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DRDO has made outstanding contributions in the vital areas of safety management; development of systems, devices, processes and materials in the fields of fire, explosives and environment safety for defence services and Ministry of Defence (MoD) establishments through its concerted R&D efforts. Centre for Fire, Explosive and Environment Safety (CFEES), an establishment of DRDO plays a unique regulatory and advisory role in MoD such as safety advice, audit, regulations and compliance of Storage and Transport of Explosive Committee (STEC), siting of explosive storages facilities and fire advisory. CFEES is also a member of Environment Safety Committee (ESC) for MoD and Ministry of Environment and Forests (MoEF). Apart from advisory and regulatory duties, the R&D work accomplished by the laboratory encompasses wide spectrum of activities like design and development of systems, design of new facilities and processes in three areas, viz.,-(i) fire science and fire protection engineering; (ii) explosive safety in the areas of storage and handling of ammunition and explosives; (iii) environmental management and hazardous waste treatment for MoD. The R&D achievements

of the lab provided continuous technology inputs for evolving more efficient and practical safety practices in the area of fire, explosives and environment safety. Besides providing consultancy to government departments, public and civil sector units on explosives/fire/environment safety related matters, the lab conducts special courses and provides training to personnel of defence services, inter service organisations, ordnance factories and other defence undertakings. It also plays a pivotal role in safety management in DRDO. Risk and Hazard Analysis (RHA) is mandatory under the manufacture, storage and import of Hazardous Chemicals Rules, 1989 for those establishments handling or processing the specified chemicals in excess of the stipulated threshold limits.

CFEES has carried out RHA and EIA studies for more than 30 MoD special projects and installations. It is the nodal agency for implementation of the National Environmental Policy (NEP) and Safety Health and Environment (SHE) for DRDO labs and disaster management for northern group of DRDO labs.





CFEES has been entrusted with the responsibility of providing a vision in the field of fire science and engineering; explosive and environment safety; integrated safety advice and safety regulations; safety audit; advice and siting of explosives storage facilities for various MoD establishments.

The Centre has excelled in a gamut of technologies related to explosive storage structures which includes design and development of propellant (HD1.3) storage/ process structure upto 50T NEC per compartment based on Unit Risk Principle and safe storage of sensitive (HD1.1) explosives upto 2T and design of High Performance Magazine (HPM) for 5T NEC (HD1.1) per compartment capacity with non-propagation type sandwich partition wall comprising composite materials. All the designs have been validated through fully instrumented blast trials and recommended by STEC panel for incorporation in STEC regulations for use by MoD users. Design configuration of underground explosive/ ammunition storage to reduce the Outside Quantity Distances (OQDs) for storage of ammunitions has been validated through fully instrumented blast trials. Hardened Aircraft Structure (HAS) capable of withstanding the direct hit of 2000 lb bomb with a penetration capacity of 1.8 m has been designed, vetted and given to Air Force for implementation at critical stages. A large number of siting of explosive facilities been carried out for MoD establishments, 73 Nos. of Army; 35 Nos. of Air Force; 3 Nos. of Navy, 68 Nos. of OFB; 42 Nos. of DRDO; 01 No. of DGQA. Trials have been conducted for hazard classification and TNT equivalence of (i) Pinaka Rocket Propellants; (ii) NEPE Rocket Propellants; (iii) Akash Booster Motor Rocket Propellants; (iv) LRSAM Rocket Motor. System has been established for disposal of unserviceable shell of 105 mm HESH by low order detonation.

CFEES has taken up a project to replace Halon 1301, an internationally banned gas under Montreal Protocol, as extinguishing medium in the crew and engine compartment of battle tanks. An alternate, Heptafluoropropane (HFP) HFC227ea, has been developed and validation trials have been conducted in the presence of users. A National Halon Bank has been established for meeting

From the Desk of Director, CFEES

the futuristic demands of Halon for MoD establishments. Recovery, recycling and reclamation programme is under development and re-charging services for LCA, MBT and other armoured vehicles have been provided. Aerosol based Fire Fighting System (AFFS) has been designed developed and demonstrated at CFEES in 1 m³ chamber for fire suppression applications in next generation aircraft engine and LCA. A state-of-the-art microcontroller based *IFDSS system for protection against fire engine and crew* compartment in AFVs, with a capacity to extinguish fire within 150 ms, has been developed and inducted into the Services. Development of water mist based IR signature suppression system for hot exhaust plume of naval ship (as per users requirements) has been completed. Polymer based Fire Suppression Gel (FSG) has also been developed for minimising water loss during fire extinguish for defence and civil sector.

The Centre plays a vital role in the field of environment management and hazardous waste treatment. Catalysts for onboard reforming applications to generate hydrogen for ethanol as clean energy source have been developed. A decontamination apparatus based on metal oxide nanomaterials for removal of hazardous toxic contaminants is under development. Eco-friendly technique for disposal of PET wastes has been established. Decontamination paint as surface protective coating for radiation shielding and protection of sub critical components for naval applications has been developed. RHA studies are being carried out to evolve preventive and protective safety measures to minimise risk and provide inputs for emergency planning in MoD establishments handling hazardous materials. CFEES is spearheading explosive safety programme for designing various types of protective structures, Magazine Fire Fighting System (MFFS) and also National Mission Programmes on designing, architecturing of nano safety manual/guidelines for DRDO.

This issue gives an overview of some of the technologies developed by CFEES.

Dr. Chitra Rajagopal Outstanding Scientist & Director CFEES



In its pursuit to strengthen and enrich the technological basis in the field of fire science, explosive safety and environmental engineering, CFEES has delivered materials, processes and systems which have benefited DRDO, MoD and have spin-offs to the civil sector.

FIRE SAFETY PRODUCTS

Fire Suppression Gel

Water is universal choice for extinguishing fire due to its natural profuse abundance but most of the watergoes waste as run-off during fire extinguishment. Fire Suppression Gel (FSG) composition when mixed with water, swells and holds water. On application, FSG sticks on burning objects and prevents the water from running off or evaporating too quickly. It is useful in preventing, retarding, suppressing and extinguishing the fire effectively.

The FSG minimises time to extinguish the fire as well as the quantity of water used.

Technical Specifications

Particle size of composition	0.01 - 2 mm	
Application quantity	0.5 to 1.5 % (w/v) of total water	
Water holding capacity	Min. 250 g water/g of FSG	
Reduction in fire extinguishing time	30 - 50 %	
Reduction in water quantity used	30 - 50 %	
Shelf life	More than 5 years	



A patent has been filed on Fire Controlling Composition and Method thereof, No. 2765/DEL/2013

Fire Suppression Gel



Potential Application Areas

Defence Services : Defence stores, ordnance factories, ammunition process industries, commercial cargo ships, aircraft hangers, cargo bays, etc.

Civil Sector : Process industries, commercial complexes, residential and high rise buildings, hotels, houses, stockyards, forests, ware houses, etc.

Process Technology for Heptafluoro -propane (HFC 227ea): Halon Alternative Fire Extinguishing Agent

Heptafluoropropane is the leading chemical agent for replacement of Halon1301 for total flooding applications. This fire extinguishing agent is highly effective, non-corrosive and electrically non conductive than the Halons and leaves no residue on applications and non-ozone depleting in nature. It is ideally suited for many critical fire suppression applications and has become the market leader in providing rapid and safe fire protection for high value assets in both occupied and unoccupied critical applications.

Globally HFC227ea is acceptable for use as a total flooding fire extinguishant for the protection of occupied space as per NFPA 2001 (2012 ed.). In

the present scenario HFC227ea available as the new environmentally acceptable solution to replace Halon1301. Heptafluoropropane has internationally been approved as Halon1301 substitute for new generation fire protection systems. All international standards are now specifying HFC227ea in their fire safety regulation both in civil and military applications. Under the strict implementation of Montreal Protocol, which prohibits production and use of Halon as fire suppressing agents, HFC227ea has a promising commercial value.

CFEES has developed an economic and commercially viable technology for the production of heptafluoropropane (HFC227ea). The process involves hydro fluorination of hexafluoropropene with anhydrous hydrofluoric acid using in-house developed metal oxide based catalyst. Transfer of technology has already been executed with industrial partner M/s Gujarat Fluorochemicals Pvt. Ltd. under DRDO-FICCI-ATAC program.

More than 80 per cent of the fire protection uses are in the civil sector industries such as oil, power, telecommunication, etc. Large demand of HFC227ea by civil sector may bring down expected cost of indigineous HFC227ea making it cheaper than imported HFC227ea. Interestingly, the performance of HFC227ea vis-à-vis Halon1301 has been proved outstanding for retrofitment in fire





Snapshots of Fire Performance Trials in Derelict T-72 Tank





HFP Catalyst Under Processing



Final Form of Catalyst

suppression system for T-72 tank and meeting the requirements of GSQR 666 and NFPA 2001 for fire suppression timing for both crew and engine compartments.

Fire Protective Clothing: Light Weight Fire Approach and Fire Proximity Suit

Light Weight Fire Approach Suit and Fire Proximity Suit are multi-layered suits used for fire fighting and rescue operations. Light weight fire approach suit consists of three layers made up of high performance blended fibres. Outermost layer : Moisture barrier : Innermost layer : Blended woven fabric Coated blended fabric Fire retardent cotton fabric

The light weight fire proximity suit has one extra layer of non-woven fabric as thermal barrier between moisture barrier and innermost layer FR cotton fabric. The weight of complete fire approach and proximity suits are approx. 6.4 kg and 7.2 kg respectively. Both the suits have five components, viz. jacket with sleeves for protection of the torso portion including the arm but excluding the hands, trousers for protection of legs excluding the feet, a pair of gloves, a pair of boots, and hood incorporating helmet and visor.

These suits can be used in fire fighting applications in establishments of armed forces, civil fire services, oil rig platforms, oil depot, ONGC, steel, plastic and chemical industries, etc.



Light Weight Fire Approach Suit Light Weight Fire Proximity Suit

Salient Features

- ✤ 30 per cent lighter in totality over the existing aluminised suit
- Light weight fire approach suit withstands for
 10 min against the exposure of fire source





Components of Light Weight Fire Suit



Snapshots of Live Fire Test Trial

of 50 L aviation turbine fuel at 5 m distance during live fire test trial while fire proximity suit withstands for 4.35 min at 3 m distance during the same live fire test trial

- Excellent maneuverability and freedom of movement to the fireman
- Meets the performance requirements of NFPA 1971 and JSQR 8415-007 except breathability in moisture barrier fabric

Patent: Patent filed vide application No. 438/ DEL/2010 and 439/DEL/2010 and granted design patent (Patent No. 227967, 2010 and 227968, 2010).

Status

Technology is available for transfer to industrial partners.

Miniaturised Instant Fire Detection and Suppression System for Armoured Fighting Vehicles

Miniaturised Instant Fire Detection and Suppression System (IFDSS) caters the eventuality of fire inside the battle tank. Designed and developed at CFEES, IFDSS is a micro controller based fire detection and suppresion system for AFVs. This system detects and suppresses the fire within 150 ms in the crew compartment and saves crew and vehicle from any fire eventuality. Size of main control unit and other sub-units are miniaturised and compact for quick use. To detect the fire inside the crew compartment, specially designed dual spectrum IR detectors are used.

The IR detector unit comprises of two narrow band sensors, namely the near infrared and mid infrared sensors. These fire detectors are tested as per MIL STDs and found fully immune to false alarms. The confirmation of fire is sent to master control unit over CAN Protocol. In engine compartment, a pair of linear thermal detector (fire wire) is used, which continuously monitors the temperature of the engine compartment.



Linear thermal detectors detect the temperature rise in engine compartment and provide audio visual alarm at 120 °C. If temperature further rises, then the fire extinguisher gets activated at 140 °C to suppress the fire. Automated fire extinguishers with Halon1301 are used for suppression of fire.

Salient Features

- ✤ Saves both crew and vehicle
- ✤ Compact in size
- ✤ Microcontroller based operation
- Discrimination between true fire signature and non fire stimuli
- Combat and peace mode operation
- ✤ Detection of fire within 20 ms by extremely fast flame detectors
- ✤ Built-in Test (BIT) facility
- ✤ Logging of important fire events for post fire analysis
- ✤ Complete fire out below 150 ms

Application

IFDSS has been tested and evaluated as per applicable MIL STDs for environmental and EMI/



Miniaturised Instant Fire Detection and Suppression System

EMC tests and then subjected to various user trials. System outperforms the GSQR parameters and recorded fire out time is 76.3 ms against 150 ms.

After successful user trials the system inducted into the Services and is being used by armed forces for various AFVs.

Water Mist Based Fire Suppression System

The CFEES water mist system is the dual fluid system which uses air or N_2 at 5-7 bar to atomise water supplied at 2-5 bar. It utilises Specifically designed multi orifice nozzles based on internal mixing of the two fluids, air and water which generate a mist of very fine droplet having Volume Mean Droplet Diameter (VMD) less than 50 µm and Sauter Mean Diameter (SMD) less than 30 µm. The droplet size distribution of the mist can be varied across a wide range by changing the relative water and air pressures. The nozzles used in the water mist systems are characterised at various operating conditions using the droplet size analyser based on laser diffraction principle.



Nozzle Characterisation



Water Mist based Fire Suppression System (WMFSS) has been validated successfully as per International Maritime Organisation (IMO) fire test protocol (IMO-Circ 1165) and demonstrated to users in a real scale sub marine fire simulation compartment (590 m³) installed and commissioned at CFEES.

The water mist technology indigenously developed is at par with the internationally available technology and has very high potential for fire suppression application in civil and defence sector. It provides the most effective and environmentally benign alternate solution to Halon based fire protection systems. This technology can also be applied effectively for cooling applications.



Internally Mixed Atomiser



Water Mist Gun System



Portable, Hand-held and Back-pack Low Pressure Water Mist Gun System

Portable, handheld and backpack low pressure twin fluid water mist gun system has been designed and developed by DRDO. The system is useful as a first- aid fire fighting for class A, class B and electrical fires in naval platforms like ships, submarines, POL stations, defence stores, trains, process industries, civilian buildings, hotels, warehouses, village farm stockyard, etc.

The system successfully passed fire tests as per international standard EN 3:7-2004 for B233,

A55 and live electrical fires. A patent on the design of the system has been filed [No. 694/DEL/2011]. This technology was transferred to industry (M/s ASKA Equipments Pvt Ltd, New Delhi) for commercialisation as it has wide applications in civil sectors also.



Flammable Liquid Pool Fire Suppression

EXPLOSIVE SAFETY TECHNOLOGIES

High Performance Magazine

High Performance Magazine (HPM) is a concept of storing higher explosive capacity (5 Ton NEC) per compartment. This concept is realised by incorporating non-propagating type sandwich partition walls between adjacent compartments of the explosive storage building, which prevents sympathetic detonation of explosives/ ammunition stored in adjacent compartments, in case of detonation in one compartment.

However, there is a possibility of delayed detonation in the adjacent compartments and the structural integrity of the adjacent compartment is not assured.

This design addresses the need for ammunition storage in critical locations where land is not available, but operational requirements compel the storage of ammunition.

Design Features

The specialised partition walls, designed and developed by CFEES, comprises of sand-air-sand configuration which not only reduces the intensity of blast overpressure but also offers significant resistance against the shock and fragmentation effects.

HPM compartments have discontinuous floor which mitigates the propagation of ground shock to adjacent compartments. The design of the nonpropagation wall has been validated by successfully conducting the instrumented blast trial.

Advantages

- No sympathetic detonation in adjacent compartments
- Reduction in QDs for HD1.1 explosive storage units, resulting saving in land
- Non-compatible ordnance can be stored in different way of the same building
- Abroad spectrum of ordnance types (missiles, mines, bombs, torpedoes, etc.) can be accommodated

Over the years, the induction of latest ammunition has resulted in increased demand for siting of new explosive facilities. Siting of these facilities has become difficult due to non-availability of requisite Inhabited Building Distances. The





High Performance Magazine Structure with Blast Gauges

Underground Explosive/ Ammunition Storage for 1.1 Ton NEC HD 1.1

concept of underground storage can solve this problem to a large extent by reducing the requirement of quantity/ safety distances by 1/3rd to 1/4th with respect to the conventional above ground explosive storage magazines.

Design Features

For the blast trial, an underground explosive/ ammunition test facility was constructed at Field Firing Range (FFR) by cut and cover method. The test facility consisted of two storage chambers (donor and acceptor) with a separation distance of 2.0 Q^{1/3} and with earth cover of 4.6 m. Donor and acceptor chambers were designed to withstand 10g ground shock acceleration and 0.5 MPa internal pressure for 1100 kg net explosive content with explosive loading density of approx. 10 kg/m³. The tunnel was designed for 5 bar internal pressure. Hazard mitigation features like expansion chambers, debris traps and portal barricades were provided to mitigate the hazardous effects of the explosion. The design of the underground structure has been validated by successfully conducting the instrumented blast trial.

Advantages

- Easier to camouflage and guard
- More protected against aerial attack and external fires
- Suitable for extreme temperature areas



Portal Barricade After Explosion at Adit of UG Structure



Unit Risk Principle Based Explosive Storage Structure upto 2T NEC HD 1.1 and Propellant storage Structure upto 50T NEC HD1.3

Unit Risk Principle (URP) is based on the division of bulk explosive into a number of stacks, stored in different compartments such that initiation of explosives in one compartment does not result in initiation of the explosives in adjacent compartment. In URP, the safety/ quantity distances are governed by the net explosive content of one compartment resulting optimal utilisation of land.

Design Features

For HD 1.1, the unit structures are designed for 2 bar blast over pressure using Laced Reinforced



URP-based Structure (for HD1.1) Before Trial

Concrete (LRC) technique with a wall thickness of 300mm and are separated by intervening sandwich partition wall. For HD 1.3, the unit structures are designed for 2 bar blast over pressure using Reinforced Cement Concrete (RCC) and are separated by intervening RCC partition wall of 450mm thickness. A special venting arrangement has been provided in the roof for the release of deflagration products.

Design Validation Field Trial

The Design of URP for 2T NEC (HD1.1) per compartment and 40T NEC HD 1.3 per compartment have been validated through fully instrumented field trials. Blast loading parameters as well as ground vibrations at critical locations were monitored in case of HD 1.1 storage structures. In case of propellant storage structures



URP-based Stucture (for HD1.1) After Trial



URP-based Structure (for HD1.3) Before Trial



URP-based Stucture (for HD1.3) After Trial



of HD 1.3 values of heat flux and temperature were measured at critical locations.

Advantages of URP Design

- ✤ Reduction in QD give 40-60 per cent reduction in land area for storage of HD 1.1 explosives
- Safety and serviceability of explosives stored in adjacent compartment
- ✤ Reusability of the adjacent compartments in case of HD 1.3
- Enhanced safety of personnel in the adjacent compartment

Hazard Classification and TNT Equivalence Trials of Akash Booster Propellant

Akash missile has composite propellant booster grain in free standing configuration and a special fuel rich grain with variable burn rate. As a common practice, propellant part of the missile is also treated as explosive of HD1.1 which require larger safe distances.

Due to non-availability of these QDs in northern and eastern sectors, there was urgency of working out on the TNT equivalence of Akash propellant. Solid propellant rocket motors cannot be subjected to the conventional tests, i.e., single package test; stack tests; and bonfire tests due to their exorbitant cost, logistics and safety issues, therefore trials were conducted as per Gap Test.

The TNT equivalence of the propellant was conducted as per SLSGT test. Shock velocity profile was measured by deploying pressure gauges at various distances from the motor.

Based on trial results, the Akash booster motor without nozzle and igniter has been categorised as HD1.3 and its TNT equivalence is 0.47.

Nitrate Ester and Plasticised Polyester/ Polyether Polyol (NEPE) is an advanced energetic propellant. Since this propellant contains HMX in a proportion where it was mandatory to conduct its hazard division and TNT equivalence.

Similar to other solid propellant, rocket motors were subjected to the Gap Test. Based on trial results, the NEPE rocket motor without nozzle and Igniter should be categorised as HD1.1 and it is having TNT Equivalence of 1.38.

Similarly, Pinaka Mk-II rocket motor without nozzle and igniter was subjected to hazard classification trials and has been categorised as HD 1.3 and is having TNT equivalence of 0.7.



Crater and Witness Plate



Witness Plate & Fragments of Pinaka Motor Casing





Propellant Grain



Sheared Piece of Witness Plate by NEPE Propellant

Hazard Classification Trials of LRSAM Rocket Motor

Long Range Surface to Air Missile (LRSAM) has integrated propulsion system of two casebonded composite propellant rocket motors, namely Pulse-I and Pulse-II. LRSAM rear sections had to be transported by air. To enable air transportation of rear section of LRSAM, it was mandatory to finalise the hazard classification and UN number as per UN regulations. Therefore, this solid propellant rocket motor was subjected to Gap Test. Based on the results of the test, the LRSAM rocket motor without nozzle and igniter was categorised as HD1.3.



Witness Plate



Test Set-up



Fragments of Casing



Hazard Classification Trials of Round 30 mm VOG-17s

Round 30 mm VOG-17S is grenade ammunition fired from 30 mm automatic grenade launcher. For this ammunition no technical information was available. Therefore, CFEES conducted trials for assignment of correct hazard classification. Based on the hazard classification trials conducted as per UN Test Series 6, Round 30 mm VOG 17S, packed in hermetically sealed metal can and further housed in a wooden box categorised as HD1.1.



Trial Set-up for Single

Disposal of UXO's by Low Order Detonation Using Sheet Explosives

DRDOsuccessfullyestablishedanewtechnique for disposal of unserviceable 51 mm, 81 mm, and 120 mm mortar bombs by low order detonation using sheet explosive. Same methodology was developed for disposal of 105 mm HESH shells so that about 1200 Nos. of these shells (UXOs) can be disposed off. In this novel method, a shock is induced in the HE filling to achieve deflagration or low order detonation, by detonating few grams of sheet explosive on the shell casing. Trials were conducted by placing sheet explosive of varying dimensions longitudinally on the shell body towards the nose end. The sheet explosive was detonated and resulted in low order detonation of shells. It was established that sheet explosive of specified size when placed longitudinally on the shell body at specified gap from cap nose of the shell, a specified contact length was able to dispose off 105 mm HESH shells by low order detonation.



Shell 105 mm Tank HESH



Opened Shell



Witness Plate After the Trial



Hazard Classification of Pechora Missile Warhead

Hazard classification of fully assembled Pechora missiles was carried out to confirm or to rule out the propagation of explosion from one package to adjoining packages when stacked in a condition similar to storage configuration. The hazard classification of any military explosive or ammunition is based on three tests, viz., single package test; stack test; and bonfire test in accordance with UN document on 'Manual of Tests and Criteria'.

The single package test for Pechora missile was waived, as the tare contained only one article. The stack test was conducted with three numbers of warheads in tares kept at a distance from each other to simulate the actual stacking configuration during storage. The middle warhead was initiated and resulted in high order detonation. Hazard classification of Pechora missile warhead was confirmed as HD1.1



Crater



Trial Set-up



ENVIRONMENT SAFETY PRODUCTS AND TECHNOLOGIES

Bio-remediation of Explosive Contaminants in Waste Water and Soil

Manufacturing and handling of large quantity of energetic materials generate effluents and waste materials containing recalcitrant nitro-organic constituents which are highly toxic in nature. It is essential to develop an eco-friendly technology for treating this waste. In nature, diverse types of micro organisms and energy sources are available. This diversity makes it possible to break down a large number of different organic chemicals. Bioremediation using native microflora has proven potential in the lab scale level for degrading the explosives present in aqueous phase and in soil matrix. isolated and identified by 16 S rRNA gene sequencing analysis

- Among the 23 microbial isolates identified, 9 microbial cultures were found to have degradation potential for 2, 4, 6 TNT and 2, 4 DNT which was confirmed by lab scale studies and evaluated for treating the TNT contaminated water and soil media
- Complete biotransformation of TNT was achieved by using Pseudomonas moselliin. Soil bio-remediation with Delftiatsuruhatensis revealed complete degradation of TNT in soil which was confirmed by HPLC analysis. Scanning electron microscopic studies showedenhancedmicrobialcolonisationinsoil by Delftiatsuruhatensis

Salient Features

 Native microflora present in actual explosives contaminated waste water and soil samples of an explosive manufacturing facility were

Merits

Treatment of recalcitrant explosive chemicals by bio-remediation is an eco-friendly technology. An



Lab-scale Bioreactor used for Degradation Evaluation of TNT and DNT



eco-friendly remediation approach will contribute to pollution reduction and sustainable environmental management of explosives manufacturing facilities.

Potential Applications

Similar approach can be used for treating other similar effluents and waste materials containing organic and inorganic contaminants.

Metal Oxide Nanoparticles as Reactive Adsorbents Against Toxicants

Metal oxide nanoparticles has gained momentum in recent times in the field of contaminants removal from environment due to large surface area and reactive sites.

Nanoparticles of MgO, AI_2O_3 , CaO, TiO₂ and SiO₂ were synthesised via aerogel route and characterised using N₂BET, SEM, TEM, XRD, TGA and FT-IR techniques. They have surface area upto 628 m²/g and density as low as 0.035 g/mL.

Metal oxide nanoparticles have been impregnated with reactive compounds (Pd/



Synthesis of Metal Oxide Nanoparticles

Fe, Pt/Fe, Co/Fe, Cu/Fe,Ni/Fe, oxaziridine, polyoxometalate, trichloroisocyanuric acid and NaOH).

High removal efficiency for diethylchlorophosphate (1011 mg/g) and nitrobenzene (1155 mg/g) achieved under static conditions with MgO and SiO_2 + Fe (10 % w/w) nanoparticles respectively. Complete removal of TNT and RDX (25 ppm) from water achieved using SiO_2 + Fe and SiO_2 + Co systems in 7 and 24 h respectively.



Metal Oxide Nanoparticles

Synthesis of Graphene Oxide Nanosheets

Graphene, a single layer of graphite has recently attracted considerable attention due to its electronic and structural properties. Most of the reported methods for grapheneoxide synthesis involve chemical oxidation of graphite using inorganic salts and strong acid (Hummers method).

CFEES has developed a modified process without use of inorganic salts, e.g. sodium nitrate. The process successfully demonstrated at batch level and 10 g per batch graphene oxide nanosheets has been synthesised. Feasibility for up-scaling to the continuous scale production up



to 100 g per batch (further up to 500 g per batch) is under progress.

Synthesised graphene oxide nanosheets have been characterised by various analytical techniques such BET surface area analysis,



HV-200.0kV Direct Mag: 40000x

TEM Image of Graphene Oxide Nanosheets



Graphene Oxide Nanosheets

Fourier Transform Infrared Spectroscopy (FTIR), Thermal Garvimetric Analysis (TGA), Scanning Electron Microscopy (SEM), Transmission Electron Spectroscopy (TEM), Energy Dispersive X-ray Spectroscopy (EDAX), X-ray Diffraction (XRD) and Raman spectroscopy.

Potential Application Areas

- ✤ Environmental remediation against toxic chemicals.
- ✤ Useful for development of reactive adsorbents, nano-composites and sensors.



Graphene Oxide Nanosheets

Core-shell Polysiloxane-MOFs Micro spheres as Stationary Phase for Gassolid Chromatographic Separations

Metal Organic Frameworks (MOFs) are an emerging class of porous materials, which in view of their high surface area, thermal stability and uniform pore size are potential candidates for various applications like gas storage, gas separation, sensors, drug delivery, catalysis, etc. The availability of in-pore functionality and possibility of outer surface modification in MOFs



render these materials extremely interesting in the context of gas or liquid separation. These features allow separations based on both molecular sieving as well as based on chemical interactions. It is also possible to tune the pore size and geometry of the MOFs for use in specific applications.

This feature can make significant impact towards development of adsorption based separation processes for structural isomers, which generally relies on the subtle matching of size and shape of the adsorbates and the adsorbent micropores, commonly known as shape selective effect. The MOFs are formed as a result of self assembly of metal ions with linker molecules. Techniques for large scale preparation of MOFs at gram level generally rely on solvo-thermal methods, which lead to the formation of powders. The particle size of the MOF powder depends on the reaction conditions (usually less than 0.5 μ).

This innovation was created to solve issues associated with high pressure drop while using MOFs in the packed columns for gas chromatographic separation applications. Coreshell methodology used in this innovation, wherein the core is of the polymer, an inert template and shell is of MOF. With this, optimised particle size of MOFs required for packed columns (177- 250 μ) were achieved.

Benefits

The technique is universally acceptable to envisage the chromatographic application of various functional MOFs.

- ✤ Large single crystal of MOF can be used in packed stainless steel columns for laboratory level similar applications.
- Higher efficiency of separation with low pressure drop is achievable through this technique eliminating the need for pelletisation due to reduction in active surface area.
- Better control over column characteristics compared to capillary columns

Applications

- Opens up pathway for application of functional MOFs in chromatographic separations
- Can be used for the application of functional MOFs in the field of biomedical engineering
- Can be used for the application of functional MOFs as pre-concentrators for chemical war fare agents



Column Made of MOFs







Chromatogram Separation of Natural Gas Mixture. 1. Methane, 2. Ethane, 3. Propane, 4. Carbon dioxide, 5. Iso-Butane, 6. n-Butane, 7. n-Pentane, 8. n-Hexane using MOF Column

Preparation of MoF

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