

3D porous carbon structures derived from whey: manufacturing processes and applications

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Objectives and novelty

Producing Porous Carbon Structures (PCS) with complex designs and customized morphologies is not a straightforward task. Over decades of research on porous carbons, significant progress has been made in controlling nanoscale parameters such as porosity and surface chemistry. However, at the macroscopic scale, porous carbons are still presented in traditional formats such as powders, granules or pellets. Monoliths are also a recurrent form for porous carbons, but usually shaped in very simple geometries.

This doctoral Thesis aims to address three main objectives. The first one is the development of PCS with tailored morphologies that overcome current design and production limitations. Moreover, these structures must exhibit sufficient consistency to allow handling, storage, and transportation. In this work, the goal is to achieve this purpose through simple methodologies, using sustainable precursors and employing straightforward technologies mainly based on Additive Manufacturing (AM). The second objective is to utilize the fabricated PCS in applications where their geometric design provides advantages over the use of simpler morphologies. The ability to potentially obtain PCS with any morphology opens up possibilities for their use in applications previously unconsidered, such as tissue engineering or process intensification in chemical engineering. Finally, the third objective is to contribute to the valorization of whey and, consequently, reduce the environmental impact of this dairy by-product, which is generated in large quantities and poses significant management challenges.

Results

Whey possesses a set of characteristics that enable the fabrication of structures with controlled morphologies, either through the sintering of whey powder or from aqueous pastes [1]. For this purpose, different methods can be used, including molding [2], machining [3], and additive manufacturing techniques, such as Selective Laser Sintering [4] and Direct Ink Writing [5] (Fig. 1). These structures can then be transformed into PCS through carbonization and activation processes. During thermal treatment, whey exhibits thermally stable behavior, ensuring that the final PCS retain the same morphology as the original whey pieces. The only deformation experienced during this process is an isotropic

shrinkage of a known magnitude, allowing for precise control over the exact dimensions of the final product (Fig. 2a).

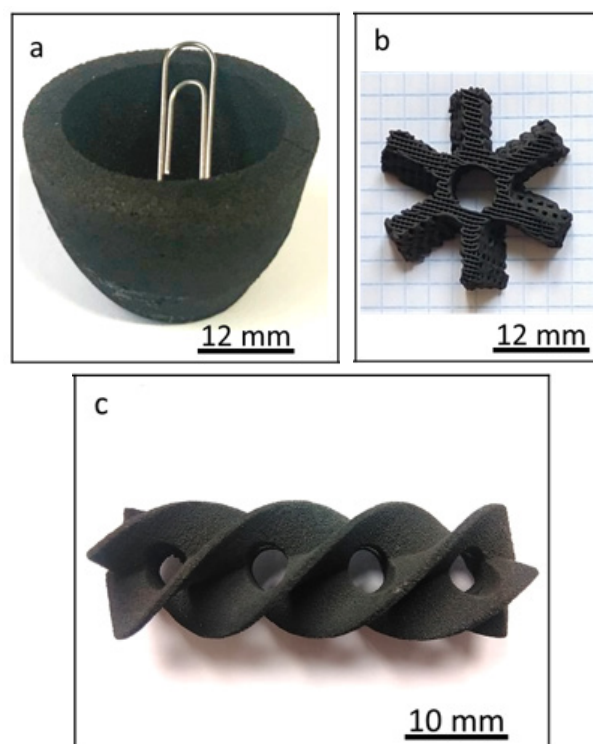


Figure 1. Examples of PCS obtained by casting and machining of whey powders (a), Direct Ink Writing (b) and Selective Laser Sintering (c).

PCS derived from whey are highly porous materials (ranging from 50% - 70% of porosity) with a hierarchical porosity that includes micropores, mesopores, and macropores (Fig. 2b), with BET surface areas up to 500 m²/g. This porosity can be increased by activation processes, obtaining as a result activated carbons with BET values higher than 1000 m²/g [6]. They also contain a significant amount of nitrogen in the range of 2% to 3%. Depending on the mineral content of the primary whey, the carbonized structures exhibit varying ash content, which can be easily reduced or removed through acid washing. However, their most notable property is the exceptionally high mechanical strength of the porous carbon components, as demonstrated by abrasion, flexural, and compression strength tests.

The freedom of design enables their use in innovative applications previously unexplored in the field of porous carbons. For instance, they can be employed in the development of bone scaffolds for tissue engineering [7]. In chemical engineering, they

can be applied to process intensification through the fabrication of catalytic agitators or continuous porous carbon reactors with optimized geometries [8,9].

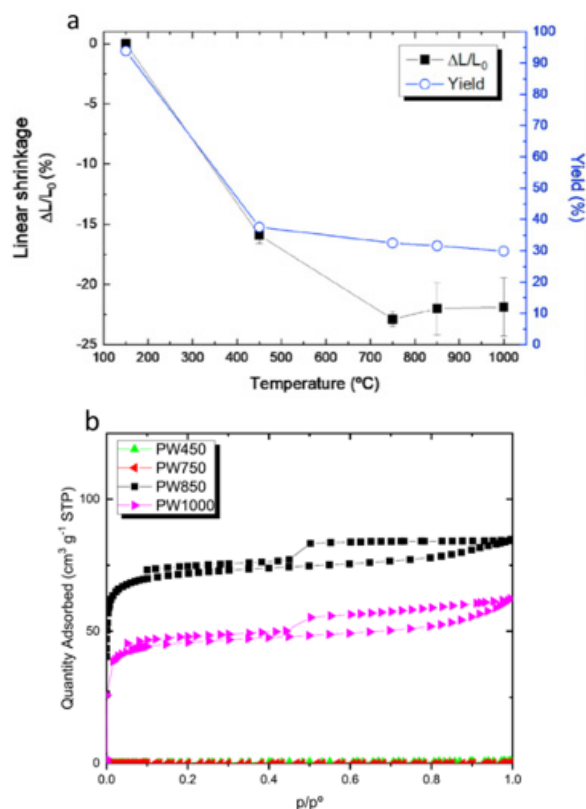


Figure 2. Shrinkage and yield (a) and Nitrogen isotherms (b) of PCS at different carbonization temperatures.

In addition to being used independently, whey can be combined with other powdered materials for the fabrication of composite structures, PCS or pellets [10]. This can be achieved via dry processing, by mixing powders followed by sintering, or via wet processing, by forming mixed aqueous pastes. Using the latter method, whey pastes can serve as binders for the production of activated carbon pellets. Specifically, the binder can be made directly from liquid whey. This provides a more sustainable and cost-effective alternative to traditional binders, mostly pitch and bentonite, as well as an opportunity for the industrial-scale valorization of liquid whey, processing thousands of tons.

Conclusions

- It is possible to obtain PCS with controlled morphologies using whey as a precursor. On one hand, its composition, primarily based on lactose and proteins, enables the formation of 3D whey structures through techniques such as molding or additive manufacturing, whether using whey powder or aqueous pastes. On the other hand, its thermally stable behavior allows pre-formed components to be carbonized without losing their shape, resulting in PCS with controlled and customized morphologies. Once fabricated, they can be easily machined, expanding the range of achievable geometries.

- Beyond morphological control, the most notable property of whey-based PCS is their high mechanical strength. Considering that they are porous materials, various mechanical tests (abrasion index, flexural strength, and compression strength) reveal greater consistency compared to other porous materials, including activated carbons, extruded cordierite, sintered hydroxyapatite, or trabecular bone. They also exhibit higher consistency than most carbons with similar porosities reported in the literature.
- The combination of whey with other powdered materials enables large-scale valorization of liquid whey, which poses significant environmental management challenges, offering a sustainable and cost-effective alternative to traditional binders such as pitch or bentonite.
- The ability to endow ECPs with complex, consistent morphologies—hard to obtain before the arrival of AM—allows their use in applications previously unexplored for porous carbons, such as the fabrication of bone scaffolds. Additionally, morphological control provides advantages in other applications, such as process intensification in chemical engineering through the development of catalytic agitators or the design of reactors with optimized geometries.

Related publications

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- [6] Llamas-Unzueta, R., Ramírez-Montoya, L. A., Menéndez, J. A., & Montes-Morán, M. A. (2023). Customised Microporous Carbon 3D Structures with Good Mechanical Properties and High Nitrogen Content Obtained from Whey Powders. *C, Journal of carbon research*, 9(4), 100. <https://doi.org/10.3390/c9040100>

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^[8] Llamas-Unzueta, R., Reguera-García, A., Sanz, I., Martín, C., Quintanilla, A., Menéndez, J. A., & Montes-Morán, M. A. (2024). 3D printed catalytic stirrers with permeable blades made of porous carbon. *Additive Manufacturing*, 104233. <https://doi.org/10.1016/j.addma.2024.104233>

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