Preface

The research work required to do a substantial addition to the existing knowledge includes, but not limited to the literature surveys in electrochemical spectroscopy and allied fields, design and developments, simulations and fabrications, experimentation and analyses, measurements and hypothesis, functionalization and immobilization, selectivity and sensitivity, detailed mathematical and statistical analyses, and result validations. The presented research work has successfully been applied to develop an exciting transducer that could not only selectively detect its target but is also capable enough to quantify harmful synthetic organic impurities in our foodstuff. This research work has not only provided a real-time tool to perform week-long chemical and biochemical assays in minutes, but had also played its part as a source for community awareness about the said chemicals that we keep ingesting knowingly or unknowingly. Consequently, these teratogenic, endogenic and carcinogenic chemicals become our body burden, and we fall prey to some incurable diseases that shorten our lifespan.

Phthalates are the most ubiquitous chemicals that pose a grave danger to the human race due to their extraordinary use as a plasticizer in consumer product industry. All contemporary detection methods require a high level of skills, expensive equipment and long analysis time as compared to the technique presented in this research work that introduces a real-time non-invasive assay. A novel type of silicon substrate based smart interdigital transducer was fabricated by employing thin-film micro-electromechanical system semiconductor device fabrication technology. Electrochemical impedance spectroscopy was used in conjunction with the fabricated sensor to detect hormones and phthalates in deionized water, energy drinks, and juices. Various concentrations of phthalates as low as 2 parts per billion to a higher level of 200 parts per million in deionized water, energy drinks, and juices were detected distinctively using the new planar interdigital sensor based EIS sensing system. The sensor was functionalized by a self-assembled monolayer of 3-aminopropyltriethoxysilane embedded with molecularly imprinted polymer to introduce selectivity for the phthalate molecule. Spectrum analysis algorithm converted the experimentally obtained impedance spectra into useful information about the analyte by applying complex nonlinear least-square

curve fitting in order to obtain equivalent electrochemical circuit and corresponding circuit parameters describing the kinetics of the electrochemical cell. Principal component analysis was applied to monitor the effects of a surface-immobilized molecularly imprinted polymer layer on the evaluated circuit parameters and its electrical response.

The major contribution of this research work is the development of a smart sensing system that owns the capabilities to detect hormones and endocrine disruptor compounds in foodstuffs. Low-cost and robustness are the merit points that make this system unique. This system is sufficiently flexible to be readily coupled with any existing chemical and biochemical sensing technique.

The contributions of this research work can be summarized as follows:

1. Explore a novel interdigital capacitive sensor design that owns enough penetration depth of the fringing electric field, sufficient to allow bulk sample testing.
2. Analyse the new sensor design by finite element analysis and select the most precise configuration of interdigital electrodes under given boundary conditions.
3. Fabricate smart sensor interdigital configuration on a silicon substrate using MEMS-based semiconductor fabrication technology.
4. Analyse and achieve stability and reproducibility in impedance measurement characteristics of the smart sensor design.
5. Establish automatic data acquisition setup to measure impedance characteristics of an electrochemical cell using the fabricated sensor.
6. Real-time detection and analysis of hormones in a bulk aqueous medium by applying electrochemical impedance spectroscopy technique.
7. Real-time detection and analysis of two phthalate esters in polar, electrolytic, and acidic media using electrochemical impedance spectroscopy technique in bulk samples.
8. Develop and tailor robust technique to induce selectivity for the phthalate analyte in the smart sensing system.
9. Explore and apply the self-assembled monolayer (SAM) technique to immobilize analyte-selective functional material on highly polished silicon substrate based smart sensor.
10. Analyse the performance of the intelligent selective sensor and its validation by commercially available detection methodology.

One of the main objectives of this research was to develop an assay system that is robust enough to have an in-field application without any technical expertise requirement for the user and is sufficiently rapid to be used for real-time monitoring of beverage products at industrial level. In order to achieve this objective, a real-time label-free assay protocol was tailored and applied by introducing “artificial antibodies” to induce selectivity in the designed sensing system. The results obtained by the testing system were validated using commercially available high-performance liquid chromatography, diode array detection at an incident wavelength of 224 nm. DEHP tainted energy drinks and juices were also investigated by the developed technology in order to validate its operation in the real-world scenario.

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