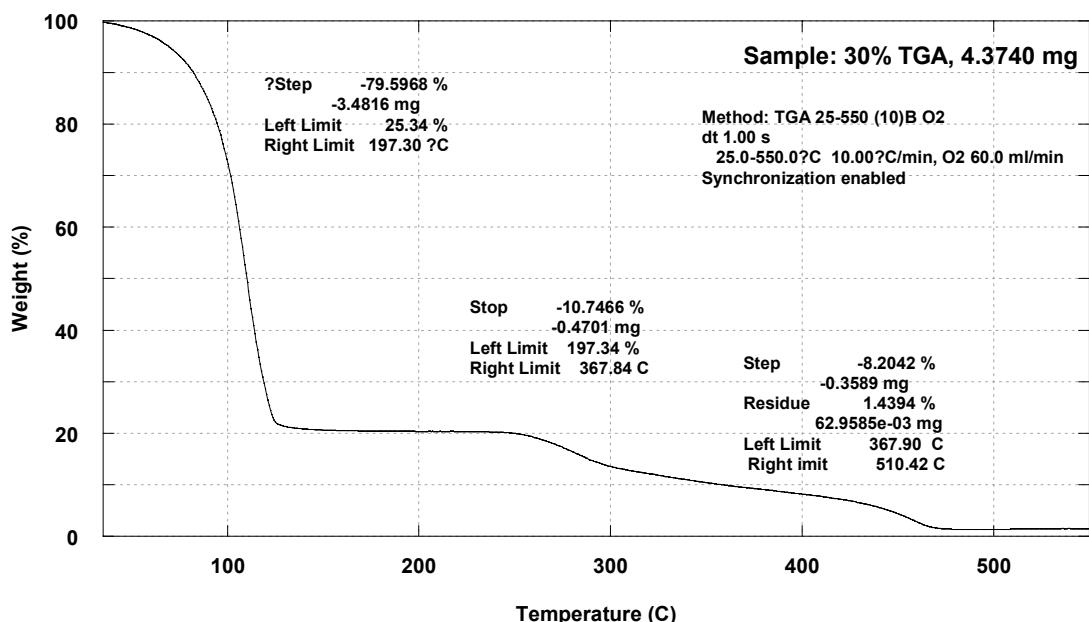


Supplementary Material (ESI) for Soft Matter  
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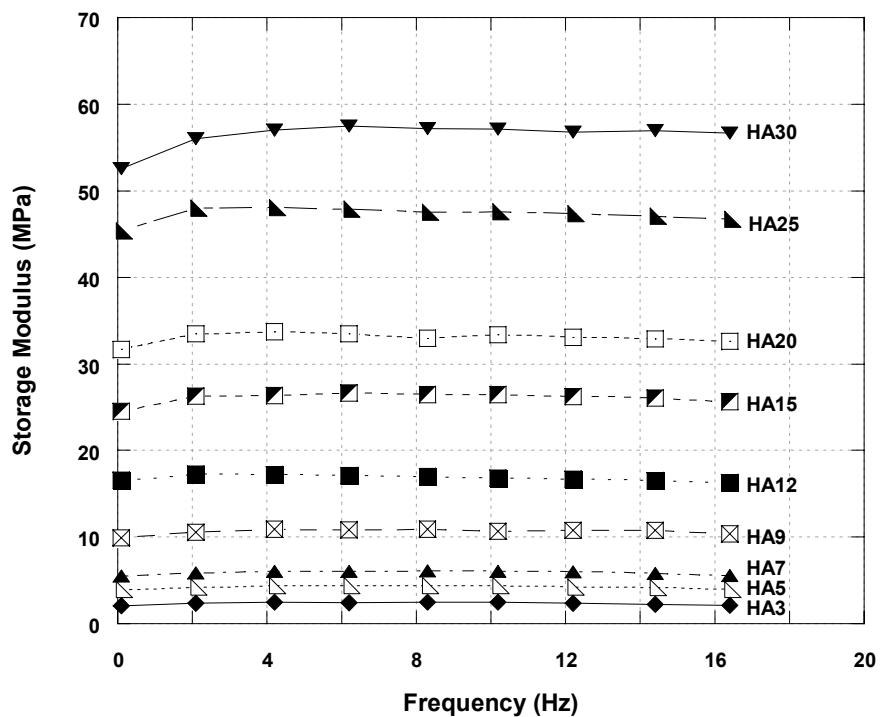
## **Supplementary Information**

### **High-concentration compact agar gels from hydrothermal synthesis**

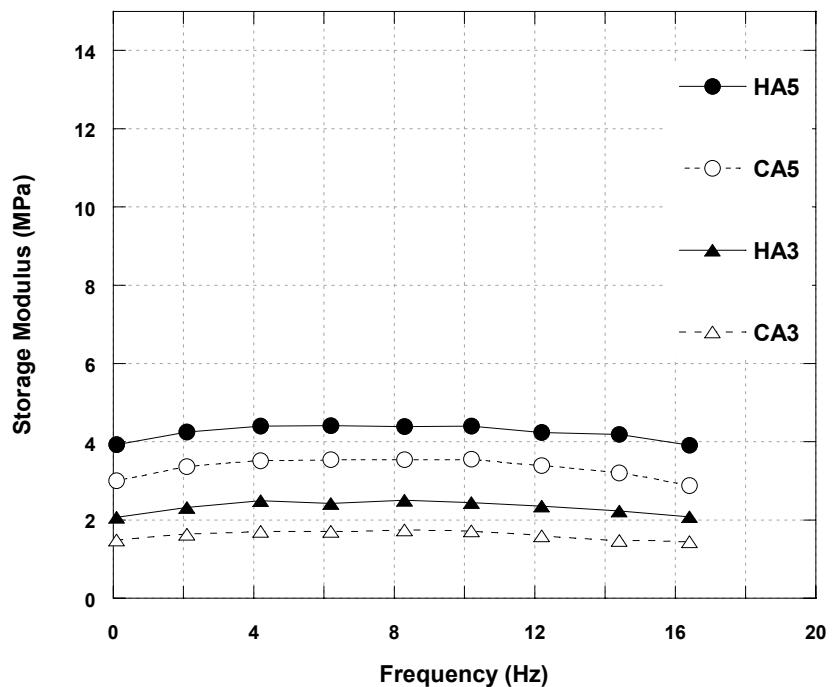
**Omar Ayyad,<sup>a\*</sup> David Muñoz-Rojas,<sup>a†</sup> Núria Agulló,<sup>b</sup> Salvador Borrós,<sup>b</sup> Pedro Gómez-Romero<sup>a\*</sup>**



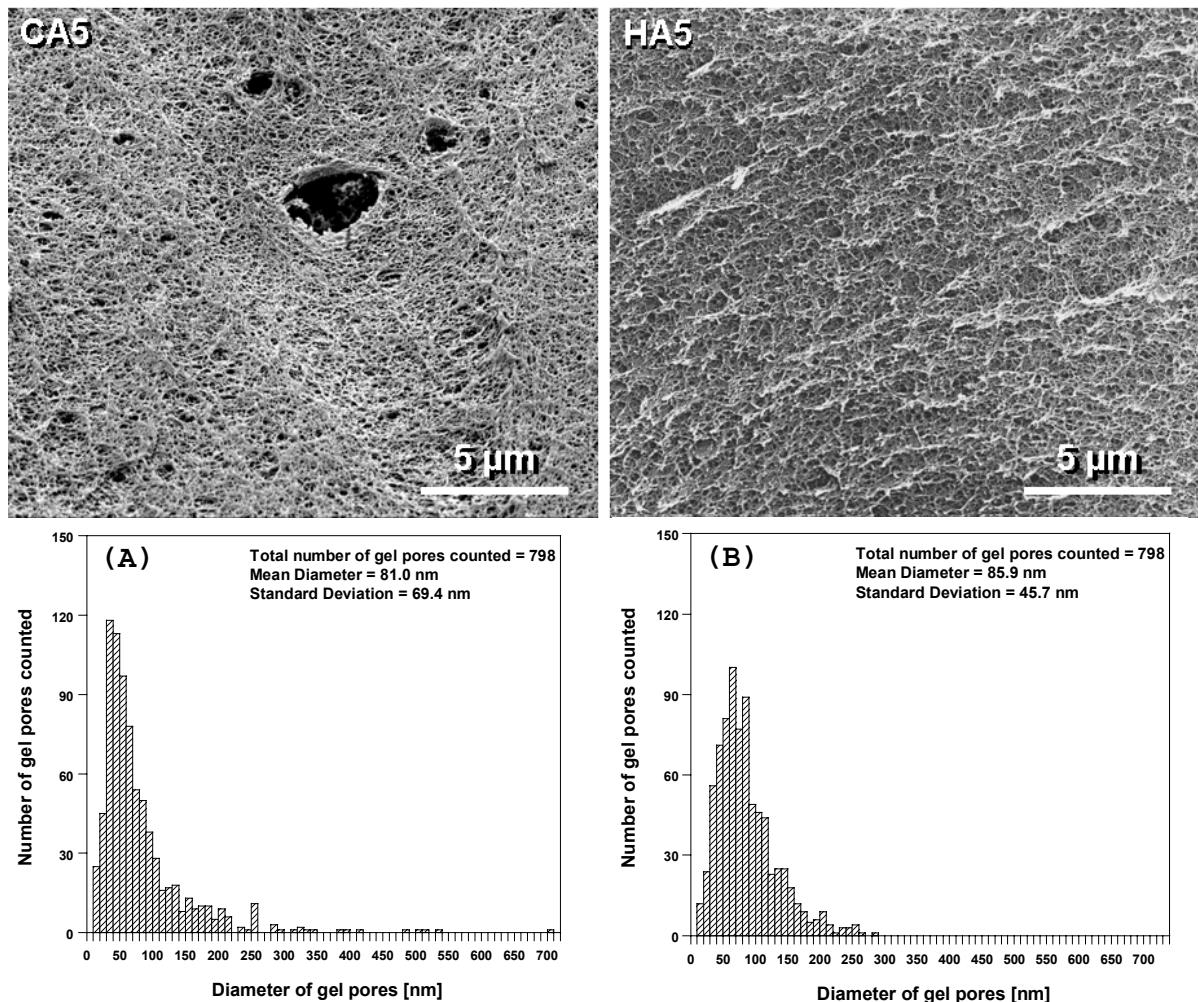
**Figure S1.** TGA curve of HA30 hydrogel.



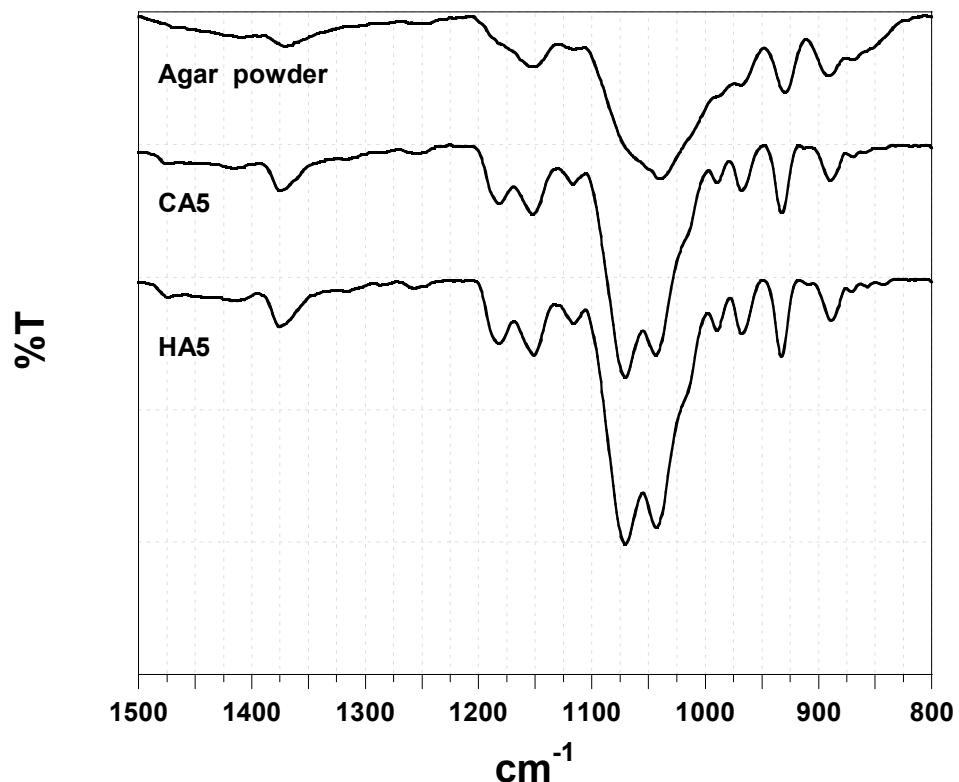
**Figure S2.** Plot of the Storage modulus as a function of frequency for the whole series of hydrothermal agar hydrogels (HA3 – HA30).



**Figure S3.** Storage modulus as a function of frequency for HA3 and HA5 hydrogels prepared hydrothermally with their corresponding hydrogels prepared by conventional procedure. CA3 and CA5, stand for agar hydrogels prepared conventionally by dispersing 3, and 5 g of agar powder, respectively, in 100mL de-ionized water, and heating over a hot plate in a Pyrex beaker up to a temperature of ~95 °C, and then stirred at that temperature for 5 min. The hydrogels were formed upon cooling down to room temperature.



**Figure S4.** Top: SEM images of CA5 (left) and HA5 (right) gels. The latter does not present the characteristic defects typically found in the conventional gel due to inhomogeneities and air bubbles. Bottom: Corresponding pore size distribution for CA5 (A, Left) and HA5 (B, Right), respectively. The standard deviation of pore size distribution for the hydrothermal gel (46 nm) is significantly smaller than that of the conventional gel (69 nm). Therefore, both images and pore size distribution confirm a superior homogeneity for HA samples..



**Figure S5.** FTIR spectra of the starting powder material, conventional (CA5) and hydrothermal (HA5) gels showing the integrity of the agar polymer even after hydrothermal treatment. It should be noted that the very weak peak at ca.1250 cm<sup>-1</sup> is assigned to total sulphate,[1] The peak at ca. 935 is assigned to 3,6-anhydro-L-galactose modes (3,6-AG). The ratios A(935)/A(1250) have been used as a relative measure of sulphate concentration in agar. The ratios we obtain from these spectra are 14.7 (powder), 11.9 (CA5) and 11.7 (HA5). These are higher values than those obtained for reported native agar (6.41) or even alkali-treated agar (9.83) [1] which indicates a lower sulphate concentrations in our samples as compared with those of the literature.[1] These results are in good agreement with elemental analyses of all our agar samples (including the starting powder) which present S contents < 0.1%

[1] R.D. Villanueva, A.M.M. Sousa, M.P. Gonçalves, M. Nilsson and L. Hilliou, *J. Appl. Phycol.* (2010), 22, 211-220 DOI: 10.1007/s10811-009-9444-7