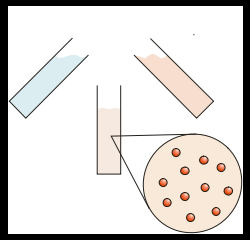

Precipitación de Oxidos Metálicos en Medios Acuosos:

Partículas Monodispersas

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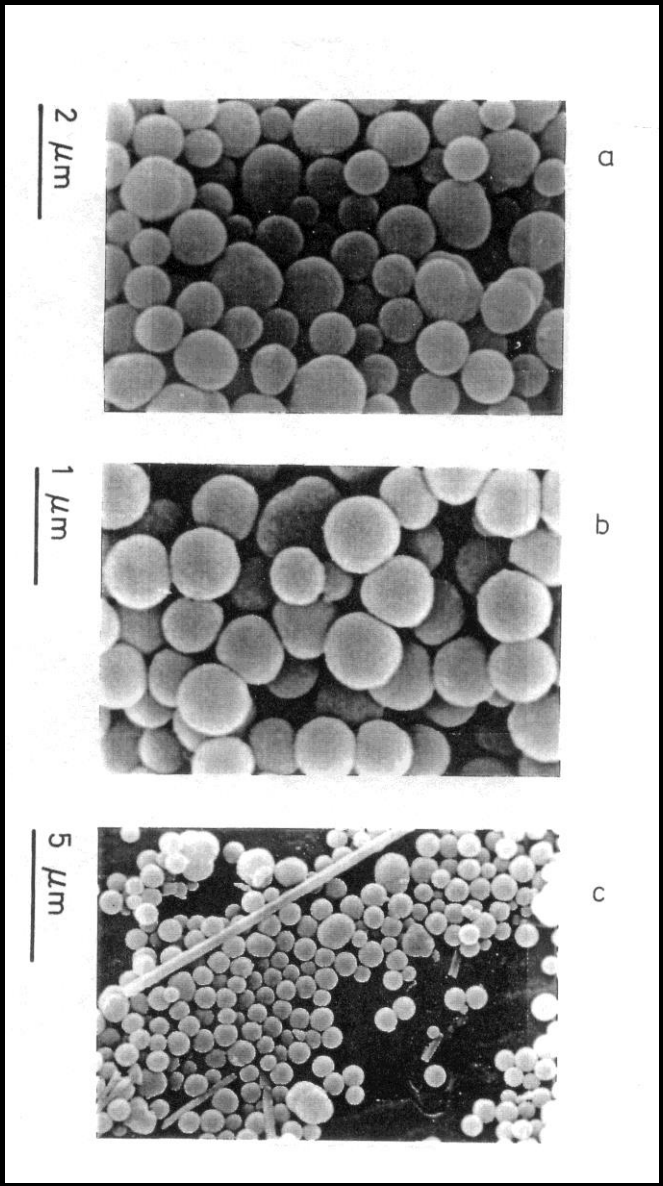
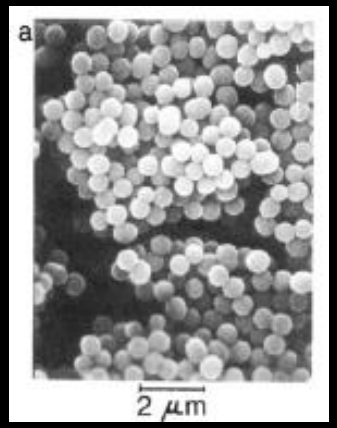


Partículas obtenidas a partir de soluciones de $Cu(NO_3)_2$ alcalinizadas por hidrólisis de urea

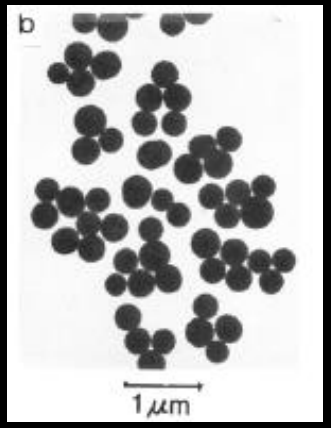
a) [Cu(II)] = 0,0025 M

b) [Cu(II)] = 0,0050 M

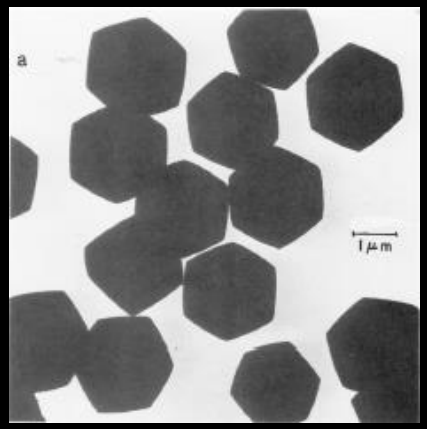
c) [Cu(II)] = 0,010 M



Aluminum (hydrous) oxide obtained by aging at 105 ^{o}C for 24 h a solution of 2.0×10^{-3} M $Al(NO_3)_3$ and 3.0×10^{-3} M $(NH_4)_2SO_4$



Chromium (hydrous) oxide particles obtained by aging at 75 ^{o}C for 24 h a solution of 4.0×10^{-3} M $CrK(SO_4)_2.12H_2O$

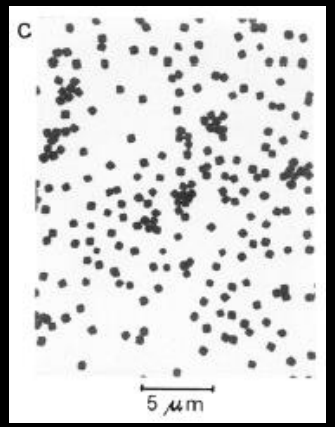


Ferric basic sulfate (alunite), $Fe_3(SO_4)_2(OH)_5.2H_2O$, particles obtained by aging at 98 ^{o}C for 2.8 h an 8.8×10^{-2} M aqueous ferric sulfate solution

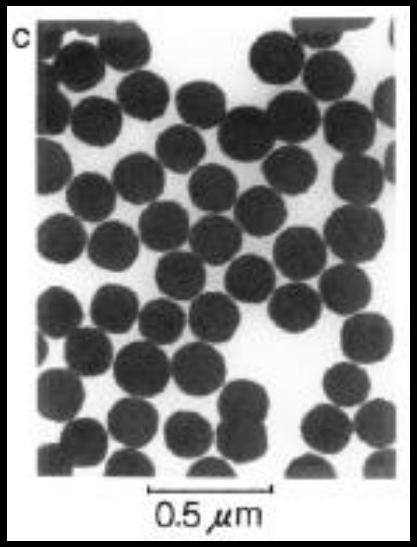
Akageneite (β-FeOOH) particles obtained by aging at 100 ^{o}C for 24 h a solution of $4.5x10^{-3}$ M $FeCl_3$ and 0.01 M HCl

Hematite particles obtained by aging at 100 ^{o}C for 10 days a solution of $3.0x10^{-2}$ M $FeCl_3$, and $5.0x10^{-3}$ M HCl

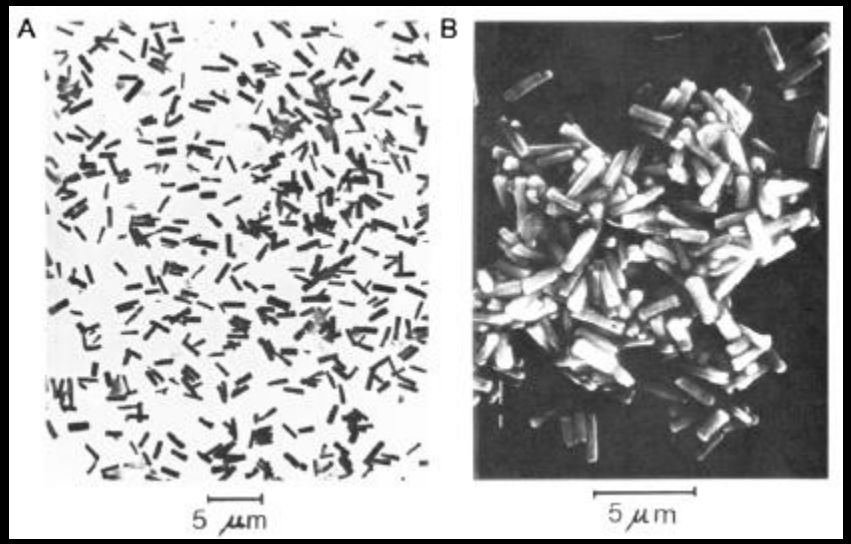
Hematite (α-Fe_2O_3) particles obtained by aging at 100 ^{o}C for 48 h a solution of $2.0x10^{-2}$ M $FeCl_3$ and $3.0x10^{-4}$ M NaH_2PO_4



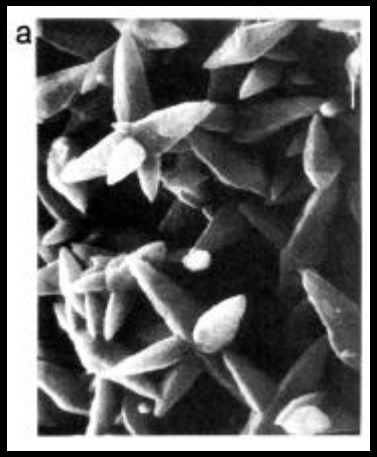
Hematite (α-Fe_2O_3) particles obtained by aging at 100 ^{o}C for 16 h a solution of $1.9x10^{-2}$ M $FeCl_3$, $1.2x10^{-3}$ M HCl, and 40% (vol) ethanol



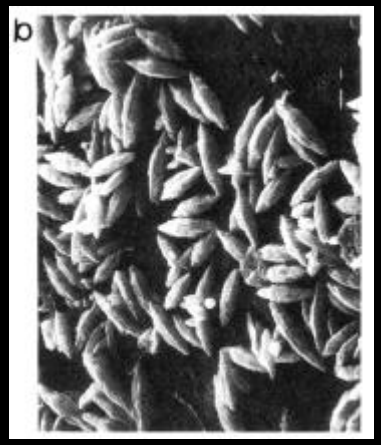
Cerium(IV) oxide particles obtained by aging at 90 ^{o}C for 48 h a solution of $1.2x10^{-3}$ M $Ce(SO_4)_2$ and $8.0x10^{-2}$ M H_2SO_4



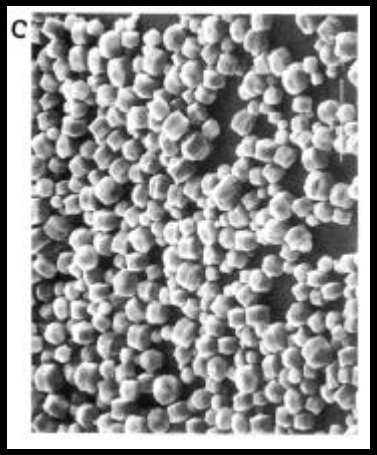
Rodlike particles obtained by aging at 90 ^{o}C for 12 h a $2.5x10^{-3}$ M $Ce(SO_4)_2$, 0.01 M H_2SO_4 and 0.4 M Na_2SO_4 solution



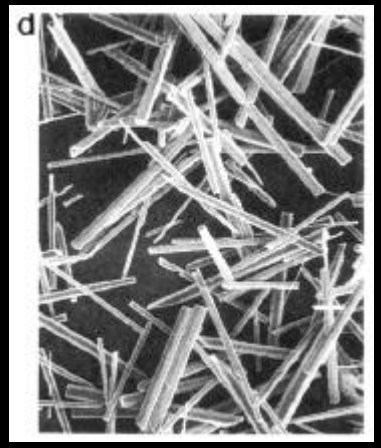
ZnO (zincite) particles obtained by aging 5.0×10^{-3} M $Zn(NO_3)_2$ and 1.9×10^{-2} M NH_4OH (pH 7.7) at 90 ^{o}C for 3 h



ZnO (zincite) particles obtained by aging 1.0×10^{-4} M $Zn(NO_3)_2$ and 3.2×10^{-2} M NH_4OH (pH 8.8) at 90 ^{o}C for 1 h

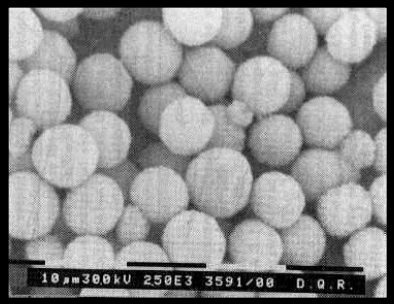


ZnO particles obtained by aging 3.2×10^{-3} M $Zn(NO_3)_2$ and 0.1 M triethanolamine (TEA), pH 8.9, at 90 ^{o}C for 1 h

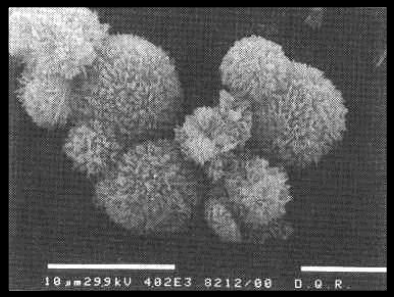


ZnO particles obtained by aging a 4.0×10^{-2} M $Zn(NO_3)_2$, 0.2 M triethanolamine (TEA), and 1.2 M NaOH (pH 12.1) at 150 ^{o}C for 2 h

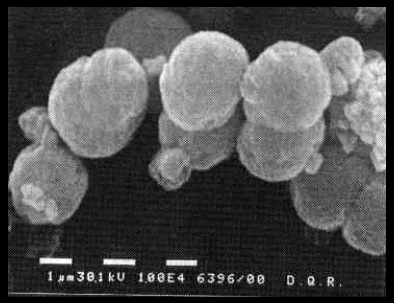
am-$Cu_2(OH)_2CO_3.xH_2O$



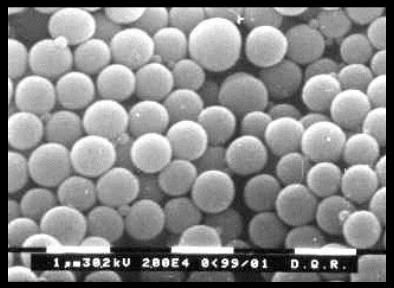
$Zn_5(OH)_6(CO_3)_2$



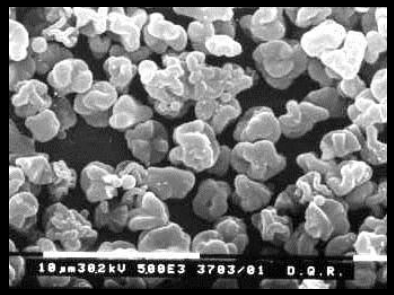
$(Zn,Cu)_5(OH)_6(CO_3)_2$



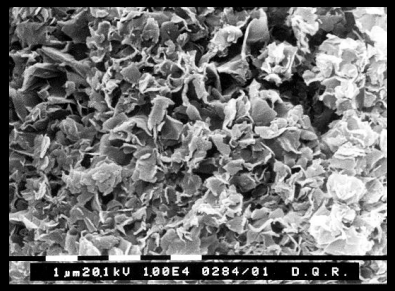
$Cu_{(1-x)}Zn_xO$



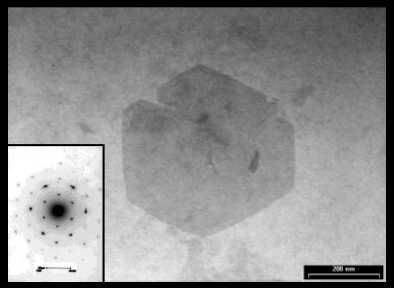
$Gd_2Cu(OH)_4(CO_3)_3.xH_2O$



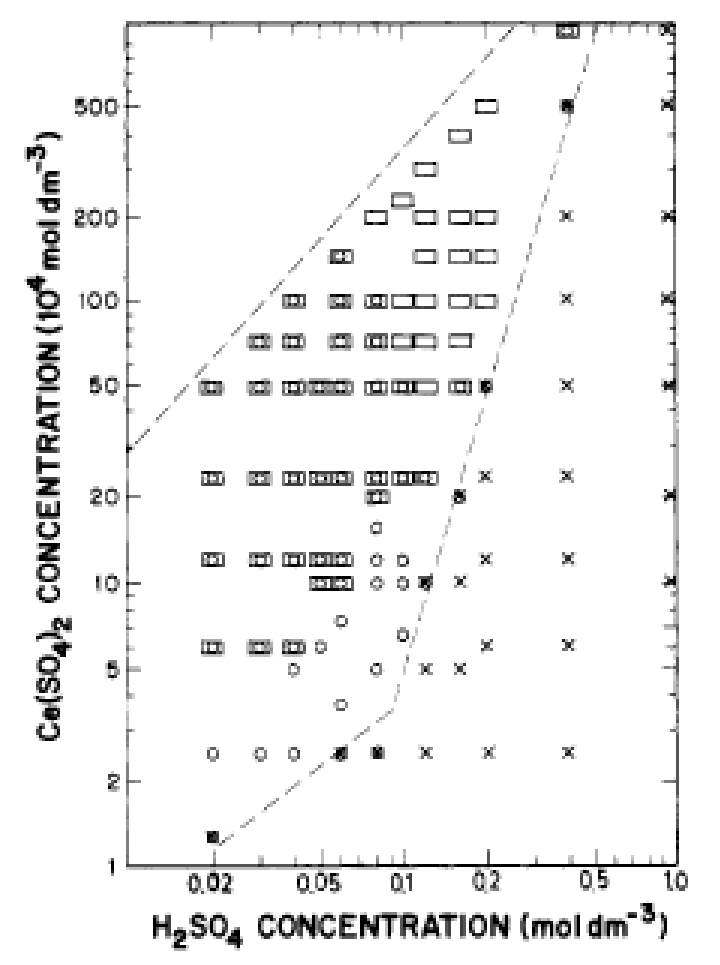
$Gd_2Cu(OH)_4(CO_3)_3.xH_2O$



α-$Ni_{(1-x)}Cu_x(OH)_2\ xH_2O$



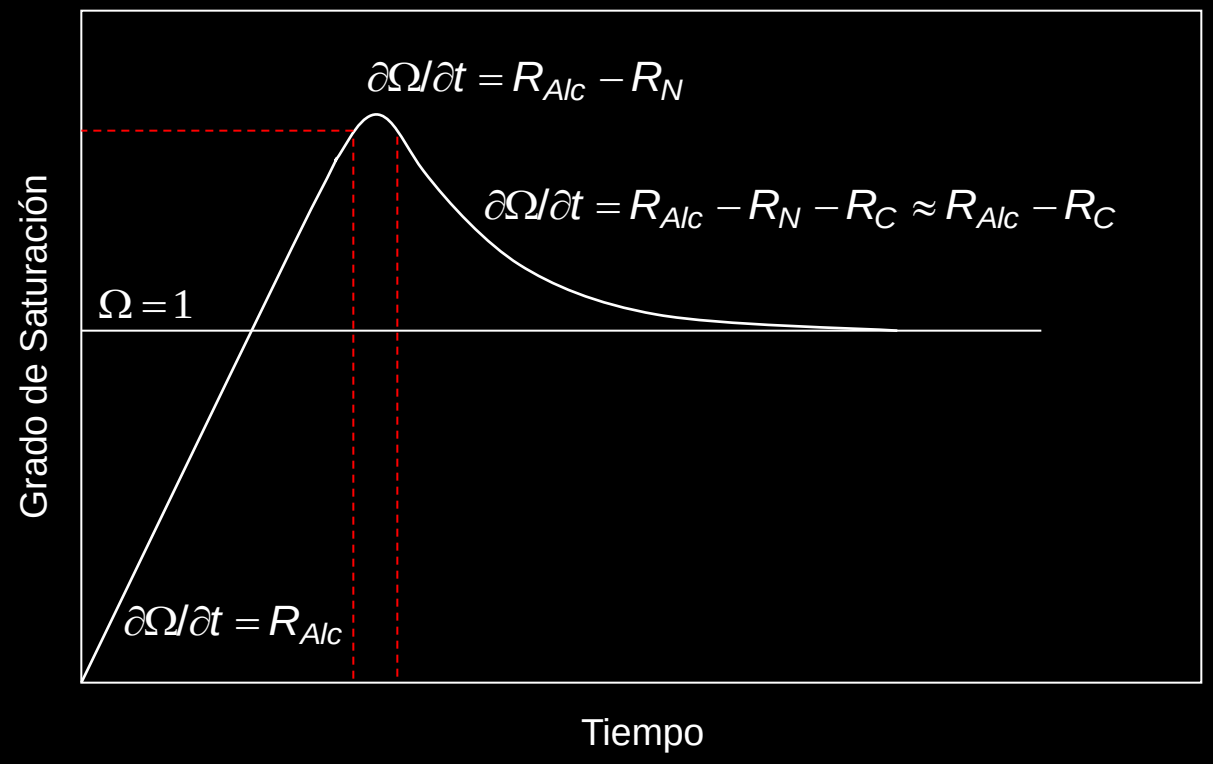
$Ni_{0.68}Cr_{0.32}(OH)_2(CO_3)_{0.16}.xH_2O$



Precipitation domain for solutions containing $Ce(SO_4)_2$ and H_2SO_4 aged at 90 ^oC for 12 h. Symbols designating different kinds of particles: ○ spheres; □ rods; □○ rods mixed with spheres; ☹ a very small amount of spheres; X no particle formation.

Síntesis de partículas uniformes *(tamaño y forma)*

Requisito: Separar las etapas de nucleación y crecimiento



Se logra mediante un adecuado control de la velocidad con que aumenta el grado de sobresaturación

Requisitos para la Síntesis de Partículas Monodispersas.
Conocer:

- Solubilidad (*termodinámica*)

$$x\,M(OH)_z\,(s) + (xz\text{-}y)\,H^+ = M_x(OH)_y^{(zx-y)+} + (xz\text{-}y)\,H_2O$$

- Hidrólisis (*cinética*)

$$M(H_2O)_n^{z+} + H_2O^* \longrightarrow M(H_2O)_{n-1}H_2O^{*z+} + H_2O$$

$$M(H_2O)_{n-1}H_2O^{z+} + OH^- \longrightarrow M(H_2O)_{n-1}OH^{(z-1)+} + H_2O$$

$$M(H_2O)_{n-1}OH^{(z-1)+} + OH^- \longrightarrow M(H_2O)_{n-2}(OH)_2^{(z-2)+} + H_2O$$

Equilibrios de Solubilidad

$$M(OH)_z(s) + zH^+ = M^{z+} + zH_2O; \quad K_{S0} = \frac{[M^{z+}]}{[H^+]^z}$$

$$K_{S0} = \frac{K_{PS}}{K_W{}^z}$$

Equilibrios de Hidrólisis

$$M^{z+} + H_2O = MOH^{(z-1)+} + H^+; \quad K_1 = \frac{[MOH^{(z-1)+}][H^+]}{[M^{z+}]}$$

$$M^{z+} + 2H_2O = M(OH)_2^{(z-2)+} + 2H^+; \quad K_2 = \frac{[M(OH)_2^{(z-2)+}][H^+]^2}{[M^{z+}]}$$

. . . . . .

$$xM^{z+} + yH_2O = M_X(OH)_y^{(zx-y)+} + yH^+; \quad K_{x,y} = \frac{[M_X(OH)_y^{(zx-y)+}][H^+]^y}{[M^{z+}]^x}$$

Balance de Masa

$$S = \sum x[M_X(OH)_y^{(zx-y)+}]$$

Balance de Carga

$$0 = C_B - z_A C_A + [H^+] - [OH^-] + \sum (zx - y)[M_X(OH)_y^{(zx-y)+}]$$

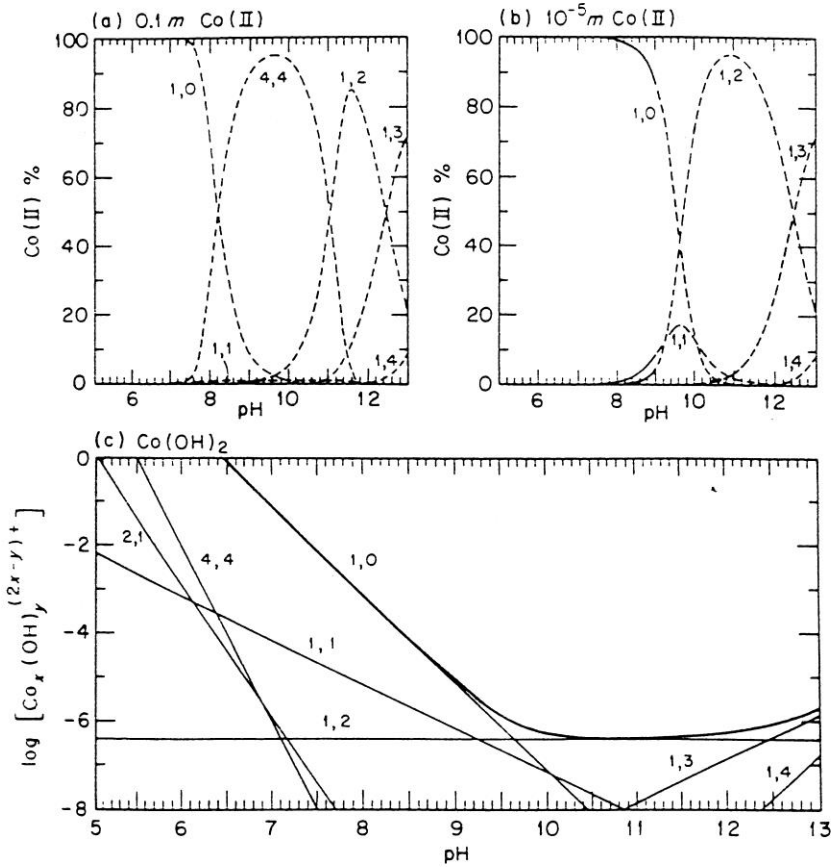


Fig. 10.14. Distribution of hydrolysis products (x,y) at $I = 1\ m$ and 25° in (*a*) 0.1 *m* Co(II), (*b*) 10^{-5} *m* Co(II), and (*c*) solutions saturated with respect to $Co(OH)_2$. The dashed curves in *a* and *b* denote regions supersaturated with respect to $Co(OH)_2$; the heavy curve in *c* is the total concentration of Co(II).

$$Co^{II}_{X}(OH)_{y}^{(zx-y)+}$$

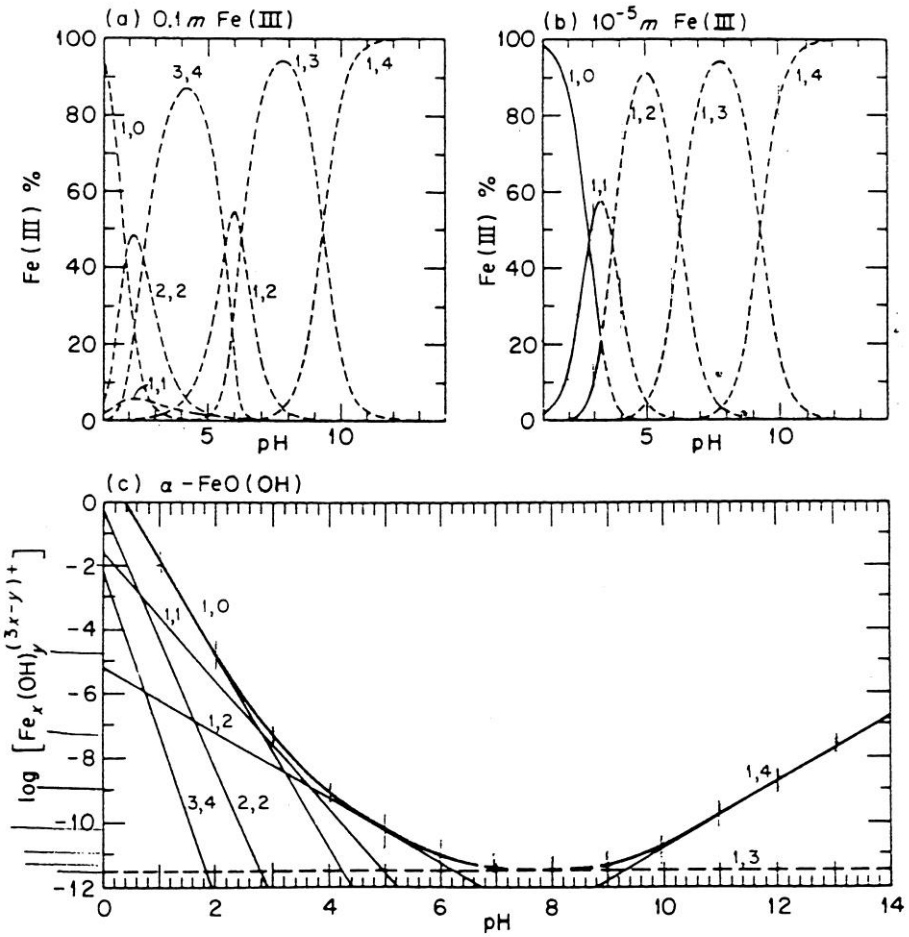


Fig. 10.13. Distribution of hydrolysis products (x,y) at $I = 1$ m and 25° in (*a*) 0.1 m Fe(III), (*b*) 10^{-5} m Fe(III), and (*c*) solutions saturated with α-FeO(OH). The dashed curves in *a* and *b* denote regions supersaturated with respect to α-FeO(OH); the heavy curve in *c* is the total concentration of Fe(III).

$$Fe^{III}_{X}(OH)_{y}^{(zx-y)+}$$

$Sn^{II}_{X}(OH)_{y}^{(zx-y)+}$

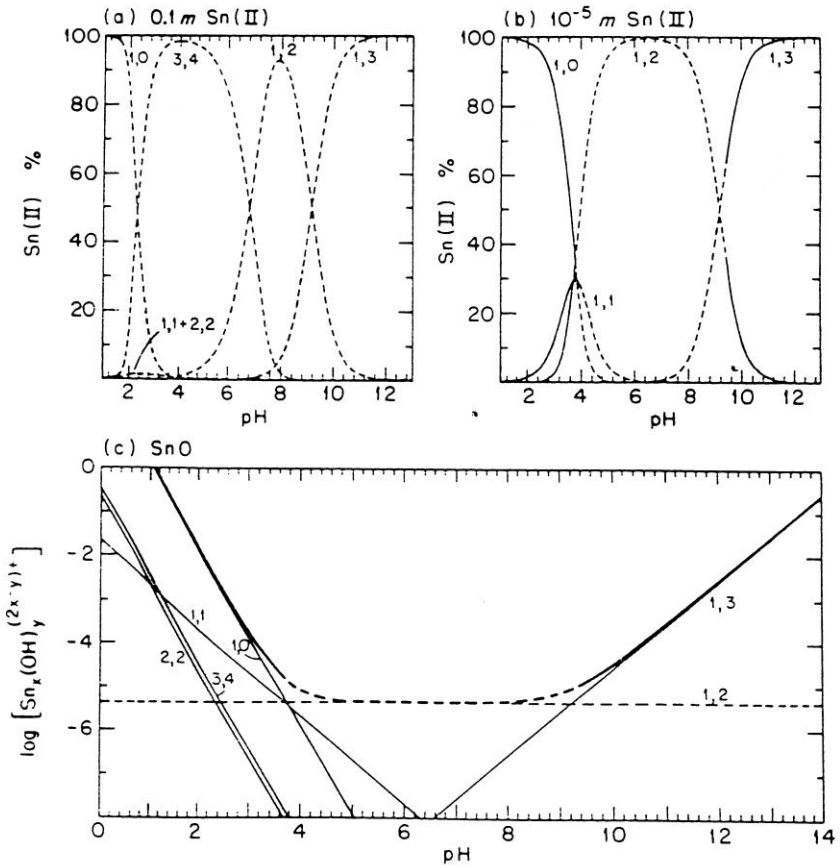


Fig. 15.6. Distribution of hydrolysis products (x,y) at $I = 1\ m$ and 25° in (*a*) 0.1 *m* Sn(II), (*b*) 10^{-5} *m* Sn(II), and (*c*) solutions saturated with SnO. The dashed curves in *a* and *b* denote regions supersaturated with respect to SnO; the heavy curve in *c* is the total concentration of tin(II). The minimum is dashed because of the uncertainty with which the $Sn(OH)_2$ species is known.

$Cr^{III}_{X}(OH)_{y}^{(zx-y)+}$

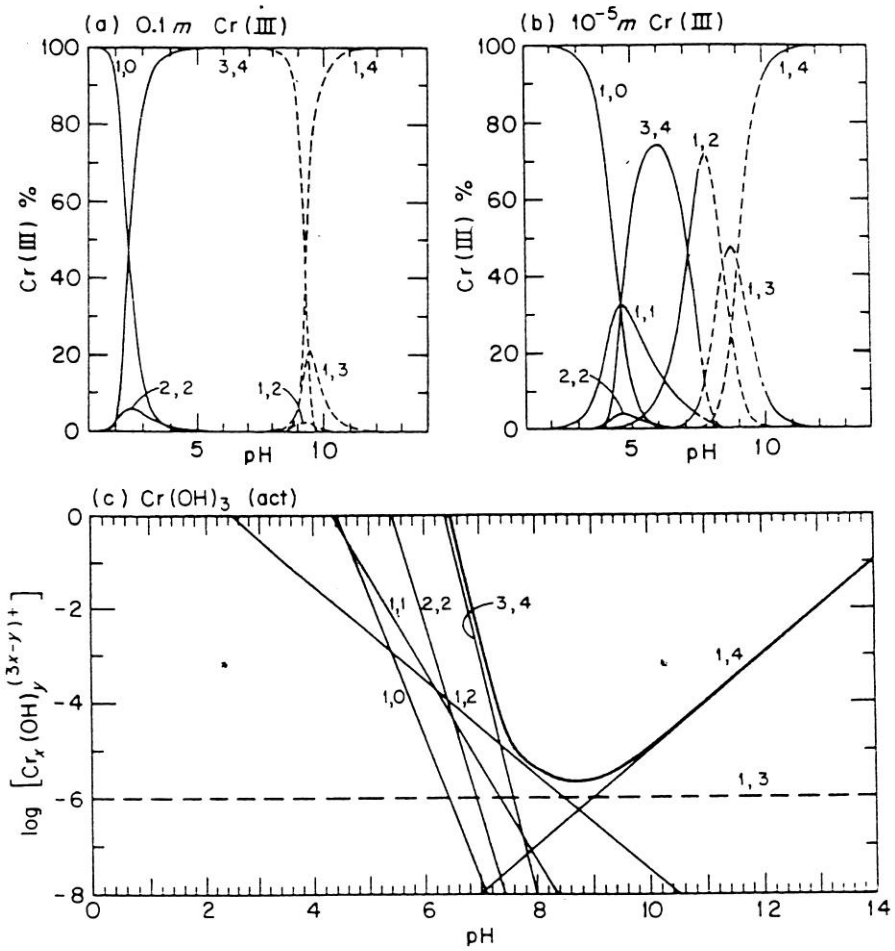


Fig. 10.8. Distribution of hydrolysis products (x,y) at $I = 1\ m$ and 25° in (*a*) 0.1 *m* Cr(III), (*b*) 10^{-5} *m* Cr(III), and (*c*) solutions saturated with $Cr(OH)_3$. The dashed curves in *a* and *b* denote regions supersaturated with respect to $Cr(OH)_3$; the heavy curve in *c* is the total concentration of chromium(III).

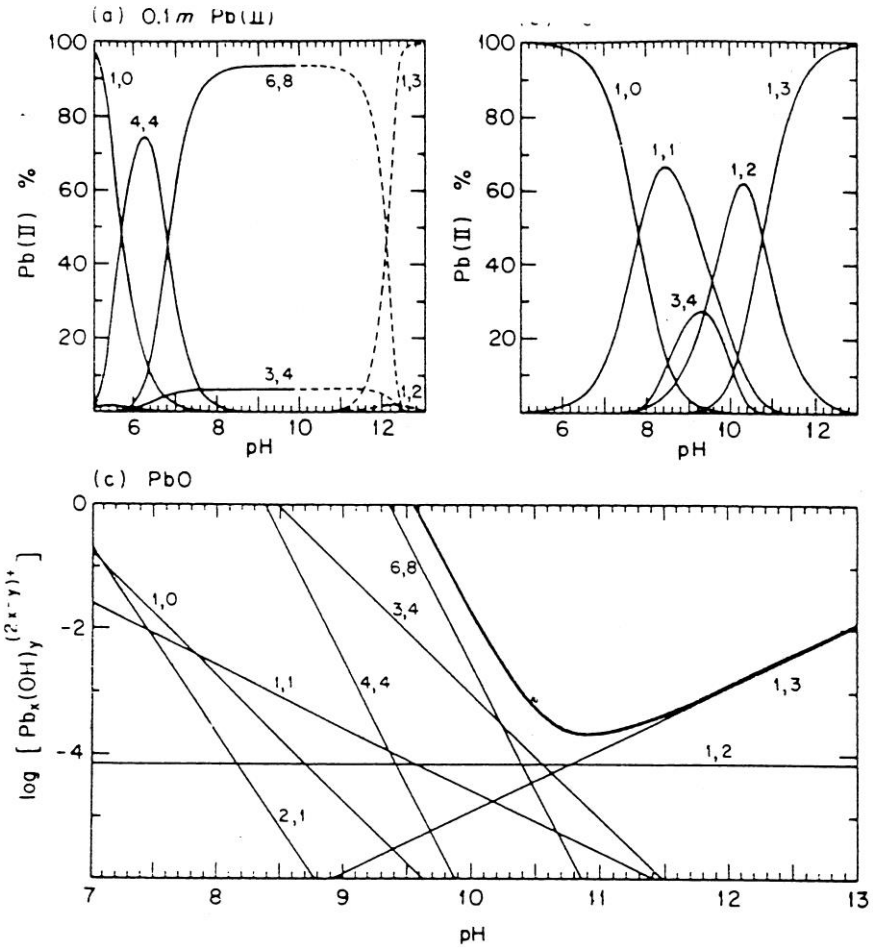


Fig. 15.9. Distribution of hydrolysis products (x,y) at I = 1 m and 25° in (a) 0.1 m Pb(II), (b) 10^{-5} m Pb(II), and (c) solutions saturated with PbO. The dashed curves in a and b denote regions supersaturated with respect to PbO; the heavy curve in c is the total concentration of lead(II).

$$Pb^{II}_{X}(OH)_{y}^{(zx-y)+}$$

Equilibrios de Solubilidad

$$M(OH)_z(s) + zH^+ = M^{z+} + zH_2O; \quad K_{S0} = \frac{[M^{z+}]}{[H^+]^z}$$

$$K_{S0} = \frac{K_{PS}}{K_W{}^z}$$

Equilibrios de Hidrólisis

$$M^{z+} + H_2O = MOH^{(z-1)+} + H^+; \quad K_1 = \frac{[MOH^{(z-1)+}][H^+]}{[M^{z+}]}$$

$$M^{z+} + 2H_2O = M(OH)_2^{(z-2)+} + 2H^+; \quad K_2 = \frac{[M(OH)_2^{(z-2)+}][H^+]^2}{[M^{z+}]}$$

. . . . . .

$$xM^{z+} + yH_2O = M_X(OH)_y^{(zx-y)+} + yH^+; \quad K_{x,y} = \frac{[M_X(OH)_y^{(zx-y)+}][H^+]^y}{[M^{z+}]^x}$$

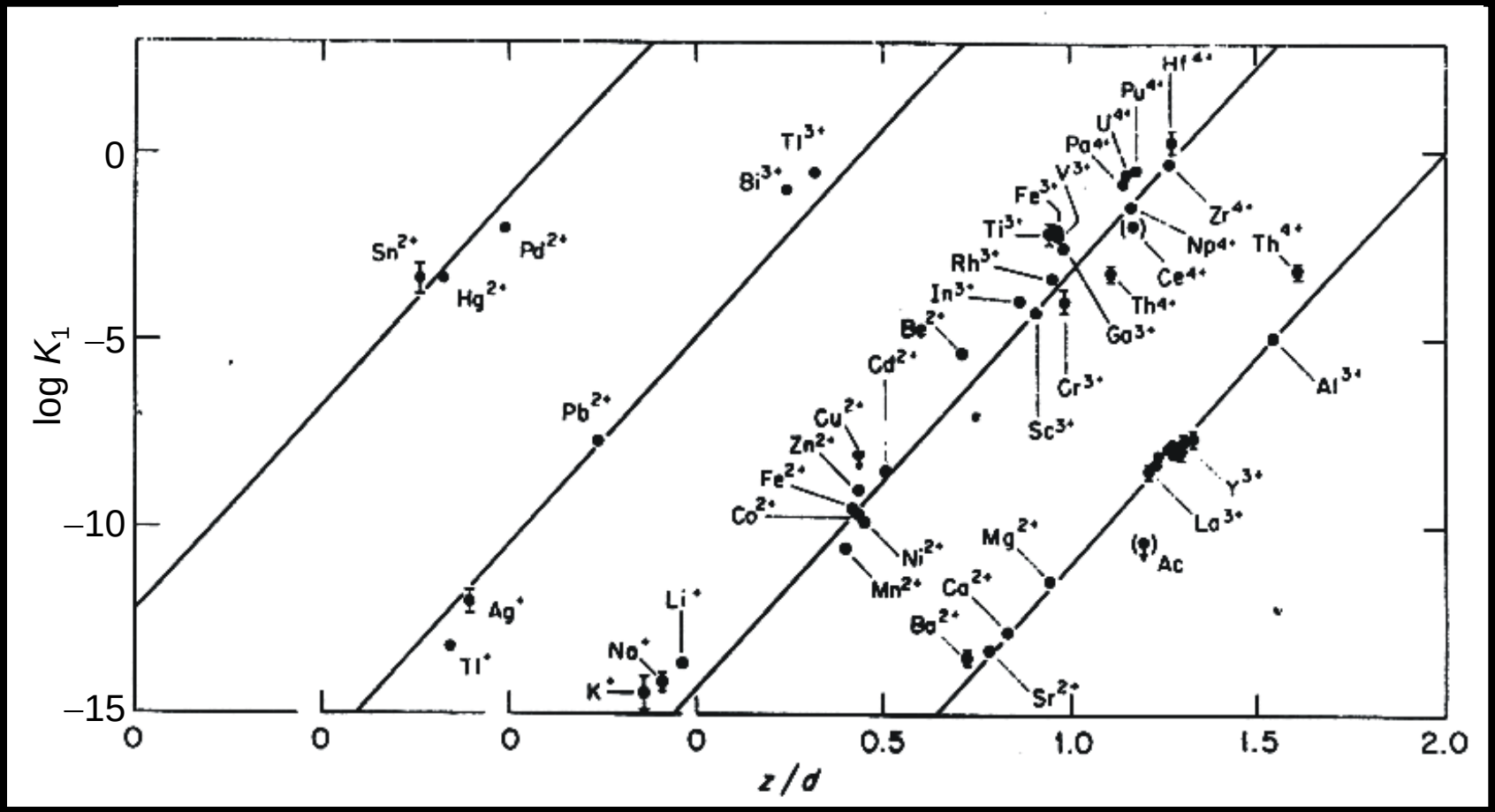
Balance de Masa

$$S = \sum x[M_X(OH)_y^{(zx-y)+}]$$

Balance de Carga

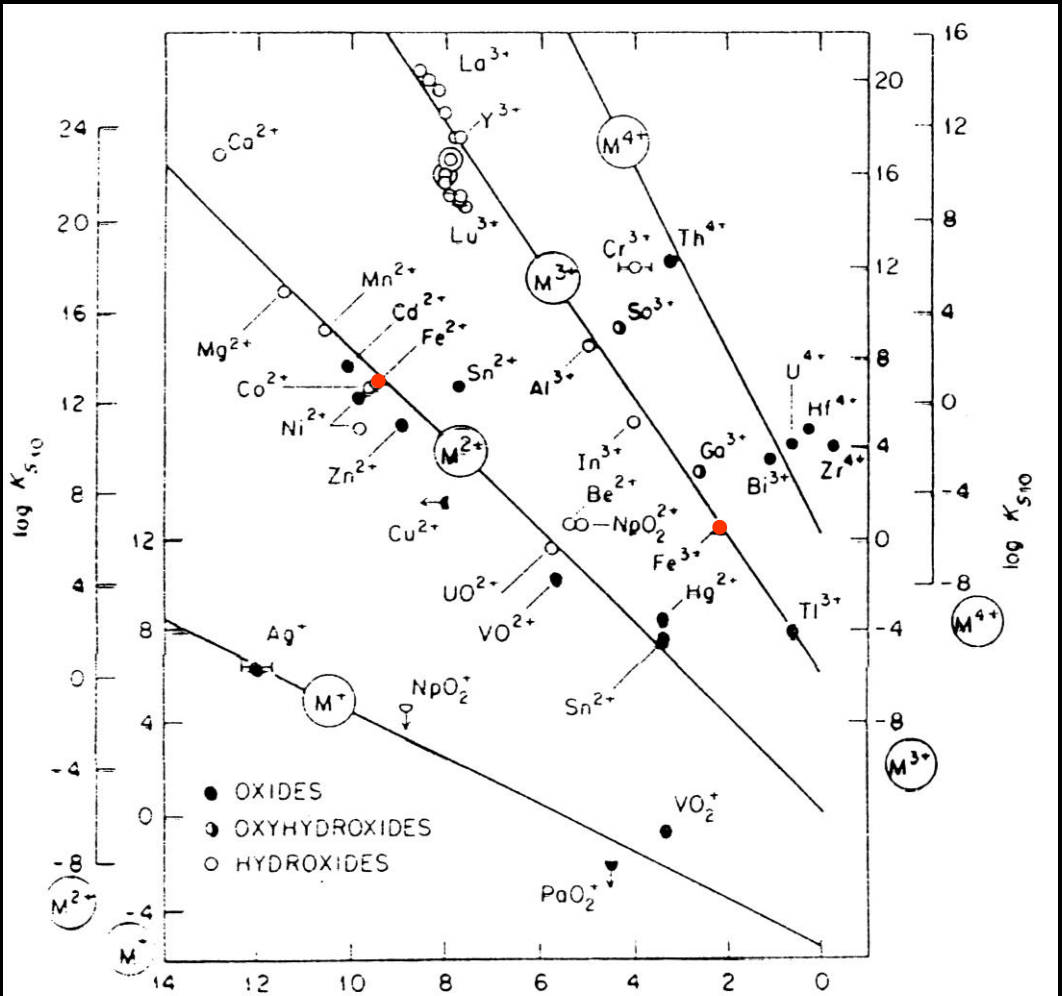
$$0 = C_B - z_A C_A + [H^+] - [OH^-] + \sum (zx - y)[M_X(OH)_y^{(zx-y)+}]$$

Dependencia de K_1 con la relación carga-radio



$$\log K_1 = A + 11 \times (z/d)$$

Relación Hidrólisis y Solubilidad: *Relación entre K_1 y K_{S0}*



Equilibrios de Solubilidad

$$M(OH)_z(s) + zH^+ = M^{z+} + zH_2O; \quad K_{S0} = \frac{[M^{z+}]}{[H^+]^z}$$

$$K_{S0} = \frac{K_{PS}}{K_W{}^z}$$

Equilibrios de Hidrólisis

$$M^{z+} + H_2O = MOH^{(z-1)+} + H^+; \quad K_1 = \frac{[MOH^{(z-1)+}][H^+]}{[M^{z+}]}$$

$$M^{z+} + 2H_2O = M(OH)_2^{(z-2)+} + 2H^+; \quad K_2 = \frac{[M(OH)_2^{(z-2)+}][H^+]^2}{[M^{z+}]}$$

. . . . . .

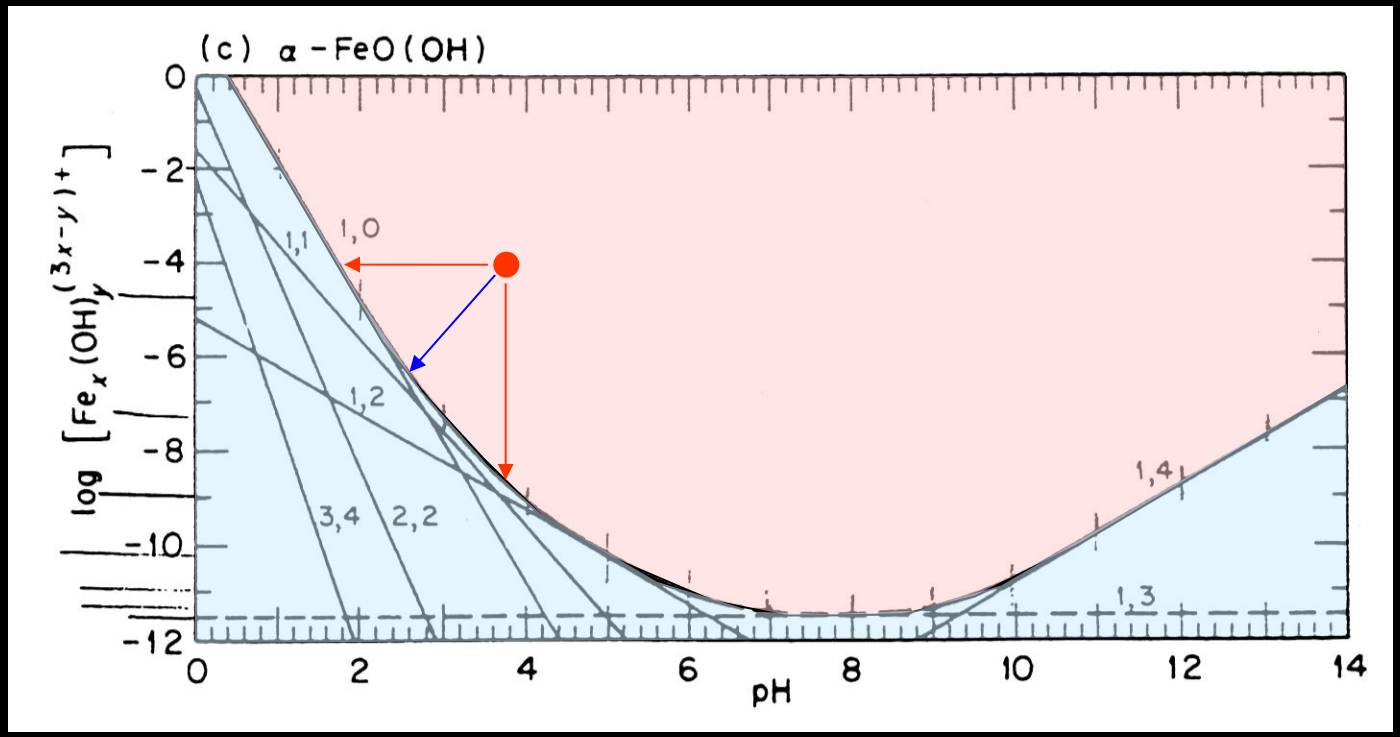
$$xM^{z+} + yH_2O = M_X(OH)_y^{(zx-y)+} + yH^+; \quad K_{x,y} = \frac{[M_X(OH)_y^{(zx-y)+}][H^+]^y}{[M^{z+}]^x}$$

Balance de Masa

$$S = \sum x[M_X(OH)_y^{(zx-y)+}]$$

Balance de Carga

$$0 = C_B - z_A C_A + [H^+] - [OH^-] + \sum (zx - y)[M_X(OH)_y^{(zx-y)+}]$$



$$M^{z+}(aq) + z\ OH^{-}(aq) \longrightarrow M(OH)_z(s);\ 1/K_{PS}$$

$$\overline{\Delta G} = \overline{\Delta G}^{o} - RT\ln(a_{M^{z+}} a^{z}_{OH^{-}}) = -RT\ln(a_{M^{z+}} a^{z}_{OH^{-}})/K_{PS}$$

$$\overline{\Delta G} = -RT\ln\Omega$$

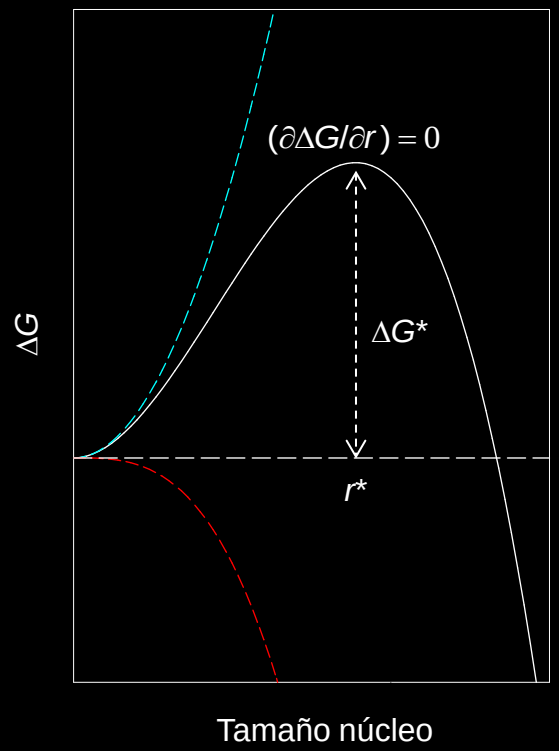
Nucleación

$$M^{z+}(aq) + zOH^{-}(aq) \longrightarrow M(OH)_z(s);\ 1/K_{PS}$$

$$\Delta G = n\overline{\Delta G} = -nRT\ln\Omega\ +\ A\gamma$$

$$\Delta G = -\frac{4\pi r^3}{3\overline{V}}RT\ln\Omega\ +\ 4\pi r^2\gamma$$

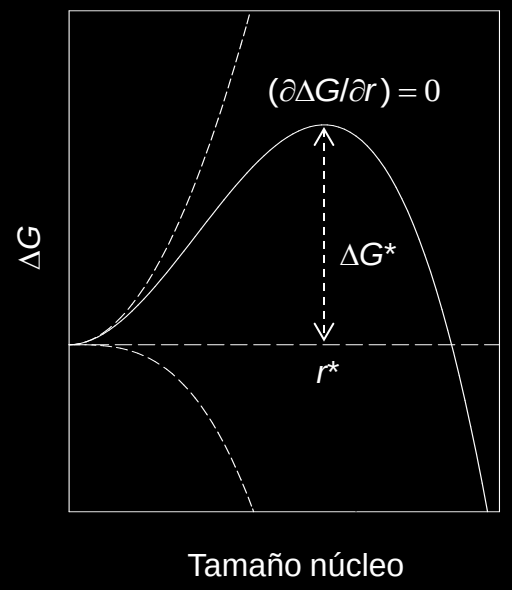
$$\Delta G^* = \frac{16\pi\overline{V}^2\gamma^3}{3(RT\ln\Omega)^2} \qquad r^* = \frac{2\gamma\overline{V}}{RT\ln\Omega}$$



Velocidad de Nucleación

ΔG^* es una barrera cinética a la formación de núcleos

La nucleación puede verse como un proceso activado



$R_N \propto \exp(-\Delta G^*/RT)$	probabilidad de nucleación (fluctuación)
$R_N \propto D = RT/6\pi r_i \eta$	velocidad de encuentros
$R_N \propto \nu_o$	frecuencia encuentros exitosos
$R_N \propto n_0$	número de centros de nucleación

$$R_N = k_N^{'} n_0 \exp(-\Delta G^*/RT)$$

$$\Delta G^* = \frac{16\pi \overline{V}^2 \gamma^3}{3(RT \ln \Omega)^2}$$

Velocidad de Nucleación

ΔG^* es una barrera cinética a la formación de núcleos

La nucleación puede verse como un proceso activado

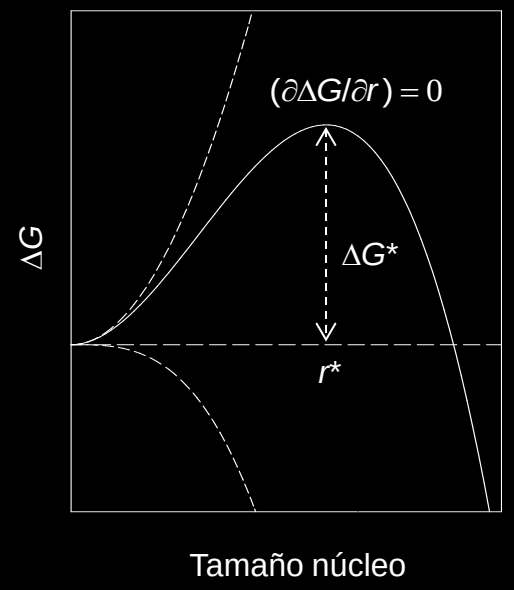
$R_N \propto \exp(-\Delta G^*/RT)$	probabilidad de nucleación (fluctuación)
$R_N \propto D = RT/6\pi r_i \eta$	velocidad de encuentros
$R_N \propto \nu_o$	frecuencia encuentros exitosos
$R_N \propto n_0$	número de centros de nucleación

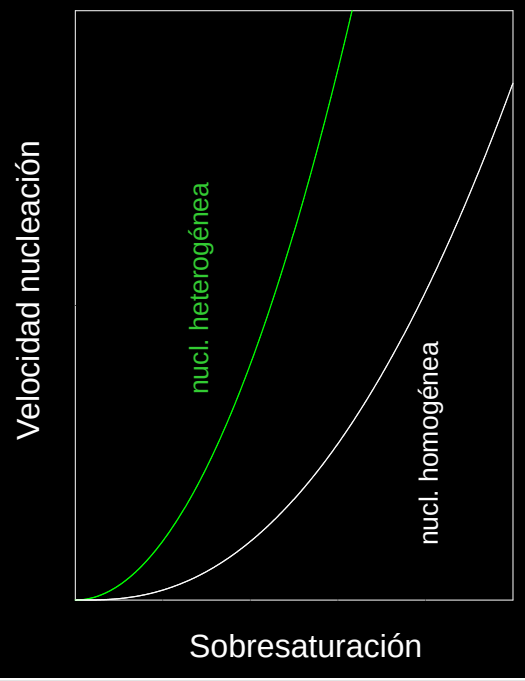
$$R_N = k_N^{'} n_0 \exp(-\Delta G^*/RT)$$

$$\Delta G^* = \frac{16\pi \overline{V}^2 \gamma^3}{3(RT \ln \Omega)^2}$$

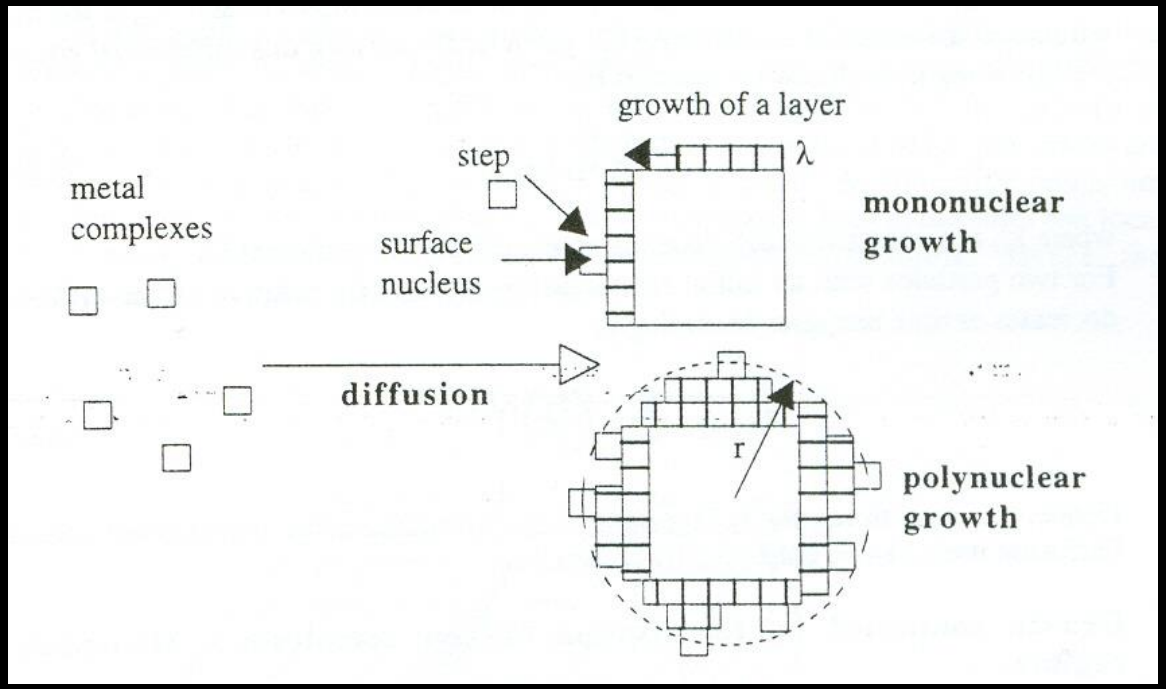
Nucleación Heterogénea

$$\Delta G^* = \frac{16\pi \overline{V}^2 \gamma^3}{3(RT \ln \Omega)^2}[1 - f(\theta)]$$





Crecimiento cristalino



Mecanismos:

- •Control difusional
- •Control superficial
  - •Régimen mononuclear (crecimiento bidimencional rápido)
  - •Régimen polinuclear (nucleación superficial rápida)
  - •Crecimiento espiralado (adición de unidades de crecimiento en dislocaciones)

Crecimiento cristalino

- Control difusional $R_C = \partial r / \partial t = k_D^{'}(\Omega - 1)/r$ supone que $r \approx \delta$

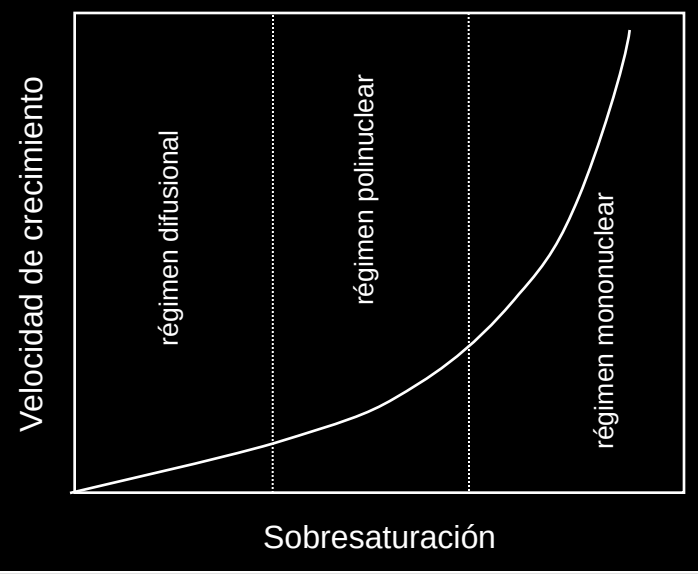
  δ (espesor de la película de difusión)

- Control superficial
  - Régimen mononuclear $R_C = k_M r^2 (\Omega - 1)^m$ proporcional al área
  - Régimen polinuclear $R_C = k_P (\Omega - 1)^{(m+2)/3}$ independiente del área

    m (ca. número de moléculas de precursor por núcleo)
  - Crecimiento espiralado $R_C = k_{BCF}[\nu(\Omega - 1)^2 / a]\ \tanh[\nu(\Omega - 1)/a]$

    ν (número de dislocaciones)

    a (constante)



Crecimiento cristalino?

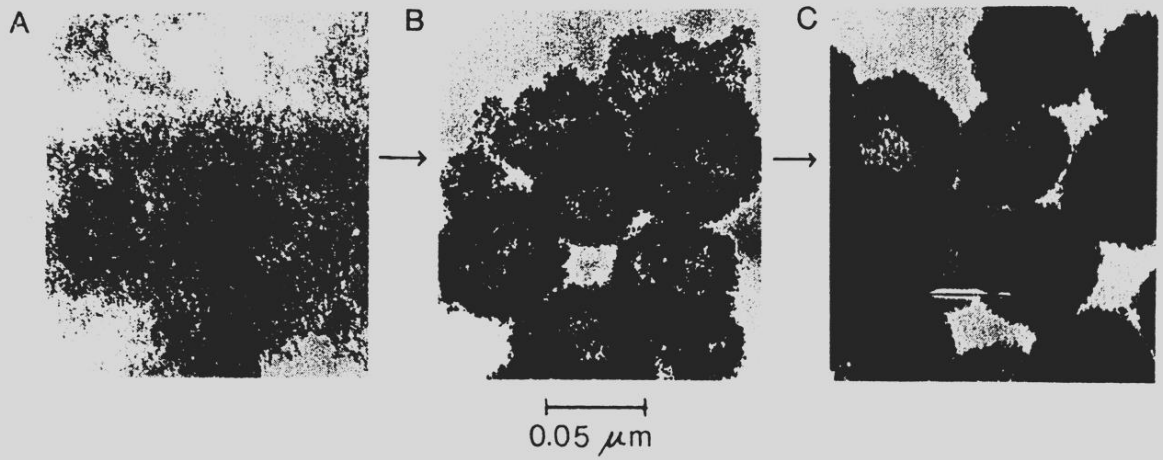
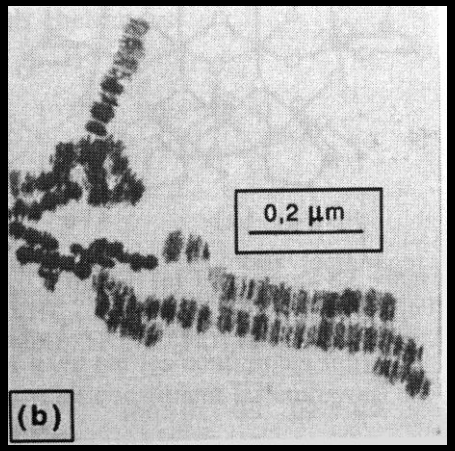
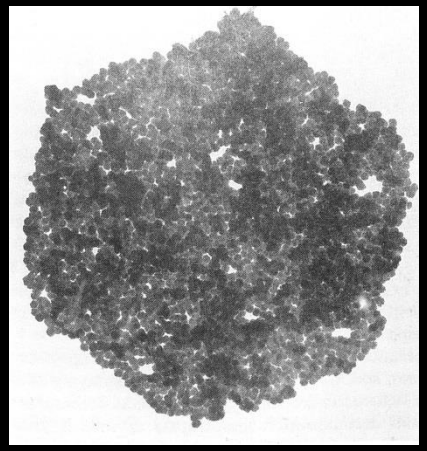


Figure 8. Formation of CeO_2 particles by forced hydrolysis of an acidic (4.0×10^{-2} mol dm^{-3} H_2SO_4) solution of $Ce(SO_4)_2$ (1.0×10^{-3} mol dm^{-3}) heated at 90 °C during a 6-h period.





Mecanismo de Precipitación

Iones metálicos

↓

Embriones

Hidrólisis:

olación
oxolación

↓

Núcleos

↓

Partículas primarias

Agregación

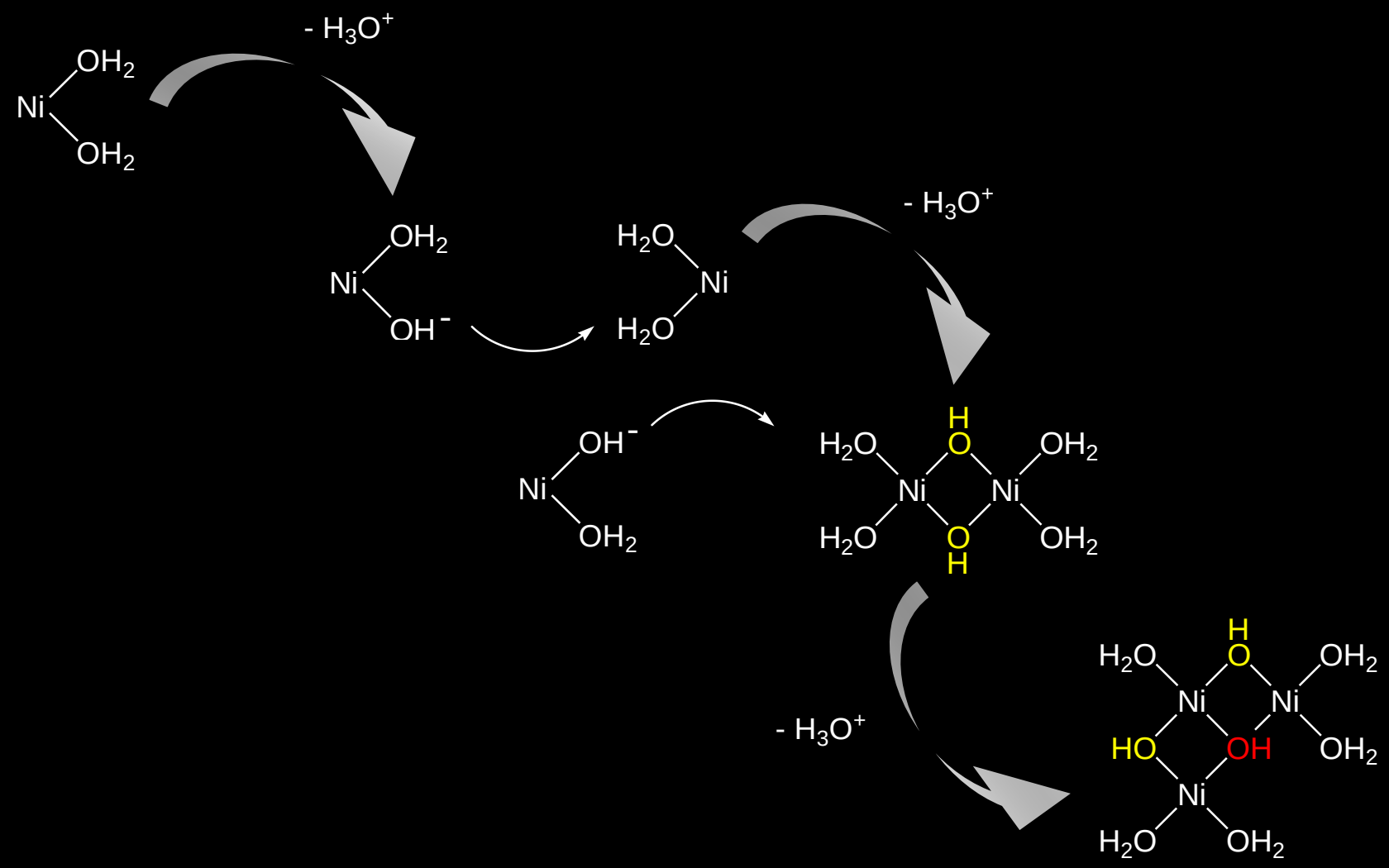
Crecimiento cristalino

Partículas

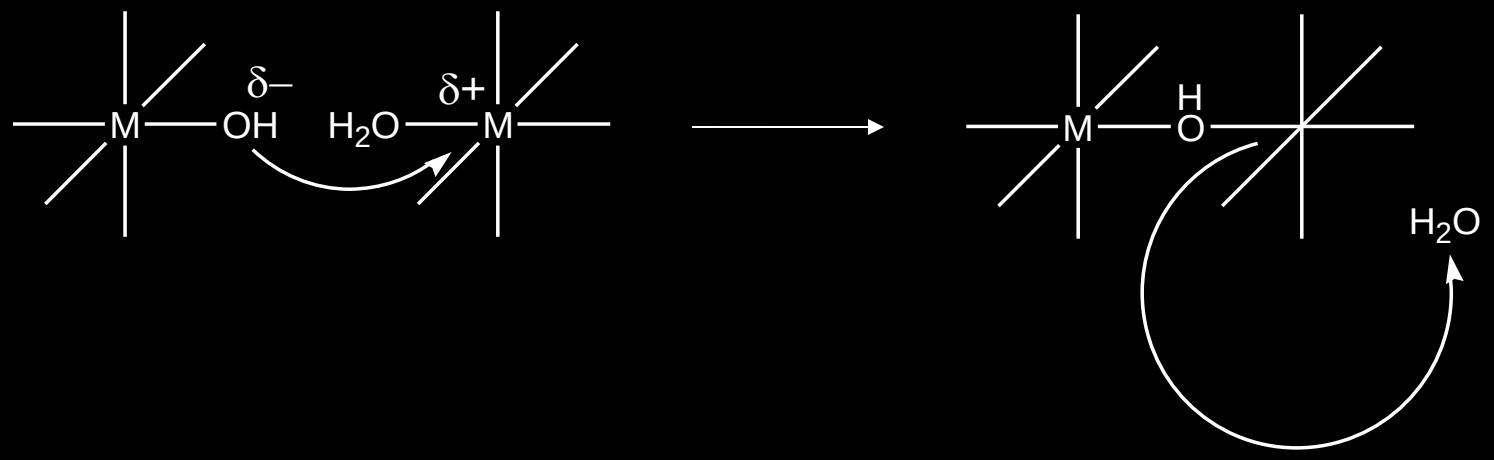
Partículas cristalinas

Disolución-reprecipitación

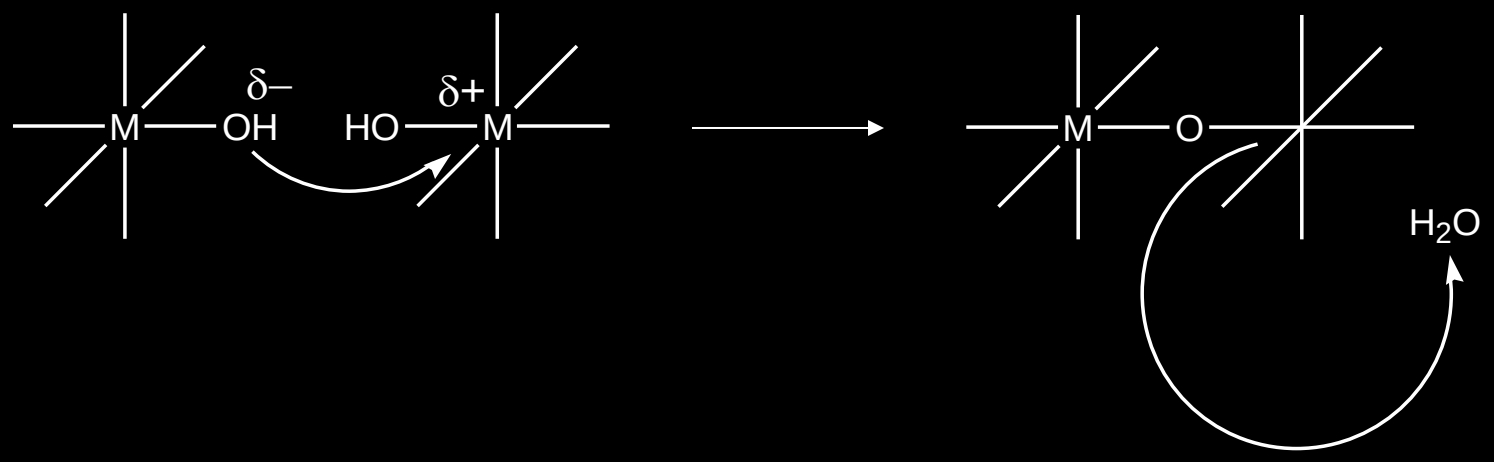
Nucleación *(Cascada de hidrólisis)*



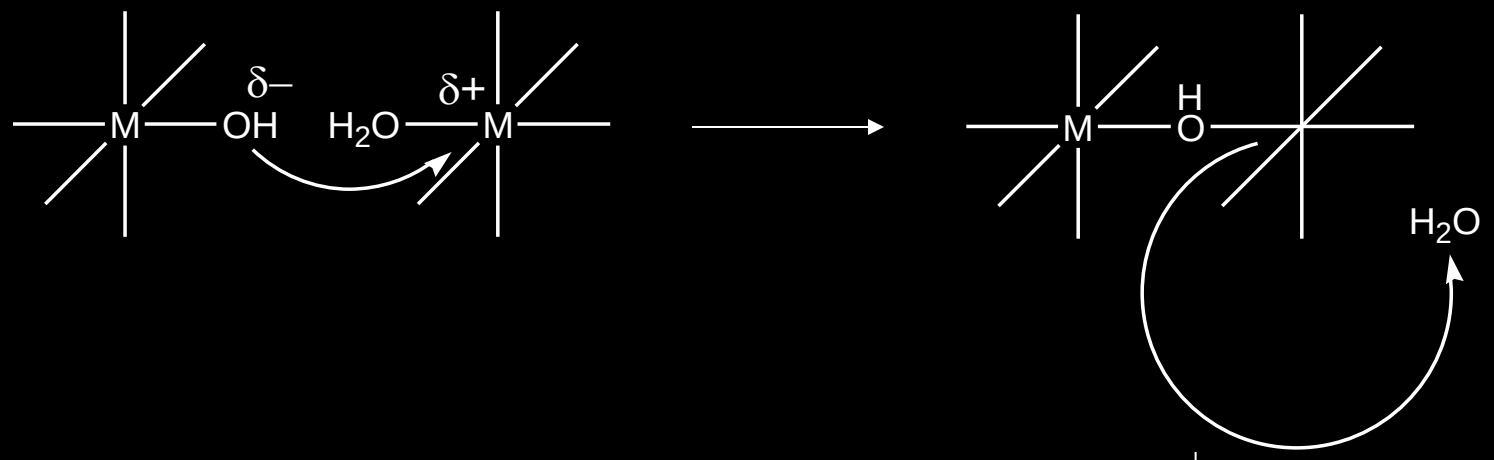
Olación



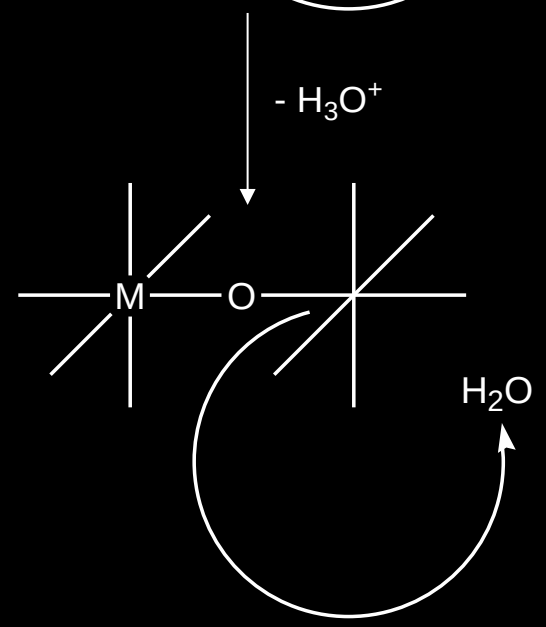
Oxolación



Olación

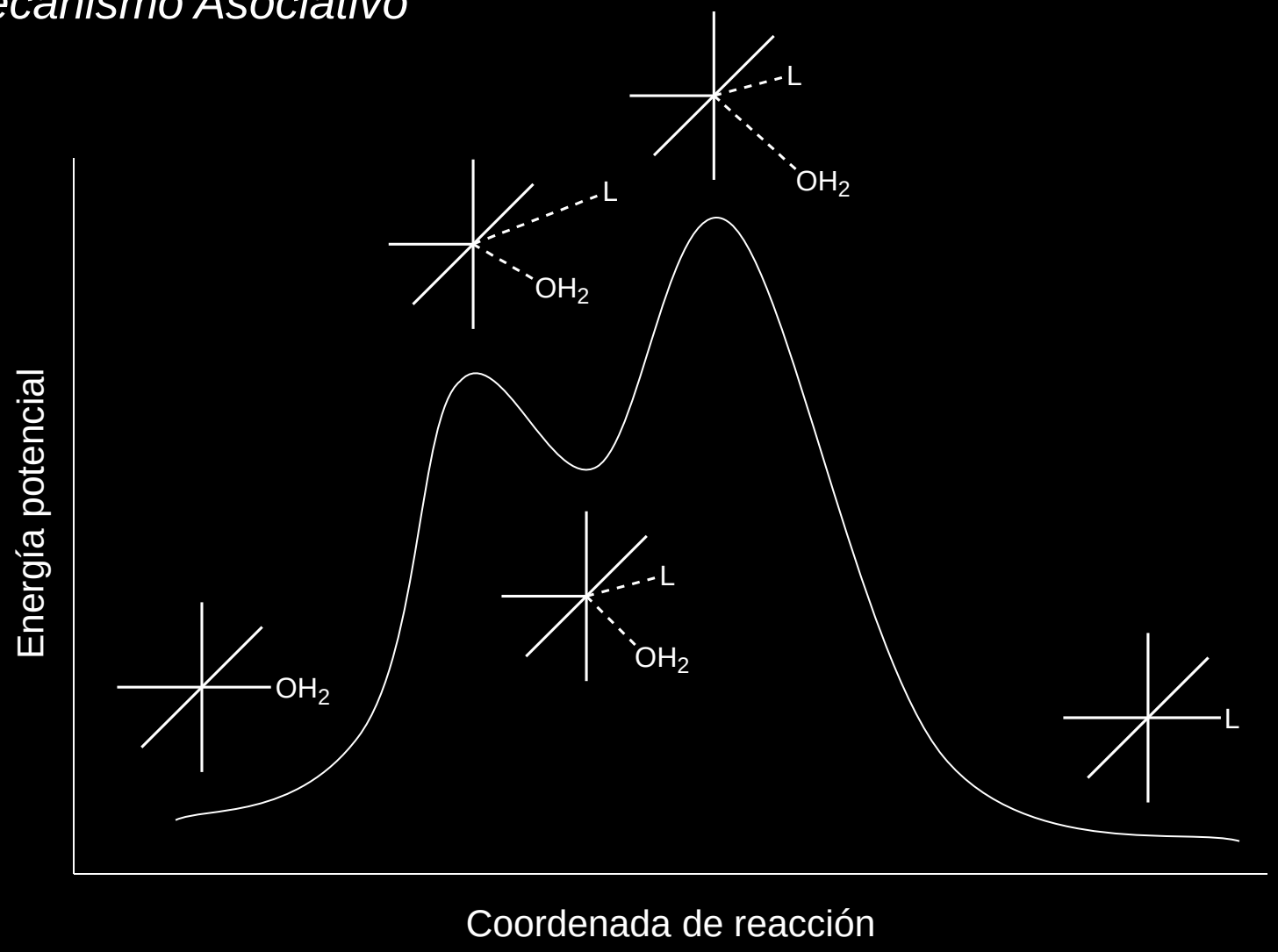


Oxolación



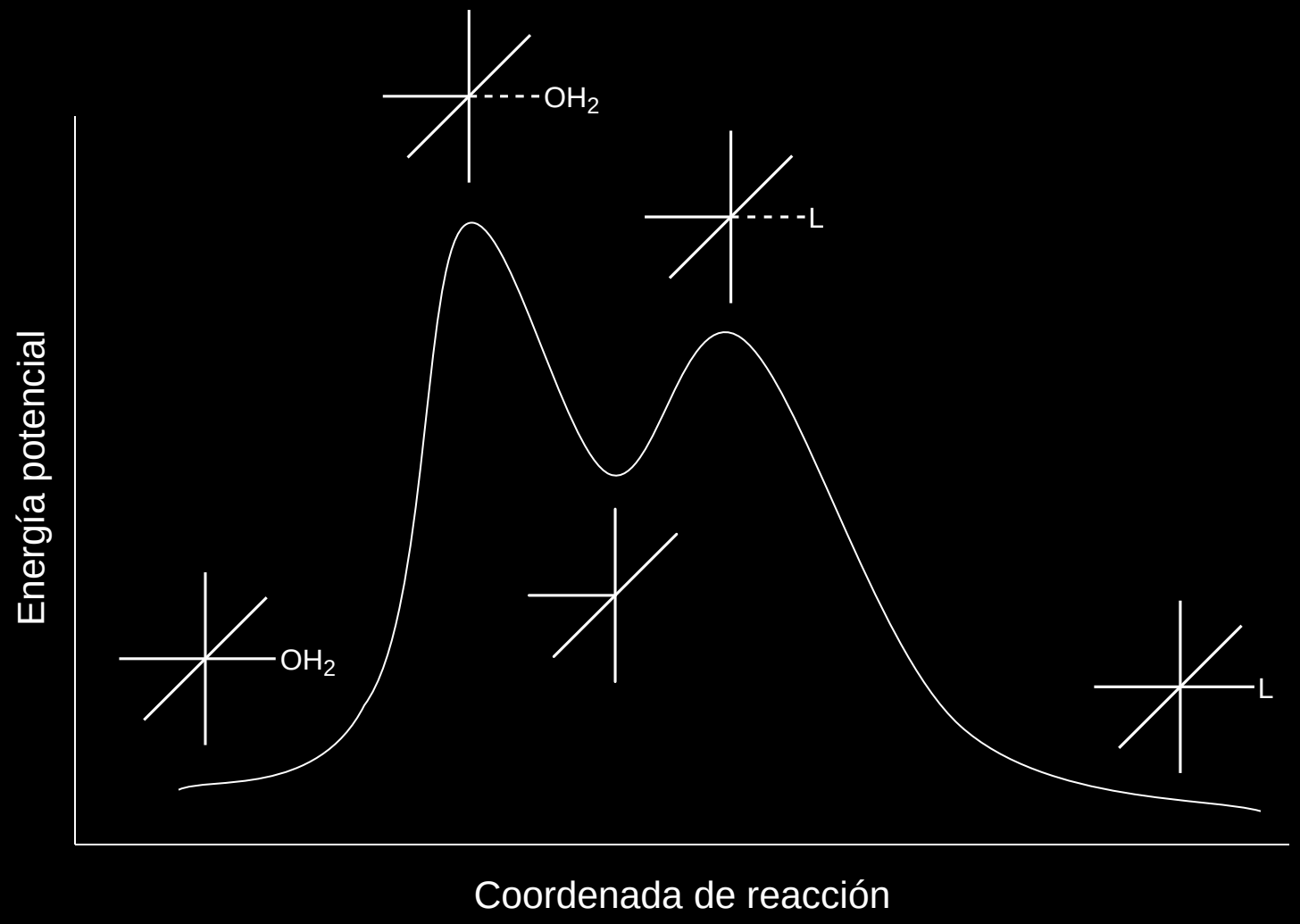
Cinética de Intercambio de Ligandos

Mecanismo Asociativo



Cinética de Intercambio de Ligandos

Mecanismo Disociativo



Intercambio de Agua

$$M(H_2O)_n^{z+} + H_2O^* \xrightarrow{k_{ex}} M(H_2O)_{(n-1)}(H_2O^*)^{z+} + H_2O$$

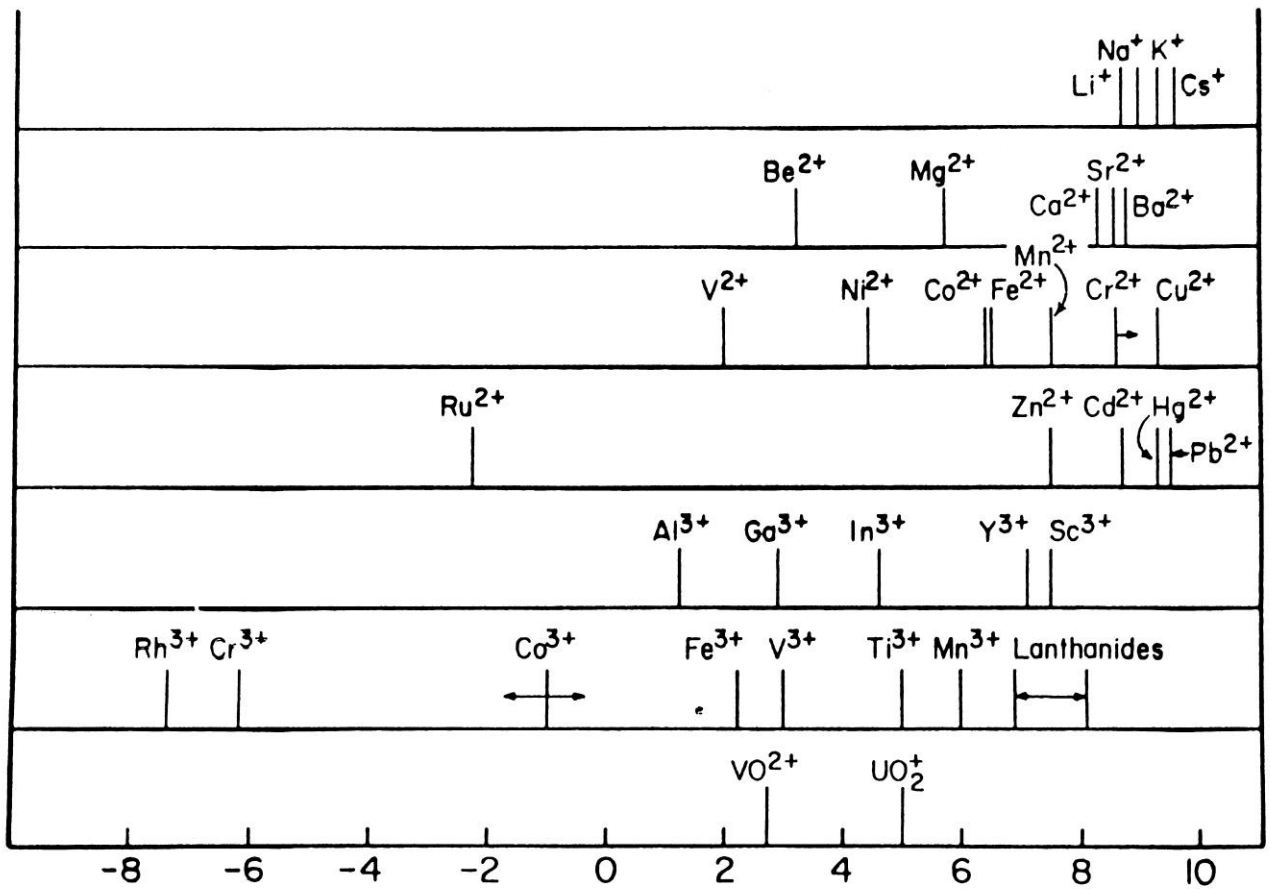
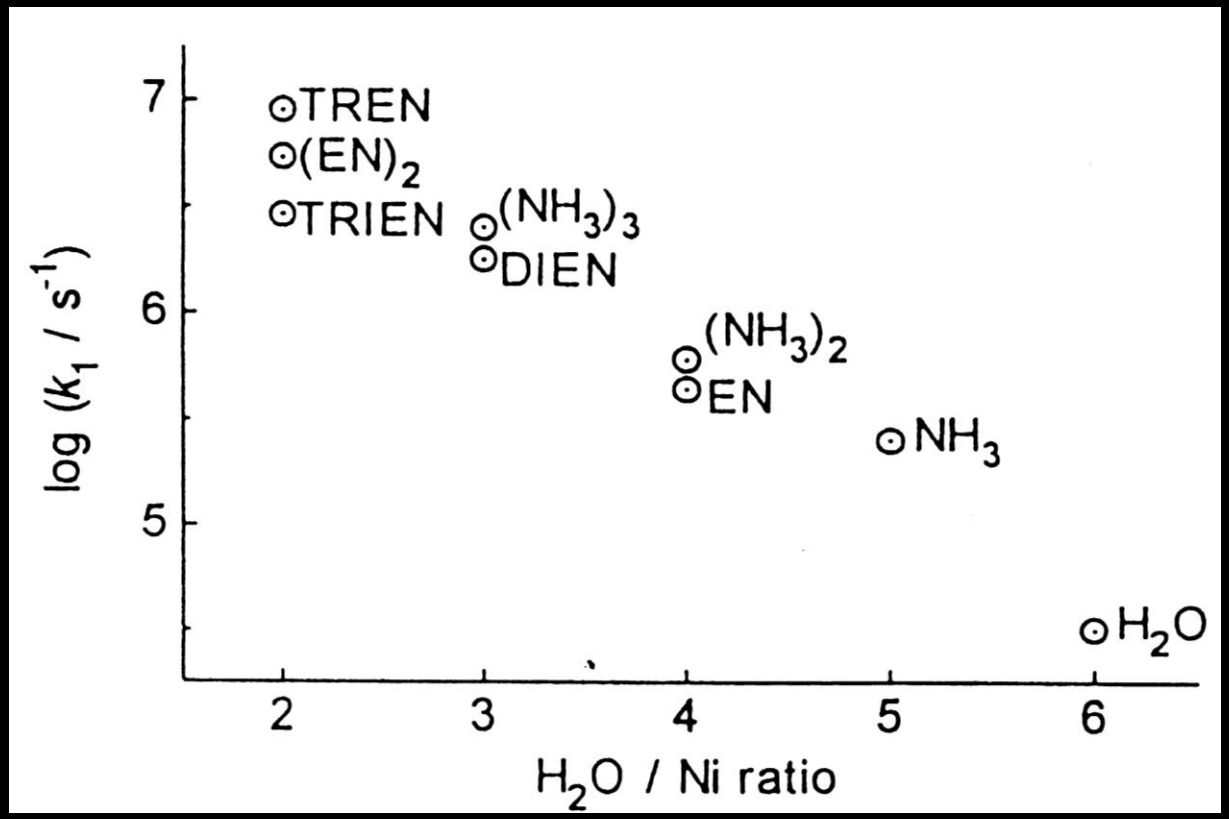
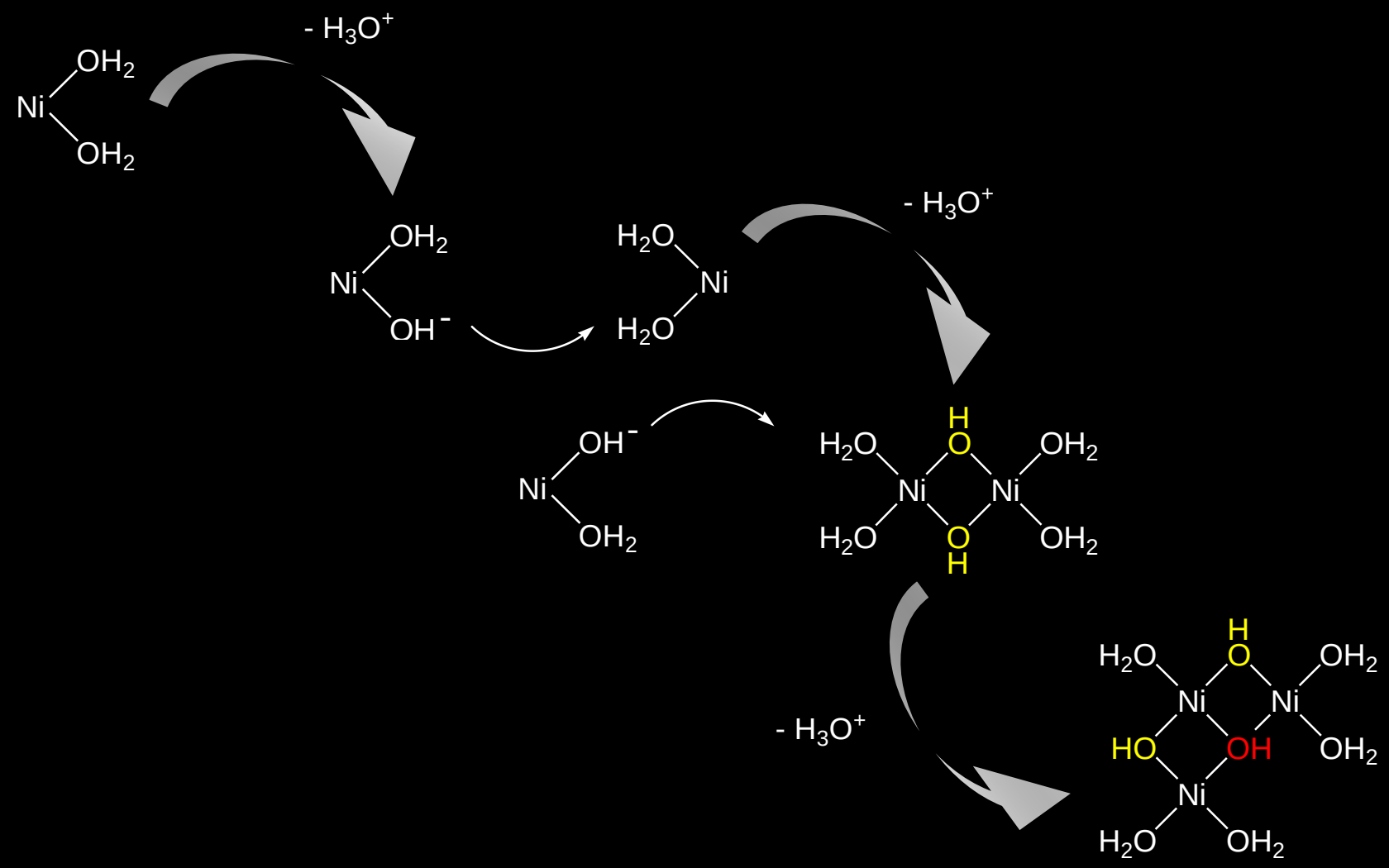


Figure 1-1 Characteristic water exchange rate constants (25°C, sec^{-1}) for aquo metal ions

Efecto de los Ligandos sobre k_{ex}



Nucleación *(Cascada de hidrólisis)*



Mecanismo de Precipitación

Iones metálicos

↓

Embriones

Hidrólisis:

olación
oxolación

↓

Núcleos

↓

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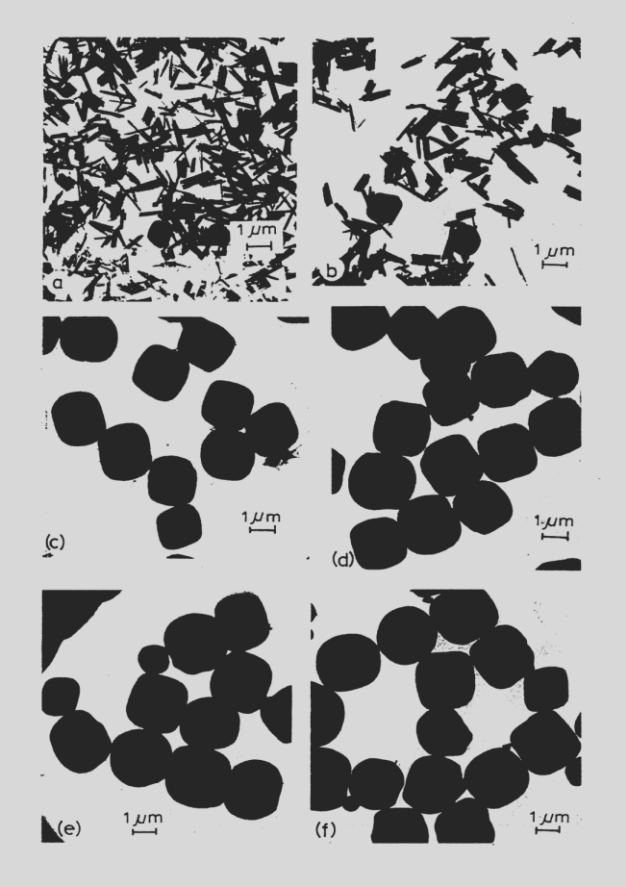
Agregación

Crecimiento cristalino

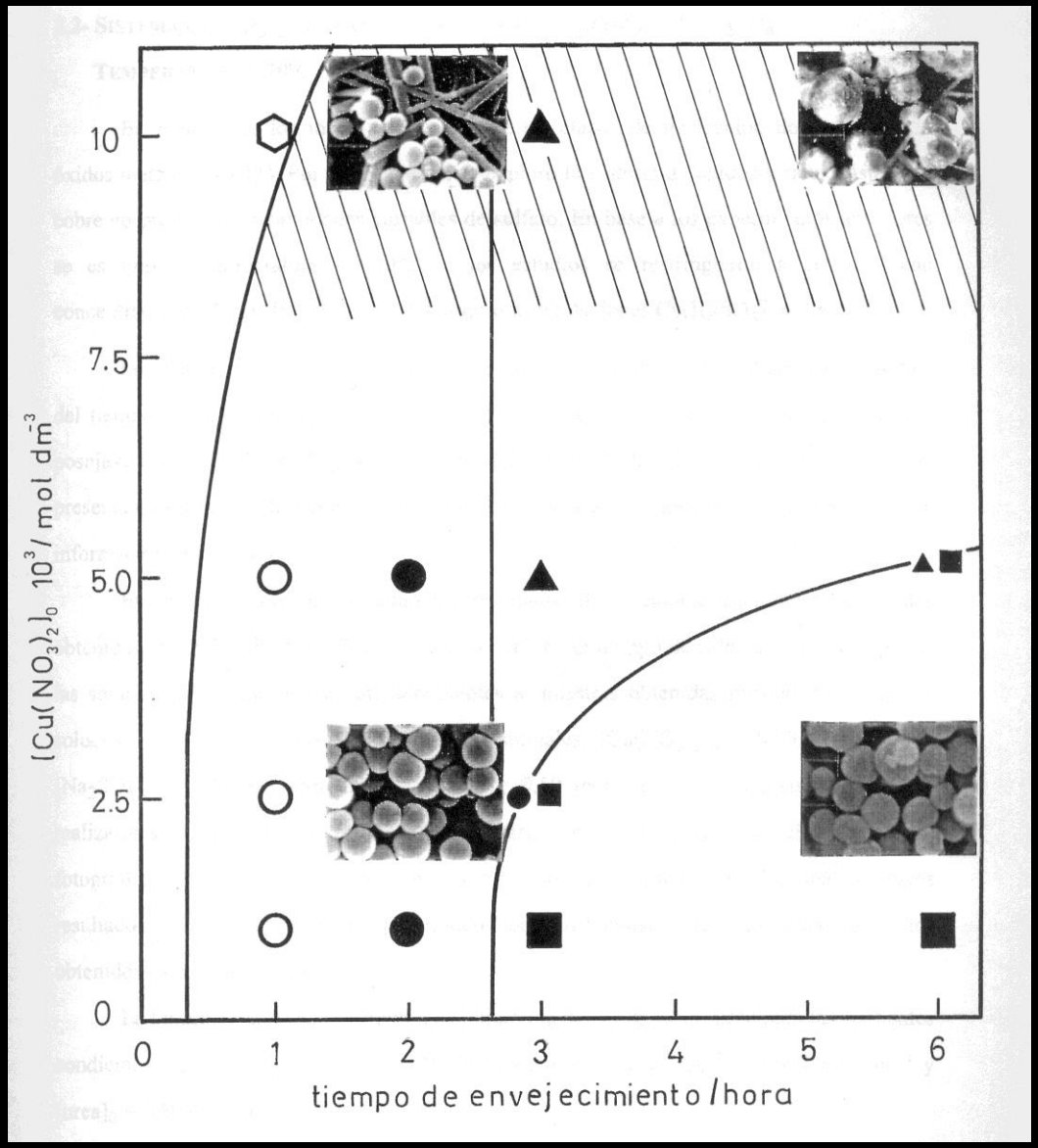
Partículas

Partículas cristalinas

Disolución-reprecipitación



Transformación β-FeOOH → α-Fe_2O_3 durante el envejecimiento de una solución 0.01 M de $FeCl_3$ a 98 ^oC


10
7.5
5.0
2.5
0
$[Cu(NO_3)_2]_0$ 10^3 / mol dm^{-3}
0
1
2
3
4
5
6
tiempo de envejecimiento / hora

La Regla de Ostwald *(el pez grande se come al pez chico)*

$$M(OH)_z(s) + zH^+ \rightleftharpoons M^{z+}(aq) + zH_2O;\ K_{S0}$$

$$-RT\ln K_{S0} = \Delta G^o$$

$$-RT\ln K_{S0}(r) = \Delta G^o - 4\pi r^2 N\gamma$$

$$N = \frac{3\overline{V}}{4\pi r^3}$$

$$\ln K_{S0}(r) = \ln K_{S0} + \frac{3\overline{V}\gamma}{RT}\frac{1}{r}$$

El sólido que precipita primero es el sólido más soluble

Síntesis de partículas uniformes *(tamaño y forma)*

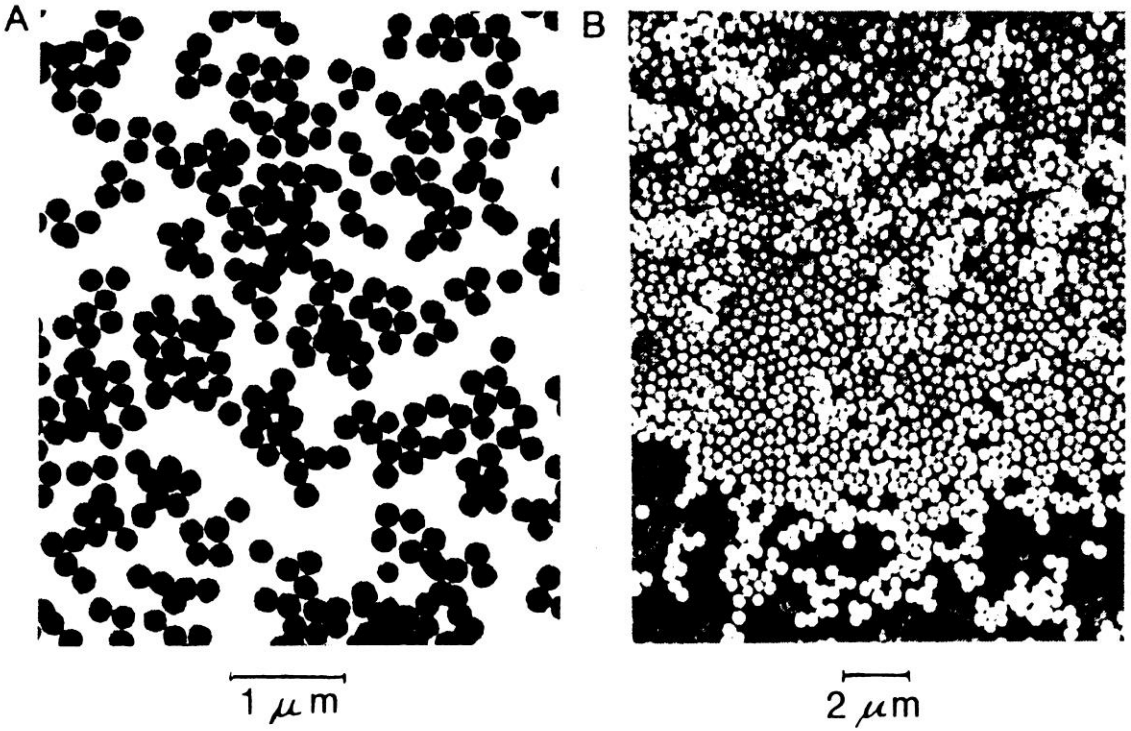
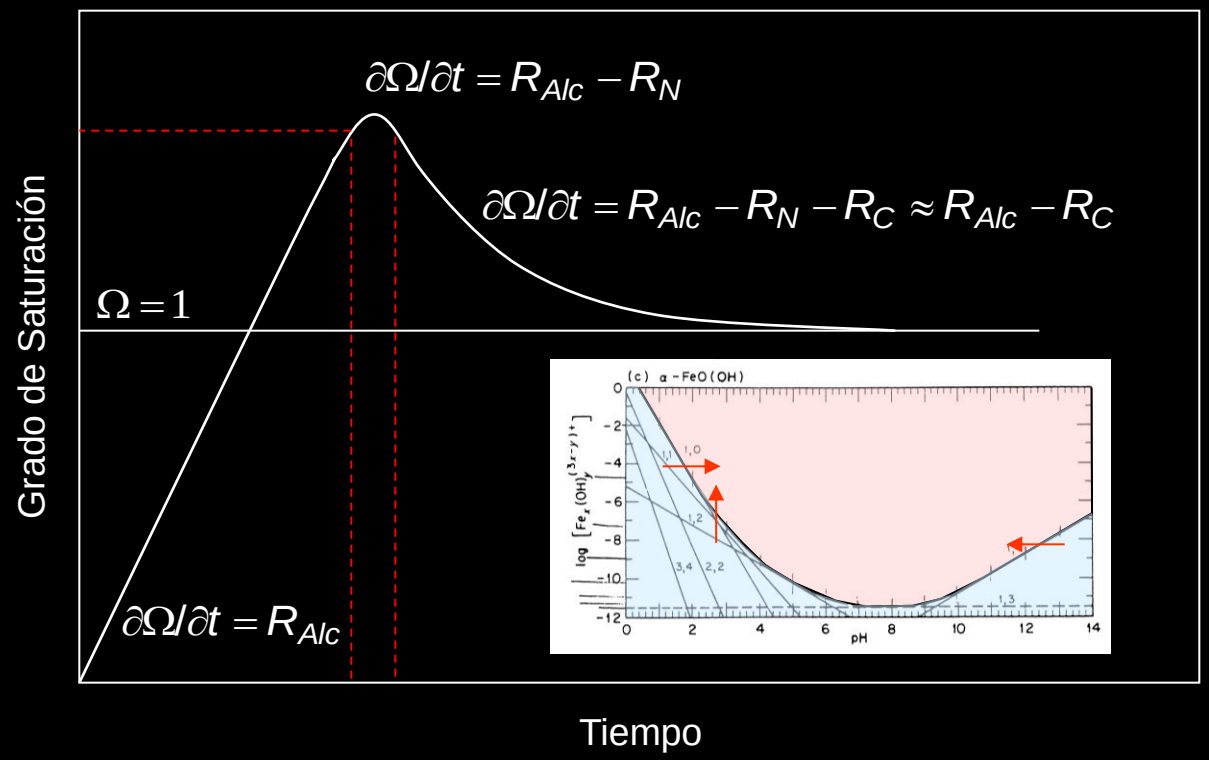


Figure 2. Transmission (TEM) (A) and scanning (SEM) (B) electron micrograph of CeO_2 particles obtained by aging at 90 °C for 48 h a solution 1.2×10^{-3} mol dm^{-3} in $Ce(SO_4)_2$ and 8.0×10^{-2} mol dm^{-3} in H_2SO_4.

Síntesis de partículas uniformes *(tamaño y forma)*

Requisito: Separar las etapas de nucleación y crecimiento



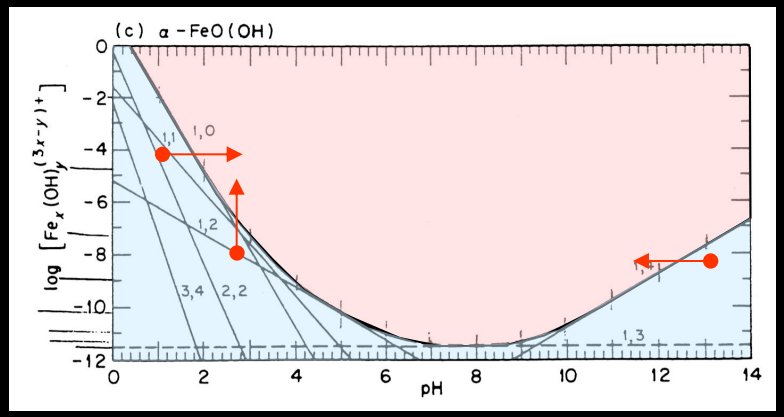
Se logra mediante un adecuado control de la velocidad con que aumenta el grado de sobresaturación

Síntesis de partículas uniformes *(tamaño y forma)*

Requisito: Separar las etapas de nucleación y crecimiento

Adecuado control del grado de la sobresaturación

Cómo sintetizarlas?



Procedimientos

- Hidrólisis forzada
- Descomposición de iones complejos
- Oxidación-Reducción
- Alcalinización homogénea

Síntesis de partículas uniformes *(tamaño y forma)*

Requisito: Separar las etapas de nucleación y crecimiento

Adecuado control del grado de la sobresaturación

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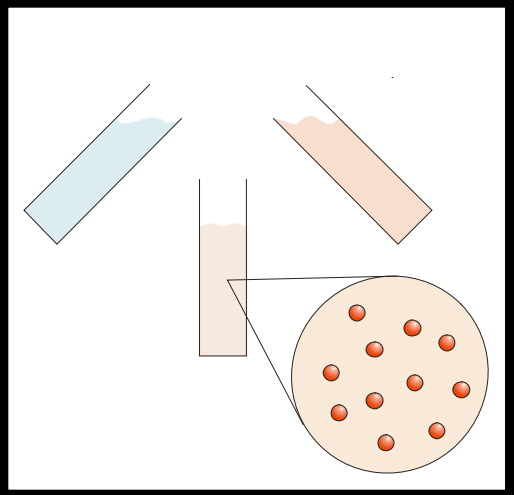

Procedimientos

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- Descomposición de iones complejos
- Oxidación-Reducción
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Hidrólisis Forzada

Principio:

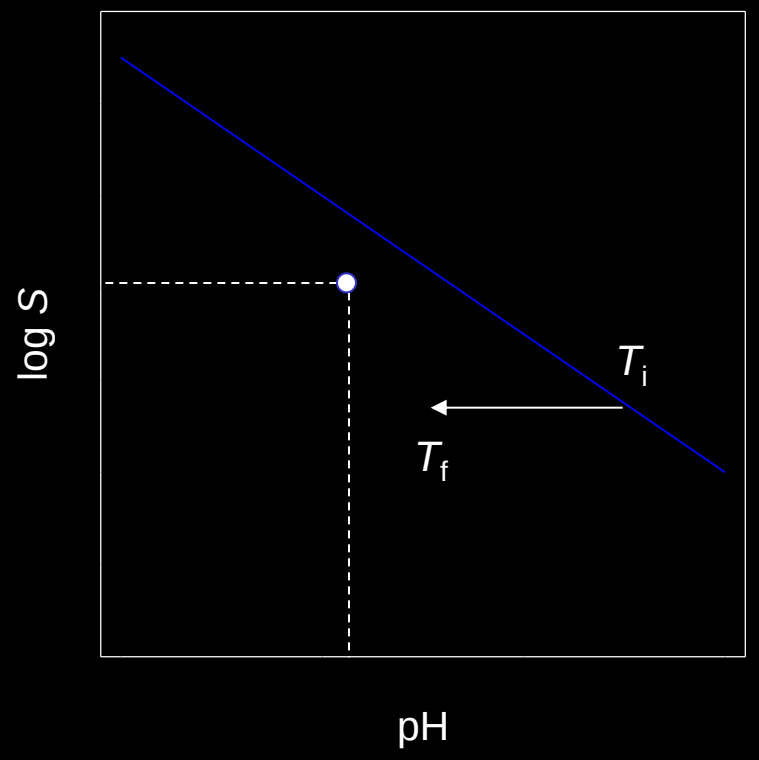
$$\frac{\partial \log K_{S0}}{\partial T} = \frac{\Delta H^{o}_{S0}}{2.3RT^{2}}$$

$$\log K_{S0} \approx a - b \times \log K_{a1}$$

Vale si la solubilidad está definida por la concentración del ión M^{z+}

$$\frac{\partial \log K_{S0}}{\partial T} \approx -\frac{b\Delta H^{o}_{a1}}{2.3RT^{2}}$$

$$\Delta H^{o}_{a1} > 0$$



Definir:

- Composición inicial del sistema
- Temperatura final
- Gradiente de temperatura
- Tiempo de reacción

Hidrólisis Forzada

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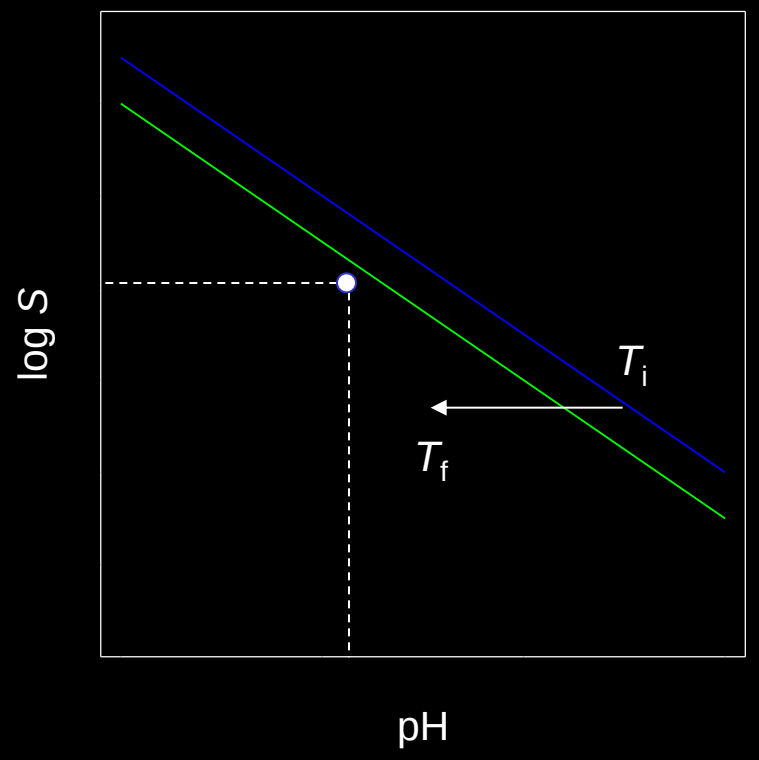

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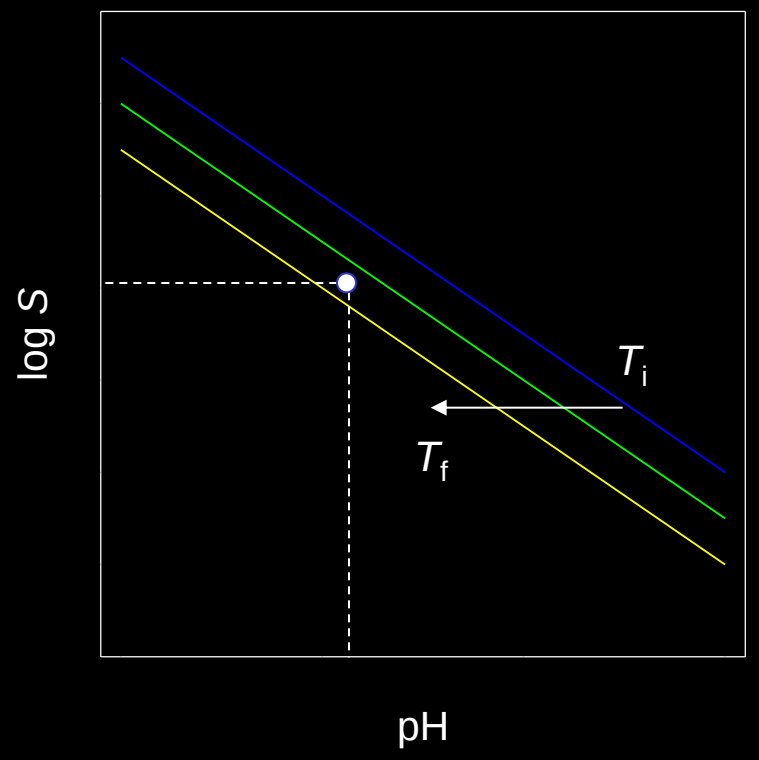

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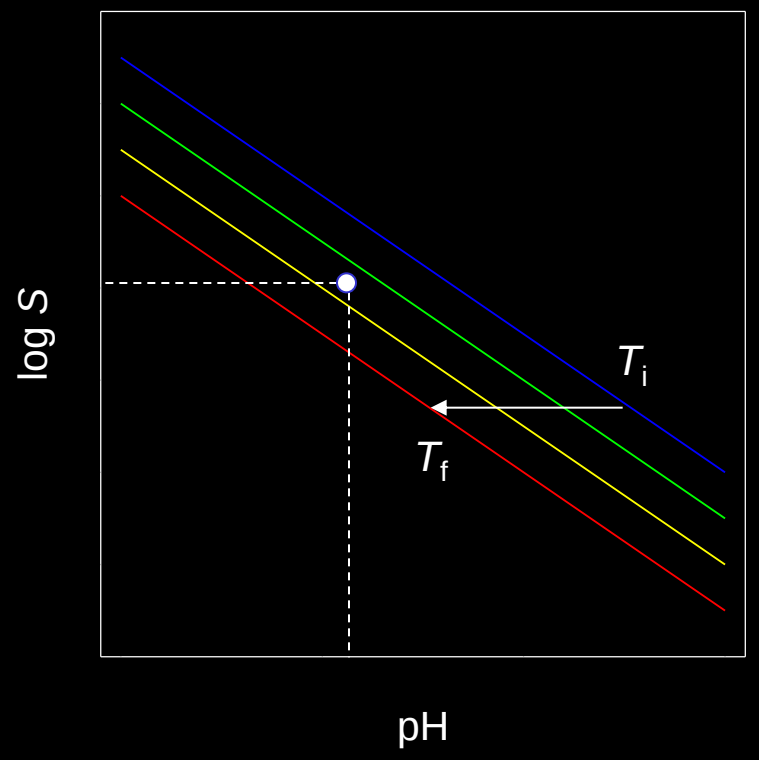


Descomposición de Iones Complejos

Principio:

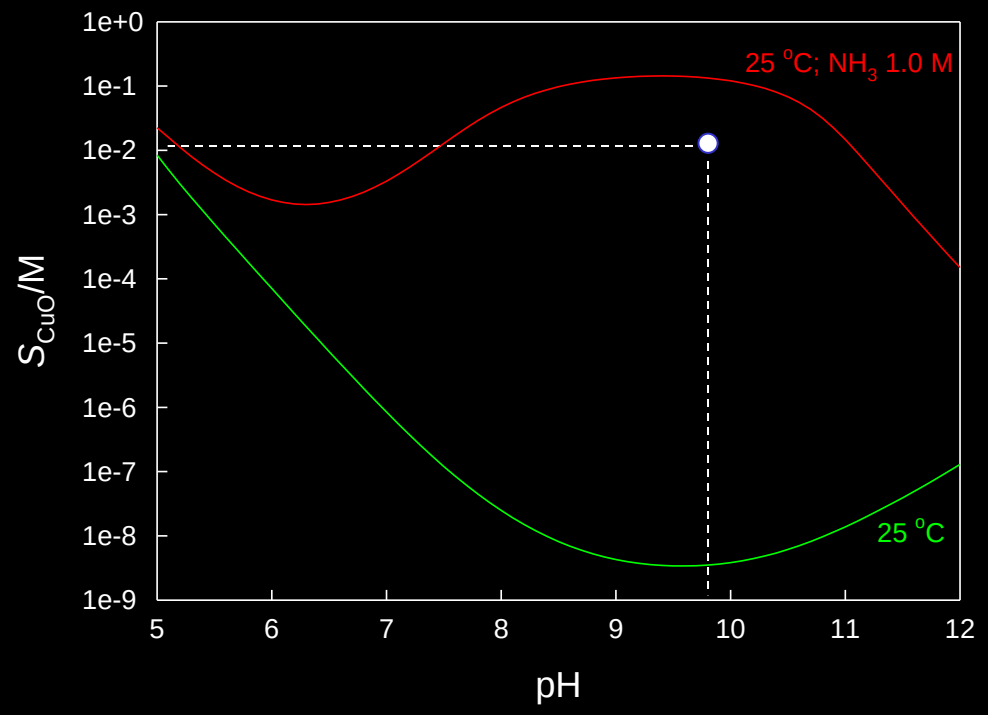
$$M(OH)_z(s) + zH^+ + nL^{m-} = ML_n^{(z-nm)+} + zH_2O$$

$$M^{z+} + nL^{m-} = ML_n^{(z-nm)+}; \quad K_{ML_n} \qquad \Delta H^o_{ML_n} < 0$$

Definir:

- Naturaleza del ligando
- Composición inicial del sistema
- Temperatura final
- Gradiente de temperatura

- Tiempo de reacción



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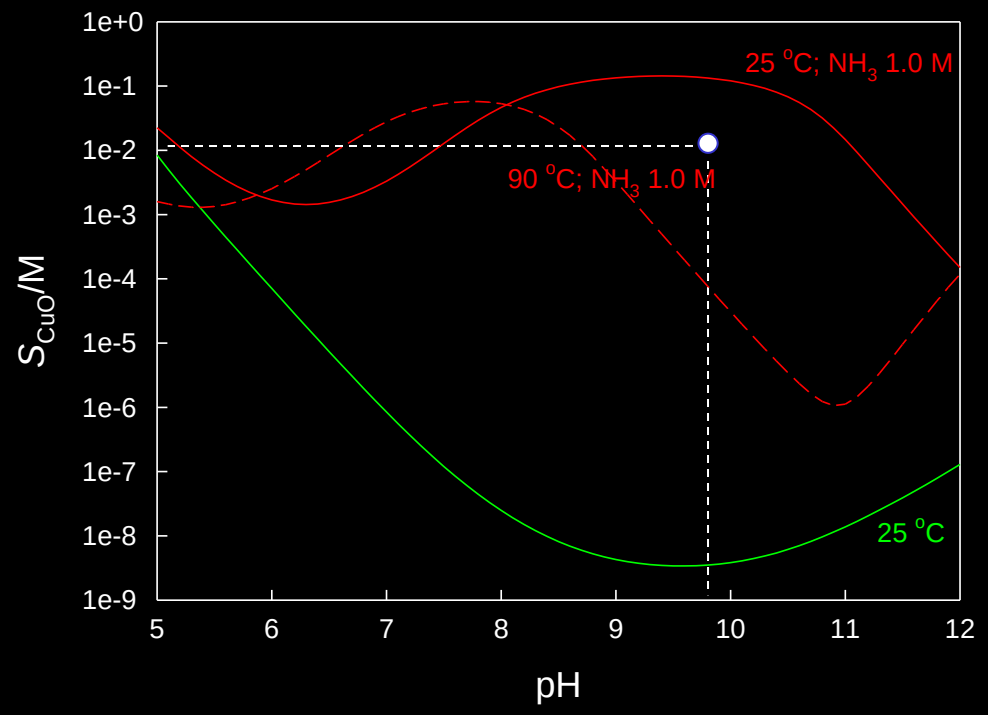
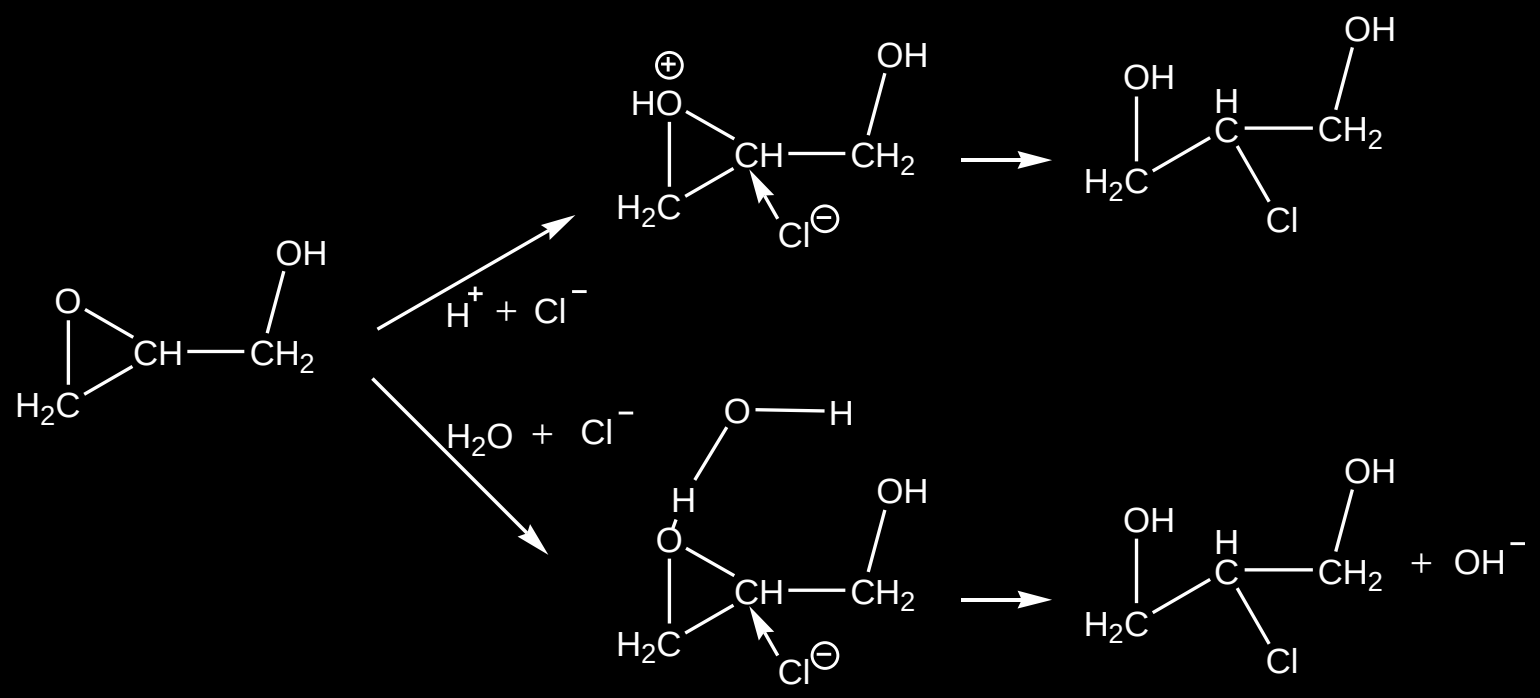


Alcalinización Homogénea

Hidrólisis de glicidol



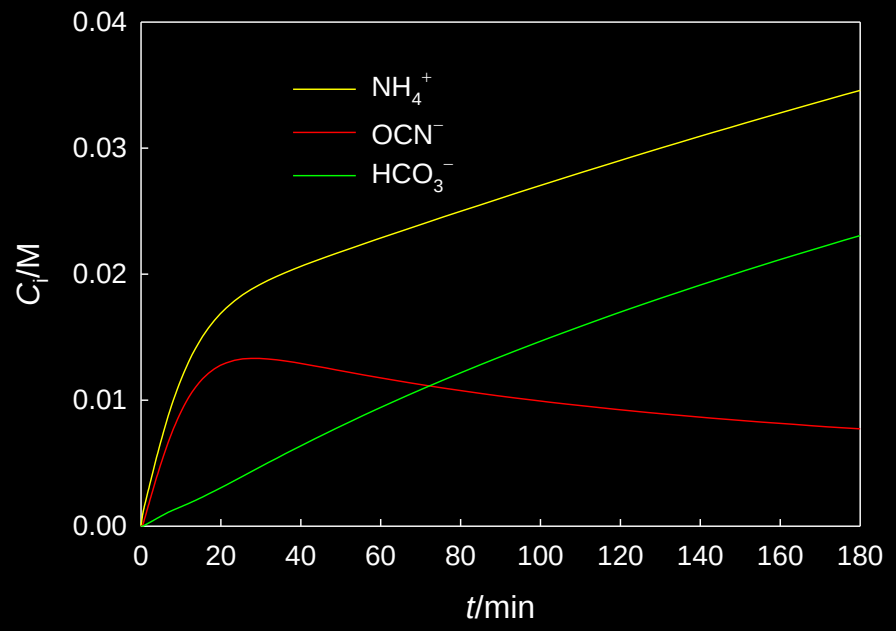
Alcalinización Homogénea

Hidrólisis de urea

$$(NH_2)_2CO + H^+ + 2H_2O \longrightarrow HCO_3^- + 2NH_4^+$$

$$(NH_2)_2CO \underset{k_{-1}}{\overset{k_1}{\rightleftharpoons}} OCN^- + NH_4^+$$

$$OCN^- + H^+ + 2H_2O \xrightarrow{k_2} HCO_3^- + NH_4^+$$



Alcalinización Homogénea

Hidrólisis de urea

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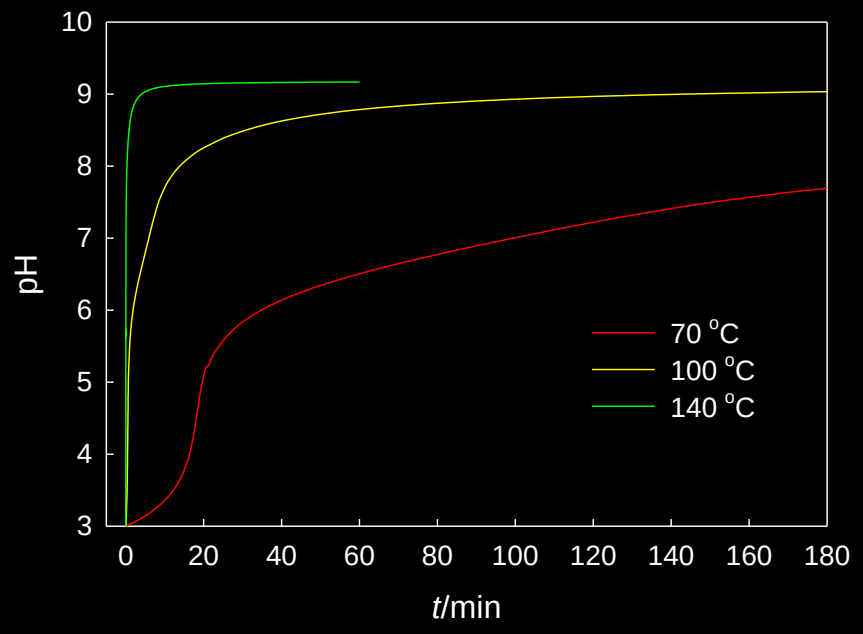
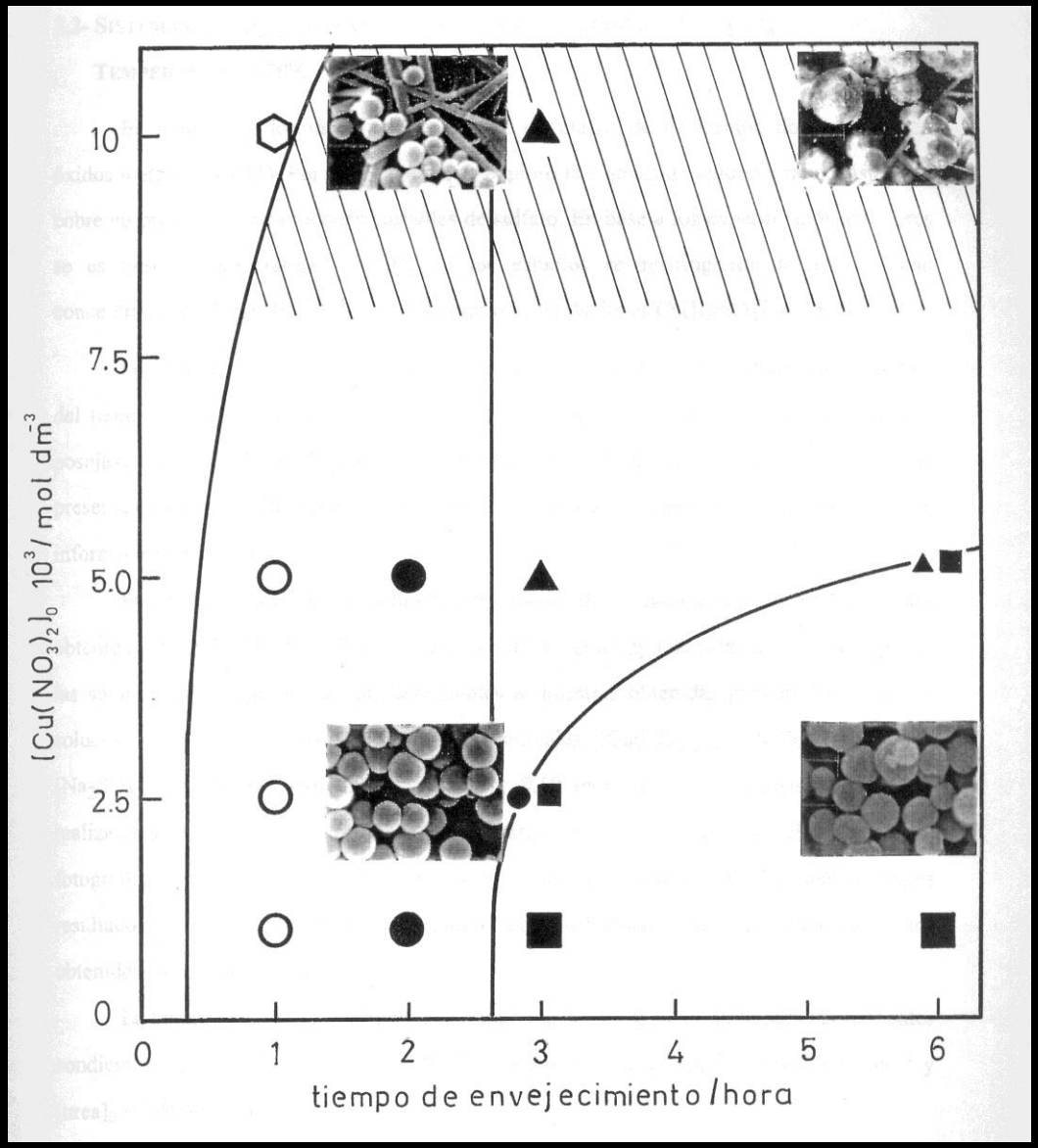
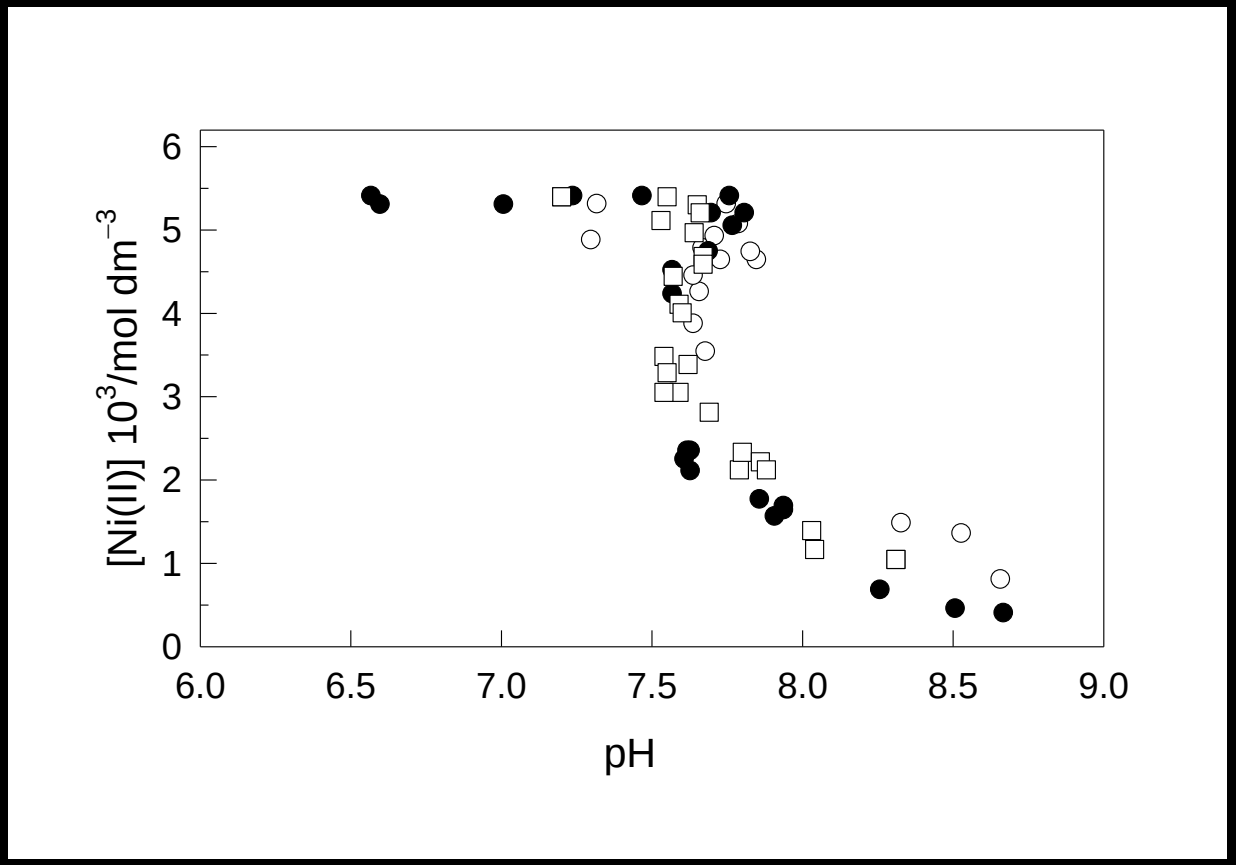
$$OCN^- + H^+ + 2H_2O \xrightarrow{k_2} HCO_3^- + NH_4^+$$

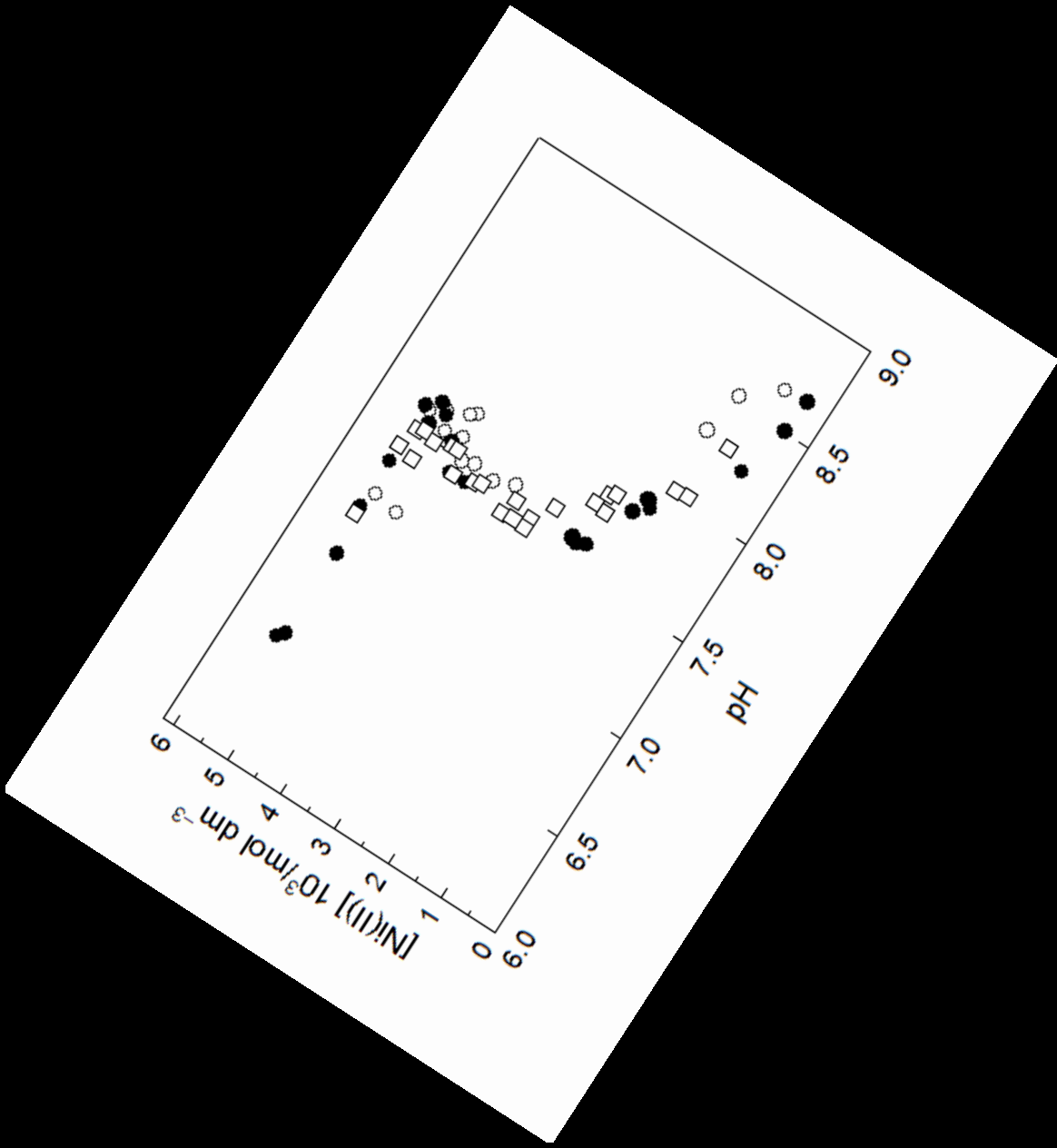
$$HCO_3^- \rightleftharpoons CO_3^{2-} + H^+$$




10
7.5
5.0
2.5
0
$[Cu(NO_3)_2]_0$ 10^3 / mol dm^{-3}
0
1
2
3
4
5
6
tiempo de envejecimiento / hora

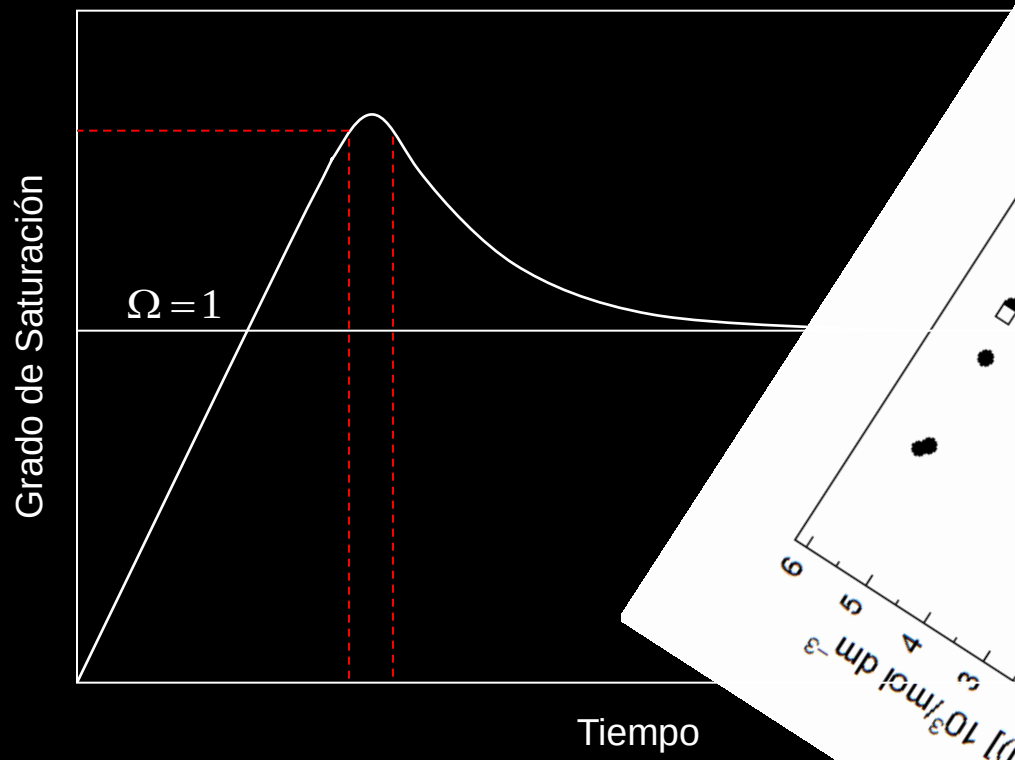


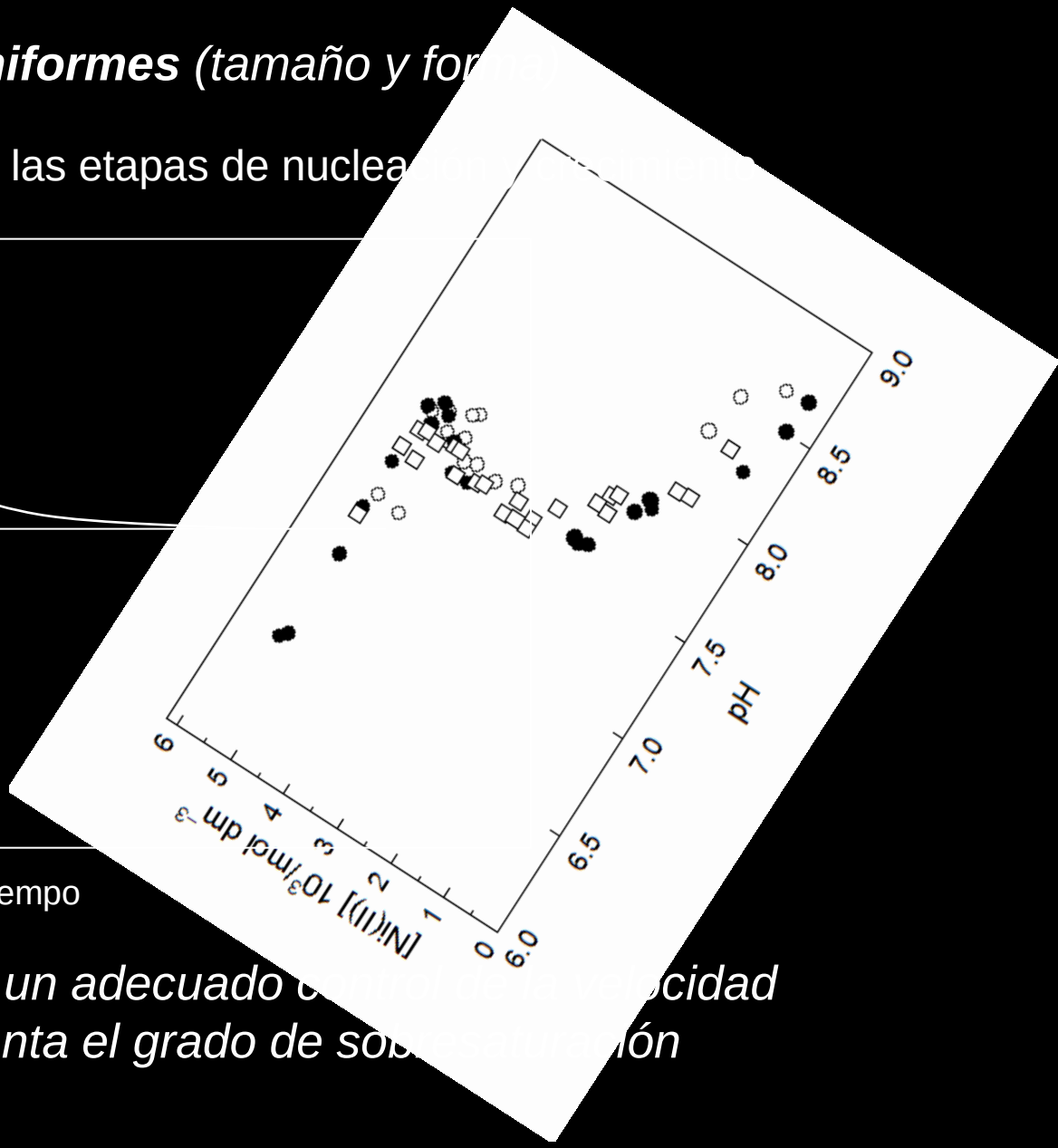
Evolución de la composición de la solución durante la precipitación de α-$Ni(OH)_2$


[Ni(II)] 10³/mol dm⁻³
0
1
2
3
4
5
6
6.0
6.5
7.0
7.5
8.0
8.5
9.0
pH

Síntesis de partículas uniformes *(tamaño y for*

Requisito: Separar las etapas de nuclea

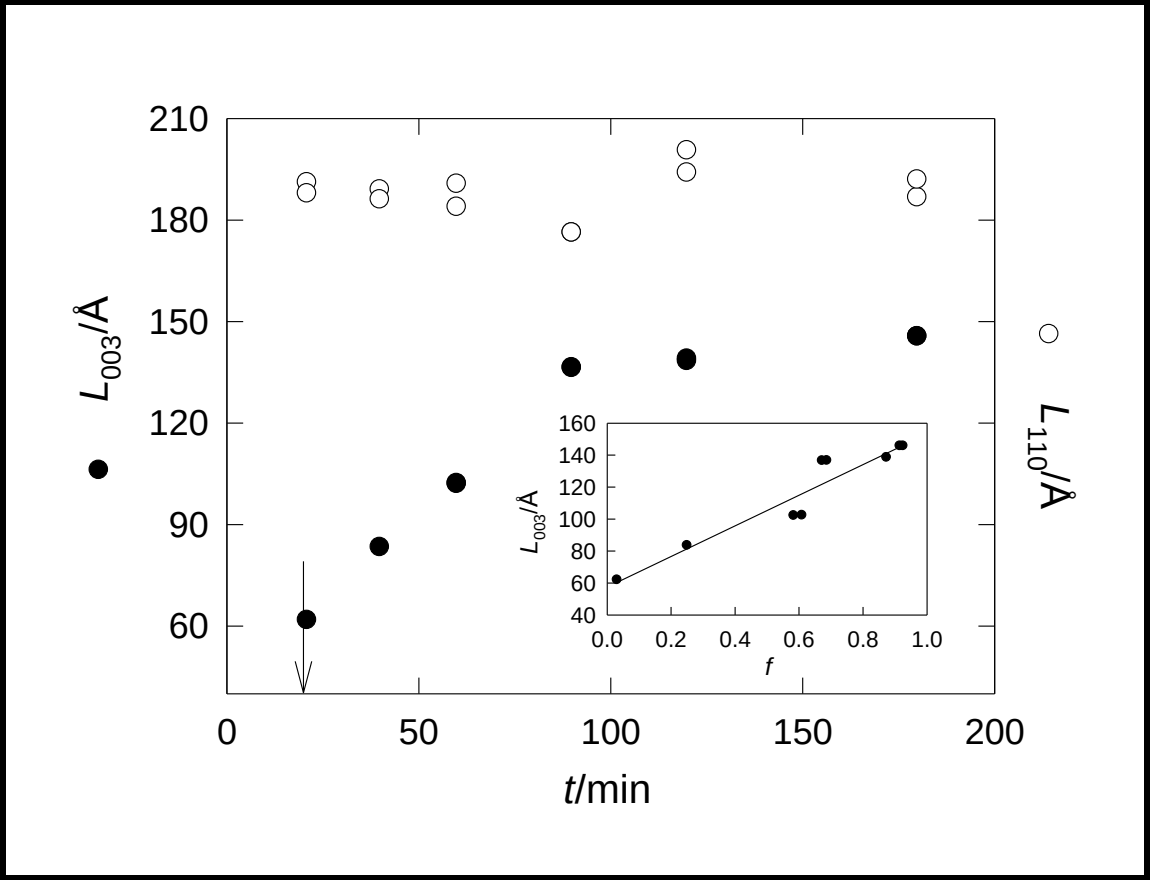


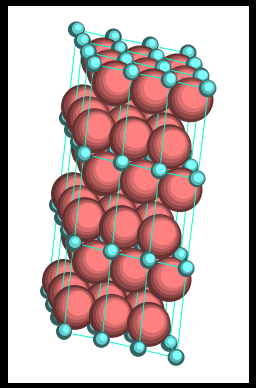


Se logra mediante un adecuado c

con que aumenta el grado de sob

Evolución del tamaño de cristalito durante la precipitación de α-$Ni(OH)_2$





Síntesis de partículas uniformes *(tamaño y forma)*

Requisito: Separar las etapas de nucleación y crecimiento

Adecuado control del grado de la sobresaturación

Cómo sintetizarlas?

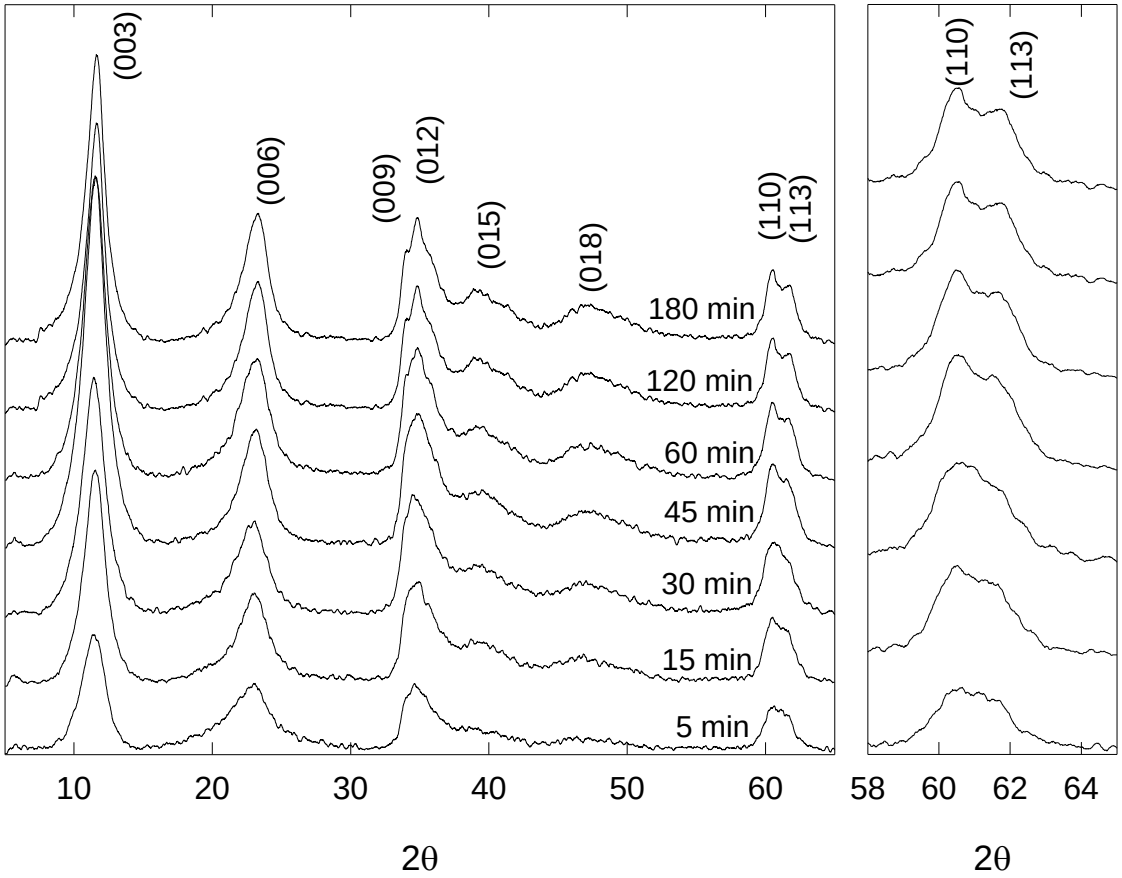
- Alcalinización homogénea

Procedimientos

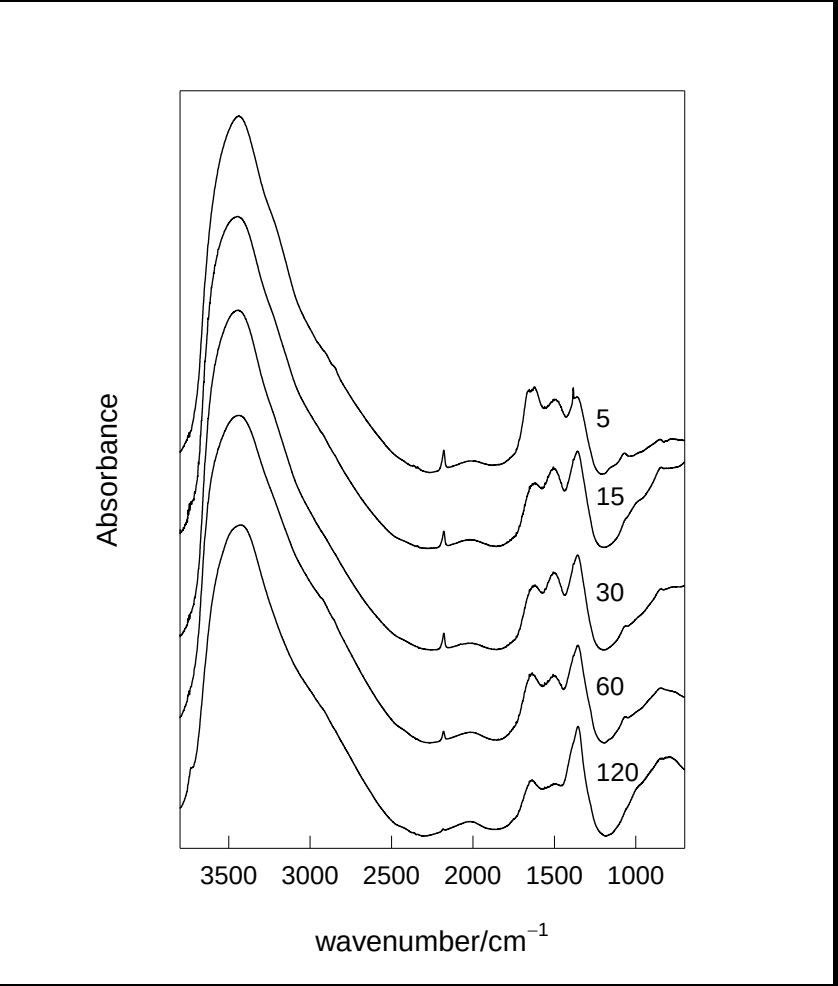
- Hidrólisis forzada
- Descomposición de iones complejos
- Oxidación-Reducción

Son adecuados para sintetizar óxidos mixtos??

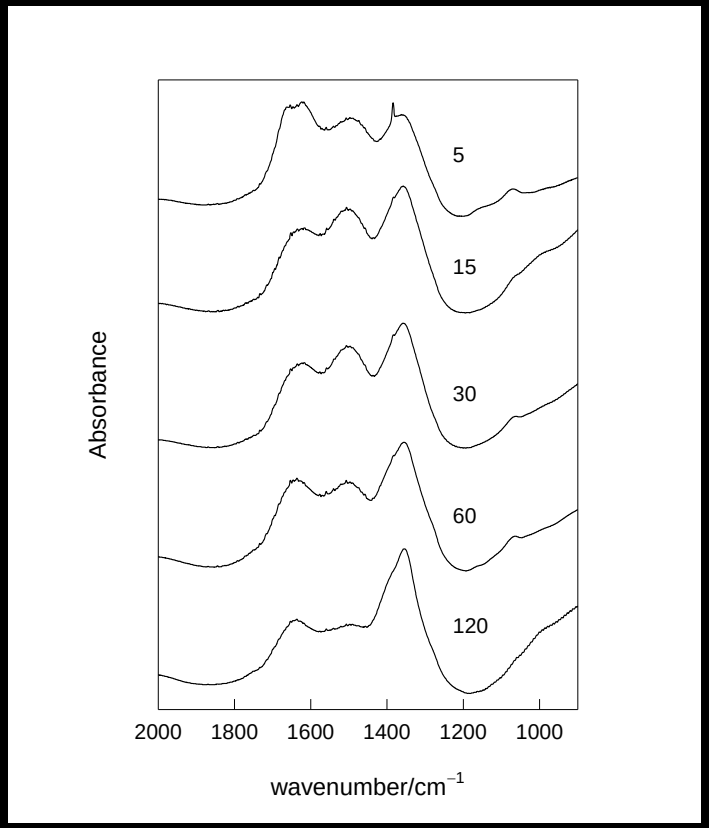
Diagramas de DRX de HDLs de Ni(II)-Cr(III)

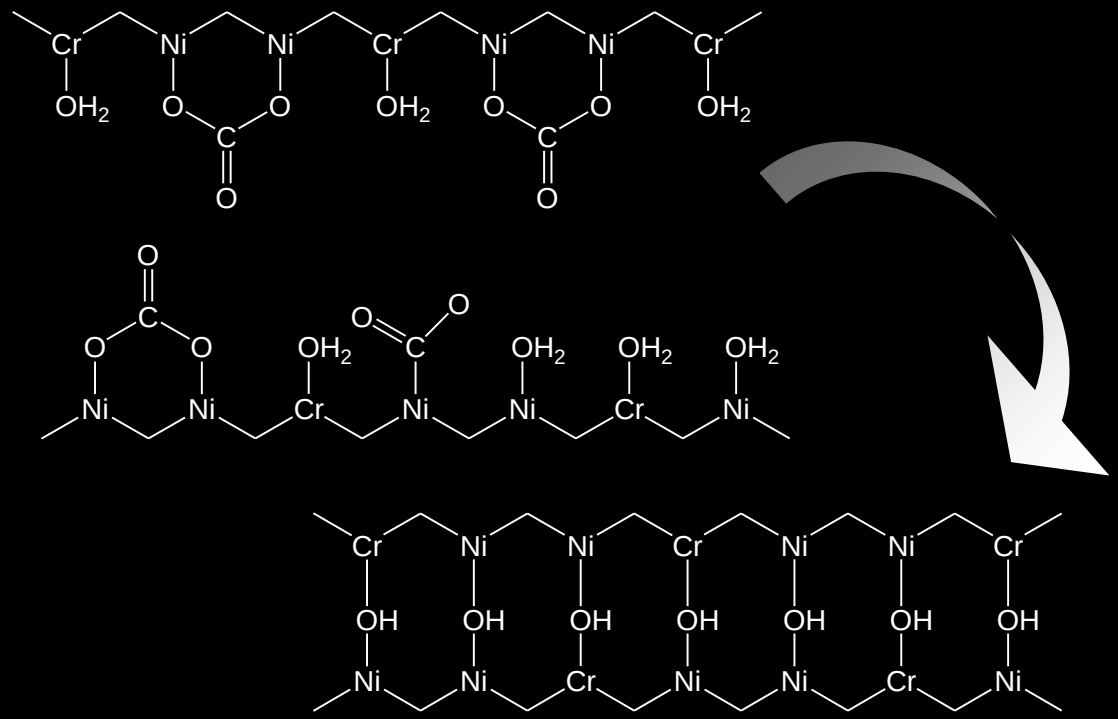


Espectros FTIR de HDLs de Ni(II)-Cr(III)



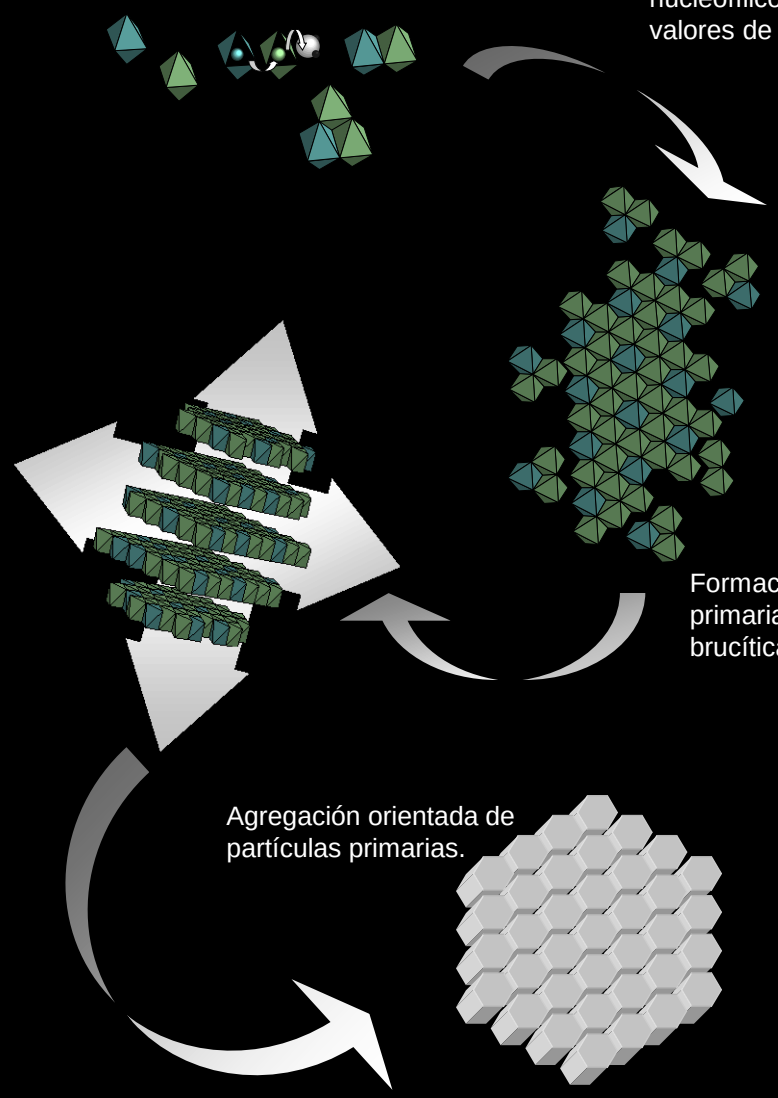
Espectros FTIR de HDLs de Ni(II)-Cr(III)





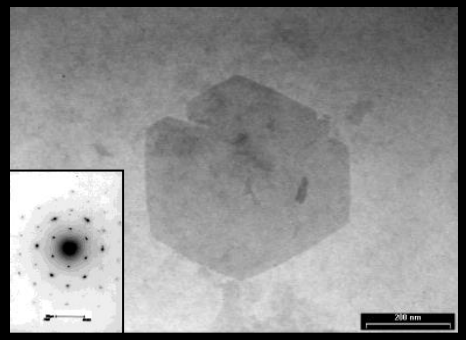
Mecanismo de Precipitación de HDLs de Ni(II)–Cr(III)

Hidrólisis. Formación de especies heteropolinucleares por ataque nucleofílico de $Cr(H_2O)_4(OH)_2^+$ sobre $Ni(H_2O)_6^{2+}$; notar los diferentes valores de kH_2O.



Formación y crecimiento de partículas primarias por condensación de capas brucíticas.

Agregación orientada de partículas primarias.



Síntesis de partículas uniformes *(tamaño y forma)*

Requisito: Separar las etapas de nucleación y crecimiento

Adecuado control del grado de la sobresaturación

Cómo sintetizarlas?

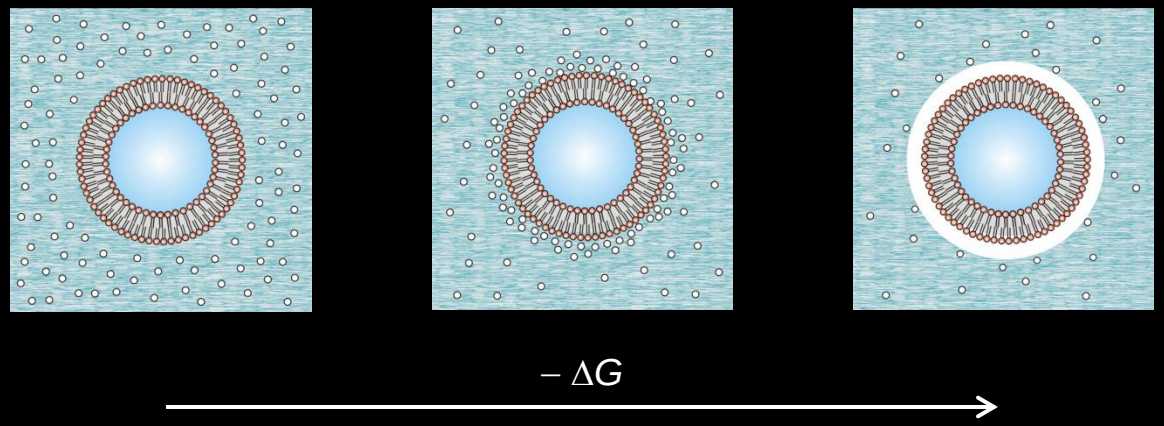
- Alcalinización homogénea

Procedimientos

- Hidrólisis forzada
- Descomposición de iones complejos
- Oxidación-Reducción

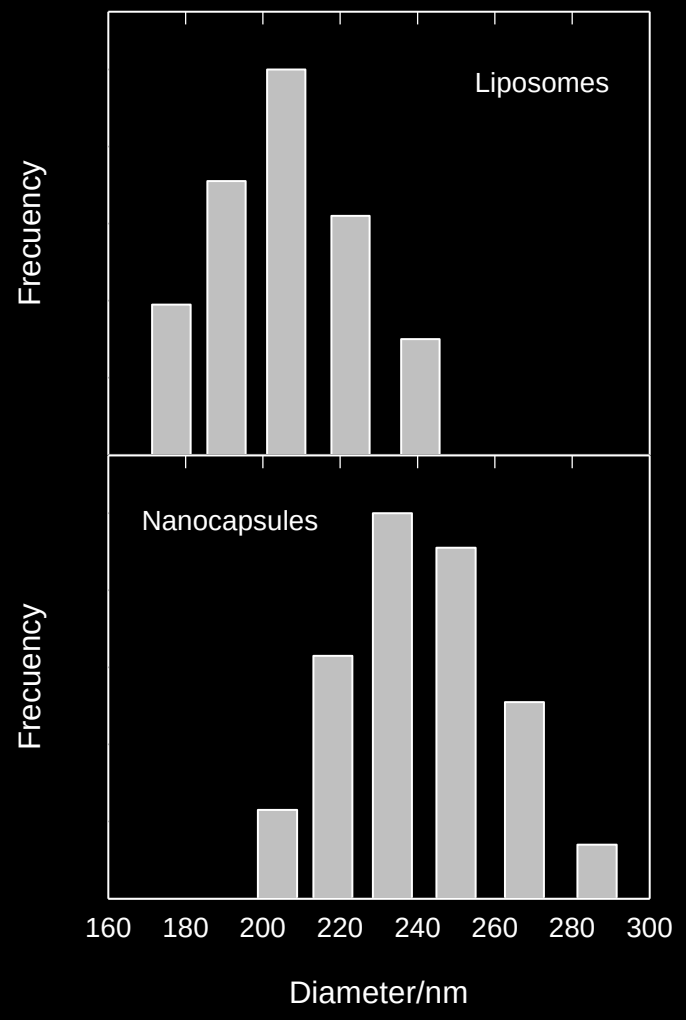
Son adecuados para sintetizar partículas Core@Shell??

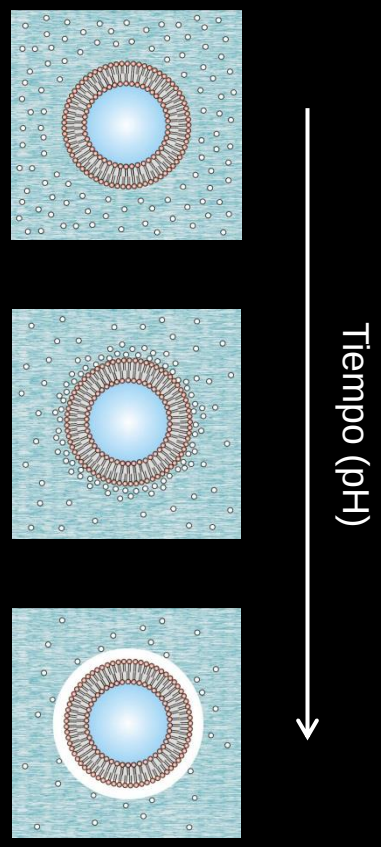
***Síntesis de partículas Core@Shell**: haciendo huevos…*



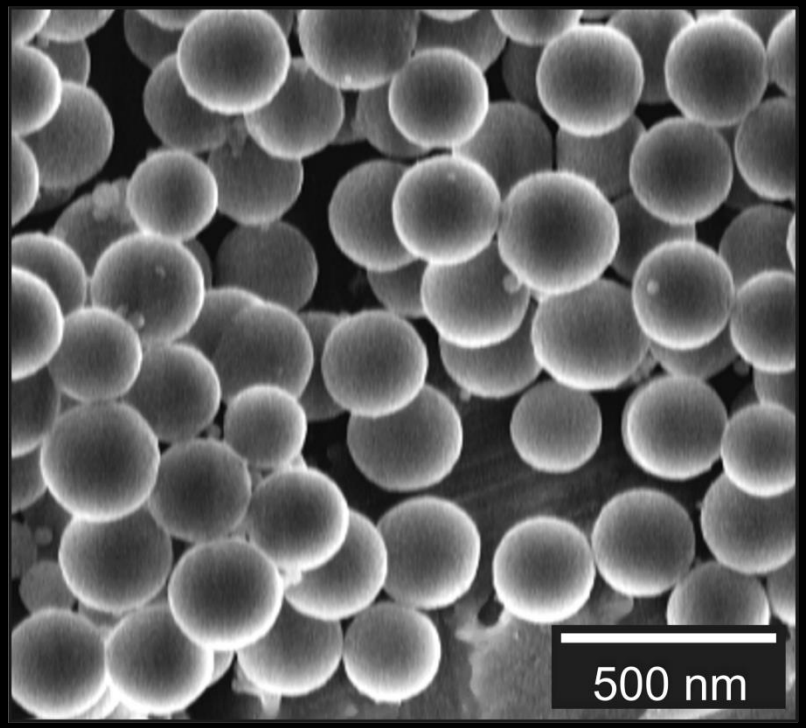
- Liposomas estables
  - Naturaleza de los fosfolípidos
  - Naturaleza de la cáscara
- Adecuada velocidad de alcalinización
  - Rápida (supervivencia de los liposomas)
  - Lenta (asegurar nucleación heterogénea)

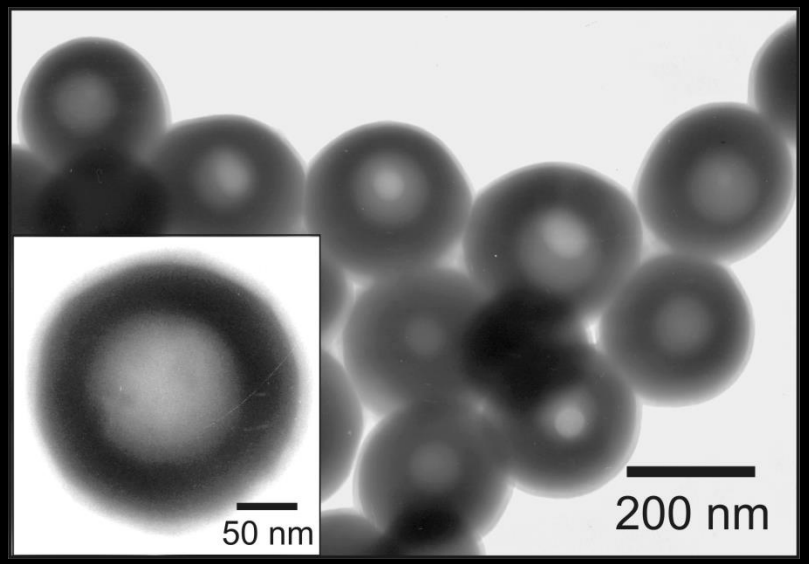
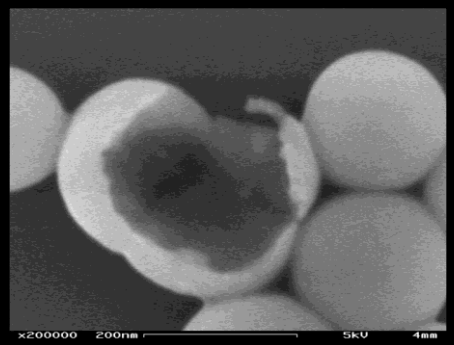
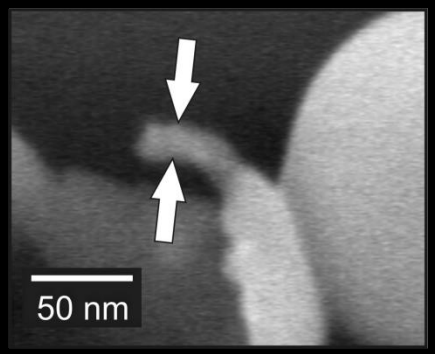
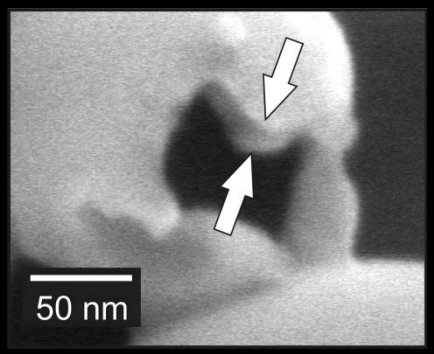
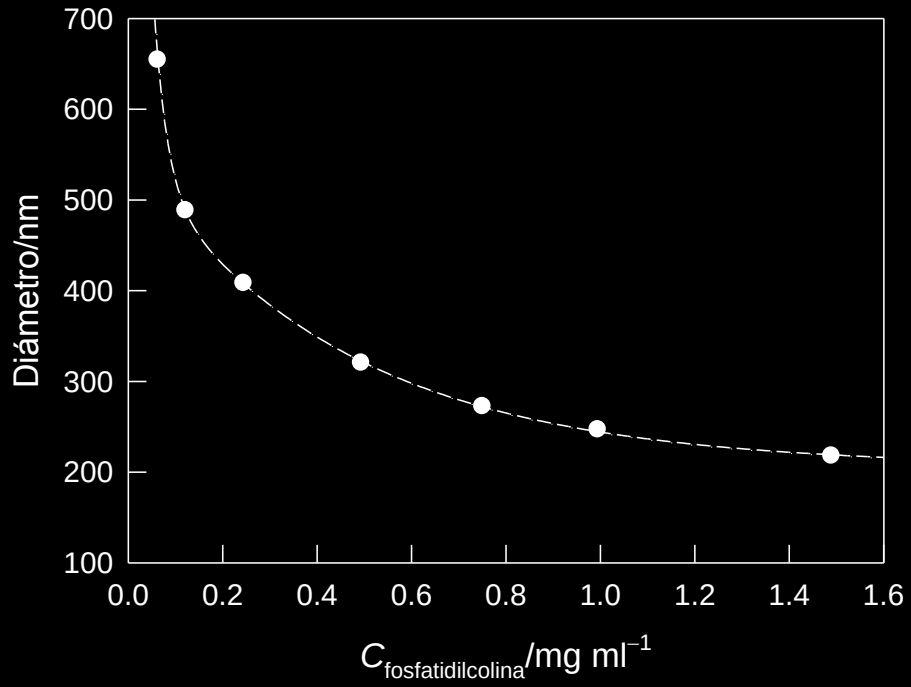
Nanocápsulas Liposoma@$Y(OH)CO_3xH_2O$





Nanocápsulas Liposoma@$Y(OH)CO_3xH_2O$





Nanocápsulas Liposoma@$Y(OH)CO_3xH_2O$