A proposal for a 100% use of bauxite residue:

The process, results on the novel Fe-rich binder and how this can take place within the alumina refinery

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The dirty by-product of the Bayer process...

- production: > 140 Mt of BR/yr
- landfilled: > 2.7 Bt
Imagine...

Stade, Germany¹

¹https://en.wikipedia.org/
²adapted from http://blogs.uni-siegen.de/
Imagine...

Stade, Germany\(^1\)

100 % use of BR

\(^1\)adapted from https://en.wikipedia.org/

\(^2\)adapted from http://blogs.uni-siegen.de/
Inorganic polymers (IP)

- Al- and Si-rich cementitious amorphous **binder**
- **polymerisation** of an alkali activated precursor
- alternative to OPC
Inorganic polymers (IP)

• Al- and Si-rich cementitious amorphous binder
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http://www.geopolymer.org/
Inorganic polymers (IP)

- Al- and Si-rich cementitious amorphous **binder**
- **polymerisation** of an alkali activated precursor
- alternative to OPC
Geopolymer is a term originally coined by French researcher Joseph Davydovits to describe a class of ceramic materials formed from aluminosilicates. Geopolymers have been known to provide enhanced physical performance to traditional cementitious binders but with the added advantages of significantly reduced greenhouse emissions, increased fire and superior chemical resistance.

A family of cementitious geopolymer mortar products designed for use in rehabilitation of pipes and structures for industry and private use.

Explore the possibilities for your own rehabilitation project or understand how other engineers and contractors have used our solutions.

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Inorganic polymers (IP)

- Fe-rich precursors such as copper, lead, ferro-nickel slags
  - Partially vitrified
  - Iron in oxidation state +II

1http://www.istgrup.com/
Goal

BR insoluble in alkaline media

Modification into Fe-rich precursor

Reduction of iron: \( \text{Fe}^{\text{III}} \rightarrow \text{Fe}^{\text{II}} \)

→ Support formation of liquid phase

Thermal treatment

Inspiration by Fe-rich precursors
• Partially vitrified
• Iron in oxidation state +II

Phase diagrams
Goal

How to increase liquid phase formation?

1083 °C

1093 °C
Goal

How to increase liquid phase formation?

1083 °C

1093 °C
Aim of present work

Development of near zero-waste process for the synthesis of IP
Overview of work

I. Characterisation of Bauxite Residue
   - XRF
   - XRD

II. High temperature processing
   - thermodynamic calculations
   - XRD

III. Synthesis of inorganic polymers
    - SEM
    - strength
Characterisation of BR

<table>
<thead>
<tr>
<th>Oxides</th>
<th>wt.%</th>
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</thead>
<tbody>
<tr>
<td>Fe$_2$O$_3$</td>
<td>47.9</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>18.9</td>
</tr>
<tr>
<td>CaO</td>
<td>11.0</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>9.6</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>6.5</td>
</tr>
<tr>
<td>Na$_2$O</td>
<td>4.0</td>
</tr>
</tbody>
</table>

XRF

XRD

°29CuK$_{\alpha}$
High temperature processing

FactSage calculation 1100 °C – Carbon addition

Graph showing the wt.% C and wt.% of various materials at different temperatures.
High temperature processing

FactSage calculation 1100 °C – Carbon addition

- $\text{Ca(Al,Fe)}_6\text{O}_{10}$
- $\text{Ca}_2(\text{Al,Fe})_8\text{SiO}_{16}$
- Corundum
- Fe
- FeO
- Mellilitie
- NaAlO$_2$
- Nepheline
- Perovskite
- Liquid phase
- Spinel
- Gas
- Ti-Spinel
High temperature processing

FactSage calculation 1100 °C – Carbon + silica addition
High temperature processing

FactSage calculation 1100 °C – Carbon + silica
High temperature processing

FactSage calculation 1100 °C – Carbon + silica

![Graph showing the phase diagram with points labeled 98.4BR_1.6C, 88.6BR_1.4C_10S, and 100BR.](image_url)
High temperature processing

Transformation of BR

- Induction furnace: \(1100 \pm 10 \, ^\circ \text{C}\) – 1 hour
- closed iron crucible, gas inlet/outlet
- inert atmosphere – Argon 99.995 %

Aim:

semi-vitrified material \(\rightarrow\) precursor for IP
XRD of precursor

a) 100BR

- c Calcium Iron Oxide,
- f Iron Calcium Silicate,
- G Gehlenite,
- H Hematite,
- I Iron,
- M Magnetite,
- m Maghemite,
- N Nepheline,
- P Perovskite,
- Q Quartz,
- S Spinel,
- W Wüstite,
- Z Zincite (internal standard)
XRD of precursor

a) 100BR

b) 98.4BR_1.6C

C Calcium Iron Oxide,
f Iron Calcium Silicate,
G Gehlenite,
H Hematite,
I Iron,
M Magnetite,
m Maghemite,
N Nepheline,
P Perovskite,
Q Quartz,
S Spinel,
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Z Zincite (internal standard)
XRD of precursor

a) 100BR

b) 98.4BR_1.6C

c) 88.6BR_1.4C_10S

- c Calcium Iron Oxide,
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- S Spinel,
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- Z Zincite (internal standard)
Synthesis of IP’s

- K-silicate activation solution, SiO$_2$/K$_2$O = 1.6, H$_2$O/K$_2$O = 16
- activation solution/solid ratio = 0.25
- Curing: 60 °C, 72 h

Steamoscope images

100BR  98.4BR_1.6C  88.6BR_1.4C_10S
Microstructure – SEM (SE)

denser microstructure
less pores, cracks
SEM & strength (3d) of IPs

**Compressive strength**

- 100BR: $13.4 \pm 0.4$ MPa
- 98.4BR_1.6C: $19.7 \pm 1.1$ MPa
- 88.6BR_1.4C_10S: $43.5 \pm 0.5$ MPa

**Flexural strength**

- 100BR: 4.2 MPa
- 98.4BR_1.6C: 5.5 MPa
- 88.6BR_1.4C_10S: 9.8 MPa
Process within alumina refinery

Bayer process → BR slurry → C → SiO₂ → mixing → filter press → BR cake → H₂O → alkaline liquor → alkalis, waterglass → IP → precursor → firing → Fe, Fe₃O₄

1http://img.tradeindia.com/
2https://encrypted-tbn1.gstatic.com
Conclusion

- BR was transformed into suitable Fe-rich precursor material for IP
  - addition of C, SiO\textsubscript{2}
  - heat treatment - 1100 °C

- Insoluble, dense IPs were successfully synthesised
  - dense microstructure
  - strength exceeding 40 MPa
Thank you for your kind attention!

To be continued ...