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# Synthesis and characterization of silver-based semiconductors towards the ultrasensitive detection of environmental pollutants using surface-enhanced Raman Spectroscopy


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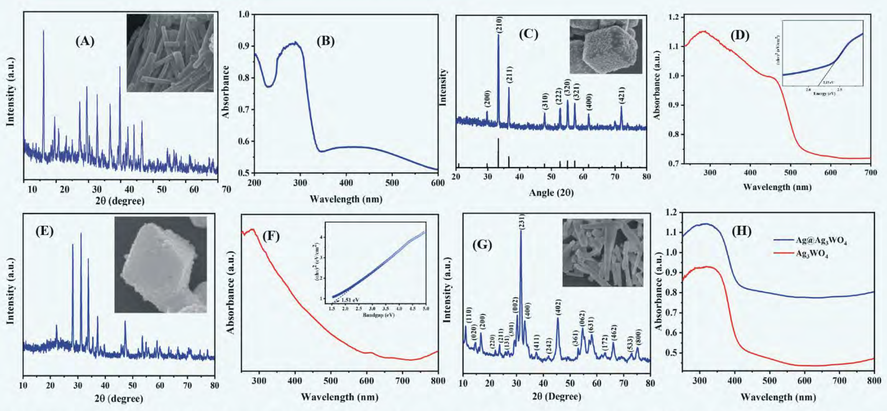
## Abstract

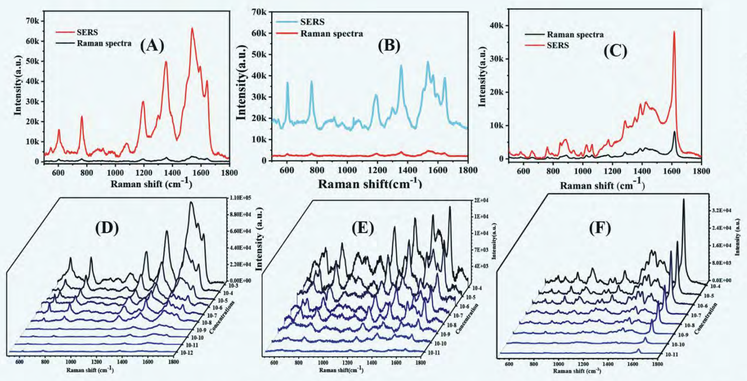

Surface-enhanced Raman (SERS) spectroscopy has emerged as an ultrasensitive, non-invasive and efficient fast-developing spectroanalytical technique. Therefore, exploration of new materials as high-performance SERS substrates is crucial for advanced development in SERS applications. Silver-based semiconductors have high chemical stability, and catalytic properties, and are used in various fields. This thesis aims to synthesis new silver-based semiconductor materials and explores their SERS activity for the detection of environmental pollutants. The silver-based metal-organic framework (Ag-BTC) and silver salts of oxyacids ($Ag_3PO_4$, $Ag_2SO_3$, $Ag_2WO_4$) were synthesized through the simple precipitation method. All the as-synthesized substrates exhibited a high SERS sensitivity, lowest detection limit and reusability for the SERS detection. The remarkable SERS sensitivity was achieved by the charge-transfer complex formation between substrate and analyte. The Ag-based semiconductors displayed great potential as SERS substrates for rapid detection of various environmental pollutants.


## Characterisation




XRD patterns and UV-DRS spectra of (A-B) Ag-BTC, (C-D) $Ag_3PO_4$, (E-F) $Ag_2SO_3$, and (G-H) Ag/$Ag_2WO_4$, respectively.

## Results

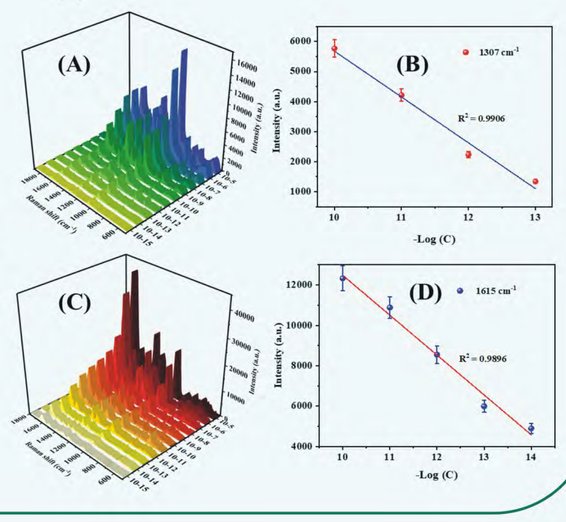


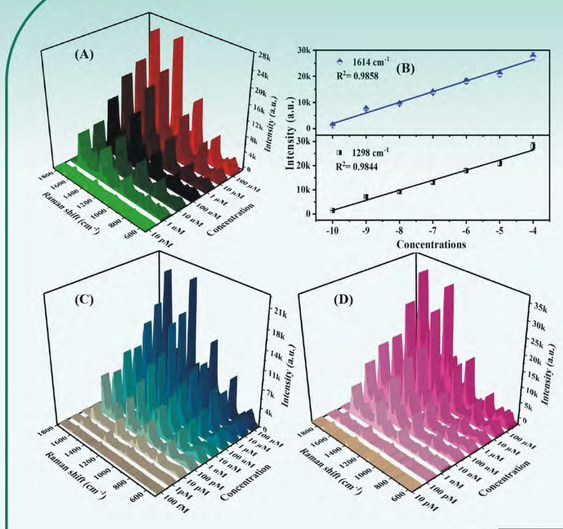

Comparison spectra of SERS and Raman absorbed on Ag-BTC for (A) Rhodamine 6G (R6G), (B) Rhodamine B (RB) and (C) Methylene Blue (MB) dyes; SERS spectra of different concentration of (D) R6G, (E) RB and (F) MB.

The analytical enhancement factor (*AEF*) values were calculated using the following equation:

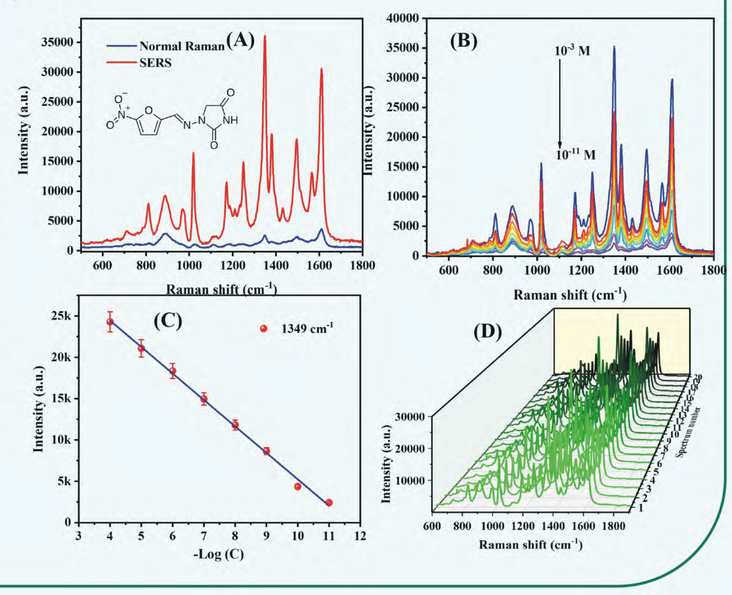
$$AEF = \frac{I_{SERS} \times C_0}{I_0 \times C_{SERS}}$$

SERS spectra of different concentration ($10^{-5}$ M to $10^{-15}$ M) of (A) $Hg^{2+}$ and (C) $Pb^{2+}$; Calibration plot for Raman intensity peaks at 1307 $cm^{-1}$ and 1615 $cm^{-1}$ of (B) $Hg^{2+}$ and (D) $Pb^{2+}$, respectively.




(A)SERS spectra of different concentration of 4-Chlorophenol (4CP) and its (B) calibration plot for Raman intensity peaks at 1298 and 1614 $cm^{-1}$; SERS spectra of different concentration (C) Sunset Yellow(SY), and (D) Methylene Blue (MB) absorbed on $Ag_2SO_3$.




(A) Comparison spectra of SERS and normal Raman of Nitrofurantoin(NFT) absorbed on Ag/$Ag_2WO_4$; (B) SERS spectra of different concentration ($10^{-3}$ M to $10^{-11}$ M) of NFT; Calibration plot for Raman intensity peaks at 1349 $cm^{-1}$ and (D) SERS spectra of NFT ($10^{-8}$ M) obtained from 20 random locations.

## Conclusion

- All the silver based semiconductor materials were fabricated by a simple co-precipitation method and investigated for the SERS activity.
- The characterization studies showed that the Ag particles were decorated uniformly on surface creating a synergistic effect due the chemical and electromagnetic mechanism.
- The prominent peaks were observable even concentration down to $10^{-11}$ M-$10^{-15}$ M indicating an excellent sensitivity.
- The AEF value for the main peaks was as around ~$10^6$-$10^9$, providing an improved SERS sensitivity. Furthermore, the substrates also exhibited excellent uniformity, stability and reusability.

## Publications


- *S Kamal*, TCK Yang, Silver enriched silver phosphate microcubes as an efficient recyclable SERS substrate for the detection of heavy metal ions. Journal of Colloid and Interface Science 605 (2022) 173–181.
- *S Kamal*, TCK Yang, A silver trimesate organic framework as an ultrasensitive surface-enhanced Raman scattering substrate for detection of various organic pollutants. Microchemical Journal 163 (2021), 105896.
- *S Kamal*, GT Pan, S Chong, TCK Yang, Ultrasonically induced sulfur-doped carbon nitride/cobalt ferrite nanocomposite for efficient sonocatalytic removal of organic dyes, Processes 8 (1), 104.
- *S Kamal*, S Balu, S Palanisamy, K Uma, V Velusamy, TCK Yang, Synthesis of boron doped $C_3N_4$/$NiFe_2O_4$ nanocomposite: An enhanced visible light photocatalyst for the degradation of methylene blue, Results in Physics 12, 1238-1244.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