Unfolding the Potential of Mechanical Activation for Fly ash Geopolymer

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CSIR-National Metallurgical Laboratory

- Founded in 1950 by Pandit Jawahar Lal Nehru, First PM of India
- Research mandate is Minerals, Metals, Materials and Waste

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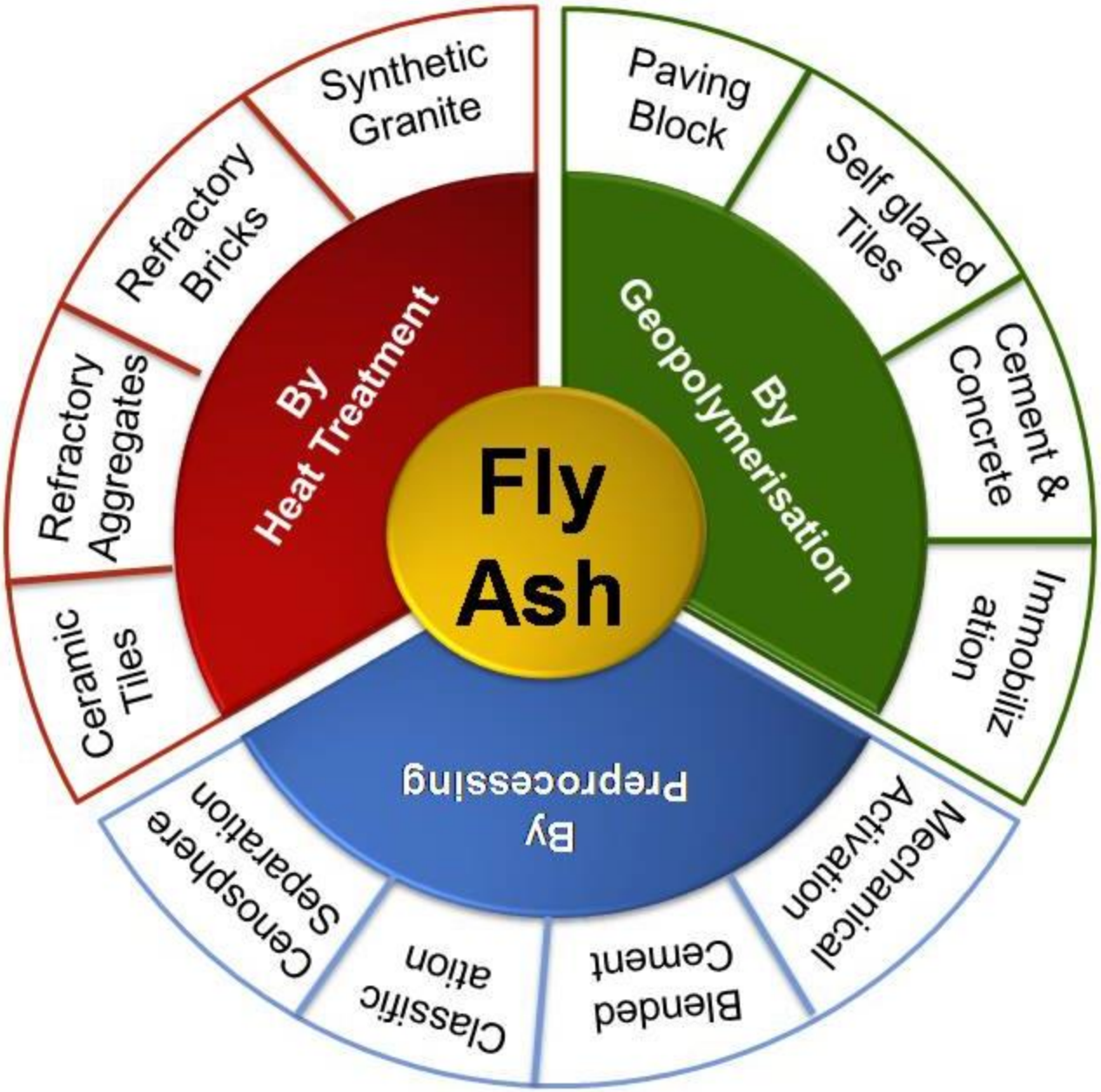
Fly Ash

Annual generation of fly ash exceeded 10 billion tonnes

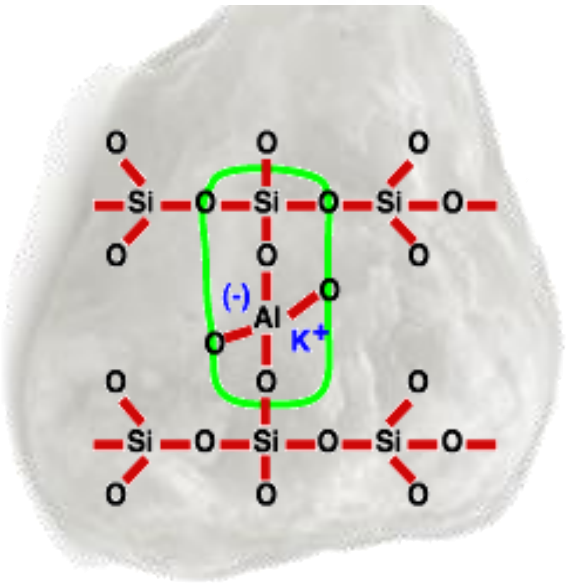
Inventory of fly ash and pond ash is even larger

More than 50% fly ash is unsuitable for product development due to its poor reactivity


Fly Ash
By Heat Treatment
Synthetic Granite
Refractory Bricks
Refractory Aggregates
Ceramic Tiles
By Geopolymerisation
Paving Block
Self glazed Tiles
Cement & Concrete
Immobiliz ation
By Preprocessing
Mechanical Activation
Blended Cement
Classific ation
Cenosphere Separation

Geopolymerisation

Science of making artificial stone at ambient or near ambient temperature



Geopolymers are inorganic binder which result from geosynthesis, i.e. reaction between alumino-silicates and oxides in alkaline media yielding 3-dimensional polymeric framework. It is represented by following equation:

$$M_n [– (Si – O_2)_z – Al – O]_n . wH_2O$$

M	: Alkaline element,
'–'	: Presence of a bond,
z	: 1, 2 or 3, and
n	: degree of polymerisation

Why Fly Ash for Geopolymer

Desirable properties

- Alumino-silicate composition, Al:Si ratio in desirable range for geopolymerisation
- Consists of glassy and crystalline phase, glassy phase reacts with alkali solution
- Good flow properties due to spherical shape
- Easy & cheap availability

Reactivity of Fly Ash in Geopolymer

- Guided by the amount of reactive glassy phase, which undergoes dissolution in alkali solution
- Influenced by the particle size, finer particles have better reactivity
- The crystalline phases such as quartz and mullite remains unreacted

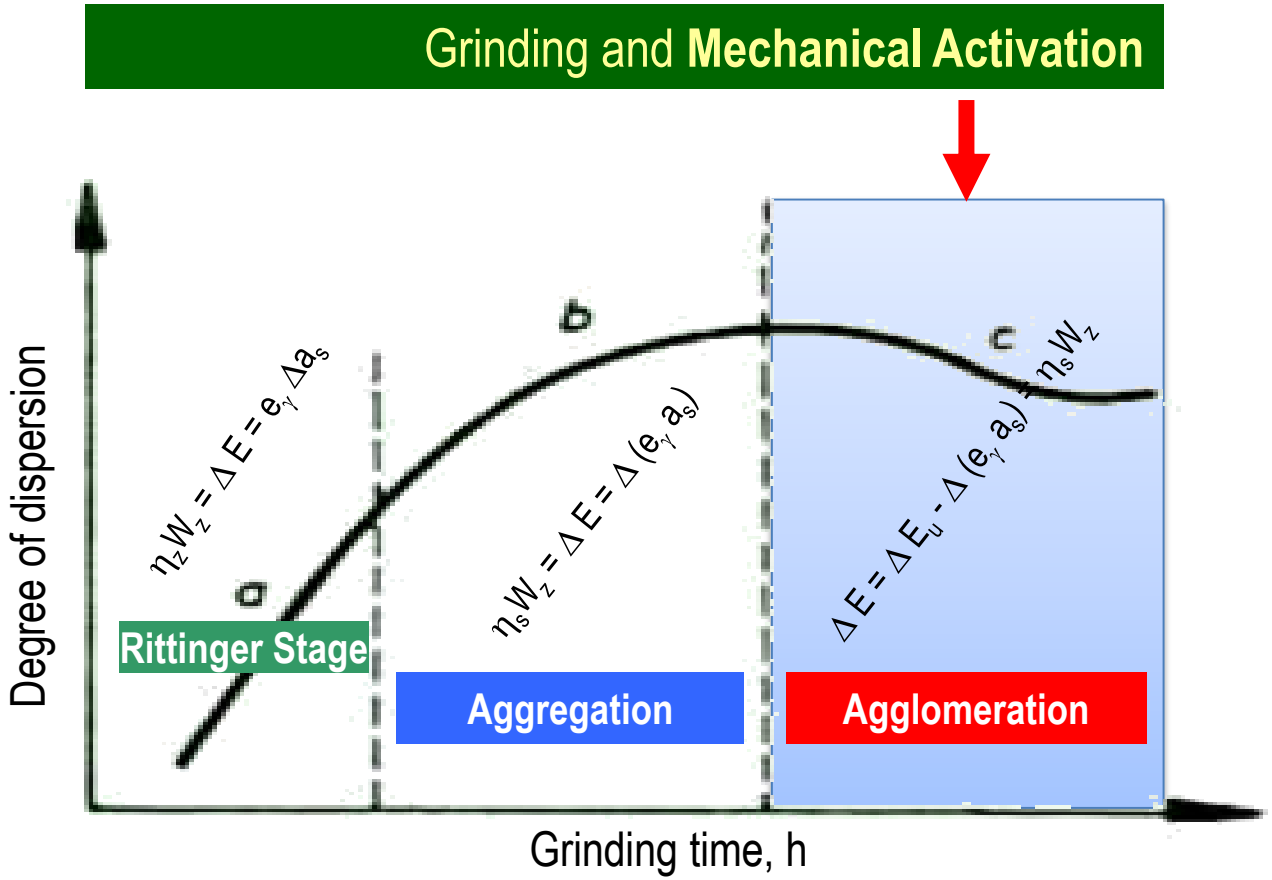
Mechanical activation is promising solution to alter fly ash reactivity

Mechanical Activation

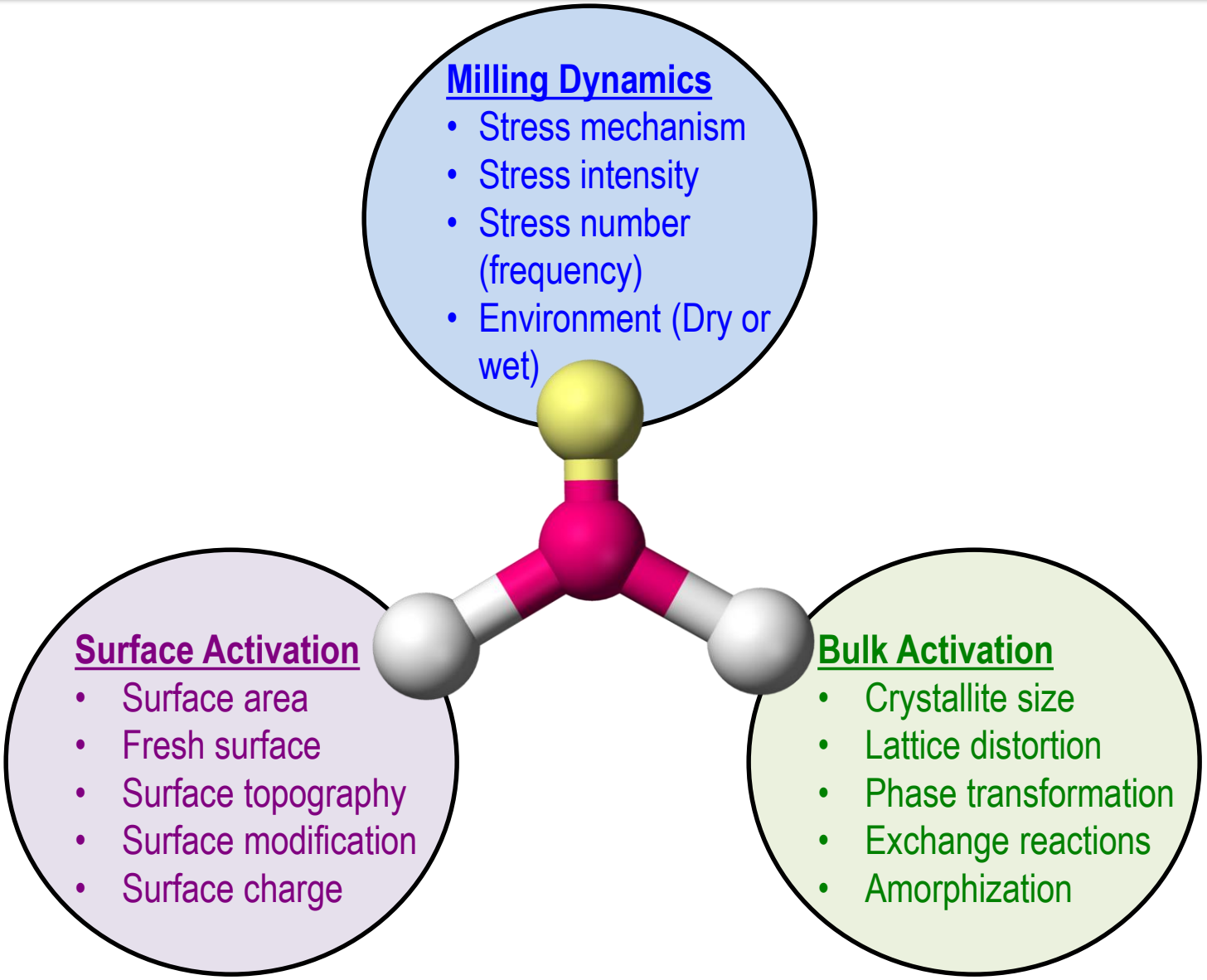
Dinosaurs eats stone to grind tough food for easy digestion

Gastroliths

Mechanical Activation



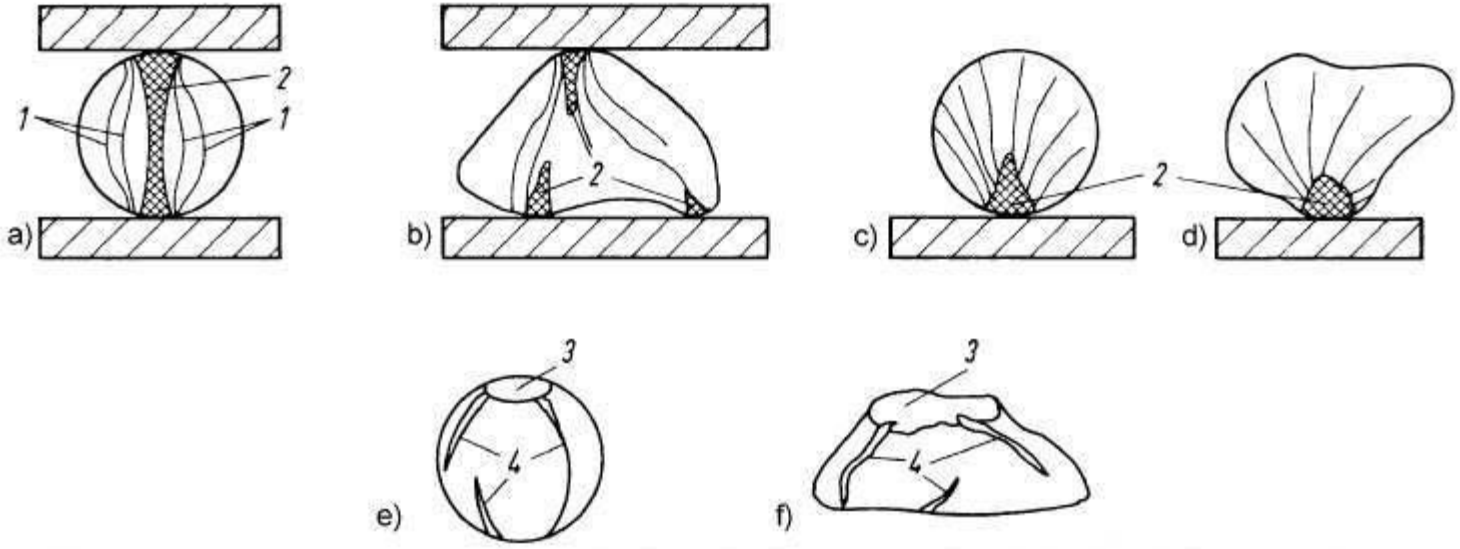
Mechanical Activation



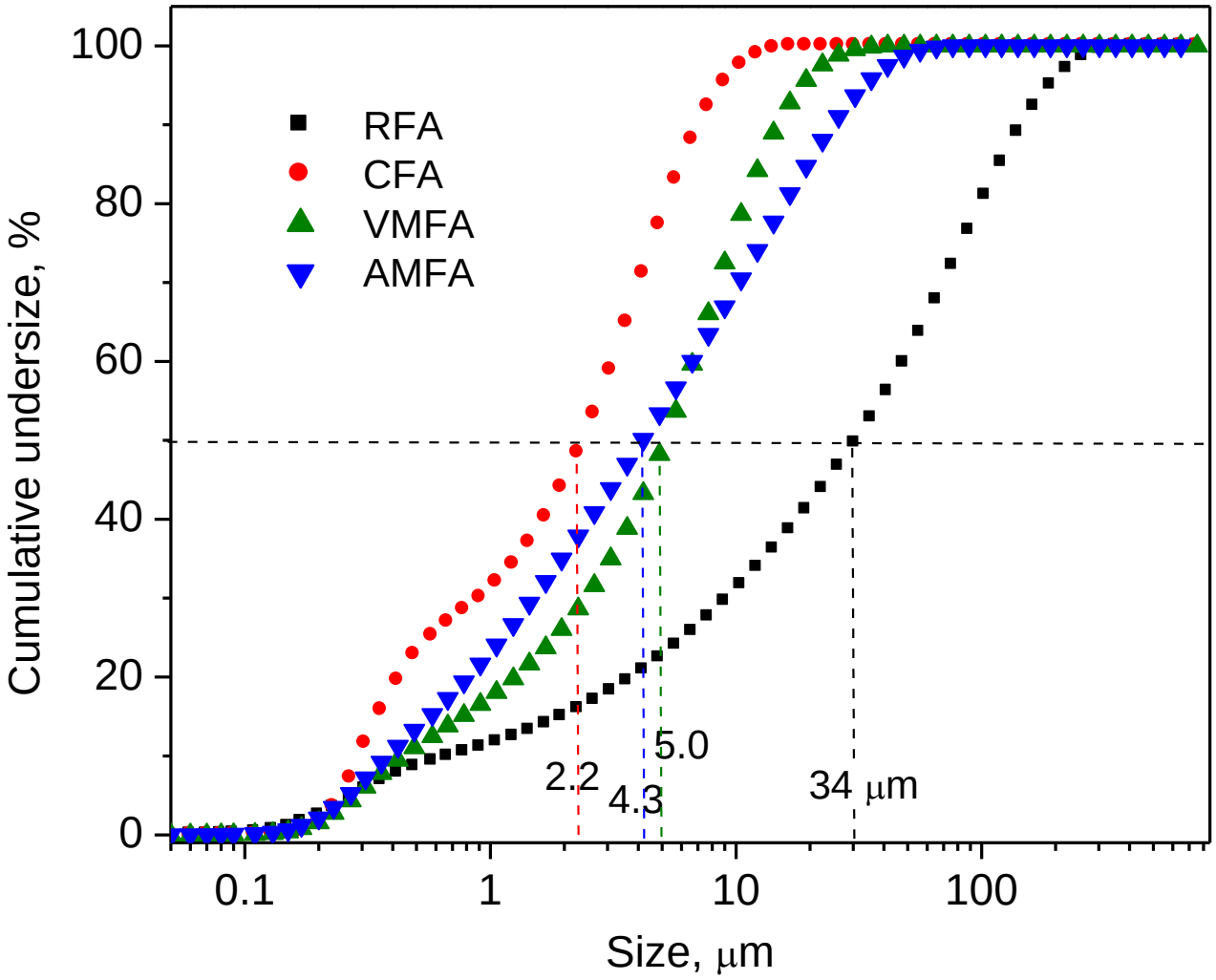
Mechanical Activation of Fly Ash

What changes occur

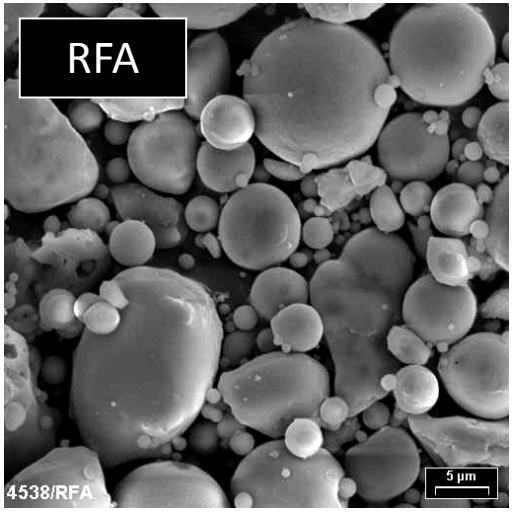
- Increase in surface area
- Stresses and defects in solid structure
- Fracture leading to dynamic creation of fresh surfaces for reactions etc.
- Energy storage in defect which releases during alkali activation leading to faster reaction

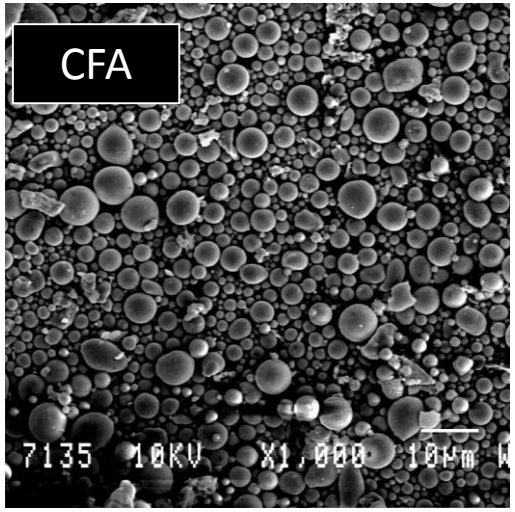


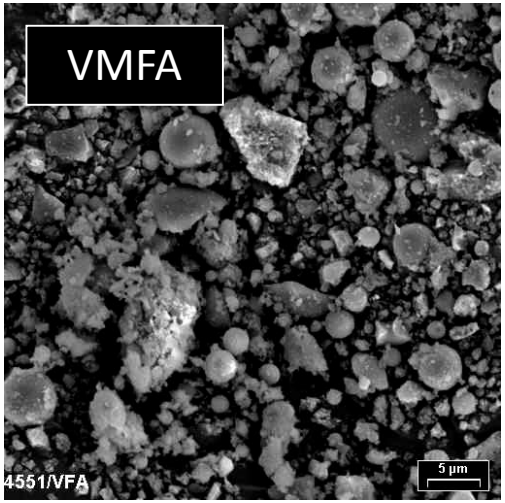
Particle Size Distribution

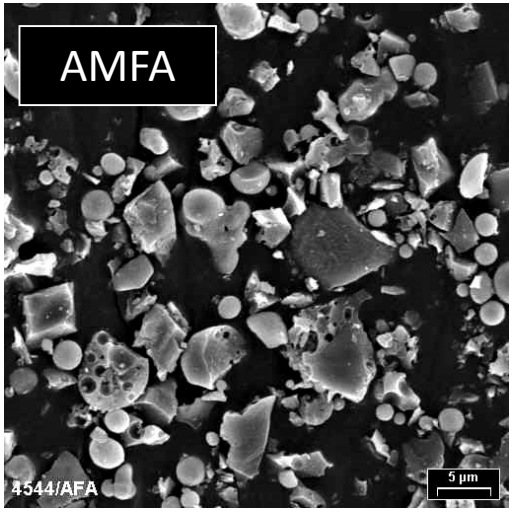


Classified & Mechanically Activated Fly Ash

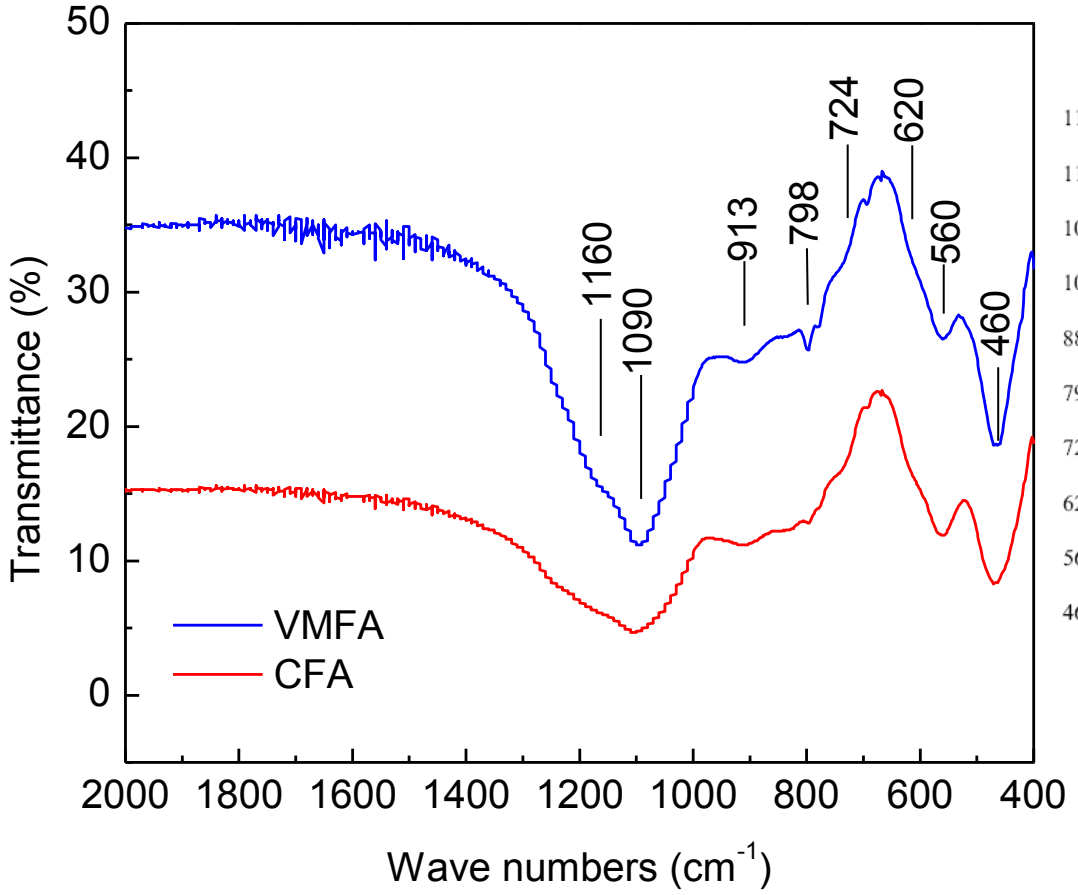






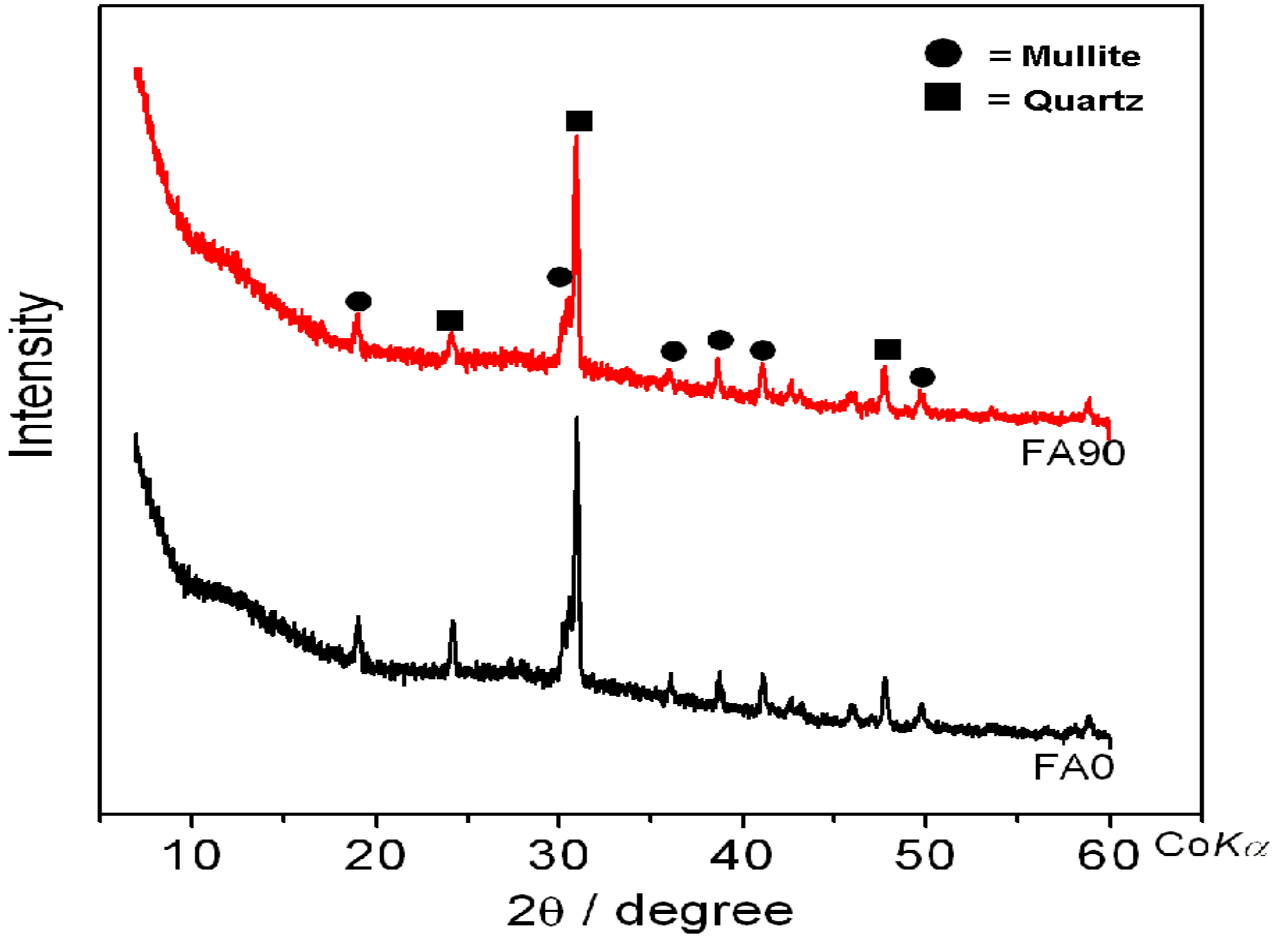


MA Vs Classified Fly Ash



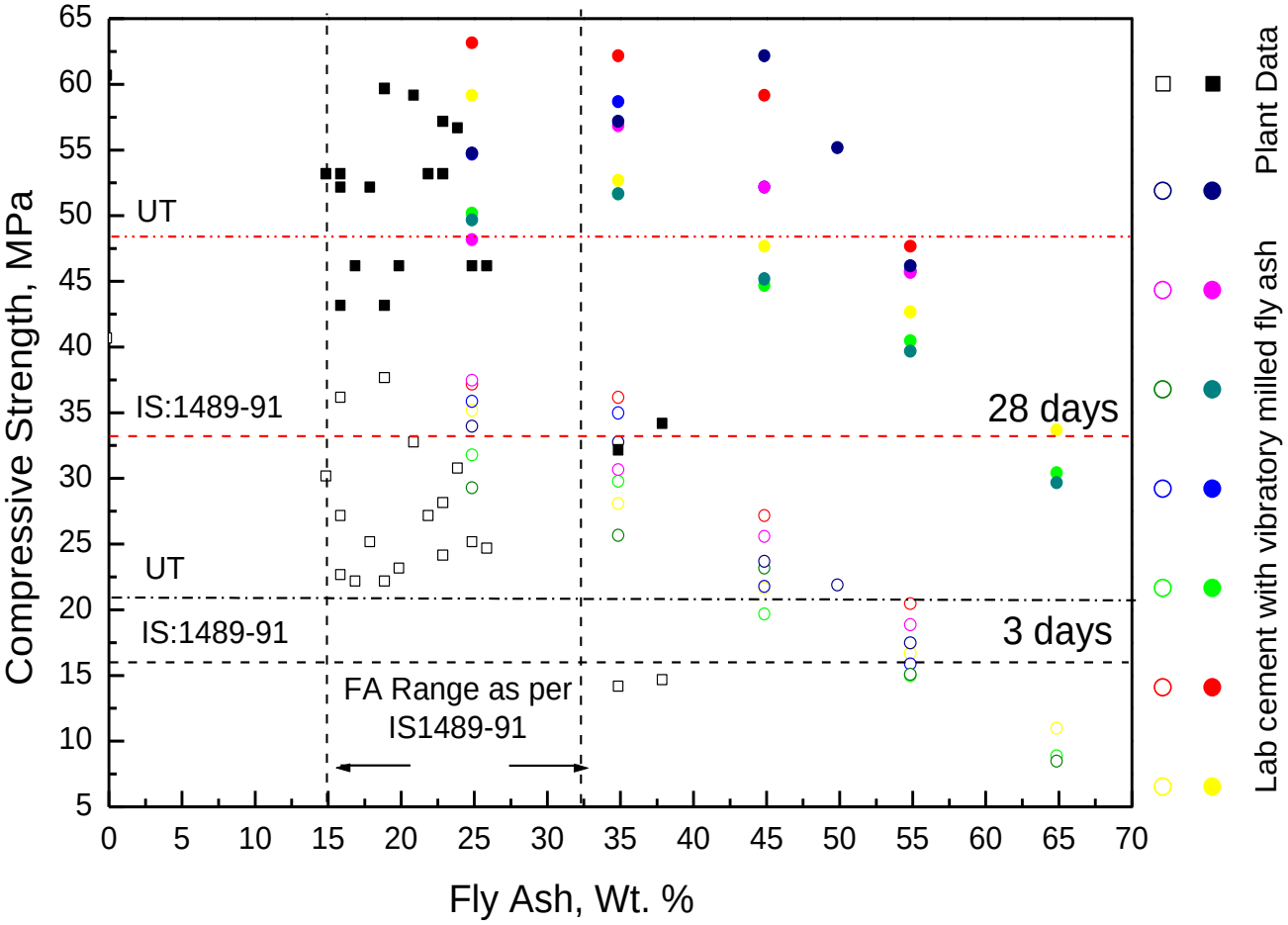
1165 (sh)	Asymmetric stretching (Si–O–Si)
1138 (sh)	Asymmetric stretching (Si–O–Si and Al–O–Si)
1080 (sh)	Asymmetric stretching (Si–O–Si and Al–O–Si)
1074 (s)	Asymmetric stretching (Si–O–Si and Al–O–Si)
882 (s)	Si–O stretching, OH bending (Si–OH)
798 (m)	Symmetric stretching (Si–O–Si)
727 (sh)	Symmetric stretching (Si–O–Si and Al–O–Si)
620 (sh)	Symmetric stretching (Si–O–Si and Al–O–Si)
561 (s)	Symmetric stretching (Al–O–Si)
466 (s)	Bending (Si–O–Si and O–Si–O)

XRD Results


= Mullite
= Quartz
Intensity
FA90
FA0
10
20
30
40
50
60
CoKα
2θ / degree

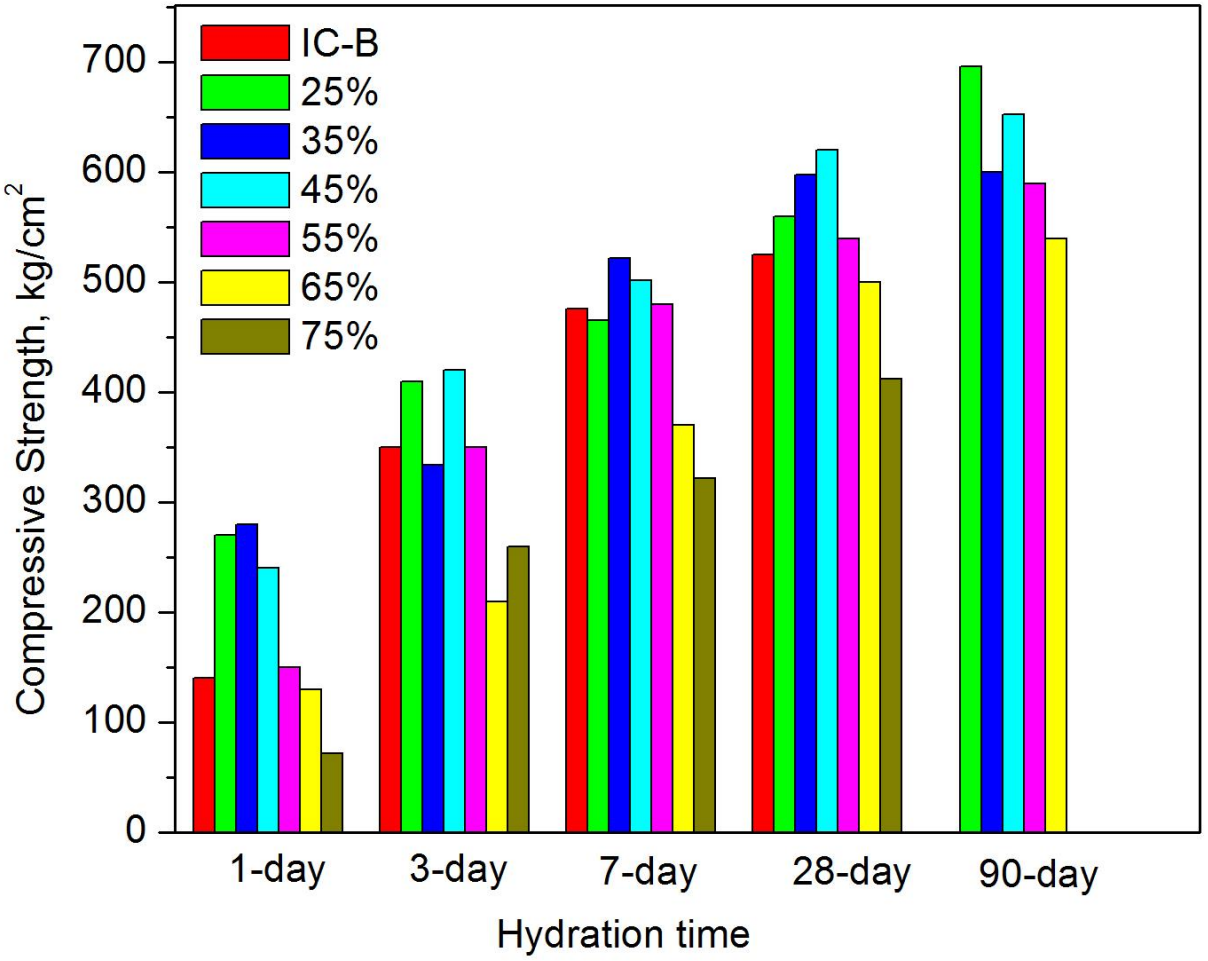
MA Fly Ash in PPC

Compressive Strength
Benchmarking, BIS Specification and Target



Patent Application No. 0644DEL2006, 10/3/2006

MA Fly Ash in PPC

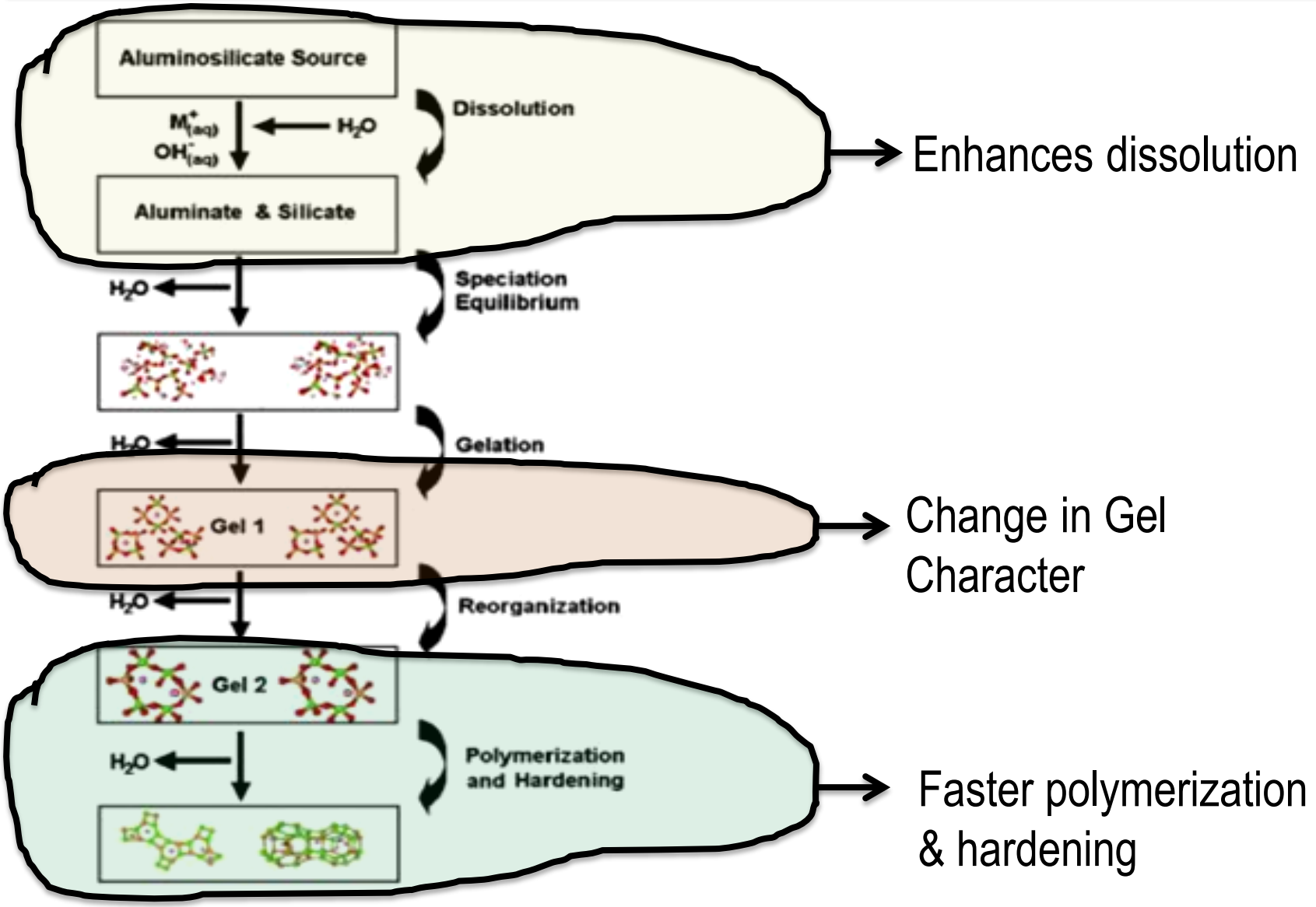


Implication of Using MA Fly Ash in PPC

Unit operation/process	Energy	PPC 25% FA	PPC 50% FA
Raw material (RM) grinding (kWh/t)	15	11	7.5
RM blending & homogenisation (kWh/t)	10	8	5
Coal grinding (kWh/t)	3	2.5	1.5
Clinker section Thermal (MJ/t) Electrical (kWh/t)	3200 40	2400 30	1600 20
Cement grinding OPC (kWh/t Cement) PPC (kWh/t Cement)	33 -	- 35	- -
PPC (kWh/t Cement) (with MA of FA)	-	-	45-52
Overall Energy Saving(25% FA-50% FA) : Thermal 800 MJ/t, Electrical : 5-12 kWh/t			

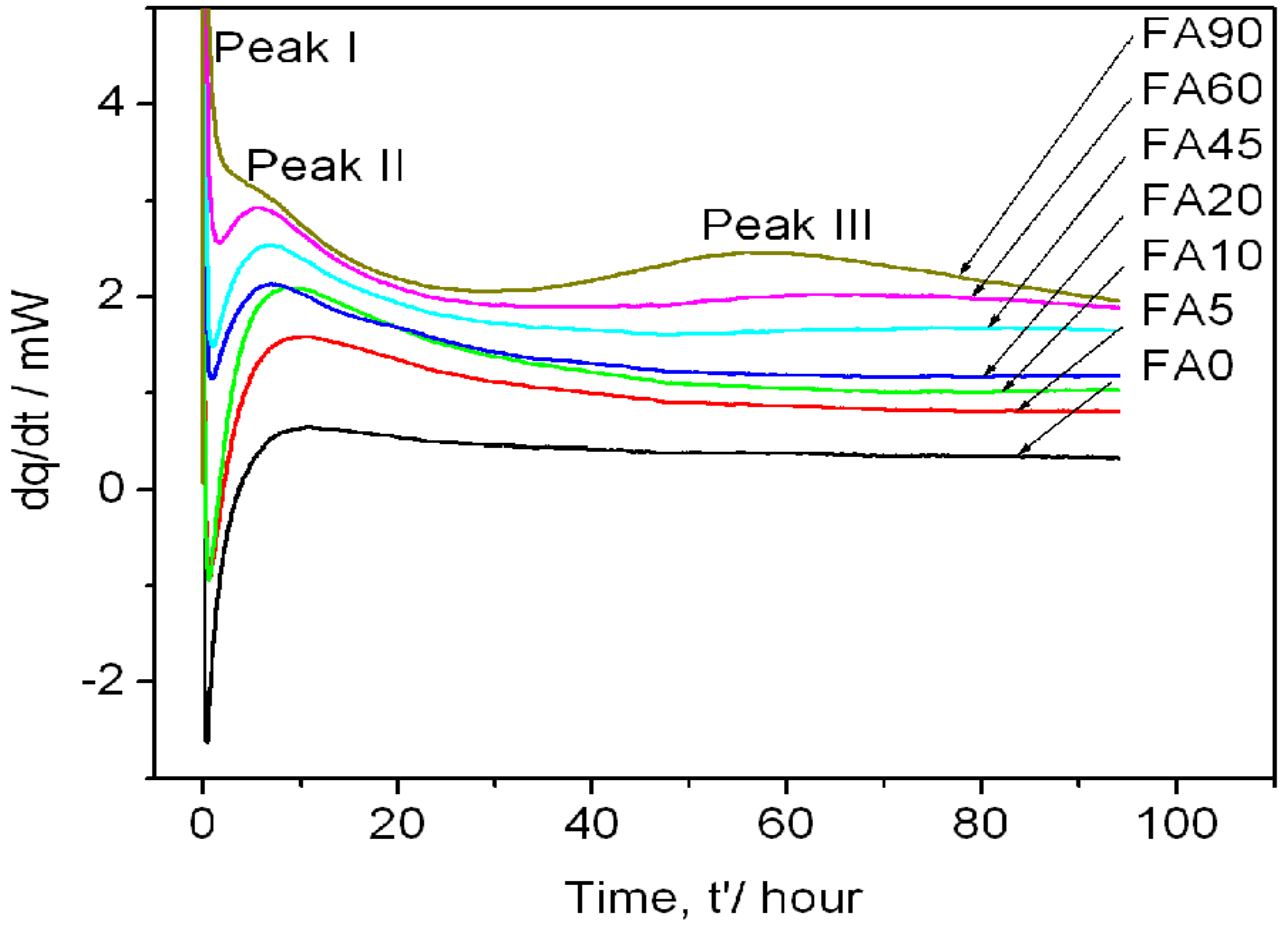
Resource	Conservation (Fly Ash 25% → 50%)
Lime stone (t/t cement)	0.25
Clay Sand etc (t/t Cement)	0.125
Coal (t /t Cement)	0.03
Refractory (g/t Cement)	90-100
Air (Nm^3/tonne Cement)	750

Mechanical Activation & Geopolymerisation

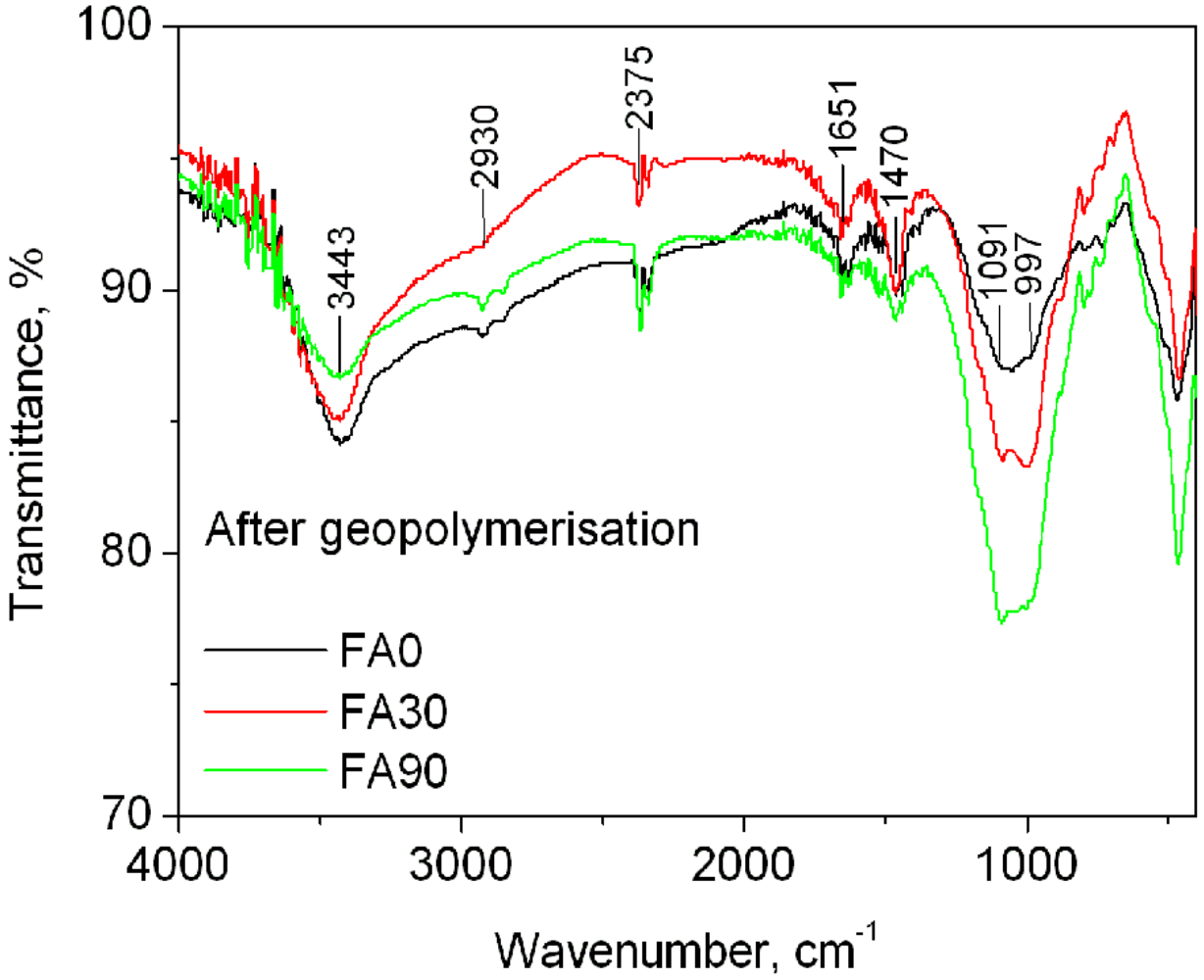


Duxton et al., J Mater Sci (2007) 42:2917–2933

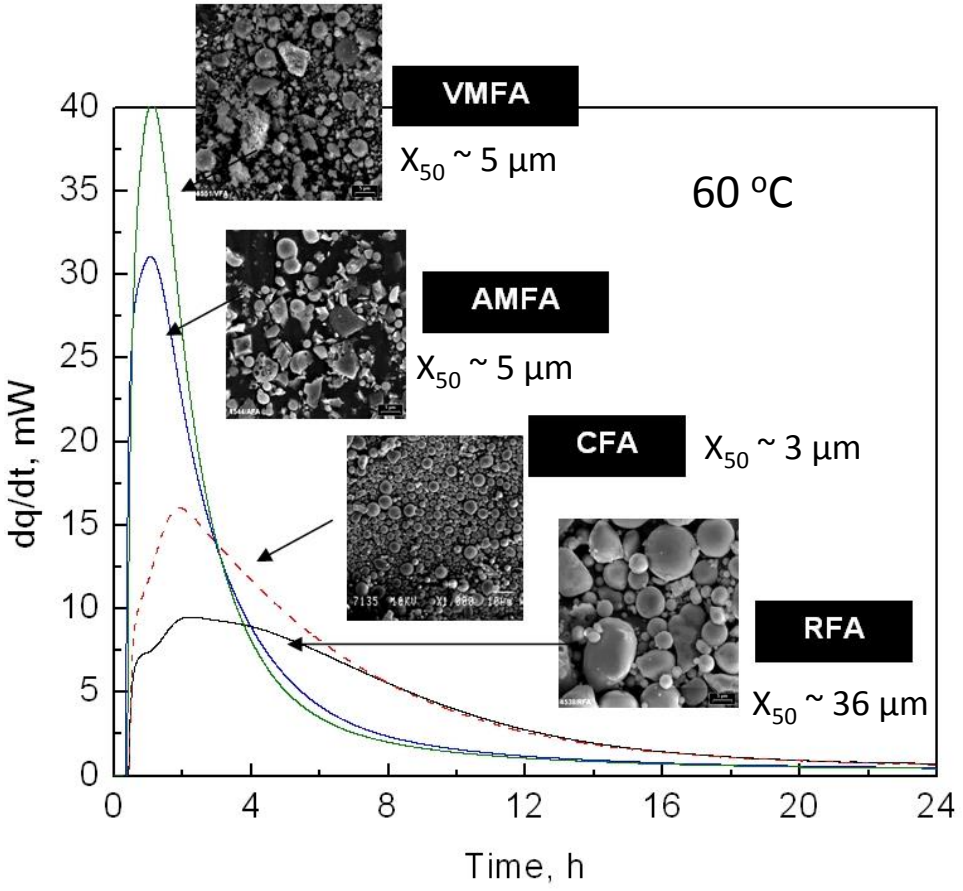
Calorimetry of Geopolymer



FTIR of Geopolymer



Reactivity is the Key

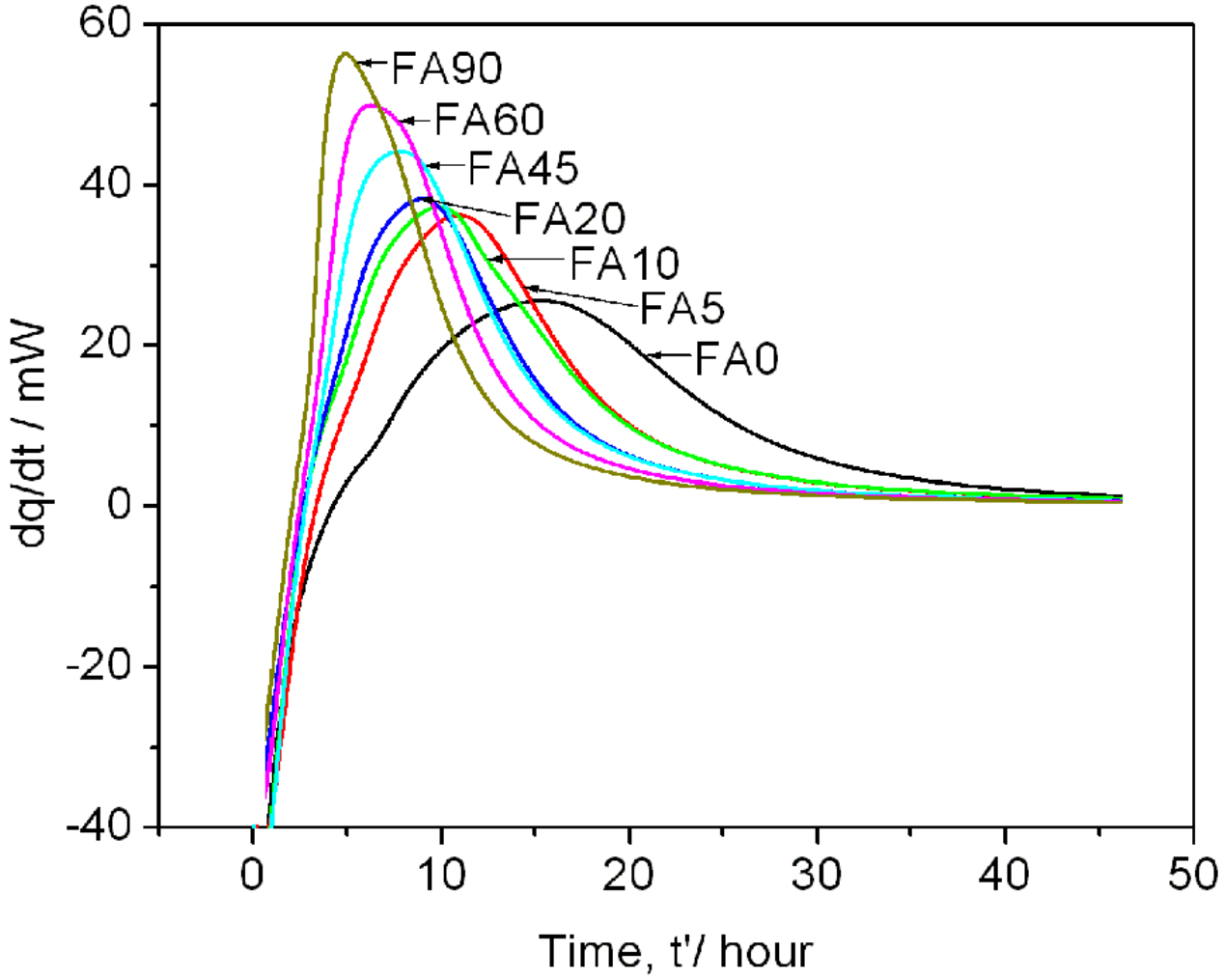


Geopolymerisation of fly ash

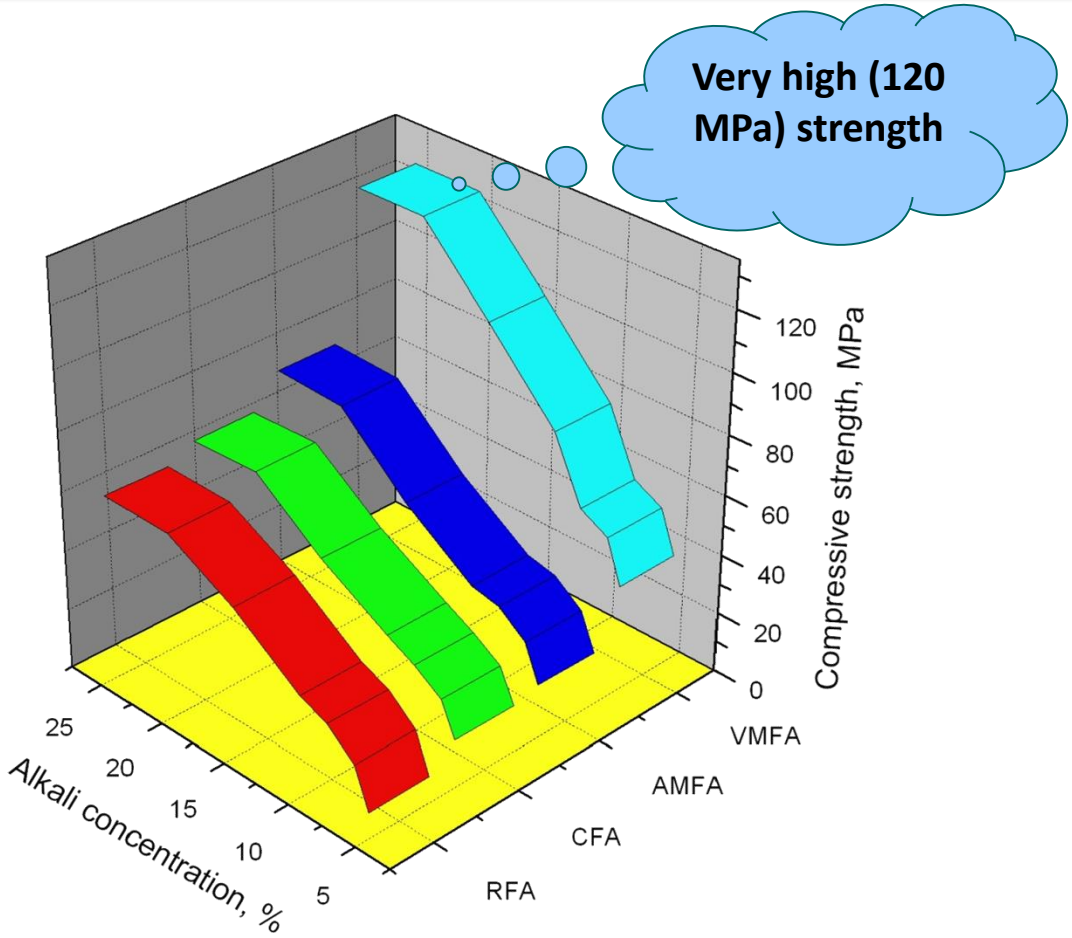
Reactivity of Fly Ash

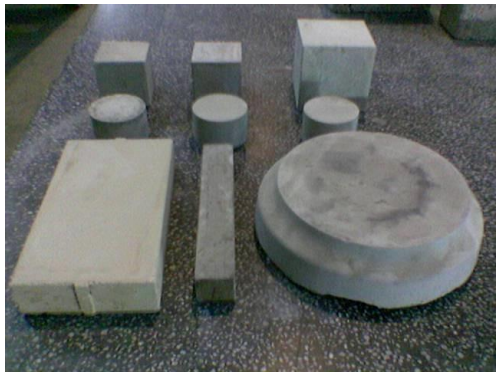
Mechanical activation more effective than increasing glass content

Activation Mechanism



Geopolymer Cement

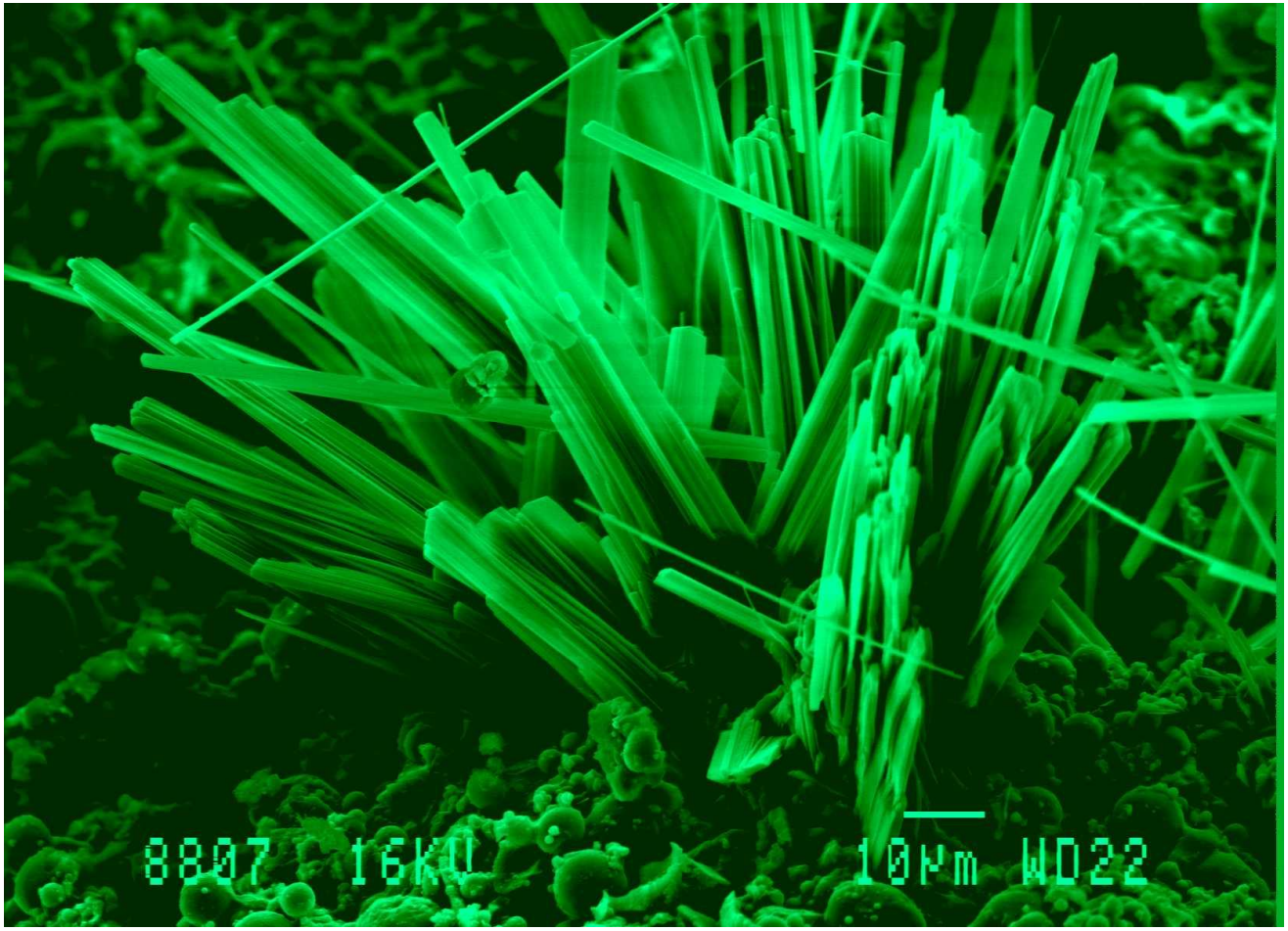




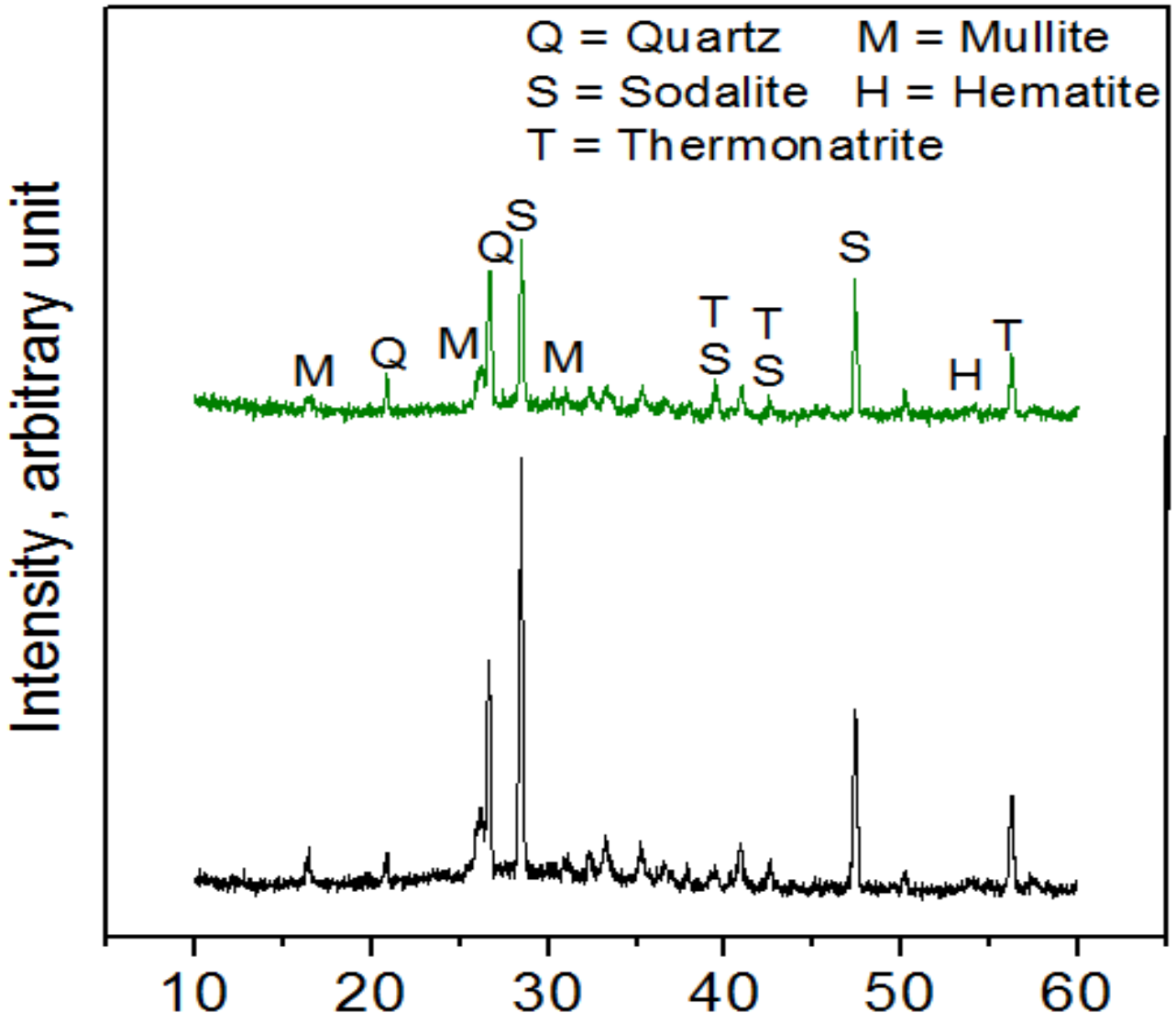
Properties can be tailored using mechanical activation

Patent : 2626/DEL/2005, 30/09/2005 and 728/DEL/2006, 30/11/2005

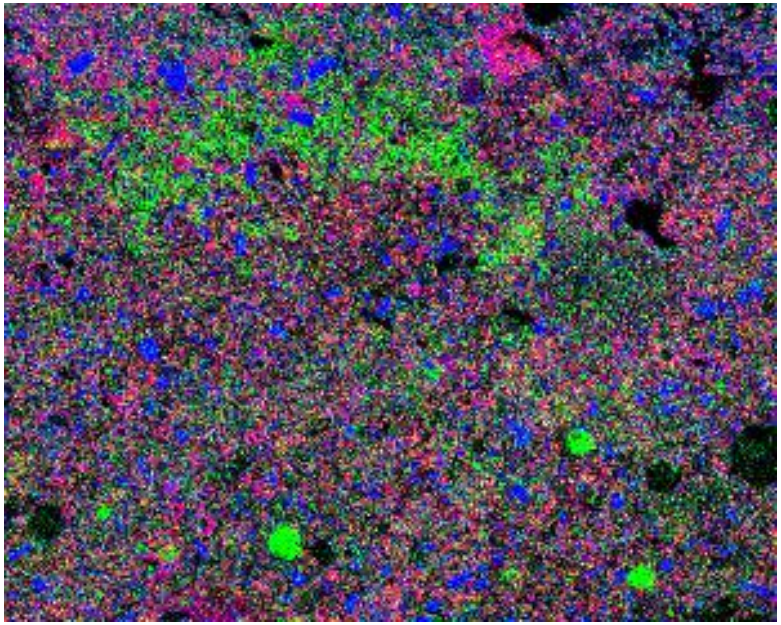
Growth of Zeolite from Geopolymer Gel



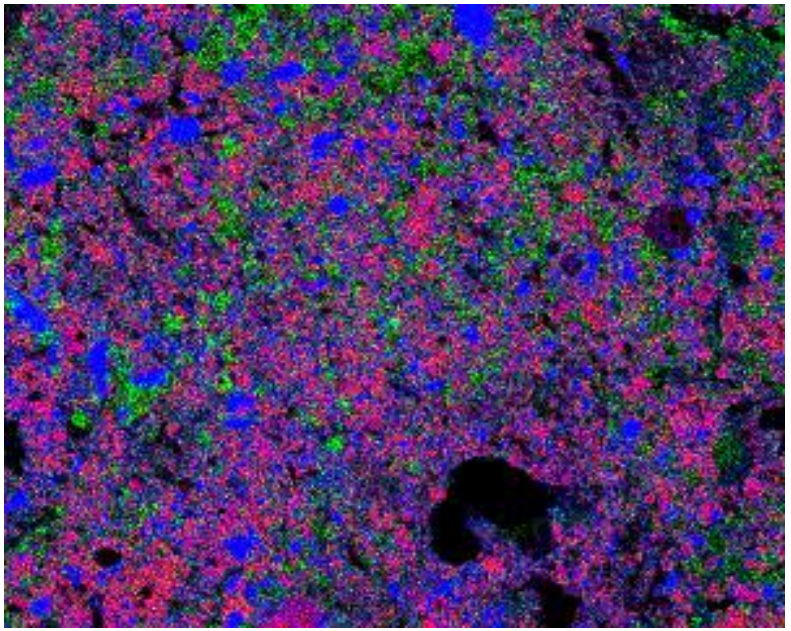
Reduction in Thermonatrite Formation



Uniform distribution of Elements



Geopolymer from Raw Fly Ash

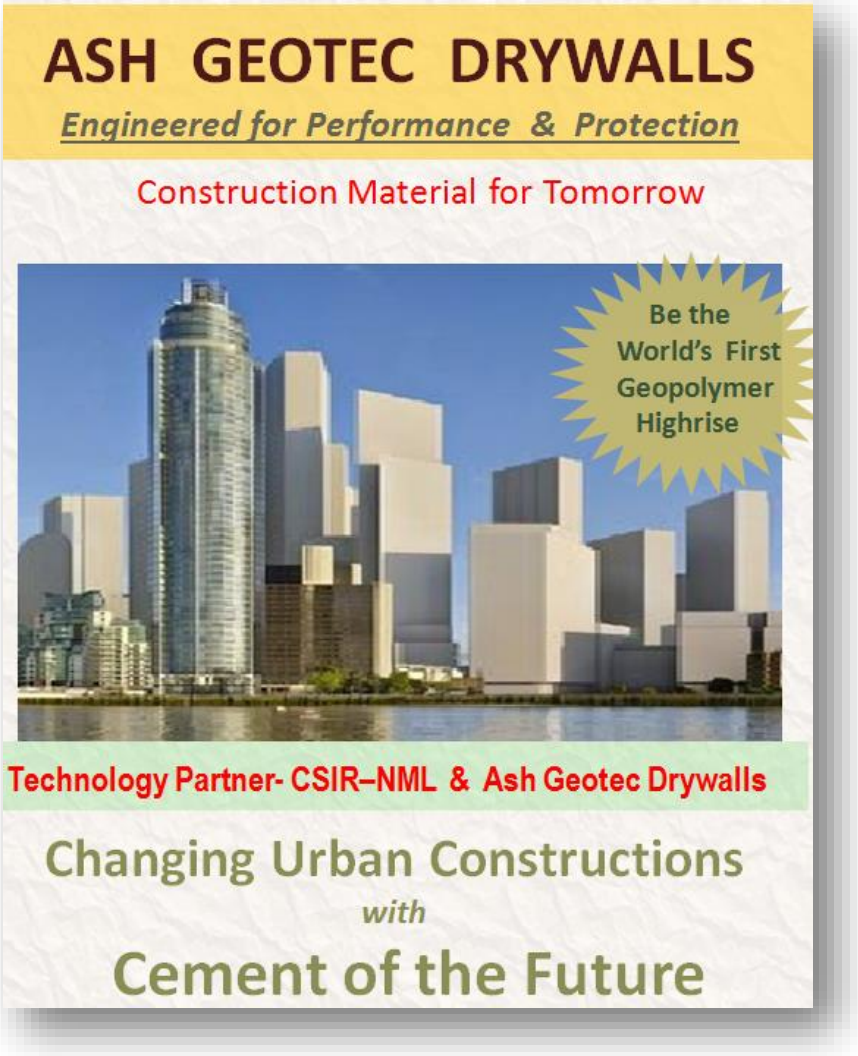


Geopolymer from MA Fly Ash

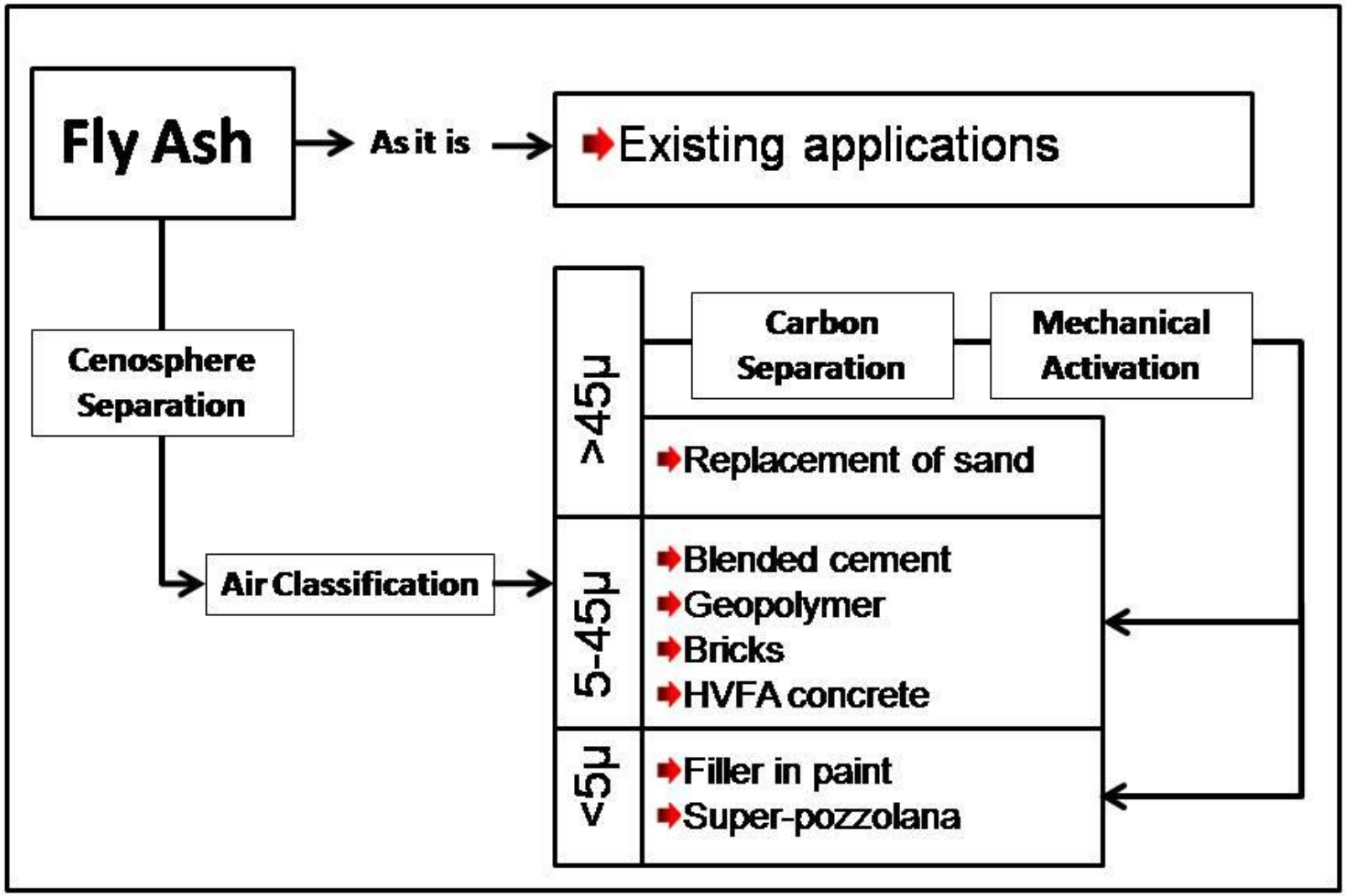
Na-green, Al-red, Si-blue

Towards Products Development

- Development of fast setting geopolymer grout using mechanically activated fly ash
- Flexible lightweight geopolymer for prefabricated wall application
- High strength geopolymer cement



Integrated Scheme



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