



# Effect of mechanical activation of fly ash on geopolymer strength

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# About our Institute

Institute of Raw Materials Preparation and Environmental Process Engineering

**Established in 1923 in Sopron**

**Department of Ore and Coal Preparation (1923-1960)**

**Department of Mineral Processing (1960-1993)**

**Department of Process Engineering(1993-2007)**

**Institute of Raw Material Preparation and Environmental Processing (2007-**

**Head: Prof. József Finkey (1923-1941)**

**Prof. Gusztáv Tarján (1941-1972)**

**Prof. Iván Tarján (1972-1995)**

**Prof. Barnabás Csőke (1995-2010)**

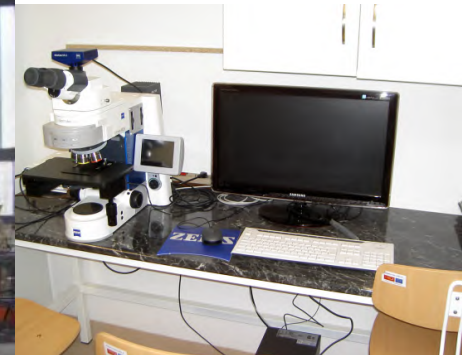
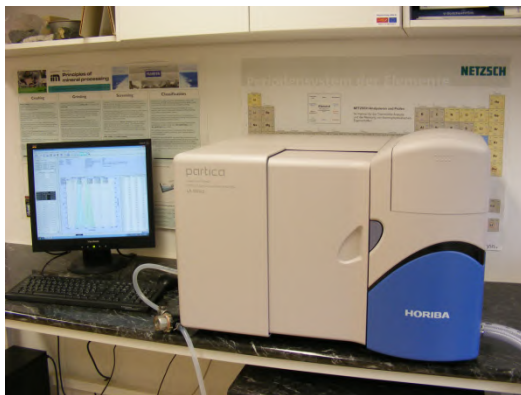
**Ass. Prof. József Bőhm (2010-2012)**

**Ass. Prof. József Faitli (2012-**



# About our Institute

Processing of raw materials and wastes (preparation) by **mechanical, physical, physical-chemical** (comminution, separation processes based on the difference of physical properties, such as density optical, magnetic, electric, thermal, surface and state properties, as well as agglomeration, mixing and homogenisation), **biological processes**.





# Problem

- Power station fly ash is generated in huge amount worldwide – only in China and India about 300 million tons per year, in Europe this number is 60 million tons.
- Depositing this enormous big quantity is problematic from several points of view, e.g. fly ash dumpsite reduce the valuable fields from agricultural activity and furthermore it means an environmental risk.
- Given the EU regulations, it is crucial to develop new technologies that allow the recycling of coal fly ash into added-value products.
- The utilization of this by-product as a geopolymer additive, can effectively solve this problem due to its immobilization capacity and special characteristics.





# Aim of the research

- To carry out laboratory scale experiments in order to investigate **the applicability** of **deposited** (decades ago) F type **fly ash** as a **starting material** (main component) of geopolymer.
- Three main characteristics were measured during experiments: (1) **compressive strength**, (2) **microstructure** (SEM), (3) **mineral composition** (XRD).
- The examination of the **effect of mechanical activation of raw material on the geopolymer properties**.
- Additives: glass waste, perlite, coal gangue, biomass fly ash.



# Materials

| Table 1. Chemical composition of deposited Class F fly ash (Berente) |                       | Tiszaújváros | Pécs  |
|--|-----------------------|--------------|-------|
| Component  | Mass concentration, % |              |       |
| L.O.I.   | 6.80                  | 2.85         | 4.21  |
| SiO <sub>2</sub>   | 53.67                 | 59.05        | 54.98 |
| Fe <sub>2</sub> O <sub>3</sub>                                       | 7.04                  | 5.42         | 8.93  |
| Al <sub>2</sub> O <sub>3</sub>                                       | 20.12                 | 26.14        | 23.82 |
| CaO  | 6.49                  | 2.3          | 2.00  |
| MgO  | 1.36                  | 1.12         | 1.16  |
| SO <sub>3</sub>  | 0.95                  | 0.25         | 0.12  |

Chemical composition of the deposited fly ash



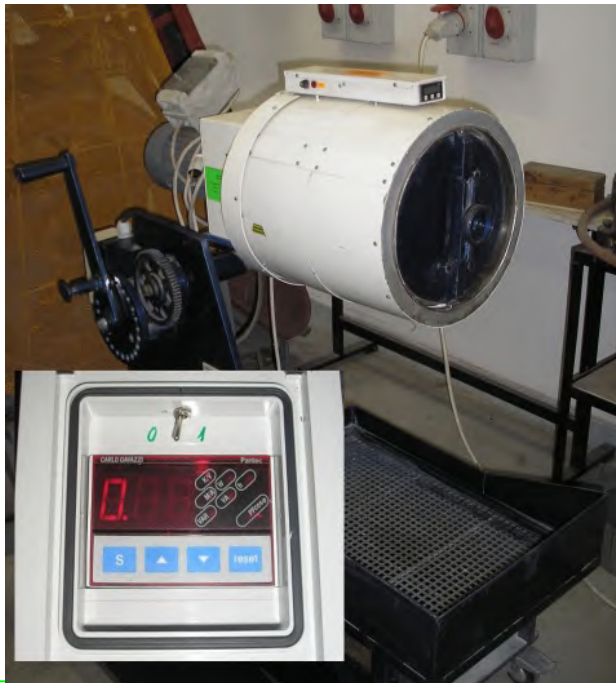
From the analysis the SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio was found to be

- Berente fly ash (brown coal 1) 2.67,
- Tiszaújváros fly ash (brown coal 2): 2.25,
- Pécs fly ash (black coal): 2.31.



# Method

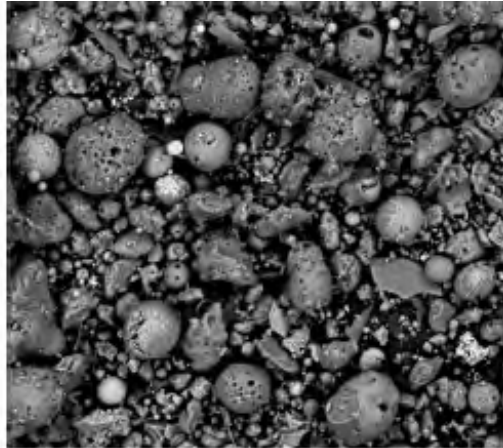
- Mechanical activation – batch ball mill (10, 20, 30, 60, 120 min).
- Geopolymer production technology – cylindrical mould (Ø50x50 mm), heat treatment at 150 °C, utilization of caustic spent liquor from alumina refinery.
- Analysis – SEM, uniaxial compressive strength, XRD.



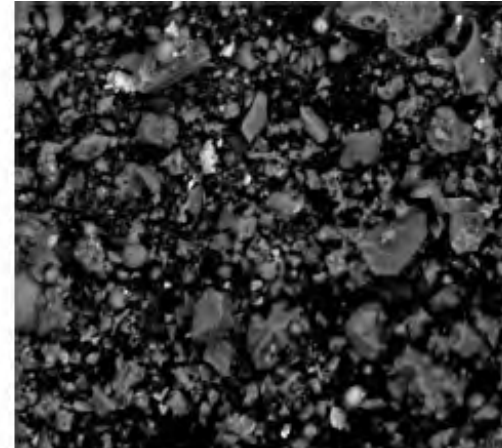


# Results

Spherical like particles



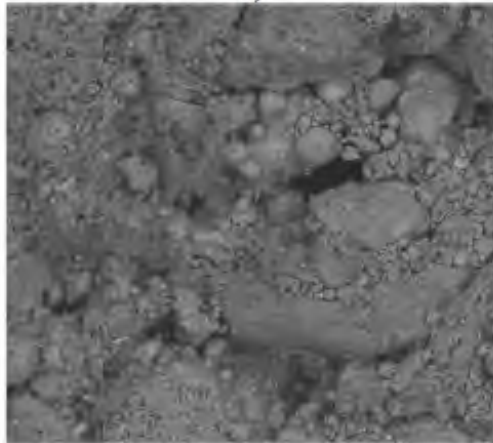
a)



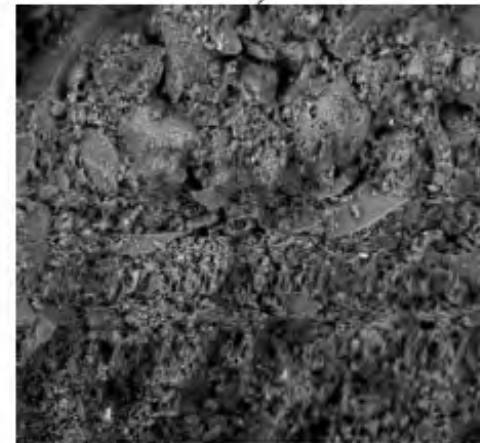
b)

Angular shape particles

Open structure, unreacted particles, heterogenous



c)

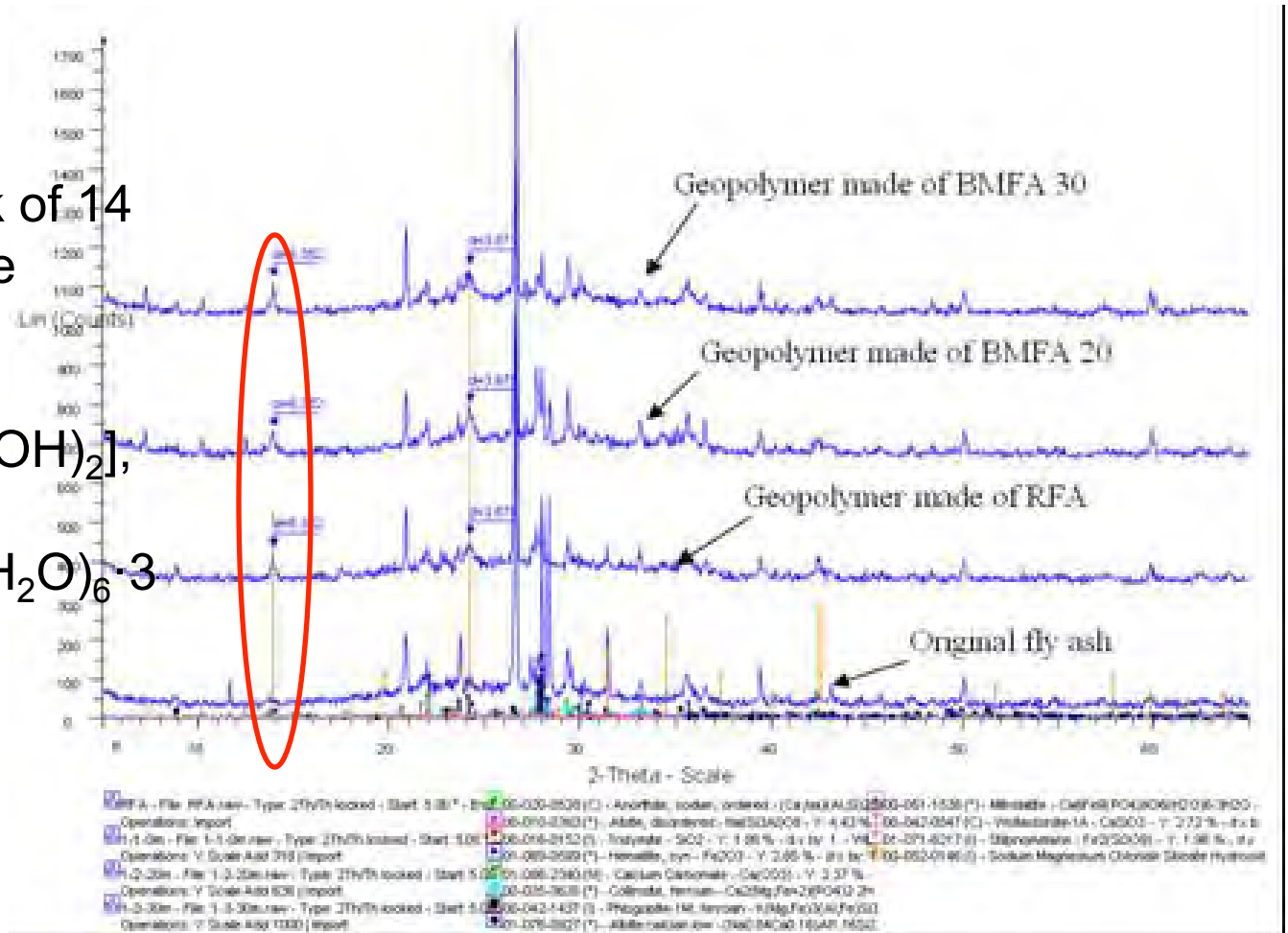


d)

Denser, more compact microstructure

Figure 2. SEM images of raw fly ash (a), ball milled fly ash for 20 min (b), and geopolymers made of RFA (c) and BMFA 20 (D)

# Results



X-ray diffraction patterns (Bruker) of raw and activated fly ash based geopolymers and the initial fly ash

New phases (peak of 14 and 24 degree) are generated:

**sodalites**  
 $[\text{Na}_8\text{Mg}_3\text{Si}_9\text{O}_{24}(\text{Cl},\text{OH})_2]$ ,

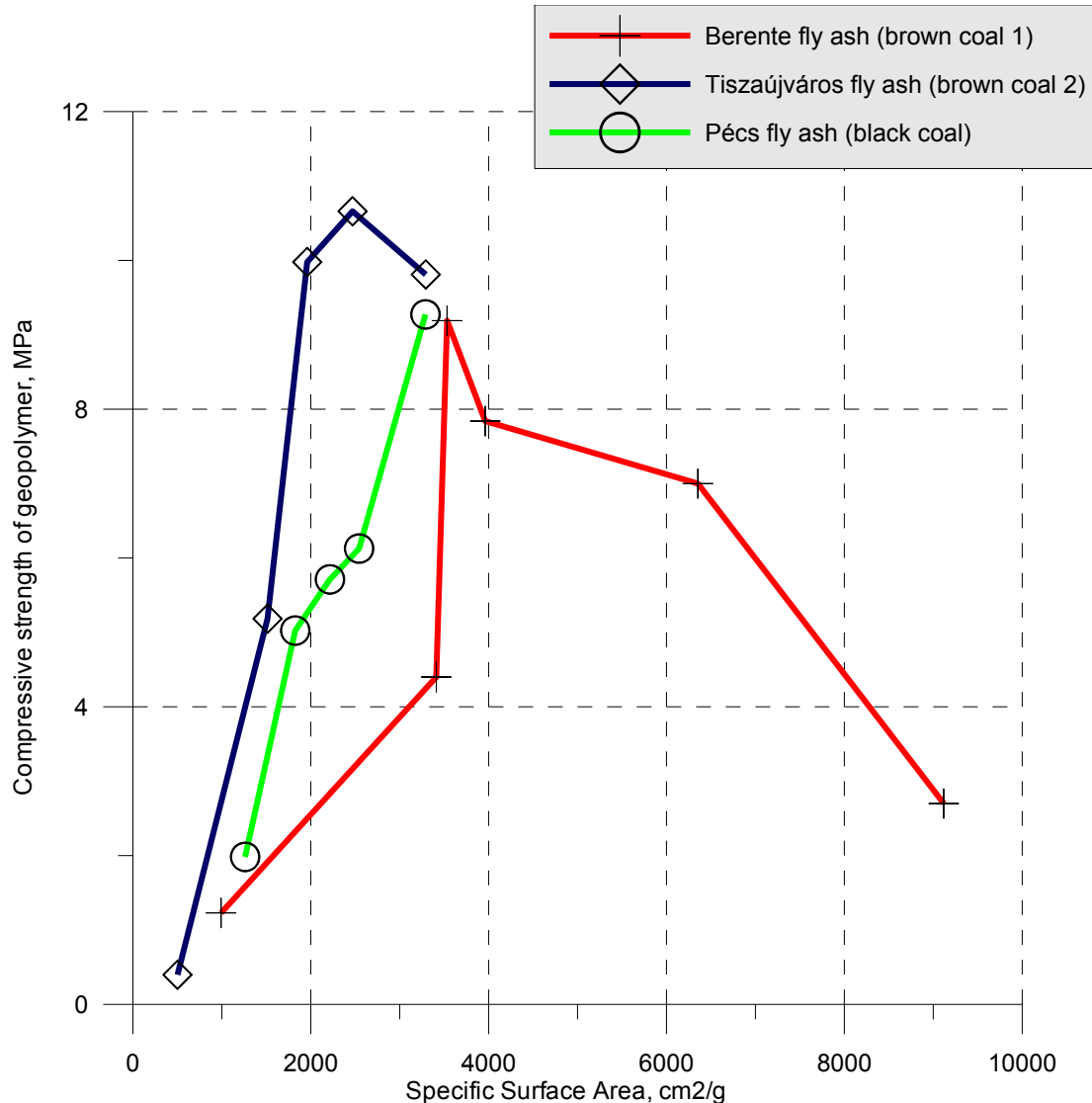
**mitridatite**  
 $[\text{Ca}_6\text{Fe}_9(\text{PO}_4)_9\text{O}_6(\text{H}_2\text{O})_6 \cdot 3\text{H}_2\text{O}]$ ,

**wollastonite-1 A**  
 $[\text{CaSiO}_3]$ ,

**stilpnomelane**  
 $[\text{Fe}_2(\text{Si}_3\text{O}_9)]$ .



# Results



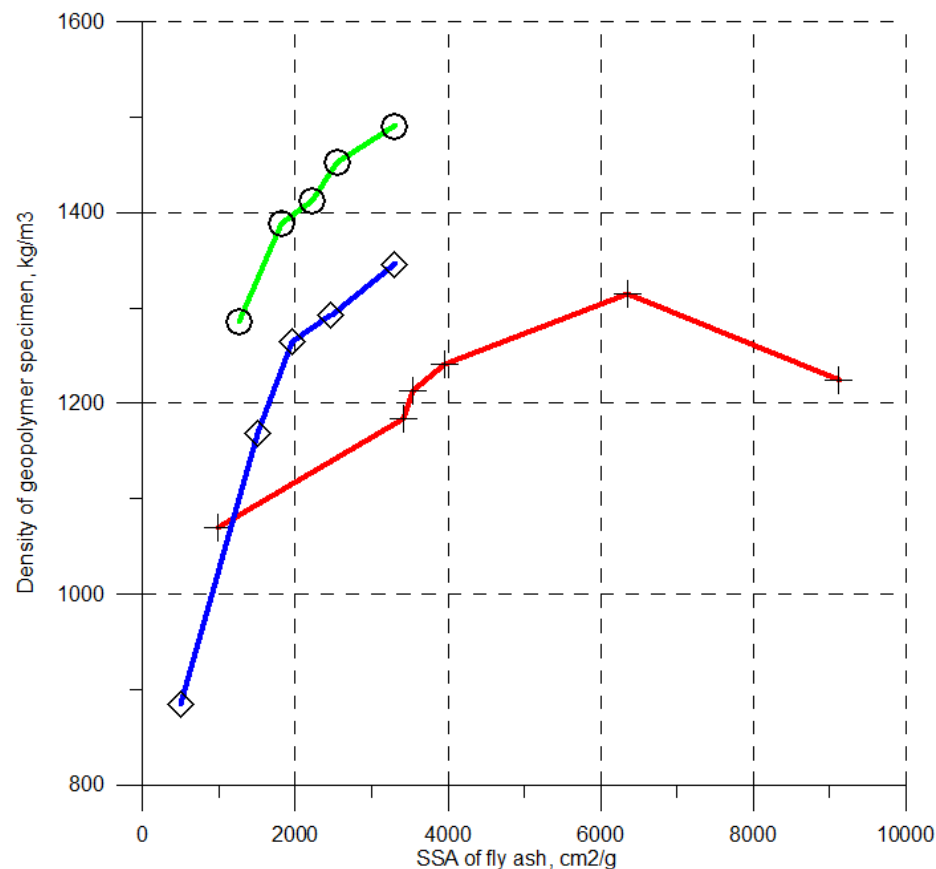
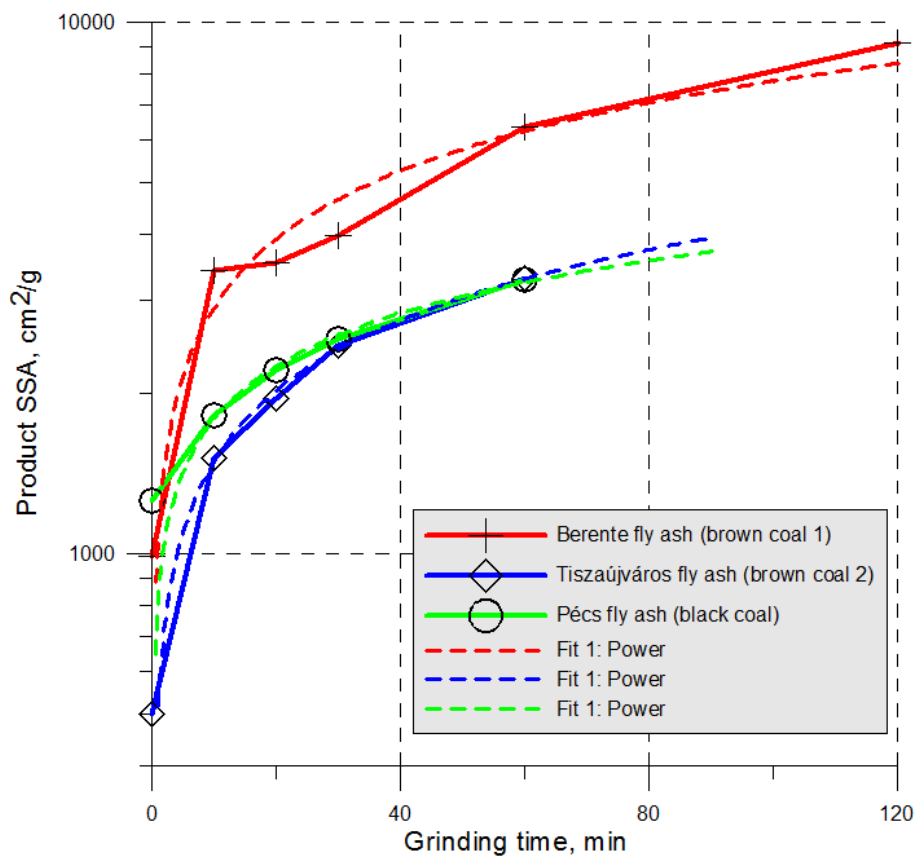
Higher specific surface area does not indicate obviously higher mechanical stability!

Optimal grinding fineness is different depending on fly ash type!

Compressive strength and specific surface area of fly ash as function of fly ash SSA

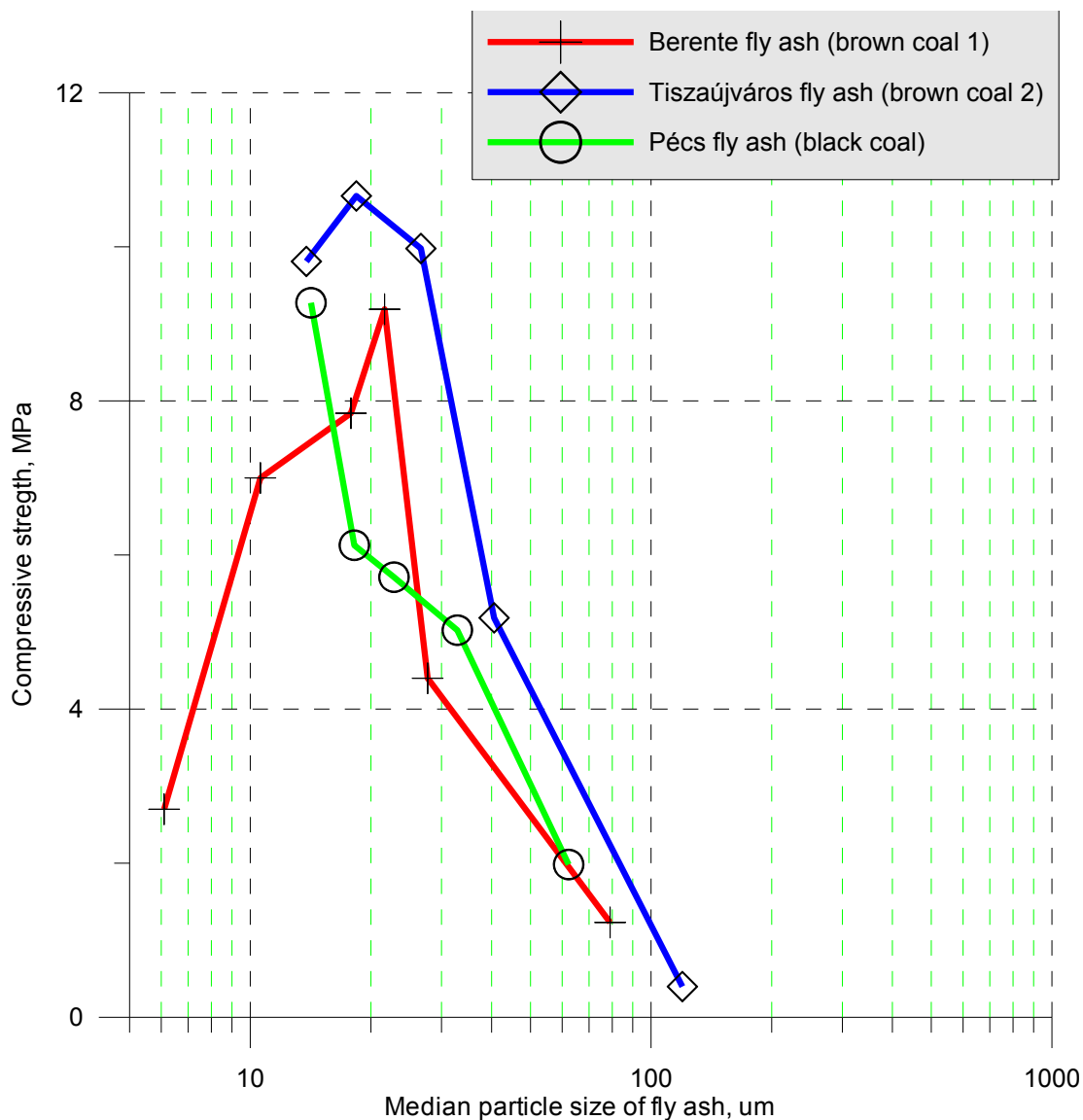


# Results





# Results



Optimal  
median size:  
- brown coal:  
~20  $\mu\text{m}$ ,  
- black coal:  
14  $\mu\text{m}$ .



# Conclusions

- The applied **deposited Class F fly ash** and spent liquor originated from alumina plant **can be used for geopolymer production.**
- The **mechanical activation** of the raw fly ash has a **positive effect on the geopolymerisation**, the fly ash fineness - **compressive strength** relation curve has maximum in case of brown coal fly ash. Black coal FA behave differently.



# Conclusions and future work

- The increasing of grinding time (finer material) resulted in **higher bulk density of geopolymer** specimens which means **denser microstructure and higher strength (until certain value)**.
- The geopolymerisation process using the above presented secondary raw materials **did not draw down volume change or crack generation** on the surface.
- Indo-Hungarian project: **Tailoring of geopolymer properties by mechanical activation** (Scientific leaders: Dr. Sanjay Kumar, Dr. Rakesh Kumar, Prof. Barnabás Csőke, Dr. Gábor Mucsi)



# Thank you for your attention!

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