Nickel nanoparticles deposited into an activated porous carbon: synthesis, microstructure and magnetic properties

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Introduction

- ✓Activated carbons (AC) are extensively used for adsorption and catalytic purposes, mainly due to their outstanding efficiency together with a wide availability and low cost.

- ✓ They are exploited in liquid phase for a number of applications, such as, catalyst or catalytic supports, to remove contaminants or for recovering specific products.

- ✓ If the selective manipulation of valuable substances associated with AC is pursued, magnetic separation could be the most effective strategy for achieving this task.

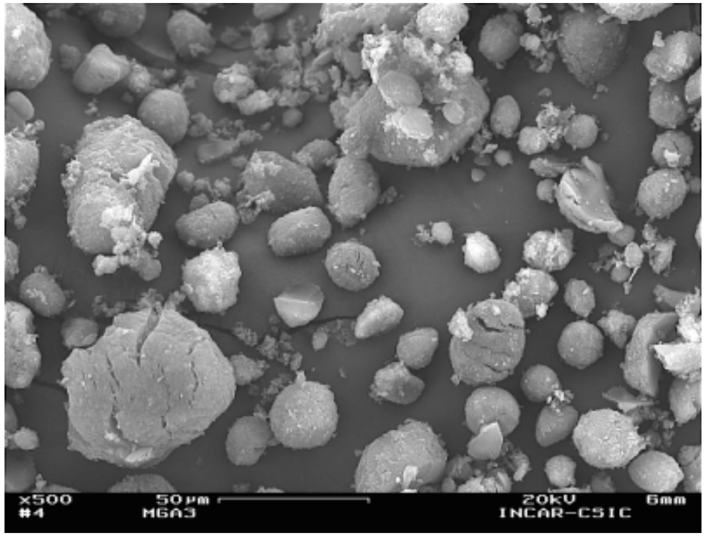
- ✓ We present a new and easy-to-follow synthesis procedure to prepare magnetically separable porous carbons, in which Nickel nanoparticles have been deposited.

AIM: Study the correlation between microstructure and magnetic properties

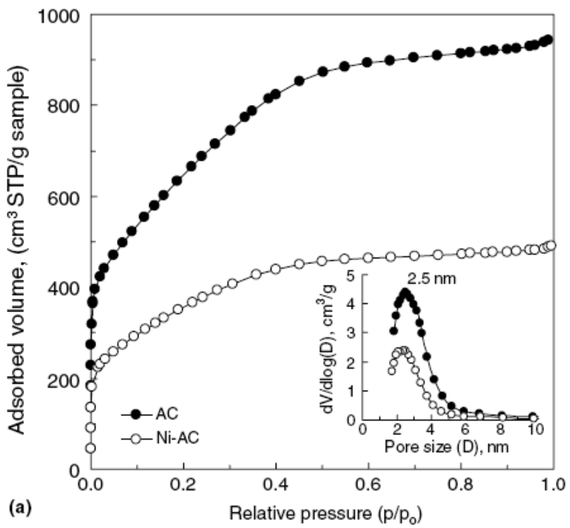
SYNTHESIS PROCEDURE AND POWDER MORPHOLOGY

- ✧ the starting material (matrix) is a commercial and low-cost AC with a large BET surface area, (2350 $m^2\ g^{-1}$) and a high pore volume (1.47 $cm^3\ g^{-1}$).
- ✧ the AC is impregnated with an aqueous solution containing sucrose and nickel nitrate
- ✧ a subsequent heat-treatment results in the formation of Nickel nanoparticles dispersed along the porous AC (the amount of Ni in the sample is ≈ 16 wt.%)
- ✧ the addition of sucrose favours protection against acid corrosion
- ✧ after acid attack (HCl) the final amount of Ni in the sample is ≈ 12 wt.%

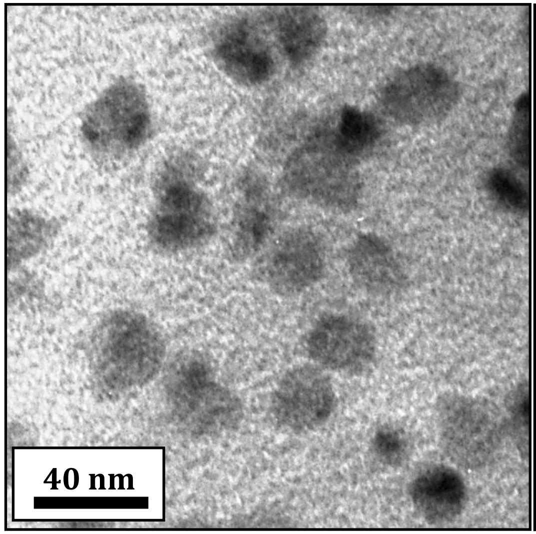
SEM
Quasi-spherical powder grains
1 – 50 μm

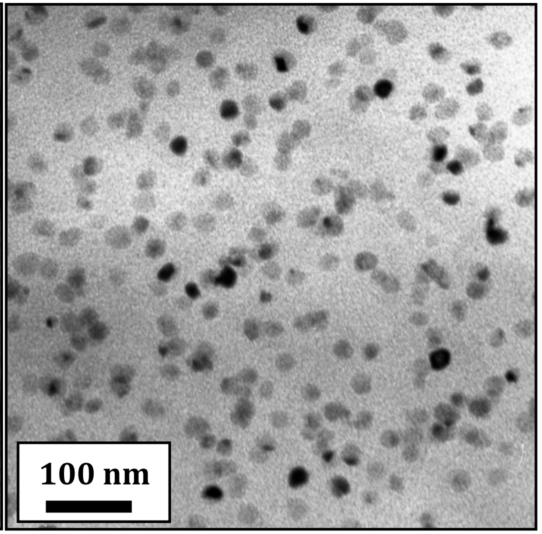


The BET surface area (BET N2) is a measurement of the extent of the pore surface developed within the matrix of the activated carbon using nitrogen (N2). Used as a primary indicator of the activity level, based on the principle that the greater the surface area, the higher the number of adsorptive sites available.



CRYSTAL STRUCTURE & MICROSTRUCTURE





TEM

Quasi-spherical and well-dispersed nanoparticles

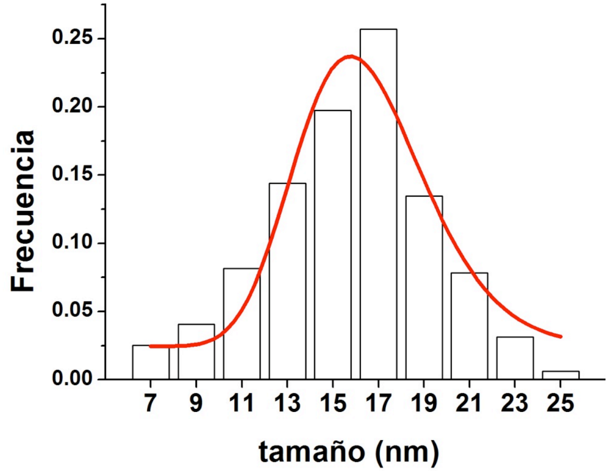
Mean size: $\langle\tau\rangle_{TEM}$ = 16(1) nm

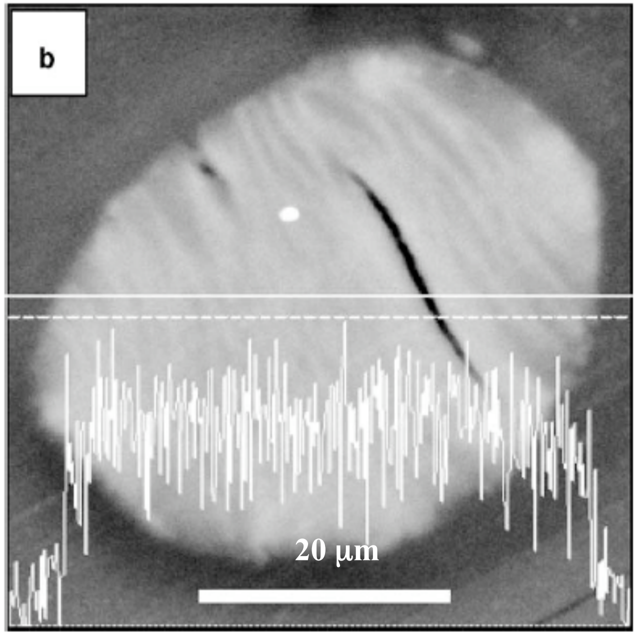
XRD

Bragg reflections corresponding to FCC-Ni

Cell parameter: a = 3.525 Å (pure Ni, a = 3.532 Å)

Mean size: $\langle\tau\rangle_{XRD}$ = 18(1) nm

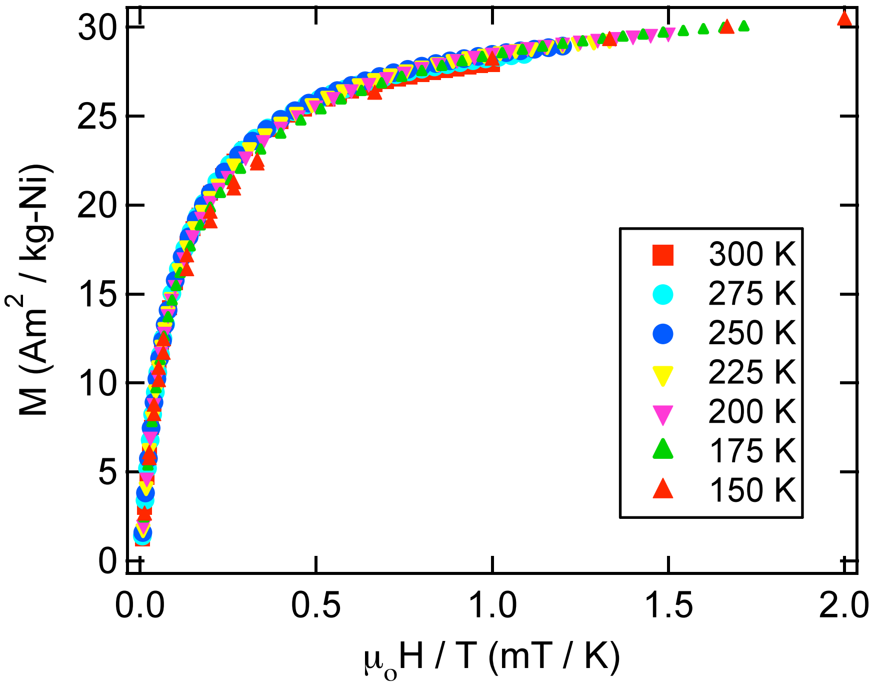




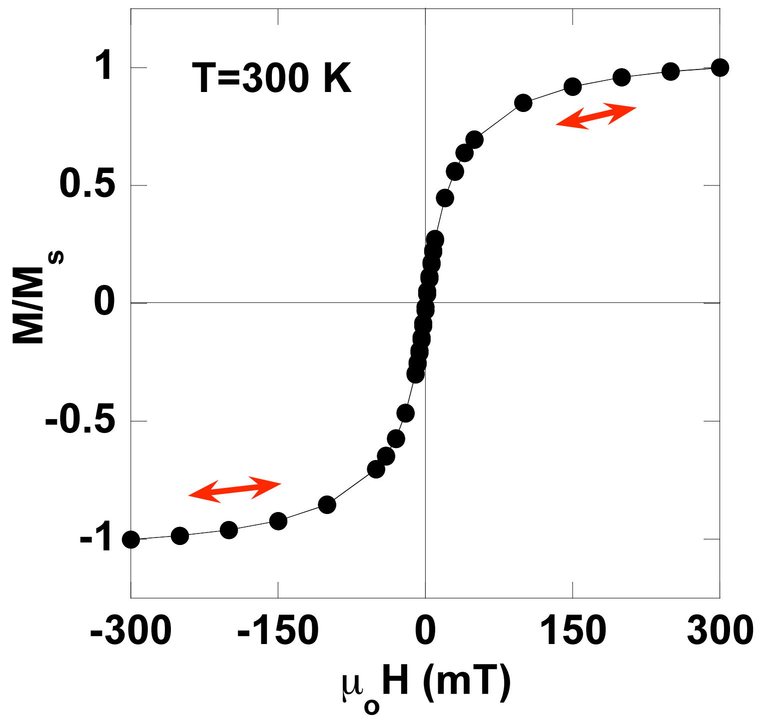
Nickel distribution obtained by energy dispersive spectrometry in the section of a particle. The noisy line represents the nickel content. The continuous line represents the selected line for analysis and the discontinuous line represents saturation of the signal. Bar length = 20 µm.

MAGNETIC PROPERTIES: 1. CONDITIONS FOR SPM

for T > 200 K M is an universal function of $\mu_0 H/T$



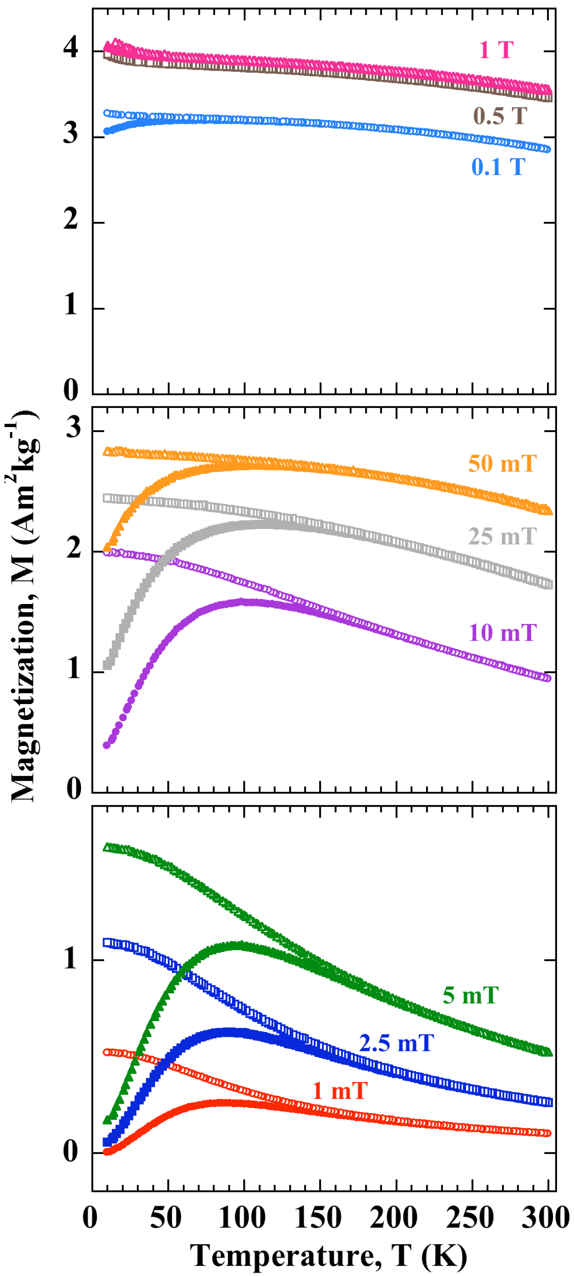
Reversible M(H) (no hysteresis)

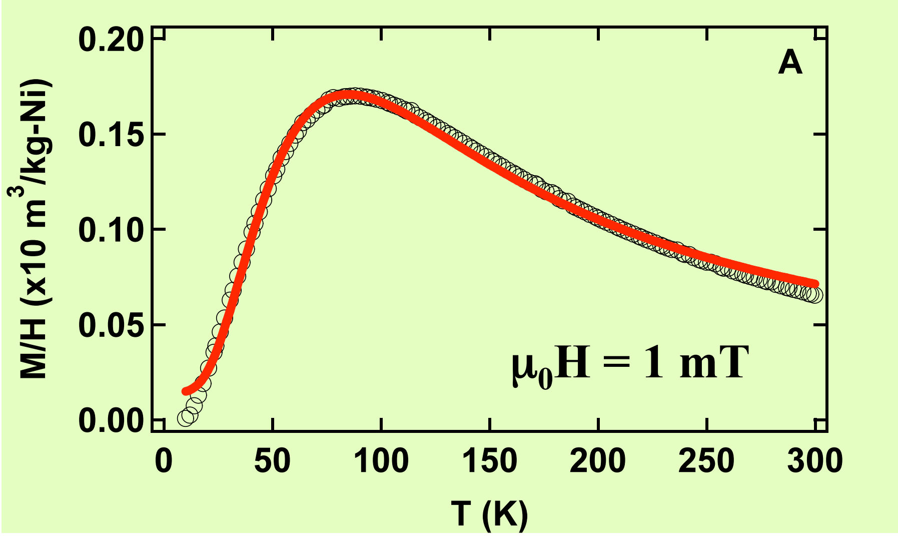


Fit to a Langevin function

d (nm)	M_s (Am^2/kg-Ni)
13(1)	30(2)

MAGNETIC PROPERTIES: 2. M vs T (ZFC-FC behaviour)

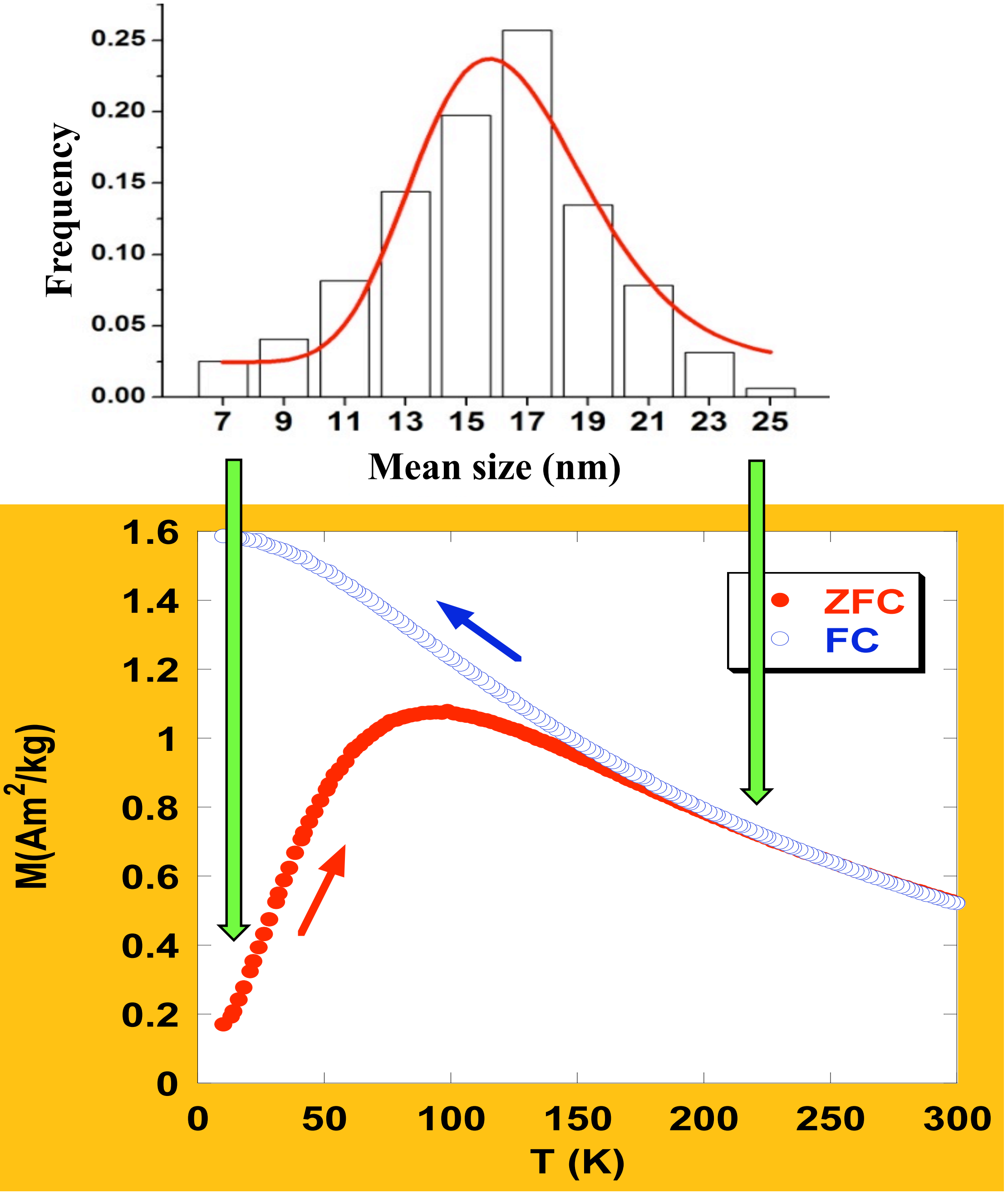




Fit to a Stoner – Wohlfarth model

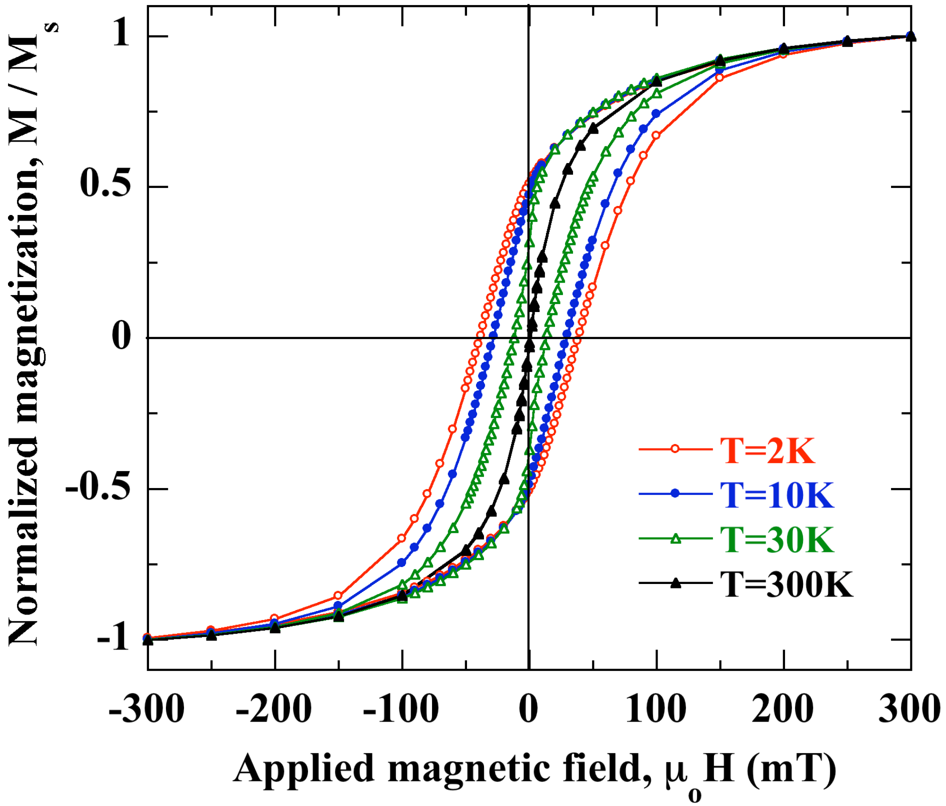
	M_s (Am^2/kg-Ni)	K_{ef} (× 10^3 J/m^3)
Ni-AC	22(3)	9.7(4)
Ni bulk	54.5	4.5

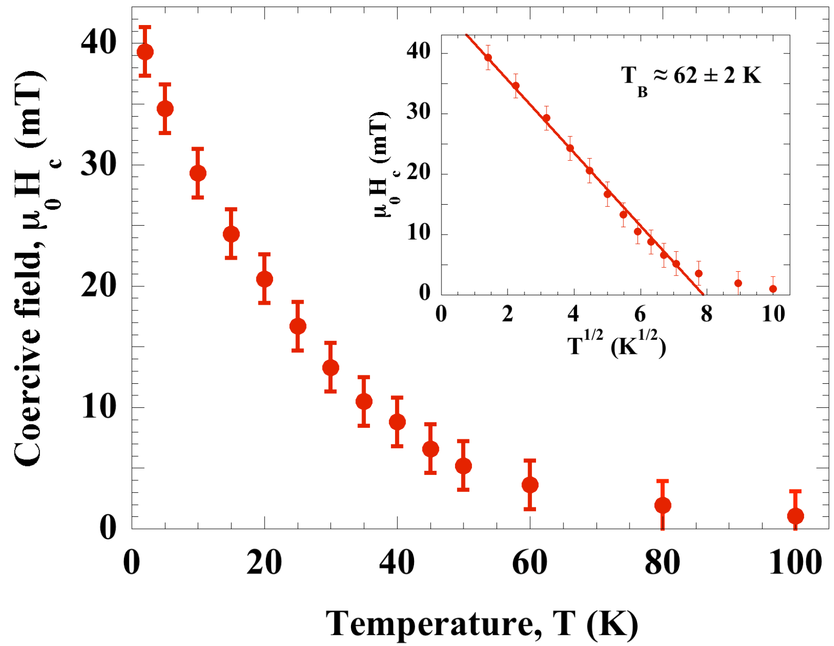
MAGNETIC PROPERTIES: 2. M vs T (ZFC-FC behaviour)



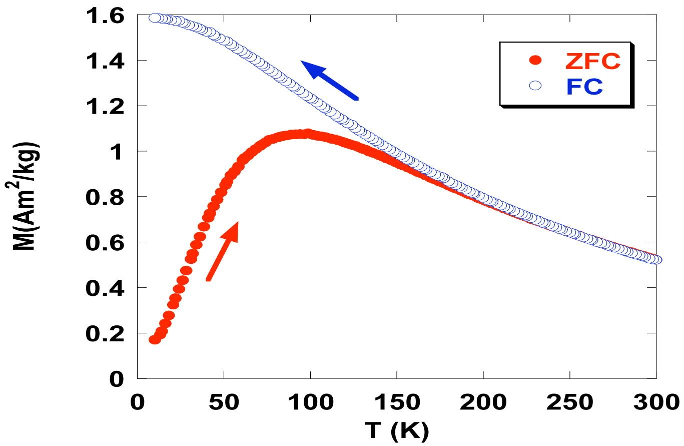
$$T_B = \frac{KV}{25 k_B}$$

MAGNETIC PROPERTIES: 3. M vs $\mu_0 H$





$$\mu_o H(T) = \mu_o H_c(0)\left[1 - \sqrt{\frac{T}{T_B}}\right]$$



SUMMARY & CONCLUSIONS

- **Ni nanoparticles can be easily inserted in activated porous carbons.**
- **The addition of sucrose protects the nanoparticles in low pH media.**
- **The Ni-AC powders exhibits magnetization values around 4 Am^2kg^{-1} and SPM at room temperature, hence, its manipulation using conventional permanent magnets for applications in magnetic separation is assured.**
- **The combination of electron microscopy, x-ray powder diffraction and magnetization vs. temperature and applied magnetic field measurements show that there is a distribution of nanoparticle sizes (≈ 7 – 25 nm) giving rise to a distribution of blocking temperatures (≈ 5 – 230 K).**
- **Saturation magnetization is around one half, while the effective magnetic anisotropy is roughly double to those of Bulk Ni.**