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FUNCTIONALLY DESIGNED ADVANCED CERAMIC MATERIAL FOR ADDRESSING OIL SPILLAGE : FIELD DEMONSTRATION.



- **Petroleum stands for Rock Oil.The word comes from the Greek word ‘petra’ (meaning rock) and the latin word ‘oleum’ (meaning ‘oil’).**
- **Petroleum or crude oil is a naturally occurring, toxic, flammable liquid consisting of a complex mixture of hydrocarbons of various molecular weights and other organic compounds that are found in geologic formations beneath the earth's surface.**
- **Petroleum is recovered mostly through oil drilling.**
- **It is refined and separated, most easily by boiling point , into a large number of consumer products, from gasoline and kerosene to asphalt and chemical reagents used to make plastics and pharmaceuticals.**

THE OIL WELL INDUSTRY HAS THREE MAJOR AREAS OF OPERATION:

1. Oil well cementing materials.

2. Drilling mud materials.

3.Issue of "wastes generated in oil well industry

NEW MATERIALS FROM "INDUSTRIAL AND AGRICULTURAL WASTES" FOR ADVANCEMENT OF PETROCHEMICAL INDUSTRY.

OIL WELL CEMENTING MATERIALS

- **The well is created by drilling a hole 12 cm to 1 meter (5 in to 40 in) in diameter into the earth with a drilling rig that rotates a drill string with a bit attached.**
- **After the hole is drilled, sections of steel pipe (casing), slightly smaller in diameter than the borehole, are placed in the hole.**
- **Cement may be placed between the outside of the casing and the borehole.**
- **The casing provides structural integrity to the newly drilled wellbore, in addition to isolating potentially dangerous high pressure zones from each other and from the surface.**

Conventional materials comprise:

- Conventional Ordinary Portland cement (OPC)

- Conventional Geopolymer cement (CGC)

An emerging New material (ALSAGC) developed by CSIR-AMPRI, Bhopal, India

- **ALSAGC is Advanced Ligno- Silico -Alminous Geopolymeric Cement.**

- **Significant features of ALSAGC:**

a) Obviates the need of external materials required in conventional geopolymer cement e.g. LIGNIN BASED COMPOUNDS.

b) ALSAGC posses 25 % increased Compressive and flexural strength compare to CGC.

c) Homogenous matrix.

DRILLING MUD MATERIAL(DMM)

-Liquid drilling fluid is often called drilling mud.

-The main functions of drilling mud include providing hydrostatic pressure to prevent formation fluids from entering into the well bore, keeping the drill bit cool and clean during drilling, carrying out drill cuttings, and suspending the drill cuttings while drilling is paused and when the drilling assembly is brought in and out of the hole.

-The drilling fluid used for a particular job is selected to avoid formation damage and to limit corrosion.

-The three main categories of drilling fluids are water-based muds (which can be dispersed and non-dispersed), non-aqueous muds, usually called oil-based mud and gaseous drilling fluid, in which a wide range of gases can be used.

Conventional Drilling Mud Materials include:



MUD



RICE HUSK



BARITE (BARIUM SULPHATE)



HEMATITE

Innovation by CSIR-AMPRI, Bhopal An emerging New material - Advanced "Magnetic" Drilling Mud Material (AMDMM) developed by CSIR-AMPRI, Bhopal

Raw materials for developing (ALSAGC) –

1. Fly Ash from Thermal power plants.

2. Rice Husk from Agricultural industry.

3. Brine Sludge from Chloral Alkali Industry.

4.Red Mud from Aluminum Production industry.

SIGNIFICANT FEATURES OF AMDMM:

- ✓ It Obviates the need of external materials required in conventional Drilling mud material namely I) Barite, ii) Hematite and Rice Husk.
- ✓ AMDMM enables controlling rheology of the drilling mud by an additional features and parameter i.e. Magnetic properties of drilling mud.
- ✓ AMDMM has a Homogenous matrix.
- ✓ Converts wastes - in to resources materials i.e. Brine sludge, Red mud and Rice husk.

WASTE GENERATED IN OIL WELL INDUSTRY.

- **Petrochemical plants generate solid wastes and sludge some which are hazardous in nature due to toxic organics and heavy metals (e.g. spent caustic).**

- **Proper facilities should be provided for handling and storage of hazardous wastes.**

- **For final disposal , recycling/reuse should be given priority.**

- **For incinerable wastes, properly designed incinerators should be installed and non-incinerable hazardous wastes should be disposed in secured landfill (TSDF)**

PRESENTLY THE POLLUTION LOADS IN OIL INDUSRTY IS FROM :

A. Barite waste from drilling mud

B. Waste water

C. Organic compounds

SOLUTION PROPOSED FOR UTILIZING THE WASTE FROM OIL WELL

A. Barite waste from drilling mud can be uses as resource material for making - **Advanced radiation Shielding materials. (Expertise of CSIR-AMPRI, Bhopal A patent In USA)**

B. Waste water

C. Organic compounds .

B & C can be treated using Geopolymeric Materials (expertise of CSIR-AMPRI, Bhopal)

PROBLEM OF OIL SPILLAGE

- **Its one of the most important problem ,having following harmful effects-**

➢ **Oil spill pollution, a severe environmental problem, which persists in marine environment or in inland water across the world, has grown to an alarming magnitude with increased levels of oil production and transport.**

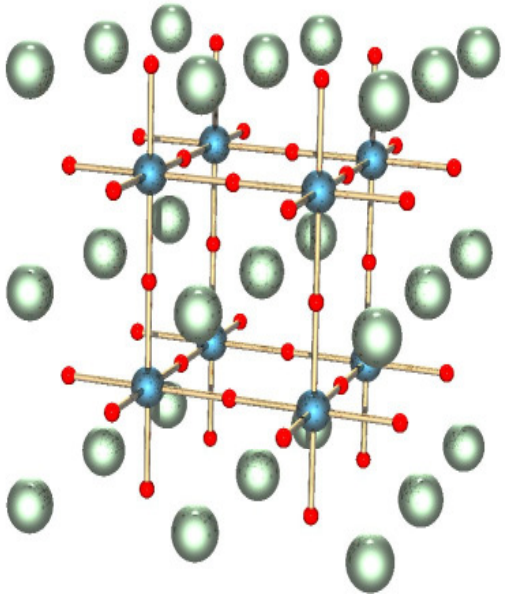
➢ **Oil spilled by damaged tankers, pipelines or offshore oil rigs coats everything it touches and becomes an unwelcome but long-term part of every ecosystem it enters.**

➢ **Developments in petrochemical industry warrants increased oil supply carried through pipelines and ships from places where it is found to the most convenient sites for refineries and chemical manufacturing plants.**

Material for addressing Oil Spillage :

- New approach towards the search for new materials is inevitable to target removal of oil from marine and off shore oil spillage problem.
- The very basis of these new material, are magnetic, high surface area and environmental friendly.
- The material is of huge industrial, environmental and scientific applications.
- The materials processing at sub-micron or nano level will be essential for the new technology.

Red Mud Derived Magnetic Iron Titanium Silicate Mineral Composite for Addressing Oil Spillage Issues



- Concept
- Structure
- Properties

- Practical Issues
- Probable Solutions
- Future Directions



AMPRI's Ceramic powder

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Concept of Developed Material

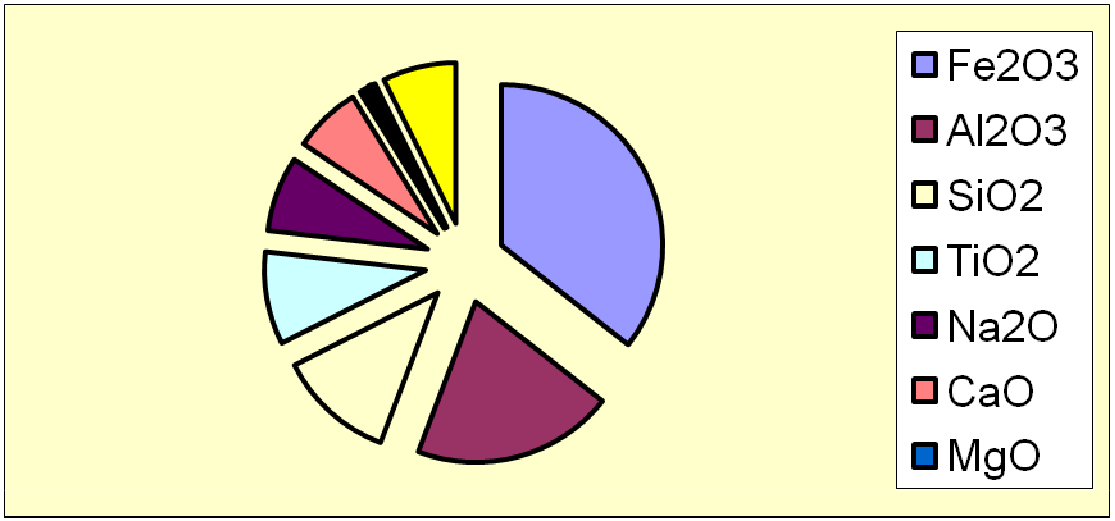
- **The development of advanced ceramic material with tailored properties like surface area which is suitable for removal of oil from marine and off shore oil spillage.**
- **Red mud (aluminium industry waste) will be used to get iron titanium silicate mineral compositions.**
- **The developed material with varying density and compositions will be used for removal of oil from marine and off shore oil spillage.**
- **The composition developed will be devoid of any hazardous elements like lead, chromium, arsenic etc, makes the material environment friendly. Developed materials are not yet mature enough to demand immediate industry attention.**
- **I had be working on the basic aspects of materials synthesis and characterization.**
- **I have enough reasons to believe that developed advanced functional ceramic can be helpful to solve the materialistic problems of oil spillage.**

- **Red mud is the waste generated during Aluminum production from bauxite. 1.0–1.6 tonnes of red mud is generated per tonne of alumina.**
- **It is estimated that over 66 million tonnes is impounded annually in the world.**
- **Disposal of red mud tailing costs industry US$ 3 per tonnes of alumina production.**
- **Redmud is used for making inexpensive and efficient adsorbent for removal of cadmium, zinc and Arsenic, fluoride, lead, chromium from aqueous solutions has also been reported recently.**

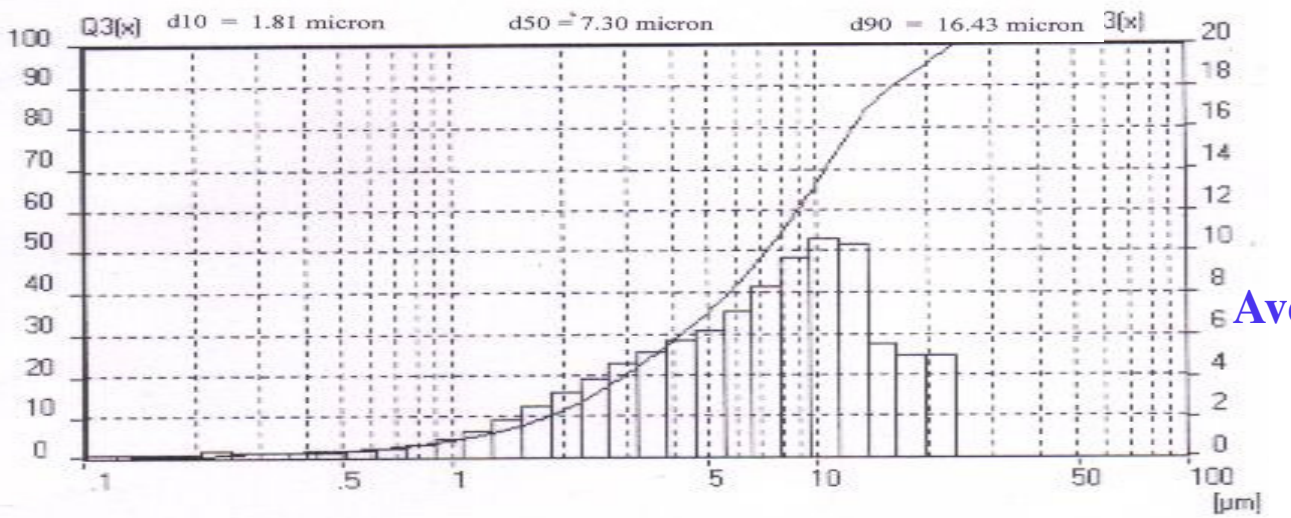




Properties of Resource Material for Designed Ceramic



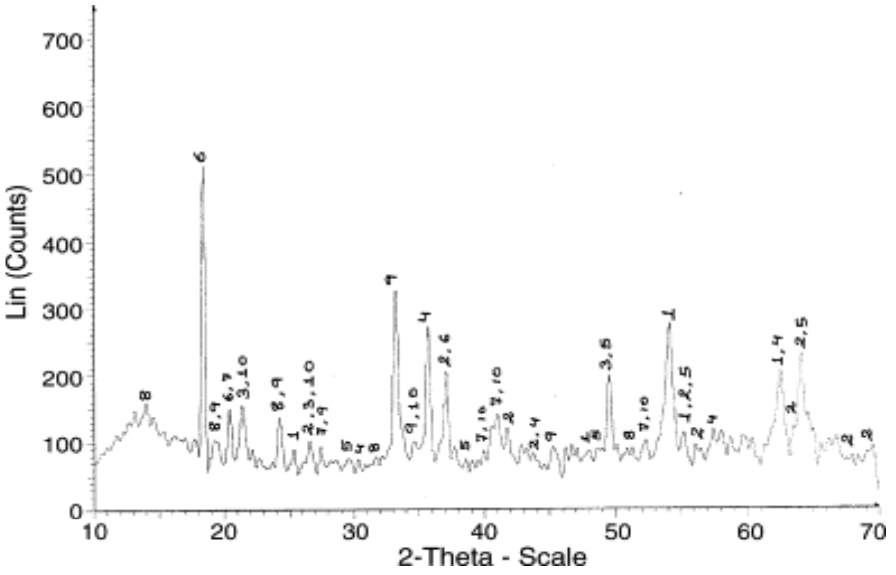
Fe_2O_3 - 31.88 %
TiO_2 - 21.20 %
Al_2O_3 - 20.10 %
SiO_2 - 8.50 %
CaO - 2.99 %
Na_2O - 6.00%.
LOI - 10.0%.



Raw Red Mud Sample
Average Particle Size : 16.43 µm

Red Mud obtained from Hindustan Aluminum Company (HINDALCO) Renukoot, India

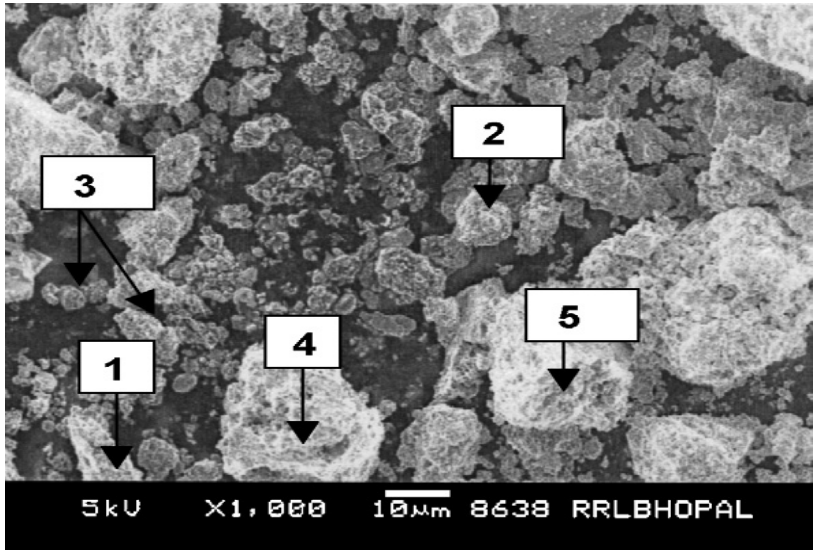
Mineralogical Composition & Surface Profile of Resource Material



Mineralogical Phases in Red Mud Sample:

1. Anatase
2. Rutile
3. Quartz
4. Hematite
5. Bohmite
6. Gibbsite
7. Bayerite
8. $Na_5Al_3CSi_3O_{15}$
9. Cancrinite,
10. Chantalite

X-ray powder diffraction analysis of red mud exhibited presence of a diverse mineralogical phases and compounds which act as a source materials for obtaining varieties desired phases.



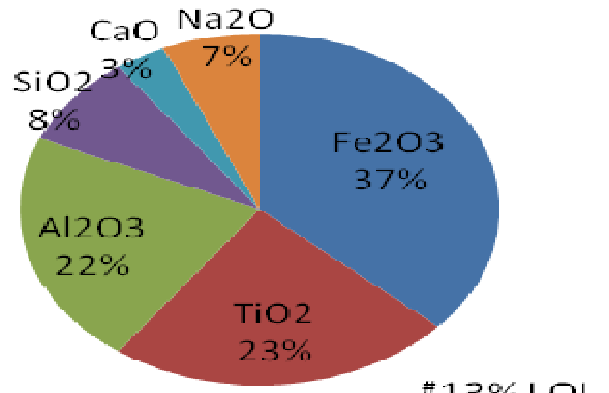
SEM of red mud (scattered morphology):

(1) Tetragonal - Anatase
(2) Tetragonal - Rutile
(3) Hematite Spherical
(4) Hexagonal Cancrinite
(5) Orthorhombic - Boehmite.

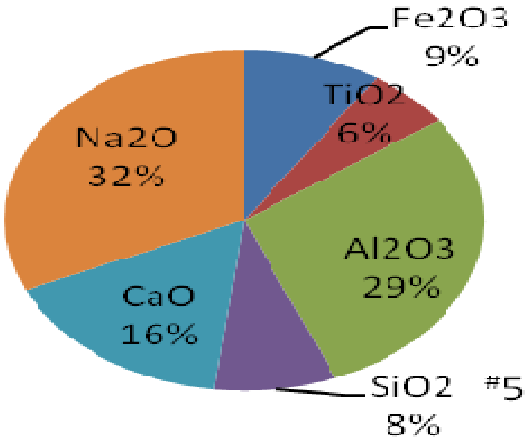
The scanning electron microphotographs exhibiting microstructure of red mud.

Red Mud obtained from Hindustan Aluminum Company (HINDALCO) Renukoot, India

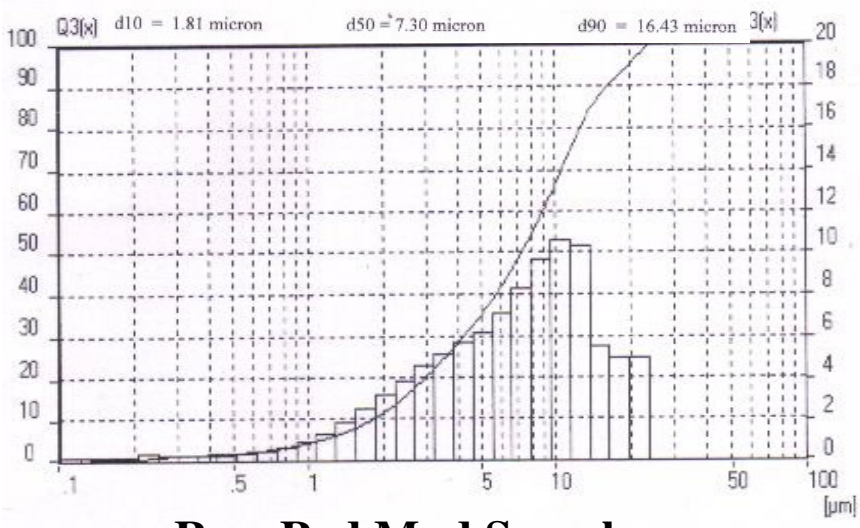
Properties of Functionally Deigned Ceramic for Addressing Oil Spillage Issue



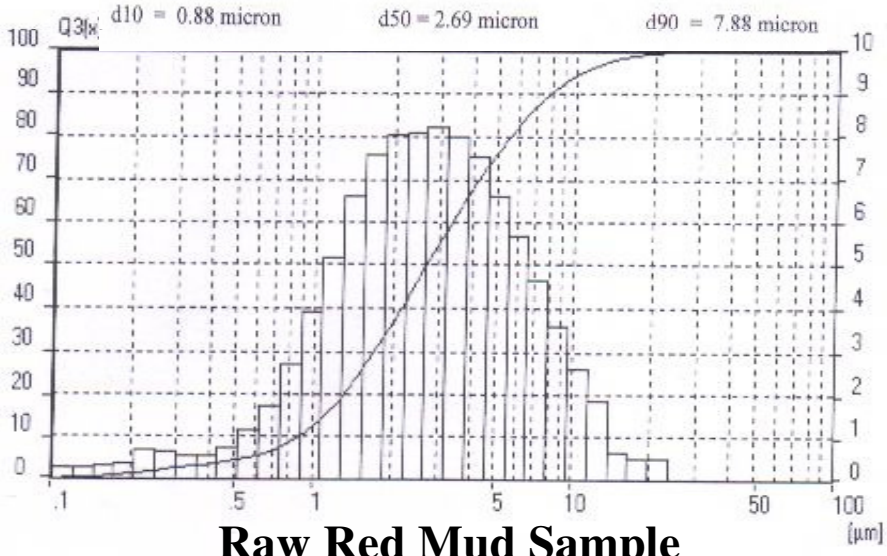
#13% LOI



#52% LOI

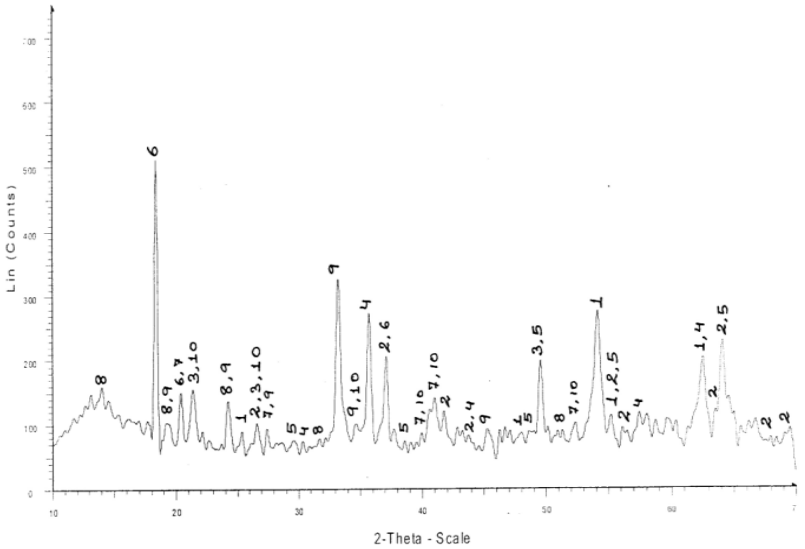


Raw Red Mud Sample
Average Particle Size : 16.43 µm



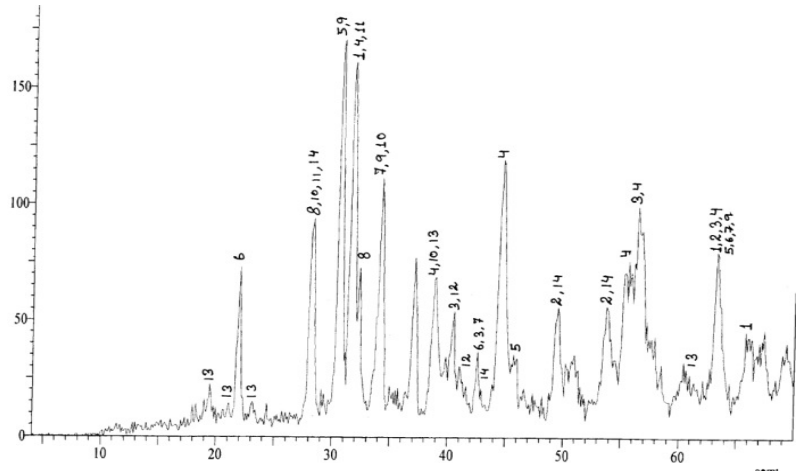
Raw Red Mud Sample
Average Particle Size : 7.88 µm

Mineralogical Composition & Surface Profile Compression



Red mud

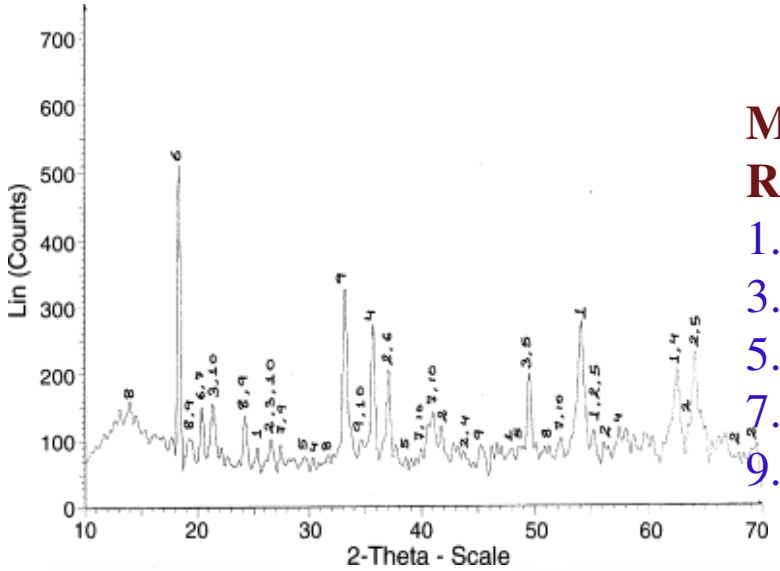
S.no.	Red mud	PDF	Crystal structure
1	Anatase	21–1272	Tetragonal
2	Rutile	21–1276	Tetragonal
3	Quartz	33–1161	
4	Hematite	33–664	Cubic
5	Bohmite	21–1307	Orthorhombic
6	Gibbsite	29–41	
7	Bayerite	20–11	Monoclinic
8	$Na_5Al_3CSi_3O_{15}$	15–469	Hexagonal
9	Cancrinite	25–776	Hexagonal
10	Chantalite	29–1410	Tetragonal



Sample 1 (Ceramic Route)

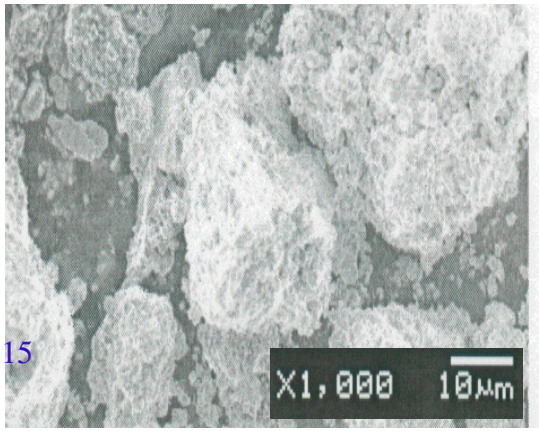
S.no.	RMSM	PDF	Crystal structure
1	Barium iron titanium oxide	26–1032	Hexagonal
2	Iron titanium oxide	13–270	Tetragonal
3	Barium titanium oxide	17–661	–
4	Barium meta titanate	8–372	Hexagonal
5	Barium iron titanium oxide	21–101	Orthorhombic
6	Barium silicate	10–45	–
7	Bafertisite	14–541	Orthorhombic
8	Barium titanium oxide	26–762	Orthorhombic
9	Barium aluminium silicate	28–125	Hexagonal
10	Barium aluminium oxide	28–121	–
11	Iron titanium oxide	9–320	–
12	Magnetite	19–629	FC cubic
13	Aluminium phosphate	20–44	
14	Iron phosphate		

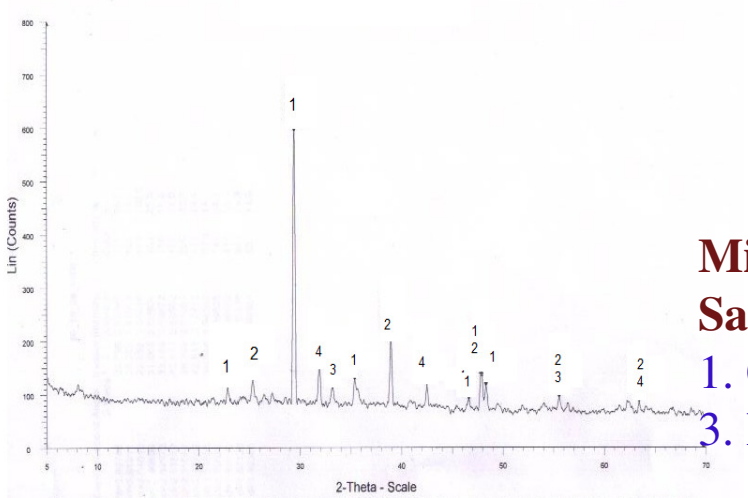
Development of Functional Ceramic by Spray Pyrolysis Technique



Mineralogical Phases in Red Mud Sample:

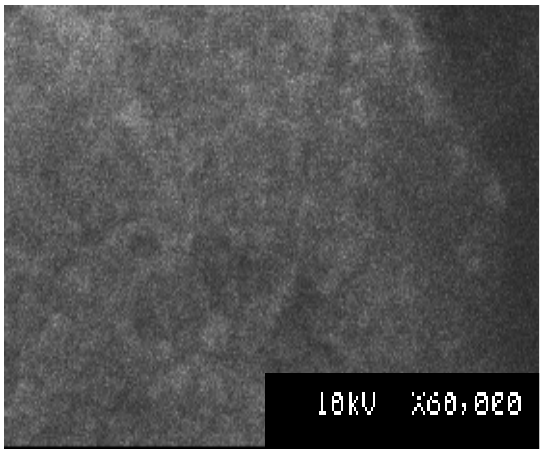
1. Anatase
2. Rutile
3. Quartz
4. Hematite
5. Bohmite
6. Gibbsite
7. Bayerite
8. $Na_5Al_3CSi_3O_{15}$
9. Cancrinite,
10. Chantalite





Mineralogical Phases in Sample 2 for oil spillage issue:

1. Calcite
2. Anatase
3. Hematite
4. Cancrinite,



Significant Features of Designed Ceramic for Addressing Oil Spillage Issue

- **Tailored properties and oil removal by sorption (adsorption & absorption both)**
  - **a) Low density (1.2 gm/cc)**
  - **b) High Porosity for oil sorption**
  - **c) Magnetic in nature for removal of oil loaded material from liquid surface.**
  - **d) High temperature stability.**
- **Oil removal from both solid and liquid surfaces.**
- **Geomimic origin: Iron Titanium Silica based material.**
- **Non toxic, Non hazardous and Environment friendly due to devoid of any hazardous elements.**
- **Non explosive, non corrosive and fire resistant.**
- **Material can be recovered and reused again.**
- **Mechanization of the processes is in progress.**

Up scaling of Developed Material for Commercial Viabilities Studies

Lab Scale development to Semi Pilot Plant Level: Processing of 3 Tons of Red mud

9 Tons Red Mud Collected
from HINDALCO, Renukoot to AMPRI, Bhopal

Ceramic Powders Synthesis using the facilities of
CDGI, Firozabad, India

Activities at CDGI under guidance of AMPRI, Bhopal India

1. Grinding of Red Mud Lumps to Red mud Powders
2. Formulation & making balls of Red Mud Powders
3. Heat Treatment of Red Mud balls at 13000C
4. Grinding of multiphase shielding material (range 100 to 200 mesh)



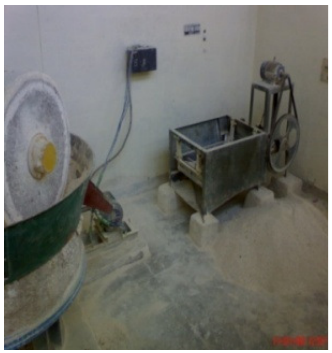
AMPRI's Ceramic powder



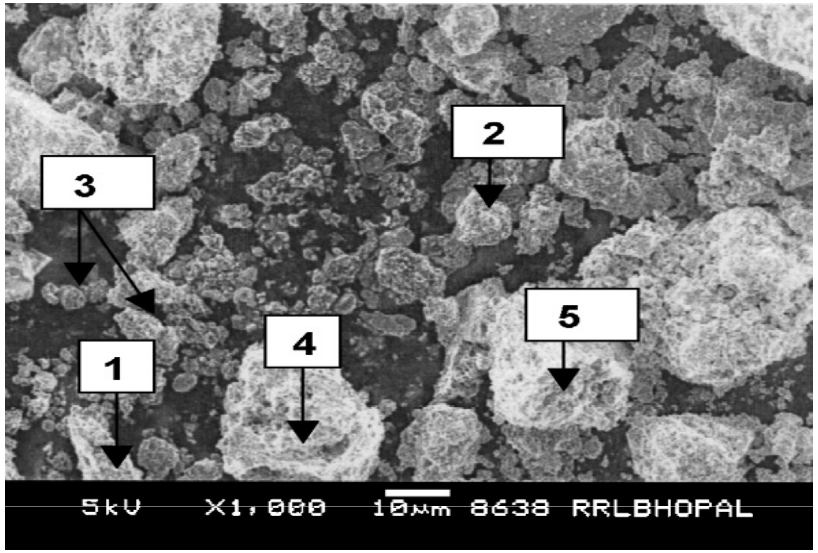


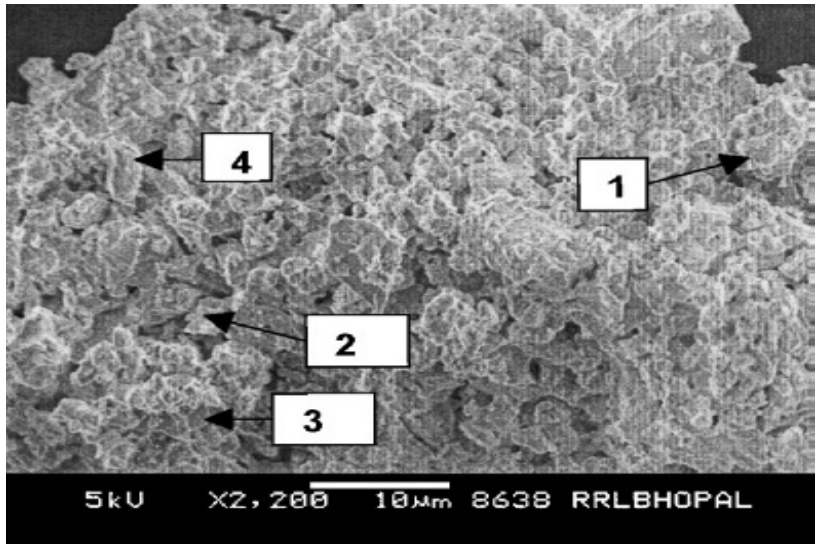






Surface Profile Compression





SEM of red mud (Scattered Morphology):

(1) Tetragonal - Anatase

(2) Tetragonal - Rutile

(3) Hematite Spherical

(4) Hexagonal Cancrinite

(5) Orthorhombic - Boehmite.

The scanning electron microphotographs exhibiting microstructure of red mud.

SEM of red mud (Modified Porosity):

(1) Hexagonal – Barium Iron Titanium Oxide

(2) Cubic - Magnetite

(3) Orthorhombic -Befertisite

The microphotographs of advanced ceramic sample clearly show the modified and continues porosity with integrated morphological characteristics.

Affected areas near Mumbai sea shore:





Floating oil at Elephanta Cave sea shore





Oils Rocks at Sea Shore of Elephanta Cave sea shore and Marine Derive, Mumbai





Oil on Mangroves and Sand at PDP, Mumbai on

Targeting : Mumbai Oil Spill removal of floating oil and oils on sea shore rocks

Operation Performed on:

1. **Floating oil on the sea water at Elephanta Cave sea shore.**
2. **Oil layer on Rocks at Elephanta Cave sea side rocks and Marine drive, Mumbai.**
3. **Sand samples at PDP, Mumbai**



Dr. R. Kumar, NEERI, Mumbai Zone and
Dr. Amritphale regarding oil removal operation



Two Tons of AMPRI material send to NEERI Mumbai Zone.





Fabrication of demo apparatus for removal of floating oil manually .

Targeting : Mumbai Oil Spill removal of floating oil and oils on sea shore rocks

During 26th Aug. to 28th Aug. 2010, Elephanta Cave sea shore and Marine drive, Mumbai






AMPRI and NEERI team at removal of oil at Mumbai sea shore






Removal of floating oil using AMPRI' developed material

Analysis of Mumbai Spill removal of floating oil and oils on sea shore rocks

Initial and Final Site: Floating oil removal from Sea and Sea Rock



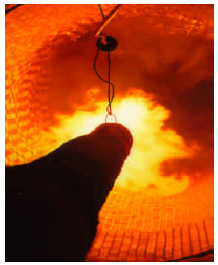


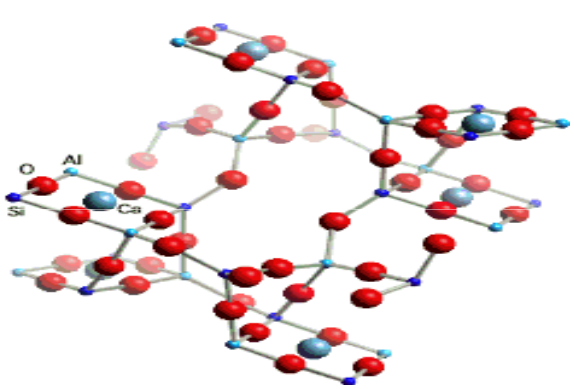

Analysis of samples at AMPRI

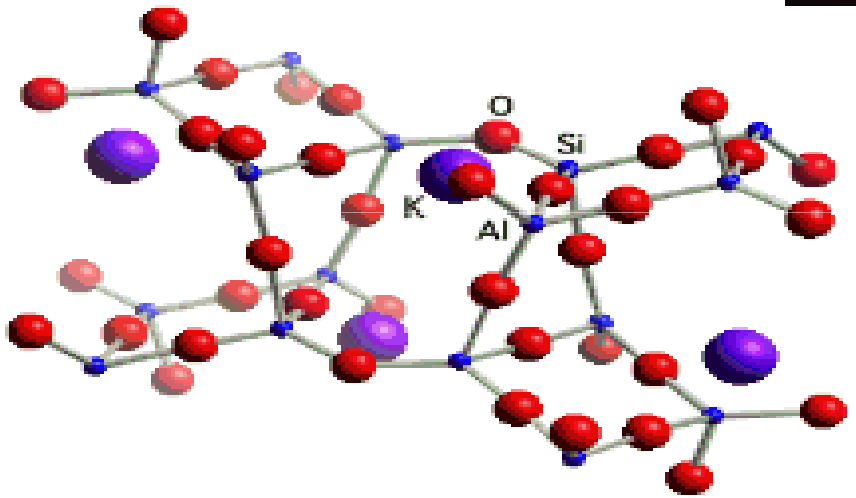




Due to severe Draw backs of Ordinary Portland cement Conventional Geopolymer Cement Concrete (CGC) emerged as possible solution







Geopolymers

Conventional Geopolymeric Materials Used for Cementing Oil Wells

✓Geopolymer cement

✓Pumpable geopolymer formulation comprising:

- an alumino silicate source,
- a carrier fluid, activator,
- boron containing compound, lignosulfate, sodium gluconate, sodium glucoheptonate, tartaric acid and phosphorus containing compound, or a mixture of them as retarder

-Lithium or potassium compound as accelerator

-cenospheres, sodium- calcium-borosilicate glass, and silica-alumina microspheres as Lightweight particle .

-Manganese tetraoxide, iron oxide (hematite), barium sulfate (barite), silica and iron/titanium oxide (ilmenite) as heavy particle .

-Water-immiscible oil-based phase.

✓Microwave Incinerated Rice Husk Ash (MIRHA) geopolymer

Portland cement

$5CaCO_3+2SiO_2 \longrightarrow 3CaO.SiO_2+2CaO.SiO_2+5CO_2$

1450ºC

Clinker Phases

Hydration (curing with water

C-S-H + $Ca(OH)_2$

Geopolymeric cement

$Al_2O_3+SiO_2$ in raw material

20-90ºC

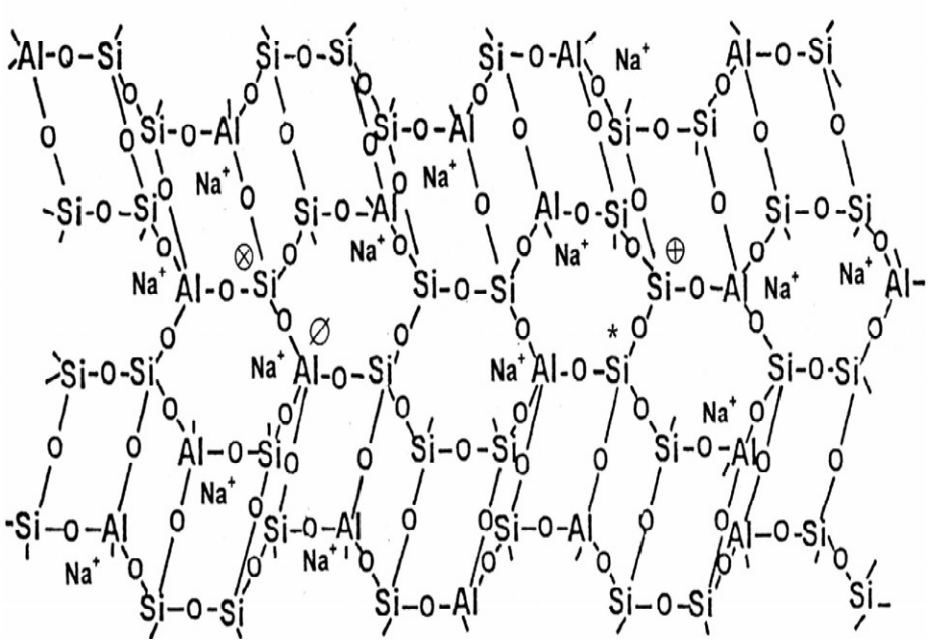
+alkali silicate solution

-Si-O-Al-O-

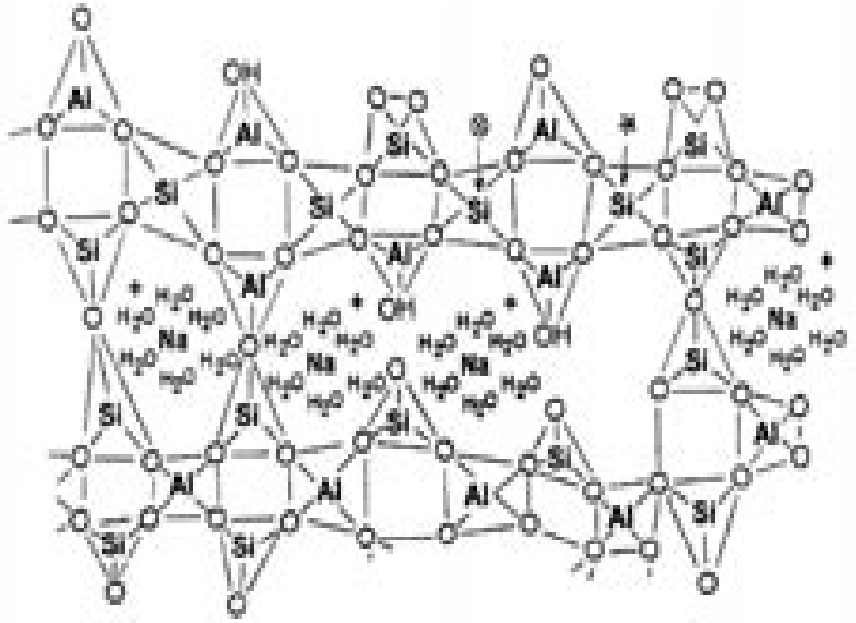
non curing

Alumino silicate 3D framework

Characteristics of OPC Compared with Conventional Geopolymer



Davidovits model of geopolymer structure

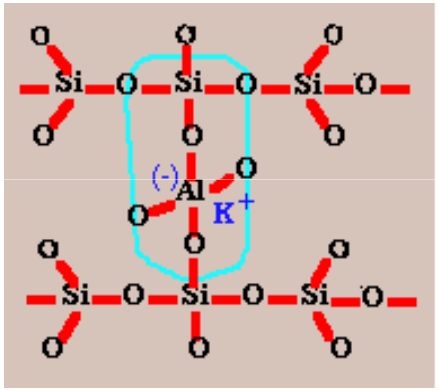


Barossa model of geopolymer structure

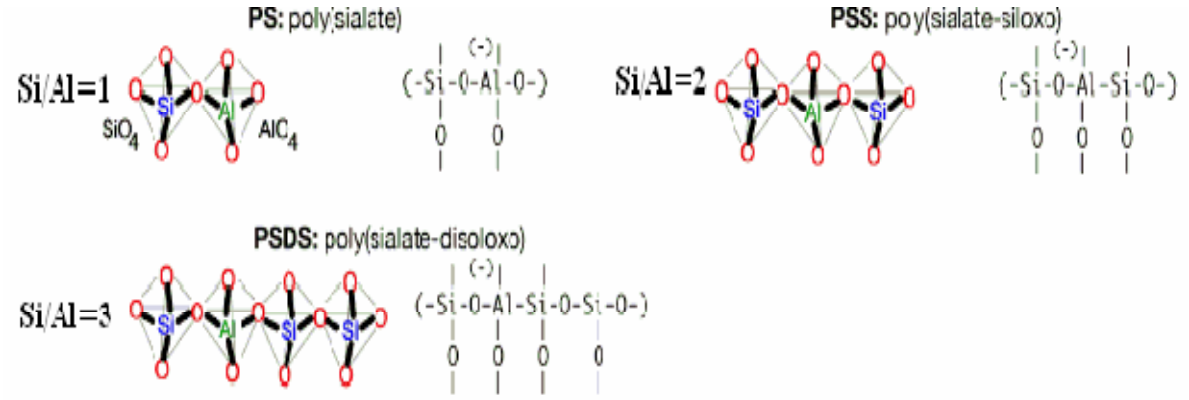
- Amorphous (gel like), partially amorphous or crystalline
- Network configurated of SiO_4 and AlO_4 tetrahedrons united together by oxygen bridges

Geopolymers (earthly originated inorganic polymeric materials)

- New family of synthetic aluminosilicate materials Mimicking synthesis of natural volcanic Zeolitised rocks
- Macromolecules linked by covalent bonds having – Si-O-M-O- backbone, Where M denotes principally aluminium and other Metals such as iron

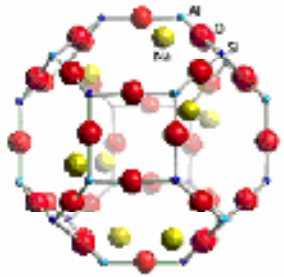


Geopolymeric structure showing Al-O-Si bonds

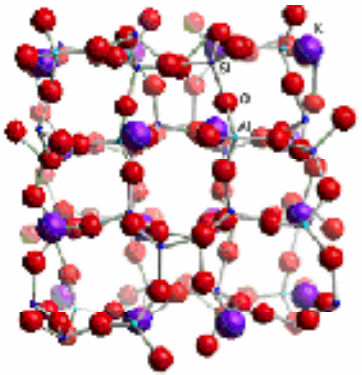


Three basic forms of geopolymer

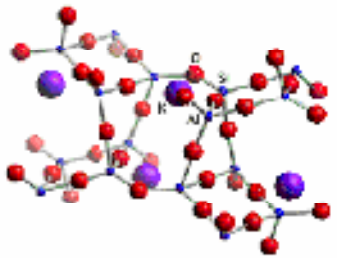
Polymeric structures from polymerization of monomers



PS-Si/Al=1



PSS-Si/Al=2

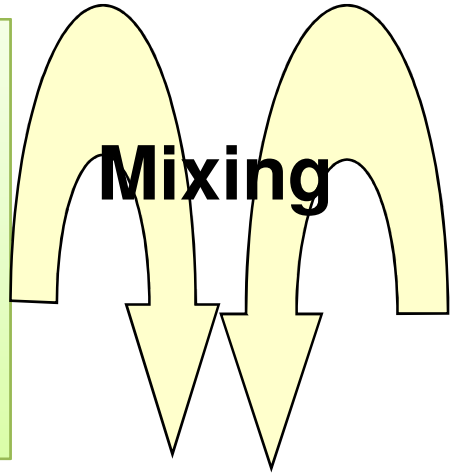


PSDS-Si/Al=3

Formation of Geopolymer

Binding Material

- Metakolin
- Clay
- Slag
- Fly Ash

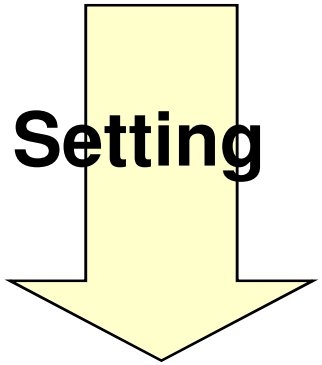


Alkaline Activator

- NaOH/ KOH
- Sodium Water Glass
- Potassium Water Glass-
- ------

+ Water

Binder



Geopolymer

Advantages of Geopolymeric Materials Used for Cementing Oil Wells

- ✓Economical in production
- ✓Sustainable in reducing waste products
- ✓Consumes less energy
- ✓doesn't undergo chemical attacks
- ✓durable
- ✓resistive in acidic environments
- ✓can be used in CO_2-rich environments.
- ✓High Strength
- ✓Excellent volume stability
- ✓Anti-corrosive
- ✓Can be used in marine environment
- ✓Possess higher strength compared to OPC.
- ✓Controllable thickening and/or the setting times of suspension.
- ✓Emits approximately 90% less CO_2 and requires 50% less energy in production due to its low process temperature compared to OPC.
- ✓The cost of geopolymer concrete manufacture is 10-30% less than that of OPC concrete

Word of appreciation by father of geopolymers

- Video clipping



GPCamp-2013-keynote-cut-cut.mpg



Rice Husk



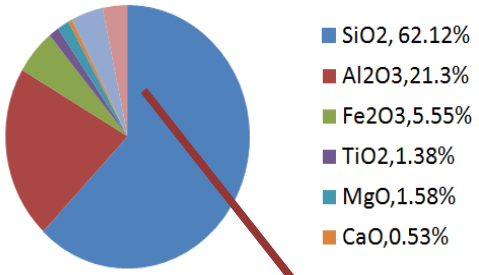
Fly Ash

INNOVATION BY CSIR-AMPRI, BHOPAL

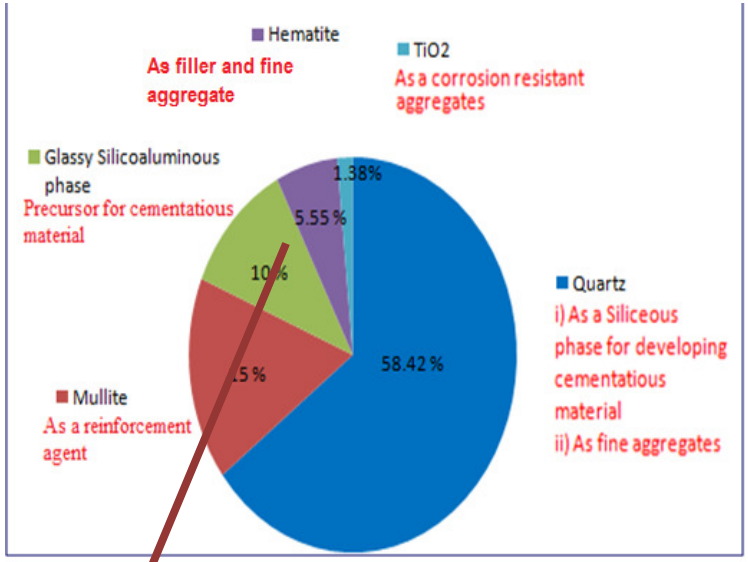
Development of curing free , Multifunctional, Ecofriendly, NEW Novel Hybrid Advanced Ligno - Silco -Aluminous geopolymeric Cementitious Material (ALSAGC)

Mechanism of the Process

Raw material number 1. Fly Ash

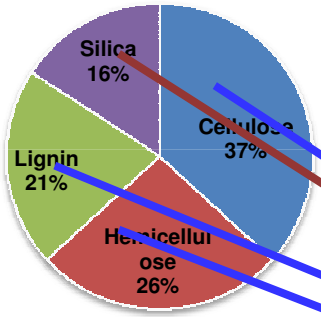


Typical Chemical Analysis of Fly Ash



Precursors From Fly Ash

Raw material number 2. Rice Husk



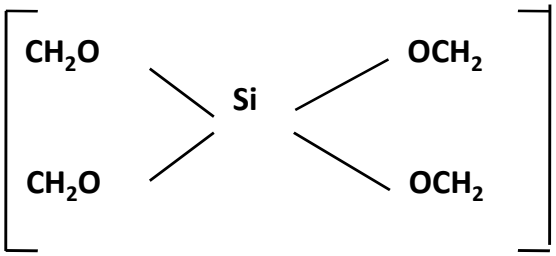
Raw material number 3. Sodium hydroxide

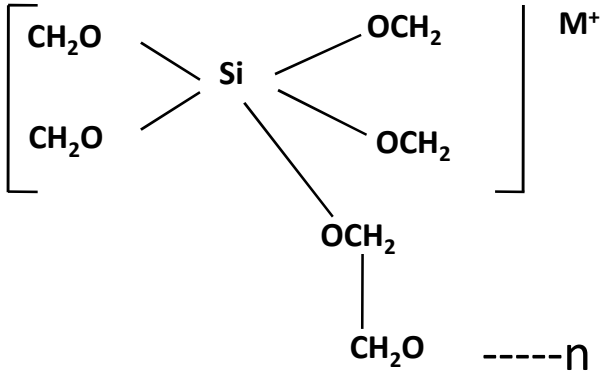
Conventional Tetrahedral silicon complexes

-OH moieties of organic precursors

Novel Penta coordinated silicon complexes

In-situ Synthesis of sodium silicate





Plausible Chemical Reactions

I. Sodium Hydroxide + Lignin in Rice husk ⟶ Sodium lignate **(Binder)**

II . Sodium Hydroxide+ Silica from Rice Husk and Silica and Alumina from Fly ash and Red Mud ⟶ Sodium silicate precursor **(Necessary for making inorganic matrix)**

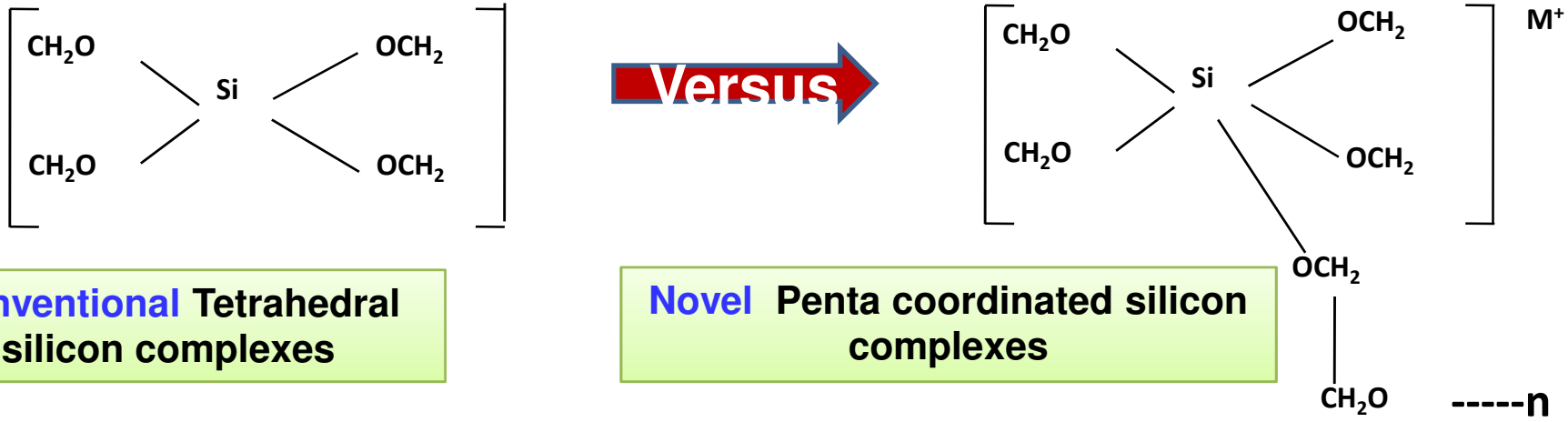
Novel Patented* Concept of CSIR-AMPRI, Bhopal for Advanced Ligno-Silico-Aluminous (LSA) Materials

(India (2301 DEL 2012) and USA (13 / 949585) .*

Conventional Geopolymeric Material

Advanced LSA Material

-OH moieties of organic precursors



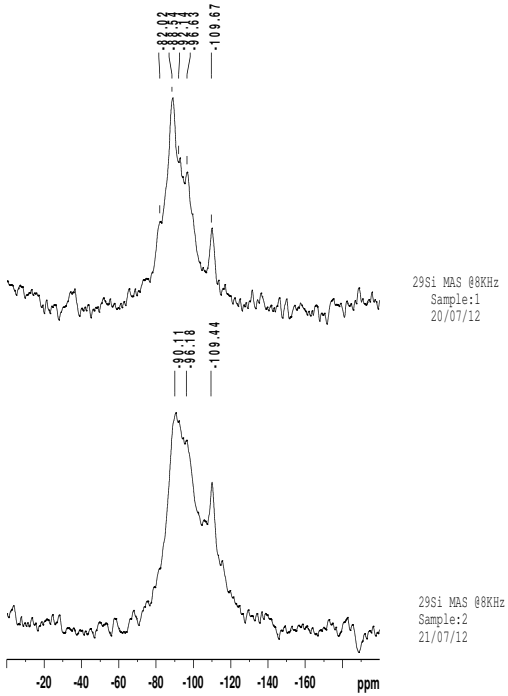
Conventional Tetrahedral silicon complexes

Novel Penta coordinated silicon complexes

✓Advanced LSA Material enables improved Engineering Properties e.g. workability, compressive strength etc.

✓Know-How transferred to M/s JSPL, Raigarh and demonstration completed successfully.

^{29}Si NMR Studies



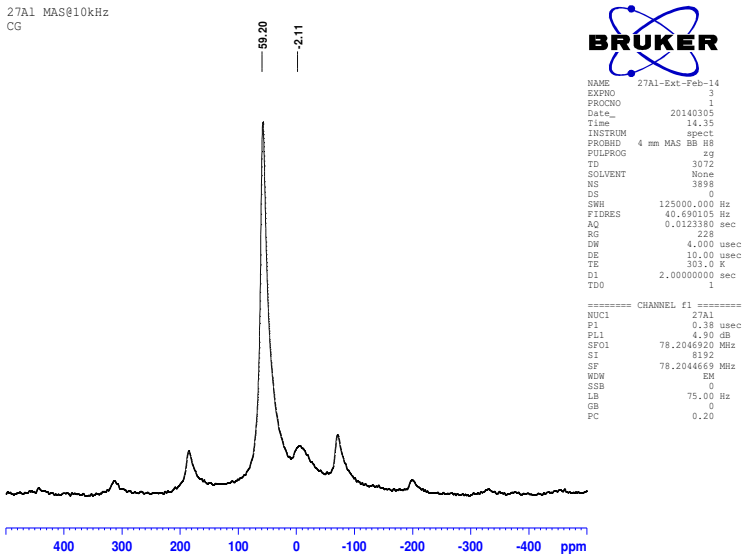
Conventional geopolymeric material: ^{29}Si NMR of conventionally developed geopolymeric material indicated NMR Signals at δ-82 ppm due to tetracoordinated geometry and signals at δ -92, δ -96 and δ -109 ppm indicated pentacoordinated geometry around silicon atom.

Advanced hybrid organic- inorganic Geopolymeric material: ^{29}Si NMR at δ -90 ppm, δ -96 ppm and at δ -109 ppm indicated pentacoordinated geometry around silicon.
Absence of NMR signals at δ -82 ppm, indicates absence of tetracoordinated geometry around silicon atom.

Conclusion of ^{29}Si NMR Studies:

- ✓ Thus complete transformation of tetracoordinated silicon to pentacoordinated silicon is confirmed in presence of alkali and organic molecules from rice husk in hybrid inorganic organic geopolymer.
- ✓ The bond length increases up to 5 % in pentacoordination which imparts improved flexibility , reactivity and thus functionality in hybrid inorganic organic geopolymer.
- ✓ Presence of organic as well as inorganic linkages in IR studies further confirmed the participation of molecules from Rice husk.

^{27}Al NMR Studies

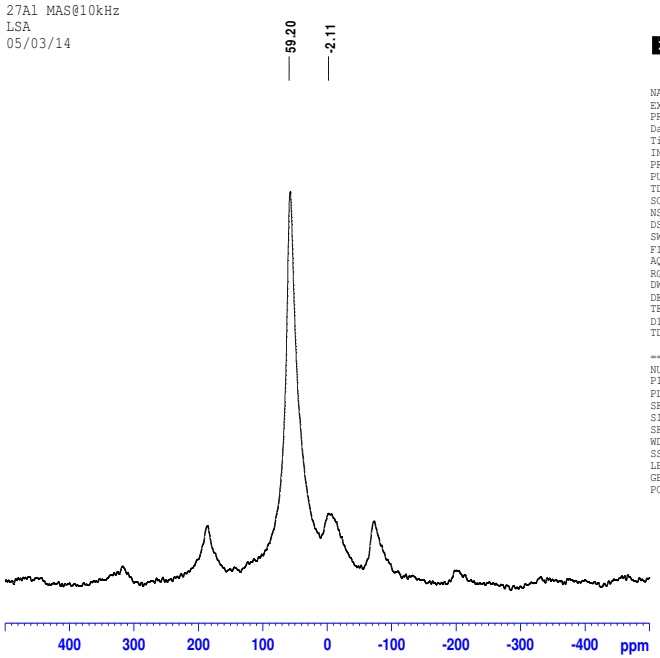


BRUKER

```
NAME            27Al-Ext-Feb-14
EXPNO                        3
PROCNO                       1
Date_                 20140305
Time                     14.35
INSTRUM                  spect
PROBHD    4 mm MAS BB H8
PULPROG                     zg
TD                        3072
SOLVENT                   None
NS                        3898
DS                           0
SWH                125000.000 Hz
FIDRES              40.690105 Hz
AQ                   0.0123380 sec
RG                         228
DW                     4.000 usec
DE                    10.00 usec
TE                     303.0 K
D1                  2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       27Al
P1                      0.38 usec
PL1                      4.90 dB
SFO1                78.2046920 MHz
SI                         8192
SF                  78.2044669 MHz
WDW                          EM
SSB                          0
LB                      75.00 Hz
GB                           0
PC                        0.20
```

^{27}Al NMR of Conventional geopolymer



BRUKER

ampri CSIR-AMPRI

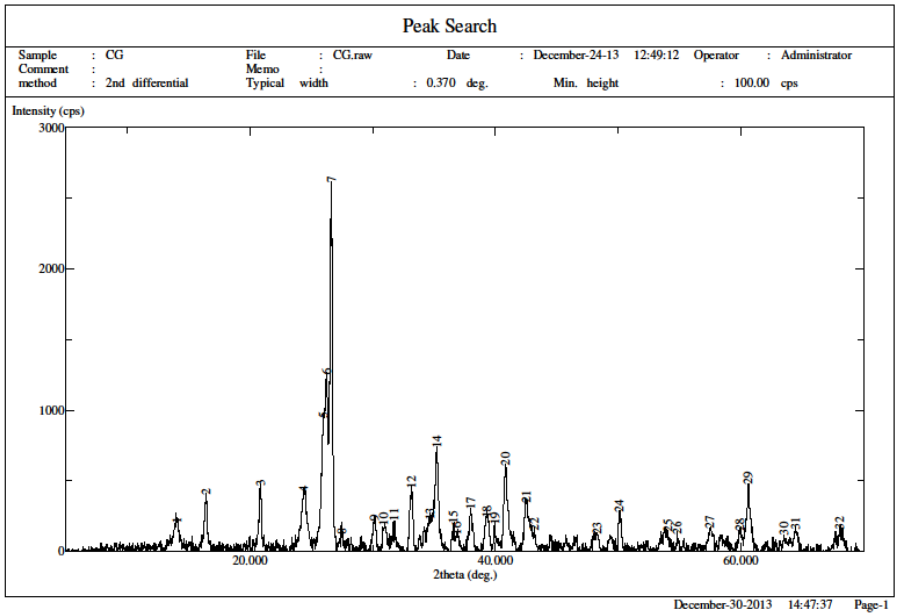
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EXPNO                        4
PROCNO                       1
Date_                 20140305
Time                     15.31
INSTRUM                  spect
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PULPROG                     zg
TD                        3072
SOLVENT                   None
NS                        4000
DS                           0
SWH                125000.000 Hz
FIDRES              40.690105 Hz
AQ                   0.0123380 sec
RG                         228
DW                     4.000 usec
DE                    10.00 usec
TE                     303.0 K
D1                  0.50000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       27Al
P1                      0.38 usec
PL1                      4.90 dB
SFO1                78.2046920 MHz
SI                         8192
SF                  78.2044669 MHz
WDW                          EM
SSB                          0
LB                      75.00 Hz
GB                           0
PC                        0.20
```

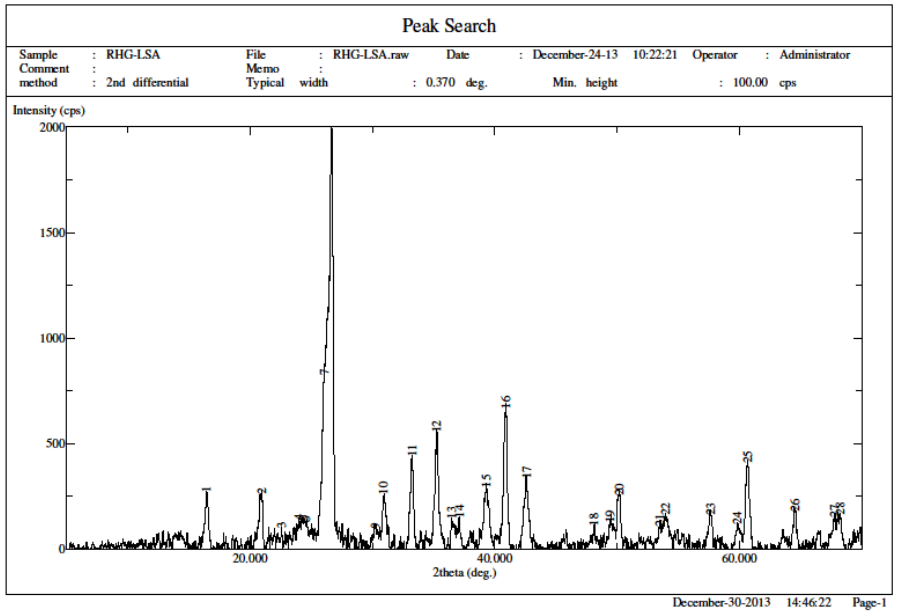
^{27}Al NMR of hybrid inorganic organic geopolymer

Conclusion: The presence of ^{27}Al NMR signal at 55 ppm which is characteristic of geopolymer formation is found in both conventional geopolymer as well as in hybrid inorganic-organic geopolymer and thus confirmed the formation of geopolymeric material.

XRD Studies



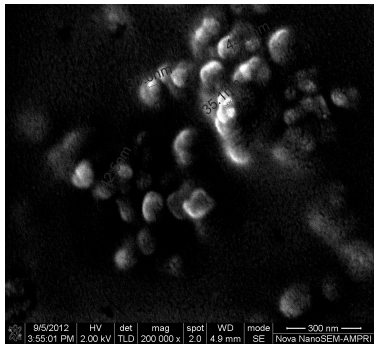
XRD pattern of Conventional geopolymeric material



XRD pattern of Hybrid inorganic organic geopolymeric material

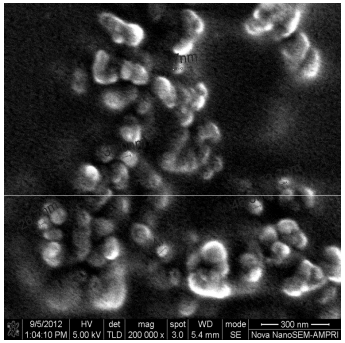
Conclusion: The XRD studies confirmed the simultaneous presence of inorganic phases namely Cancrisilite ,Herschelite, sodium aluminium silicate, quartz, mullite, hematite and organic phases namely native cellulose, α D-glucose and sucrose leading to the formation of Hybrid inorganic organic geopolymeric material.

FESEM Studies



The absence of organic molecules in conventional geopolymeric material restricts size of particles as low as 36.7 nm only

FESEM -conventional geopolymeric materials

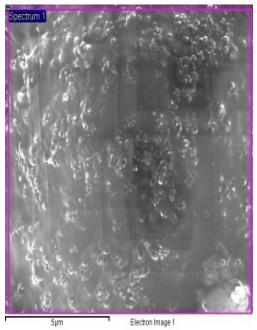


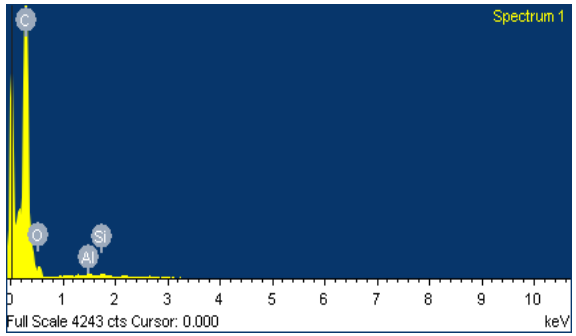
The presence of organic molecules in hybrid inorganic-organic geopolymeric material helps in obtaining size of particles as low as 26.7 nm

FESEM-Hybrid inorganic-organic geopolymeric material

Conclusion: The lesser nanosize obtained in hybrid inorganic organic materials helps in continuous growth of silicon- aluminium oxygen polymeric chains which increases amorphous nature and densifies the matrix leading to homogeneous, strong and flexible matrix with improved properties.

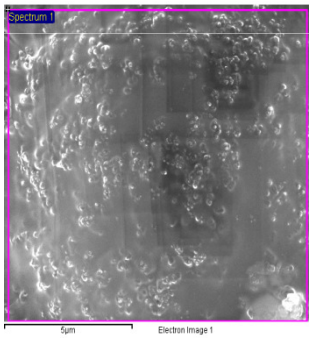
EDX spectra of hybrid inorganic-organic geopolymeric material

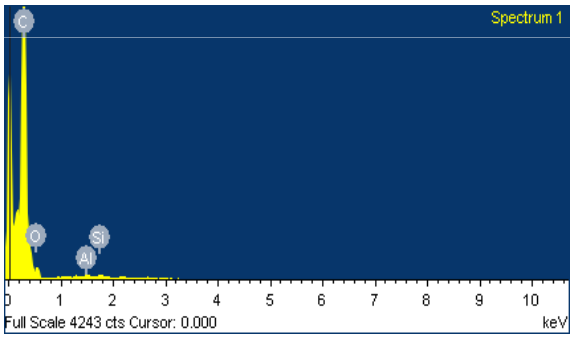




Element	Weight%	Atomic%
C K	88.16	91.96
O K	9.90	7.75
Al K	0.22	0.10
Si K	0.20	0.09
Au M	1.52	0.10
Totals	100.00	

Example

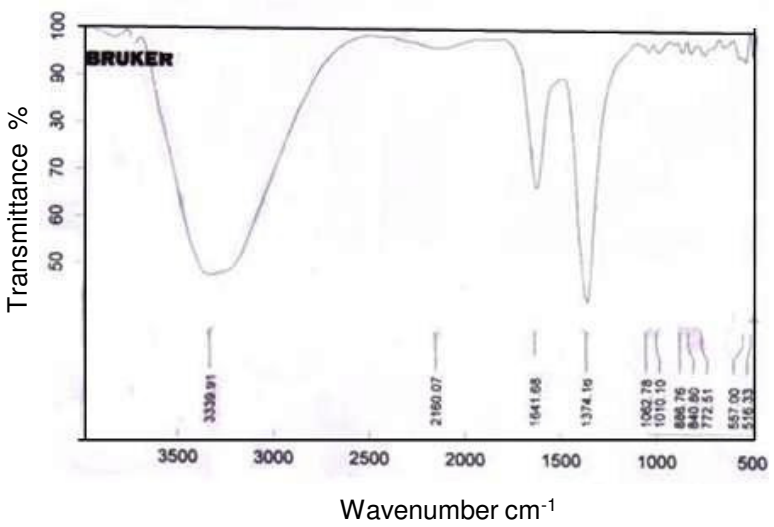




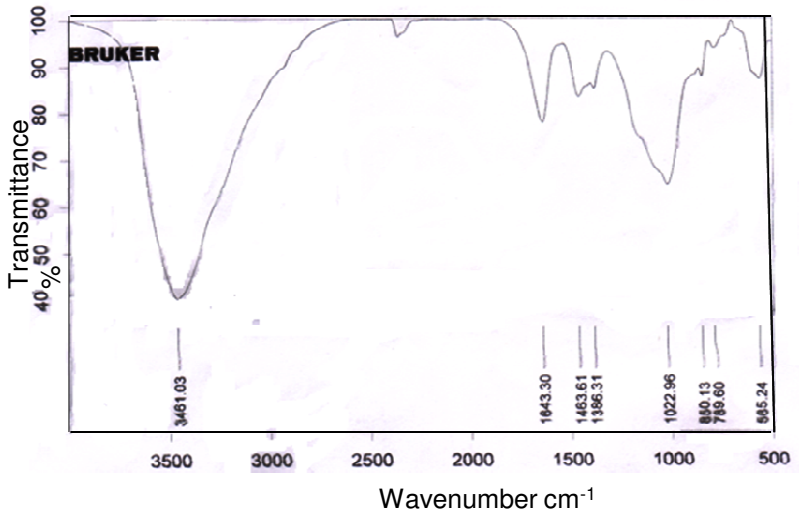
Element	Weight%	Atomic%
C K	88.16	91.96
O K	9.90	7.75
Al K	0.22	0.10
Si K	0.20	0.09
Au M	1.52	0.10
Totals	100.00	

Conclusion: The elemental analysis by EDX indicates presence of both carbon from organic molecules and Al and Si from fly ash, confirming the Hybrid nature of the geopolymeric material.

IR Studies



IR spectra of conventional geopolymeric material



IR spectra of hybrid inorganic-organic geopolymeric material

Conclusion: Thus additional peaks due to organic molecules are observed in IR spectra of Hybrid inorganic organic geopolymer in addition to inorganic phases observed in conventional geopolymerS. Thus confirming the participation of organic molecules from rice husk in presence of alkali during Geopolymerisation.

Superior Properties and comparison of Developed Advanced Ligno - Silco -Aluminous geopolymeric Materials.

1-Containing Hybrid Organic-Inorganic Matrix
2-Better Workability
3-Less Brittle Nature
4UniformHomogeneos Matrix
5-Better Compressive strength
6-Enhanced Oil Recovery due to Lignin in Rice Husk

Engineering Properties *As per IS : 516-1999*		
Properties	**Advanced Geopolymeric Materials**	**Conventional Geopolymeric Materials**
Compressive Strength	**20- 30 MPa**	**16-25 Mpa**
Flexural Strength	**2 - 4 MPa**	**1-2 Mpa**
Density	**2300 - 2500 Kg/m^3**	**1850-2100 Kg/m^3**

Applications

Advanced Ligno-Silico-Aluminous Materials

Low Tech

Green concrete
- ✓ Irrigation channels
- ✓ Fire Proof Blocks
- ✓ Heat insulating panels

Medium Tech

- ✓ Cement Free advanced reinforced green concrete
- ✓ Paver Blocks
- ✓ Alkali Activated Inorganic Path (Roads)

High Tech

- ✓ Aerospace
- ✓ Automotive Industries
- ✓ Biomedical applications
- ✓ OIL WELL INDUSTRY

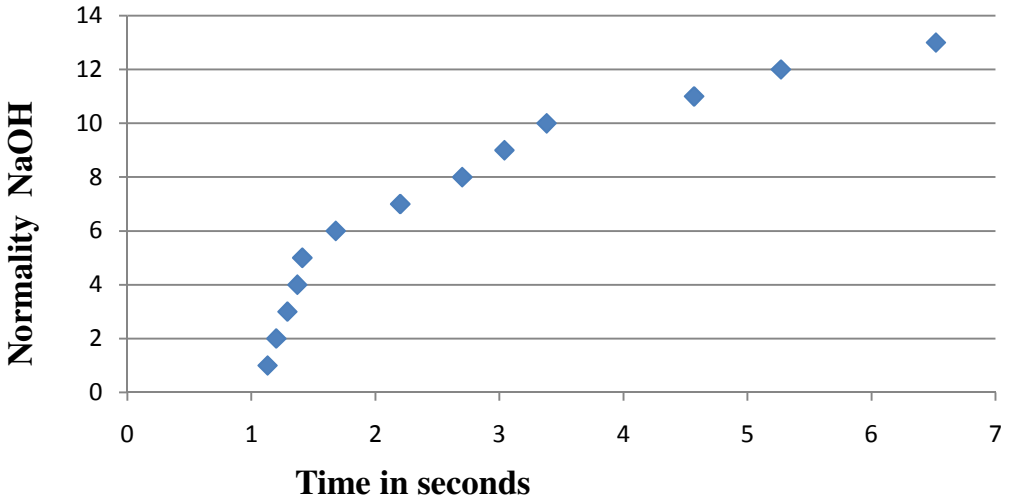
Strategic

- ✓ DESIGN MIX of shielding concrete
- ✓ shielding materials based on nano materials
- ✓ On-site and In-situ chemical encapsulation of Radioactive wastes

LIGNO-SILICO ALUMINOUS ALKALINE LIQUID AS DRILLING FLUID

The LSA can be used as oil well drilling fluid.

By taking constant quantity of rice husk and heating it with different known Normality of NaOH solution , the viscosity of filtrate has been measured at constant temperature and similar concentration of the filtrate .



The graph obtained proved that with the increase in the concentration of NaOH , the time taken for complete flow of filtrate also increases i.e viscosity increases.

The developed material can be used as good drilling mud as it should have high viscosity to carry cuttings to the surface and suspends weighting agents in the mud

2- UTILIZATION OF SEA SAND AND SEA WATER FOR MAKING LIGNO- SILICO-ALUMINOUS MATERIAL FOR APPLICATION IN OIL WELL CEMENTING

Novelty of the Work:

Sea sand and sea water cannot be used in making Conventional Cement Concrete

1. **Ligno- Silico- Aluminous Material can be made using sea sand and sea water.**
2. **Precious saving of river sand and fresh water.**
3. **Environment friendly, addresses global warming.**
4. **Water curing free.**
5. **Can be used for oil well cementing**

Methodology Adopted:



Collection of Sea Sand & Sea Water at Puri Beach



Sample Preparation using Sea Sand and Sea Water

•Engineering Properties:

Compressive Strength (MPa)

	7-DAYS	28-DAYS
Puri Sea Sand/Sea Water	21.7 – 24.2	28.5 – 30.6
Mumbai Sea Sand	15.5-18.0	19.2 – 24.4

•Evaluation of other properties as per IS in progress



Rice Husk

3-UTILIZATION OF LSA FOR MAKING : PANELS MANMADE ON SITE



Fly Ash

Studies carried Out on Advanced Ligno-Silico-Aluminous Materials at CSIR-AMPRI

➢ Panels: Manmade on Site





On-site Manual Preparation of Panels



Panel
1 x 1 feet



Irrigation
Channel



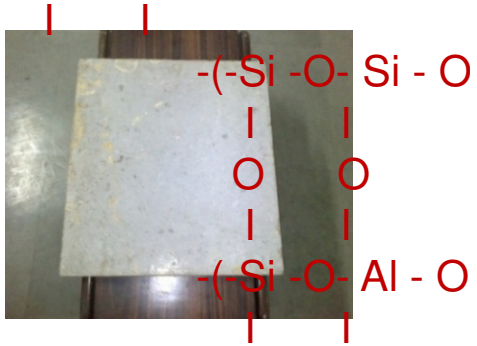
Cube
(15 cms x 15cms
x 15 cms)

➢ Prefabricated Partition Panels- By Mechanization





Mechanical Mixing Mechanical Pressing



Panel 2 x 2 feet

Properties of Advanced, Ligno- Silico- Aluminous (LSA) Cementitious Heat Insulating Panels/ Blocks

Mouldable

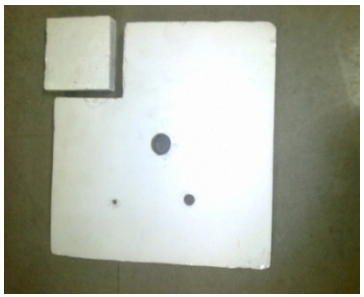


Block For Acoustic Property



Block, 10 x 10 cms

Machineable



Block, 1 x 1 feet



Panel , 2 x 2 feet

Properties-Obtained

Property	Value
Density (Kg/m^3)	1200-2500
Compressive Strength (MPa)	20-35
Flexural Strength (MPa)	3-4
Water Absorption (%)	5-8
Machinability	Possible
Mouldability	Possible

Properties- Targeted

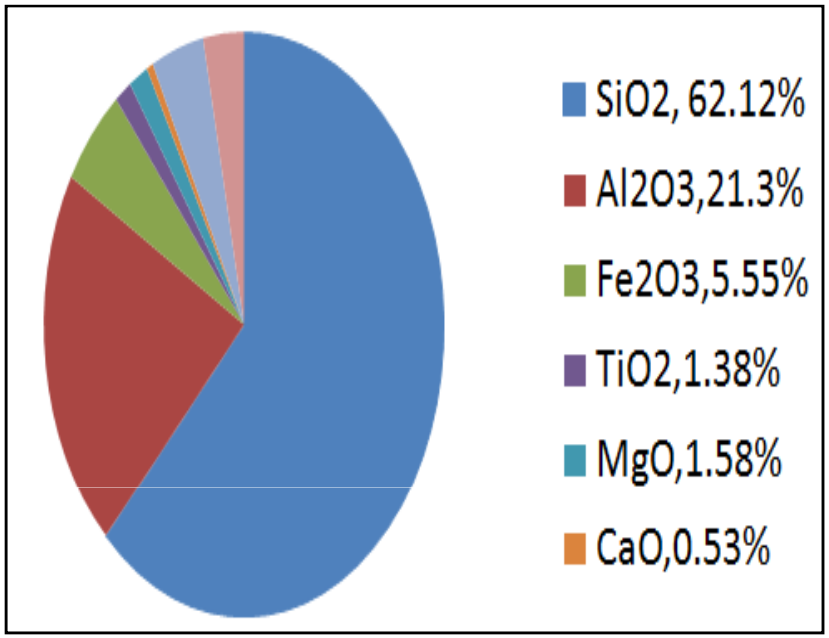
Property	Value
Density ((Kg/m^3) (IS 2185-1978 Part I and II)	900-2900
Compressive Strength (MPa) (IS 2185 1978 Part II)	30-40
Flexural Strength (MPa) (IS 516 -1959)	5-7
Water Absorption (%) (IS 2185-1978 Part I and II)	<40
Thermal Conductivity (W/m deg K) (50 mm to 75 mm) (BS4370-Part 2)	0.21-0.22
Fire Reistance (100 mm) (IS 3809-1995)	4h

* Patent being filed

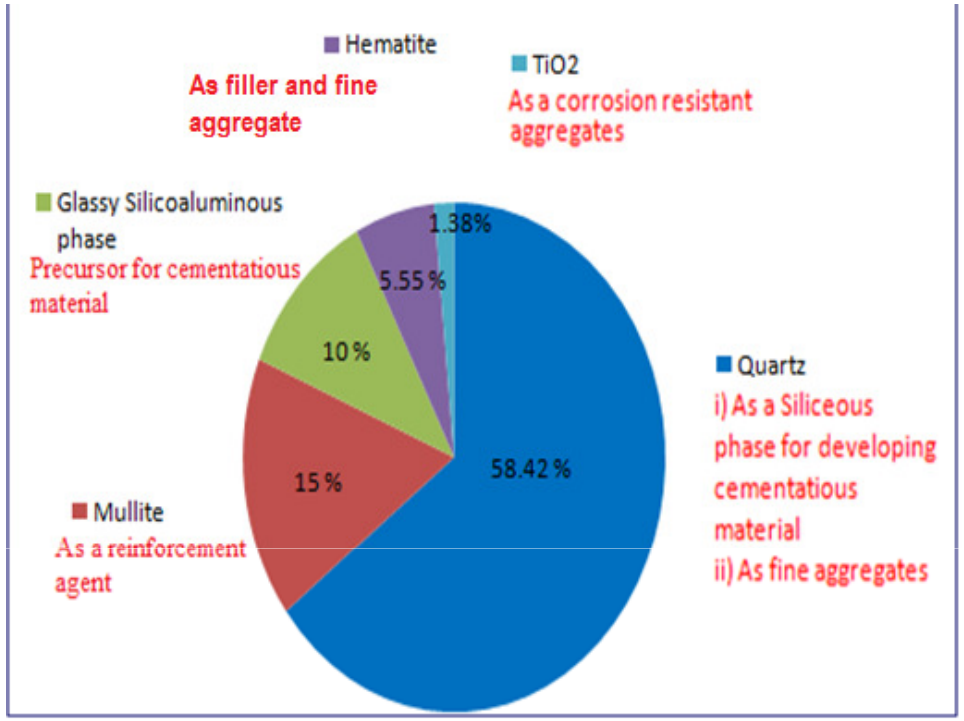
4-Development of Advanced Cement Free Green Concrete

Mechanism of the Process

Chemical Analysis of Fly Ash



Precursors From Fly Ash



Binder

Alkaline Activators: NaOH

Advanced Green Concrete



Mixing of raw materials



Pouring of slurry of raw materials in of sample cubes



Raw material filled in sample cubes



Prepared sample cubes



Reinforced Green Concrete Beam of Dimensions 6''X6''X12'



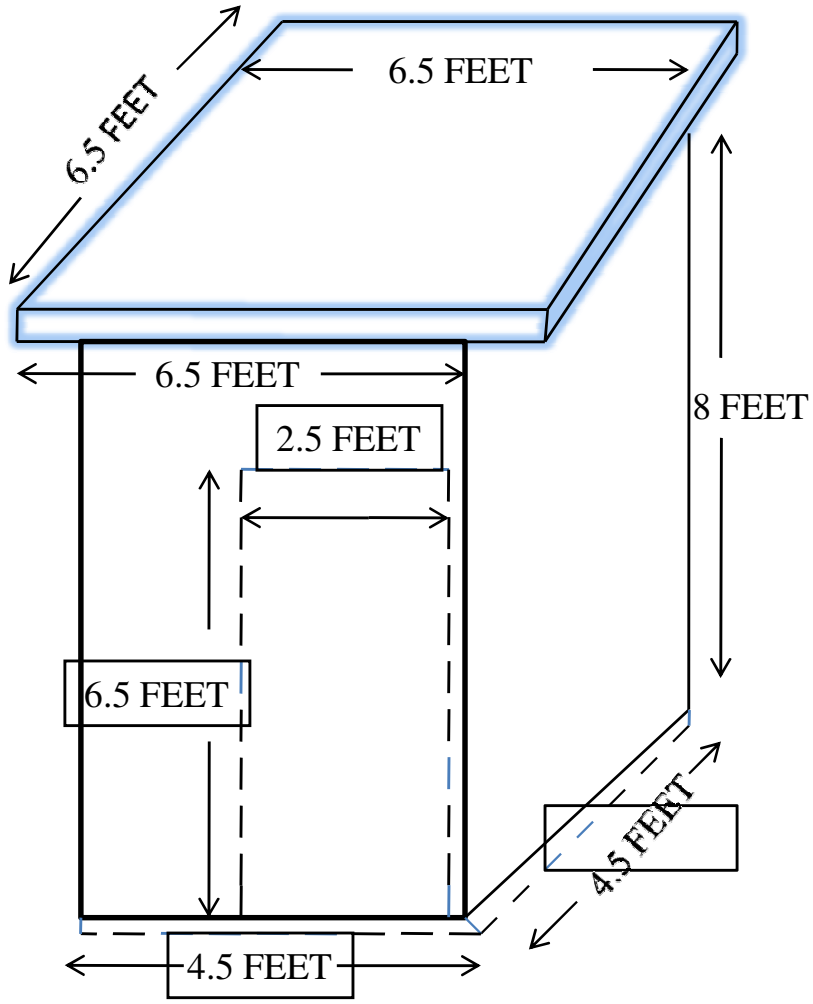
Green Concrete Base Slab Dimensions 3.0'X3.0' Developed at CSIR-AMPRI



Green Concrete Cubes



Green Concrete Cubes Compressive Strength Testing



3 Dimensional View of Structure being Constructed at CSIR-AMPRI

Demonstration of Cast in-Situ Cement Free Reinforced Green Concrete Structure at CSIR-AMPRI Premises

Various Stages of Construction Activities











Slab Construction

Side Views of Structure

Wonder Material for the Next decade
Cement Free Concrete





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