ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

2014 International Pyroprocessing Research Conference

PRESENT STATUS OF PYROPROCESS TECHNOLOGIES AT ENEA LABORATORIES

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ENEA – C.R. Casaccia, Roma, Italy

5th International Pyroprocessing Research Conference

Idaho Falls, Idaho, October 19-23/2014

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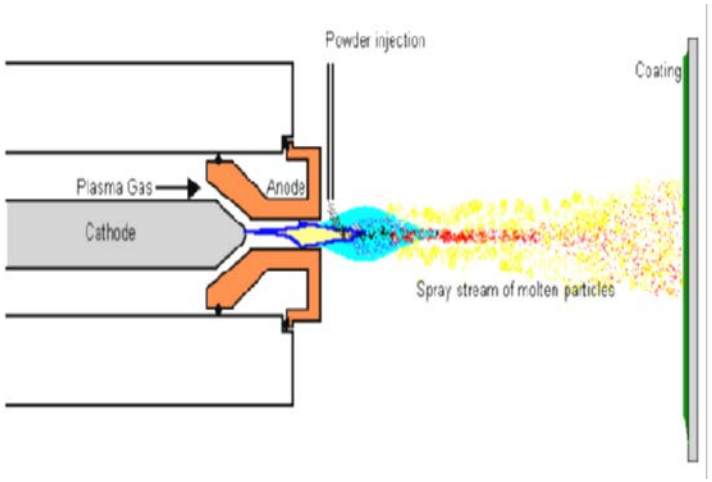
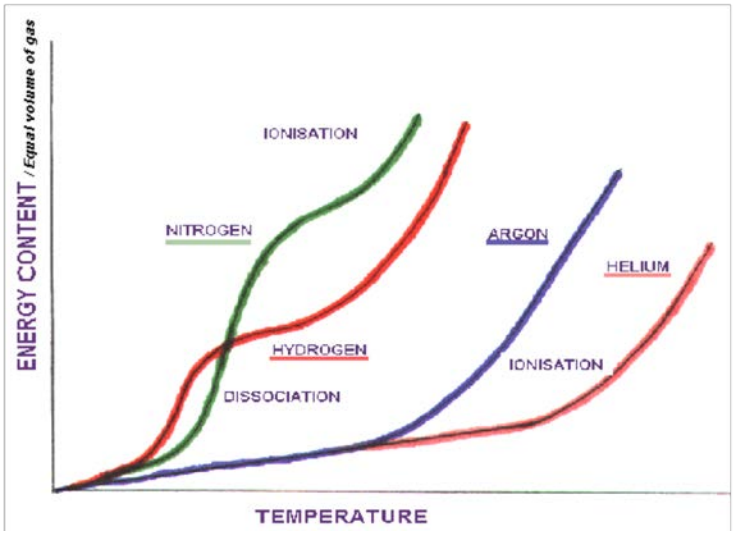
Pyrel III Pilot Plant

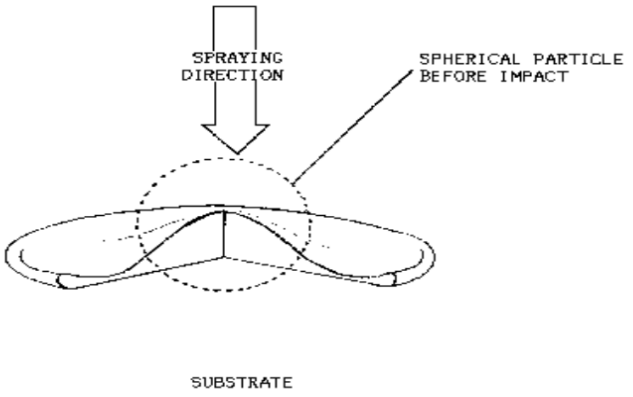
Protection of the steel crucible against corrosion

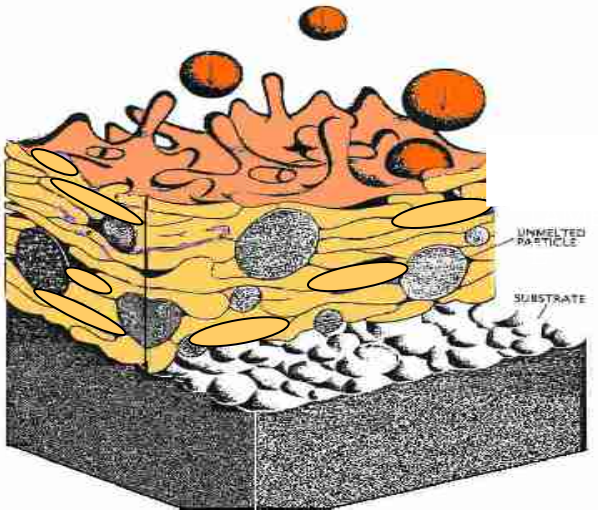
5th International Pyroprocessing Research Conference
Idaho Falls, Idaho, October 19-23/2014

Powder Injection Plasma Spraying Process, PIPSP

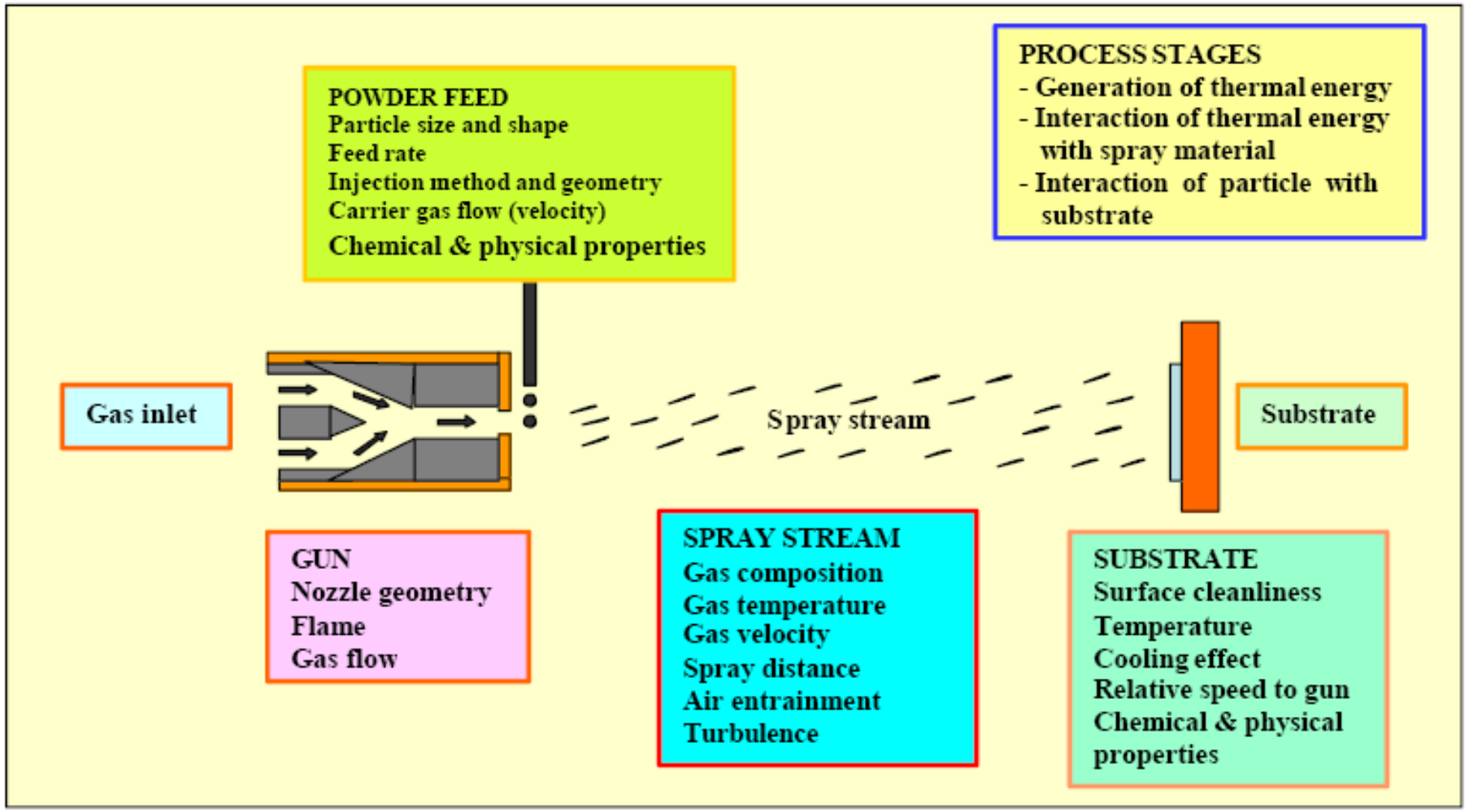




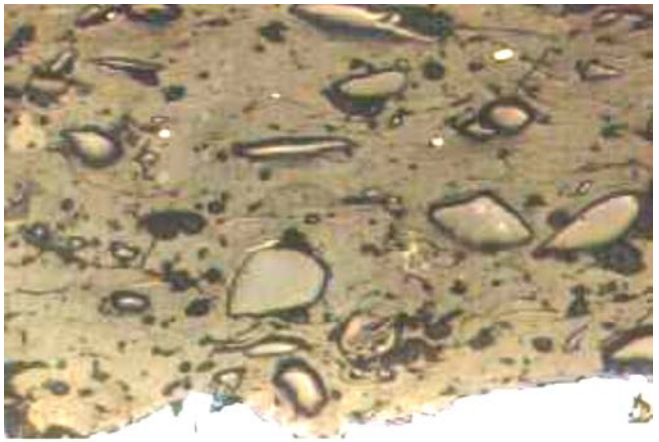


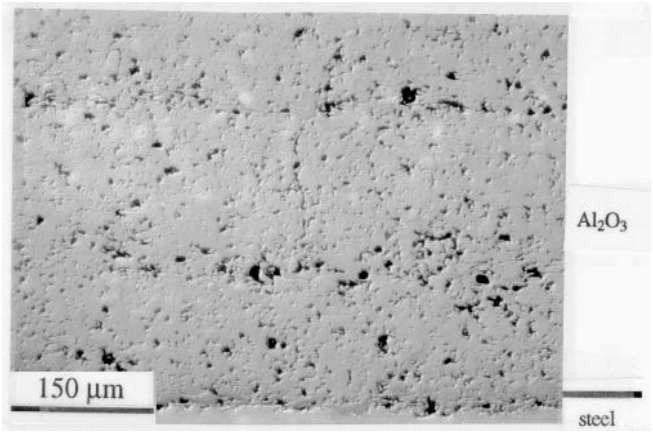


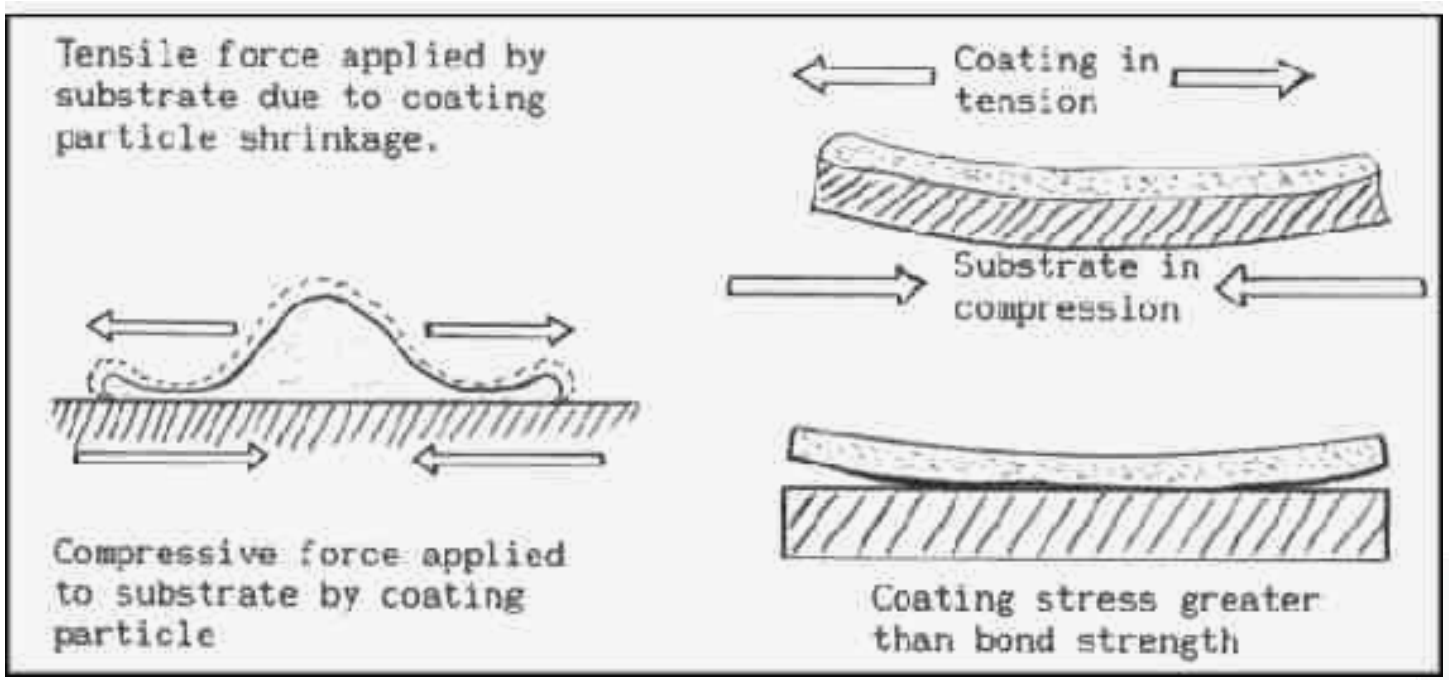
Powder Injection Plasma Spraying Process, PIPSP



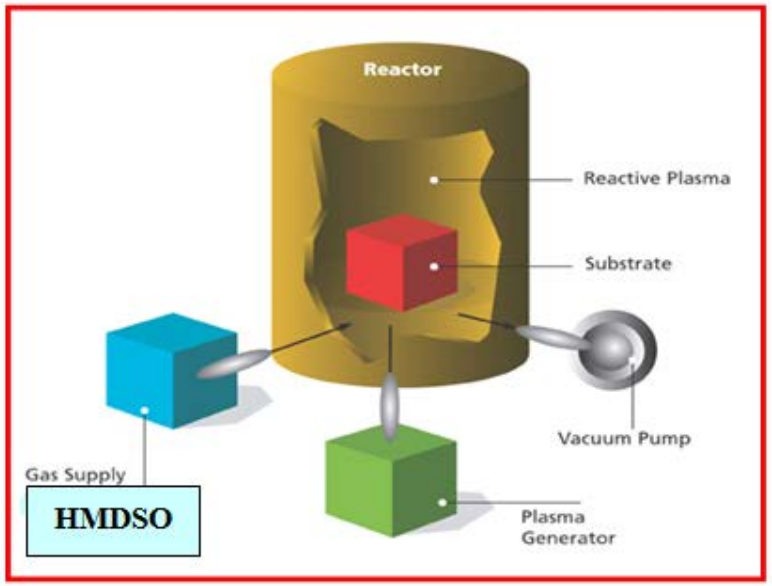
Powder Injection Plasma Spraying Process, PIPSP

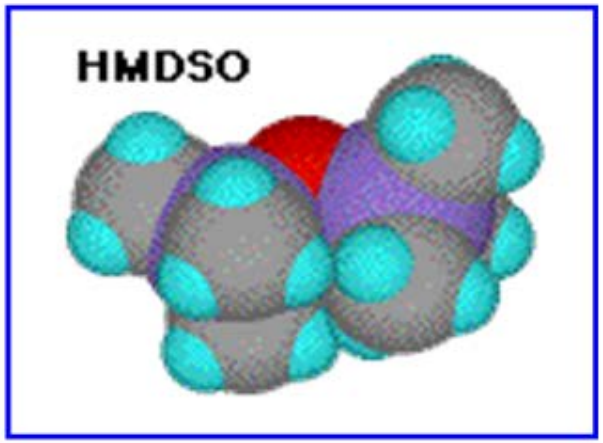




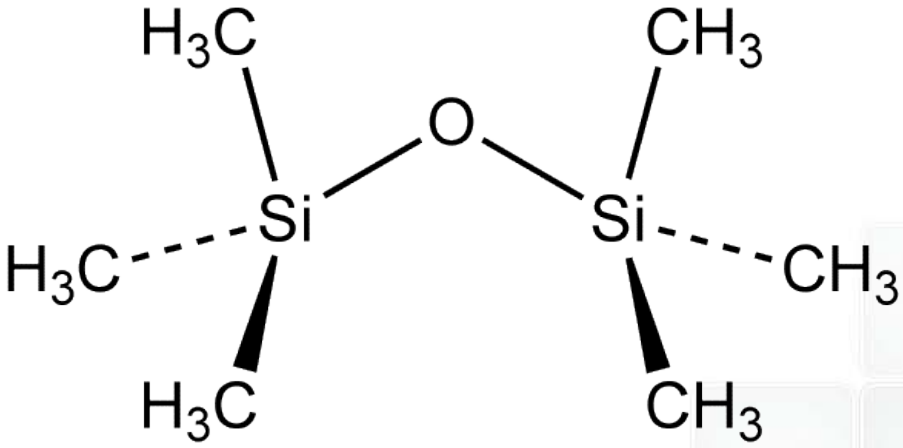


Plasma Enhanced Chemical Vapour Deposition, PECVD





HMDSO: hexamethyldisiloxane



PYREL III PILOT PLANT

Steel crucible before (top) and after (bottom) internal coating

Protection of the steel crucible against corrosion

Crucible with eutectic salt at the end of an experimental campaign

Salt partially removed after washing with hot water

Salt partially removed after washing with hot water

Salt partially removed after washing with hot water

Protection of the steel crucible against corrosion

Crucible removed from the plant

Detail of external corrosion

Crucible after external clean up

Crucible after external clean up

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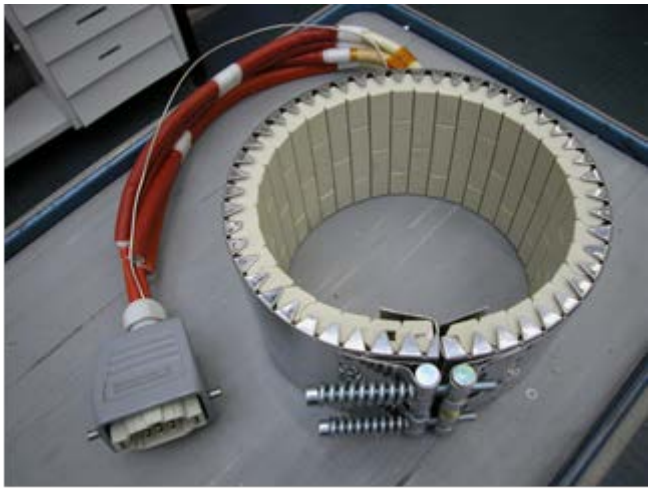
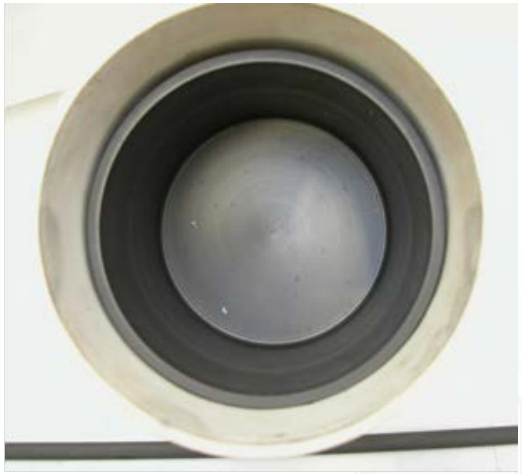
Pyrel III Pilot Plant

Plant components

Components of Pyrel III Pilot Plant



Components of Pyrel III Pilot Plant



Components of Pyrel III Pilot Plant

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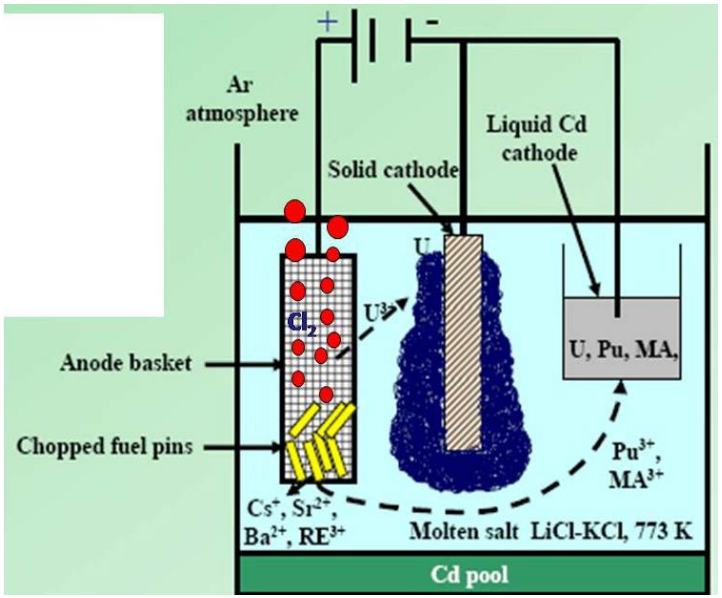
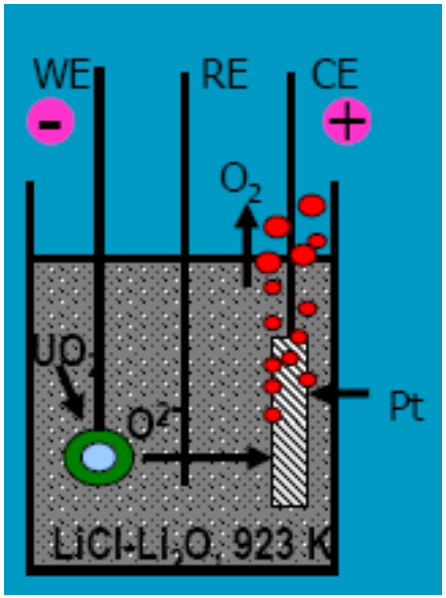
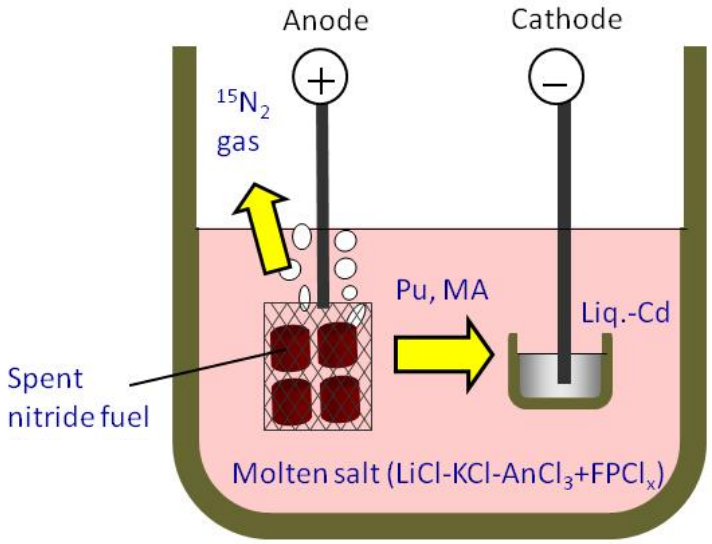
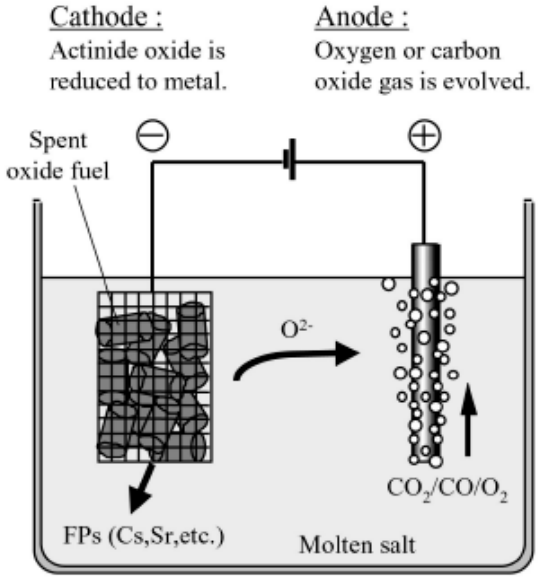




Need for a
Gas trapping system





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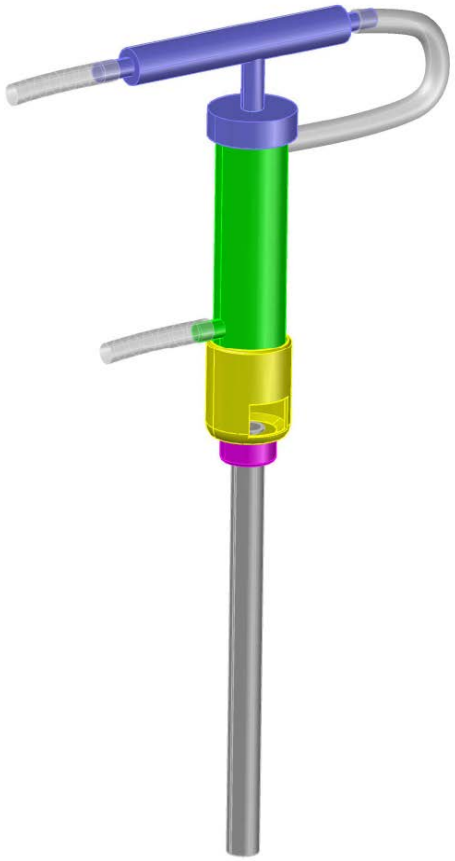
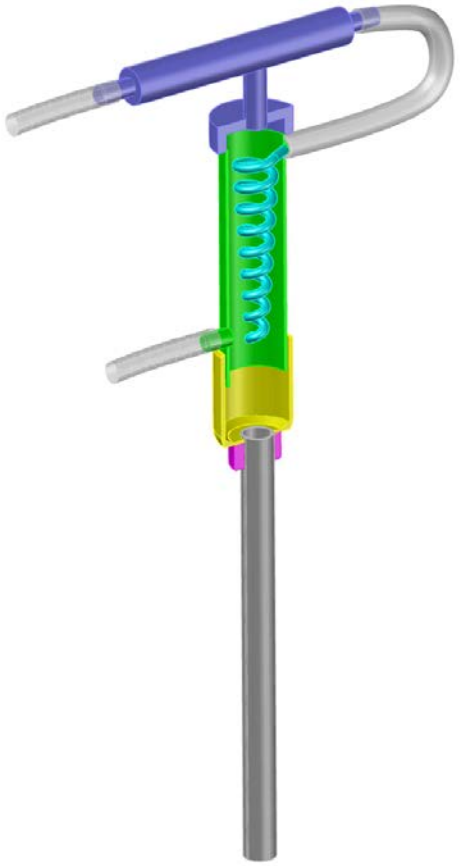
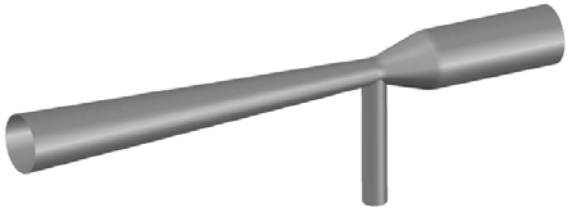
Components of Pyrel III Pilot Plant

Gas trapping system

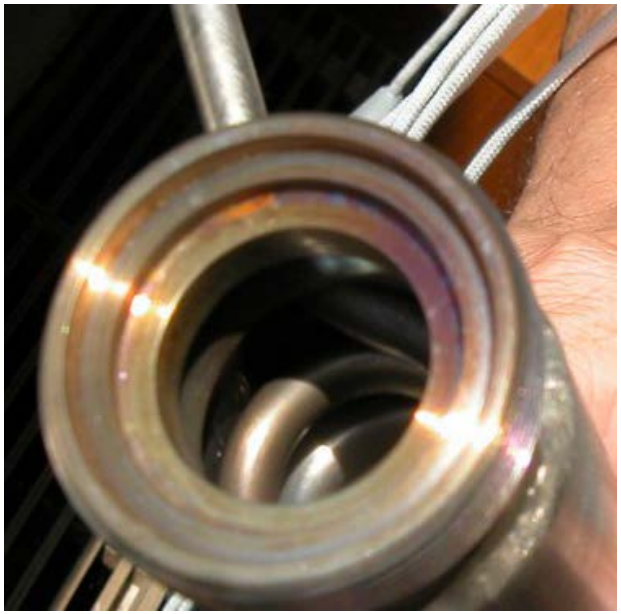
Components of Pyrel III Pilot Plant





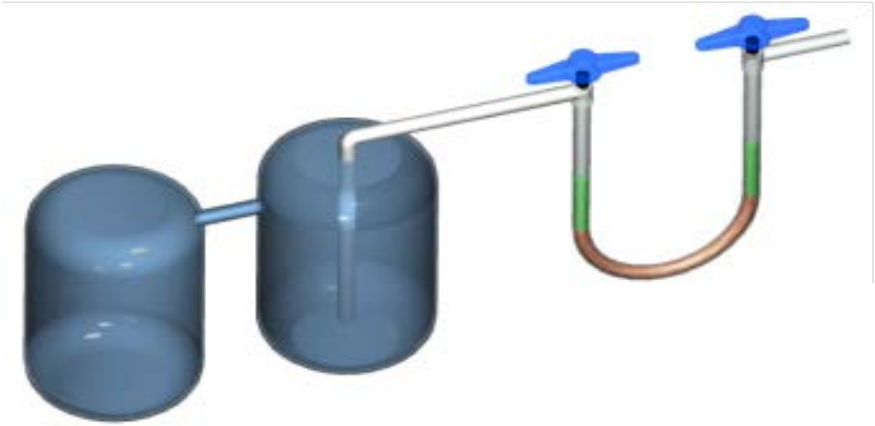


Components of Pyrel III Pilot Plant



Components of Pyrel III Pilot Plant

Outline of the experimental apparatus to detect the presence of chlorine, oxygen or carbon anidride in argon current from the glove-box of Pyrel III plant



$Cl_{2(g)} + 2NaOH \rightarrow NaCl + NaClO + H_2O$

$NaCl + AgNO_3 \rightarrow \underline{AgCl} + NaNO_3$

$CO_{2(g)} + Ca(OH)_2 \rightarrow \underline{CaCO_3} + H_2O$

$6Li_{(s)} + N_{2(g)} \rightarrow 2\underline{Li_3N_{(s)}}$

Outline of the experimental apparatus to detect the presence of nitrogen in argon current from the glove-box of Pyrel III plant

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Treatment of chloride salt wastes from pyroprocesses

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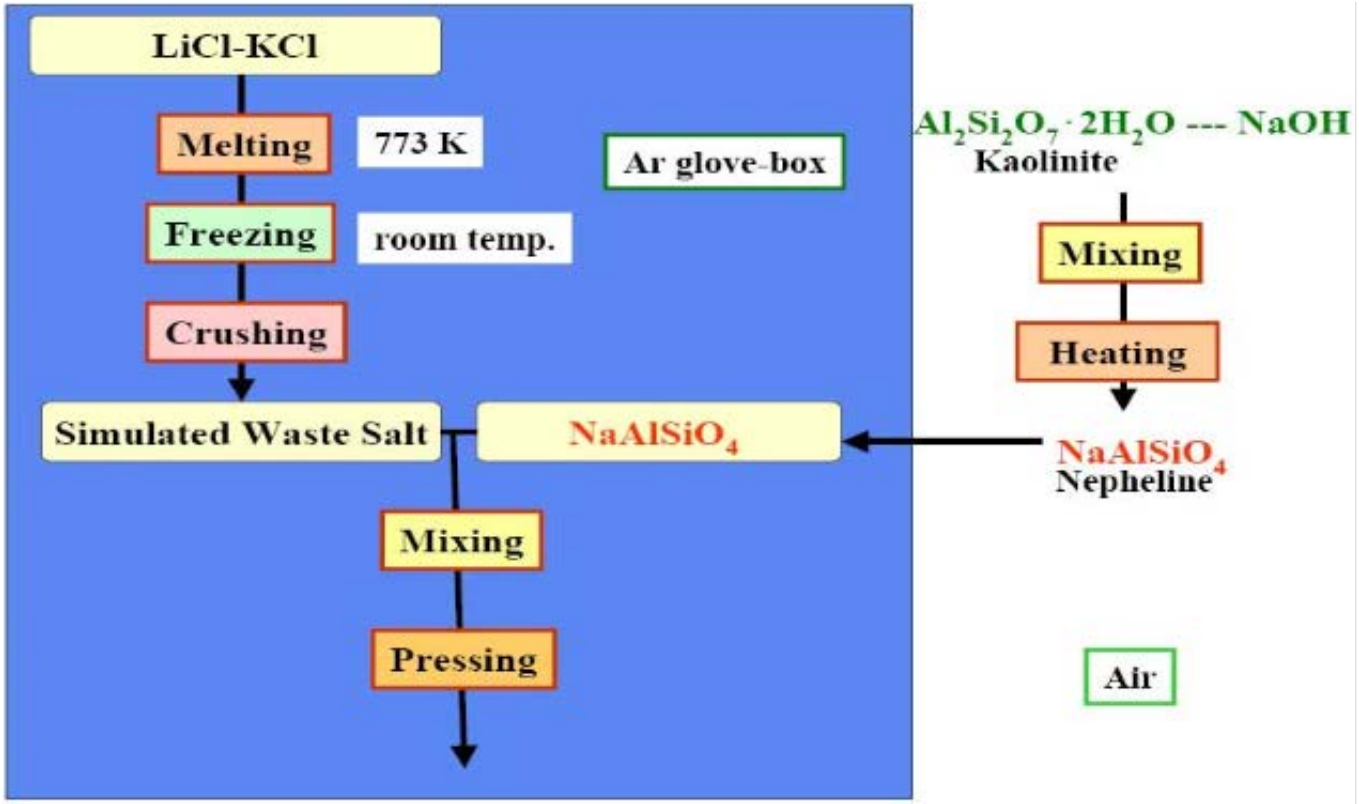
Conditioning of salt wastes with Sodalite

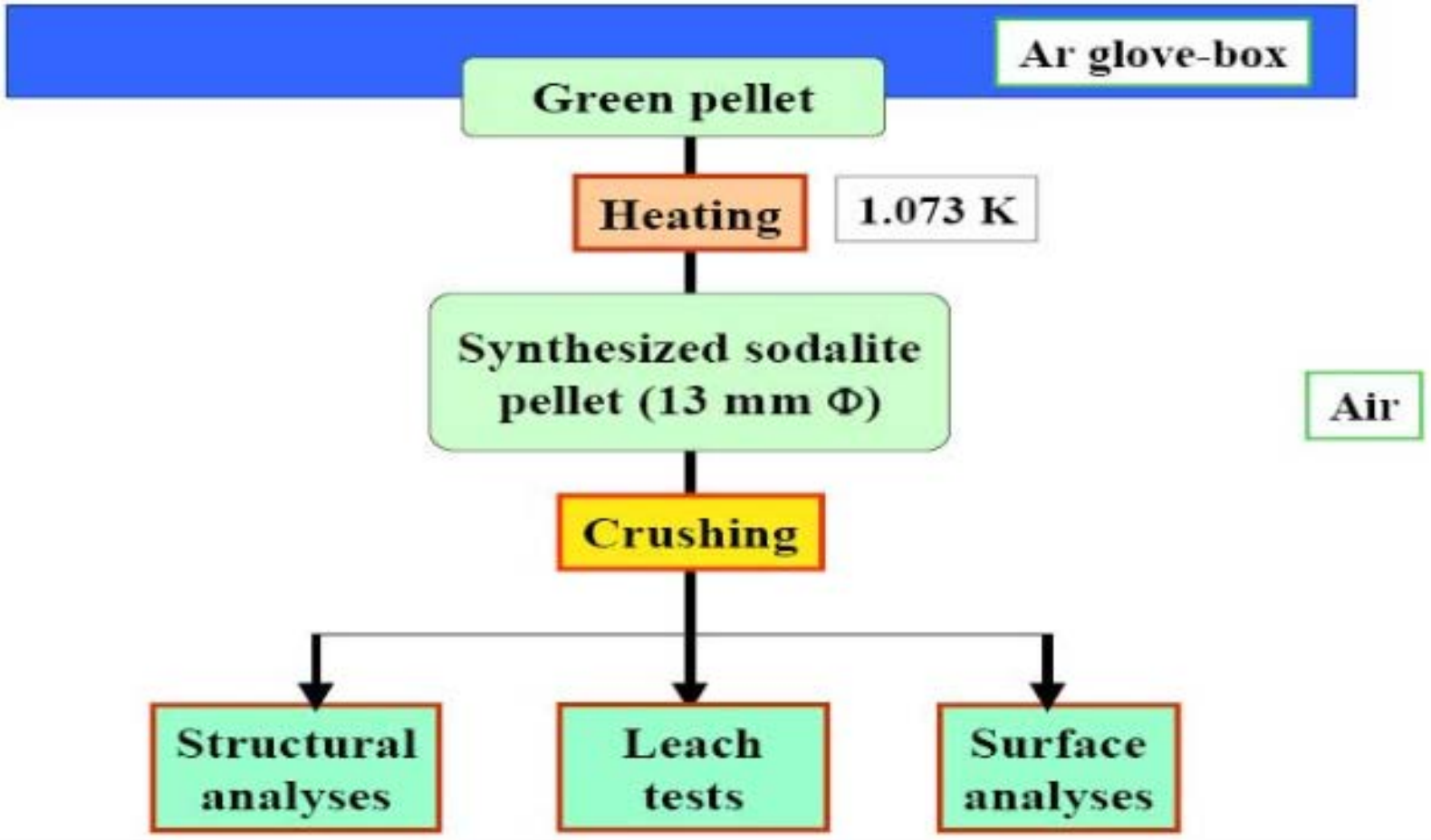


Synthesis of Sodalite from Kaolinite through Nepheline, by Hot Isostatic Pressing

$$\underset{\text{Kaolinite}}{Al_2Si_2O_7 \cdot 2H_2O} + 2NaOH \rightarrow \underset{\text{Nepheline}}{2NaAlSiO_4} + 3H_2O$$

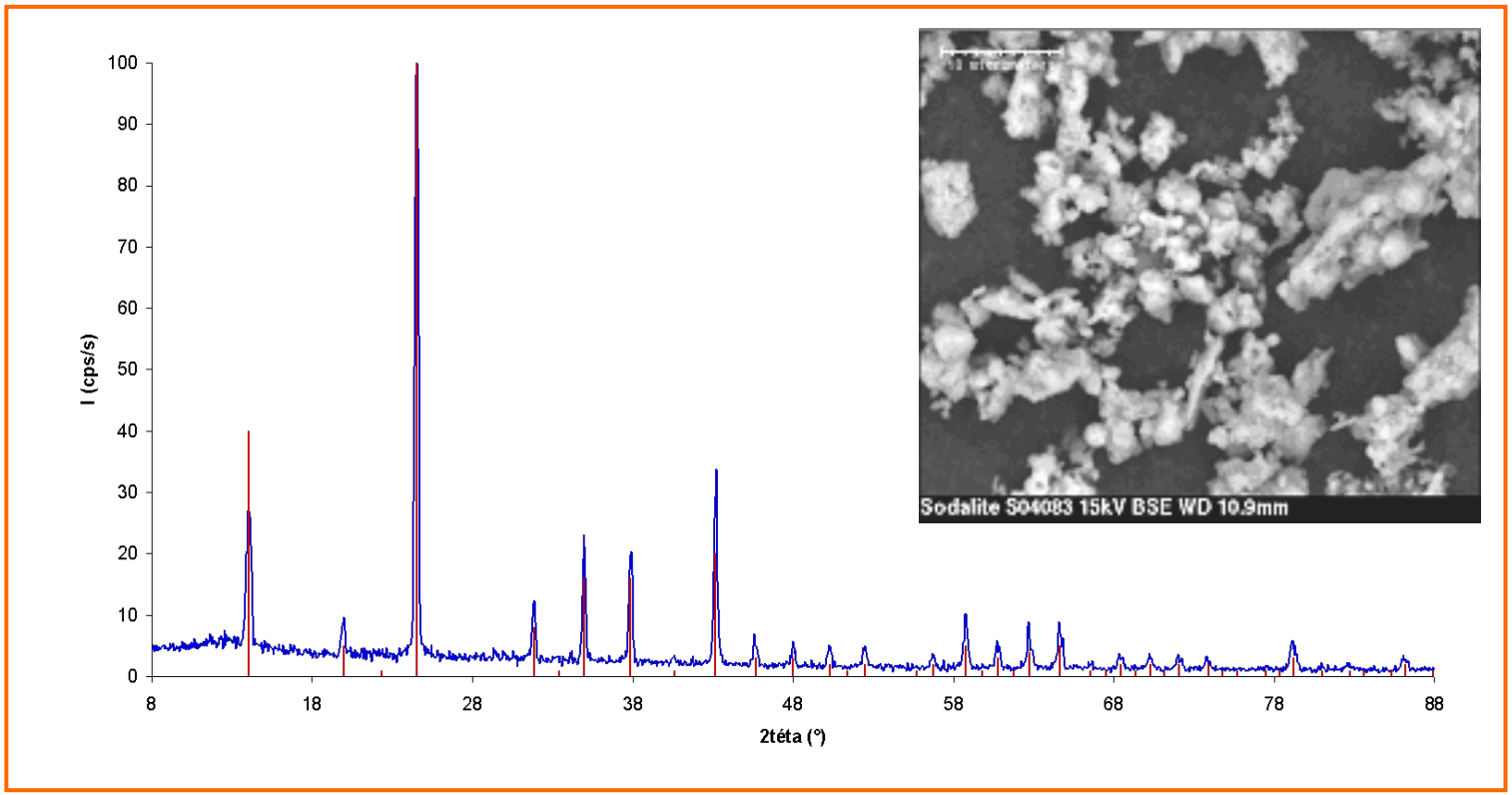
$$\underset{\text{Nepheline}}{6NaAlSiO_4} + 2\,(Li, K)Cl \rightarrow \underset{\text{Sodalite}}{Na_6(Li, K)_2[(AlO_2)_6(SiO_2)_6]Cl_2}$$

Conditioning of salt wastes with Sodalite



Synthesis of Sodalite from Kaolinite through Nepheline, by Hot Isostatic Pressing

Conditioning of salt wastes with Sodalite

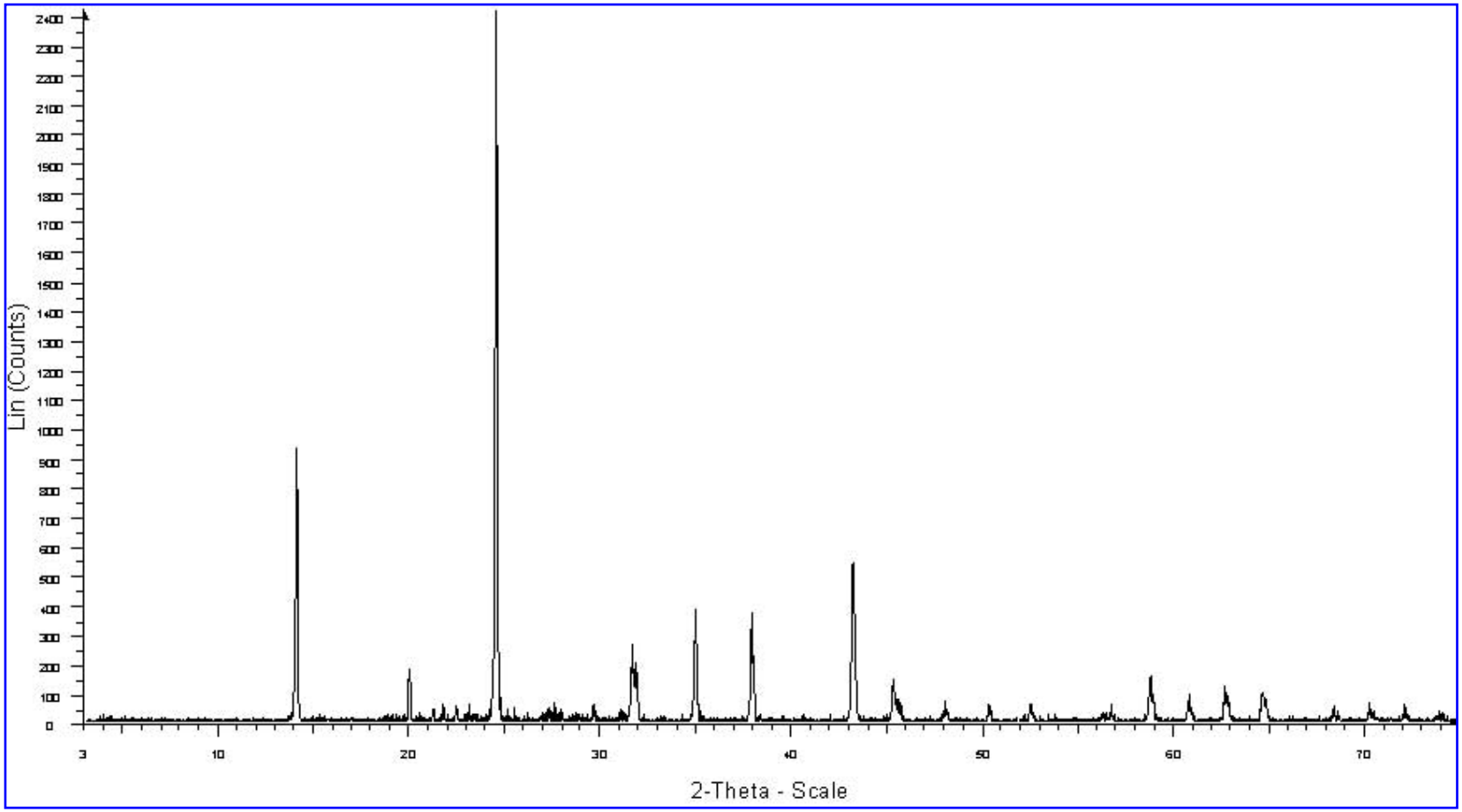


XRD spectrum of sodalite prepared from nepheline, according to HIP method

Conditioning of salt wastes with Sodalite

Pressureless Consolidation process at laboratory scale

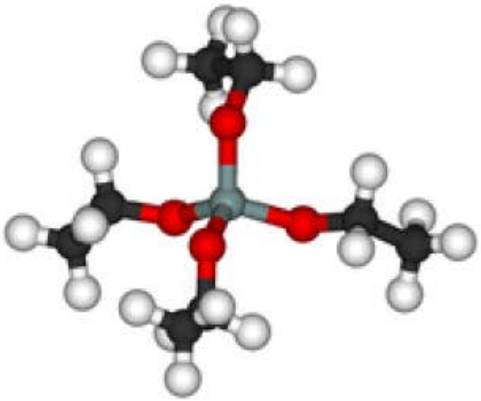
Conditioning of salt wastes with Sodalite



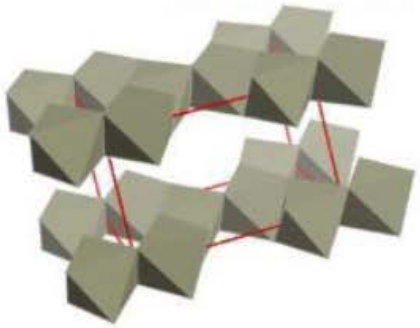
XRD spectrum of sodalite prepared from nepheline, according to Pressureless Consolidation method

Conditioning of salt wastes with SAP

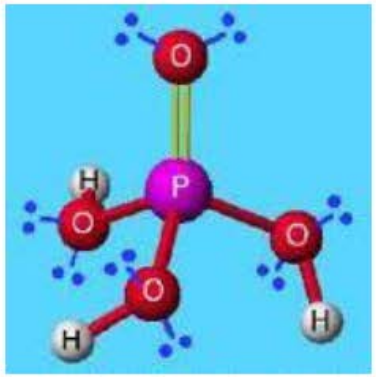
Sources of Si – Al – P



TEOS,
Tetraethyl orthosilicate
or tetraethoxysilane,
$Si(OCH_2CH_3)_4$
Gelling agent



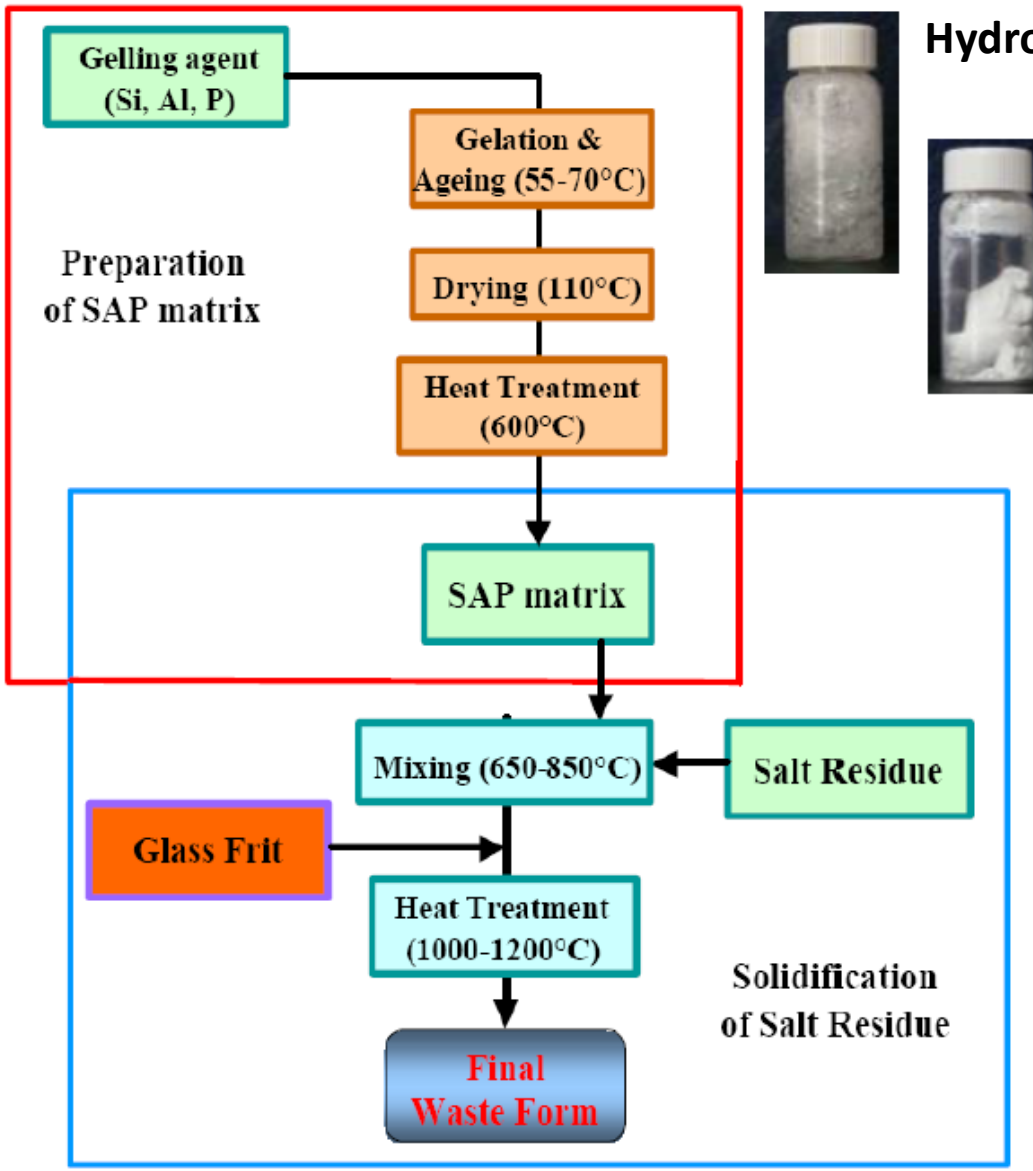
Aluminum (III) chloride,
$AlCl_3$
Property promoter



Phosphoric Acid,
H_3PO_4
Acidic catalyst/stabilizer

Conditioning of chloride salt wastes with SAP matrix

Conditioning of salt wastes with SAP

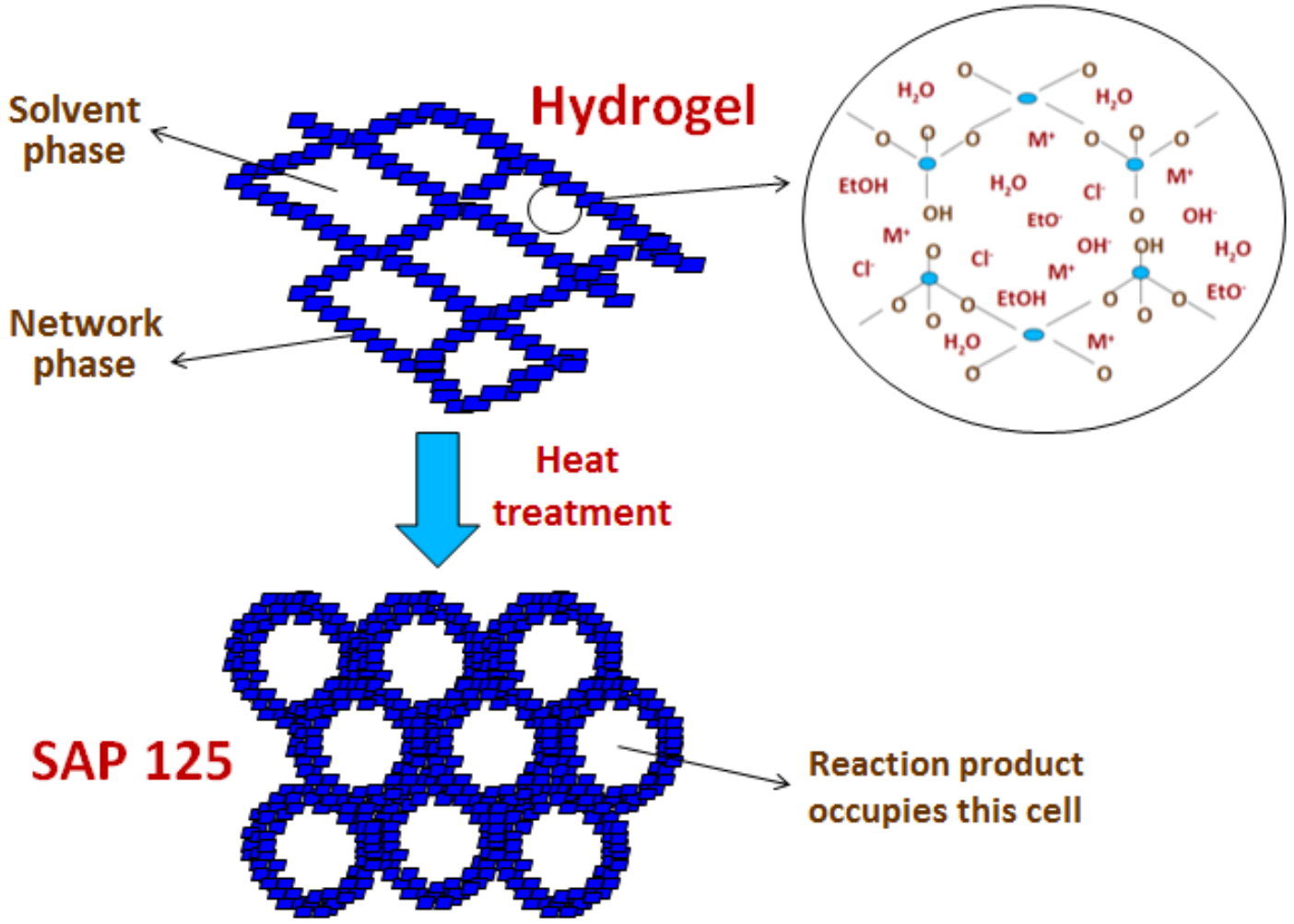


Hydrogel

SiAlP

SAP 125

Conditioning of salt wastes with SAP



Conditioning of salt wastes with SAP

Hydrolisis and polycondensation reactions

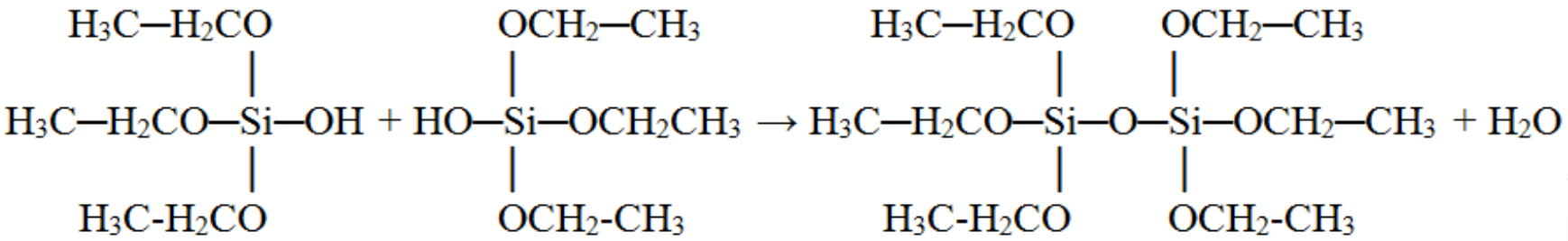
$$AlCl_3 + H_3PO_4 \rightarrow POCl_3 + Al(OH)_3$$

$$POCl_3 + CH_3CH_2OH \rightarrow POCl_{3-x}(OCH_2CH_3)_x + HCl$$

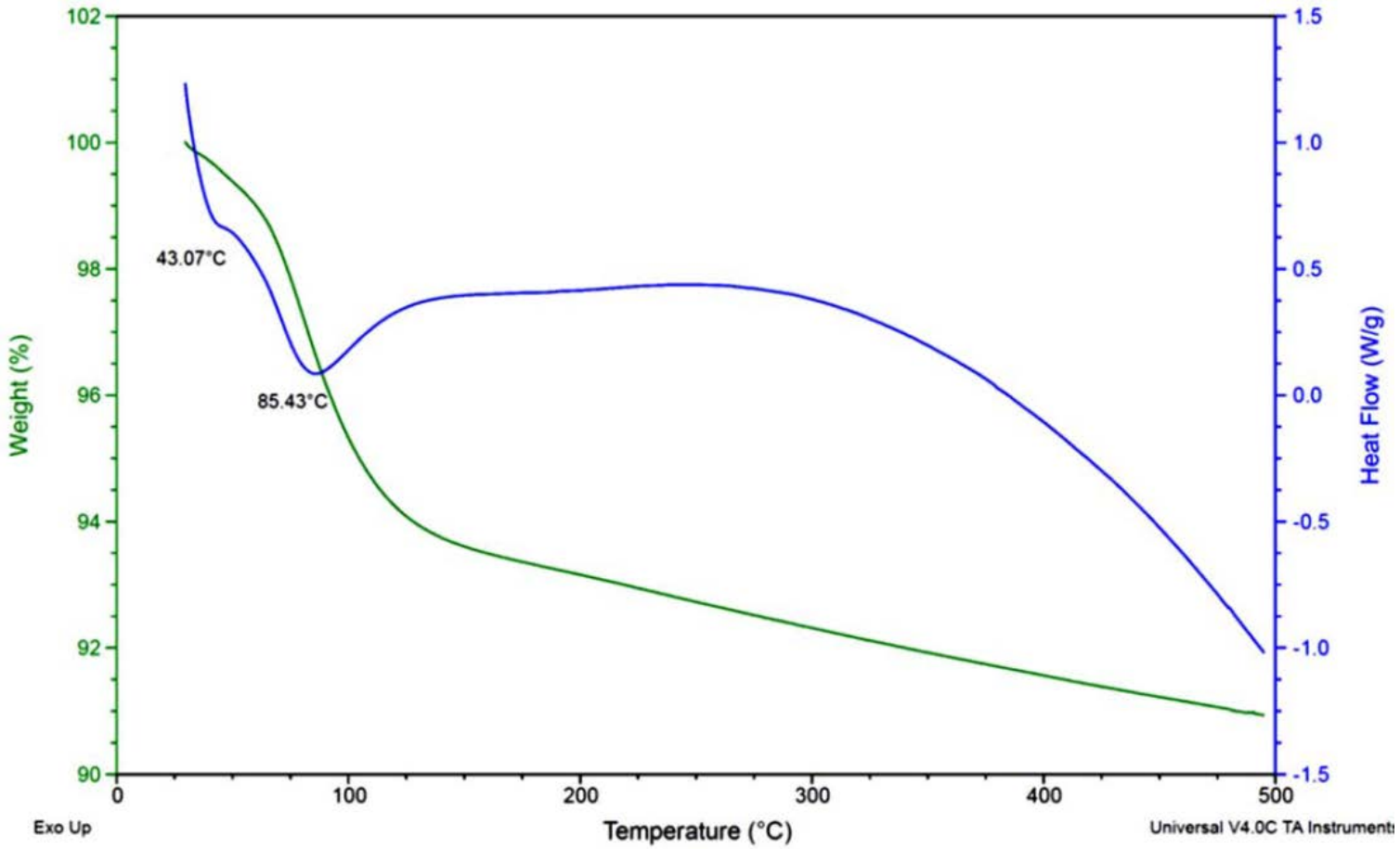
$$POCl_3 + H_2O \rightarrow POCl_{3-x}(OH)_x + HCl$$

Reaction of TEOS

$$Si(OCH_2CH_3)_4 + H_2O \rightarrow Si(OCH_2CH_3)_3(OH) + CH_3CH_2OH$$



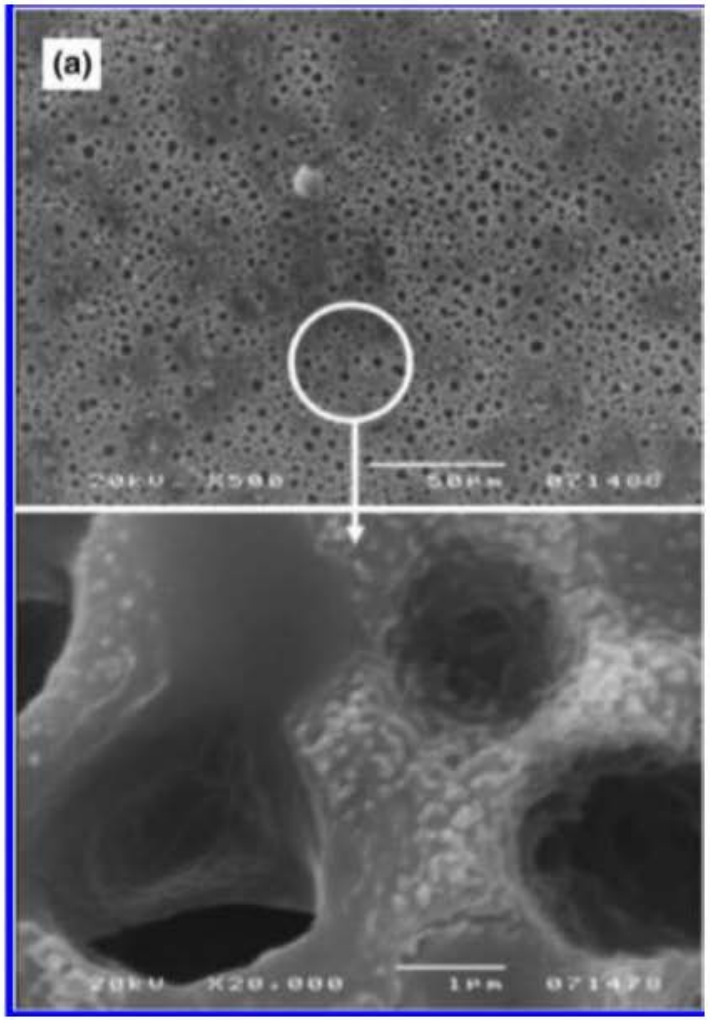
Conditioning of salt wastes with SAP

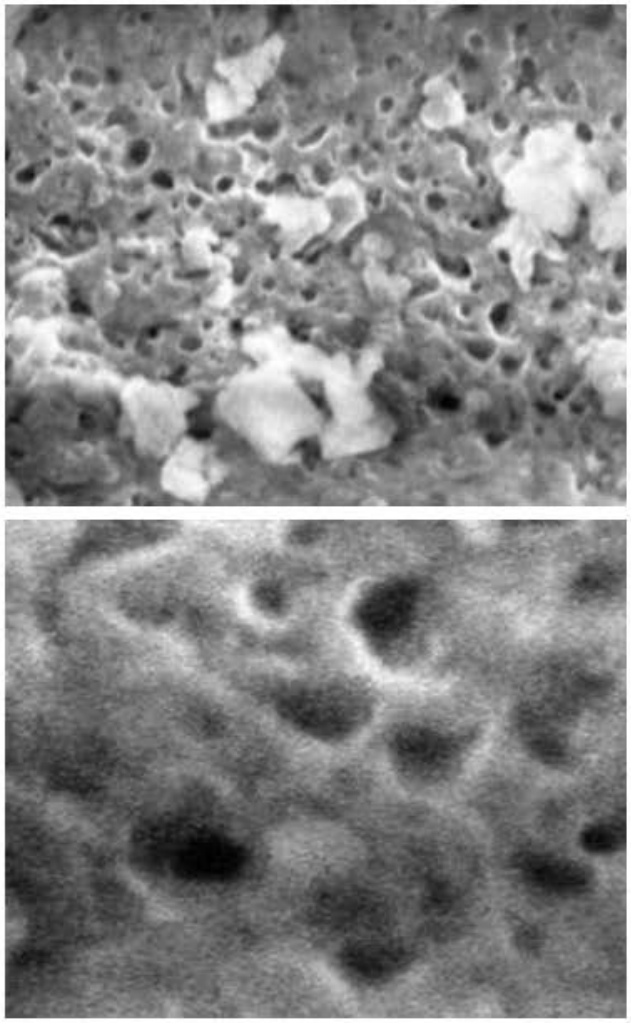


DTA (*top*) and DSC (*bottom*) curves for SiAlP sample

Conditioning of salt wastes with SAP

KAERI





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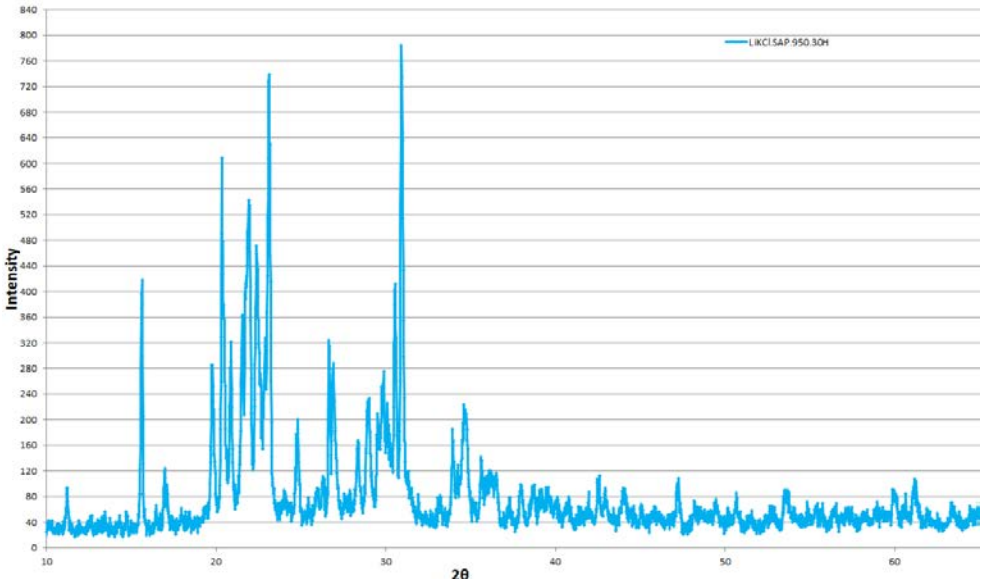
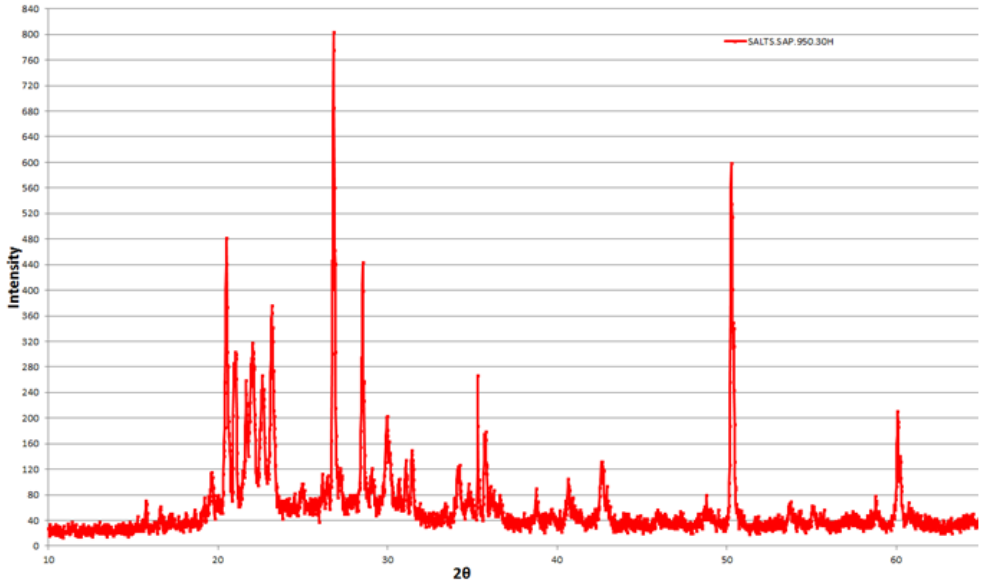
Conditioning of salt wastes with SAP



XRD of LiKCl.SAP.950C.30H



XRD of Salts.SAP.950C.30H

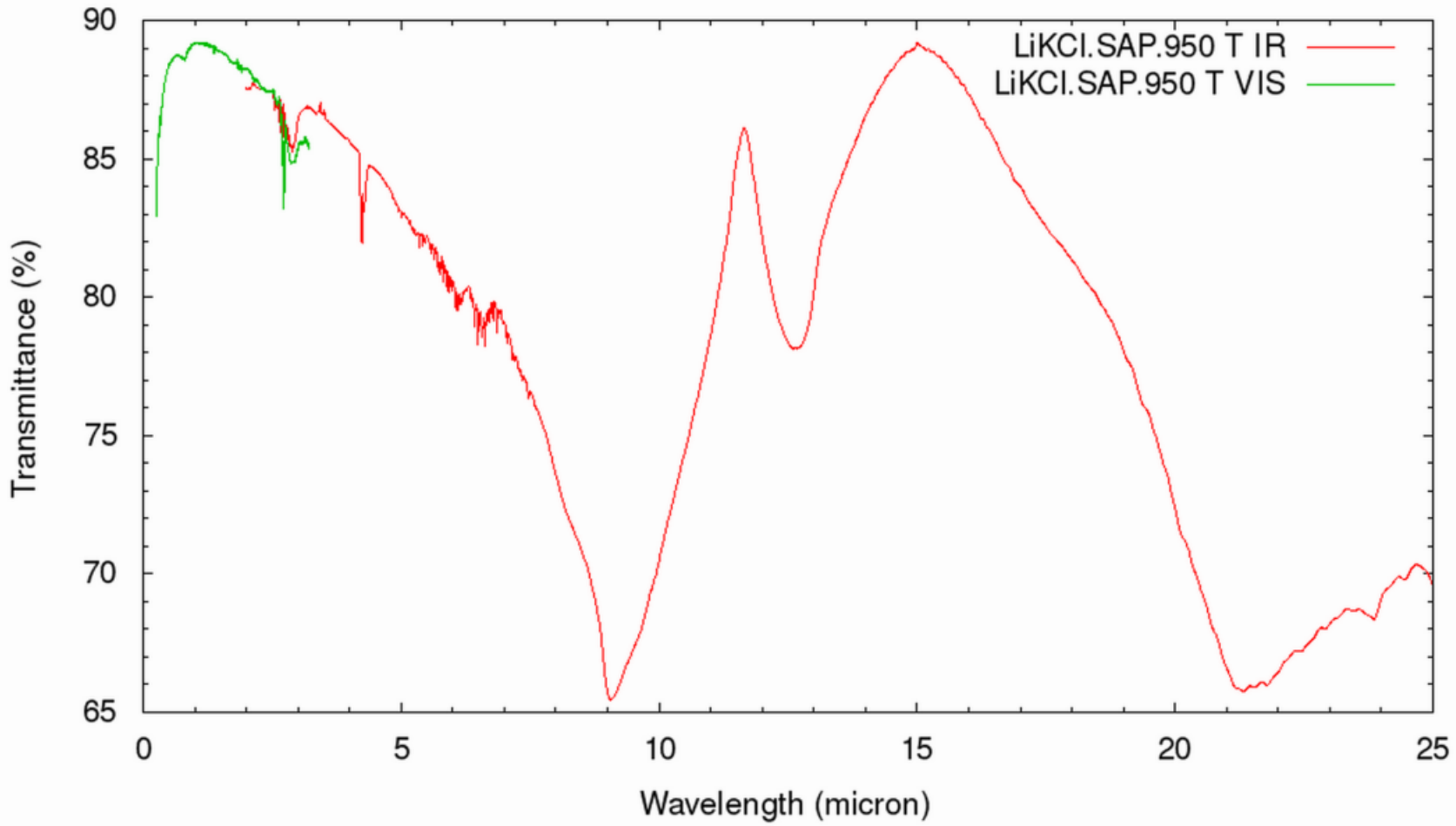
Conditioning of salt wastes with SAP

Examples of reactions between LiCl-KCl and SAP 125

$$LiCl + SAP \rightarrow Li_3PO_4 + Li_xAl_xSi_{1-x}O_{2-x} + Cl_2$$

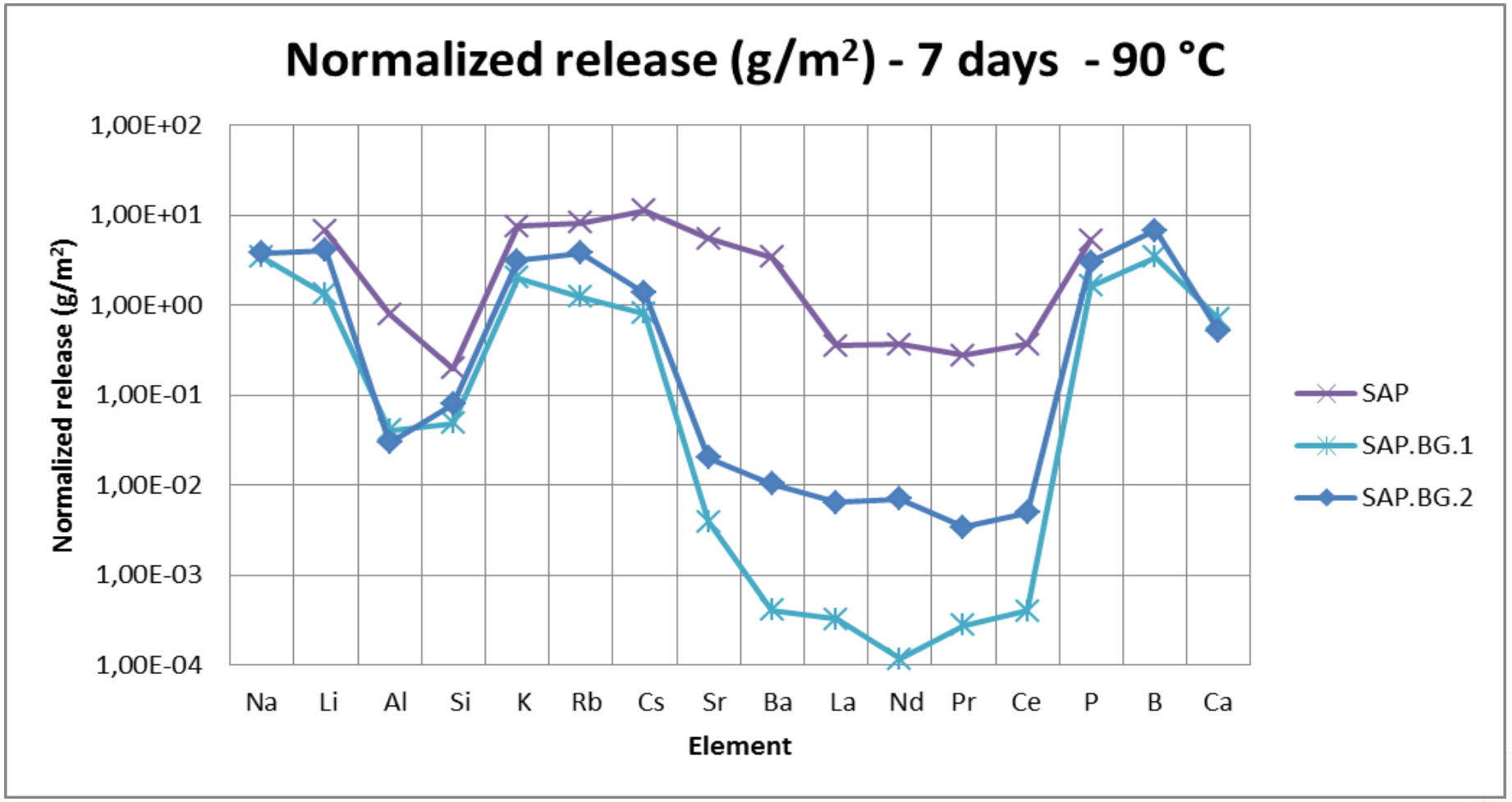
$$LiCl + KCl + SAP \rightarrow Li_3PO_4 + (Li, K)\text{-aluminosilicate} + Cl_2$$

Conditioning of salt wastes with SAP



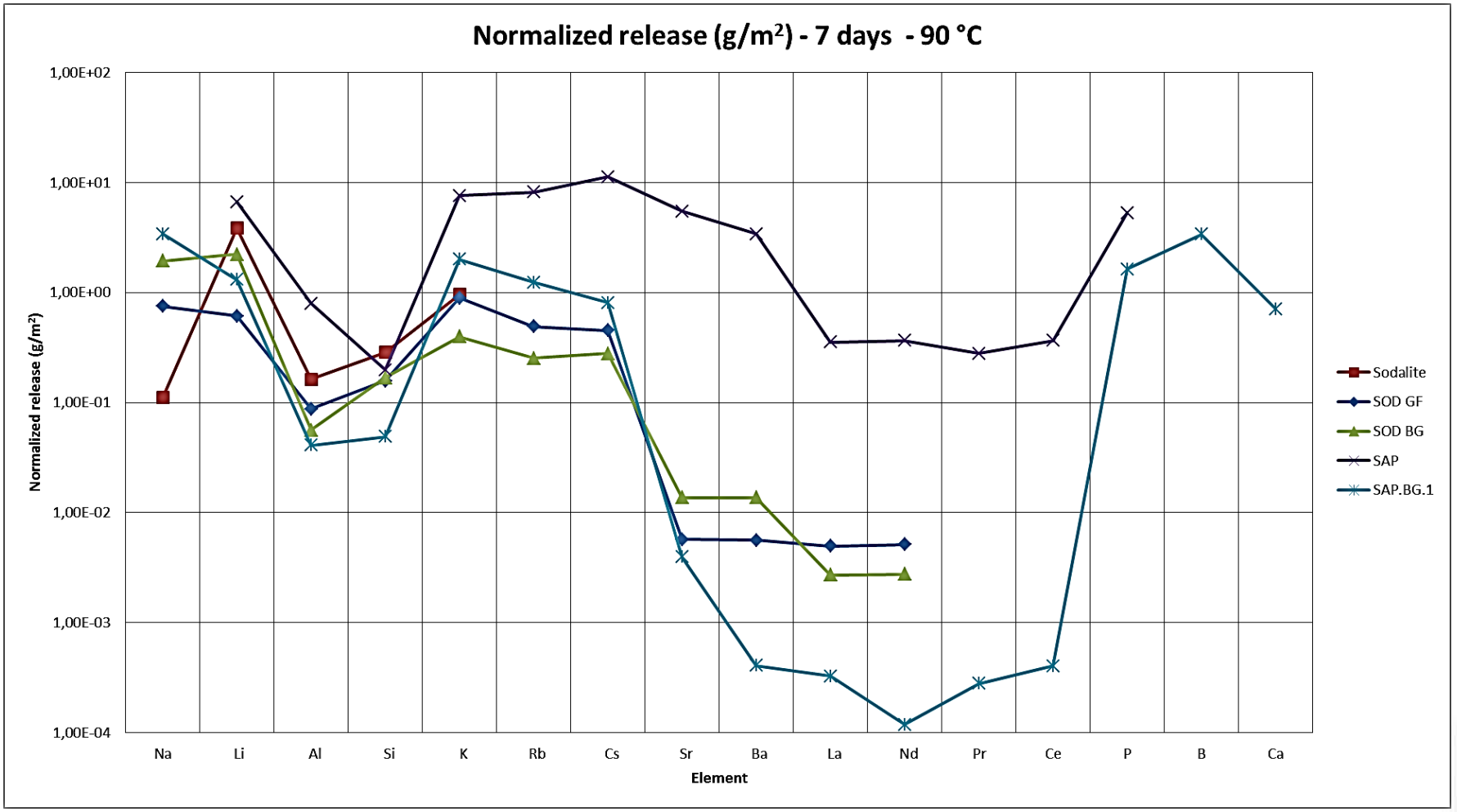
FTIR spectrum of LiKCl.SAP.950C.30H

Conditioning of salt wastes with SAP



Normalized release of SAP samples blended or not with borosilicate glass
SAP.BG.1: after heating at 1150 C for 4 hours;
SAP.BG.2: after heating at 1150 C for 1 hour

Conditioning of salt wastes with SAP



Normalized release of SAP and SOD samples blended or not with glass

Conclusions

- Experiments have been made to protect the steel crucible against corrosion, through an internal coating of alumina applied by Powder Injection Plasma Spraying Process (PIPSP), followed by Plasma Enhanced Chemical Vapour Deposition (PECDVD);
- a gas trapping system has been designed and applied at the top of a guide-tube connected to the crucible, in order to remove and transfer outside the box the gas generated during pyroprocesses;
- different methods for treatment of chloride salt wastes coming from pyroprocesses have or are being studied. The present research is focused on a new matrix, SAP (SiO_2-Al_2O_3-P_2O_5), synthesized by a conventional sol-gel process.

PYROPROCESS TECHNOLOGIES AT ENEA LABORATORIES

Thank you
for your attention

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