



Proceedings of the
Fourth International

Slag Valorisation Symposium Zero Waste

15-17 April 2015
Leuven, Belgium

Editors Annelies Malfliet and Yiannis Pontikes

KU LEUVEN

BIOMASS ASH AS ALKALINE ACTIVATOR IN INORGANIC POLYMER SYNTHESIS

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Introduction

The research into inorganic polymers is driven by the CO₂ output of the cement industry, which accounts for 5-8 % of the total anthropogenic emissions.¹ With respect to CO₂ output, inorganic polymers have the benefit of a low temperature synthesis route. Traditionally, temperatures below 100 °C are used.² This is a huge benefit compared to the 1450 °C used for cement clinker production.³ In addition, a large amount of CO₂ is released during clinkering because of the limestone dissociation reaction. These facts constitute the synthesis of inorganic polymers a more environmentally friendly process compared to ordinary Portland cement production.⁴

The inorganic polymer can still be improved in terms of environmental impact. The largest impact originates from the alkaline activator, more specific the sodium (or potassium) silicate solution.⁴ Highly concentrated alkali silicate solutions are produced from cullet. Cullet is synthesized by melting a mixture of sodium carbonate and a silica source at temperatures above 1000 °C.⁵ This high temperature, together with CO₂ evolution from the reaction, leads to a high environmental impact. Therefore, research into inorganic polymers of lower footprint is leading towards alternative methods to produce silicate solutions, the possibility of using less silicate solution, or even the possibility of not using silicate solution at all. The present study investigates the latter option. To achieve this goal a natural alkali source is investigated. More specific, ashes from the combustion of maize, originating from agricultural waste. Plant ashes contain high levels of potassium and silicon.⁶ The use of biomass ashes adds industrial relevance to the research. Biomass combustion is becoming a common source for electricity production, because it is considered to be more environmentally friendly compared to the combustion of fossil fuels.⁷ However, the ashes produced have limited applications so far and are not fully valorised.

Material

Metakaolin from South Wales, UK was used as aluminosilicate source. The mineralogical composition is given in Table 1. Bottom ashes from the combustion of maize plants were investigated as activator. They have a high alkalinity and contain a high amount of potassium and silicon. The chemical composition is given in Table 2.

Table 1: Mineralogical composition of metakaolin determined by quantitative XRD on the non-calcined kaolin with the assumption that after calcination all the kaolinite is transformed (estimated relative error 5 %)

wt.%	metakaolinite	2:1 clays	K-feldspar	quartz
Metakaolin	68	21	8	3

Table 2: Chemical composition of maize ashes determined by semi-quantitative XRF (estimated relative error 10 %)

wt.%	SiO ₂	CaO	K ₂ O	P ₂ O ₅	Al ₂ O ₃	MgO	Fe ₂ O ₃	SO ₃	L.O.I.
Maize ash	18	1	32	4	1	2	< 1	1	45

Methods

Mortar samples with a mass ratio of CEN sand to metakaolin of 1.5 and solid (metakaolin + maize ash) to water of 3.2 were prepared using a Hobart mixer, pressed with a uniaxial pressure of 59 MPa and cured at 80 °C for 2 days. Different ash to metakaolin ratios were tested. A Shenk Trebel was used for compressive strength testing after 7 days with a crosshead speed of 2 mm/min on a 15 x 15 x 15 mm³ sample size. The microstructure was investigated using a Philips XL30 FEG scanning electron microscope (SEM). To study the nature of the binder phase, attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy was performed using an Alpha-P of Bruker with a diamond crystal, supported by Opus software.

Results and discussion

After the curing period of 2 days at 80 °C, one sample of every batch was immersed in water for 72 h at 80 °C and was found insoluble. The results for the compressive strength, Figure 1, demonstrate that an ash to metakaolin weight ratio of 0.9 exhibits the highest strength, more specifically 26.7 MPa. This strength satisfies the

specifications for bricks that are exposed to severe weathering conditions according to ASTM standards C62 and C652.

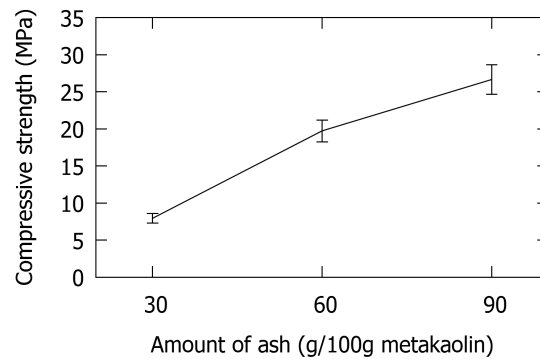


Figure 1: Compressive strength after 7 days of maize ash activated, metakaolin based inorganic polymers

The SEM images of the strongest sample, Figure 2, show a homogeneous binder phase. The relatively low strength in comparison with other inorganic polymers can be explained by the low strength of undissolved metakaolin particles and/or the presence of cracks.² A modification of the curing conditions is expected to address the latter issue.

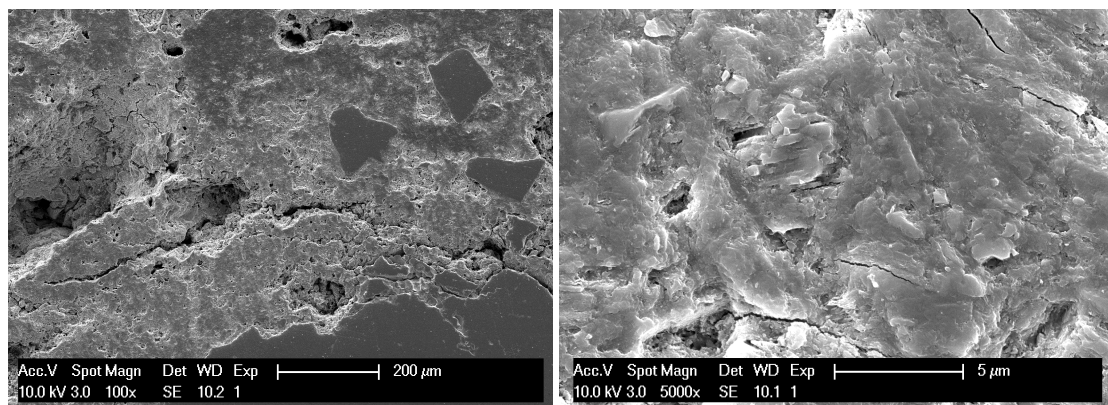


Figure 2: SEM images of maize ash activated, metakaolin based inorganic polymer with an ash to metakaolin ratio of 0.9

The ATR-FTIR spectra, Figure 3, show a shift of the Si-O and Al-O stretching band (1035-983 cm^{-1}) to lower wavenumbers after polymerization. This is commonly observed in geopolymer synthesis.^{8,9} The samples for which a more extensive shift in wavenumber is observed, underwent the geopolymerization reactions in a greater extent and obtained a higher compressive strength.

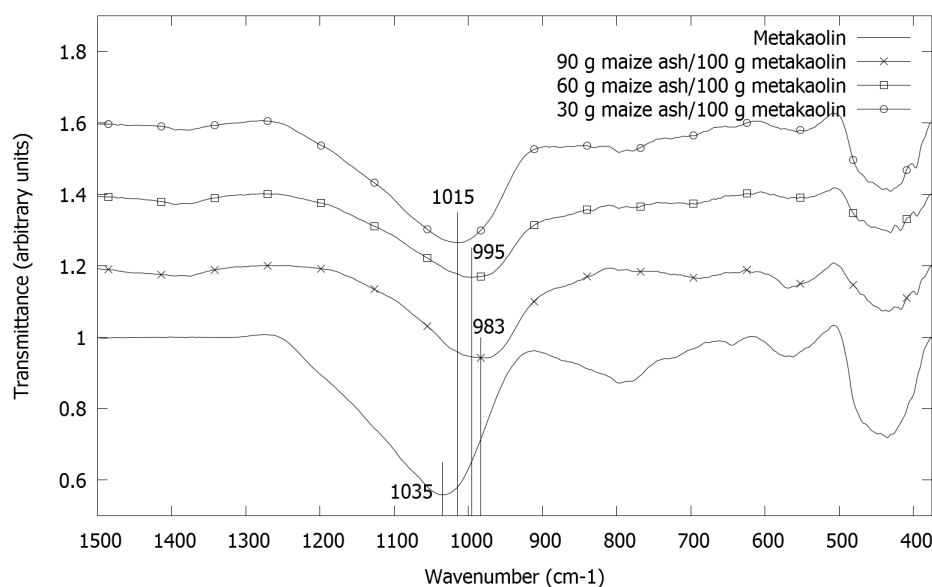


Figure 3: ATR-FTIR spectrum of maize ash activated, metakaolin based inorganic polymer

Conclusion

An inorganic polymer was synthesized using maize ashes as activator and metakaolin. No additional alkaline solution was used during synthesis, only water. This ensures a low cost and low environmental impact. The compressive strength was 26.7 MPa for pressed samples with weight ratios 0.9 maize ash/metakaolin, 1.5 sand/metakaolin and 3.2 solid (maize ash + metakaolin)/water, after 7 days of which 2 days curing at 80 °C. The obtained structure of the binder, investigated by ATR-FTIR, is comparable with common metakaolin based geopolymers. A shift in wavelength of the Si-O and Al-O stretching band is observed and related to the reaction extent. Higher reaction extents resulted in higher compressive strengths.

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