

# Preparation and Characterization of Materials Obtained from NMP and Brick Wastes

/JULIO JAVIER VARA RODRÍGUEZ (1,2\*), ASCENSIÓN MURCIEGO MURCIEGO (1), NATIVIDAD ANTÓN IGLESIAS (2), JUAN DIEGO FERNÁNDEZ SILVA (2). ESTHER ÁLVAREZ AYUSO (3)

(1) Área de Cristalografía y Mineralogía. Escuela Politécnica Superior de Zamora (USAL). Avda. Cardenal Cisneros, 34. 49022 Zamora (Spain)

(2) Área de Ciencia de Materiales e Ingeniería Metalúrgica. Escuela Politécnica Superior de Zamora. Universidad de Salamanca. Avda. Cardenal Cisneros, 34. 49022 Zamora (Spain)

(3) Department of Environmental Geochemistry. IRNASA (CSIC). Cordel de Merinas 40-52. 37008 Salamanca (Spain)

## INTRODUCTION

Nowadays wastes from industrial, construction and demolition activities, represent an important environmental problem and the recycling of them should be a priority not only because of environmental problems such as pollution of soil, water and air around areas, but also for better utilization of resources. The recycling rate in the case of brick wastes is around 70-85%, and must be free of any kind of impurity. At present, brick wastes are mostly used as part of mortar and concrete mixes (Kesegic et al., 2008). Meanwhile, salt slag (a typical residue produced by the secondary aluminum industry and generated during aluminum scrap/dross melting) is a conglomerate of crystallized salt, metal beads and non-metallic product (NMP). These latter are composed mainly of  $\alpha$ - $\text{Al}_2\text{O}_3$ ,  $\text{MgAl}_2\text{O}_4$ ,  $\alpha$ - $\text{Al}(\text{OH})_3$  and impurities ( $\text{SiO}_2$ ,  $\text{AlN}$ , etc.) (Gil, 2005). Construction and demolition waste are mostly considered non-hazardous, while salt slag not have that qualification.

The purpose of this work is the preparation and the chemical, mineralogical and microstructural characterization of green and sintered aggregates obtained from NMP and brick wastes, in order to investigate their possibilities of recycling for production of several useful commercial products.

## MATERIALS AND METHODS

Green aggregates from NMP (75%wt.) and brick (25%wt.) mixtures were made for this study. Bricks come from building wastes and NMP was supplied by Befesa (Valladolid). Firstly, the process to produce aggregates involves crushing, grinding, and sieving raw materials to obtain fine size particles ( $<63 \mu\text{m}$ ).

These powders were then mixed with distilled water and rice starch as a binder, giving to green aggregates the necessary consistency for handling. Afterwards, cylindrical Teflon mold was filled with the mixture and brought to a drying oven to remove the mixing water and to consolidate the piece. Green aggregates were then sintered at  $1300^\circ\text{C}$  in a muffle furnace. Green and sintered aggregates (GA and SA, respectively) were weighed and measured in order to calculate their volume and density. A portion of each aggregate was finely ground to be analysed by X-ray diffraction using a Bruker D8-Advance diffractometer at University of Salamanca. Polished microslides of GA and SA were obtained and studied by polarizing microscopy (PM).

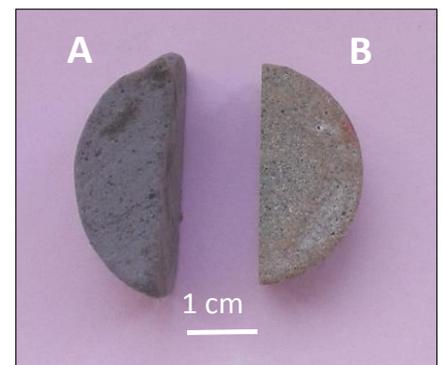


fig 1. Green (A) and sintered (B) aggregates.

Selected areas by PM were analysed using an electron probe microanalyzer (EMPA) JEOL SuperProbe JXA-8900M at the CNME (Madrid). The analysed elements were O, Mn, K, Si, Na, Fe, Ca, S, Mg, Al, P, F, Ti, Cu, Cr and Cl.

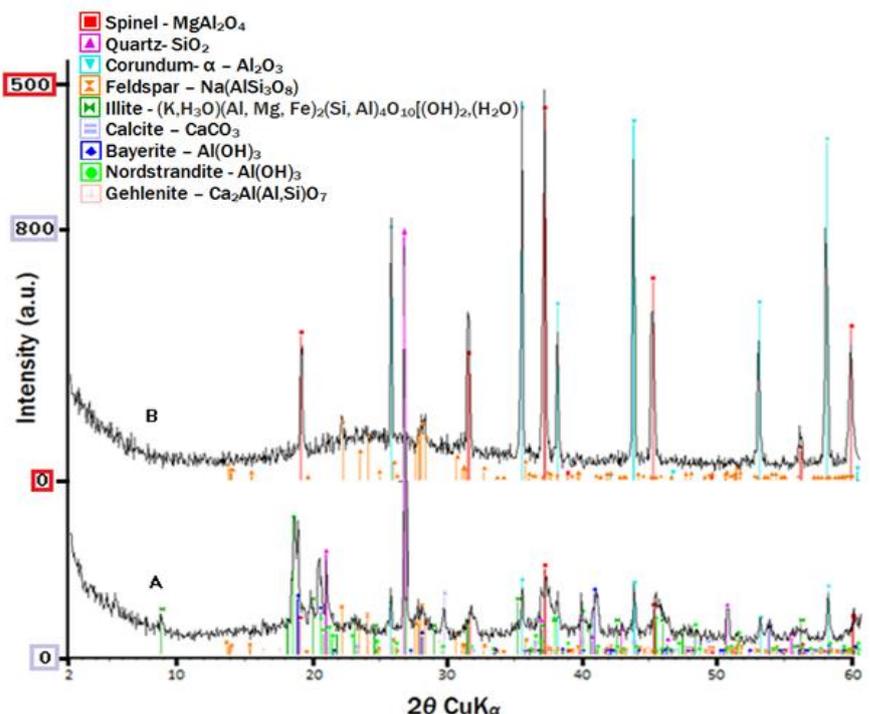


fig 2. X-ray diffraction patterns of green (A) and sintered (B) aggregates.

## RESULTS AND DISCUSSION

### Volume and density

The average values of volume and density were 7.11 cm<sup>3</sup> and 1.12 g/cm<sup>3</sup>, respectively, for GA, and 4.07 cm<sup>3</sup> and 1.69 g/cm<sup>3</sup> for SA. Shrinkage and densification of the sintered aggregate show the existence of a liquid phase during the sintering process. This also involves changes in color and texture of the samples (Fig. 1).

### X-ray diffraction

Identified crystalline phases in GA were (Fig. 2A): quartz, the most abundant, feldspar, illite, spinel, corundum, calcite, bayerite, nordstrandite and gehlenite. In SA (Fig. 2B) only three crystalline phases appear: spinel, corundum and, in minor amount, feldspar. The number of phases has decreased strongly with the increasing temperature and their degree of crystallinity is very high. Bayerite and nordstrandite disappear, turning to  $\alpha$ -alumina, produced from the dehydroxylation of these. The background hump between 15° and 30° in the X-ray diffractogram provides additional evidence of the presence of an amorphous phase (probably SiO<sub>2</sub>).

### Polarizing microscopy

Quartz, calcite, feldspar, phyllosilicates, corundum and spinel were identified in GA but none could be recognized in SA. In both samples some opaque phases were found.

### Electron microprobe analysis

Backscattered electron images (BSE, Fig. 3) show that in GA there is a conglomerate of particles physically attached. However in SA the microstructure has changed totally, with a homogeneous distribution of phases due to the diffusion process. The interconnected porosity may be related to the production of gases during sintering. Rounded holes, probably due to pull out during polishing, are frequent (Fig.4). Forty five punctual analyses carried out on different grains in GA (Fig. 3A) revealed the presence of quartz, calcite, Al hydroxides, K feldspars, Al-Mg and Al-Mg-Si oxides (spinel phase) phyllosilicates, corundum, and metallic phases (alloys) of different chemical composition: some consisting of Al (53-60%wt.)-Fe(25-37%wt.)-Si(6-18%wt.) with Cr, Mn and Cu up to 2, 3 and 4%, respectively, and others constituted by Fe(74%)-Al(20%)-Si(4%) and Al(44%)-

Fe(25%)-Mn(15%)-Si(13%), both with traces of Cu and Cr.

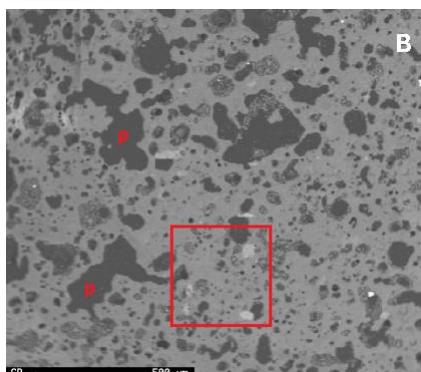
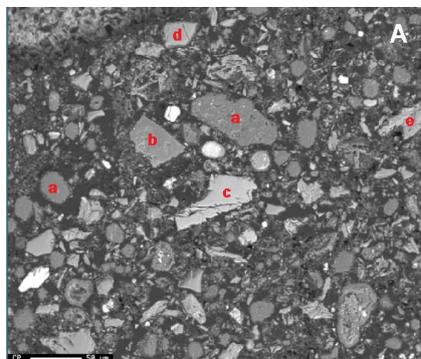


fig 3. BSE images of green (A) and sintered (B) aggregates. a: corundum, b: bayerite/nordstrandite, c: K-feldspar, d: quartz, e: phyllosilicates, p: pore. Red square is showed in fig.4.

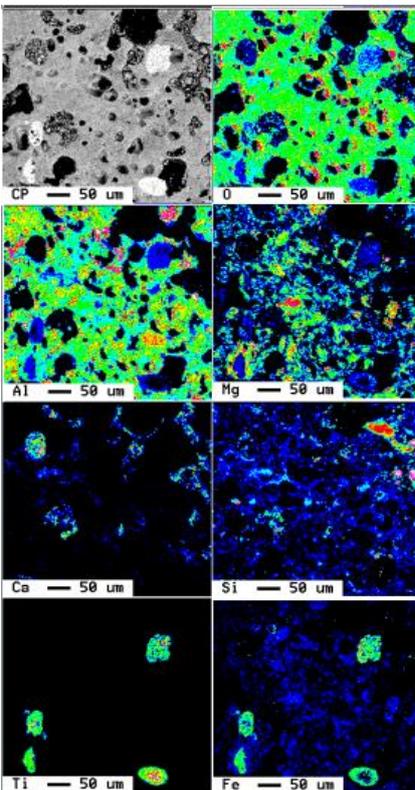


fig 4. BSE image (CP) and WDS X-ray maps showing the distribution of different elements. Color scale indicates the relative number of X-ray counts per pixel (low: blue, med: green-yellow, high: red-pink).

Thirty punctual analyses were carried out on grey and white tonalities in BSE images of SA (Fig. 3B and 4). The first ones corresponding to Al-Si-Mg-O phases (Al<sub>2</sub>O<sub>3</sub>: 47-65, SiO<sub>2</sub>: 22-29 and MgO: 5-14%wt.), the most abundant, and the second ones, to Ti-Al-Fe-Mg-O phases (TiO<sub>2</sub>: 35-40, Al<sub>2</sub>O<sub>3</sub>: 20-34, FeO: 11-17 and MgO: 4.5-8%wt.), corresponding probably to spinel phases (Hashishin et al. 2004, Mohajer et al., 2014). Feldspars of variable composition (CaO up to 8, Na<sub>2</sub>O up to 4 and K<sub>2</sub>O and MgO up to 2%wt.) and SiO<sub>2</sub> phases were present in minor quantity. Corundum grains were also identified. Metallic phases of Fe (65-69%wt.)-Si (23-33%wt.), Fe(36)-Si(35)-Mn(27%wt.) and Fe(65)-Si(6)-Cu(12)-Cr(8%wt.) appeared in minor amount. Elemental distribution showed in X-ray map (Fig. 4) reveals the diffusion process related to sintering. Zones with the highest Si contents seem to correspond to SiC used in microslide fabrication.

## CONCLUSIONS

The reuse of NMP and bricks will reduce the cost of disposal and will also lead to less environmental problems. Furthermore, the SA could be used in a variety of applications, such as the production of cements or in refractory applications. The presence of spinel and corundum in sintered aggregate is important because they are thermally stable, and it allows the use at high temperatures. The determination of its mechanical properties will allow to propose other potential uses.

## ACKNOWLEDGEMENTS

Special thanks to Francisca Sanz González, José Manuel Compañía Prieto and Alfredo Fernández Larios.

## REFERENCES

- Gil A. (2005): *Management of the Salt Cake from Secondary Aluminum Fusion Processes*. *Ind. Eng. Chem.*, **44**, 8852-8857.
- Hashishin T., Kodera Y., Yamamoto T., Ohyanagi M., Munir Z. (2004): *Synthesis of (Mg,Si)Al<sub>2</sub>O<sub>4</sub> Spinel from Aluminum Dross*. *Communications of the American Ceramic Society*, **87** [3], 496-499
- Kesegic I., Netinger I., Bjegovic D. (2008): *Recycled Clay Brick as an Aggregate for concrete: overview*. *Technical Gazette*, **15** [3], 35-40.
- Mohajer A.S., Ghanbarnezhad S., Sharifi L., Mirhosseini H., Irvani Y.M. (2014): *The Effect of TiO<sub>2</sub> on Sinterability and the Formation of Spinel Phases on MgO - Al<sub>2</sub>O<sub>3</sub> - TiO<sub>2</sub> Systems*. *International Journal of Research Science & Management*, **1** [1], 13-20.