Enhancing the carbonization efficiency of photopolymer-based monoliths

N. Corrochano*[1], M.I. Pariente[1], Y. Segura[1], R. Molina[1], F. Martínez[1,2], J.L. Diaz de Tuesta[1]

[1] *Grupo de Ingeniería Química y Ambiental*

[2] *Instituto de Tecnologías para la sostenibilidad; ESCET, Universidad Rey Juan Carlos, c/Tulipán s/n 28933 Móstoles, España*

* *noelia.cguijarro@urjc.es*

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Carbon materials play a key role in various fields due to their properties and versatility, and are commonly used in powdered, pellet or bead form. 3D-carbon-based materials can overcome the disadvantages that other forms present, improving electrical conductivity, fluidization, among others [1]. Photopolymer monoliths (PMs), synthesized by stereolithography 3D-printing, can be transformed into carbon monoliths employing a well-studied carbonization process that allows to preserve their structure [2]. In this work, the carbonization of PMs is deeply studied and optimized through a design of experiments (DoE) to maximize carbon monolith yield and size, while achieving high BET specific surface area (S_{BET}). The PMs (prepared from a tailored resin [1-2]) were synthesized by a DLP 3D printer and subsequently carbonized using thermogravimetric analysis (TGA). A DoE for two factors (11 runs) was used to optimize the oxidation temperature (T_{OX} = 200–400 ºC) and heating rate (HR = 0.5–6.5 ºC/min) of the carbonization process, which involves two stages: 1) oxidation pretreatment with air at T_{OX} and 2) pyrolysis under N_2 flow up to 850 ºC. Yields at T_{OX} and 850 ºC were determined by TGA and a digital microscope was used to measure the monolith sizes (diameter and height) (Fig. 1 A-C). The data results were successfully fitted using a second order interaction model, providing R^2_{adj} > 0.9, and the results suggested that the optimal conditions to obtain high yields and sizes are at TOX = 400 ºC and HR = 0.5ºC/min (Fig. 1 D). In addition, "run 7" (T_{OX} = 400 ºC and HR = 3.5 ºC/min) has provided the highest S_{BET}, reaching 1175 m^2/g.

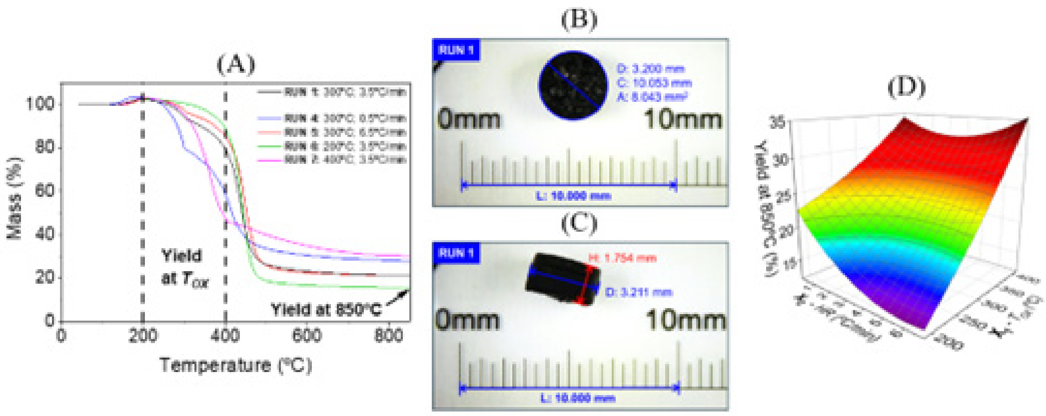

Figure 1. (A) TGA for CCD runs; (B) diameter and (C) height of carbon monolith for "run 1"; and (D) response surface of yield.

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