Electronic Supplementary Material (ESI) for Nanoscale.

One-pot laser-assisted synthesis of porous carbon with embedded magnetic cobalt nanoparticles

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Experimental Part

XRD analysis

Two different acquisition conditions were applied according to the importance of the microstructural effect. The standard acquisition is used when the microstructuctural effect is rather weak, *ie*: with a step length of 0.05°2θ and counting time 15s·step^{-1}. The step size is increased up to 0.2°2θ with a counting time 100 s·step^{-1} otherwise. Operating conditions for anode are 40 kV and 40 mA. Instrumental broadening contribution has been evaluated by measuring a Standard Reference Material (SRM) that shows a minimal amount of physical line broadening caused by defects and small crystallite size. The National Institute of Standards and Technology (NIST) make available several Standard Reference Material (SRM) for this purpose. The Instrumental Resolution Function (IRF) was established using NIST SRM-1976a reference material. SRM-1976a is a sintered corundum plate certified for measurement of instrument sensitivity, and, although not certified as a profile-shape standard, this SRM also exhibits a minimum of sample-induced profile broadening [1]. Choice of such a reference material has also been governed by the linear absorption coefficient μ of Al_2O_3 (124cm^{-1} at λ=1.54Å) which is nearest to our compounds than the well known SRM-660a (LaB_6, μ=1099cm^{-1} at λ=1.54Å).

NMR analysis

Samples were packed in 4 mm diameter cylindrical zirconia rotors with Kel-F rotor end caps and spun at the “magic angle” at 12 kHz in a Bruker DB MAS probe.

Cross-polarisation (CP) spectra were acquired using a 90° 1H pulse of 3.7μs duration, a 1 ms contact time and a 5s recycle delay. Free induction decays were acquired with a sweep width of 85 kHz. 8K data points were collected over an acquisition time of 48 ms. All spectra were

processed with a 24 Hz Lorentzian line broadening. Variable amplitude cross-polarization was used to minimize intensity variations of the non-protonated aromatic carbons that are sensitive to Hartmann-Hahn mismatch at higher MAS rotation rates. Direct polarisation (DP) with CW (continuous-wave) decoupling spectra represent the accumulation of 3000 scans and were acquired using a 45° 13C pulse of 2.5μs duration and a 20s recycle delay. Chemical shifts were externally referenced to adamantane at 29.45 ppm.

Results

Table 1S: Main XRD results from individual peak shape refinement: Selected reflections are not subject to overlapping.

Hybrid Material	FWHM (°2θ)	Reliability factors(1,2)		
		Rp,%	Rωp,%	χ^2
CGY-L@CA	3.0(3)	1.4	1.8	1.23
CGY-L@CN	2.4(2)	1.1	1.5	1.00
CGY-L@CC Co_{fcc}	0.34(4)	2.8	3.4	1.12
CGY-L@CC Co_{hcp}	0.16(2)	2.5	3.2	1.45
	0.22(1)	2.3	3.0	1.33
	0.25(9)	3.1	3.7	1.00
	0.35(9)	3.5	4.4	0.96

(1): conventional Rietveld R-factors for points with Bragg contribution
(2): definitions following [1]

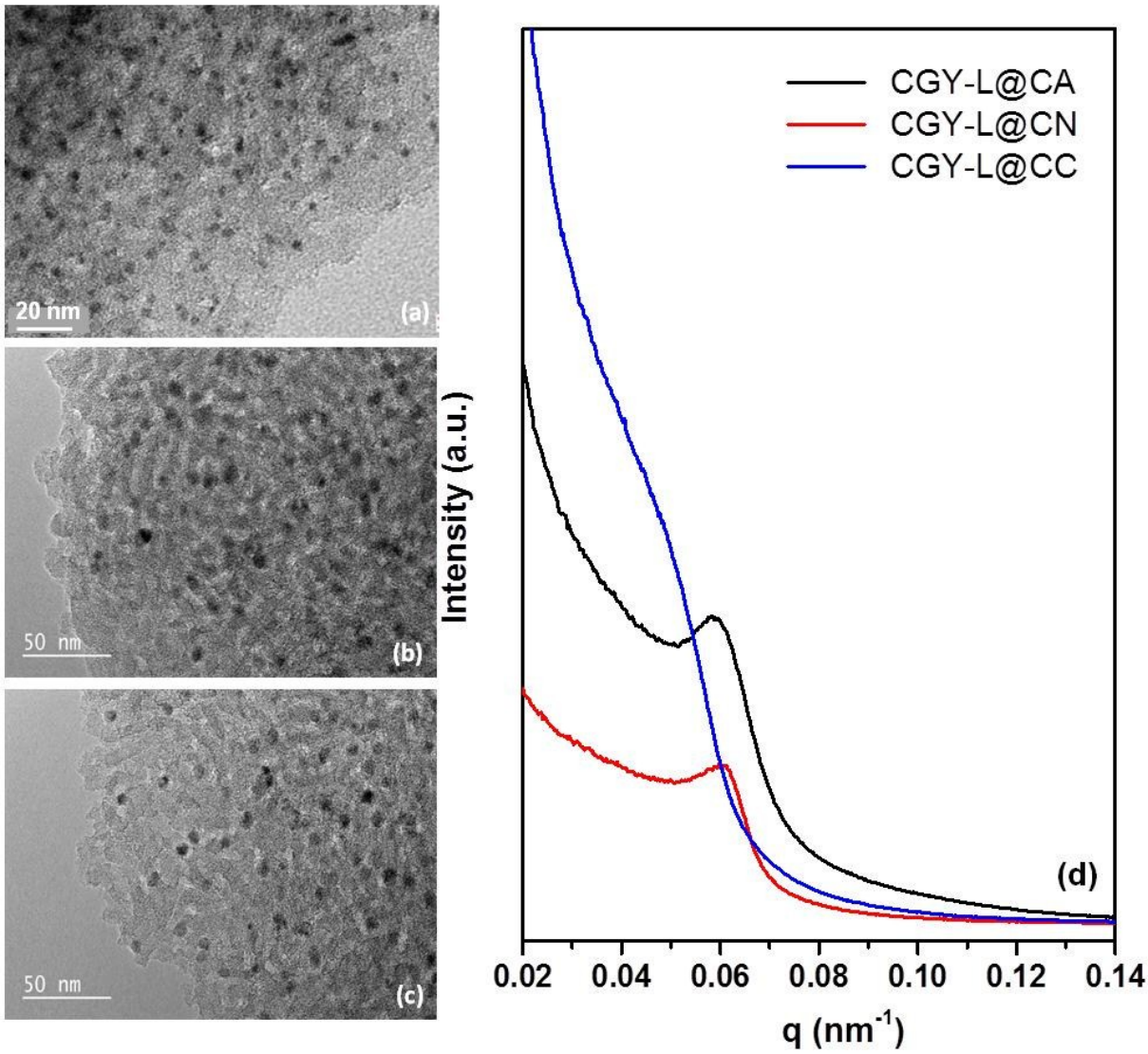

Figure S1: TEM pictures of (a) CGY-L@CA (b) CGY-L@CN and (c) CGY-L@CC materials synthesized by EISA classical approach along with their corresponding SAXS patterns (d)

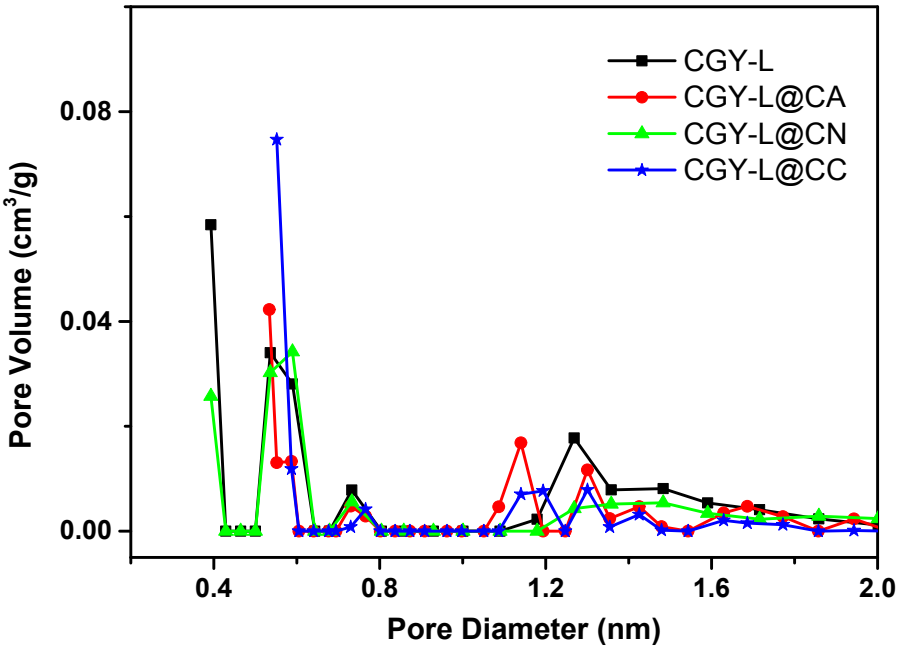

Figure S2: DFT micropore size distribution of carbon CGY-L and carbon loaded with cobalt nanoparticles

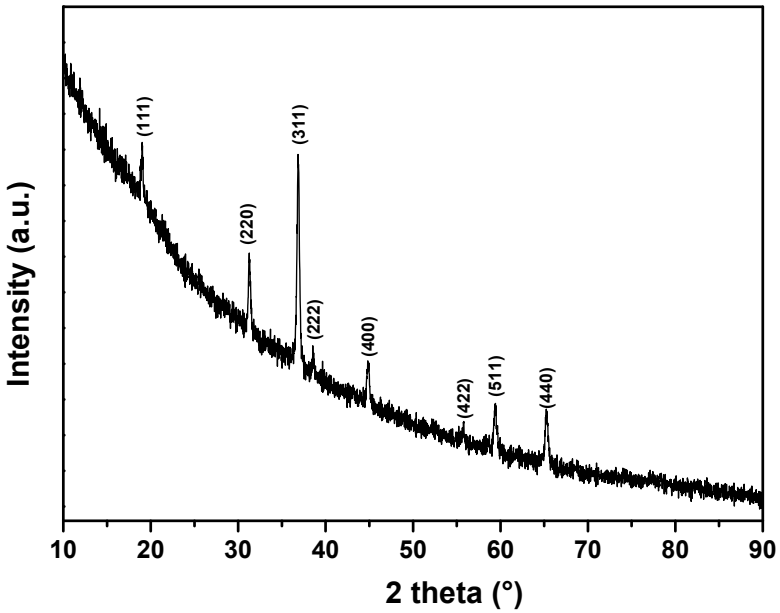

Figure S3: XRD spectra of a CGY-L@C residue obtained after TGA measurement

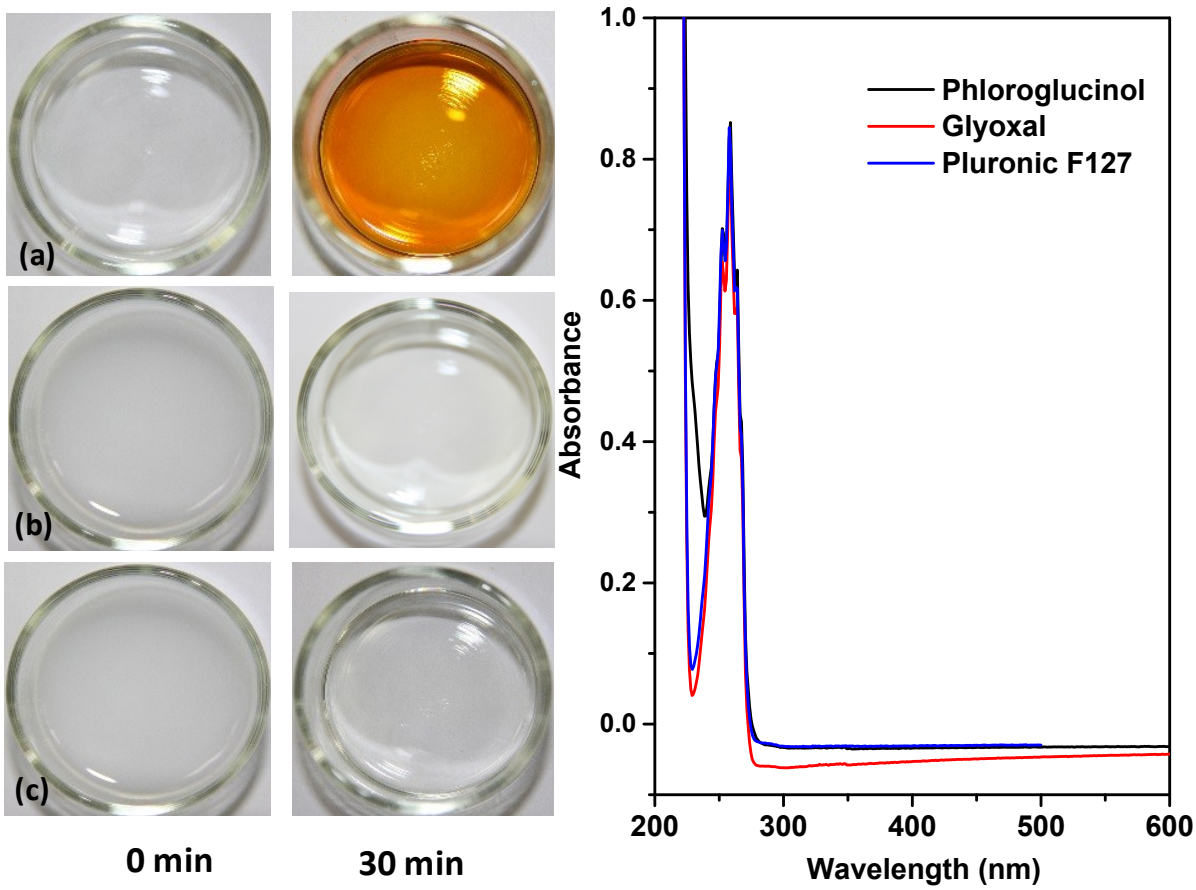

Figure S4: Images of (a) phloroglucinol (b) glyoxylic acid and (c) pluronic ethanolic solutions before and after irradiation

Reference List

[1] Delhez R, de Keiser ThH, Langford JI, Louër D, Mittemeijer EJ, Sonneveld EJ. The Rietveld Method. edited by R.A.Young, pp.1-38.IUCr/Oxford University Press 1993.