Tests of materials emploied 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 the 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 can identify the feldspar residues and silica sand. Some times also small quantities of calcium or/ and magnesium carbonates beside the content of clayed mineral.

- The content of sodium, potassium oxides identify the content of corresponding feldspar. Clay mineral and calcium, magnesium carbonates correspond with determined amount of L.O.I.

Based on classic and simplified formulas of feldspar ($Na_2O\ Al_2O_3\ 6SiO_2$) could be calculated the content of silica oxide and alumina in feldspar, the clayed mineral is usually calculated using formula of kaolinit $Al_2O_3\ 2SiO_2 2H_2O$. The loss of water content is identified as L.O.I., which should also be in accordance with losses of CO_2 from carbonates.

- e.g. use of stechiometric proportions:

  $Na_2O = 2 \times 22.98 = 45.96 + 16 = 62$

  $Al_2O_3 = 102$

  $6SiO_2 = 6 \times 60 = 360$

2c) morphology of clayed mineral (kaolinit)

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 as geopolymer base matrix.

Main differences were identified as:

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

From the morphology of the kaolinite could be estimated the temperature of „activation“ and time of temperature dwell. (Our laboratory will present in short time the testing method to identify the type of kaolinit morphology based on kaolinite crystal type and corresponding affinity to the aluminum coordination change).

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, natural volcano ashes and slate clays which take part of materials thermally transformed, naturally or by human activity.

Preparation and stability

The preparation type influences on:

a) workability,

b) mechanical properties,

c) long term stability:

- Water content,
- Proportion of Si/Al molar ration,
- 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 geopolymer matrix poured into the mold, vibrated to avoid the air bubbles, sets and hardens in 12-16 hours at ambient temperature.
- Best results are obtained when enveloped in plastics (protection of the surface water 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.
- Potassium activated material resolves the problem – the best results is a combination of potassium/calcium activators use.
- 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 – e.g. if filled by 30- 40 wt. % with powdered mica, composite resists to the high temperature, if filled by 85 wt. % by granulated calcite, composite results in highly porous calcareous solid, etc.

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

- Obvious quality should be: Insolubility in water, resistance to the thermal shocks and resistance to the acid attacks.

- The matrix, regarding the type of preparation, from dissolved clay particles in aqueous alkalis to the solid, results in porous matter and any type of future application should respects the matrix porosity.

Geopolymer composites

- The geopolymer composites are mixtures of several (generally inorganic) materials (e.g. sand, calcite, chalk, feldspar and mica) to imitate e.g. - ceramics.

- The composite, even the cementitious matrix is well calculates and prepared, has 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 or five layered plates) 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).

Microscope observation on sandstone join

Microscopy of joined limestone

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. (Specifically studied case was the inhibition of industrial waste and hazardous metals in geopolymer matrix).
- In conservation and maintenance of historical monuments should be exploit the advantage of perfect imitation of the original material - easy and durable application.
- Geopolymer sandstone-like (calcite-like) layer applied on damaged sandstone (calcite-like) could create „sacrificed layer“ applied on original historic piece and protect the original.

Thank you for your attention