DESIGN OF STRUCTURED ADSORBENTS FOR APLICATIONS IN GAS ADSORPTION PROCESSES – CONVENTIONAL SHAPING *vs* 3D-PRINTED FORMULATION

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Microporous materials highly activated and with potential to be used as adsorbents in many applications for gas separation/purification are usually available as powders. These solids usually have a great and reversible gas uptake, high gas selectivity, good chemical and thermal stability, but are unsuitable to be used in gas adsorption processes, such as Pressure Swing Adsorption (PSA) or Simulated Moving Bed (SMB).
Zeolites, carbons and more recently metal-organic frameworks (MOFs) are examples of those materials. Their use in adsorption-based processes are dependent of their upgrading from powders (micrometer scale) to particles (pellets, spheres or granules at millimeter scale). This would overcome large pressure drops and consequent energy consumptions when packing adsorbent columns in those processes. Thus, shaping adsorbents is an important step to use them in industry, although it greatly affects their capacity and selectivity towards a specific gas separation.
In this work, we explore techniques to shape powdered adsorbents, followed by their textural and mechanical characterizations, and the study of their adsorption properties towards the main components of post-combustion flues gases (CO_2 and N_2). Materials densification is proposed by employing two approaches:

- Conventional shaping through binderless mechanical compression and binder-containing extrusion; and
- Formulation by 3D printing (or additive manufacturing) to produce packed bed morphologies that precisely replicate computer aided design (CAD) models.

Porous separation media are important for fluid-solid contacting in many unit operations, including adsorption. Due to practical limitations, media particles are typically packed randomly into a column in a shaped form, allowing fluid to flow through the interstitial voids. Key to the effectiveness of packed columns are the flow-related properties of mass transfer, fluid distribution and dispersion, and back pressure, which in turn depend upon packing geometry. Until now, no alternative was found to overcome this limitation and have optimal ordered packing arrangements at the micron scale. 3D-Printing (or additive manufacturing) brings a wide range of benefits that traditional methods of manufacturing or prototyping simply cannot. With this approach, complex ordered geometries, that are not possible by conventional extrusion, can be designed and printed for a porous media, being the equipment resolution the only limiting step to overcome.
The effect of parameters like compression force, particle sieving, binder nature, binder/adsorbent ratio were firstly studied using conventional shaping techniques, as a basis for the consequent development of 3D-printed formulations. The structured samples are then characterized and adsorption equilibria studies are performed on them to evaluate their performance as media for gas adsorption separation processes. A volumetric/manometric adsorption unit built in-house was used for this purpose. Relevant experimental data is obtained, which allows to conclude that 3D-printed media can be an alternative porous media for application in gas adsorption processes.

Figure 1 - Formulations of: a) binder-containing extruded pellets, b) binderless compressed granules and c) 3D-printed structures