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ENVIRONMENTAL ANALYTICAL CHEMISTRY

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SESSION CHAIR: DR. M.N.V. PRASAD
Department of Plant Sciences, University of Hyderabad, India

SESSION CO- CHAIR: DR. YUCEL KADIOGLU
Ataturk Universit, Faculty of Pharmacy, Department of Analytical Chemistry ,Turkey

SESSION INTRODUCTION

TITLE: COMPARISION OF RETINOL PLASMA LEVELS IN PATIENTS WITH BLADDER CANCER AND HEALTHY VOLUNTEER WITH HPLC-DAD METHODS AFTER A SINGLE ORAL -CAROTENE ADMINISTRATIONB

DR. YUCEL KADIOGLU, Ataturk Universit, Faculty of Pharmacy, Department of Analytical Chemistry ,Turkey

TITLE: BIOANALYTICAL TECHNIQUES TO DETECT TRACES OF PESTICIDES AND TOXINS IN THE FOODS AND ENVIRONMENTAL SAMPLES

DR. M. S.THAKUR, Scientist, Central Food Technological Research Institute, India

TITLE: DEVELOPMENT OF AN ANALYTICAL METHOD FOR THE DETERMINATION OF STORAGE LIPIDS IN CALANUS FINMARCHICUSS

DR. ABDULLA AHMAD YUSUF, Fisheries Research Services (FRS), Marine Laboratory, Torry Aberdeen, UK

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TITLE: USING ATR- FTIR FOR DETECTION OF IMMOBILIZED BIOMOLECULES ON AMINO ACID FUNCTIONALIZED HAP NANOPARTICLES

DR. M.N.V. PRASAD, Department of Plant Sciences, University of Hyderabad, India

TITLE: DETERMINATION OF MELOXICAM AND FLUFENAMIC ACID IN PHARMACEUTICAL FORMULATIONS AND BIOLOGICAL FLUIDS USING LANTHANIDE SENSITIZED LUMINESCENCE

DR. SALMA M. Z. AL-KINDY, Department of Chemistry, College of Science, Sultan Qaboos University, Sultanate of Oman

TITLE: ARSENIC REMOVAL FROM GROUND WATER BY TREATMENT AND REMEDIATION METHODS

DR. IMRAN ALI, Department of Chemistry, Jamia Millia Islamia (Central University), India

TITLE: ELECTROCHEMICAL BIOSENSOR FOR DETECTION OF CADMIUM IN MILK

MR. SACHIN SURYAN, Punjabi University, Department of Biotechnology, India

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Comparision of Retinol Plasma Levels in Patients with Bladder Cancer and Healthy Volunteer with HPLC-DAD Methods after a Single Oral β-Carotene Administration

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The disease-preventing activity of β-carotene could be ascribed either to their conversion into retinoid or to their activity as intact molecules.The aim of study were to develop and validate a simple, precise, accurate and specific HPLC-DAD method for determination of retinol from human plasma and to examine whether retinol levels of patients with bladder cancer are increased or decreased when compared to healthy volunteers after a single oral administration of 20 mg of β-carotene. The chromatographic conditions of the HPLC-DAD methods using vitamin K_2 as the internal standard (IS) were optimized. Retinol and IS were extracted into n-hexane and chloroform containing butylated hydroxytoluene solvent system.The method has a wide linear over the 0.5-10 µg/mL of concentration range (the endogenous retinol has a concentration of approximately 0.79µg/mL). The precision (RSD %) of this method was less than 7.9%, and accuracy (RE) was better than ± 9.1 (n=6). The developed and validated method could be successfully applied to determination of retinol measured in plasma samples (one milliliter blood samples were collected at 0 (before dosing) and 2.5 h after dosing) from six healthy volunteers and six bladder cancer patients following oral administration of single dose of β-carotene. Obtained data in this study were compared by Student-t test (at 95% coeffidence level). There is no significant difference between plasma concentration of retinol of healthy volunteers and bladder cancer patients (for 0 h; t_h=1.374; P>0.05, for 2.5 h; t_h=0.208; P>0.05).

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Bioanalytical Techniques to Detect Traces of Pesticides and Toxins in the Foods and Environmental Samples

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Food and environmental safety is a prime concerned of modern human society. The economy of India is mainly depending on agriculture. The green revolution achieved in India has been possible only because of the effective management of land and water resources along with inputs like fertilizers and pesticides. The increasing use of pesticides/herbicides/insecticides in recent years for achieving higher agricultural yields has posed considerable problems in general health programs. These organic toxins enter animals and human beings directly as well as indirectly through the food chain or drinking water. The high toxicity of organophosphorus neurotoxins and their large use in modern agriculture practices has generated public concerns. Short duration exposure of these pesticides can potentially create health hazards. Thus, there is a need for the detection of these pesticides at high sensitive level with fast, reliable and economically feasible analytical technique. Biosensor is an alternative tool to detect the pesticide at sensitive level with rapidly and cost effectiveness. There is also a need to develop a simple dipstick technique having field applicability and rapidity for the detection of food toxins like pesticides. Following useful system have been developed at CFTRI for the detection of pesticides and food borne pathogens/toxins.

1. Biosensor based sensitive detection techniques.
2. Application of nanoparticles for bioanalytical applications.

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Development of an Analytical Method for the Determination of Storage Lipids in *Calanus finmarchicus*

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A method was developed for the determination of the major storage lipids, wax ester and triglycerides, in the copepod *Calanus finmarchicus*. A variation of the Folch method was used to extract the lipid. The method was scaled down to enable the extract ion of either pooled (~l mg) or individual (~200 µg) copepods. The major lipid classes were identified using TLC and quantified using HPLC coupled with evaporative light scattering detection. Analysis of laboratory reference materials indicated that this method underestimated the minor triglyceride component, but gave a good estimate of the major wax ester component. The fatty acid and fatty alcohol composition of the *C. finmarchicus* were determined following trans-esterification of the lipid extract in methanol. Fatty acids and fatty alcohols were initially identified by comparison with authentic standard and by mass spectroscopy. Using GC with flame ionisation detection the normalised area percentage of the fatty alcohols and fatty acid m ethyl esters was deter mined simultaneously in one run for either pooled or individual copepod samples. These methods were applied to *C. finmarchicus* collected from the Irminger Sea, North Atlantic in 2001 and 2002.

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Knowledge Explosion in Environmental Remediation Techniques and Strategies

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Bioremediation is an emerging and effective innovative technology for treatment of a wide variety of contaminants. This technology includes phytoremediation (plants) and rhizoremediation (plant and microbe interaction). Rhizoremediation, which is the most evolved process of bioremediation, involves the removal of specific contaminants from contaminated sites by mutual interaction of plant roots and suitable microbial flora. This discussion paper presents an exhaustive evaluation with respect to developments, current practices and perspectives of a variety of approaches of bioremediation. Bioremediation approach is currently applied to contain contaminants in soil, groundwater, surface water, or sediments including air. These technologies have become attractive alternatives to conventional cleanup technologies due to relatively low capital costs and the inherently aesthetic nature. This lecture would deal with bioremediation mechanisms such as phytosequestration, rhizodegradation, phytohydraulics, phytoextraction, phytodegradation, and phytovolatilization. Arsenic, Mercury, Chromium, Fluoride, Cyanide, abandoned mines, fly ash disposed sites, engineered phytotreatment technologies, biological permeable barriers; and Organics viz., petroleum hydrocarbons, pesticides explosives are some of the examples chosen for this presentation. Quite a variety of plants, natural, transgenic, and/or associated with rhizosphere micro-organisms are extraordinarily active in these biological interventions and cleaning up pollutants by removing or immobilizing. While diverse microbes are the most active agents, fungi and their strong oxidative enzymes are key players as well in recycling recalcitrant polymers and xenobiotic chemicals as well. The proactive role of MoEF and industries for implementing bioremediation and envisaged action plan are also discussed. Institutions involved in bioremediation research, frequently asked questions covered in power point presentation.

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Determination of Meloxicam and Flufenamic Acid in Pharmaceutical Formulations and Biological Fluids using lanthanide Sensitized Luminescence

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Two highly selective and sensitive luminescence methods for the assay of anti-inflammatory drug meloxicam (MX) and flufenamic acid (FFA) in biological fluid and tap water are described. The assay of MX was based on europium sensitized luminescence. The method makes use of radiative energy transfer from the enolate ring to europium ions in methanol and in aqueous system. Optimum conditions for the formation of the enolate Eu^{3+} complexes were investigated. In methanol, the Eu-MX complex was found to depend on the concentration of Tris buffer and Eu^{3+}. In aqueous system, maximum sensitization was obtained in the presence of 0.28% Tween –80, 0.01M tris buffer pH 8.0, 6 μM of 1, 10- phenanthroline, 1.75 mM Eu^{3+} and 7 μM of gadolinium ions as co-luminescence reagent. Under the optimum conditions linear calibration curves between 0-1000 and 0-800 ppb for MX in methanol and in aqueous medium respectively were obtained with the detection limit being 6.0 ppb. The proposed method was successfully applied for the determination of MX in Mobic and Co-Oxicam tablets and in tap water and urine samples. Excellent recovering were obtained for the MX samples. The second method, a novel sequential injection analysis (SIA) approach was used for the determination of FFA in samples of urine and tap water. The method was based luminescence sensitization of terbium by complex formation with FFA. The luminescence signal was monitored at λ_{em} = 565nm when excited at λ_{ex} = 298nm using time resolved mode. Experimental factors that influenced fluorescence reaction were systematically optimized in aqueous medium using chemometric optimization. Under the optimum conditions linear calibration curves between 0-1200 ppb for FFA in aqueous medium were obtained with a LOD of 80 ppb. When applied to Urine and Tap water samples the procedures were found to be free from matrix interferences except for Fe3+ that had significant interference effect. The results obtained for the assay of FFA in urine and tap water samples demonstrated good accuracy and precision.

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Arsenic Removal from Ground Water by Treatment and Remediation Methods

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Arsenic contamination in ground water contamination is a serious problem as it is acute toxic. Ground water of many parts of the world are being contaminated by arsenic. Therefore, the development of arsenic treatment and remediation technologies is required urgently. The physical (ion exchange, reverse osmosis, electrokinetics, adsorption), chemical (coagulation and sedimentation, lime softening, oxidation) and biological (bacterial oxidation, phytoremediation) treatment methods are available for the removal of arsenic from the ground water. The units developed and used at commercial scale include different kinds of filters, bucket type units, fill and draw, kalshi etc. The remediation methods discussed include air oxidation, reactive barriers, utilization of deeper aquifers and sanitary protected dug wells. But still no technology is available which can be used for the removal of arsenic from the ground water at economic, efficient and commercial levels affordable in the developing and under developed countries (especially Bangladesh, India and other effected countries). Further improvements are required to develop fast, efficient and economic arsenic removal technologies.

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Electrochemical Biosensor for Detection of Cadmium in Milk

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Exposure to cadmium is among the main threats to human health from heavy metals. Among heavy metals cadmium and lead are the main contaminant of milk (*Alonso et al., 2003, Ayar et al., 2008*). The permissible limit of Cd in milk has not been defined though it is 3 ppb for mineral water as defined in CODEX STAN 193-1995 rev.2009. In the present study an electrochemical biosensor has been developed for the detection cadmium in milk. *Bacillus badius* whole cells, an isolate of biosensor technology lab, were used as biocomponent in the study, immobilized at the tip of carbon paste electrode. The principle of biosensor was based on the inhibition of urease activity, was measured in terms of NADPH through cyclic voltammetry at a scan rate of 100mV/s. *Bacillus badius* is a urease producing micro-organism; urease and Glutamate dehydrogenase of the cell were used as bi-enzymatic machinery in the assay. Ammonium ion produced by urease activity along with NADPH was utilized in the reaction catalyzed by Glutamate dehydrogenase. Excess of NADPH was measured through cyclic voltammetry. Inhibition of urease leads to lesser production of ammonium ion thereby increase in unutilized NADPH and therefore increases the current. The developed biosensor was applied to natural milk and spiked milk samples. A detection limit of as low as 0.5ppb has been achieved. Cadmium is showing specificity over lead there is no interference of lead. Application of whole cells in the study as bi-enzymatic machinery is making the practice cost effective.

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Wide Scope of Polymeric Membrane Based Ion Selective Electrodes

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Ion Selective Electrodes (ISEs) are the chemical sensors of longest history and probably the most frequent routine application. The most commonly used ion selective electrode is the pH described it in 1906. By the mid 1960's the Orion Research Inc was producing calcium electrodes for the use in blood gas analyzers (Frant 1994). Since then numerous electrodes has been developed for the analysis of samples containing many different ions. A chemical sensor detects the presence of specific chemical or class of chemical in any sample. These are miniaturized analytical devices, which can deliver real-time and on-line information on the presence of specific compounds or ions in complex samples. Usually the analyte recognition takes place followed by the conversion of chemical information into an electrical or optical signal.

The basic ISE setup includes a potentiometer, a probe (selective for each analyte of interest) and various consumables used for the pH or an ionic strength adjustment, which makes the cost of initial setup low as compared to other techniques. ISE determinations are not subject to interferences such as colour in the sample. Thus in past two decades, there has been a growing interest in search for ionophores (electroactive material) that can chemically recognize specific ion and offer either new or improved selectivity for different ions. The limited availability of such materials makes it difficult to develop efficient sensors and hence this is a demanding field of research.

ISEs now being exploited as detectors in flow injection analysis systems. In such systems, the dynamic behaviour of an ISE assumes great importance, since this will determine the signal shape and the sampling rate. Characterizing the dynamic behaviour of ISEs constitutes the third area of advancement in this research field. Physiological and biological applications of ISEs have been a goal since the inception of the field. Miniaturization in instrumentation seems to be the current paradigm in the ISEs study which leads to the development of microelectrodes for intracellular measurements of ion activities. Finally, commercial application of these electrodes is the major area to be worked upon.