

Supplementary Information

Polyethyleneimine Assisted Formation of Ag-SiO₂ Hybrid Microspheres for H₂O₂ Sensing and SERS Applications

Swati Mehta^{1,2}, Jitendra Bahadur^{1,2*}, Debasis Sen^{1,2}, Divya Nechiyl³, H. Bhatt^{4,2}, Naveen Kumar⁵, Jyoti Prakash^{2,3}

¹Solid State Physics Division, Bhabha Atomic Research Centre, Mumbai, 400085, India

²Homi Bhabha National Institute, Mumbai, 400094, India

³Materials Group, Bhabha Atomic Research Centre, Mumbai, 400085, India

⁴High Pressure and Synchrotron Radiation Physics Division, Bhabha Atomic Research Centre, Mumbai, 400085, India

⁵Atomic and Molecular Physics Division, Bhabha Atomic Research Centre, Mumbai, 400085, India

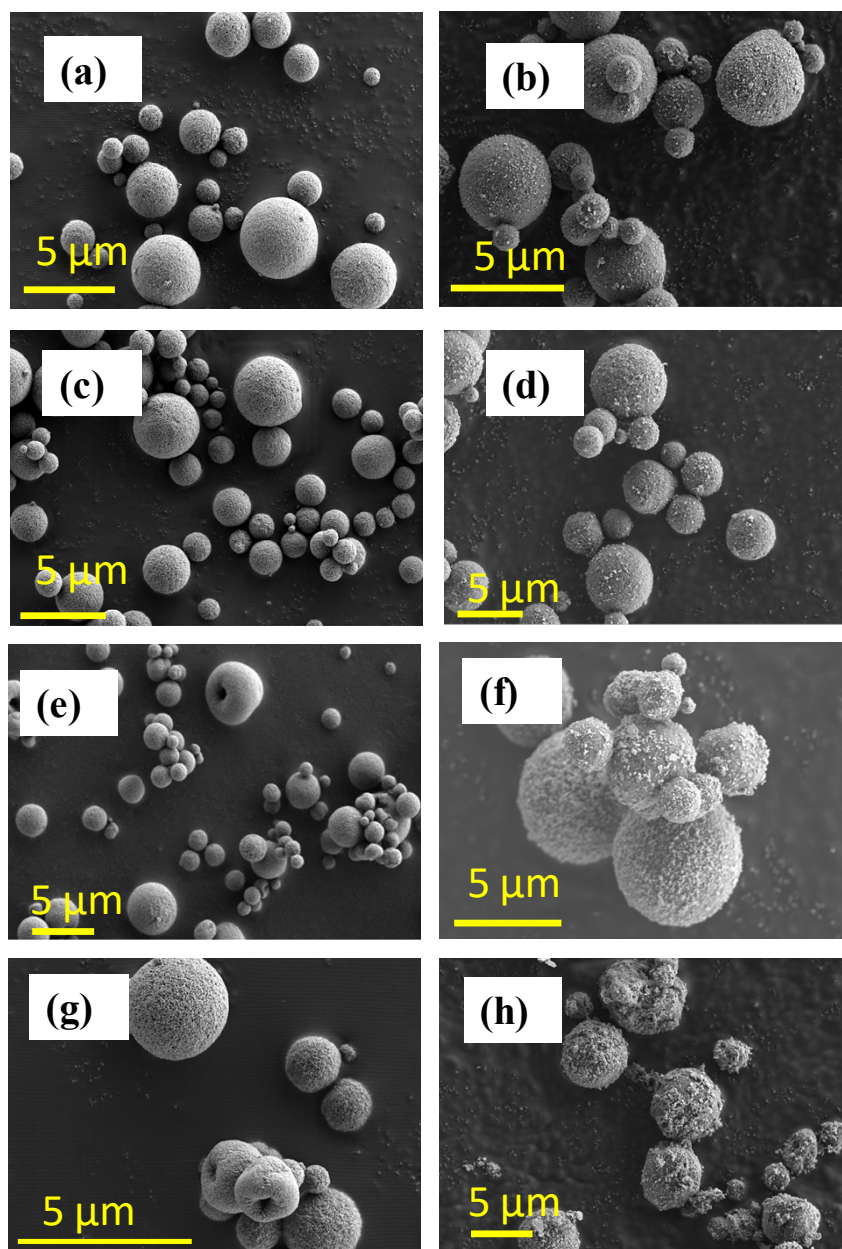


Figure S1: FESEM micrographs of silica-PEI microspheres and Ag-SiO₂ microspheres (a) Si-p5; (b) Si-p5-Ag (c) Si-p11; (d) Si-p11-Ag; (e) Si-p20; (f) Si-p20-Ag; (g) Si-p33; (h) Si-p33-Ag.

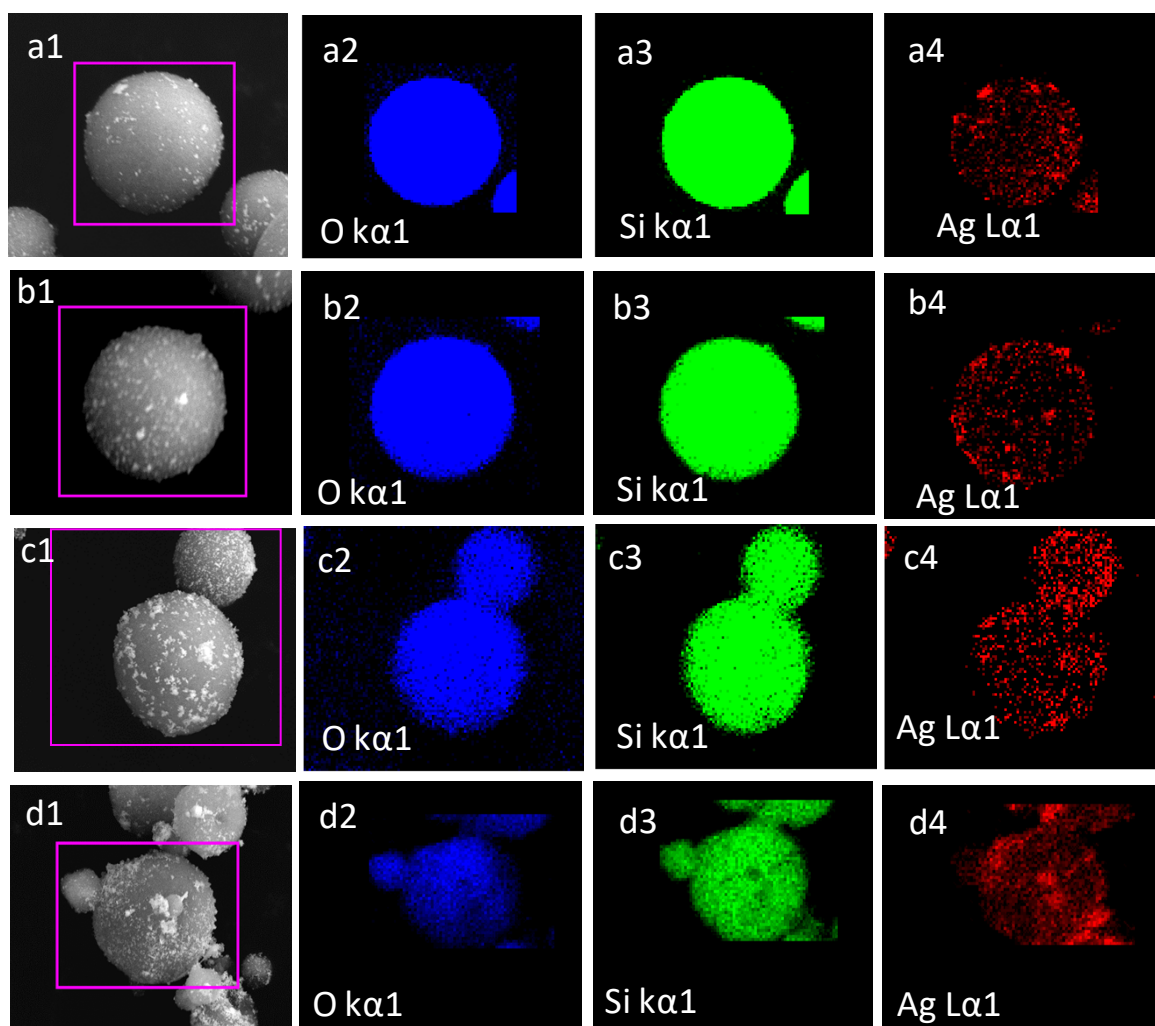


Figure S2: Energy Dispersive X-ray elemental mapping of Ag-SiO₂ microspheres obtained for (a) 5 wt% (b) 11 wt% (c) 20 wt% (d) 33 wt% PEI loading

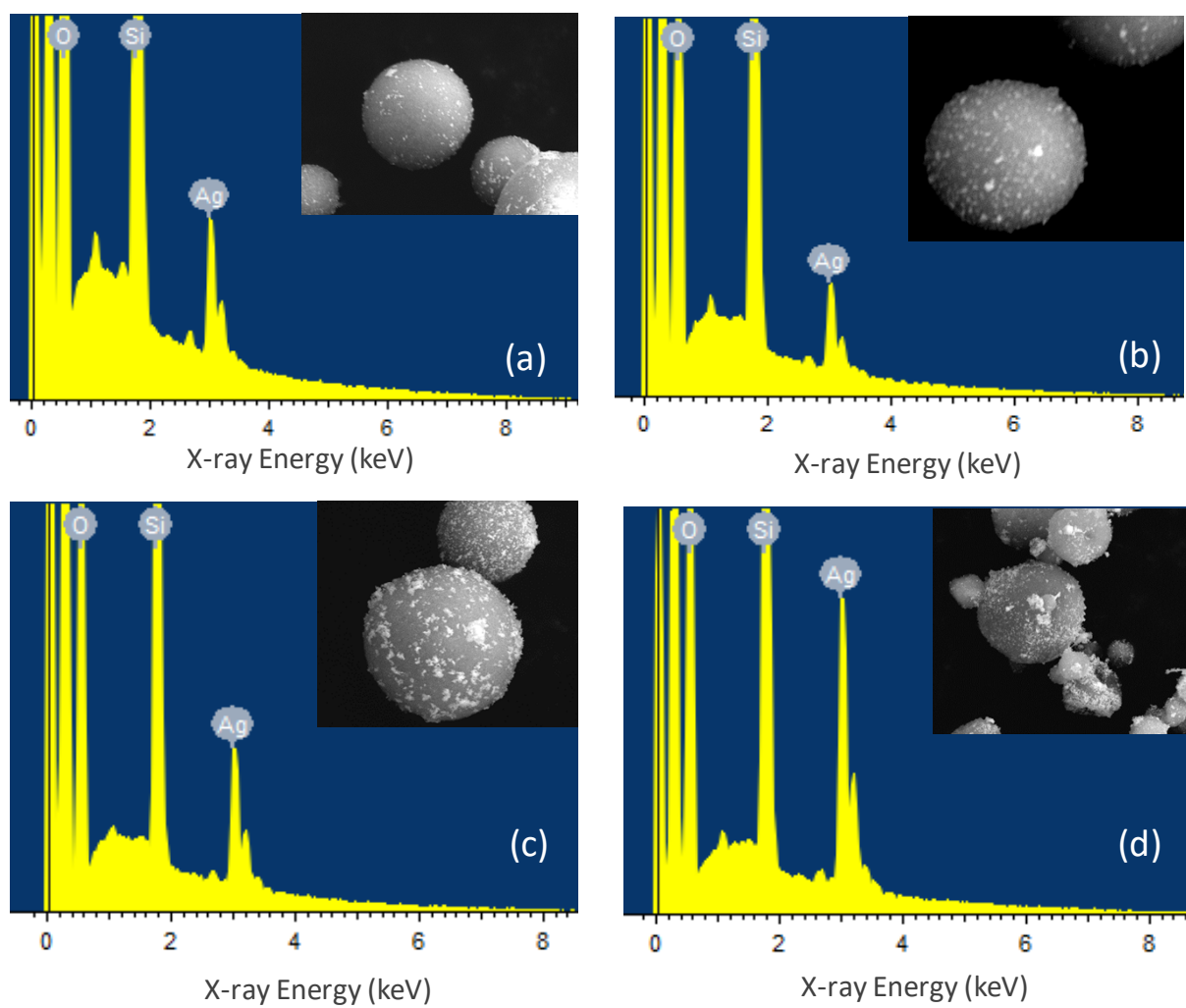


Figure S3: Energy Dispersive X-ray (EDX) spectra of Ag-SiO₂ microspheres obtained for (a) 5 wt% (b) 11 wt% (c) 20wt% (d) 33 wt% PEI loading

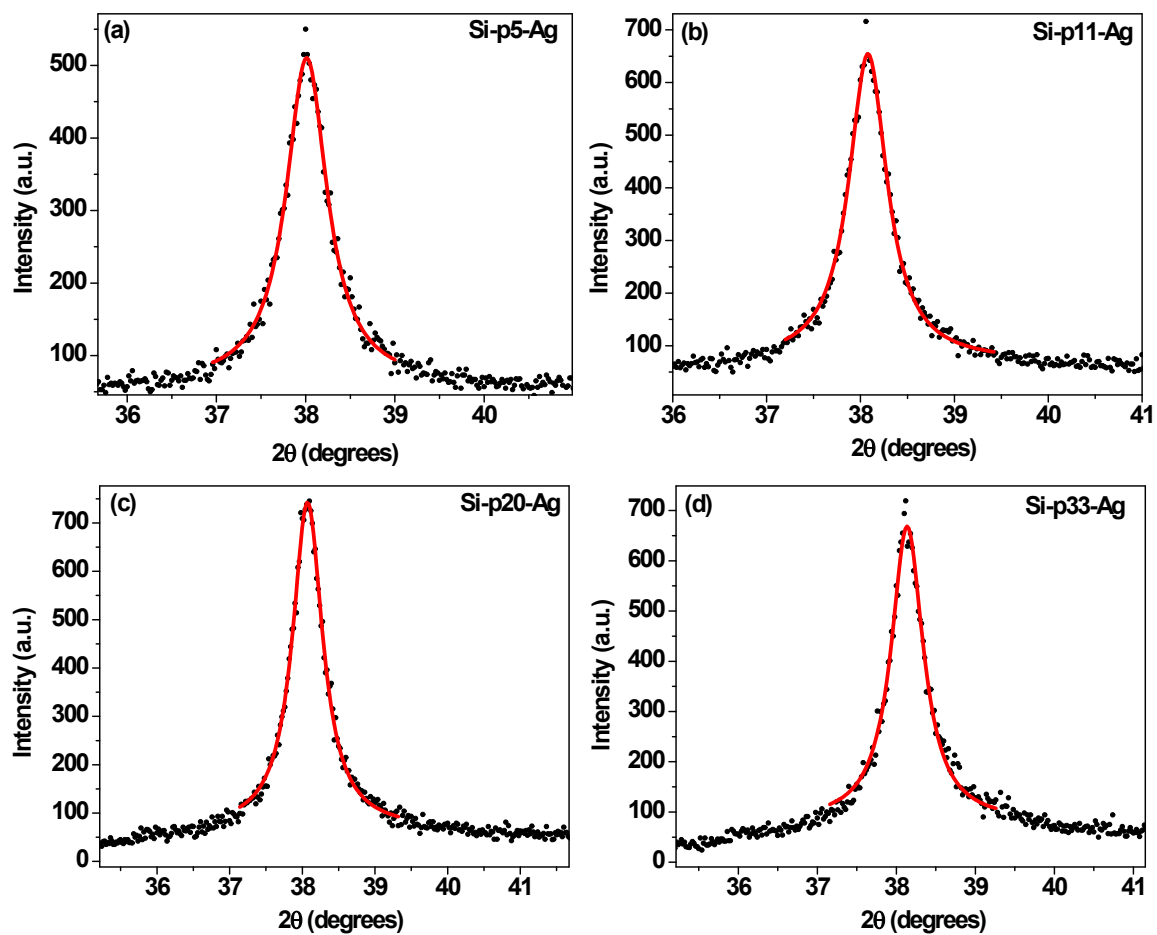


Figure S4: Lorentzian fit of the (111) peak of the XRD data for the Ag-SiO₂ microspheres.

Figure S5: UV-Visible spectrum of Ag-SiO₂ microspheres composite dispersed in water

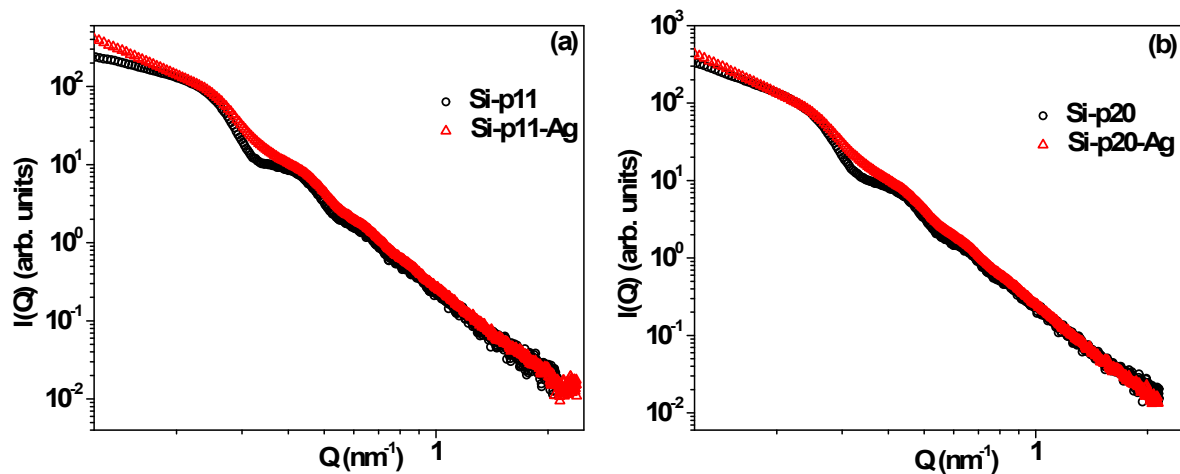


Figure S6: Comparison of SAXS profiles of silica-PEI and Ag-SiO₂ microspheres for (a) 11 wt% (b) 20 wt% PEI loading. Hollow circles (black) represent the SAXS data of pristine silica-PEI microspheres while profiles shown with hollow triangles (red) indicate SAXS data of Ag-SiO₂ microspheres.

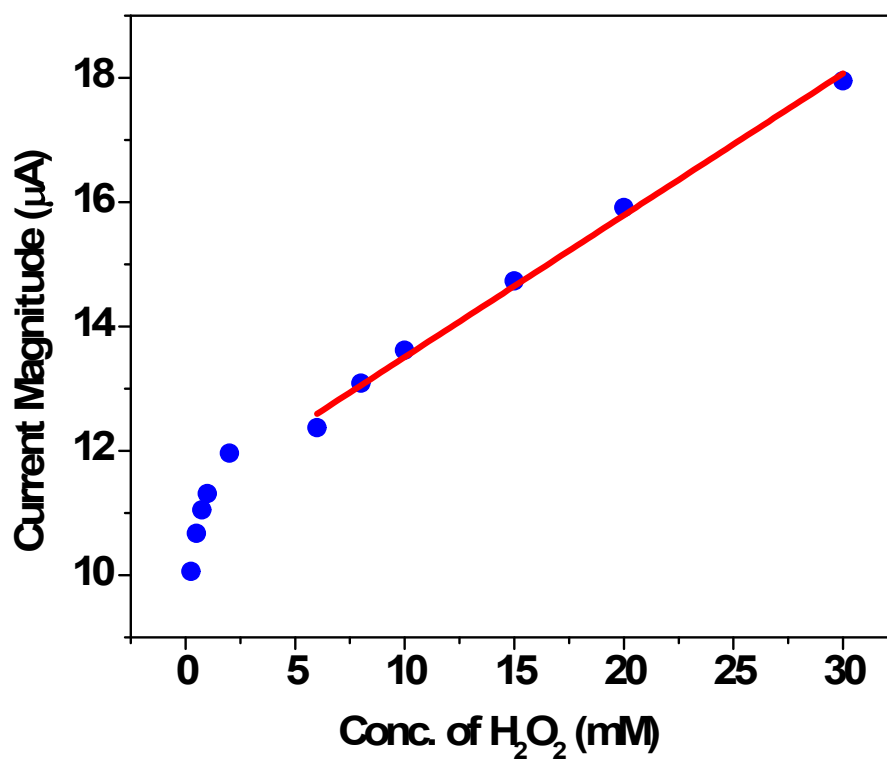


Figure S7: Variation in the magnitude of reduction current peak with H_2O_2 concentration. Solid line represents the fitting of the linear range of the variation in current versus concentration of H_2O_2 .

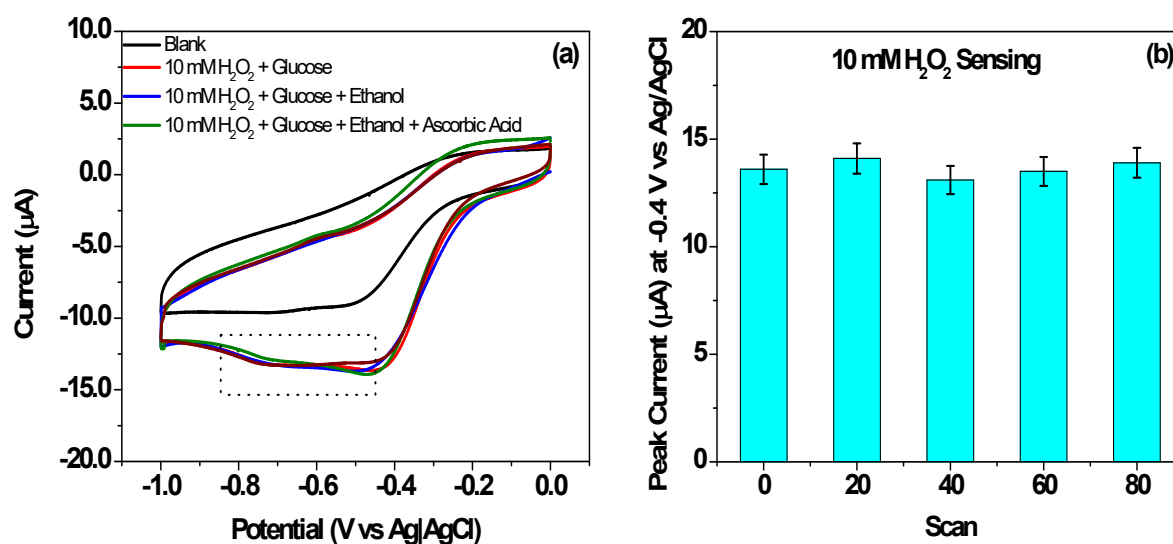


Figure S8: (a) CV measurements for the electrochemical detection of 10 mM H_2O_2 in the presence of various interfering agents (glucose, ethanol, ascorbic acid). (b) Current corresponding to the reduction peak potential at -0.4 V vs Ag|AgCl with varying CV scans.

Table S1: Comparison of Ag nanoparticles-based sensor for electrochemical sensing of H_2O_2

	Reductant source	Analyte Detected	LOD	Reference
Silver Nanoparticles	Polyethyleneimine	H_2O_2	1.08 mM	This Study
	Lavender leaf extract	H_2O_2	-----	1
	Dextrose	H_2O_2	-----	2
	Locust Bean Gum Polysaccharide	H_2O_2	-----	3
	Starch	H_2O_2	0.9 μM	4
	Euphorbia hirta (AEE) leaf extract	H_2O_2	10^{-7} M	5

	Agar & Ascorbic acid	H ₂ O ₂	0.03 μM	6
	Tea Extract	H ₂ O ₂	0.73 μM	7
	Isoimperatorin	H ₂ O ₂	0.036 μM	8

Figure S8: *Schematic for the instrumentation of Raman scattering measurements carried out on Ag-SiO₂ microspheres.*

Table S2: Comparative study of the distinct support materials for Ag nanoparticles used as SERS substrates

Support Material	Probe Molecule	LOD	Reference
Ag@SiO ₂ Microsphere	Rhodamine 6G (R6G)	10 ⁻⁶ M	This study
Ag@ SiO ₂	Prostate Specific Antigen (PSA)	0.11 pg/mL	9
PDMS + lotus leaves	Triazophos	10 ⁻⁸ M	10
Adhesive tape	Triazophos	25 ng/cm ²	11
Cotton Swabs	Thiabendazole	1 ng/cm ²	12
PDMS	Thiram	10 ⁻⁵ M	13
PDMS+PS microsphere	Thiram	10 ⁻¹⁰ M	14

References:

1. B. Kumar, K. Smita and L. Cumbal, *Nanotechnology Reviews*, 2016, **5**, 521-528.
2. S. Mohan, O. S. Oluwafemi, S. C. George, V. Jayachandran, F. B. Lewu, S. P. Songca, N. Kalarikkal and S. Thomas, *Carbohydrate polymers*, 2014, **106**, 469-474.
3. C. K. Tagad, S. R. Dugasani, R. Aiyer, S. Park, A. Kulkarni and S. Sabharwal, *Sensors and Actuators B: Chemical*, 2013, **183**, 144-149.
4. P. Vasileva, B. Donkova, I. Karadjova and C. Dushkin, *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 2011, **382**, 203-210.
5. V. Kumar, R. K. Gundampati, D. K. Singh, D. Bano, M. V. Jagannadham and S. H. Hasan, *Journal of Photochemistry and Photobiology B: Biology*, 2016, **162**, 374-385.
6. S. Basiri, A. Mehdinia and A. Jabbari, *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 2018, **545**, 138-146.
7. P. Salazar, I. Fernandez, M. C. Rodríguez, A. Hernandez-Creus and J. L. González-Mora, *Journal of Electroanalytical Chemistry*, 2019, **855**, 113638.
8. M. Mavaei, A. Chahardoli, Y. Shokoohinia, A. Khoshroo and A. Fattahi, *Scientific reports*, 2020, **10**, 1762.
9. H. Chang, H. Kang, E. Ko, B.-H. Jun, H.-Y. Lee, Y.-S. Lee and D. H. Jeong, *Acs Sensors*, 2016, **1**, 645-649.
10. Z. Zhu, X. Shi, Y. Feng, M. He, C. Ye, H. Zhou, M. Zhang, W. Zhang, J. Li and C. Jiang, *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 2023, **288**, 122211.
11. X. Gong, M. Tang, Z. Gong, Z. Qiu, D. Wang and M. Fan, *Food chemistry*, 2019, **295**, 254-258.

12. L. Kong, M. Huang, J. Chen and M. Lin, *New Journal of Chemistry*, 2020, **44**, 12779-12784.
13. A. Alyami, A. J. Quinn and D. Iacopino, *Talanta*, 2019, **201**, 58-64.
14. S. HaiYang, W. Zhengkun, Z. Yong and Z. Jie, *Optics Express*, 2023, **31**, 16484-16494.