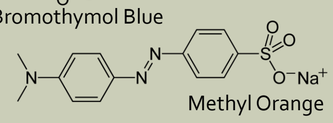
Università degli Studi della Campania *Luigi Vanvitelli*

## Convegno Nazionale Italiano sui Geopolimeri
## Bari, 16 and 17 February 2023

UNIMORE – Università degli Studi di Modena e Reggio Emilia – Dipartimento di Ingegneria "Enzo Ferrari"

# Synthesis and Characterization of Coloured Metakaolin-Based Geopolymers

Bromothymol Blue



Methyl Orange

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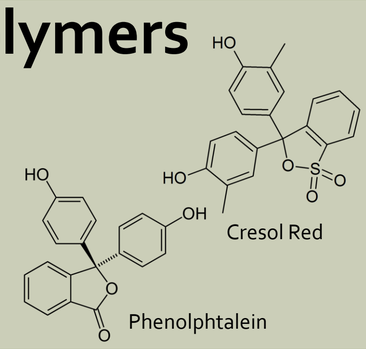


Cresol Red

Phenolphtalein

## Objective

Over time, several coloured materials have been employed in buildings, paintings, ceramics, and mosaic restoration. In recent years, geopolymers, due to their high chemical and mechanical resistance, have attracted great attention to be used in many areas, including restoration. In this work, coloured geopolymers are realized starting from white metakaolin (MK) paste and pH indicators, such as bromothymol blue (B), cresol red (C), phenolphthalein (P), and methyl orange (M), as dyeing agents. The geopolymers (GP), cured at 25 (labelled 1) and 40°C (labelled 2) are chemically analysed at different ageing times (from 7 to 56 days) through the Ionic Conductivity and pH measurements and FT-IR. Eventually, the colour hues are assessed in the CIELAB colour space before and after immersion in water.

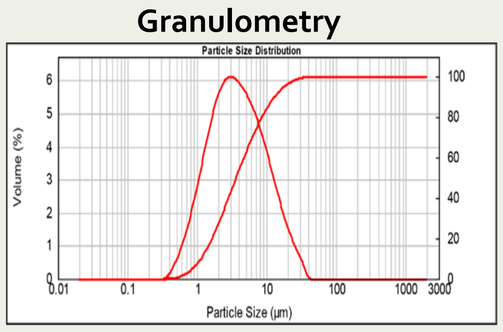
## Metakaolin characterization


MK

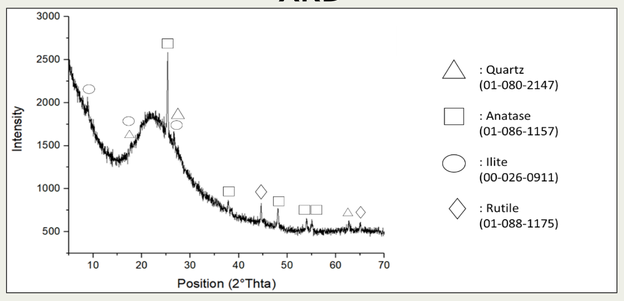
### XRF

| Chemical oxide composition (wt%) | $SiO_2$ | $Al_2O_3$ | $TiO_2$ | $Na_2O$ | Other oxides |
|---|---|---|---|---|---|
| MK | 53 | 40.5 | 5 | - | 1.5 |

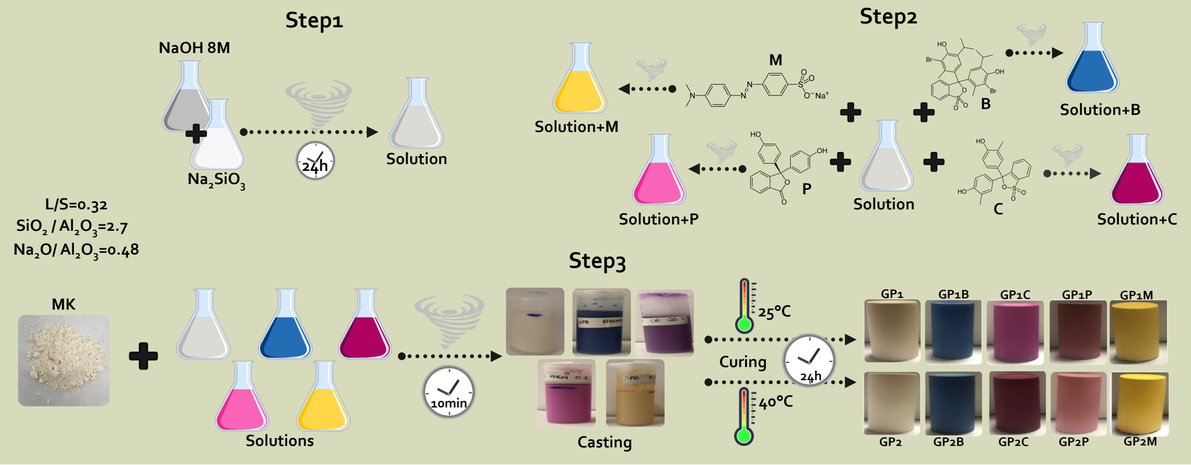
### Granulometry




### XRD

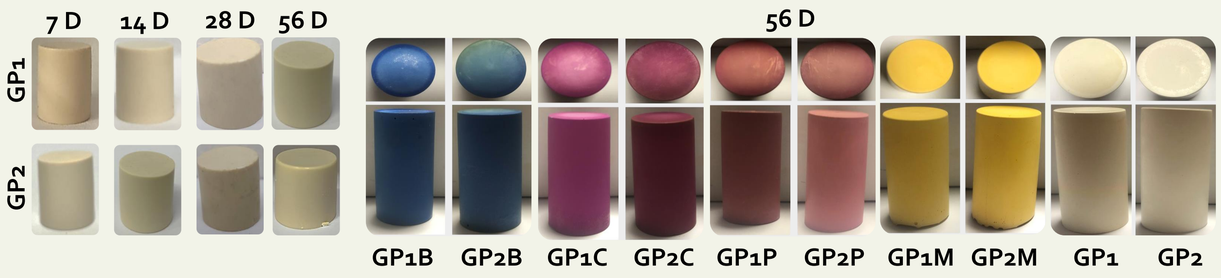



## Geopolymer syntheses




# Results

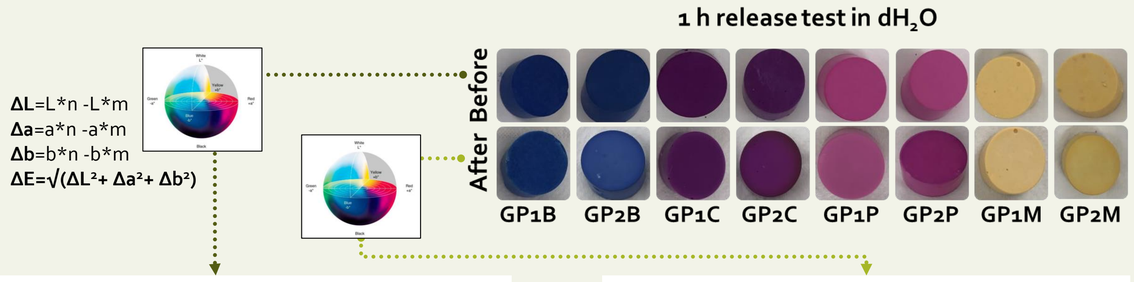
## Geopolymer characterization




- White/milky colour
- No bubbles
- Finger pressure resistant.

- Curing at 25 and 40°C affects the colour hues of the geopolymers;
- Few bubbles;
- Finger pressure resistant;
- Slight surface acidifcation of GP2B due to the carbonation phenomenon.

## Release study and CIELAB analysis



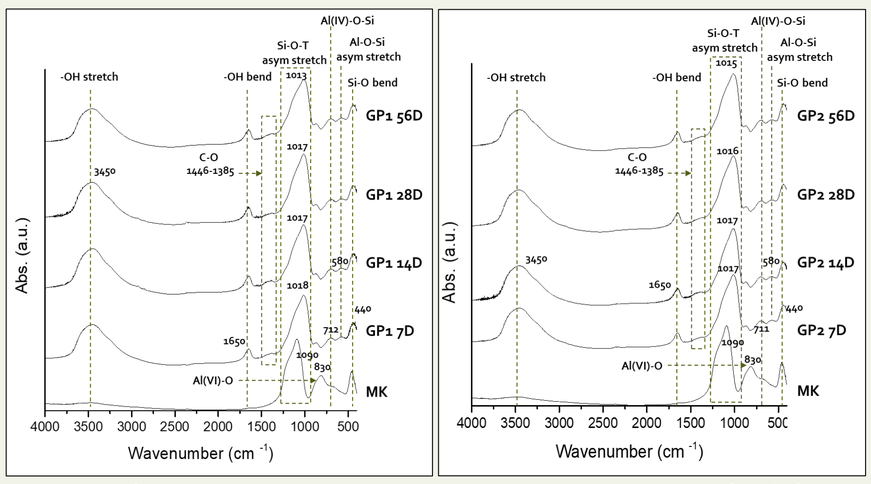

CIELAB Colour Space before release test

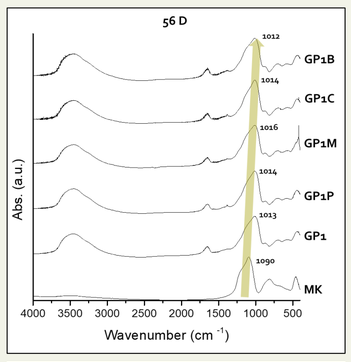
| Sample | ΔE | Sample | ΔE |
|---|---|---|---|
| GP1 | 0 | GP2 | 0 |
| GP1B | 44.55 | GP2B | 41.31 |
| GP1C | 47.46 | GP2C | 42.37 |
| GP1P | 49.32 | GP2P | 64.71 |
| GP1M | 57.89 | GP2M | 56.44 |

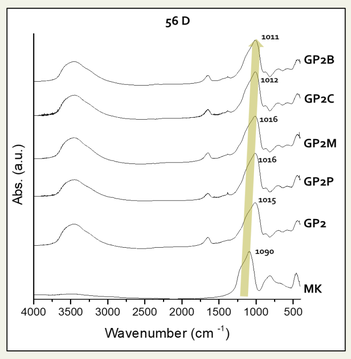
CIELAB Colour Space after release test

| Sample | ΔE | Sample | ΔE |
|---|---|---|---|
| GP1 | 0 | GP2 | 0 |
| GP1B | 61.44 | GP2B | 57.47 |
| GP1C | 67.37 | GP2C | 59.30 |
| GP1P | 70.09 | GP2P | 52.54 |
| GP1M | 48.14 | GP2M | 48.27 |

## FT-IR analysis

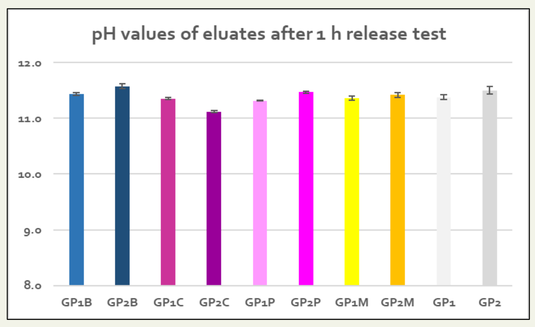


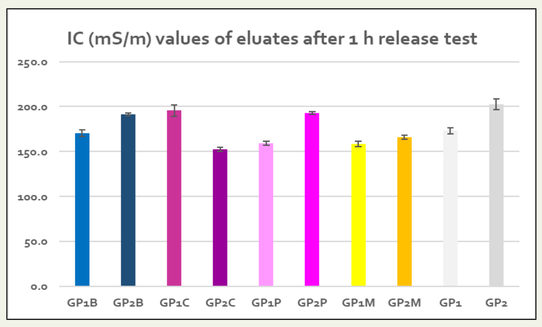



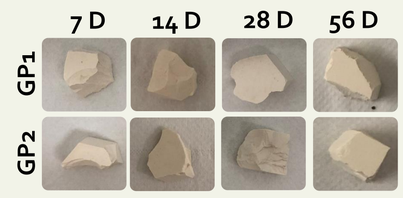

- Geopolymersization occurence ad Si-O-T (T=Si or Al) shifts at lower wavenumber for all the geopolymer samples;
- Carbonation phenomenon occurrence (presence of C-O vibration modes);
- Presence of pH indicators does not affect the geopolymerizations.

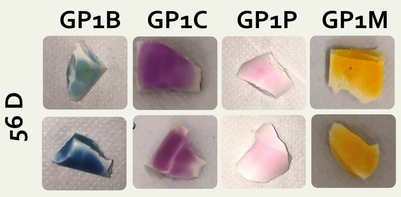
## pH and IC measurements

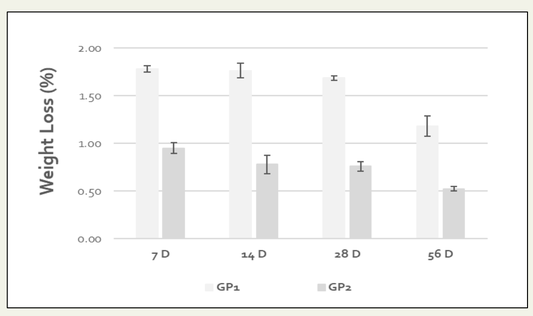


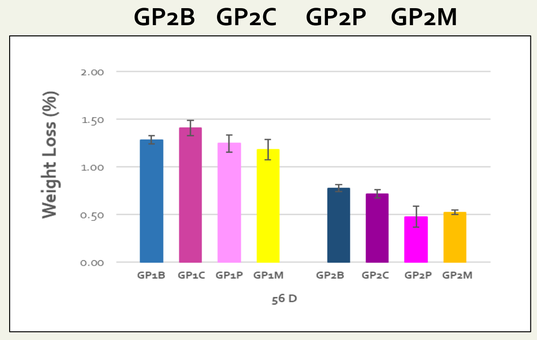




## Weight Loss Test

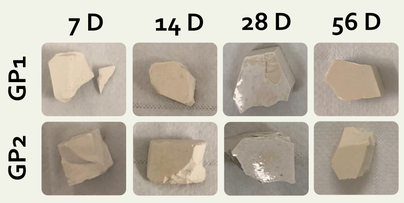


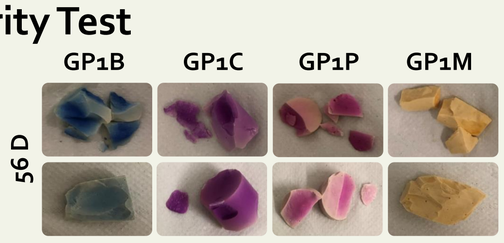




## Integrity Test

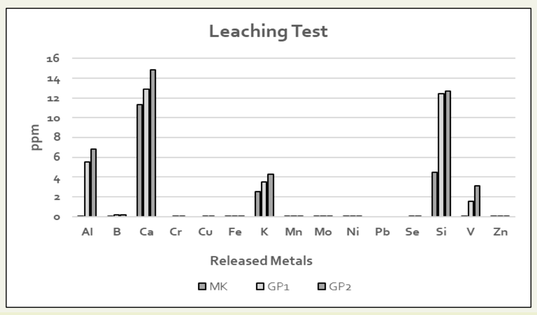



- Water clear after 24 h of test;
- Only GP1 at 7 D did not pass the test;
- pH ranging from 9.0 to 10.5.




- Colour release during 24 h of test;
- No integrity for all the GP1 series;
- pH ranging from 9.5 to 10.0;
- GP2B and GP2M were not broken after the test.

## Leaching




- Curing at 25 and 40°C affects the ionic metal release;
- Increase of Ca, Si, Al, K and V release after geopolymerization occurrence.

## Conclusions

- The investigated geopolymers formulations showed good compatibility with organic dyes;
- No significant changes in microstructural 3D networks, as indicated by the vibrational spectroscopy, as well as the chemical stability, as indicated by the release of unreacted alkaline media influencing the pH and ionic conductivity of the leachate water;
- Colours did not visibly suffer from curing at 40 °C, except for the case of phenolphthalein, which naturally decomposes at pH higher than 10 to the colourless form;
- Colours were retained after immersion in water for a short time.

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