

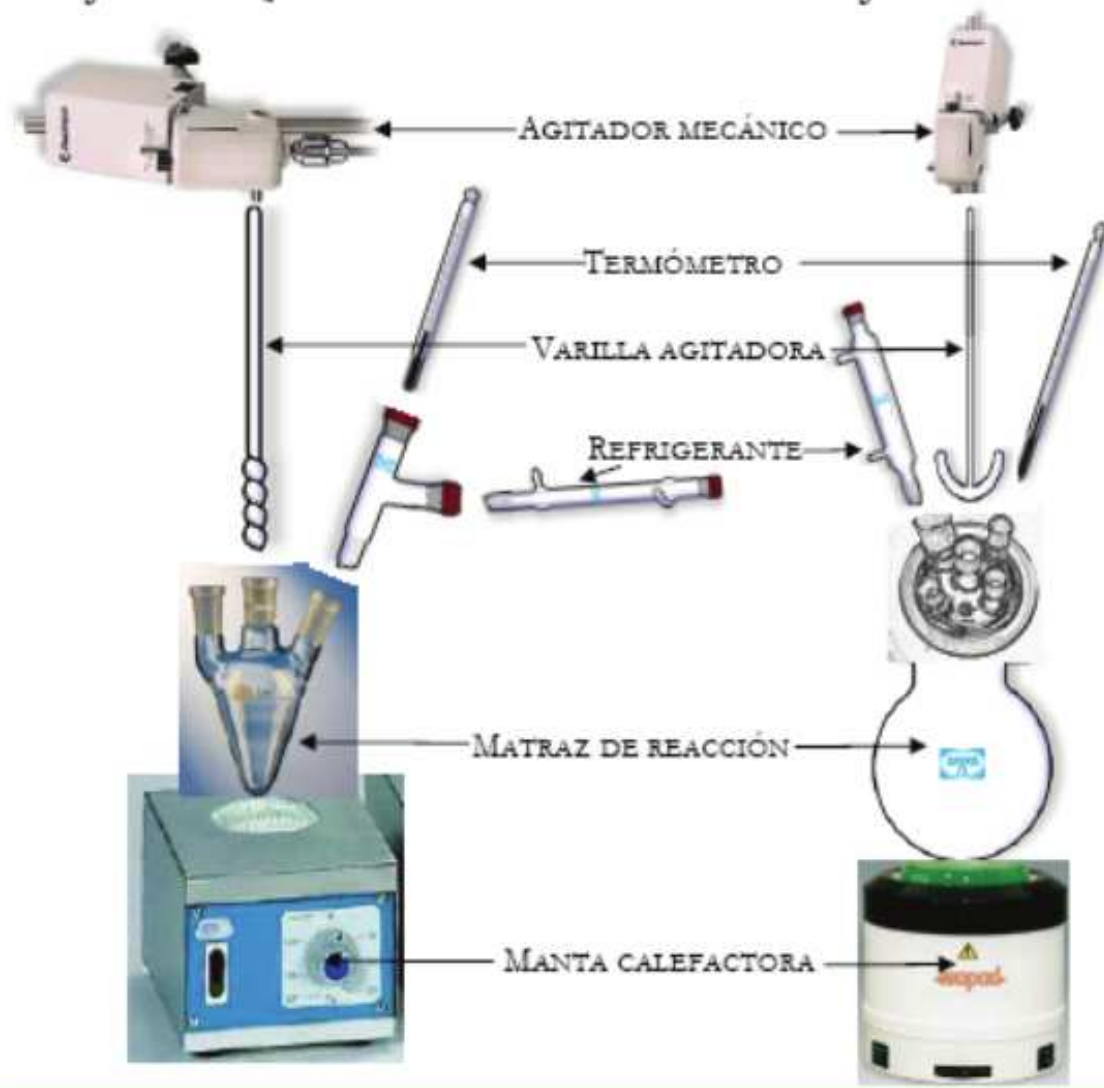
Síntesis y caracterización de nanopartículas de Fe_3O_4

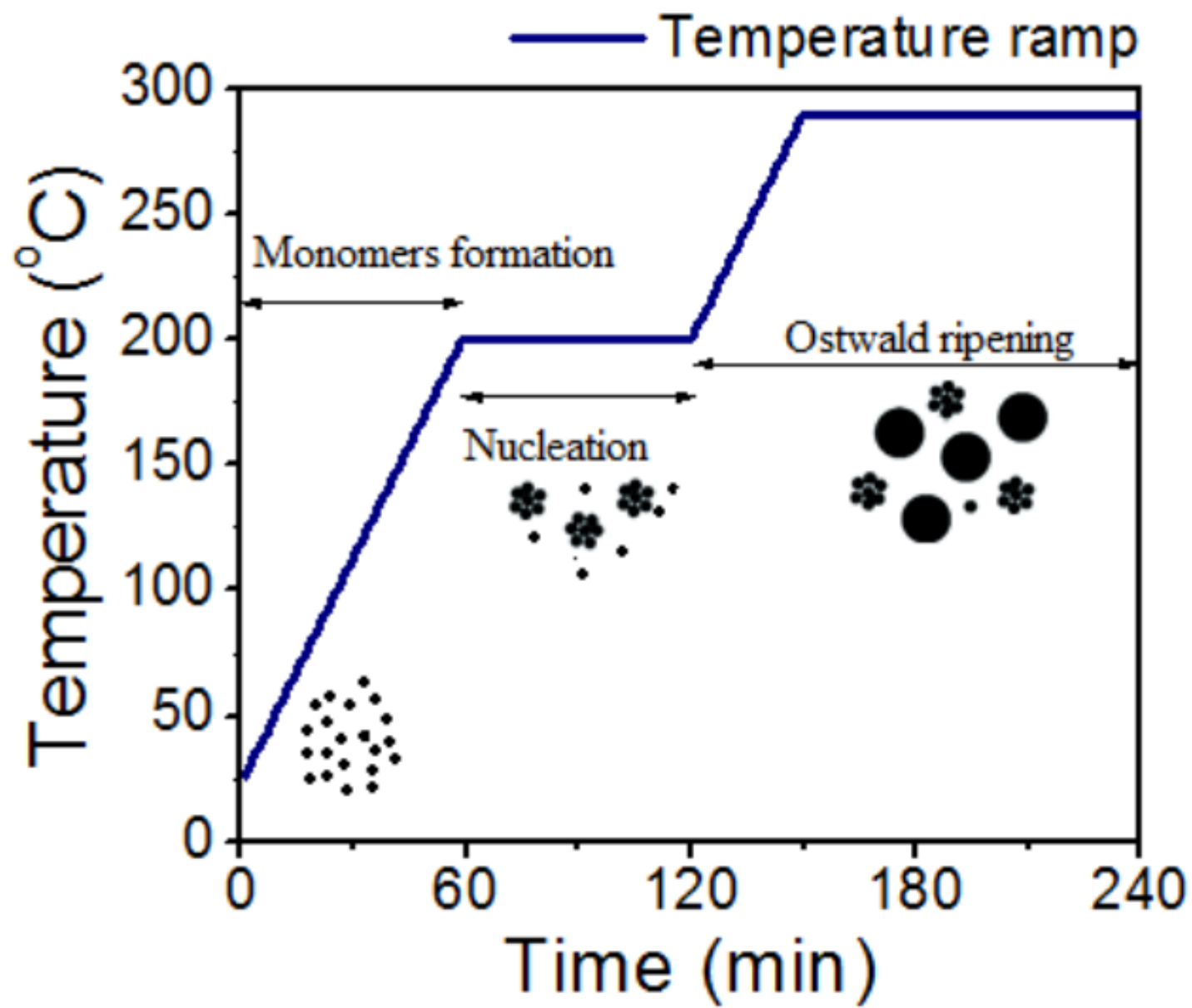
Juan Manuel Orozco

Obtenção das nanopartículas

MONTAJE A PEQUEÑA ESCALA

MONTAJE A GRAN ESCALA





Síntese das nanopartículas

Método de LaMer

Victor K. LaMer and Robert H. Dinigar. *J. Am. Soc. Chem.* 72 (1950)

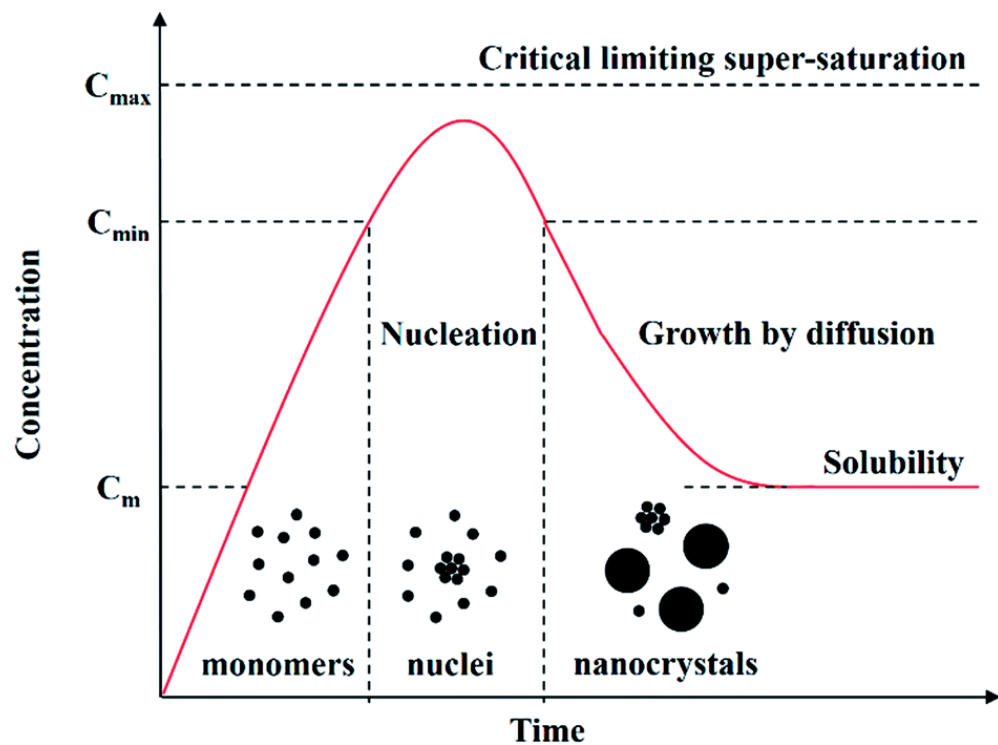


Figura 1. Síntese de nanocristais. Processo de LaMer.

Jin Chang and Erick R. Waclawik. *RSC Adv.*, 4 (2014)

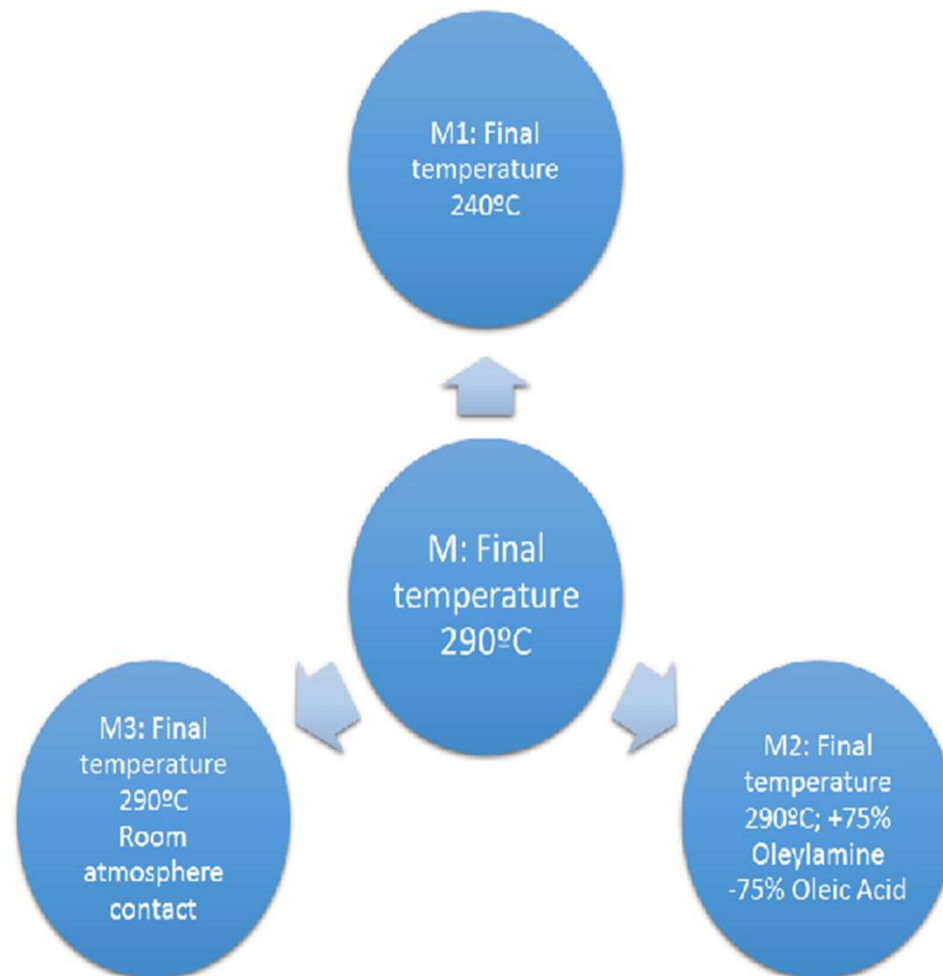
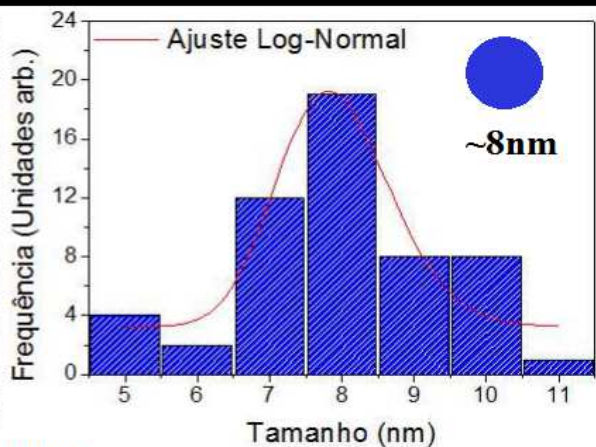
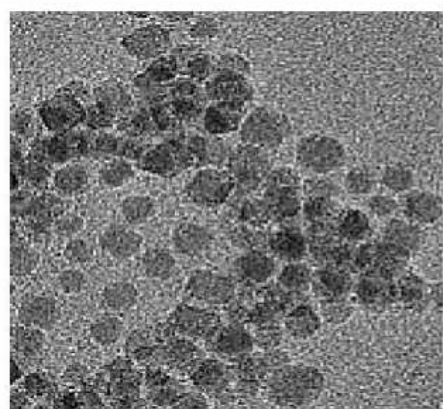


Figura 2. Variações feitas na síntese das nanopartículas.

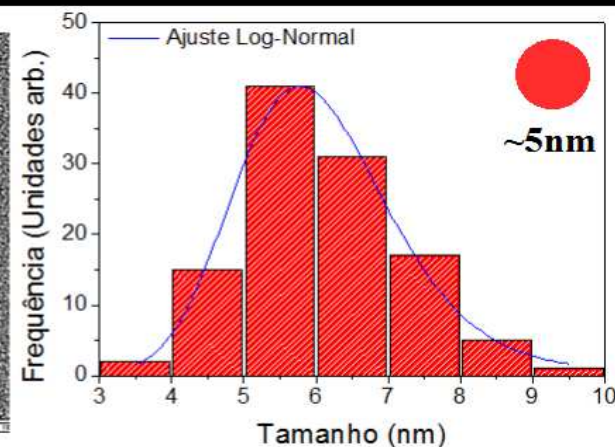
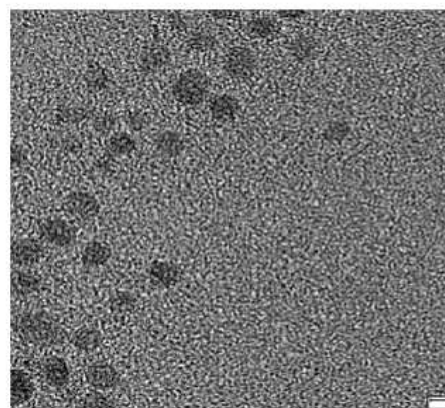
Caracterização estrutural

Caracterização Estrutural

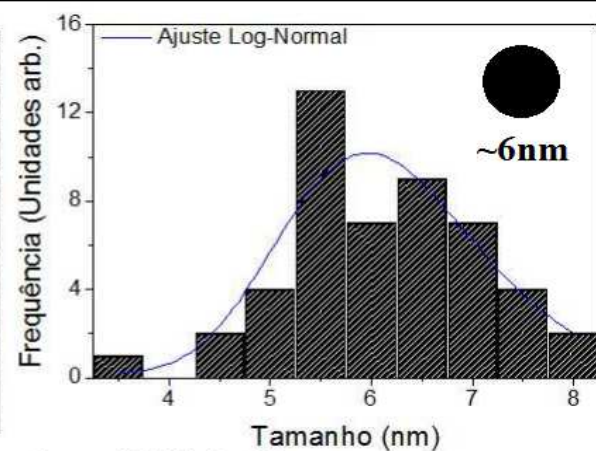
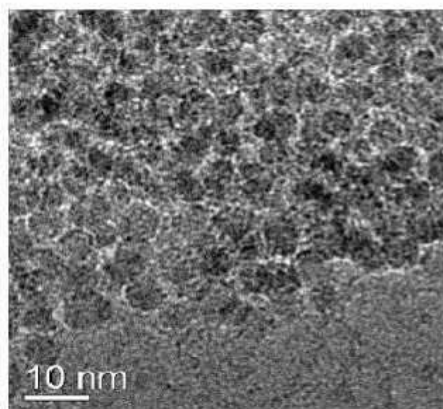
Imagens TEM e Distribuições de tamanhos



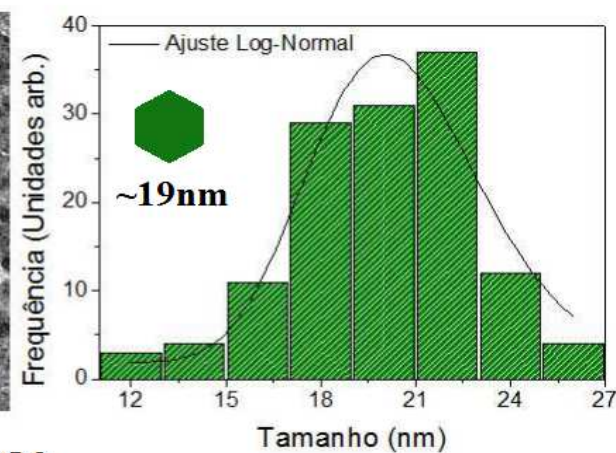
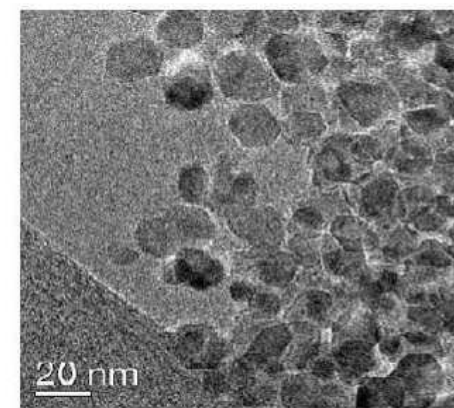
Amostra padrão J02. Tf = 290°C



Amostra J07. Tf = 240°C



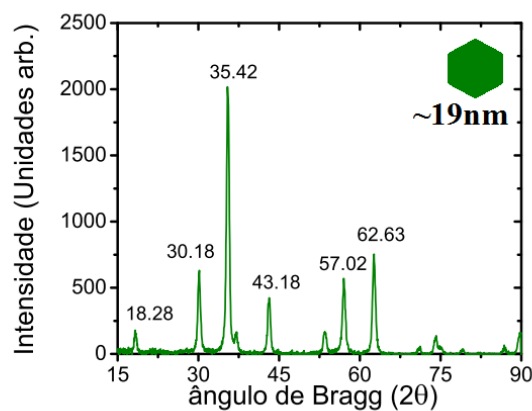
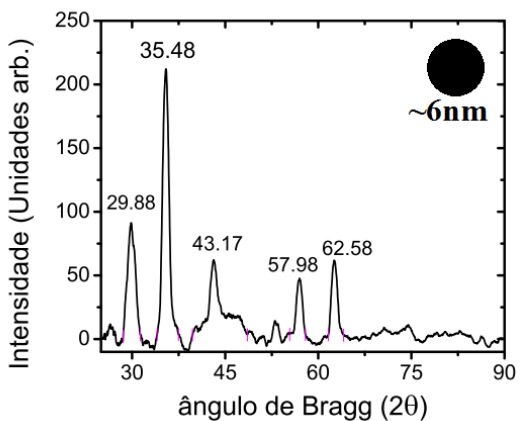
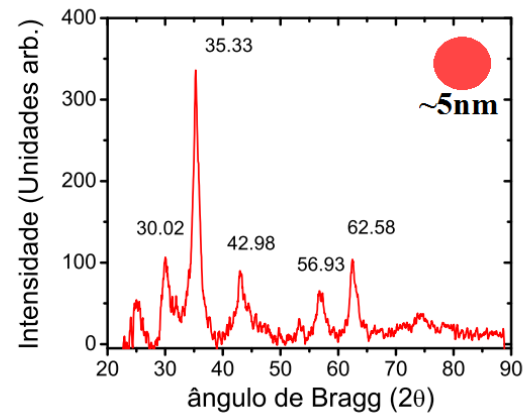
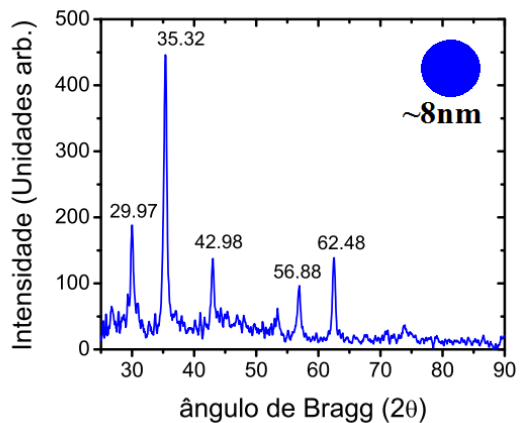
Amostra J09. +75% Oleilamina, -75% Acido oléico



Amostra J11. Contato com O2

Caracterização Estrutural





Difração de Raios X e Estimação do tamanho dos nanocristais



Equação de Scherrer :

$$\tau = \frac{K \lambda}{\beta \cos(\theta)}$$

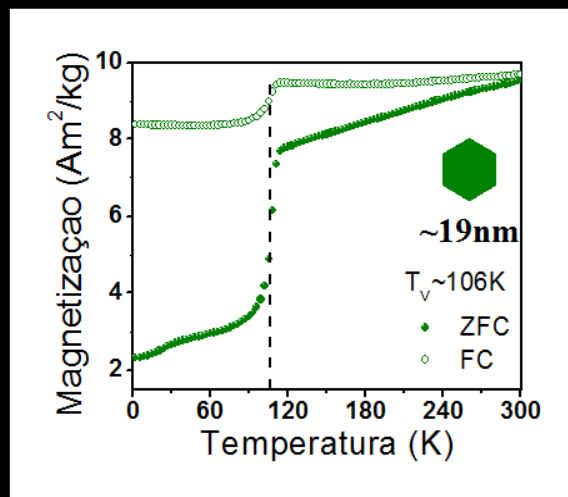
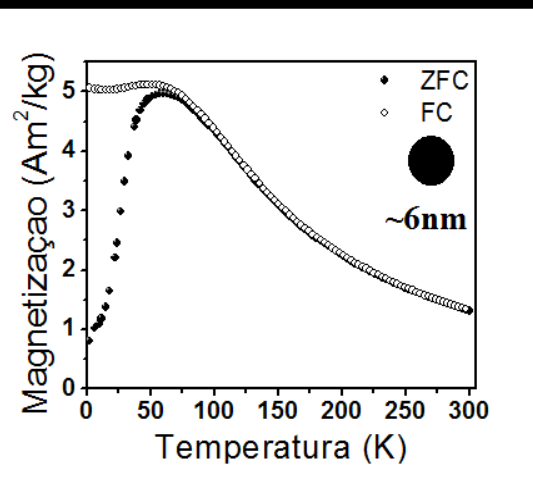
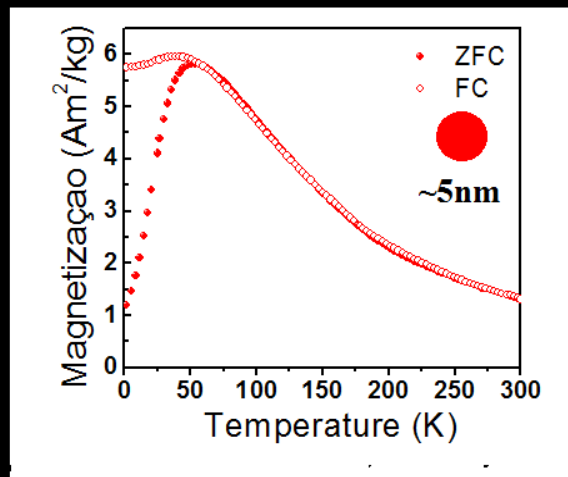
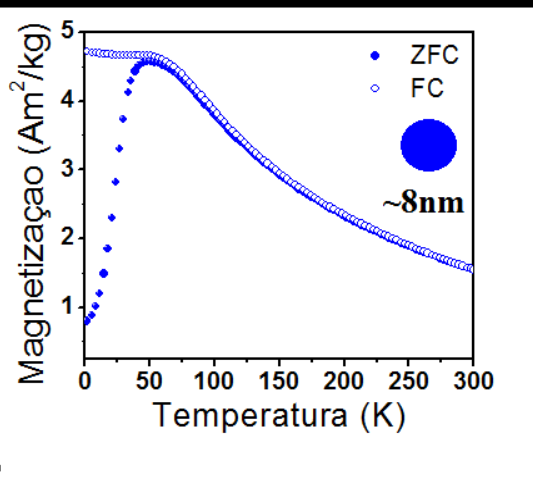
- K : Fator de Forma
- λ : Comprimento de Onda
- β : FWHM
- θ : Ângulo de difração

Amostra	θ (grad)	β (rad)	Tamanho (nm)
 ~8nm	17.7	9.8×10^{-3}	*
 ~5nm	17.7	20.2×10^{-3}	8.6 ± 0.9
 ~6nm	17.7	18.9×10^{-3}	9.2 ± 0.9
 ~19nm	17.7	10.2×10^{-3}	17.8 ± 1.2




Caracterização magnética

Resultados

Magnetização zero field cooled e Field Cooled



Momentos magnéticos efetivos obtidos mediante a lei de Curie:

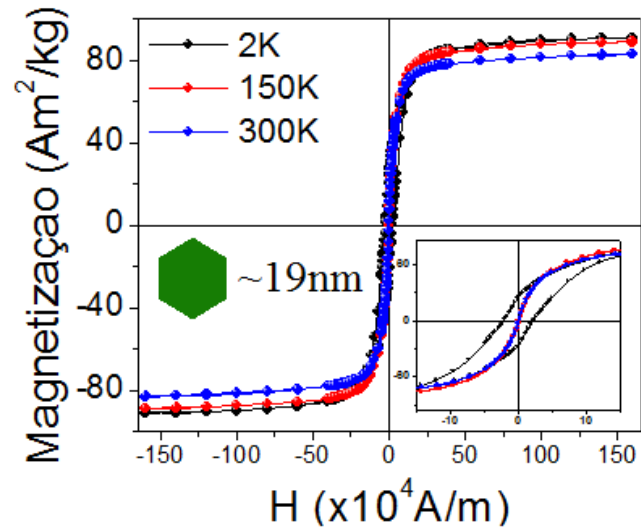
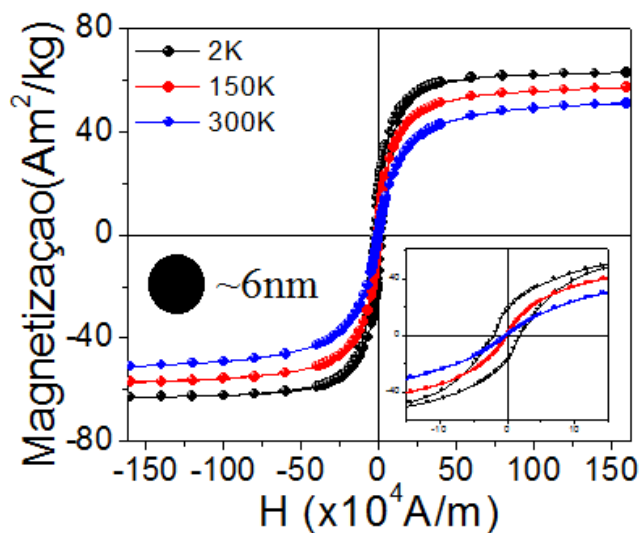
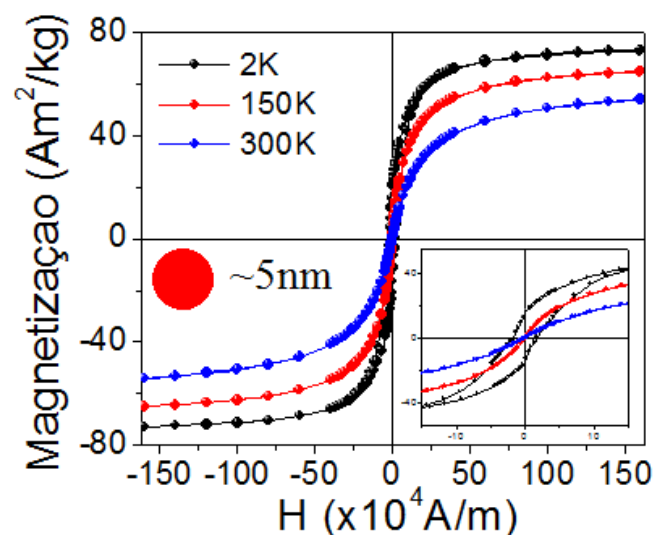
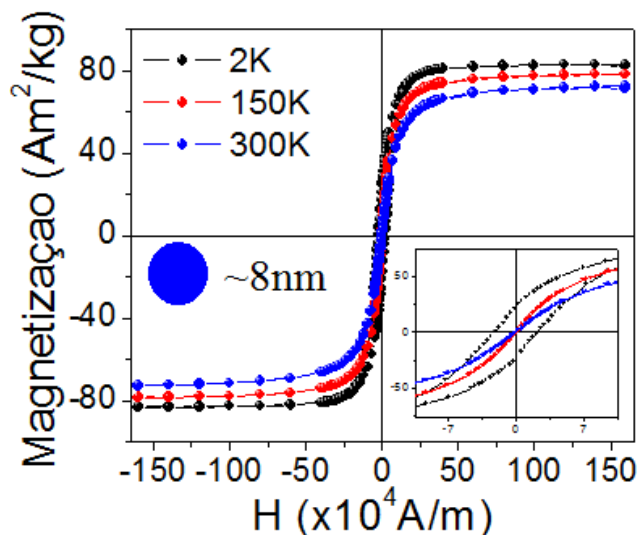
Amostra	μ_{ef} ($\times 10^3 \mu_B$)
 ~8nm	6.5 ± 0.4
 ~5nm	3.2 ± 0.2
 ~6nm	4.5 ± 0.2

Campo aplicado de 500e em cada caso no intervalo de temperaturas superiores à temperatura de bloqueio.

$$\chi = \frac{M}{H} = \frac{nC}{T} \quad \left| \quad C = \frac{n\mu^2}{3k_B}\right.$$

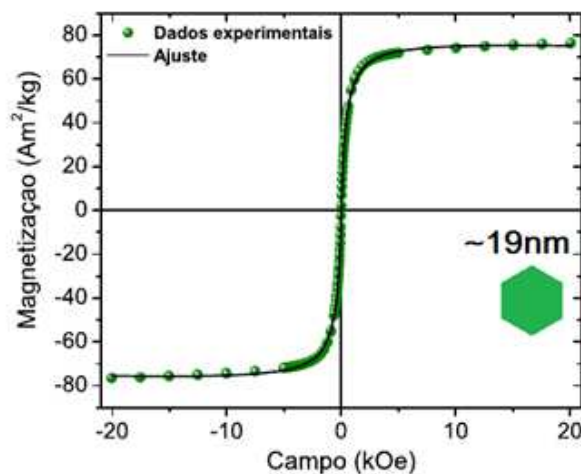
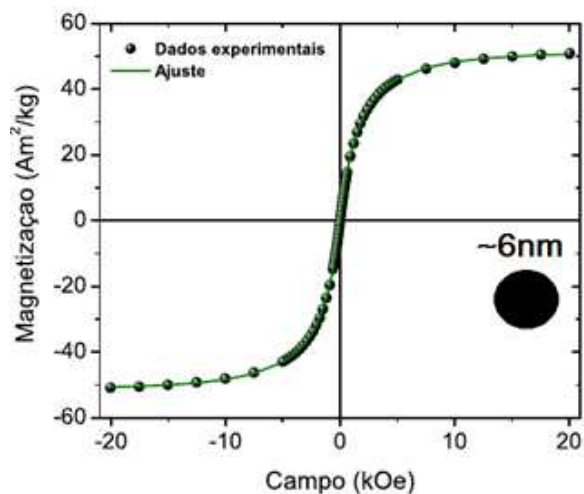
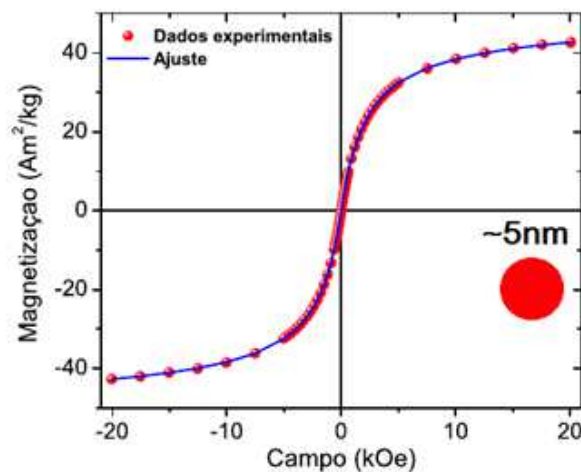
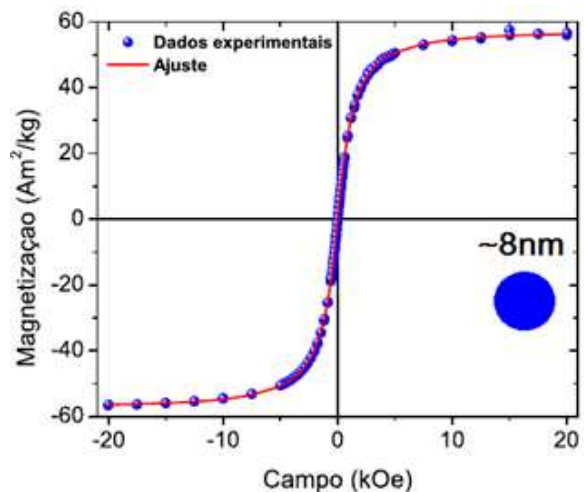
Caracterização magnética

Ciclos de histerese







Caracterização magnética

Ajuste das curvas M vs H para $T = 300K$



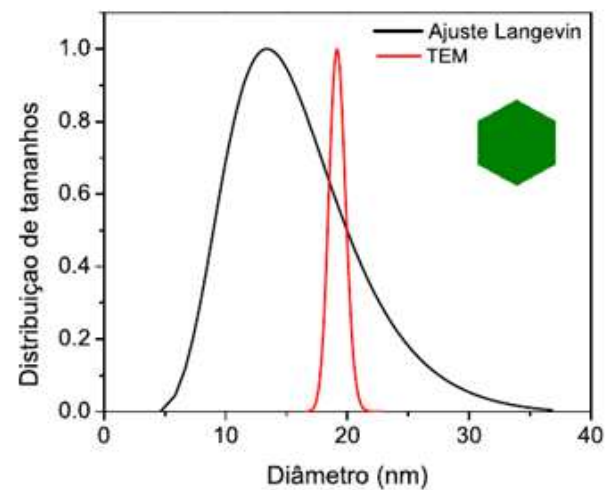
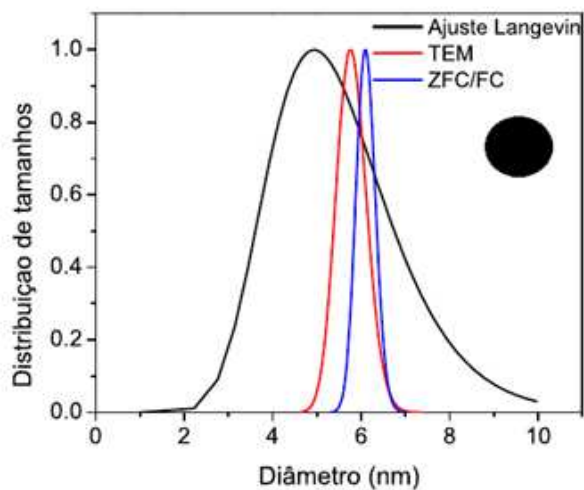
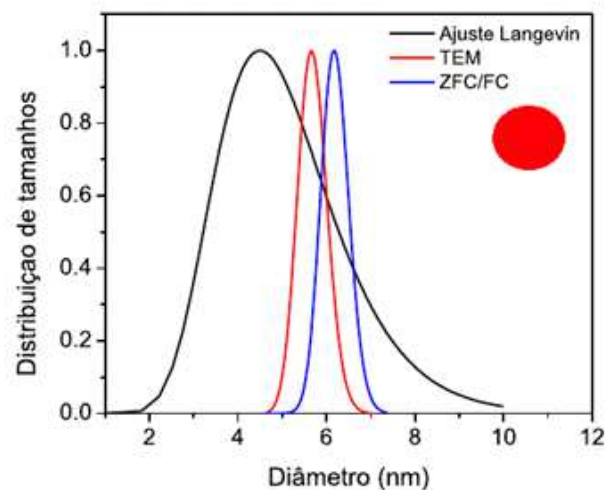
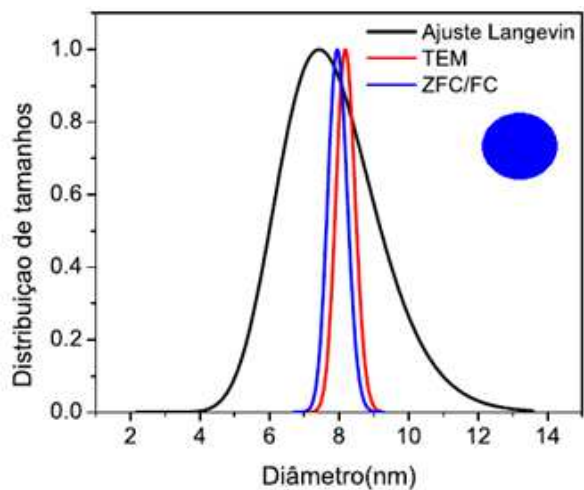
Momentos magnéticos efetivos obtidos:

$\sim 8\text{nm}$ 			
σ	μ_0	$\langle \mu \rangle$	(μ) Lei de Curie
0.5	5.2	6.1	6.5
$\sim 5\text{nm}$ 			
σ	μ_0	$\langle \mu \rangle$	
0.9	1.9	2.7	3.2
$\sim 6\text{nm}$ 			
σ	μ_0	$\langle \mu \rangle$	
0.8	2.6	3.6	4.5
$\sim 19\text{nm}$ 			
σ	μ_0	$\langle \mu \rangle$	
1.0	6.8	11.5	





*Valores de μ em $\times 10^3 \mu_B \sim 9.27e^{-21} \text{Am}^2$

Resultados

Comparação entre as distribuições de tamanho obtidas



Resumo dos resultados obtidos:

Amostra	TEM	XRD	ZFC/FC	Ajuste Langevin
 ~8nm	8.3	*	8.0	7.2
 ~5nm	5.8	8.6	6.2	6.0
 ~6nm	5.9	9.2	6.1	6.0
 ~19nm	19.3	17.8	—	14.0

Aplicações em Hipertermia magnética

Hipertermia magnética

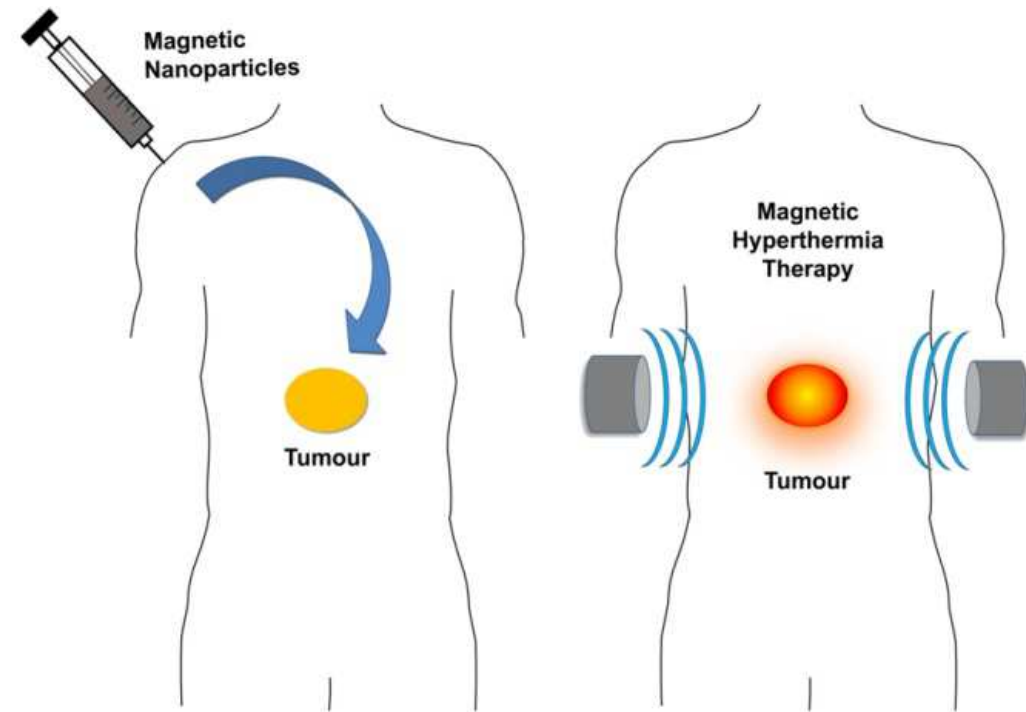


Figura 9. Magneto hipertermia como uma técnica para tratamento do câncer.

Ângela Andrade, Roberta Ferreira, José Fabris and Rosana Domingues. Coating Nanomagnetic Particles for Biomedical Applications, Biomedical Engineering - Frontiers and Challenges (2011).

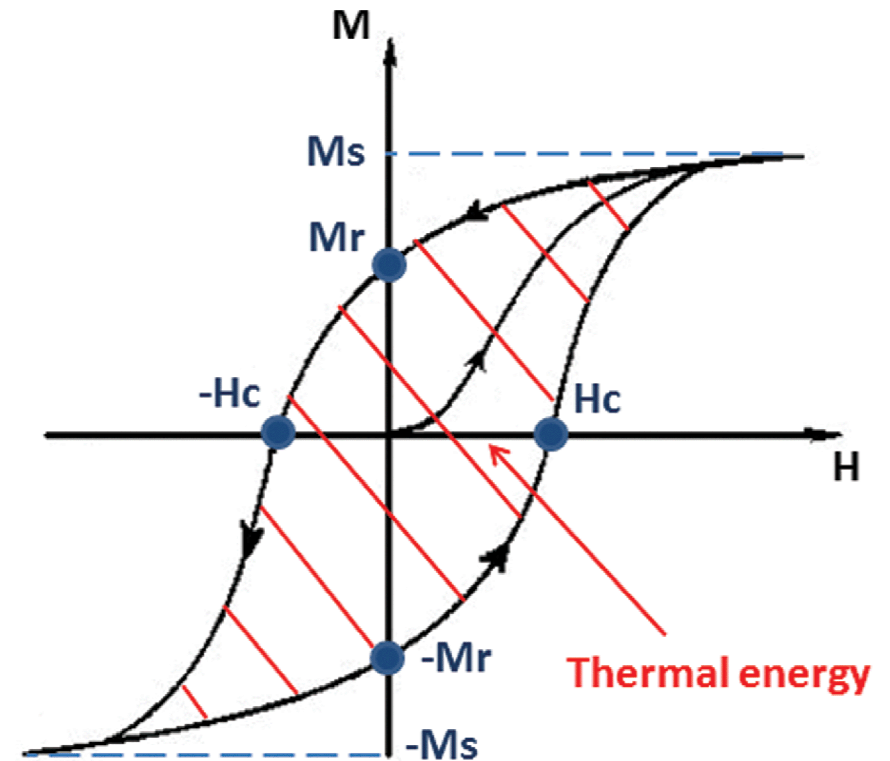
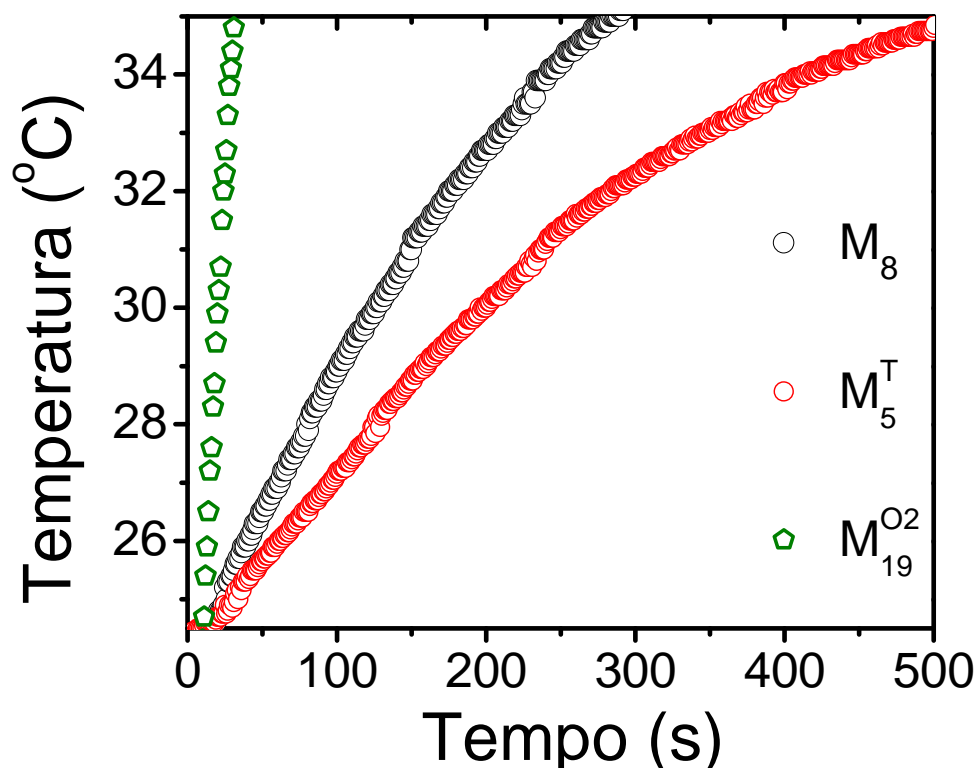


Figura 10. Energia dissipada pelo sistema baixo a ação do campo alternante.

Aziliz Hervault and Nguyen Thi Kim Thanh. Nanoscale 6: 11553 (2014)

Hipertermia magnética

Energia dissipada em função do tempo



* Campo e frequência aplicados: 52kA/m, 260kHz

Taxa de absorção específica (siglas em inglês, SAR) está dada pela relação:

$$SAR = \frac{\rho C}{[C]} \frac{\Delta T}{\Delta t} [W/g]$$

ρ : Densidade do solvente

C: Calor específico do solvente

[C]: Concentração da solução

Amostra	SAR (W/g)
● ~8nm	5.8 ± 0.4
● ~5nm	3.0 ± 0.1
● ~19nm	41.3 ± 2.7

Hipertermia magnética

Relação com a teoria de resposta linear

Susceptibilidade fora de fase:

$$\chi''(f, \tau) = \chi_0 \left(\frac{2\pi f \tau}{1 + (2\pi f \tau)^2} \right)$$

Relacionada com a potência dissipada pela teoria de resposta linear:

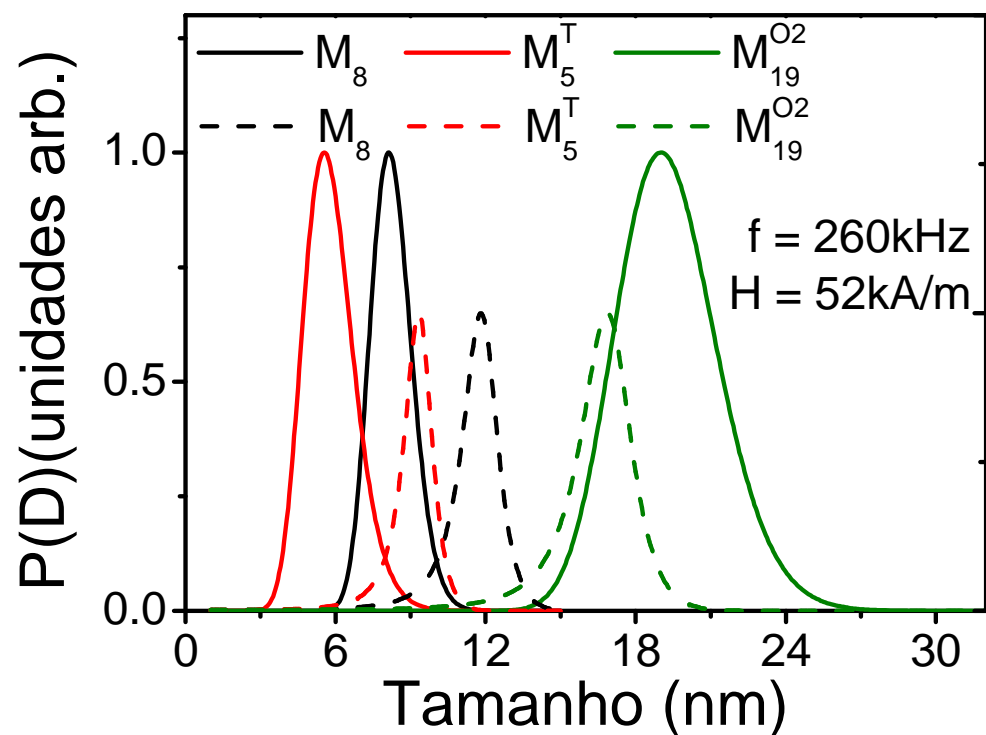
$$P = 2\mu_0 \pi f H_0^2 \chi''(f, \tau)$$

Amostra	SAR (W/g)
● ~8nm	7.13
● ~5nm	3.21
● ~19nm	20.81

Hipertermia magnética

Aproximação usando as distribuições de tamanho

Uma aproximação mais exata à potência dissipada pelo sistema tem em conta a distribuição dos tamanhos das partículas:



$\chi''(D)/\chi_0$ (unidades arb.)

$$P = \pi f \mu_0 H_0 \frac{\int_0^\infty \chi''(D) P_N(D) dD}{\int_0^\infty P_N(D) dD}$$

