# Abstract

Citrus fruit juices exhibit health-beneficial activities due to the presents of nutritive phytochemicals such as octanol, hespiridin, neohespiridin, nomilin, limonin, etc. Therefore, it finds its place as the major constituent product of the juice and the food industry around the world. Most of the citrus fruits such as kinnow, mosambi, orange, pomelo, lemon, grapefruit, etc are grown by Indian farmers and these belong to the family Rutaceae. The fruits are available during its season and it is accepted in the form of fresh juice. Therefore packaging fruit juices and products could be a substitute for their seasonal availability to make them available all year round. However, citrus juices faced formidable problems in terms of bitterness. The consumer's acceptance of citrus fruit juices is affected by the bitterness of the juice making it inappropriate for long time storage. Bitterness is mainly caused by the accumulation of two different chemical compounds: limonin from the limonoid terpine group and naringin from the flavonoid phenolic group in the fruit. The bitterness developed at the instant of juice extraction is due to naringin and the delayed bitterness that is developed after the extraction of juice is due to limonin. This delayed bitterness that increases with time causes the problem of long term storage of the juices. The intact fruit hardly contains limonin; however, its non-bitter precursor, limonate- A-ring lactone (LARL), is found to be endogenously present in the cell cytoplasm probably at a neutral to slightly alkaline pH in membranous sacs. When these sacs of fruits are ruptured during juice processing, the LARL encounters the net acidic pH of the juice, which gradually catalyzes the closure of the ring to form limonin. The quality of juice becomes objectionable if limonin exceeds ∼6 ppm (∼13 μM) as per the study. The other bitter limonoids are also present in the citrus juices, but at much lower concentrations than limonin and hence do not contribute significantly to the bitterness problem. Therefore, it is very important to quantify and reduce limonin levels in citrus fruit juices for improvement in the fruit quality and economic growth of the same.

The bitterness can be controlled to a semi-acceptable level by chemical, biochemical and physical means. Most of the analytical techniques reported for detection such as HPLC, TLC, GLC, RIA and EIA have several advantages in the analysis, but all the techniques have other limitations as they alter the chemical composition of the juice affecting the its quality, are time-consuming, expensive, use toxic chemicals and are not designed for on-site testing. The sensors developed to date such as amperometric biosensor, Organic Electrochemical Transistor are also expensive, complex and time-consuming in terms of fabrication. The methods/devices are also not suitable for the measurement of limonin reduction which is essential for the processing and storage of juice. Therefore, there is a huge scope of research for the development of devices to overcome the present limitations.

Here, we like to focus on the design and development of Interdigitated Electrodes (IDE) based sensor which can be applied in the quantification as well as measure reduction of limonin in citrus juices. We have used the planar interdigitated structure in our work for capacitive sensor which exhibits a large number of applications due to its miniature size, fast response, less fabrication cost, less power consumption, etc.

The thesis presents the development of two different devices – one using cerium oxide nanoparticles as a sensing material for detecting limonin in citrus fruit juices and another using magnesium silicate to detect as well as reduce limonin level by selective adsorption of limonin in the surface of magnesium silicate. The cerium oxide nanoparticles are green synthesized using Dillenia Indica aqueous extract in overcoming the limitations of the physical and chemical synthesis process. The chemical structure, morphology, and optoelectronic characterization of synthesized nanoparticles have been done using XRD, SEM, EDX, and UV-vis spectroscopy. The sensors have been fabricated on the cellulose paper substrate over the Ag electrodes IDE pattern. The synthesized materials ($CeO_2$) have been drop-coated over the pattern. The other sensor with magnesium silicate as a sensing and reducing element has been prepared by the same drop-coating methodology. The performance of the sensors has been analyzed in terms of their accuracy, sensitivity, specificity, reusability, detection limit, response time, reproducibility, shelf life, etc. The HPLC (analytical) technique has been used for the validation of the sensor results. The sensory evaluation, TPC, TFC, and antioxidant properties are also studied for the debittered juice samples. While comparing the developed devices with earlier reported methods it is observed that the devices exhibit high accuracy, low cost, fast response, high sensitivity, selective, on-site measuring capability, etc. Finally, we conclude that these approaches may lead to significant progress in next-generation low-cost, efficient sensors for detection and debittering in citrus fruit juices.

Keywords: *Limonin; cerium oxide nanoparticles; magnesium silicate; bitterness; IDE sensor*