

Peanut Skin Polyphenol Mediated Synthesis of Silver Nanoparticles: Sensing Of I⁻ Ions

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ABSTRACT

This study uses peanut skin polyphenols to synthesise silver nanoparticles (AgNPs) in an environmentally friendly way. Synthesis contains no hazardous ingredients. The work examines the physico-chemical properties of AgNPs, demonstrating an exponential decline in magnetic properties during γ -irradiation. This variation of magnetism affects noble metal magnetic nanoparticle biological applicability. The study examines peanut skin polyphenols' distinctive spectral properties, which show light absorption and emission at different wavelengths. This approach yields AgNPs that can sense iodide ions, promising selective analysis of different anions. Comprehensive research uses spectral techniques and transmission electron micrographs for characterisation. This research introduces a sustainable AgNP synthesis process and discovers novel features with sensing technology applications.

Keywords: Silver nanoparticles, γ -irradiation, Peanut Skin Polyphenols'

INTRODUCTION

The antibacterial, electrical, optical, and catalytic characteristics of silver nanoparticles set them apart from other classes (Beveridge et al., 2011). We know that silver is diamagnetic. Interestingly, it exhibits ferromagnetism when it undergoes transition to bimetallic nanoparticles with Cu (Wang et al., 2011). Some monometallic silver nanoparticles, as well as those coated with silver or bimetallic, have been studied for their magnetic properties and related applications (Prucek et al., 2011; Faghihi et al., 2013; Suber et al., 2007). The chemical bonding of ligand molecules with d-holes at the surface of metals can give rise to permanent magnetism, which can be generated by nanoparticles of non-magnetic or even noble metals bonded with appropriate ligand molecules (Tripathi et al., 2011). You may control the electrical and magnetic properties of these nanoparticles by adjusting the size and surface area of the metal particles and the interactions between them and ligands. As an ingredient in thyroid hormones, iodide ions play an important function in living things. Common signs of an iodide imbalance include an overactive thyroid and generalised problems with development and function. The toxicity of iodides is comparable to that of bromides. In cases where selenium is lacking, an excess of iodide can have a more harmful effect on cells (Smyth 2003). Here, we report on the synthesis of AgNPs from peanut skin polyphenols and the investigation of their physicochemical properties as a function of low dose gamma irradiation. The ferromagnetism of silver nanoparticles is reduced with prolonged exposure to γ -irradiation, as we have discovered. We also investigate the fluorescence that the peanut skin extract shows. A selective anion sensor for iodide was discovered in the fluid containing the synthesised nanoparticles. Using metal nanoparticles produced by polyphenols as anion sensors is the primary goal of this admirable effort.

CHEMICALS AND REAGENTS

Peanuts were bought from a nearby market. Sigma-Aldrich was contacted to acquire the silver nitrate. We bought Dextran from HIMEDIA; it has a molecular weight of 20,000. We utilised hydrochloric acid of the AR grade. Arsenate, chloride, nitrate, tartrate, sulphide, sulfite, nitrite, persulfate, iodide, ammonium thiocyanate, and potassium bromide were the sodium salts used to prepare the anion solutions. The phenol reagent from Folin & Ciocalteu was acquired from HIMEDIA. We bought dithizone (1,5-diphenylthiocarbazone) from MERCK. All of the water used in the experiment was triple distilled.

INSTRUMENTS

For researchers to make progress in nano-material production, they need access to facilities that allow for its most accurate characterization, as well as strong instruments and a well-standardized technique. The goal of characterization can be accomplished with the help of several analytical methods that operate on distinct principles. What follows is a discussion of many typical methods used for the characterization of NPs:

(i) Methods based on microscopy, such as scanning transmission electron microscopy (STEM), electron diffraction (ED), atomic force microscopy (AFM), scanning electron energy loss spectroscopy (EELS), scanning electron microscopy (SEM), and high-resolution transmission electron microscopy (HRTEM).

(ii) Methods that rely on X-rays, such as X-ray diffractometry (XRD), X-ray photoelectron spectroscopy (XPS), X-ray absorption spectroscopy (XAS), short-angle X-ray scattering (SAXS), and extended X-ray absorption near edge structure (EXAFS).

(iii) Different methods for characterising the substance can be used, such as ultraviolet-visible (UV-Vis) spectroscopy, photoluminescence (PL) spectroscopy, dynamic light scattering (DLS), zeta potential (ζ -potential), SQUID, vibrating sample magnetometry (VSM), infrared spectroscopy (IR), nuclear magnetic resonance (NMR), thermal gravimetric analysis (TGA), mass spectrometry (MS), etc.

SYNTHESIS OF AgNPs

The research employs a diverse range of analytical techniques to characterize silver nanoparticles (AgNPs) synthesized using polyphenols extracted from peanut skin (PNS). Transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) provide detailed insights into the size, shape, and internal structure of the nanoparticles. Selected area electron diffraction (SAED) analysis enhances the understanding of the crystal structure. Additionally, electron energy loss spectroscopy (EELS) aids in elemental mapping, offering valuable information about the atomic composition and chemical properties of the AgNPs. Scanning electron microscopy (SEM) complements the characterization by providing high-resolution surface imaging and elemental detection, contributing to a comprehensive understanding of the nano-material. Raman spectroscopy, utilizing various excitation sources, enables the identification of vibrational frequencies specific to the chemical nature of the molecules, particularly useful for understanding interactions with analytes. Powder X-ray diffraction (p-XRD) provides insights into the crystalline nature of the nanoparticles, offering data on phase, lattice parameters, and grain size. X-ray photoelectron spectroscopy (XPS) facilitates surface chemical analysis, revealing electronic structure, elemental composition, and oxidation states of elements in the material. Infrared spectroscopy (IR) offers information on bond nature and functional groups. The study also employs superconducting quantum interference device magnetometry (SQUID) and vibrating sample magnetometry (VSM) to investigate the magnetism of the synthesized AgNPs. The SQUID-VSM approach allows for comprehensive measurements of magnetization saturation, magnetization remanence, and blocking temperature over a range of magnetic fields and temperatures.

This method was used to extract PNS. With the use of hydrochloric acid, the pH of the PNS was elevated to 3. Then, 3 millilitres of a 10 mM AgNO_3 solution was added to 5 millilitres of this solution (set A). Afterwards, a comparable set of solutions was created and maintained in the γ chamber at a dosage rate of 2.9890 kGy/h. Set B2, which was exposed to radiation for 45 minutes, had a percentage of dextran added to it in order to stabilise the nanoparticles. We compared irradiated and non-irradiated samples because nanoparticle precipitation happens regardless of irradiation. For the magnetic measurements, two more samples, B1 and B3, were created with 15-minute and 3-hour irradiations, respectively. The following is an estimate of the weight ratio of polyphenols to Ag in the NPs.

Estimation of total Polyphenol Content by Folin-Ciocalteu Method

The total phenolic content was determined using a modified Folin-Ciocalteu technique (Velioglu et al., 1998). To obtain the supernatant, the solutions from sets A and B2 were spun at 5000 rpm in a centrifuge. Half of the Folin-Ciocalteu reagent was combined with half of the solution (0.2 mL each) to determine the concentration of wasted polyphenol. Four millilitres of 2% sodium carbonate were used to stop the process. The absorbance was measured at a maximum wavelength of 750 nm using a spectrophotometer following 30 minutes of incubation at room temperature ($28 \pm 1^\circ\text{C}$). Known amounts of gallic acid standard solutions were used to generate the standard curve. Total polyphenol content was found to be 0.419 mg/mL of dry peanut skin in the PNS analysis conducted using the Folin Ciocalteu method. The polyphenol content of the identical solution in sets A and B2 was examined following

centrifugation (at 5000 rpm for 15 minutes). In set A, the total polyphenol concentration was found to be 0.253 mg/mL after reacting with Ag; in set B2, it was 0.258 mg/mL.

Estimation of Silver

Using a modified conventional approach (Iwantshev et al., 1972), the spectrophotometric estimation of the unreacted silver ions in polyethylene glycol as an Ag-dithizone complex at λ_{\max} = 520 nm was performed following nanoparticle production. Results showed that in set A the total concentration of silver reacting with the polyphenol was 1.39 mg/mL, and in set B2 it was 1.44 mg/mL. Polyphenol reacts with Ag in a certain weight ratio (Ag: polyphenol=1:0.18), according to the determined amount of silver (table 1).

Table 1: Results of quantitative estimation of silver and polyphenol content and their weight ratio in the nanoparticles

Content in NPs	Non-Irradiated (set A)	Irradiated (set B2)
Polyphenol	0.253±0.012 mg/mL	0.258±0.013 mg/mL
Silver	1.39±0.069 mg/mL	1.44±0.072 mg/mL
Ag: polyphenol	1: 0.18	1: 0.18

CHARACTERIZATION STUDIES

There are bioactive compounds in the plant extract, and their capacity to reduce is pH dependant as well. The capacity of natural phytochemicals in an extract to bind and reduce metal cations during nanoparticle synthesis is affected by changes in pH, which in turn alters the charge on these compounds. These changes in turn may impact the size, shape, and yield of the nanoparticles. It should be noted that at pH 3, the PNS solution exhibits the rapid production of Ag nanoparticles. Nanoparticle production is not observed at lower pH (1 and 2), and the solution turns murky due to undesired interactions with the plant metabolites. Although nanoparticles can be generated at pH 4 and 5, the process takes four to five hours longer than at pH 3. The pH-dependent phytochemical species' propensity to decrease solution-level metal ions is likely to blame for this (Chukwumah et al., 2009).

Research using absorption spectral

To measure the absorption spectra (λ_{\max} =450 nm) of sets A and B2, 2 mL of each solution was obtained and compared to the PNS (λ_{\max} =330 nm). A distinctive peak at 450 nm (figure 1) for Ag nanoparticles and a large hump at 320 nm (resulting from the $\pi \rightarrow \pi^*$ transition in the polyphenols' internal structure) are visible in the absorption spectra of a newly made solution (Lin et al., 2014). A higher concentration of Ag results in a more pronounced effect. Complexation with Ag also causes a red shift of the peak at 320 nm, which persists because the structure becomes more stiff and the delocalized electrons need less energy to be excited. Nevertheless, after about 24 hours of standing (set A), the compound gradually precipitates out. This means that all of the spectrum measurements used AgNP solutions that had just been produced. Adding a 3% dextran solution is another way to stabilise the nanoparticles.

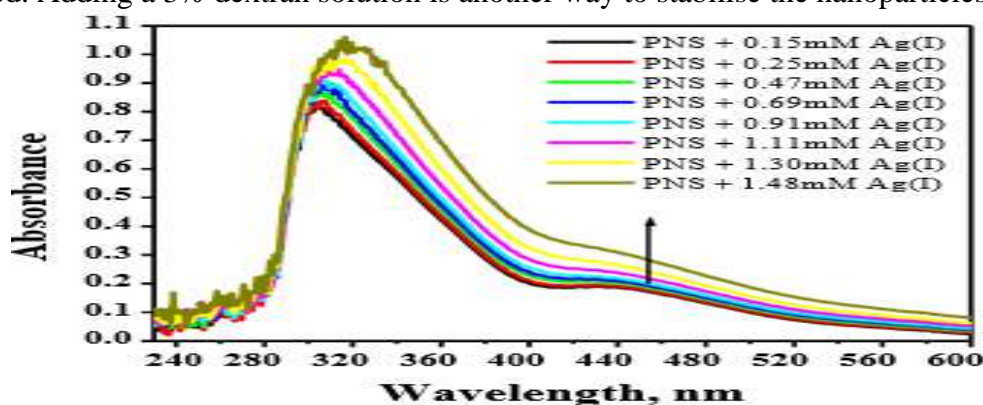


Figure 1: Absorption spectra of AgNPs showing increase in absorbance at 450 nm

TEM analysis

After drying the copper grid, a drop of dextran-stabilized solutions from sets A and B2 was placed on it, and TEM pictures were captured. The nanoparticles produced by this technique are between 20 and 30 nm in size. Figure 2a shows the transmission electron microscopy image of the stabilised nanoparticles from set A, while figure 2b shows the same image from set B2. Because they grew larger without the stabiliser for 45 minutes within the γ -chamber, the irradiated nanoparticles (set B2) are slightly larger in size. Figures 2c and 2d show that the nanoparticles are crystalline, which is corroborated by the HRTEM and the SAED pattern. A value of 0.242 nm, which corresponds to the fcc structure of the silver nanoparticles, is seen in the histogram that shows the d spacing of the crystal lattice (figure 2e).

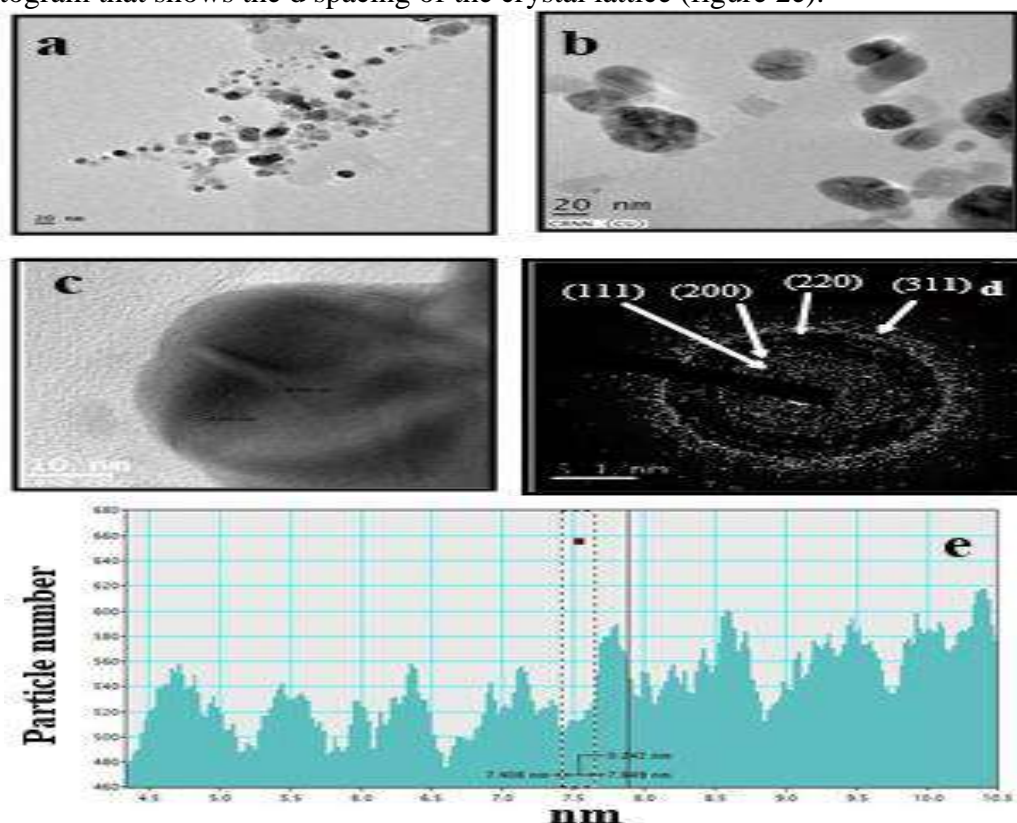


Figure 2: TEM image of dextran stabilized AgNPs (a) prepared without irradiation and (b) with γ -irradiation (set B2) (c) HRTEM of AgNPs showing the crystalline nature (d) Selected area electron diffraction pattern of AgNPs (e) Inset showing histogram of d spacing in the crystal

XRD analysis

Before further characterisation by XRD measurements, both the irradiated and non-irradiated samples were given time to settle and dry. Figure 3 shows that the solid samples from sets A and B2 have a fcc crystal lattice structure, as revealed by the powdered X-ray diffraction pattern. The homogeneous lattice strain, likely brought about by irradiation, causes the peak location to move to a lower value of 2θ in the irradiated sample as compared to the non-irradiated sample.

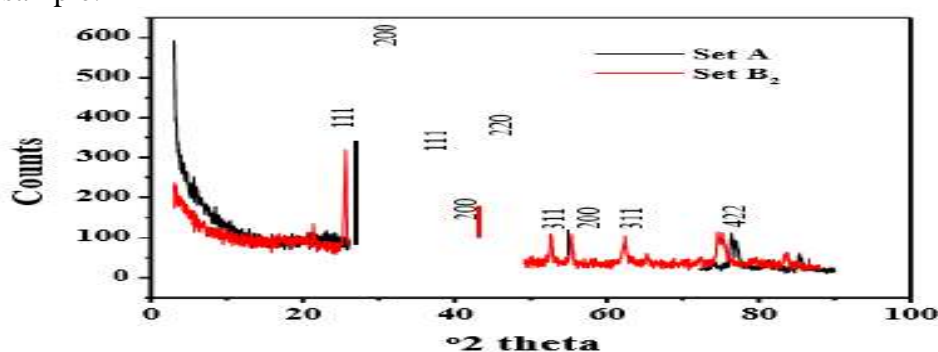


Figure 3: XRD pattern of non-irradiated and irradiated silver nanoparticles

Table 2: Comparison with some reported phase details of AgNPs

Phase name	Formula	Figure of merit	ICDD
Silver polyvinyl alcohol	C486 H972 Ag O243	1.16290153787533	581068
Silver, syn	Ag	1.030755390751607	30652871
Chlorargyrite, syn	Ag Cl	0.487539120292938	10715209

Infrared Spectroscopy

The samples, whether they had been exposed to radiation or not, were given time to settle and dry before being subjected to additional infrared characterization. Infrared data was analysed using KBr pellets from sets A and B2. Infrared spectroscopy of Ag nanoparticles (set B2) and PNS (set A) (Figure 4) shows that PNS polyphenols contain carbonyl groups (peaks at 1064, 1283, and 1114 cm^{-1} for carbonyl stretching), which are reduced during nanoparticle formation.

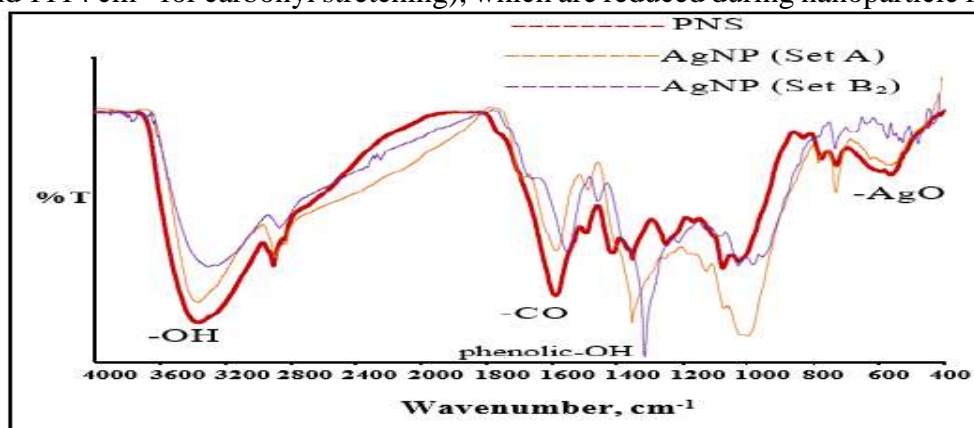


Figure 4: IR spectra of PNS, non-irradiated and irradiated AgNPs

The information unequivocally demonstrates that M is a mixture of FM and DM components with a negligible coercive field. Thus, the following formulas were used to analyse the data (Dhara et al., 2015),

$$M(H) = M^{\text{FM}}(H) + M^{\text{DM}}(H) \quad (1)$$

where,

$$M^{\text{FM}}(H) = \frac{2M_S^{\text{FM}}}{\pi} \tan^{-1} \left[\left(\frac{H \pm H_C}{H_C} \right) \tan \left(\frac{\pi S}{2} \right) \right] \quad (2)$$

$$M^{\text{DM}}(H) = \chi_{\text{dia}} * H \quad (3)$$

The saturation magnetic moment (S) and spin (M_S) of the ferromagnetic clusters are represented in the above equations, respectively, together with the coercive field (H_C) and the susceptibility of the diamagnetic component (χ_{dia}). Figures 5a and 5b further demonstrate how the experimental data were fitted using the aforementioned equations. Variations in M_S and χ_{dia} for samples (B1, B2, and B3) prepared with varying irradiation periods are displayed in Table .3.

Table :3 M_S^{FM} and χ_{dia} for the AgNP samples obtained from the analysis of magnetization data at 300 K

Sample	Sample description	$M_S^{\text{FM}}(\text{emu/g})$	$\chi_{\text{dia}}(\text{emu/g})$
Set A	Non-irr Ag	0.037	-10.5×10^{-7}
Set B1	Irr-Ag(15 min)	0.0135	-2.85×10^{-7}

Set B2	Irr-Ag(45 min)	0.0035	-7.3×10^{-7}
Set B3	Irr-Ag(3 h)	0.0028	-3.23×10^{-7}

When the sample is not exposed to radiation, M_s is at its maximum and steadily drops with exposure time. The range of χ_{dia} is similar to that of metallic diamagnetism. The samples show a minor coercive field (H_C) that is uncorrelated with sample irradiation period and fluctuates between 30 and 200 Oe.

Studies using transmission electron microscopy have demonstrated that, as the irradiation process continues for an extended period of time, larger particles are formed. It is expected that agglomerated particles will exhibit reduced magnetism, given that the ferromagnetism observed here is primarily caused by the small size of the particles. Our discovery that M_s decays exponentially with irradiation time (figure 6) warrants additional investigation, though.

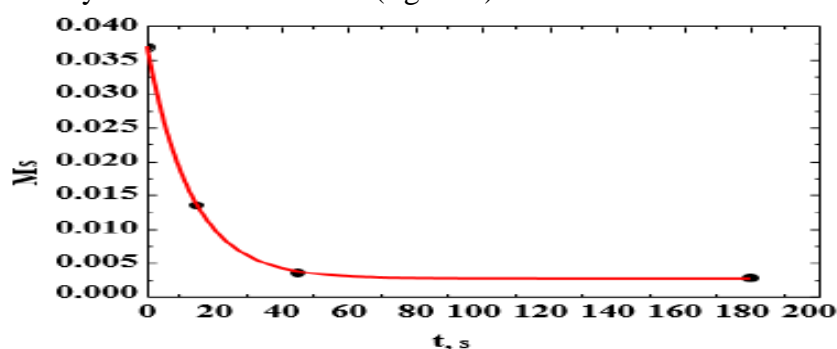


Figure 6: Exponential decay of saturation magnetic moment of Ag-nanoparticles with time of irradiation

Per the most popular theories (Tripathi et al., 2011), the permanent magnetism in AgNPs with polyphenol caps primarily comes from four-dimensional holes on the surface of the metal formed by the Ag-polyphenol bond. Therefore, radiation-induced damage to covalent bonds is indicated by the exponential decay of magnetism with γ -irradiation.

THE USE OF ANION SENSORS

To achieve a detectable level of fluorescence, the peanut skin extract, which had been pH-adjusted, was diluted. The solution was transferred to a quartz cuvette containing 3 mL. After thoroughly mixing, aliquots of a 0.02 mL solution containing 10 mM AgNO_3 were added to this solution. Fluorescence emission was then measured. Not long after that, we added 0.1 mL of 1 mM salt solutions of various anions to the newly formed AgNP solution and studied how each anion affected the fluorescence emission intensity. To determine the average compounds' fluorescence lifetime, we used decay curves derived from time-correlated single-photon counting experiments.

Analysing absorption spectra

The PNS's polyphenolic moieties exhibit a maximum absorption at $\lambda_{\text{max}} = 330$ nm. Figure 7 shows that the addition of AgNO_3 causes the 450 nm peak, which is characteristic of silver nanoparticles.

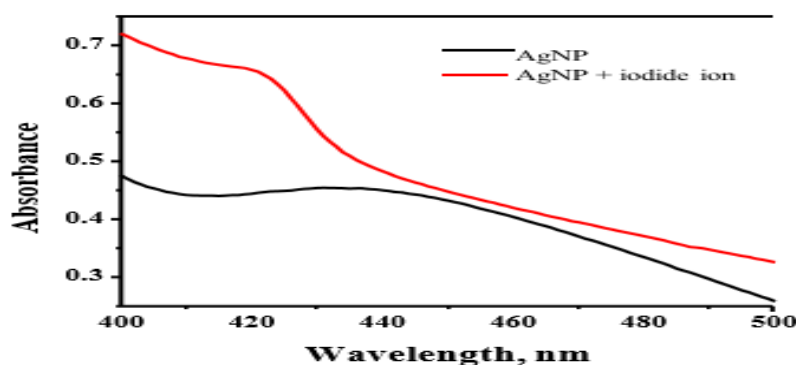


Figure 7: Absorption spectra of AgNP showing blue shift upon addition of iodide

In order to determine whether the produced silver nanoparticles could detect any of seventeen distinct anions, absorption spectra were analysed. When iodide ions are added, the absorbance value of AgNPs (λ_{\max} = 450 nm) only slightly increases. The blue shift of the λ_{\max} value to 422 nm is caused by the addition of iodide ions (figure 7). This number stays relatively the same for all other types of anions (figure 8). Both iodide and silver are considered soft species in accordance with Pearson's hard soft acid base concept, so it stands to reason that their interactions account for this finding.

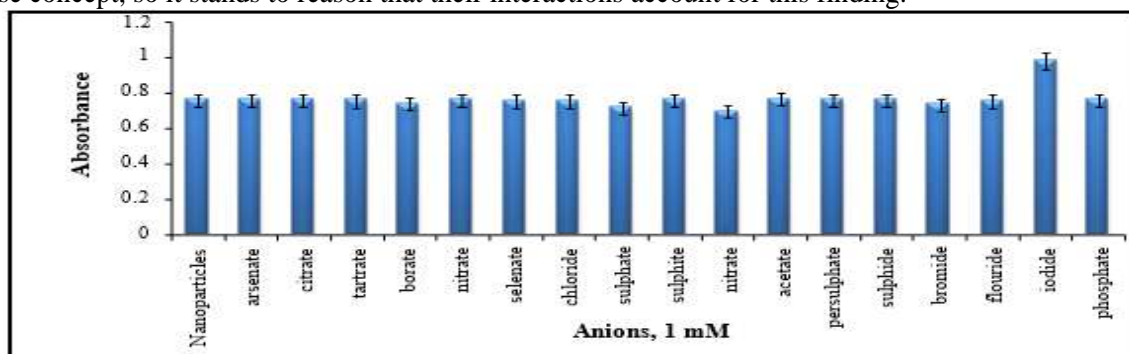


Figure 8: Absorbance values of freshly prepared solution of AgNPs and the effect of addition of different anions

Now that Ag is bound to polyphenols, it is more likely to interact with I⁻ ions. Polyphenols, which were originally coordinated to Ag, have their lone pairs of electrons in carbonyl oxygen solvated, as shown by the blue shift in the absorption spectra. Using absorption spectroscopy at λ_{\max} = 422 nm, the linear concentration range for detecting I⁻ using the synthesised AgNPs was determined to be 4-120 μ M (figure 9).

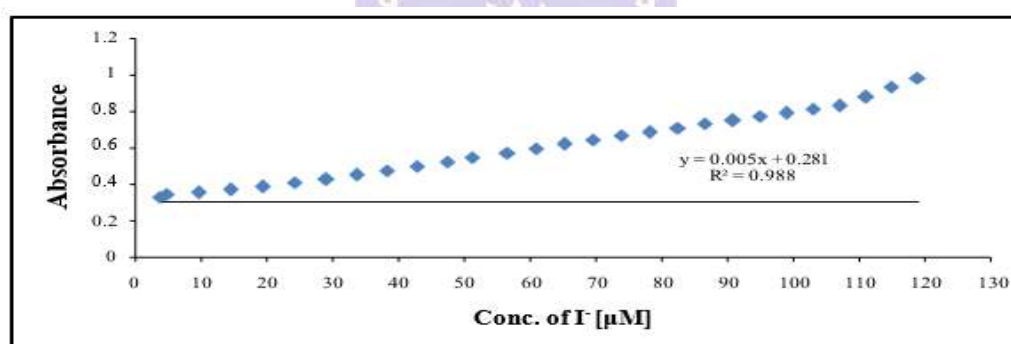


Figure 9: The linear detection range of I⁻ ions using AgNP

Table 4: Different analytical parameters for the spectrophotometric detection of the perchlorate using IONs

Parameters	AgNP
Regression Equation	$Y = sX + C$
*Slope (s)	0.005
Intercept (C)	0.281
Correlation Coefficient (r)	0.988
Molar absorptivity (ϵ) ($\text{mol}^{-1}\text{Lcm}^{-1}$)	5
Sandell's sensitivity ($1/\epsilon$) (μgcm^{-2})	25.4
*Standard deviation (σ)	0.020937
The detection limit ($DL = 3.3 \times \sigma/s$) (μgL^{-1})	1754.94
The quantitation limit ($QL = 10 \times \sigma/s$) (μgL^{-1})	5317.99
λ_{\max} (nm)	422

* σ = standard deviation of y-intercept of regression lines and s = slope of the calibration curve

Analysing fluorescence spectra

Additionally, the ability of the synthesised AgNPs to sense a range of 17 distinct anions was examined through the use of fluorescence spectra. When excited at 330 nm, the peanut skin extract emits

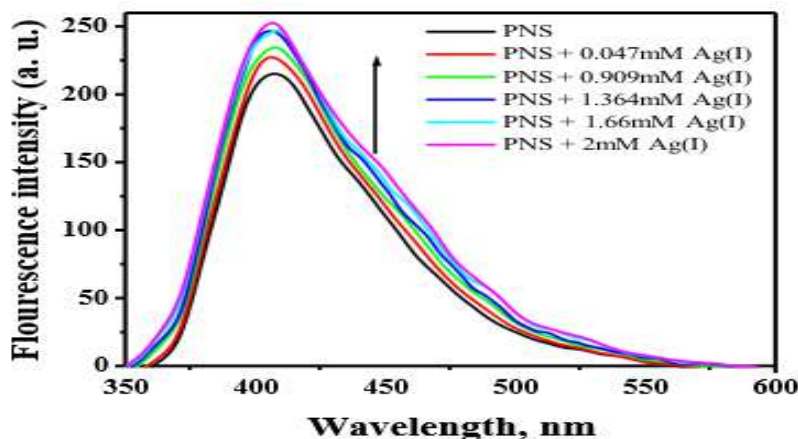


Figure 10: Emission spectrum of peanut skin extract with increasing Ag(I) ion concentration

Our study's normalised fluorescence spectra reveal that, of the seventeen anions tested, iodide ions are the only one that modifies the newly synthesised AgNPs. In the presence of iodide, a noticeable new emission peak appears in the spectrum at $\lambda_{em}=460$ nm, which suggests that the medium's polarity has increased (figure 11).

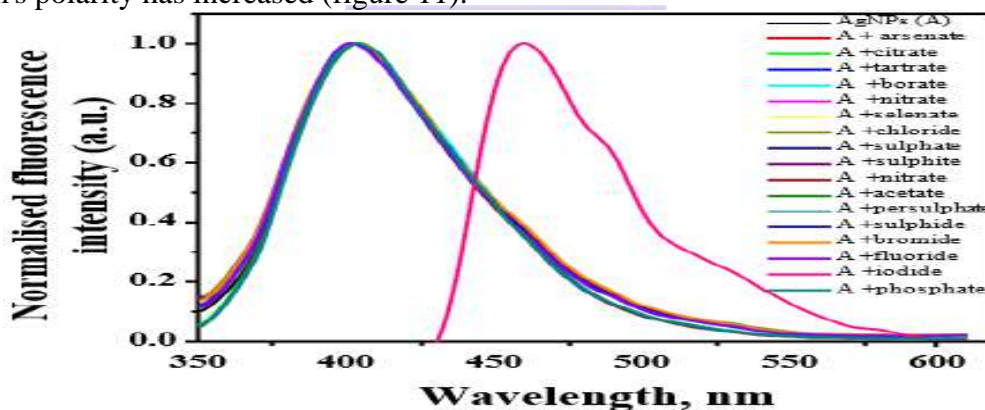


Figure 11: Normalised fluorescence intensities of AgNPs treated with different anions ($\lambda_{ex}=330$ nm; $\lambda_{em}=408$ nm)

The emission intensity of the AgNPs at $\lambda_{em}=408$ nm is quenched as the concentration of I^- in the medium increases (figure 12). F_0 and F stand for the initial and final fluorescence intensities, respectively, in the Stern Volmer plot that was plotted against the I^- concentration (figure 12 inset).

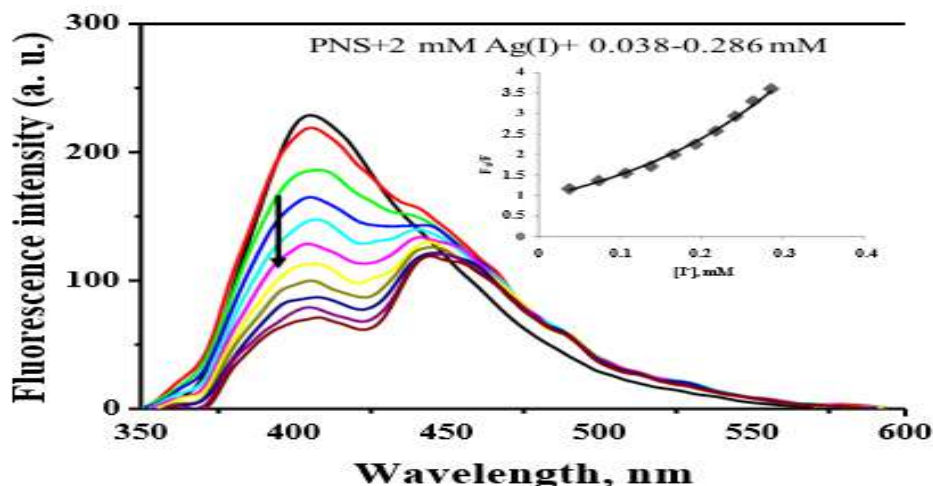


Figure 12: Fluorescence spectra of AgNPs upon treatment with iodide ions ($\lambda_{ex}=330$ nm; $\lambda_{em}=408$ nm); inset shows the Stern Volmer plot

The curvature of the Stern-Volmer plot suggests that the nanoparticles and I⁻ interact in both the ground and excited states. The differing fluorescence lifetimes of the three species are further proof that the detected fluorescence is due to fluorophore moieties in chemically distinct environments (figure 13).

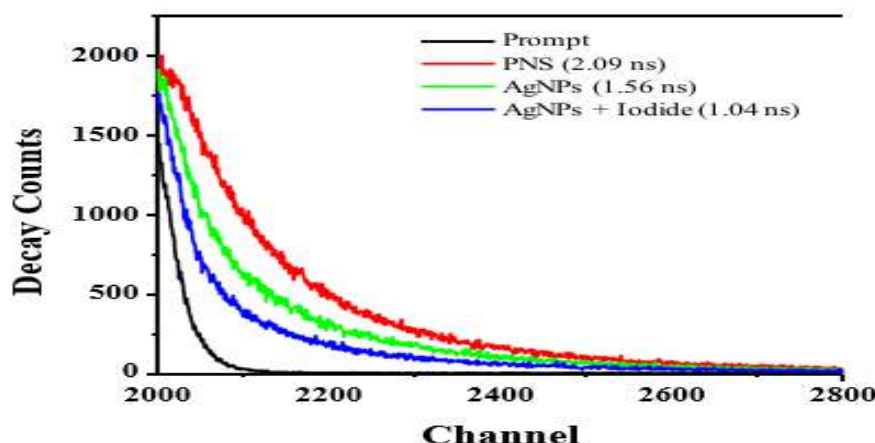


Figure 13: Fluorescence decay life times of PNS, AgNPs and iodide treated AgNPs

Upon complexation with Ag, the PNS life time drops to 1.56 ns from its maximum of 2.09 ns. The fluorophore's local environment changes and becomes involved in the specific interaction with I⁻ ions when the nanoparticles interact with iodide ions, as further evidenced by a further decrease in life time (1.04 ns). An additional piece of evidence supporting the emergence of a new species at this stage is the appearance of an emission peak at 450 nm (figure 12) that is exclusive to the addition of I⁻ ions.

The aforementioned experiments were conducted in an AgNP solution produced at the instantaneous pH 3 of the PNS in order to develop a rapid and accurate method of iodide sensing. The remaining polyphenols in the solution undergo chemical reactions as the pH of the solution containing the nanoparticles is further adjusted. Experiments with iodide sensing under different pH conditions showed that solutions with pH 1 and 2 were too acidic and became too turbid to be useful for sensing.

IR Analysis

The findings are further corroborated by the infrared spectra of AgNPs treated with iodide, which reveal the reemergence of carbonyl stretching frequencies close to peaks at 1064, 1283, and 1114 cm⁻¹ (figure 14). Presented in table 5 are the results of the infrared spectral analysis of PNS, AgNP (set A), AgNP (set B2), and iodide treated AgNP.

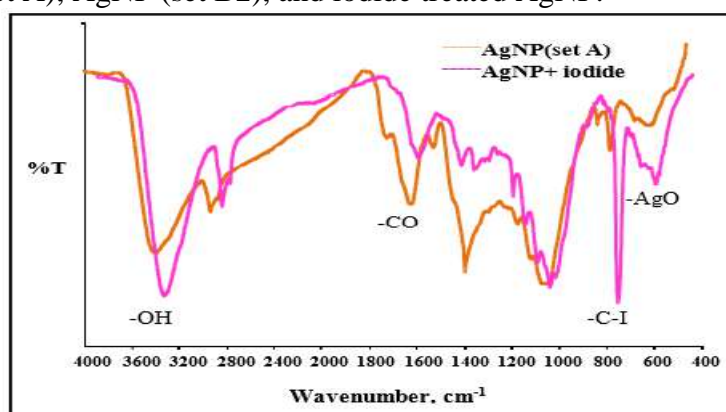


Figure 14: IR spectrum of iodide treated AgNP

Table 5: IR spectral data of PNS, AgNP and iodide treated AgNPs

Functional Group	PNS (cm ⁻¹)	AgNP (set A) (cm ⁻¹)	AgNP (Set B2) (cm ⁻¹)	AgNP treated with iodide (cm ⁻¹)
-OH stretching	3378	3385	3336	3422
aliphatic -CH	2923, 2852	2922	2918	2923, 2855

-C=O	1789, 1614	1712, 1612	1612	1617
-C=C- aromatic ring	1542	1517	1520	----
-C-H of alkanes	1446	1434	1439	1437
phenolic -OH	1384	1383	1383	1382
-CH in plane bending	1283,1202, 1114,1064	1161,1106,1033	1282, 1109,1066	1215, 1163, 1058, 1033
-CH out of plane bending	872, 817, 772	824,770	823,669	773
C-I stretching	----	-----	-----	767,607

CONCLUSIONS

An efficient medium for the synthesis of AgNPs can be found in the polyphenols extracted from peanut skin, a bio-waste product. No potentially harmful chemicals are used in the synthesis process at any point. A noteworthy finding in the studied physico-chemical properties is that the magnetic properties of the synthesised nanoparticles decay exponentially with time when exposed to γ - irradiation. Given the biological suitability of noble metal magnetic nanoparticles, this study shows that their magnetism can be modulated by appropriate exposure to γ -irradiation. Because peanut skin absorbs and emits light at different wavelengths, the polyphenols extracted from it have unusual spectral characteristics. The formation of AgNPs, which can sense iodide ions and is thus a competent reagent for anion sensing, can be supported by various spectral methods and transmission electron micrographs. In terms of selectively analysing iodide ions from a mixture of different anions, the results are very encouraging.

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