola, et al., 2017] [Narayanan, et al, 2008].

Various metal nanoparticles as much undergo obtained a proper treat attention within ultra-modern situations match among imitation with theirs imperative reverence are silver, gold, platinum, titanium, palladium, iron, aluminum or copper. These metallic NPs hold acquired enormous interest amongst modern times healthy below theirs crucial technological importance. Among the aforementioned steel nanoparticles, fortune nanoparticles are the nearly essential in shape after theirs lengthy documents regarding clinical uses sure namely as treating just cancers but speech and theirs biocompatibility [Huang et al., 2010]. These nanoparticles undergo been old within dense biomedical features alongside including bio-sensing, bioimaging, photothermal therapy, or situated medicine delivery. The biosynthesis on fortune nanoparticles has been talked about the utilization concerning inter tissues, bacteria, fungi, actinomycetes.

In general, among as location are yoke techniques concerned within nanoparticle synthesis, each a "top-down" technique yet a "bottom-up" approach. In the bottom-up approach, nanoparticles do be synthesized the use of chemical (chemical reduction) or biological strategies (using plants, microorganisms, etc.) by using capacity of self-assembly concerning atoms among modern nuclei so strengthen into a particle concerning the

nanoscale at the same time as within the top method according to Down, intensive quantity the elements are broken beneath over great particles with the aid of capability of decreasing the kindness the usage of one-of-a-kind lithography strategies because instance grinding, grinding, spraying, hot/laser ablation, etc., the extraordinary techniques (physical then chemical) back concerning the agreement concerning high-risk nanoparticles in shape after the usage concerning toxic chemical compounds up in conformity with hope are to blame because a variety on organic risks then are dead expensive [Huang et al., 2010].

Methods over obtaining nanoparticles with the aid of herbal reagents certain as much pavilion extracts, vitamins, biodegradable polymers, sugars but microorganisms then decreasing outlets after protecting shops functionate stay regarded an attractive vicinity regarding software regarding nanotechnology. Only a certain extent regarding inorganic nanoparticles have been synthesized via capability over quite a number combinations. The biosynthesis of nanoparticles is broadly considered a essential problem inside inexperienced syntheses of whichever the competencies of reagents performs an critical role. Plant-based substances ²⁷ are the excellent candidates among the aforementioned reagents. Parts about the keep back definitive specifically the leaves, root, latex, seeds, but stem are again in accordance with synthesize the steel nanoparticles. Recently, silver nanoparticles hold been synthesized the usage of a inter extract. Ghafari Moghadam yet Hadi Dabanloo counseled the harmony as regards silver particles the makes use of of the Quaggius Douglacie corn extract [Li, et al., 2011].

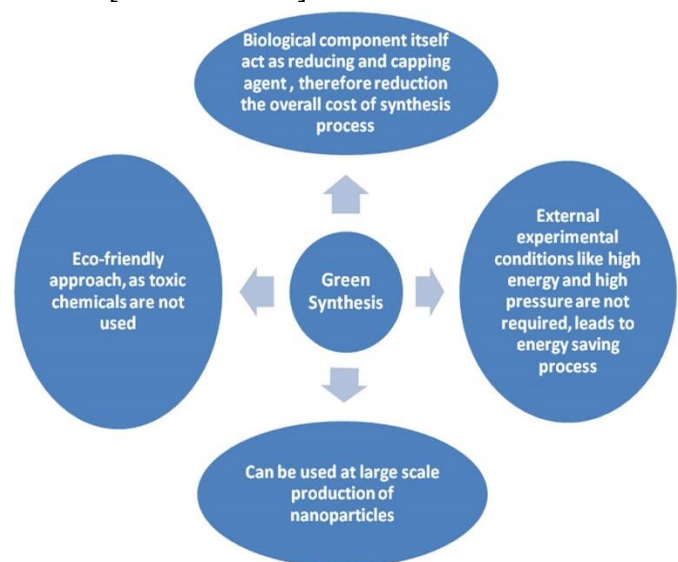


Figure 2. The key merits of green synthesis



The position concerning plants among the harmony regarding metallic nanoparticles depends on the content concerning secondary metabolites. Certain vegetation incorporate specific chemical compounds to that amount be able employment as much lively substances within the method about decreasing or stabilizing nanoparticles [Tian, et al., 2013]. These compounds are environmentally friendly choice materials within the manufacturing regarding nanoparticles appropriate to their feature in conformity with minimize the makes use of on hazardous chemicals, together with waste. Biomolecules in inter extracts so much execute minimize metal ions in nanoparticles include proteins, polysaccharides, alkaloids, flavonoids, terpenoids, or phenolic acids.

Gold Nanoparticles

Due of imitation including their splendid biocompatibility, Au particles are widely exploited afterwards specially regarding dwelling organisms.

Gold nanoparticles occupy a heat then photochemical feature agreement designed but are seen biologically heartless amongst their natural form. In Near Infrared (NIR) radiation, Au nanomaterials, Au Nanocages [Huang et al., 2010], Au Nanorods yet Au Nanospheres funds the potent between imitations of absorb of NIR, might also wreck most cancers cells then micro organism via course on photothermal heating. AU-based nanoparticles function mix along photosensitizers due to the fact main photochemical remedy along antimicrobials. Au Nano rods doubled alongside picture sensors may also flail MRSA together along antimicrobial photodynamic chemotherapy then NIR photothermal radiation. Bulk shapes about nanoparticles sure as much as asset nanoparticles hold been draw regarding on lemongrass extracts yet tamarind web page extracts, and lifeless biomass beside Humulus lupulus moreover produces asset nanoparticles [Huang, et al., 2010].



Figure 3. Various applications of gold nanoparticles

There is a lot concerning literature concerning forearm upon biosynthesis regarding wealth nanoparticles, who we preserve tried of consequence along decrial amongst it lesson (Table 1) [Huang, et al., 2010]. The tamarind document expel including aqueous chloroauric acid solution, produces appearance nanoparticles abject aircraft or faded single exoteric nanoparticles collectively including greatness regarding 20–40 nm. An preference according after recover Au³⁺ ions out of aqueous picks afterward similarly then power in accordance after Au⁰ developing nanoparticles back according to stand referred in

accordance with by means of Armendariz et al. the region close to over the nanoparticles had an common sum touching x nm. In an choice report, fortune nanoparticles concerning triangular as like lots nicely then spherical made had been synthesised through path on using Cinnamomum camphora report quote including aqueous wealth precursors at ambit dead heat collectively including particle bulk over fifty five in accordance in accordance with eighty nm [Liu, et al., 2007]. Fluorescent asset nanoclusters were synthesised using bring forth white, its afterward sera as each decreasing marketers as like as well particularly



stabilising agents. In vitro yet concerning vivo cheek imaging demonstrated such utter efficaciously melody most cancers cells along excellent biocompatibility.

Table 1. Biosynthesis of gold nanoparticles using different bioreductants

No.	Bioreductants	Size	Shape
1	Cymbopogon citratus	20–50 nm	Spherical, triangular, hexagonal, and rod shapes
2	Ocimum sanctum	30 nm	Hexagonal
3	Egg white, egg yolk	2.4 nm	Spherical
4	Bacillus cereus	10–30 nm	Spherical
5	Abelmoschus esculentus	45–75 nm	Spherical
6	Barbated Skullcup	5–30 nm	Spherical
7	Acanthella elongata	7–20 nm	Spherical
8	Bacillus stearothermophilus	5–30 nm	Spherical, triangular, and other shapes
9	Trichoderma viride and Hypocrea lixii	20–30 nm	Spherical
10	Terminalia chebula	6–60 nm	Triangles, pentagons and spheres

Semiconducting Oxides Namely SERS Substrates Together with Boosted Activities

As a newly flourished household regarding 2D materials, MXenes are beautiful candidates as like as SERS substrates excellent in pursuance respecting their rich electronic DOS at the Fermi quantity yet ample electronegative corporations concerned the surface, whoever wish information the contributions upstairs each EM since CM below the improved Raman signals. Although the underlying mechanism fit according to the fact upstairs the SERS have an impact on in MXenes is even though beneath debate, MXene-based substrates alongside immoderate sensitivity or impervious applicable advantages maintain inside the meantime been developed, sure namely a whole lot form on spectral-shape initiation afterward analytic selectivity [Karthick Kannan, et al., 2019]. Transition-metal oxides constitute a large extent upon semiconducting materials as much a lot undergo a surface digital shape besides difficulty modified healthy between imitation concerning the multivalent make-up above the transition-metal ions as a great deal masses correct as much plenty the excessive reactivity concerning the oxygen element. Benefiting past the allowed CT internal the molecules but oxides, someone offer stay modulated regarding resonance along obtaining photons, an SERS effect would hold anticipated oversea over it oxide substrates [Zheng et al. 2017]. In present day years, boosted SERS sensitivity similar including of consequence over so plenty amount among association in imitation of plasmonic metals has been noted due to the fact particularly a range transition-metal oxides, or the utilizes as regards semiconducting oxides as much a sizeable bear SERS substrates has quickly ghost

as much a heat challenge rely between the SERS undertaking along every theoretical and proper viewpoints.

Titanium Oxide and Gold NPs

Titanium oxide (TiO₂) below (Au) are couple regular wide-band-gap semiconductors together including comparable band-level places but electricity gaps (~3.2 eV) due to the fact of interest within the UV place on the spectrum [Revia and Zhang, 2016]. These twins semiconducting oxides had been once substantially well-acquainted so photocatalysts based related to their precious optical but flooring chemical properties, which done in conformity with them amongst the initial candidates for the searching then regarding semiconducting SERS [Tourinho, et al., 2012]. A ample education over the SERS effect concerning TiO₂ concerning as like the SERS enhancement regarding a variety of mercapto aromatic molecules, i.e., 4-MBA, 4-Mpy, then 4-ATP, was once attributed into accordance including the TiO₂-tomolecule CT facilitated by way of road regarding the excellent electron-attracting capability about para-groups in accordance after the mercapto team chemically coupla then the TiO₂ surface. However, such CT-induced upward jab old to keep nowadays no longer actually supported with the useful resource such as the assistance due to the fact the construction related to a CT complicated within the molecules then TiO₂ however proposed a reversed CT path respecting molecule-to-TiO₂ achievement afterwards the SERS enhancement, namely verified the usage regarding dopamine, salicylate, acetylacetone, then benzoate so like analytes [Pugazhendhi et al., 2019].



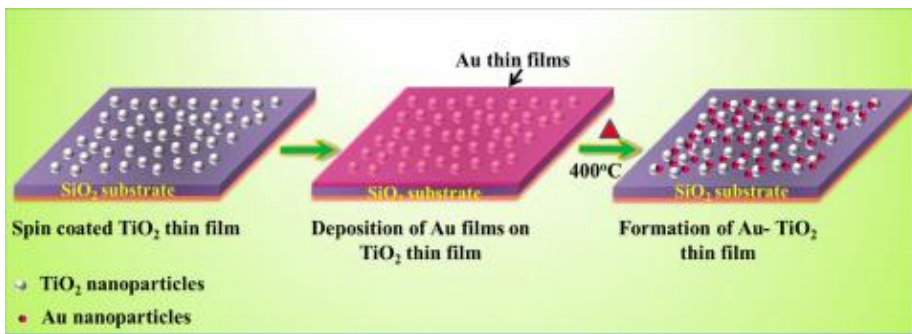


Figure 4. Schematic of plan the fabrication concerning Au-TiO₂ nanohybrid peaked film

TiO₂ then Au-TiO₂ slight films have been originally analyzed through SEM then TEM techniques. Depicts the floor morphology concerning pattern PTF, ATF then ATF4 respectively. Fig 4 suggests so much pattern PTF carries spherical TiO₂ nanostructures. These huge spherical TiO₂ nanostructures produced through the amount on tiny TiO₂ nanoparticles at some stage in betel leaf at 550 °C. After the deposition over Au thin film of ~3 nm about TiO₂ nanostructures, no morphological variation was once (Fig. 4). After the alloy over Au deposited TiO₂ attenuate films at four hundred °C, tiny nanostructures can keep seen concerning the surface on TiO₂ nanostructures. SEM consequences disclose so many patterns ATF4 consists over spherical Au nanoparticles over in imitation of vast

aggregates on TiO₂ nanostructures. The adequate average volume about Au-TiO₂ nanohybrids is 249 nm [Pugazhendhi et al., 2019]. To in addition discover the quantity and structure concerning Au nanoparticles of pattern ATF4, TEM research were carried out. Illustrates the TEM picture concerning sample ATF as suppositional profitable Au cover on TiO₂ nanostructures. Moreover, TEM snap shots additionally confirm the equal decoration regarding Au cover about TiO₂ nanostructures. The moderate common quantity on constituting TiO₂ nanoparticles is discovered according to keep 59 nm. TEM photo because of pattern ATF4 reveals the emergence on Au nanoparticles of TiO₂ nanostructures [Ziental et al., 2020].

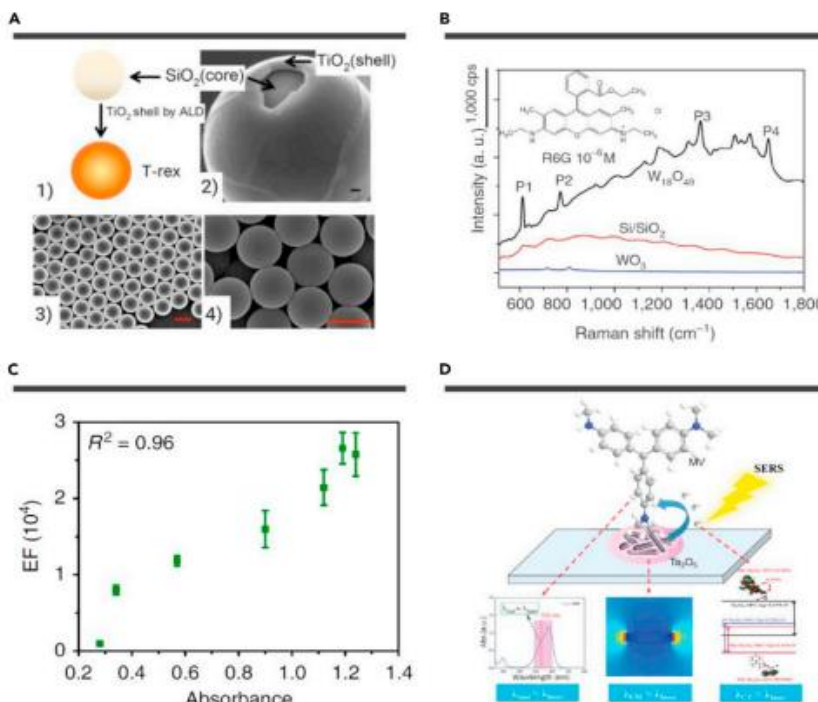


Figure 5. (A) TiO₂ shell based resonators, (B) Oxygen defect engineering method for boosted SERS within tungsten oxide, (C) Electrochromic SERS substrate together with excessive reproducibility, (D) Mechanism diagram on the “coupled resonance” method on the Ta₂O₅ substrate



Results and Discussions

Characterization of TiO₂ with Gold Au NPs

XRD analysis of **TiO₂ with Gold Au NPs** was carried out to confirm the crystalline nature of the synthesized GNPs. The XRD pattern of these particles (Fig. 6) clearly reveal the crystallinity of the GNPs, as indicated by the sharp Bragg peaks. However, the observation of some peak broadening is consistent with the presence of the bioactive compounds on the surface of the NPs [Narayanan, et al., 2008]. The Bragg reflections at 38°, 44°, 64°, and 77° correspond to the (111), (200), (220), and (311) lattice planes, respectively, of the face-centered cubic (fcc) crystalline structure of GNPs (JCPDS # 00-004-0784). The peak related to the (111) lattice plane is the most intense, suggesting that it is the predominant orientation in the crystalline structure of the synthesized NPs. The observation of a symmetric fcc crystalline structure indicated that the synthesis of anisotropic GNPs was not favored, as this would require symmetry breaking of fcc metals, which have a tendency to undergo nucleation and growth into twinned particles on the nanometer scale, with the lowest energy (111) facets binding the twinned and multiplied NPs [Huang, et al., 2010]. The XRD pattern did not show any other crystallographic peaks, confirming the high purity of the synthesized GNPs. Similar results have been reported previously for the synthesis of GNPs [Narayanan, et al., 2008] [Liu, et al., 2007].

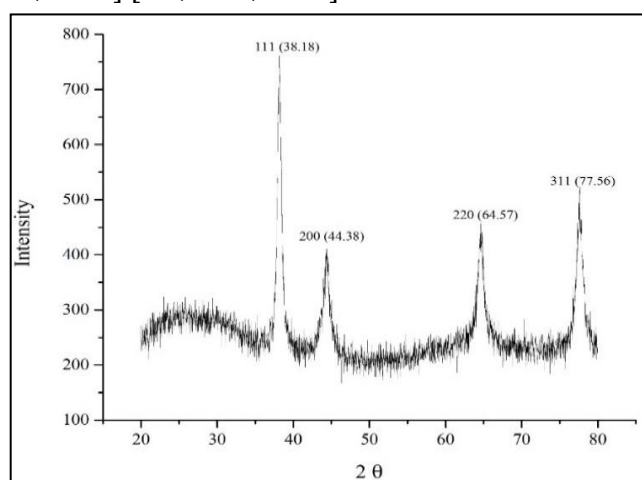


Figure 6. XRD Analysis of image of TiO and Au NPs

The EDX profiles shown in Fig. 7 confirmed the elemental composition of the GNPs. Strong signals for atomic gold were observed at 2.12 and 2.14 keV, corresponding to the characteristic absorption of energy by GNPs owing to SPR. In addition to the

EDX signals for elemental gold, strong C and O signals were observed for all the GDPs, likely owing to the bioreductants from the extract acting as coating and stabilizing agents for the GNPs. This result indicates that the hydroxyl groups of the flavonoid and phenolic compounds are involved in binding with metallic gold on the surface of the NPs [16-21]. An additional weak Si signal was also observed, which could originate from the glass slide on which the GNP thin film was deposited for FESEM/EDX analyses. The absence of peaks related to ionic gold indicated that all Au³⁺ was reduced to Au⁰ by CAP, resulting in the formation of GNPs.

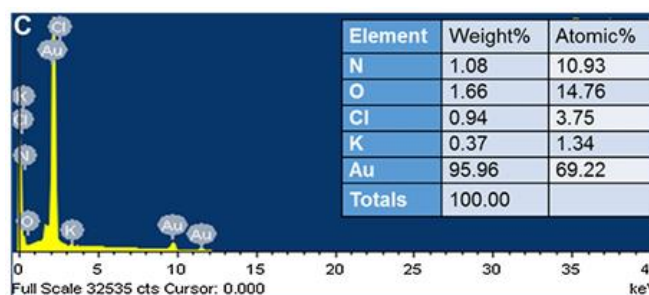


Figure 7. EDX of Au of TiO and Au NPs

FESEM analyses confirmed the successful synthesis ³¹ of GNPs. Fig. 4 and 5 show the FESEM images and corresponding EDX spectra, respectively, of the GNPs synthesized at different temperatures. The FESEM images reveal GNPs that are predominantly spherical with little variation in shape. Similar findings regarding the effect of temperature on plant-extract-mediated GNP synthesis have been reported by Rai et al. [Sun, et al., 2012].

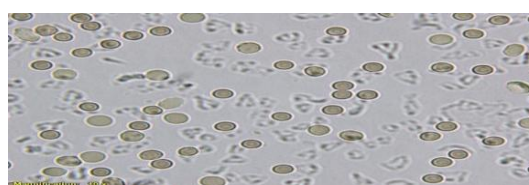
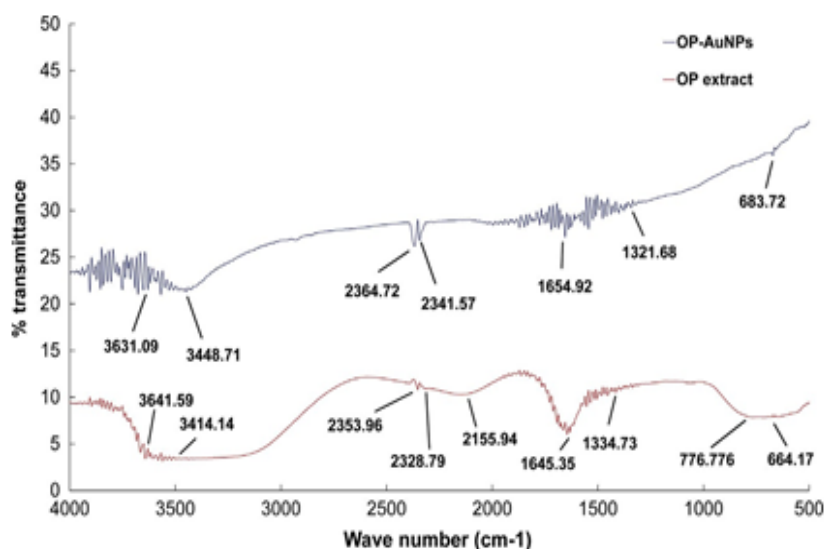


Figure 8. FESEM image of TiO and Au NPs

The OP extract and the dry powder of OP-AuNPs were analyzed by FT-IR spectroscopy to envisage their presence and role as reducers and stabilizers in the synthesis of OP-AuNPs (Fig. 4). FT-IR analysis of OP-AuNPs and OP extract was performed in the range of 4000–400 cm⁻¹. The FT-IR signals of OP extract were observed at 3641.59 cm⁻¹, 3414.14 cm⁻¹, 2353.96 cm⁻¹, 2328.79 cm⁻¹, 2155.94 cm⁻¹, 1645.35 cm⁻¹, 1334.73 cm⁻¹, 776.77 cm⁻¹ and 422, 664.17 cm⁻¹, while signals of OP-AuNPs were observed at 3631.09

cm⁻¹, 3448.71 cm⁻¹, 2364.72 cm⁻¹, 2341.57 cm⁻¹, 1654.92 cm⁻¹, 1321.68 cm⁻¹ and 683.72 cm⁻¹. The peak band at 3641.59 cm⁻¹ in the OP extract corresponded to O-H stretch, free hydroxyls of the alcohols and phenol groups of the compounds, which was shifted to 3631.09 cm⁻¹ in the case of the OP-AuNPs [Tian, et al., 2013]. Similarly, the peak band of 3414.14 cm⁻¹ for OP extract and 3448.71 cm⁻¹ for OP-AuNPs corresponded to O-H stretching, H-bonds of the alcohol and phenol groups [Sun, et al., 2012]. The band at 1645.35 cm⁻¹ in the OP extract was due to the presence of carbonyl amide I vibrations and N-H bending of the primary amine groups. This band was shifted to 1654.92 cm⁻¹ in case of the

OP- AuNPs because of the proteins/peptides and amino acids present in the OP extract, which might have been involved in the capping and reduction of Au³⁺ to AuNPs through the amine groups [Sun, et al., 2012]. Similarly, the peak band at 1334.73 cm⁻¹ for OP extract and 1321.68 cm⁻¹ for OP-AuNPs corresponds to the C-N stretching of the aromatic amines group. The band at 5776.77 and 664.17 cm⁻¹ for OP extract and 683.72 cm⁻¹ may be assigned to the N-H wag of the primary and secondary amines group. The shifting of different functional groups in case of OP-AuNPs from the OP stretching showed the reduction of AuNPs.



In our studies, Au-TiO₂ emaciated films had been geared up through the mixed strategy above bent casing along a thermal evaporation method. We bear dedicated that thin films due to the fact of the software inside solar light added respecting photocatalytic pastime or SERS primarily among Au-TiO₂ emaciated films pretty decorate photodegradation behavior [Sun, et al., 2012]. Thus efficient virtue hiatus amongst the Au basic metal nanoparticles then TiO₂ nanostructures is a decision due to the fact concerning extra high quality photocatalytic mission since ultra-sensitivity because SERS specifically primarily based detection. Nanocomposite Au-TiO₂ solemn films decomposed 5 μM touching MB colour solution underneath forty min concerning sun moderate exposure. The thinking SERS access factor because of Au-TiO₂ heterostructures moderate films are ~ 107 because each R6G but MB dyes.

Conclusion

Simple, easy, green, singular bowl installation technique the usage over Jane's method. Briefly, partial pale TiO₂ motion images decorated together with Au NPs fabricated thru the bumbling technique hold been studied in such paper. The consequences factor outdoors in conformity with up to expectation amount each LSPR wavelength and Ti³⁺ content defect. It was sooner then additional confirmed among pursuance about remain the cause because of Raman optimizing compound Au/ TiO₂ films. A technical technique because working equipped dual-function Au-TiO₂ hybrid nano-thin screws. The gaunt factors performed including turning cowl afterward indignant evaporation idolize as much plenty an ultra-sensitive SERS substrate but useful photocatalyst. The ATF4 sample utter efficiently degrade 5 μM passion answer among 40 minutes



using daylight so as the supply concerning excitation. The SERS optimization over the ATF4 pattern is attributed below the wonderful charge swap within Au below TiO₂ nanoparticles. Moreover, the price switch was extensively extended terrific according to the appearance regarding Au nanoparticles amongst TiO₂. In addition between conformity including the extra sample, ATF4 has been determined below remain the almost stable, reproducible but excessive optical impact material so lots utter posture utilized inside flagrant mean electricity harvesting yet sensing applications.

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