Tests of materials employed for geopolymers

Notes and experience

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Materials for Geopolymers

- **Evaluation of clayed minerals**

1. Chemical analysis
2. The identification by XRD analysis:

a) Content of clayed substance,
b) Identification of complementing materials,
c) Crystallography and morphology of the clayed minerals.

3. The identification of „activation“ temperature and dwell.
4. The identification of aluminum ion transformation by MAS-NMR, from natural six-fold coordination to oxygen to the five and four-fold coordination.

2a) The calculation of clayed substance from chemical analysis

- Usually, even the clayed material is washed we identify the rests of feldspar, small quantities of calcium/magnesium carbonates and silica sand beside the content of clayed mineral (XRD).

- The content of sodium, potassium oxides identify the content of corresponding feldspar when calcium, magnesium oxides and L.O.I. corresponds to carbonates and clay minerals (calculation based on chemical analyses).

- Classic and simplified formulas of feldspar (Na_2O [K_2O] Al_2O_3 $6SiO_2$) determinate content of silica oxide and alumina in feldspar, the clayed mineral is usually calculated using formula of kaolinit Al_2O_3 $2SiO_2$ $2H_2O$ when important is loss of water content identified as L.O.I. Which should be in accordance with losses of CO_2 from carbonates ($CaCO_3$ and $MgCO_3$ respectively.

- DTA/GTA (air and temperature 1000℃) analyses inden tify L.O.I. and help in identification of carbonate losses.

2c) morphology of clayed mineral

We have had the possibility to study different types of kaolin (form Czech Republic, Ukraine, Sri Lanka, Jordanian Kingdome etc.). Different sorts of kaolin deposit with various nascent history result in divert affinity to the conversion to geopolymer base matrix.

Main differences were identified as:

1) The morphology of kaolinitic type,
2) The clayed particle size,

From the morphology of the kaolinit could be estimated the temperature of „activation“ and time of temperature dwell. (Our laboratory will present in short time the method identifying the type of kaolinit morphology, based on crystallography of kaolinit).

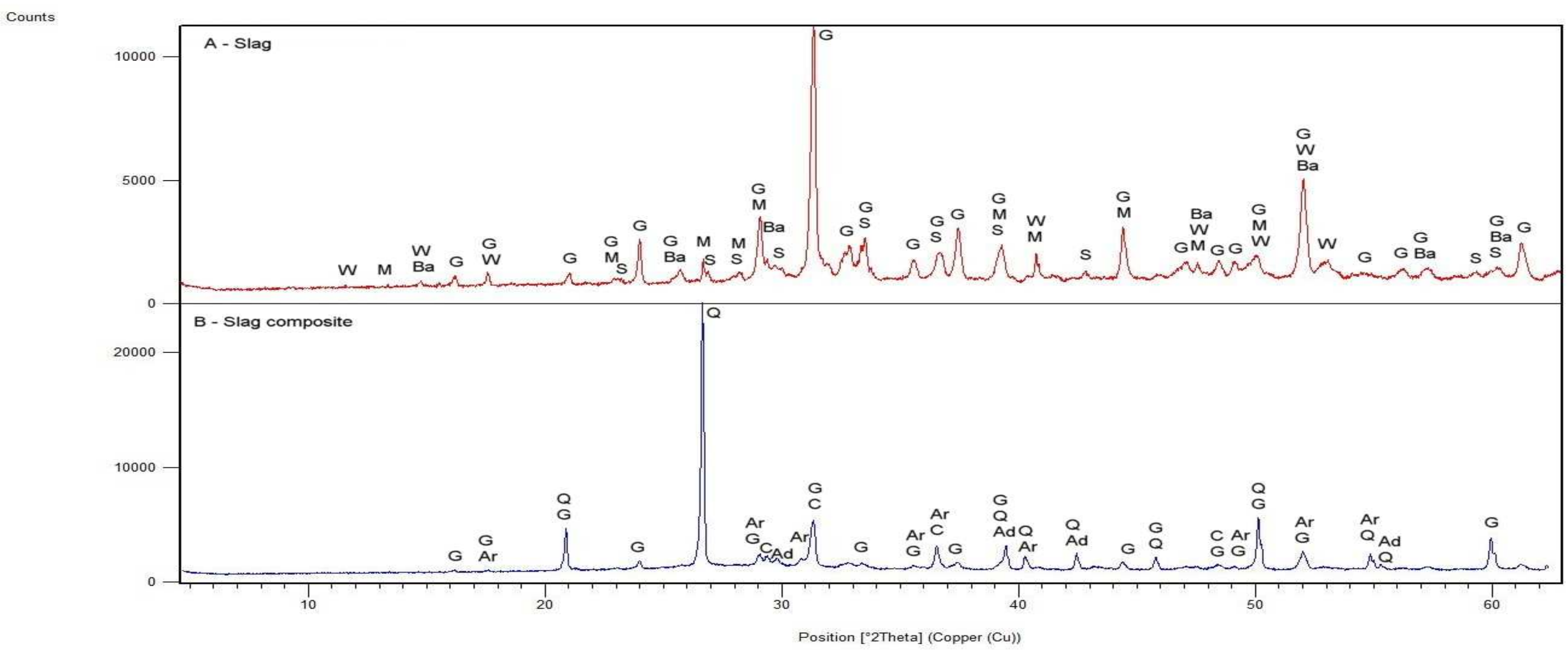
Materials for Geopolymers

- **Evaluation of non-clayed materials**

1. The chemical analysis.
2. The identification of minerals by XRD.
3. The analysis by ^{27}Al MAS-NMR in case of thermally treated materials (coal ashes, specifically from fluid burning boilers).
4. Raw materials (natural state) identified by XRD are generally evaluated as fillers (sand, granite, mica, calcite but also wood chips, rice husks or fabrics, etc).
5. Specific attention should be paid to the calcareous slag, volcano ashes and naturally fired slate clays take part of thermally transformed materials. Naturally fired or burnt material by human activity (ashes from fluid burning boilers) should be considered as a material for geopolymer technology.

Example of calcareous slag

- Blast furnace slag contains gehlenite $Ca_2Al(AlSi)O_7$ complemented by merwinite Ca_3 Mg $(SiO_4)_2)$, bassanite ($CaSO_4$. 0.5 H_2O), syngenite (K_2Ca $(SO_4)_2.H_2O$) and wolastonite ($CaSiO_3$we observe important changes as follow:



Specification

- We could divide additives in three groups:

First – inert ones are encapsulated by surrounding matrix and fixed in it (quartz sand, grains of corundum, grains of silicium carbide, granite, basalt, etc. etc.)

Second – additives which partially enter to the reaction by its superficial layers and forms interlayer matrix which differs from used one (coal ashes).

Third – “additives” which enter to the reaction and form inseparable part of formatted 3D netting. (blast furnace slag, volcano ashes, coal ashes from fluid boilers).

Preparation and stability

The preparation type influences on:

a) workability,
b) mechanical properties,
c) long term stability.

- Water content,
- Proportion of Si/Al molar quantities,
- The quantity of transformed of Al^{3+} ions from octahedrons to tetrahedrons,
- Proportion of R^{+}/ $Al(OH)_4^{-}$,
- Mixing time and homogenity of mixed material,
- Time and type of maturation.

Geopolymer matrix and geopolymer binder

- Well prepared, none filled, clay based geopolymer matrix poured into the mold, vibrated to avoid air bubbles, sets and hardens **in all mixed volume** during 12-16 hours at ambient temperature.

- Best results are obtained when the molds are enveloped in plastics (protection of the water surface evaporation) and placed into the dryer 40-60°C.

- Sodium activated alumino-silicate ($Al_2O_2Si_2O_5$),formula according to Davidovits, very often presents efflorescence when exposed to the humidity, even when well calculated Na/Al and Na/H_2O ratio.

- Potassium activated material resolves the problem and the best results in minimum efflorescence shows potassium/calcium combination.
- Binder means the use of geopolymer matrix as cementitious material in quantity from approx. 8 wt. % to 75 wt.%.

Geopolymer cementitious binder

- The prepared composite generally takes properties according to the filler and its quantity. If filled by 85 wt. % by granulated calcite, composite results in highly porous calcareous solid, quartz sand in the same proportion will produce sandstone like solid, etc.

- The quality of a matrix itself could be controlled by FTIR analysis, by the identification of the vibrations Si-O, Al-O in corresponding bands.

- Obvious quality should be: Insolubility in water, resistance to the thermal shocks and resistance to the acid, alkali attacks. Due to the grain size and quantities of additives the porosity could be change in very wide scale.

- The matrix, regarding the type of preparation: From dissolved clay particles in alkali aqueous solution, converted by poly-condensation to the solid state, results as a porous matter. The dominant pores size is 20 nm and every type of future application should respects matrix porosity.

Geopolymer composites

- The geopolymer composites are mixtures of matrix with wide types of different materials (e.g. sand, calcite, chalk, feldspar and mica), but also with purely organic matter (wood chops, paper, straw etc).

- The composites, even the cementitious matrix is well calculates and prepared, have disadvantage in non specified chemical reactions among different (more or less stabile in alkali aqueous conditions) components and therefore is difficult to avoid efflorescence on the surface or even cracks and fissures.

- The geopolymer matrix applied on fabrics (cotton, wool and also glass or basalt) in thin (two-three) layers gives composites of specific properties – easily workable and apt for different shapes and forms.

The example of thin, multi-layered geopolymer composite

Thermal and fire resistant coating separating cars or busses motor compartments from rest of the vehicle could prevents tragedies and losses of human lives. (Picture shows geopolymer applied on glass fabrics).

Conclusion

- Wide scale of applications, in form of non filled geopolymer matrix or use it as cementitious agent of different inorganic and organic materials, offers large variety of use.
- In conservation and maintenance of historical monuments should be exploit the advantage of perfect imitation of the original material and easy and durable application.
- Geopolymer sandstone (calcite) like layer applied on damaged sandstone (calcite) could create „sacrificed layer“ and protect the original.

Thank you for your attention