



## Polysaccharide-derived mesoporous materials (Starbon<sup>®</sup>) for sustainable separation of complex mixtures

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### Supplementary material

#### Preparation of polysaccharide-derived mesoporous materials (A800) treated with by-products of a microwave assisted hydrothermal process of orange peel

One gram of A800 was added to a 10 mL solution prepared in acetone containing 0.05 g of resulting mixture of saccharides of rhamnose, galactose, glucose, GalA obtained from the low temperature hydrothermal microwave process of orange peel<sup>1,2</sup>, and stirred overnight at room temperature. After washing with acetone, the treated A800 carbonaceous material was dried at 40 °C overnight. Afterwards, 0.5 g of this material was subsequently carbonised at 300 °C at a heating rate of 1 °C min<sup>-1</sup> under an inert atmosphere. Both materials, dried at 40 °C (i.e. before pyrolysis) and the material subsequently pyrolysed at 300 °C, were used to prepare SPE cartridges, which were used to perform the extraction tests of the 10 selected bioactive phenolic compounds. The recovery results found for the all sorbents studied can be seen in Table 1.



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**Table 1.** Recovery (Mean and Relative Standard Deviation) of the selected phenolic compounds by SPE using C18, PGC and sustainable mesoporous carbonaceous materials as sorbents (S, A and PcB Starbon® series, including the novel treated A800 solid phases).

Sorbent	1. Umbelliferone	2. Daidzein	3. Naringenin	4. Catechin	5. Quercetin	6. Diadzin	7. Hesperidin	8. Naringin	9. Rutin	10. Isoquercitrin
PcB0	*	89.6 (3.5)	89.4 (2.8)	107.4 (4.9)	118.4 (4.0)	105.1 (5.3)	80.4 (10.5)	104.8 (6.3)	89.7 (2.7)	104.1 (0.6)
PcB300	34.3 (1.7)	85.1 (5.0)	60.8 (3.0)	0.0 (0.0)	0.0 (0.0)	42.7 (4.9)	52.5 (4.2)	97.7 (4.1)	3.1 (8.8)	0.0 (0.0)
S300	87.8 (0.7)	79.1 (2.7)	84.4 (3.7)	40.6 (0.3)	74.7 (7.4)	86.6 (7.8)	90.7 (4.2)	88.9 (3.6)	90.5 (3.4)	82.1 (3.2)
A300	96.0 (4.8)	81.1 (1.4)	87.8 (2.0)	65.8 (2.1)	10.6 (7.8)	86.7 (2.6)	95.3 (2.8)	80.2 (1.0)	87.9 (4.1)	84.5 (3.4)
PcB450	56.8 (7.5)	98.5 (4.3)	66.1 (14.0)	0.0 (0.0)	0.0 (0.0)	50.5 (13.9)	56.8 (13.9)	88.6 (10.8)	0.0 (0.0)	0.0 (0.0)
S450	9.6 (11.9)	55.0 (5.4)	60.5 (11.2)	0.0 (0.0)	0.0 (0.0)	64.1 (8.0)	70.5 (2.3)	75.7 (3.9)	54.7 (4.6)	62.3 (2.7)
A450	58.1 (7.1)	68.6 (7.8)	53.0 (7.6)	0.0 (0.0)	0.0 (0.0)	38.6 (10.6)	41.9 (4.9)	66.9 (7.4)	6.6 (5.9)	0.0 (0.0)
PcB600	8.5 (5.7)	73.0 (2.7)	51.6 (1.4)	0.0 (0.0)	0.0 (0.0)	27.6 (13.5)	61.9 (2.2)	68.4 (9.6)	0.0 (0.0)	0.0 (0.0)
S600	31.2 (12.3)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	15.7 (13.2)	88.7 (1.4)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)
A600	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	4.0 (16.3)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)
PcB800	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)
S800	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)
A800	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)
C18	98.4 (1.2)	97.5 (1.1)	96.9 (1.9)	92.9 (4.8)	76.3 (7.8)	103.7 (5.8)	101.7 (1.3)	100.3 (4.7)	101.8 (2.4)	100.1 (4.2)
PGC	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.7 (7.9)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	1.1 (13.0)	0.0 (0.0)	0.0 (0.0)
Treated A800	35.6 (9.2)	15.8(7.3)	53.1 (5.6)	0.0 (0.0)	0.0 (0.0)	40.6 (7.6)	88.6 (5.0)	10 (7.6)	0.0 (0.0)	0.0 (0.0)
Treated and heated A800	0.0 (0.0)	0.0 (0.0)	25.6 (10.1)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	74.3 (3.4)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)

\* Interference from PcB0 (method based on the addition of *t*-butanol to hydrogels before the freeze drying step).<sup>3</sup>

C18 and PGC SPE cartridges (0.1 g; 1mL) were obtained from Dionex Corporation (Sunnyvale, CA, USA) and Thermo Fisher Scientific Inc. (Waltham, MA USA).  
(n = 3)



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