# ISASINDIA Newsletter

*Volume 19, No 9* *September 2019*

## EDITORIAL

Welcome to the new issue of ISASINDIA Newsletter, the e-newsletter of the Indian Society of Analytical Scientists (ISAS). ISAS Newsletter aims to showcase the wide range of analytical science activities being run by ISAS as well as linking with maximum number of the Indian analytical scientists community beyond our membership. September 2019 has been a busy and hectic month for ISAS, organizing the Indian Analytical Science Congress 2019. The ISAS Life time Achievement award 2018 was presented to Dr K N. Ninan during the congress. The congress showcased cutting edge analytical and bio-analytical science to researchers, students and teachers as well as scientists from several R&D organizations across the country. The abstracts of the plenary and invited talks presented in the congress are included in the newsletter. A seminar on 150 years of periodic table of elements and advances in hyphenated chromatographic techniques was held at Mar Ivanios College, Thiruvananthapuram on September 18, 2019. Nearly 250 PG students and researchers attended the seminar. Meeting of the New EC AND General body meeting were held on 20th September 2019. Further details are included in the newsletter. Please send your feedback and any content for the next issue by November 30 th 2019 by emailing the Editor-isaskerala@gmail.com.

Editorial Committee

# Message from President ISAS

The Indian Analytical Science Congress , 2019 (IASC-2019) recently concluded at Hotel Apollo Dimora, Thiruvananthapuram was indeed a feather in the cap of ISAS which is poised to usher with a fresh zeal and spate of activities, as a National Professional Body of Experts, towards National Development.

The wide spectrum of topics in Analytical Sciences, presented as Plenary and Invited Talks by world class experts in a variety of technical fields, was a new dimension of this latest Congress in the IASC series conducted by ISAS.

Thanks to the meticulous planning and execution by the organizing team, a large number of Senior experts from VSSC, DAE, NIIST, Universities, etc. displayed outstanding levels of performance.

Dr KN Ninan, Former Deputy Director, VSSC who did pioneering work in establishing many indigenous facilities, was bestowed with the prestigious "Life Time Achievement Award 2018 " of ISAS, at the Inaugural Session of the Congress.

Participation of a large number of leading experts from various institutions in India, a substantial presence of student delegates, contributions by many sponsors and presence of exhibitors and their technical talks, etc. made the event a grand success.

ISAS News Letter is an Excellent Media for outreaching a vast number of users of Analytical Instruments and Systems. Hence this News Letter is an excellent channel, useful for the Instrument Manufacturers and Suppliers, to continue to popularise and market their products.

I am happy to note the excellence of contents and quality of presentation, as well as the high levels of acceptance of ISAS News Letter amongst our members.

I wish every one a happy reading of ISAS News Letter.

(Dr. P. P. Chandrachoodan)

President, ISAS.

# Seminar organized by ISAS at Mar Ivanios College Thiruvananthapuram.

ISAS In association with chromatographic Society of India and Mar Ivanios College, Thiruvananthapuram organized an one day seminar on 150 years of Periodic Table of Elements and Advances in Hyphenated Techniques at the Mar Gregorios College, Thiruvananthapurm on 18th September 2019.


## Inauguration of the seminar

A seminar on 150 Years of the Periodic Table of Elements & Advances in Hyphenated Chromatographic Techniques jointly organized by the Indian Society of Analytical Scientists (ISAS), Head Quarters (Mumbai) & Kerala Chapter; Chromatographic Society of India & Department of Chemistry, Mar Ivanios College (Autonomous), Thiruvananthapuram was held at Mar Ivanios College on 18th September 2019.

The seminar which was attended by more than 220 participants including Teachers, Scientists, PG students and research scholars from various other institutions was inaugurated by Dr. Georgee K.I, Principal, Mar Ivanios College in a meeting presided over by Dr. P. P. Chandrachoodan, President, ISAS.


Prof. C. G. Ramachandran Nair (former Head, Dept. of Chemistry, University of Kerala), Dr. A. P. Jayaraman (Senior Nuclear Scientist), Prof. S. Sivadas (Writer & Former Professor of Chemistry, CMS College, Kottayam) and Dr. G. Ramakrishnan (President, Chromatographic Society of India) were the esteemed resource persons. Dr. V. R. Nair (ISAS), Dr. K. K. A. Rashid (ISAS, Kerala Chapter), Dr. V. S. Jayakumar (Former Prof. of Physics, Mar Ivanios College) and Dr. Rajeev (Scientist, VSSC) were present all through the seminar.

The coordination of Ms. Suma P. Oommen, HoD of Chemistry, cooperation of teachers from the Department of Chemistry and support of the students (M.Sc Chemistry-I and II years), contributed to the success of the seminar. Students from University College, Govt Women's College, All Saints College, Mahatma Gandhi College, S N College ,University Department of Chemistry – all from Thiruvananthapuram attended the seminar.

Audience of the workshop

The seminar was a grand success. It was rich in content and valuable in creating better understanding of the topics that were discussed. The speakers were all highly respected professionals, and the presentations were excellent.

## Indian Analytical science Congress 2019

Indian Analytical science Congress 2019 was held at Hotel Apollo Dimora, Thiruvananthapuram, Kerala State during September 19-2,2019. The conference was attended by 224 participants from all over India. One award talk, four plenary

talks, and seventy Nine contributed papers were presented in the congress. An exhibition was also organized in connection with the congress. The congress was inaugurated on 19th September 2019 at 10 AM by Dr Dinesh Srivastava, Chief Executive of Nuclear fuel Complex Hyderabad. The inaugural function was presided over by Dr P.P. Chandrachoodan, President- ISAS. Dr S C Sharma, Depuy Director, VSSC, Thiruvananthapuram and Prof (Dr) C G R Nair, Former Dean faculty of Science, Kerala university, spoke on the occasion. Dr KKA Rashid Chairman-National Organizing Committee welcomed the participants and Dr R.Rajeev, Chairman local organizing committee proposed vote of thanks.


Dr Srivastava inaugurating the congress by lighting the sacred lamp

ISAS Life time Achievement Award 2018 was presented to Dr K N Ninan, Former Deputy Director VSSC on this occasion .Professor C G R Nair presented the life time achievement award to Dr K N Ninan.

Prof CGR Nair presenting ISAS Lifetime Achievement award 2019 to Dr KN Ninan

A section of audience

The inaugural Session was followed by technical sessions. A cultural programme and dinner was arranged on 19th evening. Exhibition organized in connection with the conference was inaugurated by Dr P P Chandrachoodan, President, ISAS National body on 19th afternoon. Concluding session was held on 21st at 16.30 Hours. Dr P P Chandrachoodan, President- ISAS .Dr KKA Rashid, Chairman-National Organizing Committee, Dr VR Nair ,Convener- National organizing committee, Dr Rajeev, Chairman-local organizing committee, Shri Rakesh Ranjan, Convener local organizing committee were present on the dais. Awards for best oral presentation(Contributed paper) and award for best poster presentations were distributed. Best exhibition stalls were also presented with mementos and certificates. In the feed back session delegates expressed happiness and satisfaction over the arrangements and they look forward to attending future programmes organized by ISAS. The session was concluded at 17.30 hours. Abstracts of award talk, plenary talks and invited talk are given below:

**AWARD LECTURE**

# ANALYTICAL SCIENCE IN THE EVOLUTION OF CHEMICAL SYSTEMS FOR INDIA'S SATELLITE LAUNCH VEHICLES & SPACECRAFTS

Dr. K N Ninan

ISAS Lifetime Achievement award 2018 Winner

The synergy of working together by synthetic chemists, formulation technologists, analytical scientists and scale-up engineers helped ISRO to realize several state of art chemical systems towards the self reliant Indian space program. The article gives a bird's eye-view of a few of them. The development, scale up and production of hydroxyl terminated polybutadiene (HTPB) binder and the composite solid propellant based on it is a major milestone. The polymerisation conditions for HTPB were standardised to get reproducible microstructure, as measured by 1H NMR. The GPC studies on functionality distribution and the 13C NMR data revealed the presence of tri and poly functional entities in the resin, which helped to understand and overcome the problem of reproducibility in propellant mechanical properties. Detailed mechanical characterization of the propellant gave inputs for designing propellant grains with adequate margin of safety. The studies on penetrometric pot life of propellant paste with toluene diisocyanate and slower reacting isophorone diisocyanate as the curatives gave a clue to realize porosity-free S200 solid motor, the third largest in the world. The demand for chlorine-free, eco-friendly solid propellants, led to the development of new oxidizers like ammonium dinitramide and binders like glycidyl azide polymer. Their NMR spectra (1H, 13C, 14N, 15N & 17O) confirmed the structure of the synthesized products and the TG - DSC results came handy for their estimation at mg quantity levels. Profiling the matrix resin- reinforcement composition and the specific heat and its variation with temperature of the silica - phenolic / carbon – phenolic liners of a failed SLV3 Stage 1 nozzle in 1985, using the TG-DSC method developed for the purpose, gave an insight in to the failure and provided the inputs for design of the ablative nozzle liners. There are many more instances of failure investigation analyses, leading to realization of reliable products. A host of alloys make up the rocket hardware and a judicious combination of analytical methods like chemical analysis, XRF, AAS, ICP-AES, Glow Discharge OES, C/S Analyser and Gases in metal analyser is used to confirm the quality of these alloys. In early 1980's (when instrumental methods were not available), appropriate chemical methods were developed to meet specific requirements, like (i) estimation of V & Cr in 15CDV6 by perchloric acid oxidation method, faster than the ASTM E30 method (ii) A rapid non-aqueous titrimetric estimation of ammonium perchlorate in pyrotechnic compositions and propellant paste to suit control lab needs and (iii) A colorimetric method for the detection and a non-aqueous titrimetric method for the estimation of FDMH impurity in UDMH, during its developmental stage. The technologies for the production of the liquid propellants like unsymmetrical dimethyl hydrazine,

monomethyl hydrazine and N2O4 were developed in-house and transferred to industries, along with standardised analysis procedures. With the advent of cryogenic propulsion in ISRO, analytical scientists addressed the issue of analysis of LH2 with purity = 99.995% and critical impurities of oxygen < 2 ppb & nitrogen < 200 ppb. The denial of cryogenic technology by Russia was a big challenge faced by ISRO. Dozens of polymers and special materials were indigenised, in which analytical science had a significant role in deciphering and analysing them at each stage of development and characterizing them as the final products. Similarly, the phenol / formaldehyde ratio analysis by NMR and the cure optimization studies by DMTA were immensely helpful for the indigenous development of the silica-phenolic ablative nozzle throat insert for PS2/GS2 (when the item was not available from the imported source). For the future eco-friendly semi-cryo propulsion system, a hydrocarbon fuel, ISROSENE, was developed at an industry. A rapid IR spectroscopic method was developed, validated with NMR data and transferred to the industry, which enabled timely development of the product. Light weight, high strength polymer matrix composites made with epoxies as matrix and carbon / polyaramid fibre as reinforcement are extensively used in launch vehicles and satellites of ISRO. As a part of developing advanced matrix resins for future, phenol-triazine resin chemistry has been developed for possible satellite structure applications. Similarly, polyetheretherketone (PEEK), a thermoplastic with possibility for repair and rework, and for making cryo tanks has been developed. In addition to spectroscopic characterisation of these novel matrix resins, the glass transition and melting temperatures of PEEK were established using DSC. To withstand the high heat flux during the re-entry regime of Reusable Launch Vehicles, ceramic matrix composites have been developed / are being developed. Ceramic conversion studies of the pre-ceramic polymers, followed with solid state NMR and the detection of glass formation in borosiloxane polymer by DTA helped in progress of the work. The coming decades will witness establishment of self-sustainable habitats and exploration of minerals and other resources available on the Moon and Mars, and the Gaganyaan Project is a forerunner of Indian efforts in this regard. Studies on carbohydrate derived sorbants for CO2 removal were undertaken for air revitalization in crew cabin of manned spacecrafts mission, before the commencement of the Project. NMR was used for the structural elucidation of the sorbent and its adsorption – desorption characteristics. Functionalized polysilsesquoxane based regenerable sorbent for CO2 removal for long duration human space flight and its regeneration capacity have been demonstrated. Demands of the future and our determination to make Indian space programmes contemporary and competitive, give enormous challenges to space scientists and in particular to analytical scientists who have an important role to play in the ambitious programmes of India's future space endeavours.

## Plenary Lecture -01 : MICROSCOPIC LIGHT-ABSORBING PARTICLES IN THE ATMOSPHERE AS SHORT - LIVED CLIMATE FORCERS

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In recent years, there has been a substantial increase in interest in the climate impact of lightabsorbing particles in the atmosphere (known as aerosols) due to their high atmospheric warming potential. Of special significance is black carbon (BC) aerosols, which is believed to have a warming effect second only to CO2. Even though constitutes only a small fraction of atmospheric mass, BC aerosols have the potential to influence the energy balance of the earth-atmosphere system. Despite the general consensus among climate scientists on the significant role of light-absorbing aerosols on regional and global climate, various studies and impact assessments provide widely differing and contrasting inferences and projections. In this context, the South Asian region and India assume special significance due to the diverse geographical features, high population density, rapid urbanization and industrialization, leading to a highly complex aerosol system. Theparadox of light-absorbing aerosols and comprehensive investigation of their climate impacts using comprehensive measurements onboard a variety of platforms such as a network of surface climate observatories, research ships, instrumented aircrafts, satellite remote sensing and high altitude balloons along with climate modeling is the topic of this lecture. Talk will be at basic level suitable for an interdisciplinary audience.

## Plenary Lecture -02: APPLICATION OF RADIATION AND ISOTOPIC TECHNIQUES IN ENSURING FOOD SECURITY

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It is an undisputable fact that agriculture is the backbone of Indian economy. It contributes significantly to the GDP and directly or indirectly impacts the standard of living of roughly half the country's population. It also wields an overwhelming influence on the manufacturing and services sectors. However, climate change and associated phenomena have made agriculture quite unpredictable as compared to what it used to be, particularly for our traditional farmers, who form the majority of the farming community. Apart from this, there are problems associated with decline in soil fertility. Therefore, it is very important to find ways and means to make agriculture sustainable. Basic and applied research aimed at understanding and manipulating the molecular and biochemical mechanisms of plant responses towards biotic and abiotic stresses holds the key to sustainable agriculture. Agricultural practices should not only protect farmers' income but also preserve their soil and water resources. Considering the fact that about 10-40% of what is produced by the farmers do not reach consumers because of post-harvest losses, it is equally important to effectively use technology to ensure "farm-to-fork" transfer of what is produced, if we are to meet the ever-increasing food demands of a growing population. Agriculture scientists need to orient their work in the direction to provide sustainable solutions for the eradication of hunger, including the so-called "hidden hunger" BARC is a premier multidisciplinary R&D organization engaged in research with the objective of generating knowledge and technologies for nuclear power production, application of radioisotopes in industry, healthcare and food security, apart from research in frontier areas of science and technology. BARC has a robust research programme on nuclear agriculture and food sciences. Research efforts at BARC have resulted in the development, using nuclear techniques, of a large number of improved crop varieties. One of the important advantages of radiation-induced mutation breeding approach is that it enables breeders to bring in specific improvements in a crop, without affecting the existing good attributes/qualities of the variety. Using mutation breeding, forty four new crop varieties, particularly in oil seeds and pulses, with wide geographical adaptability and resilience to various biotic and abiotic stresses, have been developed. Short-duration pulse-crops, for newer niche like rice fallow conditions, have also been developed. Legumes, especially pulses, are integral part of sustainable agriculture, as their inclusion in cereal-based cropping systems contributes to soil fertility by supplementing organic nitrogen, reducing the demand for chemical fertilizers and enriching soil microflora. They are also important component of protein diet for large population of the country suffering from protein and mineral malnutrition or "hidden hunger". In addition, farmer-friendly kits for analysing soil fertility and environment-friendly biopesticide formulations have also been developed. Strategies like sterile insect technique, under integrated pest management program, are being developed. Technologies have also been

developed for easy, field-level assessment of soil organic carbon and detection of pesticides in agricultural produce. Technologies have also been developed that provide solution to post-harvest spoilage of agricultural produce. Combination of radiation and chemical based techniques has been developed for extending the shelf life of agricultural produce such as vegetables, fruits, pulses and cereals. A number of ready-to-eat food items have been developed which not only have extended shelf life, but also meet the nutritional requirement of consumers. The talk aims to give an overview of the application of nuclear and isotopic techniques that go a long way in ensuring food security for the country.

## Plenary Lecture -03: IMPACT OF OPTICS AND ANALYTICAL SCIENCE IN INDUSTRY 4.0

Murukeshan Vadakke Matham

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Economists and historians predict in the same way that the coming big wave of cyber-physical production will define the fourth industrial revolution, also known as Industry 4.0 or the Industrial Internet of Things (IIoT). In this I 4.0 everything produced in factories will be monitored, analyzed, quality approved, packaged and shipped by self-sustained, cloud- connected, AI cyber-robots.

A recent analysis and many forums discussed in recent times about the role of optics and photonics in I4.0. In general, Industry 4.0 otherwise known as the smart factory is seen as the immediate innovation phase in manufacturing. It is very evident that lasers and photonics will be pivotal to delivering Industry 4.0 on multiple fronts. The convergence of light assisted processing and communications under pinning Industry 4.0 is stimulating the ever increasing demand for more integration and novel approaches to manufacturing for applications in individualized manufacturing, precision healthcare and autonomous vehicles. In short, Industry 4.0 would be nowhere without optics and photonics. In the case of analytical science, analytical scientists in general face two important questions-

what have we got and how much of it do we have? These days' bioanalytical techniques for healthcare are present not only in hospitals and with doctors who perform surgeries but many also available in our homes. The most widely used examples are blood glucose meters for diabetes management and home pregnancy kits. While designing healthcare diagnostic imaging tools or sensors, it is required to ensure they are simple to use and also non-invasive. Analytical techniques are often used to fight fraud, food quality monitoring, face recognition and counterfeit currency detection are some of the examples. While we are slowly entering into 4th industrial revolution, the recent developments in this area also need to be reviewed. This talk in this context, will be covering all the above mentioned aspects and the recent study using grapheme plasmonics for diagnostic sensing applications, with a special emphasize on lasers and optics underpinning their impact in I 4.0. The research presented in this talk is from the research funding MOE ( RG 192/17) and is acknowledged.

## Plenary Lecture -04: RADIOANALYTICAL TECHNIQUES OF MEDICAL INTEREST: In vItro TO In vIvo ASSAYS

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Measurement of biological materials present in the body is one of the important aspects for disease management. The biological materials needed for sustaining body chemistry comes in different categories such as carbohydrates, steroids, peptides, proteins and DNA. Bio-analytical chemistry dealing with the measurement of biological substance is a key evolving branch of chemistry. Most of the analytical techniques used in conventional analytical chemistry are adapted and used for the measurement of biological materials. These include LC-MS, GC-MS, CE-MS, HPLC, GC, UPLC, MS and NMR. All these techniques involve the three basic principles of analytical chemistry i.e. identify, separate and quantify. However, these techniques fail to analyze those molecules which are present in small concentrations and present in a complex environment such as blood and tissue samples. One of the major complications of biological analysis is that the analyte of

interest is present in an environment having structurally close entities making the separation of the required species difficult. Another equally important problem is that the materials of interest are present in extremely small concentrations, nano to picomolar levels. The radio immunoassay (RIA) technique developed by Berson and Yalow in the early sixties addressed both the above issues in one step. RIA uses a radiotracer as signal and antibody as the reagent. The use of radiotracer offered the ability to measure picomolar levels of biological substances. The use of a highly specific antibody obviated the need for separation of the analyte from the bulk sample. In RIA, the analyte and its radioactive counterpart is allowed to compete with a limited amount of antibody. As the reagent is in limited concentration, the amount of tracer bound to the antibody is inversely related to the concentration of the analyte. Over the years, RIA evolved to better techniques using more sensitive tracers such as enzymes and fluorescent molecules. However, the use of antibodies as the specific reagent continues and is finding newer applications. Millions of immunoassays are done daily across the world. Mapping the biological reactions taking place inside the body by non-invasive techniques is another important aspect of disease management. Though the concentration of the biological materials present is not measured in absolute numbers, their relative concentration measured is useful to detect disease. Molecular imaging using radiopharmaceuticals is one such technique. For e.g. positron emission tomography in fusion with computed tomography (PET-CT) is used to find glucose metabolism in vivo. Cancer cells proliferate fast and hence need higher levels of glucose. Higher uptake of a radioactive glucose (FDG) is seen in PET-CT imaging of patients suffering from cancer. Similarly, the in vivo measurement of prostate specific membrane antigen (PSMA), an enzyme over expressed during prostate cancer, is used for the identification and staging of prostate cancer. A PET-CT imaging for this purpose is developed by using a radiolabelled enzyme inhibitor as reagent. The relative amount of PSMA present in different organs and tissues of the body is used for detection and staging of prostate cancer. Radiotracers used for in vivo analysis have short life of a few minutes to a few hours. Setting up facilities to make the radiotracers for PET-CT imaging in geographically distributed regions is an important task. The Molecular Group has set up a state of art facility which includes a cyclotron and radiochemistry laboratories at Cochin to make the radiopharmaceuticals for imaging of several diseases, more importantly cancer. This facility caters to the need of several hospitals in the Southern region of India.

Invited Lecture -01 : **RECENT ADVANCES IN THE APPLICATION OF ANALYTICAL TECHNIQUES IN FORENSIC SCIENCE**


**A K Basu**

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**Forensic science is oriented towards the examination of specimens, collected from a scene of crime in order to establish the link between a criminal and crime. The main function of a Forensic Science Laboratory is to provide an unbiased scientific report to the investigating agencies and thus help the judiciary system. Forensic Science Laboratory facilitates the investigation of different crimes with the use of advanced technologies in each and every field of forensic science. Forensic case exhibit samples are of varied nature covering both materials of biological and non-biological evidence usually encountered in civil and criminal cases. Since some of the techniques are applicable only for organic and inorganic materials, does a selection of a proper analytical technique is essential for successful and careful analysis using the latest scientific tools and objectively prepares report to aid the investigating agencies. The use of appropriate method along with selective separation processes provides the best option for many challenging analytical task. The analytical method chosen should be not only specific and selective but sensitive, reliable and accurate. Combination of nuclear and other non-nuclear suitable analytical methods are employed for examination of real life samples. The use of non-destructive methods like XRF, SEM , AAS, NAA etc is extremely useful in need to preserve the specimen for later use and reconfirmation of results. The major areas pertain to forensic ballistics involving investigation of shoot outs. The use of NAA and other complementary techniques help in answering certain questions like the identification of fire arms, the range of firing , an opinion about entry and exit hole etc. Gunshot Residues ( GSR) / Firearm Discharge Residues (FDR) and matching of bullet specimens are examples where nuclear and other analytical techniques are extremely useful. It has found application in**

the analysis of hair, fingernail, paint, glass, fuels, lubricants as well as counterfeit coins or antiques etc. Analytical chemistry play a vital role in forensic toxicology and in establishing the source correspondence to decide commonness of origin of the metals. Forensics and forensic investigations are fast changing their face as every other day new types of crime are being committed. Today DNA profiling, audio video tape forensics, brain finger printing, computer forensic etc are some of the newer forensic technologies which have given entirely new dimensions to the whole system of scientific investigations. Thus, a synergic combination of sensitive methods like AAS,ICPAES, ICPMS, Glow discharge MS, GCMS, LCMS, NAA, electroanalytical methods and other sophisticated analytical techniques along with separation methods like ion exchange, solvent extraction and chromatographic techniques can lead to specific and sensitive procedures as the aid towards establishing the truth in criminal justice system.

## Invited Lecture -02: FLUORESCENT NANOMATERIALS FOR BIOSENSING APPLICATIONS

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Fluorescent noble metal quantum clusters (NMQCs) with interesting functional properties have received great attention in the present scenario due to their fascinating photophysical and optical properties such as well-defined molecular structure, discrete electronic transitions and strong luminescence. Noble metal quantum clusters (NMQCs) are comprised of several tens of atoms and the optical properties of these quantum clusters are due to their sub nano meter core size. As the size of the particle is comparable to the Fermi wavelength of the conduction electrons, it can act as a bridge between nanoparticles and atoms or molecule. Protein protected metal quantum clusters provide more attractive features such as good aqueous solubility, excellent biocompatibility and versatile surface chemistry. The combination of unique optical, electronic and catalytic properties of metal quantum clusters together with the inherent biological properties of

proteins render NMQCs as promising candidates for various biomedical applications like imaging, sensing, delivery and therapeutics. The cost of proteins derived from animal sources is escalating and hence there is anenormous interest among researchers in finding out new protein ingredients, particularly from plant sources for the synthesis of nano materials. Gluten, a cysteine rich protein derived from wheat, stands out as an excellent candidate among different plant derived proteins owing to its ease of availability and low cost. Due to the presence of cysteine and other aromatic amino acids in the gluten protein, we employed it as a template for developing a new gold quantum cluster. To the best of our knowledge, this is the first report of a cost effective, one-pot, environmentally benign synthesis of gold quantum clusters using gluten, as a template. The AuQC@gluten shows intense red emission at 680 nm and is characterized using UV-Vis spectroscopy, fluorescence, Fourier-transform infrared spectroscopy (FT-IR), transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS). There are several reports on biosensors for the detection of creatinine, most of which are based on electrochemical or colorimetric sensing, whereas none has explored the gluten-directed formation of gold quantum clusters and their application in creatinine sensing. In this context, we developed a highly selective, sensitive and cost effective novel turn-on fluorescent sensing of creatinine using AuQC@gluten with PA (picric acid) acting as a quencher. Contrary to any other protein protected gold quantum cluster, AuQC@gluten is highly stable towards reactive oxygen species like $H_2O_2$. Turn-on fluorescence sensing using gold quantum clusters are still rare since most of the sensors reported in literature are based on the fluorescence quenching of gold quantum clusters. Here creatinine could selectively recover the PA- quenched fluorescence of AuQC@gluten owing to the higher binding affinity of PA towards creatinine. The red emitting AuQC@gluten has been applied in the detection of creatinine with high sensitivity and selectivity. The detection limit is found to be 2 nM in the linear range from 20 µM to 520 µM. This method allows the accurate detection of creatinine in clinical blood samples, indicating its promising application as a valuable diagnostic tool for patients with kidney malfunctions.

Invited Lecture -04: **MULTI-ANALYTICAL TECHNIQUES IN LITHIUM ION BATTERY TECHNOLOGY**

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Lithium ion battery technology is the only possible energy storage system currently one can rely on to power the electric traction. The various electrochemical characteristic of the lithium ion cell depends on the structural properties of the electrode material, electrode coating and the interface between various components. The material properties such as defects, impurities, morphology, chemical composition and distribution across the material influence the practical achievable energy and power density, cyclic stability of the cell. The structural properties of the material, electrode and the interface can be investigated using various analytical techniques such as X-ray diffraction, Scanning Electron Microscopy, Transmission Electron Microscopy, Thermo Gravimetric-Differential Thermal Analysis, ion chromatography, Inductively Coupled Plasma Atomic Emission Spectroscopy, Mass spectroscopy, Fourier Transmittance Infra Red and Raman spectroscopy. The electrochemical performance of the cell is investigated using electro analytical methods such as potentiostatic and galvanostatic impedance spectroscopy, cyclic voltammetry, constant current and constant voltage charging/discharging. The crucial role played by the of multi-analytical techniques in the development of various cathode materials (i) LiFe1-xMnxPO4, (ii) LiNi1-x-yCoxAlyO2, (iii) LiNi1-x-yMnxCoyO2, and Li1/3Mn2/3]O2 : (1-X)LiMO2 will be presented [1-3]. (i) De-convoluted Raman peaks and scanning transmission electron microscope-energy dispersive spectroscopy (STEM-EDS) is used to identify the nano-sized impurity phases present within the crystalline lattice of hydrothermally synthesized LiFe0.75Mn0.25PO4 (ii) The use of combination of versatile techniques such as X-ray diffraction, energy dispersive X-ray analysis mapping and vacuum Fourier transform infrared spectroscopy to identify distribution of aluminium and anion substitution at oxygen site in LiNi1-x-yCoxAlyO2, synthesized by the co-precipitation assisted solid state reaction will be presented (iii) The in-situ carbon encapsulation of LiNi1/3Mn1/3Co1/3O2 and Li1/3Mn2/3]O2 : (1-X)LiMO2mechanism will be discussed using detailed analyses carried out by Raman spectroscopy, TEM, EDS mapping and thermal analysis coupled with mass spectroscopy.

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## Invited Lecture -05: ANALYTICAL PRACTICES FOLLOWED AT NFC FOR THE CHEMICAL CHARACTERIZATION OF NUCLEAR FUEL MATERIALS

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Department of Atomic Energy (DAE) has established Nuclear Fuel Complex (NFC) with a clear cut mandate of manufacturing and supply of fuel sub-assemblies on industrial scale to both PHWRs & BWRs with very stringent specifications for achieving the optimum performance of the fuel under reactor operating conditions. Natural UO2 and enriched UO2 in the form of pellets are being used as fuel in PHWR and BWR respectively. The desired specifications are achieved by having a strict control on material manufacturing process and can be achieved through monitoring the quality of materials taking part in the process and also with a strict vigil on process conditions. Chemical Quality Control (CQC) of raw materials, process intermediates and final products will ensure the desired quality of final products and thus, it becomes integral part of QA/QC program. Control Laboratory (C.Lab) was established as a centralized analytical facility at NFC for qualifying raw materials, process intermediates and final products by employing state-of-the art analytical techniques for carrying-out estimations ranging from sub-ppm to percentage levels. It is to be noted that the chemical composition of nuclear materials like fuel and other reactor core components form a unique class and is distinct from other materials.

Therefore choice of selecting a method depends on many factors like nature of sample, type of matrix, elements to be analyzed & their concentration levels and requirement to give quick analytical feedback etc. Different sampling procedures and sample preparation steps are adopted depending on nature of the sample. In some cases sample pre-concentration steps are also incorporated in sample preparation step to increase the analytical signal of analyte. With ever growing demanding situation for supply of nuclear fuel, C.Lab is geared up to meet challenging analytical requirements of all production plants at NFC and average annual analytical load comes anywhere between 6-7 lakh of estimations. In order to manage such a massive analytical load a proper synergy between good chemistry, process conditions and analytical methods becomes a necessity and laboratory is able to meet this important requirement consistently. The present paper discusses the application different analytical techniques that are being followed for chemical characterization of different nuclear materials used in nuclear fuel fabrication process at NFC. As maintenance of health of all facilities is essential to provide un-interrupted analytical services, some significant maintenance works that are carried-out in lab are also given in the presentation. Besides this, communication of analytical results to different plants is a time bound activity and forms a very important step in realizing the goal of any testing lab, especially where analytical load is huge as seen in our laboratory. The presentation gives glimpses of type communication adopted in our lab to communicate the analytical results. Timely analysis and timely communication of analytical results are like two eyes of any testing laboratory's face. Even the slightest malfunctioning of one eye can eventually lead to defeat of organizational goal.

Invited Lecture -06: IMPORTANCE OF CHEMICAL IONIZATION (CI) IN MASS SPECTROMETRY.

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The most common technique in GCMS is Electron Ionization (EI) where the neutral organic molecules are bombarded or collided with electrons emitting from a rhenium filament under high vacuum at the standard 70 ev. In this energy most of the molecules produce molecular Ions (M+) and the fragment ions to give decent mass spectra that can be used for molecular formula and structure elucidation and also for computer search against a data base . The EI technique works well in majority of the compounds (may be around 95% of the molecules). However there are certain molecules such as the long chain aliphatic hydrocarbons, certain primary alcohols, aromatic compounds with aliphatic side chains, compounds containing acetyl groups, etc, whose molecular ion ( M+) are not stable due to the extra kinetic energy they get due to the electron collision, that they undergo fragmentation before they reach the detector. Such compounds do not display the M+ peak at all in the mass spectra or display a small M+ peak. As the M+ is indicative of the molecular weight of the compound a very highly characteristic information of the molecule will not be available for the investigator. Petroleum industry in particular was very much affected with this problem that many of the long chain hydrocarbons did not exhibit the molecular ion M+ in their mass spectra. This became a big challenge for Field and Munson working in Exxon Labs in USA in the 1960s to get an alternative technique called Chemical Ionization (CI) . In this technique a reagent gas such as methane is used as a reagent gas at a higher pressure inside a modified ion source and bombarded with electrons at 70 eV. The electrons ionize methane gas into various positive ions , predominantly $CH_5^+$, $C_2H_5^+$ and $C_3H_7^+$ termed as Bronsted acids, which can either donate a proton to a neutral molecule when introduced into the ion source or form positive adduct ions. As this form of ionization will be so soft, the $(MH^+)$ ions or the adduct ions formed will not have much kinetic energy thus not ending u with fragmentation. In such ionization the protonated molecular ion will be most prominent, thus by deducting 1 amu from the $MH^+$ ion will give the molecular ion of the compound thus leading to the molecular formula. This will mean that for compounds that are not amenable to EI mass spectrometry as their molecular ions are not stable to reach the detector, the CI technique offers an excellent alternative to get the important information such as the molecular weight of the molecule. While the CI technique used for producing the positive ions can be termed as Positive Chemical Ionization (PCI) the CI technique used to form the negative ions is called Negative Chemical Ionization (NCI) technique. This presentation will detail the general introduction to Electron Ionization and takes one through PCI and NCI, the theory, instrumentation and applications.

Invited Lecture -07: ANALYSIS OF STRUCTURES CONSIDERING SERVICE AND ACCIDENTAL LOADS TO ENSURE THE SAFETY

**G R Reddy**

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**Structures define the historical, economic, development and safety status of the country. Safety of the structures of heritage, residences, offices, schools, hospitals, power plants, chemical industries, roads, rail and connecting bridges has to be ensured throughout its life in order to protect the life and economy. It is ensured by correct designs, quality construction, and quality maintenance and assuring good health of structures throughout its life. For designing the structures both service loads and accidental loads are considered. Service loads are dead weights, live loads, winter-summer temperatures, environmental conditions (i.e. corrosive, wind etc) imposed loads. Accidental loads are extreme floods, wind, blasts, accidental fire, earthquake, tsunami etc. For designing conventional structures (e.g residences, offices, industrial structures not handling hazard chemicals) National standard specifies the accidental design loads. For large structures such as dame structures of hazardous chemical industries it specifies low probable design accidental loads with high return period. It also specifies to generate site dependent design basis loads for structures of nuclear facilities. It should be low probable (i.e. 10000 years return period for Nuclear Power Plants) and have high return periods based on the capacity of the plant. In order to have economical and safe designs one should follow graded approach in selecting the design basis accidental loads for the structures. All the structures of the plants not required to design for the same low probable design basis and in fact make systematic safety and design categorisation of structures and decide the probability of design basis. Once the loads are finalised design of the structural elements such as beams, columns, walls, slabs and components and components such as joints and foundations is made for service loads as per the design codes and books. Then detailed analysis is performed for accidental loads such as earthquakes, blast, air craft impact using numerical procedures for evaluating the element and component forces. Designs are checked for accidental loads along with service loads and if required sizes of elements and components are modified and finalised the design. Then good quality construction of**

the structure is made as per the design construction drawings. During service life of the structure for its intended use, evaluate its performance in terms of deflections and stress limits. If found not acceptable, repair and rehabilitate the structure. It is also required to continuously monitor the health of the structure using Non Destructive Testing and if required perform repair and rehabilitate the structure. As the age is increased, there is a possibility of increasing the demand of accidental loads such as earthquake, floods etc. In this situation evaluate the capacity of the structure and if not meeting the demand, retrofit the structure ensuring the safety of the structure under new demands. My presentation will describe the methods of generating design basis loads, detailed analysis and design of structures with proper safety and design classifications. The procedure of health evaluation of existing structures, such as bridges, structures of nuclear facilities will be described. Also details will be discussed on the methods of retrofitting of structures if the demand of the accidental loads is exceeding the capacity to ensure the safety.

## Invited Lecture -08: X-RAY EMISSION TECHNIQUES (XRF, PIXE, XANES, EXAFS) IN BIOANALYTICAL SCIENCES

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Since time memorial it has been shown that any solid can be studied by determining its elemental, molecular and structural composition. All these information can be obtained by using X-ray Emission Techniques consisting of probes such as photons as in XRF (X-ray Fluorescence), protons as in PIXE (Proton Induced X-ray Emission) and synchrotron as in XANES and EXAFS. In EDXRF photon sources are in the form of Radioisotopes such as Fe55, Cd109, and Am241. In PIXE we use the FOTIA (Folded tandem ion Accelerator) to extract protons of around 2-5 Mev with a current of around 10 nA to obtain a signature of elemental composition of the samples. In EXAFS a Synchrotron beam of elliptical focussing is used. The beamline has a 460-mm long Si (111) crystal having 2d value equal to 6.2709 Å mounted on an elliptical bender, which can bend the crystal to take shape of

an ellipse. I in my talk will summarise how all these probes can be usefully applied to study not only their structure, but also their correlation to other trace elements in biological systems. In EDXRF and PIXE, a Si (Li) detector, amplifier and MCA is included besides the excitation modes of photons and protons. My talk will elaborate on its experimental methods, results and conclusion in bio analytical applications such as soil, blood, ayurvedic drugs, sorghum plants, Indian spices, curcumin, uranium uptake in bioremediation studies, fertilizer use, etc.

## Invited Lecture -09: EFFICACY OF CUSTOMIZED BIOPOLYMERS, SYNTHETIC RESINS AND GRAPHENE OXIDE FOR DEFLUORIDATION OF WATER

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The contamination of groundwater due to fluoride is an important problem to be addressed. The challenges are manifold and it is imperative to develop effective methodologies for fluoride remediation. The WHO limit for fluoride in water is in the range 1.0–1.5 mg L-1. This limit has been set as maximum 1.0 mg L-1 in Indian standards. Hence, the contamination of groundwater due to fluoride is an important problem to be addressed. The challenges are manifold and it is imperative to develop effective methodologies. Considering the gravity of the problem, and to surmount some of the inadequacies in the existing methods it is imperative to develop effective adsorbents with removal efficiency in the permissible limit (1.0-1.5 mg L-1). The adsorbents tested by our group include a novel Al-Zr impregnated onto cellulose and polystyrene divinylbenzene resin for defluoridation. In addition, the interaction of aluminium (III) with graphene oxide was explored as a more viable alternative for defluoridation. This method resulted in a prototype development at the laboratory scale. Various adsorption parameters such as

pH, adsorbent dosage, isotherm studies, kinetics, thermodynamic parameters, aqueous phase volume, interfering ions and column studies were investigated in detail. The modified adsorbents were characterized thoroughly using BET surface area analysis, FT-IR spectroscopy, SEM, EDX, XRD and XPS studies. The concentration of fluoride was measured using a fluoride ion selective electrode. The concentration of metal ions ($Al^{3+}$ and $Zr^{4+}$) leached into the aqueous phase after adsorption (detectable if any) was checked using ICP-AES. The methods were applied in the defluoridation of real water samples.

Reference

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## Invited Lecture -10 : RHEOLOGY AND MECHANICAL CHARACTERISATION OF SOLID PROPELLANTS

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Solid propellants consist of a polymeric binder, oxidizer, metallic fuel and other additives. To start with it is a thick paste and on curing/cross linking it forms a solid. The flowing ability of the paste in its uncured condition dictates the type of casting process, casting devices and casting condition. The relationship between the shear rate and shear stress provided by the rheological behavior law allows the prediction of the flow rate of the slurry in the casting facilities and the calculation of the time and temperature of casting or the number of grains that can be cast with in a period of time compatible with the pot life of the propellant. Cured solid propellants belong to the class of viscoelastic materials which respond in a manner intermediate between the behavior of an elastic solid and a

viscous liquid. This unique property is due to the polymeric binder which comes under the class of rubbers or elastomers which can undergo large deformation under a small applied stress. A rubber like solid is unique in that its physical properties resemble those of solids, liquids and gases in various respects. It is solid like in that it maintains dimensional stability and its elastic response at small strains is essentially hookean. It behaves like a liquid because its coefficient of thermal expansion and isothermal compressibility are of the same order of magnitude as those of liquids. It resembles gases in the sense that the stress in a deformed rubber increases with increasing temperature much as the pressure in a compressed gas increases with increasing temperature. This gas like behavior was, in fact what first provided the hint that rubbery stresses are entropic in origin. The propellant in the solid from should have adequate mechanical properties to withstand the stresses imposed during thermal cool down after curing at high temperature, handling and firing. The mechanical properties depend on the characteristics of the binder, the percentage of solids and the particle size distribution. The response behavior of a solid propellant and loading conditions are very complex and the failure criteria should address a given loading condition for a given material and bond line properties.

## Invited Lecture -11 MASS SPECTROMETRIC TECHNIQUES FOR CHARACTERIZATION OF SPACE MATERIALS

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Over the years, mass spectrometry (MS) has evolved as a powerful tool for material characterization. MS technologies have continued to improve dramatically, and advanced strategies are increasingly becoming available. In recent decades, advancements are made in MS resolution, quantitative accuracy, and information science for appropriate data processing. Hyphenation of MS with other analytical techniques resulted in resolving complexities associated with material characterization. Direct PyrolysisMass Spectrometry (DPMS), wherein the sample is pyrolysed very close to the ion source and

the primary decomposition products are immediately driven to the detector for getting the mass spectra, is used for understanding the primary degradation process of materials. The use of hyphenated techniques like Gas Chromatography-Mass Spectrometry (GC-MS), Pyrolysis-Gas Chromatography-Mass Spectrometry (PyGC-MS), Thermogravimetry-Mass Spectrometry (TG-MS), Thermogravimetry - Gas Chromatography Mass Spectrometry (TG-

GC-MS), Thermal Desorption-Gas Chromatography-Mass Spectrometry (TDGC-MS) and Liquid Chromatography-Mass Spectrometry (LC-MS) in understanding reaction pathways for design of new materials, in identifying and quantifying evolved gases and components of desorption, and for characterization of materials are described in this paper. Though, MS has applications in basic sciences, forensic medicine, plant science, and natural products, this lecture is focused towards the applications of the above mentioned techniques in characterization of space materials.

## Invited Lecture -12: BIOSENSING AND BIOIMAGING: A QUANTUM DOT APPROACH

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Quantum dots (QDs) are semiconductor nanocrystals whose electrons and holes are quantum- confined in all three spatial dimensions. They are considered as a bridge between bulk materials and atomic or molecular structures and are characterized by excellent optical and electrical properties. These nanoparticles have size-tunable emission, strong light absorbance, and very high quantum yield, long life time and photostability. Unique optical property makes it a promising alternative to organic dyes for labeling, imaging, tracking, detection, and therapy. Highly luminescent and stable QDs are prepared by coating the core with another material, resulting in core-shell quantum dots. To make them useful for biomedical applications, QDs are often conjugated to biological molecules without disturbing the biological function of these molecules. The broad absorption spectra of the QDs allows single wavelength excitation of emission from

different-sized QDs. With these advantages, many novel applications based on QD formulations have been developed for bio-sensing and bio-imaging, such as cell tracking and multichannel and multimodel imaging. The most commonly studied and used QD is cadmium selenide. As they are made up of heavy metals, the potential toxicity of the QDs is a general concern. The limitation of heavy metal– containing QDs has lead to extensive research interests in exploring alternative strategies for the design of fluorescent nano materials with high quantum yield and biocompatibility. This presentation will include the synthesis of cysteine functionalized cadmium selenium quantum dots and its use as a multi analyte sensor for copper and creatinine. A hybrid system of carbon nanotube and quantum dots facilitated a multifunctional system for photothermal therapy and imaging. Yet another study provided femto molar level detection of endosulfan using citrate capped quantum dots. Our experience in working with QD has paved the way for novel biological applications of the same, including cell imaging, targeted therapy and imaging, and biosensing leading to molecular diagnostics.

## Invited Lecture -13: MICROSTRUCTURE, MATERIAL PROPERTIES AND ANALYTICAL METHODS

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The microstructure of materials is an essential feature for the design of engineering structures with improved performances. Scientists can tailor the microstructure of a material to give it specific properties. With clear interpretation and sound understanding, microstructure can be exploited to provide information on mechanical, chemical and other properties. Microstructural changes can be induced in almost all engineering materials to alter their mechanical properties. This is usually achieved through thermal treatments involving heating and cooling under controlled conditions. Such thermomechanical treatments often produce properties that cannot be obtained in any other way. In some cases, different properties are desired in the bulk than in the surface

of a part. In these instances, specialized thermal treatments involving surface diffusion are used. It may be noted that insufficient attention to control microstructures can lead unpredicted properties, inconsistent behaviour and premature failure of materials. In last decade, a huge effort has been made in the direction of conceiving new materials with specific microstructures for the sake of producing exotic mechanical behaviours both in the static and the dynamic regime. Also new materials for use in some of the extreme conditions of stress, temperature, and environment rely heavily on the microstructural condition. Furthermore, the materials used in aerospace and nuclear energy systems needs higher reliability and therefore requires close control on the microstructures. The specifications for such materials insist on information such as grain size, volume fraction of phases, mean free path of particles, texture details, etc.

To measure the above properties precisely, one needs good analytical tools. Powder X-ray diffraction is a fundamental characterisation technique for every material which allows a fast and reliable identification of the phases present in the sample by comparison with a database. Quantitative phase analysis is achieved by the Rietveld method, while the analysis of lattice parameters delivers further information on solid solution formation and composition. Furthermore, microscopic texture investigations, microscopic roughness profile determination and in situ tension and compression tests are carried out to extract detailed information. A variety of optical microscopic techniques are used along with scanning electron microscope and an analytical transmission electron microscope to yield variety of information on microstructures. An Energy Dispersive X-ray Spectroscopy(EDS) system permits quantitative elemental analysis, line scans and quantitative elemental mapping.

Invited Lecture -14 MULTI-MATERIAL SOLUTIONS FOR CRITICAL AEROSPACE APPLICATIONS


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Advances in aerospace and related technologies have, to a large extent, been boosted by the advances made in the understanding of the materials and their processing technologies. One of the demanding aspects of materials for space applications is the challenges from contradicting requirements such materials having high strength with low density, extreme temperature gradients in small thicknesses such as those in thrust chambers and cryogenic propellant tanks. To meet this conundrum, often materials scientists provide multi-material solutions which can be amply seen in the area of rocketry and related technologies. In this presentation on multi-material solutions, several examples will be presented depicting the importance of multi material solutions in the success of Indian space program. Author's experience in providing solutions to some of the problems encountered will be shared.

## Invited Lecture -15 RECENT ADVANCES IN THE APPLICATION OF ANALYTICAL TECHNIQUES IN CATALYST MANUFACTURING AND APPLICATIONS

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The consistent innovation and technology up gradation has open a new arena in the field of catalysis. Use of catalyst is not only restricted to the conventional chemical processes or technologies but started entering into the various chemical domains. The recent advances have brought the focus of analytical scientists into the characterization methods and elemental estimations for verification and validation of catalyst performance. Due to the mushrooming growth in instrumental techniques and availability of various instruments at cheaper rates with a lot of variability in the market has made the task of scientists easier. The accuracy and precision required during development and manufacturing of catalysts have brought an emphasis on making right quality of catalyst meeting defined

specifications. The challenge for the analytical scientists is to develop analytical methods for the estimation of various elements for certifying the elemental composition of the catalyst in order to minimise the risk of performance. Though metal content estimation by using ICP-OES and X-ray Fluorescence Spectrometers has simplified the estimation process with respect to conventional techniques but the challenge remains in the sample preparation techniques for getting right results. As catalysts are generally patented products in the market, standard references are not available for verification, calibrations and standardizations in the market. Transition metals like cobalt, nickel and molybdenum in heterogeneous catalysts is generally present in high percentages whereas precious metals like silver, platinum, palladium and rhodium are used in ppm levels. This creates a challenge for the analyst to work in a test sample from macro to microgram level of estimations. In zeolites based products, the sample preparation throws a big challenge before the estimation work begins. In this technical presentation, the focus is on the challenges faced for elemental estimations during analytical method developments for heterogeneous catalysts, zeolite based catalyst and catalytic converters using ICP-OES and X-ray Fluorescence spectrometer (XRF). The elemental interferences during estimations in both the techniques have also been compared.

Invited Lecture -16: ACTIVATED CARBON FOR AIR PURIFICATION

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Enormous amount of pollutant gases from vehicular / industrial emissions lead to deterioration of the quality of atmospheric air . It is essential to design suitable methods to control the level of such pollutants. Adsorption on suitable adsorbents or catalytic conversion to environmental friendly products seems to be the ideal solution for ensuring a clean atmosphere. Removal of pollutants over activated alumina, activated carbon and

zeolites are extensively studied. Priority is often directed towards a technically and economically feasible option, preferably focusing more towards an activated carbon-based product with enhanced performance. Activated carbon or its doped versions are widely used in trapping impurities like H2S, mercaptans , CO, CO2, SO2, NH3, HCN and several volatile organic compounds. Regenerable type of activated carbonbased adsorbents for repeated cyclic operations with scope for favorable adsorption, desorption and further utilization at moderate conditions of temperature and pressure has received wide attention over the recent years. Modification of pore structure and basicity of activated carbon through chemical and thermal treatments may yield a product having improved adsorption capacities with favorable desorption at mild conditions over repeated cycles of adsorption / regeneration. Amines are the most commonly used liquid absorbent used for removing CO2. Environmental toxicity of the solvents and economic aspects as well need to be considered while designing a suitable CO2 storage / removal system. Amines impregnated on activated carbon is mostly employed for removal / storage of carbon dioxide. Adsorption desorption process has to be energetically favorable during regeneration compared to absorption over solvents. High CO2 adsorption capacity, high selectivity for CO2, fast adsorption desorption kinetics, adequate mechanical strength of adsorbent particles and finally regenerability without any significant cyclic performance loss are the basic requirements of an activated carbon-based adsorbent for CO2 removal. There is enough scope for exploring the possibilities of immobilizing various types of solvents over activated carbon. Alkali doped activated carbon for adsorbing acidic vapors, acid doped one for ammonia and amines, copper for strong acidic and basic gases, finely divided iron for CO removal are a few examples that can illustrate adsorptive properties of activated carbon-based traps.

## ISAS EC Meeting

EC Meeting of ISAS was held at the board room of Hotel Apollo Dimora. All EC members except Shri S K Malhotra, Dr D K Singh, Prof Sriman Narayanan, Dr Veerabadran, Dr Roopa Bose and Shri N Kalyan were present for the EC Meeting. Several important decisions were taken during the EC meeting.

EC Meeting in Progress

## General Body Meeting

General Body Meeting of ISAS was held at 18.00 hours on 20th September 2019.

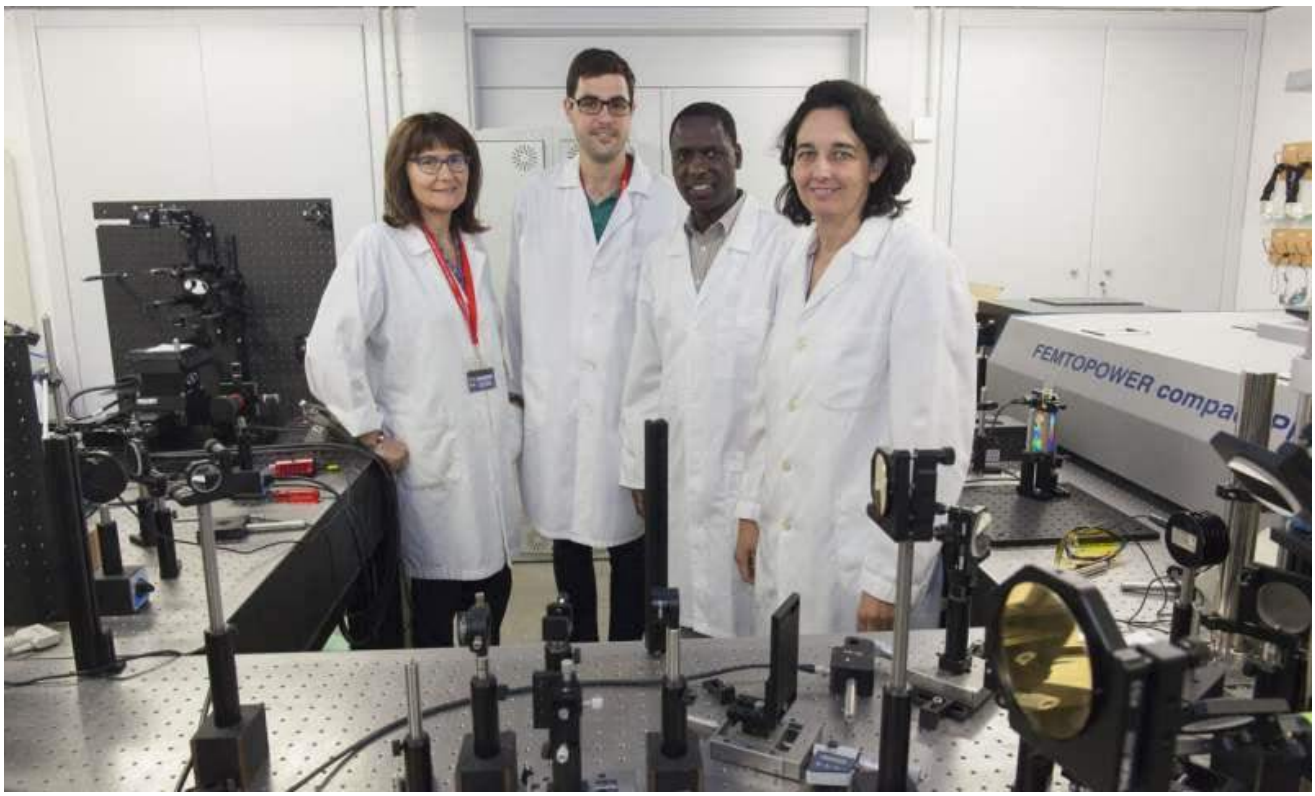

Dr P P Chandrachoodan presided over the meeting. In the absence of Dr D K Singh, Shri Arunkumar , Joint Secretary took his chair on the dais along with Dr P. Unnikrishnan. Dr KKA Rashid Election committee convener read out the name of members of the new EC. This was followed by presentation of report by Dr Daisy Joseph secretary of Previous EC. Dr P K Sharma presented the audited accounts report of last year. President of ISAS Dr Chandrachoodan requested the members to actively participate and work hard to implement the tasks assigned to each member and group. Important decisions were taken in the meeting. The meeting was over at 19.30 Hours.

**ISAS Members attending AGM**

## LATEST NEWS

# Researchers develop an optical sensor that detects very low glucose concentrations

by Asociacion RUVID




**The Optical Research Group. Credit: Universitat Jaume I**

The Optical Research Group of the Universitat Jaume I (GROC-UJI) has developed an optical nanoparticle sensor capable of detecting very low glucose concentrations such as those present in tears by means of fluorescent carbon quantum dots.

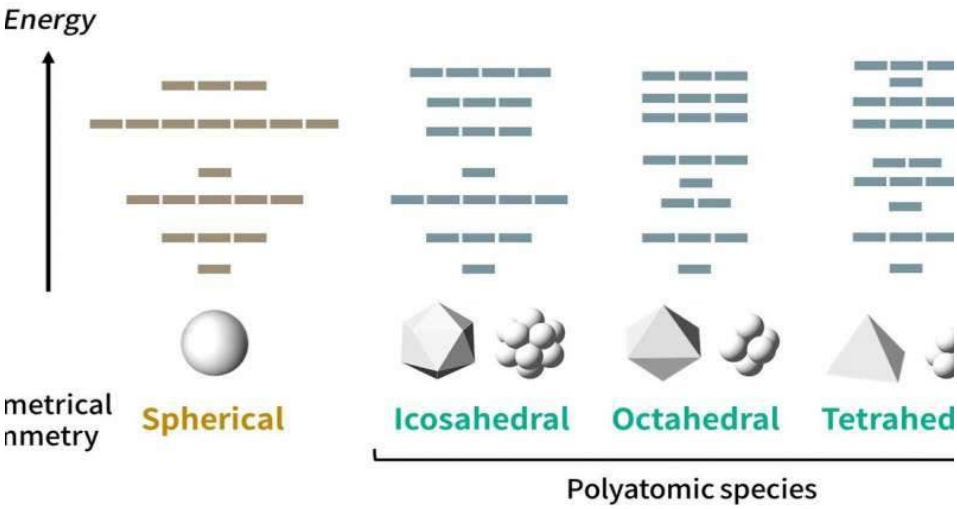
The main objective of this project is to create a tool for the diagnosis of non-invasive diabetes through the detection of ocular glucose in vitro, which can be integrated into a smartphone for both clinical and private use. Therefore, diabetics would not have to prick themselves several times a day to control their glucose levels, thus avoiding the discomfort it entails. In addition, the use of mobile phones would enable the systematic collection and management of electronic glucose level records to reduce errors and improve diabetes control.

Laser-based synthesis enables the development of green and sustainable nanotechnology, because it does not require an excess of polluting chemical products, nor does it necessarily produce waste. Furthermore, the functionalization of nanoparticles is simple and efficient, since it is obtained in situ during the synthesis process with a pulsed laser. Finally, thanks to the manufacturing process, nanosensors are not blocked by any other chemical component or residue that may cause unwanted chemical effects.

The researchers developed a technique to produce a single carbon quantum dot capable of detecting very low glucose levels, thanks to its 63 percent quantum efficiency in fluorescence, and with a high photo-stability demonstrated for more than 15 hours. This new type of carbon quantum dot opens the door to numerous applications in companies specializing in the synthesis of nanoparticles.

## Discovery of periodic tables for molecules

by Tokyo Institute of Technology



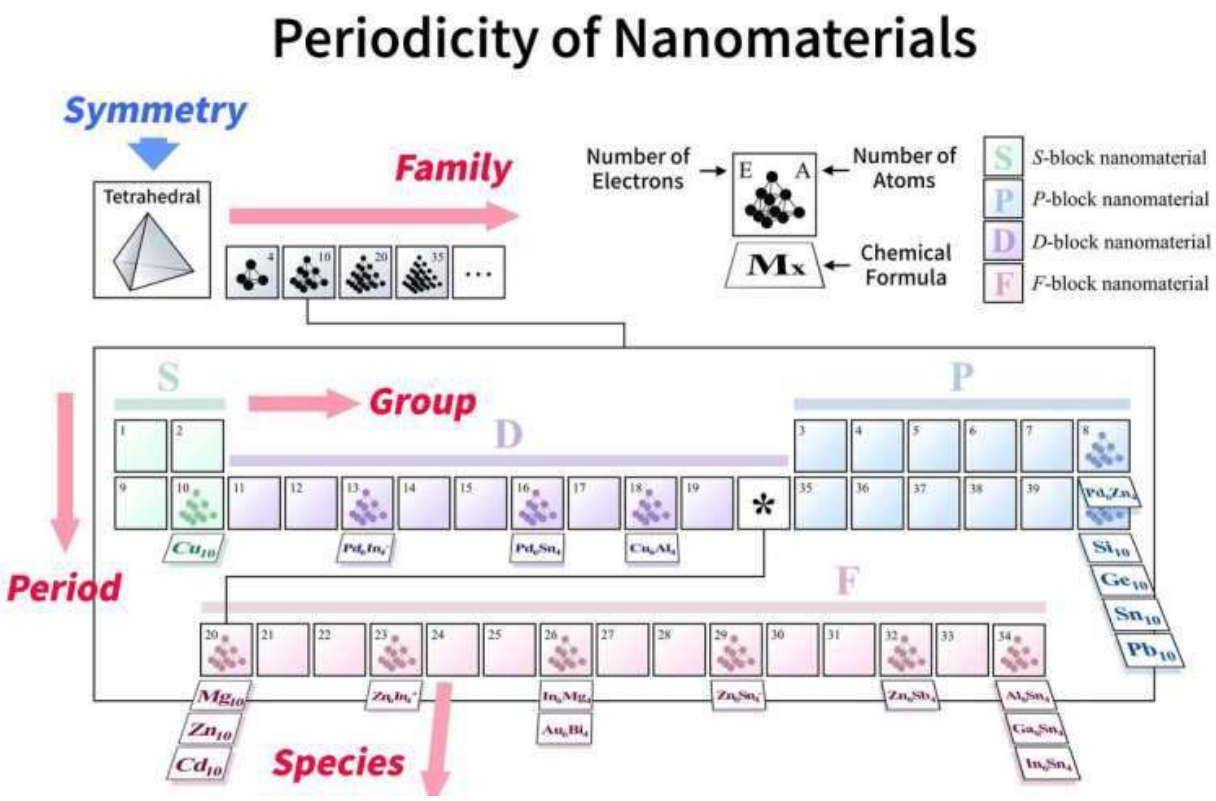

The proposed model accounts for orbital patterns obeying certain rules for many types of symmetries. Although a sphere has the highest geometrical symmetry, there is no real polyatomic species with a spherical symmetry. Credit: Tokyo Tech

The periodic table of elements was proposed in 1869, and thereafter became one of the cornerstones of the natural sciences. This table was designed to contain all the elements found in nature in a special layout that groups them in rows and columns according to one of their most important characteristics, the number of electrons. Scientists have used the periodic table for decades to predict the characteristics of the then-unknown elements, which were added to the table over time.

Could there be such a periodic table for molecules? Although some researchers have thought about this possibility and proposed periodic rules for predicting the existence of certain molecules, these predictions were valid only for clusters of atoms with a quasi-spherical symmetry, because of the limitations of their own theory. However, there are many clusters of atoms with other shapes and other types of symmetries that should be accounted for with a better model. Thus, a research team from Tokyo Tech, including Dr. Takamasa Tsukamoto, Dr. Naoki Haruta, Prof. Kimihisa Yamamoto and colleagues, proposed a new approach to build a periodic table for molecules with multiple types of symmetries.

Their approach is based on a keen observation of the behavior of the valence electrons of atoms that form molecular clusters. The valence electrons can be regarded as "free" electrons in atoms with an outermost orbital, and thus they can

interact with the electrons of other atoms to form compounds. When multiple atoms form a cluster with a symmetrical shape, their valence electrons tend to occupy specific molecular orbitals called as "super-atomic orbitals," in which they behave almost exactly as if they were the electrons of a huge atom.




In the proposed framework, there would be sets of tables for each type of symmetry organized according to four parameters: groups and periods (number of electrons), families (number of constituting atoms), and species (type of constituting elements). Credit: Tokyo Tech

By considering this fact and analyzing the effects of the structural symmetries for clusters (Fig. 1), the researchers proposed "symmetry-adapted orbital (SAO) models," which are in agreement with multiple known molecules as well as state-of-the-art quantum-mechanical calculations. The new periodic tables, which would be created for each symmetry type, would actually be four-dimensional, as shown in Fig. 2, because the molecules would be arranged according to four parameters: groups and periods (based on their "valence" electrons, similar to the normal periodic table), species (based on the constituting elements), and families (based on the number of atoms).

The SAO approach is very promising in the field of materials design. "Modern synthesis techniques enable us to produce many innovative materials based on the SAO model, such as lightweight magnetic materials," states Prof. Yamamoto. The road ahead for scientists lies in further expanding these tables to molecular clusters with other shapes and symmetries and predicting stable molecules that have yet to be developed. "Among the infinite combinations of constitutive elements, the proposed periodic table will be a significant contribution to the discovery of novel functional materials," concludes Prof. Yamamoto.

SEPTEMBER 17, 2019

# A safer way for police to test drug evidence

by National Institute of Standards and Technology

Credit: CC0 Public Domain

Scientists have demonstrated a way for police to quickly and safely test whether a baggie or other package contains illegal drugs without having to handle any suspicious contents directly. The new technique can limit the risk of accidental exposure to fentanyl and other highly potent drugs that can be dangerous if a small amount is accidentally inhaled.

The proposed method involves swiping the outside of a baggie then analyzing the swipe for drugs in the same way that airport security officers swipe carry-on

luggage to detect explosives. Researchers at the National Institute of Standards and Technology (NIST) and state forensic laboratories in Maryland and Vermont have demonstrated that this approach can reliably predict whether a package contains fentanyl, even if mixed with cocaine, heroin or other substances. Their research was published this week in *Forensic Science International*.

"What's needed is a fast and safe way to screen drug evidence so that it can be handled appropriately," said Ed Sisco, a research chemist at NIST and the lead author of the study. For instance, hazardous packages can be flagged so they are opened only under a laboratory fume hood.

The swipe method works because opening a bag contaminates its outside surface. "If you've ever opened a bag of flour, you know that some of it poofs into the air," said NIST co-author Elizabeth Robinson. "That's just the way lightweight powders behave."

Before fentanyl became a common street drug, police often field-tested evidence by scooping a bit of powder into a solution that would change color depending on what type of drug was present, if any. But many police departments now discourage or prohibit such "color tests" in the field for safety reasons. Instead, officers must send the suspected drugs to a crime lab, then wait for a result before getting a search warrant or making an arrest.

Particles can contaminate the outside of a baggie when it is opened. This video was shot with a technique called laser light sheet flow visualization and used talc as a safe powder substitute. Credit: E. Sisco and M. Staymates/NIST

Amber Burns, manager of the Maryland State Police forensic chemistry lab and a co-author of the study, said that she gets a lot of rush requests, and each request currently requires a full work-up of the evidence. Her lab plans to install an instrument called a Direct Analysis in Real Time Mass Spectrometer (DART-MS) to do the quick screening, which should speed up the process considerably. "They just need to bring me the swipe, and they can be on their way in two minutes," she said.

Alternately, departments can purchase instruments that fit in a police vehicle. Wherever the screening is done, it provides only a preliminary identification. To bring a criminal case to court, a complete work-up using standard laboratory equipment would still be necessary.

To conduct this study, the NIST scientists teamed up with Burns and her counterpart at the Vermont Forensic Laboratory, Rebecca Mead, who was also an author of the study. When suspected drug evidence arrived at their labs, Burns and Mead swiped the outside of the packages. Most were plastic baggies, though they also included envelopes, tinfoil and pill bottles. The chemists also dissolved a small amount of the suspicious material in alcohol and put a drop of the resulting solution onto a second swipe for comparison. They then sent the pair of swipes to NIST for analysis.

The NIST authors received swipes from 191 suspicious packages, which they analyzed using DART-MS and another technique called liquid chromatography-mass spectrometry (LC/MS). Those swipes contained a panoply of contemporary street drugs, including several types of fentanyl as well as heroin, cocaine, methamphetamines, ketamine and others. Many of the cocaine and heroin samples were mixed with fentanyl. The swiped packages also contained plant material sprayed with synthetic cannabinoids, which are often marketed as K2 or Spice.

Two of the packages contained carfentanil, a super-potent form of fentanyl, sometimes used as a large animal tranquilizer, which can be particularly dangerous for police and first responders. Carfentanil is roughly 5,000 times as potent as heroin.

Particles can also contaminate the outside of a baggie when it is prepared for sale. This video was shot with a technique called laser light sheet flow visualization and used talc as a safe powder substitute. Credit: E. Sisco and M. Staymates/NIST

The authors found that swiping the outside of a package correctly predicted its contents 92% of the time. In cases involving fentanyl and other opioids, the outside of the package predicted the contents 100% of the time. In other words, if

the goal is to flag fentanyl-containing packages for special handling, the technique worked every time.

The 8% of non-matches involved cases where several bags of different material were placed together by police into a single evidence bag, allowing for cross-contamination. Also, the technique did not work in most cases involving plant material in heat-sealed bags.

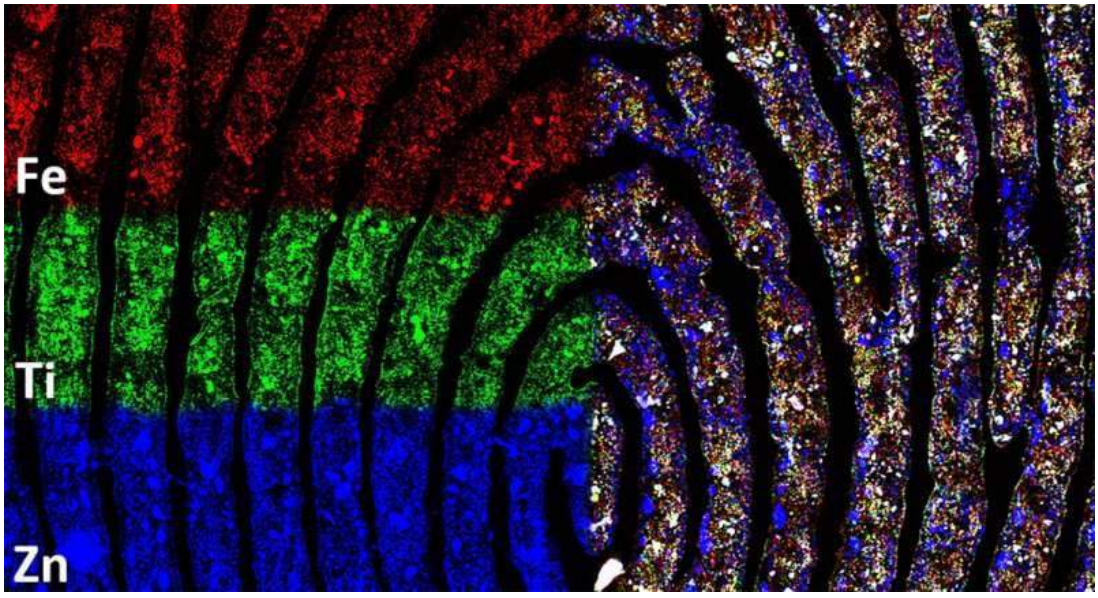
This swipe technique will do more than help police get faster answers when investigating drug crimes. It will also help at crime labs. At the Maryland lab, Burns said that upon receiving evidence they use color tests—the same tests that officers once used in the field—to quickly get an idea of what's in the bag so they can line up the right types of laboratory analysis. But those color tests don't detect many of the new designer drugs that make up an increasing fraction of the caseload.

The swipe test will work for this, however. "We plan to use this to optimize our whole workflow," Burns said.

SEPTEMBER 11, 2019

# Analysis of fingermarks with synchrotron techniques provide new insights

by Australian Nuclear Science and Technology Organisation (ANSTO)



Credit: Australian Nuclear Science and Technology Organisation (ANSTO)

The findings by lead researchers Prof Simon Lewis and Dr. Mark Hackett may provide opportunities to optimise current fingermark detection methods or identify new detection strategies for forensic purposes.

Latent fingermarks are generally described as those requiring some process to make them readily visible to the eye. These fingermarks are typically made up of natural skin secretions, along with contaminants (such as food or cosmetics) picked up from various surfaces.

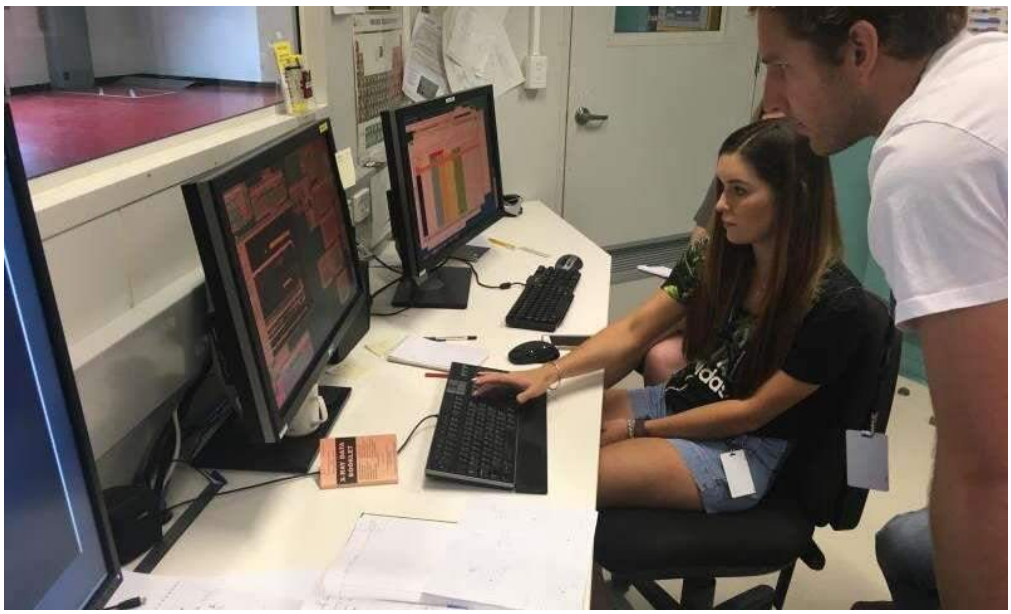
The detection of latent fingermarks is often crucial in forensic investigations, but this is not always a straightforward task.

"We know that there are issues in detecting fingermarks as they get older, and also under certain environmental conditions," said Lewis, whose main research focus is forensic exchange evidence.

"In order to improve our ability to detect fingermarks, we need to understand the nature of fingermark residue, and this includes both the organic and inorganic components. Many chemical components in fingermark residue are present at very low levels, and we don't know how they are distributed within the fingermark. This is what took us to the Australian Synchrotron."

To date, most fingermark research has largely focused on the organic material in residues. Consequently, a gap in fundamental knowledge exists when it comes to inorganic components such as metals. "Our purpose in using X-ray fluorescence microscopy was to determine if there were inorganic components present in the fingermark that we could use as a target for detection techniques, or to better understand how our current detection methods are working," said Lewis "XFM can detect elements with spatial resolution at sub-micron length scales directly and rapidly. Importantly, it reveals the location of elements within a sample, which is valuable in forensic science and a range of other disciplines," said Dr. Daryl Howard, XFM Instrument Scientist, who assisted with the measurements.

Dr. Mark Hackett, who is an expert in mapping metals for health and bioscience applications, said, "We have been able to use X-rays generated by the synchrotron to study how trace amounts of metals and metal ions can be transferred to a fingermark due to handing everyday items that range from coins to cosmetics. Further, we can use the synchrotron to determine the identity of the metals or metal ions, to differentiate between those that do and don't originate in the body. "



Curtin PhD student Rhiannon Boseley sets up a scan on XFM under the careful eye of Dr Mark Hackett. This research is part of her PhD project “Recovery and Enhancement of Fingermarks and other Physical Evidence: Towards Improved Protocols for Crime Scene Investigation." Credit: Australian Nuclear Science and Technology Organisation (ANSTO)

"I was truly surprised at the amount of metals from external sources, that we appear to carry on our fingertips" said Hackett.

Because the presence of metals can impact detection of latent fingermarks, the investigators were interested in their concentration and location within the residue.

The findings open up the possibility of understanding why fingermarks from some individuals are more easily detected than others, and this information could be used to improve detection methods.

"Our most spectacular image captured the element titanium on one fingermark, which really lit up the fingermark. Titanium is commonly used in face make-up, and even after being rinsed with water, the element persisted. This may have significance in relation to the longevity and detectability pf such impressions" said Lewis

The researchers also tried washing fingermarks for 30 minutes before XFM imaging to see what effect this might have on the residue, as detecting finger marks on wetted surfaces is known to be challenging. Following immersion in water, elements were leached out of the sweat component of the residue, but preserved in the oily matrix.

Drs Mark Tobin and Pimm Vongsvivut assisted with infrared microspectroscopy measurements at the Australian Synchrotron, which was used to reveal the distribution of organic components of finger marks, such as the oily matrix or water-soluble organic material found in sweat. In particular, the combination of infrared microscopy and X-ray fluorescence microscopy revealed that water-soluble organic material also contained much of the inorganic material found naturally in fingermark residue.

In earlier work published in 2018 in *Analyst*, Dorakumbura et al characterized the highly variable and complex nature of the organic compounds in fingermarks. They used a type of infrared spectroscopy at the Australian Synchrotron, in combination with Raman microscopy at Curtin University, to directly and non-destructively image the numerous chemical components within fingermarks at the micron scale.

It also provided the first direct evidence that the chemical composition and distribution within finger marks reflect water-in-oil and oil-in-water emulsions.

"We are not suggesting that routine application of synchrotron techniques for forensic testing is likely. It has however given us a greater understanding of the chemical complexity, transfer processes and persistence of material associated with latent finger marks," said Lewis.

# IPCC: Rapid changes are forcing people to fundamentally alter their ways of life

25 Sep 2019 Liz Kalaugher

.(Image;courtesy: iStock/borchee)

**The IPCC says its Special Report on the Ocean and Cryosphere in a Changing Climate, released this morning, provides new evidence for the benefits of limiting global warming to the lowest possible level – in line with the goal governments set themselves in the 2015 Paris Agreement.**

**"The open sea, the Arctic, the Antarctic and the high mountains may seem far away to many people," said Hoesung Lee, Chair of the IPCC. "But we depend on them and are influenced by them directly and indirectly in many ways – for weather and climate, for food and water, for energy, trade, transport, recreation and tourism, for health and wellbeing, for culture and identity."**

**According to Lee, if we reduce emissions sharply, consequences for people and their livelihoods will still be challenging, but potentially more manageable for those who are most vulnerable. "We increase our ability to build resilience and there will be more benefits for sustainable development," he said.**

A total of 670 million people in high mountain regions and 680 million people in low-lying coastal zones depend directly on the ocean and cryosphere, according to the IPCC. Four million people live permanently in the Arctic region, meanwhile, and small island developing states are home to 65 million people.

"The world's ocean and cryosphere have been 'taking the heat' from climate change for decades, and consequences for nature and humanity are sweeping and severe," said Ko Barrett, Vice-Chair of the IPCC. "The rapid changes to the ocean and the frozen parts of our planet are forcing people from coastal cities to remote Arctic communities to fundamentally alter their ways of life," she added. Barrett explained that the report provides a complete picture of water – "the lifeblood of our planet" – around the globe and its interconnections.

Released this morning in Monaco, the report details that the oceans are warmer, more acidic and less productive. Melting glaciers and ice sheets are causing sea level rise and coastal extreme events are becoming more severe. The special report was produced by more than 100 authors from 36 countries, and references roughly 7,000 scientific publications. While sea-level rose around 15 cm globally during the 20th century, it is now rising more than twice as fast, at 3.6 mm per year, according to the report. By 2100 if greenhouse emissions continue to increase strongly, sea-level rise could reach 60-110 cm.

"In recent decades the rate of sea level rise has accelerated, due to growing water inputs from ice sheets in Greenland and Antarctica, in addition to the contribution of meltwater from glaciers and the expansion of warmer sea waters," said Valérie Masson-Delmotte, Co-Chair of IPCC Working Group I. "This new assessment has also revised upwards the projected contribution of the Antarctic ice sheet to sea level rise by 2100 in the case of high emissions of greenhouse gases. The wide range of sea level projections for 2100 and beyond is related to how ice sheets will react to warming, especially in Antarctica, with major uncertainties still remaining."

# MasSpec Pen' for accurate cancer detection during surgery

by American Chemical Society

The MasSpec Pen (left) could someday help cancer surgeons determine the edges of tumors in the operating room; researchers used it to analyze thyroid tissue ex vivo (right) and are now testing it in vivo with human patients. Credit: Eberlin lab/University of Texas at Austin

A major challenge for cancer surgeons is to determine exactly where a tumor starts and where it ends. Removing too much tissue can impair normal functions, but not taking enough can mean the disease could recur. The "MasSpec Pen," a handheld device in development, could someday enable surgeons to distinguish between cancerous and healthy tissue with greater certainty in seconds, while in the operating room. Today, researchers report first results of its use in human surgeries.

The researchers will present their findings at the American Chemical Society (ACS) Fall 2019 National Meeting & Exposition.

"It's been shown with extensive clinical data that highly effective surgeries are those that remove the most cancer, but also preserve the most normal tissue," says principal investigator Livia Eberlin, Ph.D. "We created the MasSpec Pen because we thought it would be incredible if there was a technology that could actually provide molecular information right in the operating room in living tissues within a time frame that could expedite surgical decisions."

Surprisingly, the most common method that medical professionals currently use to determine tumor margins or verify a diagnosis is 100 years old: histopathology. With this technique, a tissue sample is extracted during surgery and taken to a laboratory. The sample is flash-frozen, sectioned, stained and examined with a microscope. In total, this procedure can take an average of 30 minutes. Meanwhile, the patient, who is still under anesthesia, and the surgeon are left waiting. In addition, while histopathology is effective for many surgeries, especially for cancers, the process can be subjective because artifacts from the freezing process can complicate interpretation, Eberlin explains.

To overcome these challenges, Eberlin and colleagues at the University of Texas at Austin developed the MasSpec Pen, a handheld and biocompatible device connected to a high-performance mass spectrometer. The device rapidly identifies the molecular profile of tissue exposed during a surgery by first depositing a small droplet of water on the tissue surface for about three seconds. Next, the droplet is transferred to the mass spectrometer, where molecules from the tissue are identified. Finally, machine learning algorithms comb through the molecular information and provide a predictive diagnosis that surgeons can act on.

"We have developed the MasSpec Pen so that the surgeon just has to touch the tissue with the pen, and trigger the system with a foot pedal," Eberlin says. "From there, everything is coded and automated so that the whole process is completed in under 10 seconds." A video of the device in action is posted here.

So far the MasSpec Pen has been tested on more than 800 human tissues ex vivo, including normal and cancerous breast, brain, pancreatic, thyroid, lung and ovarian tissues. The team is now testing the MasSpec Pen in vivo, in an ongoing clinical study at the Texas Medical Center with human patients during thyroid, breast and pancreatic cancer surgeries. Freshly excised patient tissue also is being analyzed and is showing promising results.

"We are continuing research and development of this technology in my lab by continuing to improve our technology and validating its performance across different cancer types," Eberlin says. "We are also exploring new applications

**in surgery including minimally invasive surgical procedures, as well as outside the operating room in forensics and agricultural applications."**

# Researchers create inexpensive system to test for dangerous levels of lead in water

*Alina Shrourou, B.Sc. (Editor)*Sep 26

The discovery of lead in Flint, Michigan's drinking water drew renewed attention to the health risks posed by the metal. Now researchers at the University of Houston have created an inexpensive system using a smartphone and a lens made with an inkjet printer that can detect lead in tap water at levels commonly accepted as dangerous. The system builds upon earlier work by Wei-Chuan Shih, associate professor of electrical & computer engineering, and members of his lab, including the discovery of an inexpensive elastomer lens that can convert a basic smartphone into a microscope. The latest discovery, described in the journal *Analytical Chemistry*, combines nano-colorimetry with dark-field microscopy, integrated into the smartphone microscope platform to detect levels of lead below the safety threshold set by the Environmental Protection Agency.

"Smartphone nano-colorimetry is rapid, low-cost, and has the potential to enable individual citizens to examine (lead) content in drinking water on-demand in virtually any environmental setting," the researchers wrote. Even small amounts of lead can cause serious health problems, with young children especially vulnerable to neurological damage. EPA standards require lead levels in drinking water to be below 15 parts per billion, and Shih said currently available consumer test kits aren't sensitive enough to accurately detect lead at that level.

By using an inexpensive smartphone equipped with an inkjet-printed lens and using the dark-field imaging mode, researchers were able to produce a system that was both portable and easy to operate, as well as able to detect lead concentrations at 5 parts per billion in tap water. The sensitivity reached 1.37 parts per billion in deionized water.

Shih and his students last year published an open-source dataset in *Biomedical Optics Express*, explaining how to convert a smartphone equipped with the elastomer lens into a microscope capable of fluorescence microscopy. That paper has been the journal's most frequently downloaded paper since its publication.

The latest application incorporates color analysis to detect nanoscale lead particles. In addition to Shih, researchers on the project include first author Hoang Nguyen and

Yulung Sung, Kelly O'Shaughnessy and Xiaonan Shan, all with the UH Department of Electrical & Computer Engineering. (O'Shaughnessy was a summer intern from the University of Cincinnati under the National Science Foundation's Research Experiences for Undergraduates program.) Applying the dataset published , the researchers built a self-contained smartphone microscope that can operate in both fluorescence and dark-field imaging modes and paired it with an inexpensive Lumina 640 smartphone with an 8-megapixel camera. They spiked tap water with varying amounts of lead, ranging from 1.37 parts per billion to 175 parts per billion. They then added chromate ions, which react with the lead to form lead chromate nanoparticles; the nanoparticles can be detected by combining colorimetric analysis and microscopy. The analysis measured both the intensity detected from the nanoparticles, correlating that to the lead concentration, and verified that the reaction was spurred by the presence of lead. The mixture was transferred to a polydimethylsiloxane slab attached to a glass slide; after it dried, deionized water was used to rinse off the chromate compound and the remaining sediment was imaged for analysis.

The microscopy imaging capability proved essential, Shih said, because the quantity of sediment was too small to be imaged with an unassisted smartphone camera, making it impossible to detect relatively low levels of lead. Building upon the smart phone microscope platform to create a useful consumer product was key, Shih said. "We wanted to be sure we could do something that would be useful from the standpoint lead at the EPA standard," he said.

http://www.uh.edu/news-events/stories/

# Biosensors and Your Health

# What's Your Body Trying to Tell You?

**Biosensors are already used for many health issues. They can:**

- **continuously detect your blood pressure without a blood pressure cuff**
- **help a person with diabetes maintain safe blood sugar levels without a finger prick**
- **advise a doctor on whether to start a patient on an antibiotic treatment for strep throat**
- **tell whether a woman is pregnant**
- **record the levels of oxygen in a patient's blood without a lab test**

- sense the amount of alcohol in a person's system from their skin
- diagnose patients in low-resource areas that don't have lab facilities

Your body alerts you to many aspects of your health. Your stomach growling tells you when to eat. A powerful yawn lets you know you're tired. Your body gives off many other valuable signals, but requires technology to detect them. Scientists are looking for new ways to track and use your body's signals to improve your health and manage disease.

Physical activity trackers and step counters are now helping people develop and maintain healthy habits. These devices have also opened doors for people to participate in health research. Now, researchers are designing more advanced devices called biosensors that measure biological, chemical, and physical signs of health.

"The variety of biosensors used by researchers, clinicians, and people from every walk of life is growing," says Dr. Šeila Selimović, a biosensors expert at NIH. "Some speed up test results so treatments can be started promptly. Others provide the benefits of continuous monitoring of health conditions. [Biosensors] function in fascinating ways. [They use] chemical attraction, electrical currents, light-detection systems, and compact wireless-sensing technologies."

The mercury thermometer is one of the earliest biosensor technologies used in medicine. In modern thermometers, mercury has been replaced by safer temperature-sensitive probes. But the goal is still the same: to detect changes in your body temperature.

Another common biosensor used at home is the pregnancy test. Home pregnancy tests use color-changing strips to detect pregnancy hormones in urine. Pregnancy tests are still done in doctor's offices. But the home test has become a reliable alternative since it was first introduced more than 40 years ago.

The rapid strep test is another commonly used biosensor. If you have a sore throat, your doctor may want to use one to test for bacteria called streptococci. The rapid strep test can provide results from a swab of the back of your throat in a

few minutes—with 95% accuracy. Your doctor may still send a throat swab to a lab to confirm a positive test result. But they can use the rapid test results to start treatment immediately.

In parts of the world where public health care isn't readily available, researchers hope to introduce rapid tests for people living in remote regions to test for infections like influenza, HIV, and hepatitis C. New biosensor technologies can now be combined with smart phone cameras and wireless signaling. These advances make health tests more portable and affordable than lab-based equipment.

Biosensors can also be used to continuously monitor a health condition. Blood-oxygen monitors are now found throughout hospitals and in patients' homes. These devices detect changes in the level of oxygen in the bloodstream. A rapid drop in oxygen can cause brain injury and requires quick medical attention. Blood oxygen monitors are ideal for people with lung and heart conditions, those undergoing anesthesia, or those being treated in intensive, neonatal, or emergency care. Other biosensors can be used to continuously monitor your blood sugar levels (for managing diabetes), blood pressure, or heart rate.

Flexible sensors are making even more types of monitoring possible. A team of engineers, led by Dr. Patrick Mercier and Dr. Joseph Wang at the University of California San Diego, is developing a flexible sensor that measures blood alcohol levels. It looks like a temporary tattoo. The sensor releases a sweat-promoting chemical into the skin and detects alcohol in the sweat. The sensor then sends the information wirelessly to a laptop or mobile device. Similar devices are being developed by other groups to monitor cystic fibrosis and other diseases and conditions.

At the University of Minnesota, a group of researchers led by Dr. Michael McAlpine has developed inks for 3-D printing sensors that are flexible, stretchable, and sensitive. These sensors can be used to detect human movements, such as flexing a finger. They can be printed directly onto skin and used to detect body signals, like a pulse. They can also detect chemicals in the environment and be used to warn of hazards.

NIH also supports research to use sensors to gather data about environmental and other factors involved in childhood asthma. These sensor systems monitor what children are exposed to and their body's reactions. For example, Dr. Zhenyu Li, a biomedical engineer at George Washington University, is developing a sensor that can be worn on a child's wrist to detect formaldehyde, an air pollutant that can trigger asthma.

"Researchers don't have tools at the moment that can monitor environmental triggers, physiological responses, and behavior without interrupting normal activities," Li says. There are many different asthma triggers, he explains. He expects to have a wearable sensor prototype that he and his clinical partners can begin testing with patients. He's also working on a device that can be placed in a child's home to detect multiple air pollutants, like those found in tobacco smoke and some manufactured wood products, such as flooring and furniture.

Biosensors can be placed inside your body as well. Dr. Natalie Wisniewski, a biomedical engineer at a medical device company in San Francisco called Profusa, is developing miniature sensors that can be injected under the skin. These sensors automatically track chemicals in your body without drawing blood. They continuously scan multiple factors at once. Normally, you need to stay in a hospital to have your body chemistry continuously monitored. With this technology, information about the chemicals in your body could be accessed around the clock, from anywhere.

Once placed under the skin, such biosensors can last for months to years. They can monitor various body functions through chemical changes. All this information can be collected on a cell phone app and shared with your physician, a caretaker, or anyone else you choose.

"Health sensors have the potential to dramatically improve the way we practice medicine and shift the focus away from reactive treatments to preventive maintenance," Wisniewski explains.

Biosensors are quickly becoming part of our normal health care routines. New sensor technologies are opening avenues to better health. Researchers are

working to develop the biosensors of tomorrow. These could provide access to better health in ways we can't yet imagine.

# Industry 4.0 and how smart sensors make the difference

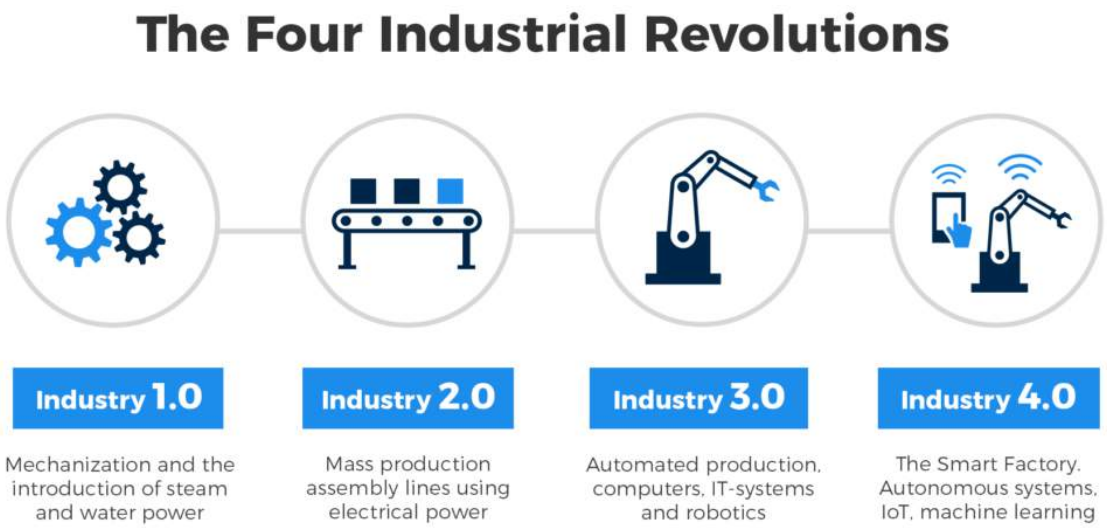
Most people working in the process industry have heard the term *Industry 4.0*. But what does it actually mean? Why should anyone be interested about it? Industry 4.0 has been claimed to be the fourth revolution of the manufacturing industry. The humankind has already seen three major revolutions in manufacturing industries:

1st was when mechanization and steam power changed the whole concept of manufacturing.

2nd revolution happened, when mass-production assembly lines and electrical energy took place and enabled a giant step in production efficiency.

3rd revolution brought automation, computers and robots to production.

It is easy to imagine how this evolvement changed the competitive landscape of many industrial processes.




It has been discussed already more than 5 years how IoT (Internet of Things) devices and sensors, digitalization and big data can change the whole manufacturing industry in the next 5-10 years. It is no more a question whether

this will or won't happen - the only remaining question is how fast this obvious evolution phase will be implemented into real actions in production.

Like a dear child, industry 4.0 has many names. Smart factory, cloud-based manufacturing (CBM), factory of the future, smart manufacturing and digital manufacturing are all synonyms for this next big revolution. Even though using several names may create confusion, the scope of digitalization is clear: increase production efficiency, reduce waste, decrease manufacturing costs, but also gain new business. Industry 4.0 is a very interesting topic for almost everyone involved in the manufacturing industries, because it combines multiple emerging technologies. This so called 4$^{th}$ revolution requires a combination of talent and expertise. The road to success is paved with multidisciplinary competencies, and it will need more collaboration than ever in manufacturing industries. The industry 4.0 manufacturing concepts of the future should gain benefit from new sensing technologies, big data, cloud computing, artificial intelligence, adaptive robots, smart valves and autonomous smart control applications for bringing maximum added value in smart processes of the next generation.

## The goals and benefits of Industry 4.0

The benefits and driving factors of this evolution should be scrutinized as well. What is the value and payback time of the investments, and is this the proper time to jump aboard the moving train?

Of course, the first obvious goal for Industry 4.0 is to enhance productivity through automation. In addition, industry 4.0 will decrease waste and improve yield, which both are very important driving factors in all development. Smart factories will also increase sustainability through real-time monitoring of production, and the autonomous control systems will reduce maintenance costs of future factories. Additionally, it can be seen that digitalization will increase the agility of production which will, of course, give crucial competitive benefits for advanced manufacturing companies.

On top of all this, the industry 4.0 will change existing business relationships and value chains. In other words, agile companies can gain the benefits of this transition phase by bringing totally new products to the market for material sensing, data management, big data or automation. The earning logics can also be

totally different than in the past, for example pay-per-results, monthly fees, or similar what we have already seen in the software industry (Software as a Service).

One of the largest studies on digitalization was published by PwC in 2016. Over 2,000 companies from 26 countries participated in PwC's 2016 Global Industry 4.0 survey*. The conclusion from this survey was clear - Industry 4.0 is no longer a future trend, but it has already moved from talking to actions. For example, the study found out that only 33% companies world-wide identified themselves as *advanced in digitalization* today. The amount of advanced companies will be increased up to 72% in the next 5 years. So, this is clear evidence that the race towards digitalization is already on, and competitive landscapes will be completely redefined in the nearby future. The same study claimed that 24% of companies believe to achieve more than 30% cost reductions in the next five years by investing into digitalization. The number was even higher in Asia, where 33% of companies believed to get significant competitive benefits in manufacturing costs in next 5 years. The majority of companies, 60%, said they would invest 2-10% of their revenue in digitalization, which is a staggeringly high proportion of their R&D investments. The investments are expected to be a total of US$ 907 bn annually until 2020. Another interesting finding in the survey was that though R&D investments were high, a majority of the companies expect Industry 4.0 investments to pay back within two years.

*) https://www.pwc.com/gx/en/industries/industry-4.0.html

Material sensing in Industry 4.0


But how can we benefit from material sensing in future smart factories? Existing technologies exploited by measurement, control and automation systems are mainly based on measuring physical parameters. Shape and distance measurements are critical for robots, flow measurement and temperature sensors are common in process control applications. Next big improvement will be deploying real-time chemical information from the measurement objects to an automation database and use it in automated control decisions. Spectroscopic technologies are well-established in off-line analysis in chemical laboratories, but it is obvious that future smart factories will gain huge benefits, when qualitative and quantitative material information can be generated with affordable and reliable sensors.

The price point of existing laboratory spectrometers is still several tens of thousands, and there still remains the challenge that these systems are very rarely feasible to be used in real-time analysis of continuous processes. Spectral Engines has developed a new Scanner Platform, which can bring this material information from laboratories to future smart factories only with a fraction of the price of existing material analysers. The Spectral Engines' motto has already been for several years to offer "Spectrometer performance at a sensor price point". This shows how Spectral Engines sees its own role as an Industry 4.0 enabler.

Spectral Engines' sensor platform is only one part of a complete sensing system. New expanded opportunities are related into machine learning, which brings

the smartness to spectral sensors. We have most likely seen only the tip of the iceberg in how deep learning, cloud computing and machine learning will be total game changers in many products and industries, including the factories of the future. Self-learning algorithms are going to play a very critical part, and cloud-connected sensors are a good example of what the IoT-connectivity can enable in the future. We are convinced that deep learning will generate so much information about future manufacturing processes, that the 4th manufacturing revolution will be a well-deserved definition for this giant leap of process industry. The change will be as dramatic as the one we saw when robots took their places at assembly lines.

A new era of process control

Real-time chemical sensors will open a new era of process control in several industries. The pulp and paper industry is a good example on how smart factory concepts have changed the production methods over the last decades, even before it was called Industry 4.0. Most likely pharmaceutical and chemical industries are the next ones where the deployment of smart sensors can be seen in a more broader way.

Also, the industrial processes of food and beverages will benefit more and more in the future from next generation smart sensor platforms. The future measurement opportunities are definitely related into moisture control, food ingredient measurements, ethanol concentration in alcoholic beverages, end-point analysis of pharmaceutical processes, and many more. The role of advanced machine learning algorithms will expand the opportunities to predictive maintenance, where the operator won't need to know every specific concentration, but monitor the income and outcome flows in processes that may indicate some problems in the process tools. The list of opportunities is endless, and based on current knowledge, it is easy to say that the development will be faster than we can even imagine.

Digitalization will be the key driving factor of future factory concepts. Industry 4.0 is going to change the landscape of all production based industries. It is obvious that there is a gap to be filled with smart sensors to generate real-time material information. Combining new affordable sensors and cloud-computing algorithms, many existing manufacturing problems can be solved. Based on the extensive PwC

market survey, where 2000+ companies were interviewed, the majority of companies (72%) would like to achieve an advanced level in digitalization.

A lot of investments have to be spent and the competitive landscape redefined in the next 5 years. The race is on.....

## IMPORTANCE OF THE IDENTIFICATION AND QUANTIFICATION OF PESTICIDE RESIDUES ON DOMESTIC AND IMPORTED FOODS

- Pesticides are used to protect crops against insects, weeds, fungi and other pests.
- Pesticides are potentially toxic to humans and can have both acute and chronic health effects, depending on the quantity and ways in which a person is exposed.
- Some of the older, cheaper pesticides can remain for years in soil and water. These chemicals have been banned from agricultural use in developed countries, but they are still used in many developing countries.
- People who face the greatest health risks from exposure to pesticides are those who come into contact with them at work, in their home or garden.
- Pesticides play a significant role in food production. They protect or increase yields and the number of times per year a crop can be grown on the same land. This is particularly important in countries that face food shortages.
- To protect food consumers from adverse effects of pesticides, WHO reviews evidence and develops internationally-accepted maximum residue limits.

Importance of Pesticide Residue Analysis

It is an undisputed fact that the area of the agriculture land on the planet will decrease and the demand for food and fibre for the ever-increasing population will keep on increasing. Protection of crop losses is as important as producing food and fibre, and crop protection chemicals have proved their worth in minimizing crop losses during green

revolution and are continuing to do so. Lately, pesticide residues in food commodities and their entry into the food-chain has become a major cause of concern all-over the world. Food safety has become crucial for all involved in the value chain and consumers have to be assured that they are not exposed to an unacceptable level of pesticide residues. After the establishment of the world trade order (WTO) and other GATT, SPS and IPR challenges in this millennium, presence of the residues above the permissible level is also a major bottleneck in the international trade of food commodities. Within the sphere of health and wellness, the topic can become quite polarizing. The conversation surrounding organic versus conventionally farmed foods is one such example, with dozens of articles extolling the virtues of organic foods, and an equal number that take an opposing view. It is undoubtedly a complex issue, and each perspective has its merits. There is a long history of pesticide discovery, development, and use in various states. Despite increasing consumer demand for organically farmed foods, pesticides are still widely used on crops grown for animal feed or human consumption. Numerous regulations, monitoring programs, and residue testing methods are employed to ensure safe pesticide application. Advances in analytical techniques including gas and liquid chromatography coupled to mass spectrometry continue to improve the sensitivity of pesticide residue detection.

THE EVOLUTION OF PESTICIDE USE: Pesticide use dates back to some of the earliest farming, as Ancient Sumerians and Romans used sulfur and salt to control pests. Later, soap, tobacco infusions, and arsenic were implemented as pesticides. Following World War II, a boom in pesticide development led to the creation of compounds such as DDT (dichlorodiphenyltrichloroethane), aldrin, dieldrin, endrin, and 2,4-D (2,4-Dichlorophenoxyacetic acid). These pesticides were initially effective, however their potent, sterilizing nature eventually led certain pests to develop resistance, and dangerous residues accumulated in food, water, and soil. The publication of Rachel Carson's Silent Spring in 1962 shed a harsh light on the impact of indiscriminate pesticide use, and despite criticism, sparked an environmental movement. The next generation of pesticides were designed with higher specificity. Insecticides (such as organophosphorus and carbamates) targeted insects, herbicides (including glyphosate) targeted plants, and fungicides and rodenticides targeted fungi and rodents, respectively. Along with a shift toward greater specificity, there was also a focus on integrated pest management (IPM). IPM is considered a more holistic approach that minimizes pesticide use and aims to manage, but not eradicate, pests. Since then, neonicotinoids and genetically modified organisms have been developed as defence against pests.

PESTICIDE REGULATION: Pesticide use has been instrumental for improving crop yields, thus supporting food production for humans and livestock. The desire for high yields

coupled to consumer demand for visually appealing, unblemished produce has driven their continued use. As such, regulation and testing methods must be in place to limit exposure and ensure safety. In India, the food safety is based on the guiding principle of risk analysis of the Codex Alimentarius Commission (CAC). In order to exploit full potential of pesticides in agriculture and public health programmes without adversely affecting the environment, it is essential to study the facts about pesticide behaviour and their persistence / dissipation under tropical Indian conditions. There is also a need to know the status of pesticide residues to ensure the safety to the consumer and to overcome the trade barriers at international level. The Government of India regulates the pesticide residues detected in various food items through Prevention of Food Adulteration Act (now through Food Safety and Standards Act, 2005). Various organizations in India such as institutions under the Indian Council of Agricultural Research (ICAR); State Agricultural Universities; Central Insecticides Laboratory (CIL); Indian Institute of Grain Storage; institutions under Indian Council of Medical Research (ICMR); Council of Scientific and Industrial Research (CSIR) and Bhabha Atomic Research Centre (BARC), and other research groups have been engaged in monitoring of pesticide residues in food commodities and environmental samples in their individual capacity primarily for academic 346148/2018/PP-| 267 2 purposes. Such studies were often overlapping and differed from one another in their results. Due to increasing public awareness and legalities involved in pesticide residues in food commodities, there was a need to harmonize the monitoring of pesticide residues in the country. DAC&FW sponsored central sector scheme, "Monitoring of Pesticide Residues at National Level" (MPRNL). Government has taken steps to ensure that pesticides adhere to specific safety standards, and re-evaluates pesticides every 15 years. In the US as part of its regulatory activity, the EPA develops a maximum legal residue limit (tolerance) for each treated food using data obtained from the United States Department of Agriculture (USDA) Pesticide Data Program. The EPA determines a tolerance that ensures use of a specific pesticide is associated with "reasonable certainty of no harm," factoring in toxicity, concentration, and residue. Since many food products are imported to the United States, the EPA tolerances are applied to both domestic and imported foods. In India the Department of Agriculture, Cooperation & Farmers Welfare, Ministry of Agriculture & Farmers Welfare had started a central sector scheme, "Monitoring of Pesticide Residues at National Level" (MPRNL) in food commodities and environmental samples during 2005-06 with the participation of various laboratories representing Ministry of Agriculture, Indian Council of Agriculture Research, Ministry of Health and Family Welfare, Ministry of Environment and Forest, Council of Scientific and Industrial Research, Ministry of Chemical and Fertilizer, Ministry of Commerce and State Agricultural Universities across the country. During 2008 to 2018, a total of 1,81,656

samples of the various food commodities such as vegetables, fruits, rice, wheat, pulses, spices, red chilli powder, curry leaves, milk, butter, fish and other marine products, meat, egg, tea, honey etc. and environmental samples like soil and water were collected from various parts of the country and analyzed during 2008- 2018 for the presence of pesticide residues, out of which 3,844 (2.1%) samples were found above MRL as prescribed under Food Safety Standard Authority of India (FSSAI), Ministry of Health and Family welfare.

SAMPLE PREPARATION TECHNIQUES : Gas and liquid chromatography (GC and LC) coupled to mass spectrometry (MS) are ideal for the identification and quantification of known and unknown compounds within a sample. As such, these techniques are widely used for pesticide residue analysis. Sample preparation for pesticide analysis often necessitates extraction steps to isolate the pesticides from the sample matrix and clean up steps to isolate the compound of interest from co-extracts. The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) method was first published in 2003 by USDA scientists for the analysis of fruits and vegetables, and has been expanded for use with animal products, grains, and other foods. Briefly, the original method involves the combination of sample with acetonitrile, magnesium sulphate, and sodium chloride. Clean up is achieved with dispersive solid phase extraction (dSPE) using anhydrous magnesium sulphate and a primary secondary amine sorbent combined with the acetonitrile extract to remove polar matrix components such as sugars and organic acids. The protocol has since been modified to include two additional versions, the AOAC 2007.01 and EN 15662.

Different matrices and compounds of interest pose some challenges during sample preparation. “Complex matrices can cause reduced or amplified recovery. While clean up processes are used, finding a balance of maintaining low detection limits and dealing with complex matrices is always a challenge,”.

IDENTIFICATION AND QUANTIFICATION: GC and LC are the most common techniques used for multi-residue pesticide analysis. Each technique has unique advantages and disadvantages, depending on the sample matrix and pesticides of interest. Pesticide polarity is an important consideration, as GC-MS is best suited to less polar compounds while LC-MS is best for highly polar compounds. For GC analysis, a pesticide must be volatile or undergo derivatization while LC enables detection of a much wider range of pesticides. “We use either or both GC or LC for all of our pesticide methods,” says Troester. “Liquid chromatography extraction methods are typically easier and achieve lower detection limits, partially due to larger injection volumes. GC methods are rugged, reliable, and offer a different detection approach than LC. This can lead to identification of compounds LC may miss.” Detection methods are also an important consideration. MS

quantifies the mass-to-charge ratio of ions, while MS-MS (tandem MS) breaks precursor ions into fragments, which are then separated and detected in a second stage. MS-MS offers greater selectivity, which is beneficial for the detection of known compounds. "The obvious advantage of MS-MS is the high level of selectivity leading to lower method detection limits. The advantage of MS is the lack of selectivity, which can be useful in identifying compounds that we were not necessarily even looking for and is helpful in identifying 'unknown' compounds."

TESTING IN THE FIELDS : Improved techniques and technologies facilitate faster, more accurate pesticide residue detection. Older methods of measuring pesticide residues tend to be more time-and solvent-intensive. For this reason, more routine testing labs are adopting simpler and greener methods of measuring pesticide residues. Now the trend is towards field instrumentation for pesticide analysis. "Laboratory-based LC-MS technologies have been employed for screening pesticides for years. However, interest is growing for more rapid screening techniques for deployment outside the lab. Such field-deployable technologies may mitigate risk at earlier points in the supply chain by allowing growers to screen crops pre-harvest to determine the best time to harvest them, thereby reducing the risk to consumers. And so, laboratories are challenging vendors to deliver rapid, field-deployable, portable technologies such as direct MS whereby samples are introduced into the mass spectrometer under atmospheric pressure without prior chromatographic separation, with automated chemometric statistics for obtaining results in real time." The impact of pesticides used in conventional farming on human health remains unclear, and at this time, pesticides are still widely used to improve crop yields. Adherence to maximum residue limits, ongoing efforts to reduce the amount of pesticides applied, and sensitive testing techniques all serve to ensure consumer health and safety. As technology improves, we may further reduce our reliance on pesticides. In the meantime, vigilance is required to ensure environmental and human health.

Reference: Lab manager, September 2019