Abstract

The increasing global emphasis on alternative and complementary medicine has elevated the importance of medicinal plants and their bioactive constituents, known as phytochemicals. However, the efficacy and safety of herbal products remain constrained by the lack of standardized and cost-effective analytical techniques for quantifying these compounds. This thesis addresses this critical gap by developing electrochemical sensing platforms based on Molecularly Imprinted Polymers (MIPs) for the selective detection of key phytochemicals—specifically piperine (PIP) and thymoquinone (TQ)**,** present in Indian spices such as *Piper nigrum* (black pepper) and *Nigella sativa* (black cumin) respectively. The molecular dimensions, auxiliary variations, and morphological modifications of the imprinted electrodes were analyzed using Fourier Transform Infrared (FTIR) spectroscopy, Field Emission Scanning Electron Microscopy (FESEM), and Ultraviolet–Visible (UV–Vis) spectroscopy, respectively.

Firstly a novel molecularly imprinted poly (N,N-dimethylacrylamide)-based graphite electrode (PDMAM-MIP@G) was fabricated for sensitive and selective detection of piperine from black pepper. The sensor was synthesized via polymerization using N,N-DMAM as monomer, piperine as the template molecule, and EGDMA as the cross-linker. The fabricated sensor showed high sensitivity (12.92 μA/μM), wide linear range (0.001–200 μM), and low detection limit (0.6 nM), with excellent stability, reproducibility, and selectivity. A CNN-driven model, validated against RP-HPLC, predicted piperine from CV data with outstanding accuracy ($R^2 \approx 0.9999$, MAE = 0.0339%). Next two novel electrochemical sensors was developed for detecting thymoquinone (TQ) in black cumin: a titanium oxide-modified graphite paste electrode (GPE/TiO_2) and a thymoquinone-imprinted polymer electrode (TQIP/G). Both sensors showed excellent sensitivity, low detection limits (0.02 μM and 0.03 μM respectively), and broad linear ranges . They demonstrated high selectivity, reproducibility, and stability. Principal Component Analysis (PCA) effectively distinguished cumin brands based on TQ content. For enhanced prediction accuracy, a 1D convolutional neural network (CNN) model was trained using DPV signals, achieving near-perfect agreement with reference RP-HPLC data ($R^2 \approx 0.9996$).

The thesis also includes the development of a low-cost potentiostat (LC-PSTAT) designed using Arduino microcontroller, LF347 OPAMPs, and integrated with Python-based software which

supports both cyclic voltammetry (CV) and differential pulse voltammetry (DPV). It showed a detection limit of 0.03 μM and a linear detection range of 1–200 μM for thymoquinone. The sensor maintained high repeatability (RSD 3.23%), reproducibility, and stability. Real-sample analysis of four branded black cumin samples using DPV and Partial Least Squares Regression (PLSR) showed a prediction accuracy of ~94% when compared with RP-HPLC.