

Low-molecular-weight (LMW) compounds play crucial roles in numerous biological, chemical, and environmental processes. Given the diverse roles and significance of LMW compounds, the development of innovative analytical techniques for their analysis is imperative. In this thesis, we present novel approaches for the analysis of LMW compounds, focusing on the fabrication of advanced analytical devices to meet the evolving needs of various scientific disciplines.

The primary objective of the research was to fabricate novel analytical devices and explore their capabilities in the analysis of volatile and non-volatile LMW compounds.

Innovative solid-phase microextraction (SPME) fibers were synthesized by modification of polypyrrole coating material with metal-organic frameworks (MOFs), specifically ZIF-8, enhancing their extraction performance for volatile organic compounds (VOCs).

For the analysis of non-volatile compounds, two types of LDI-MS targets were fabricated: gold nanostars as inorganic matrix and silver nanostructured substrates. Gold nanostars were synthesized using seed-mediated approach and characterized using XRD, DLS, SEM and TEM, UV-VIS spectroscopy, ICP-MS. At the same time, silver nanostructured substrates were synthesized using chemical vapor deposition. The effect of mass of silver precursor on particles morphology, size and distribution, and LDI-MS performance was investigated. Both LDI-MS targets demonstrated promising sensitivity towards LMW compounds, including lipids.

The potential of devices for differentiation of bacterial species has been investigated on eight bacterial strains such as *Morganella morganii* (MM), *Staphylococcus warneri* (SW), *Lactobacillus plantarum* (LP), *Enterococcus faecium* (EF), *Enterococcus durans* (ED), *Lactococcus garvieae* (LG), *Staphylococcus epidermidis* (SE), and *Escherichia coli* (EC). Statistical methods of data processing included principal component analysis, hierarchical cluster analysis, random forest model, and canonical correlation analysis. The results of the research indicated on potential of lab-made innovative devices for differentiation between bacterial strains.

Developed analytical devices demonstrated significant potential for analysis of LMW compounds and subsequent applications in various fields, including analytical chemistry, forensic sciences, food manufacturing, and environmental monitoring, offering cost-efficient alternatives to commercially available analogs.