Synthesis of Silica Spheres and $CoFe_2O_4/SiO_2$ Spheres by Microemulsion Process

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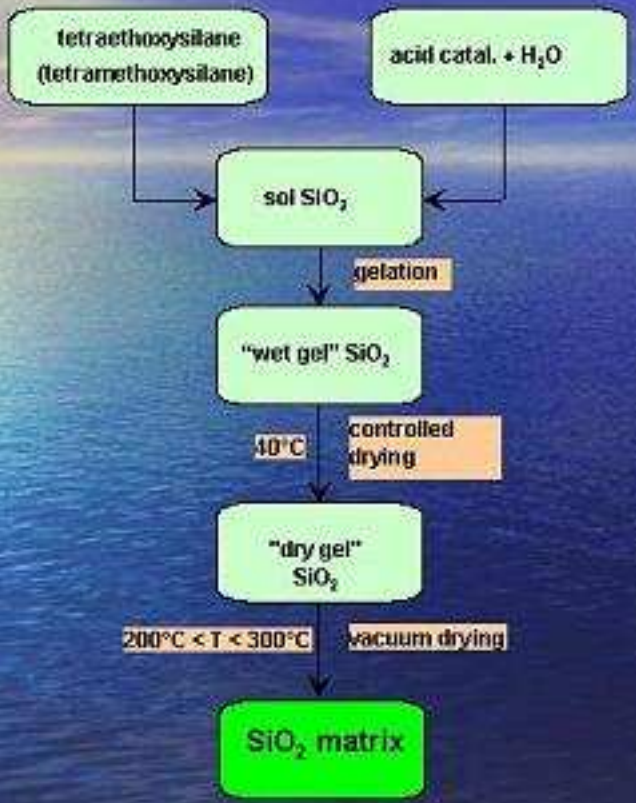
Outline:

- Sol-gel – matrix preparation
- Nanokompozite preparation
- The aim of this work
- SiO_2- 25μm spheres –preparation
- SiO_2 spheres – with size about 3μm – preparation
- $CoFe_2O_4/SiO_2$ spheres – preparation
- Conclusion

Sol-gel – matrix preparation

- Preparation of **precurzor** solutions (TEOS, TMOS, alcoxides)
- **Hydrolytic and condenzation** reactions, **sol** formation (controlling by: temperature, concentrations, pH, modificator)
- Interconnecting of coloidal sol particles – 3D network production –> **gel** (rapid viscozity increasing of the system)
- **Drying** – liquid removal from the gel porozity. Sample **schrinkage** -> strain rising -> cracking or pulverizing of the sample (problem especially in bulk materials).
- Heating to 400-1400°C -> **densification** creation of compact and hard materials. Some of them crystallize, or create glasses or ceramics

SiO_2 matrix preparation - scheme



Nanokompozite preparation

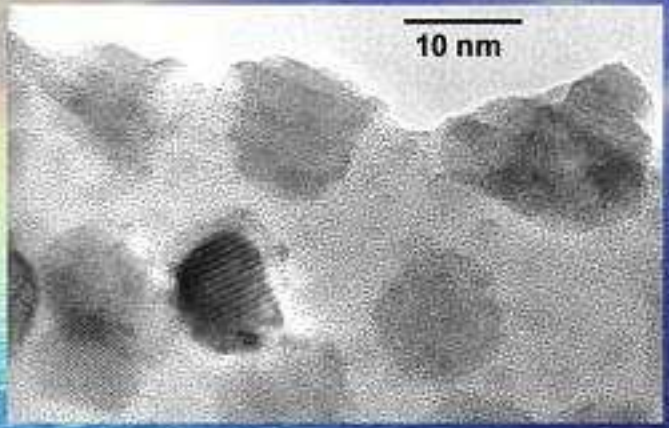
In addition to pure SiO_2:

- **Aditives** addition to starting solutions – active compounds **precurzors**
- **Modificator** addition – compoud affecting to resulting gel quality – most common modificator is formamide (*FA*, $HCONH_2$).
- TMOS, resp. TEOS hydrolysis is acid catalyzed – small amount of nitric acid addition
- Xerogel heating to temperatures at which active comopound precurzors crystallized to nanocrystals.

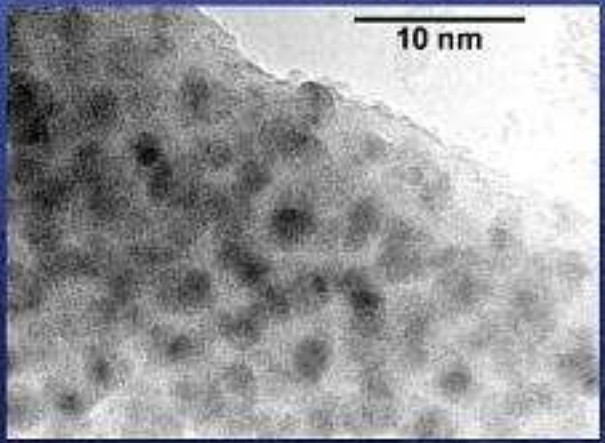
Up to now, they have been prepared in our laboratory next nanokompozites in SiO_2 matrix:

- $CuFe_2O_4$, $CdFe_2O_4$, $CoFe_2O_4$, $NiFe_2O_4$, $ZnFe_2O_4$
- $KHSO_4$, $KDSO_4$, $RbHSO_4$, $RbDSO_4$, $CsHSO_4$, $CsDSO_4$

HR-TEM $ZnFe_2O_4/SiO_2$
heated to 1100°C



HR-TEM $CdFe_2O_4/SiO_2$
heated to 900°C



Mean ferite nanoparticle size at samples heated to different temperatures

Particle size/ nm	800°C	900°C	1100°C
$ZnFe_2O_4/SiO_2$	4,2	5,6	14,9
$CdFe_2O_4/SiO_2$	3,4	6,0	--

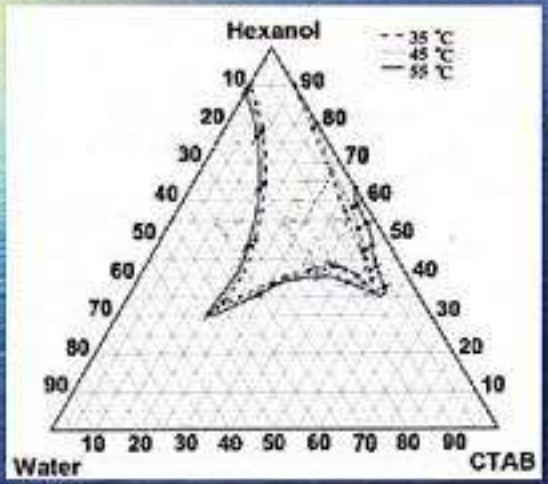
The aim of this work

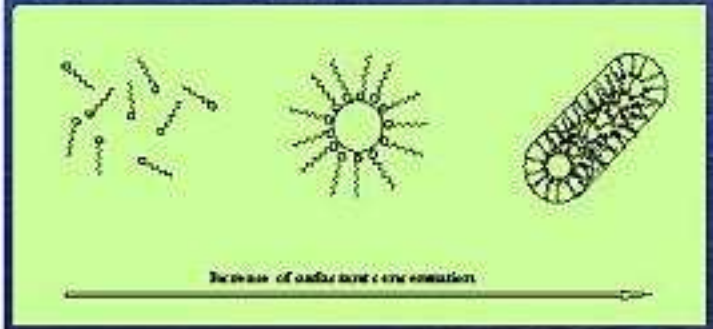
- Find out possibility of preparation of **sol-gel** silica spheres with size about 1 μm using **microemulsion** process
- Try to control the sphere size
- Try to control the size distribution
- Preparation of magnetic nanocomposites with spherical shape and size about 1 μm

Possible use of these materials

- Magnetic-located drugs carier
- Magnetic isolable sorbents for poluted soils

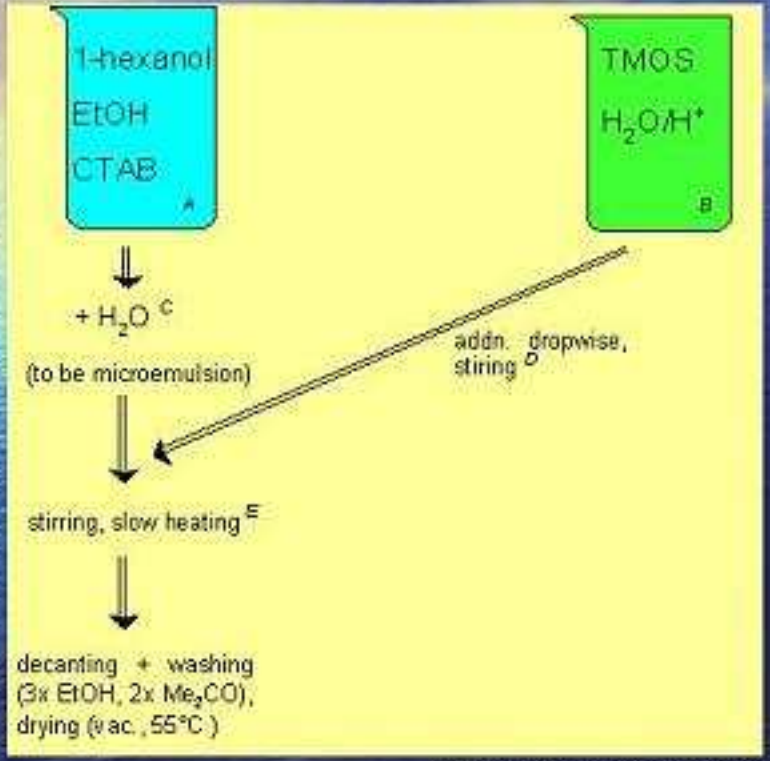
Relations in (reverse) micelar systems





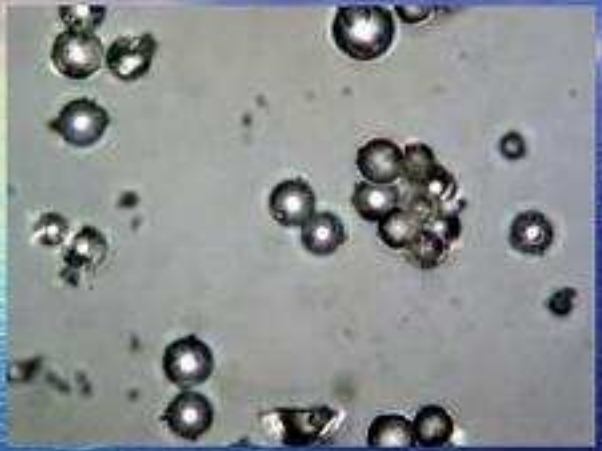
(CTAB = cetyltrimethylammonium bromide)

1. SiO_2- 25μm spheres -preparation



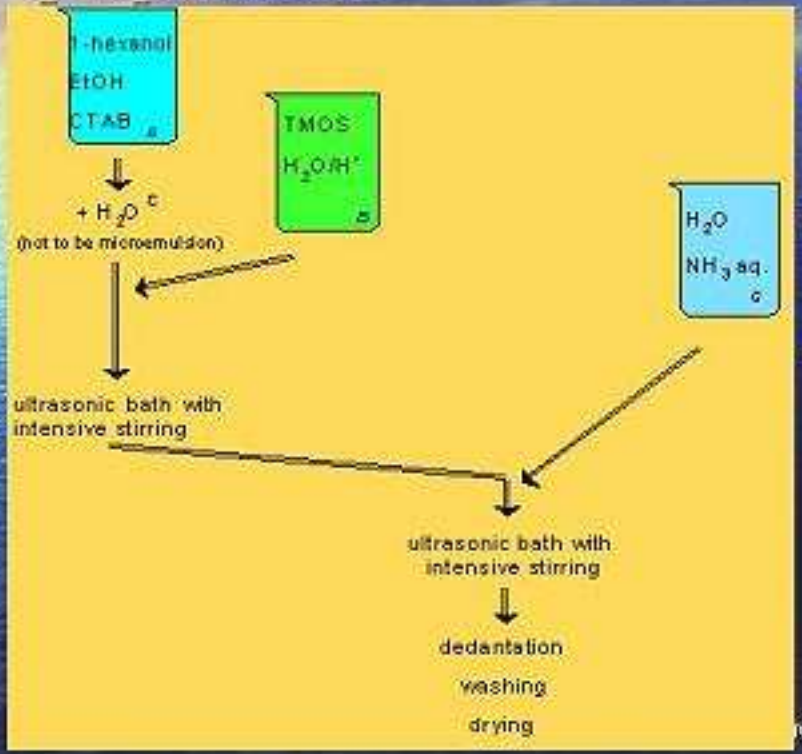
- Size of 25 μm
- Narrow size distribution
- Gelation by temperature increase

Optical microskope



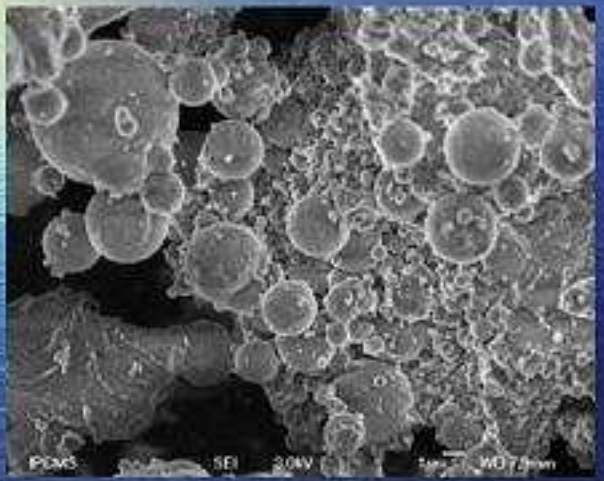
průměr 20 – 25 μm

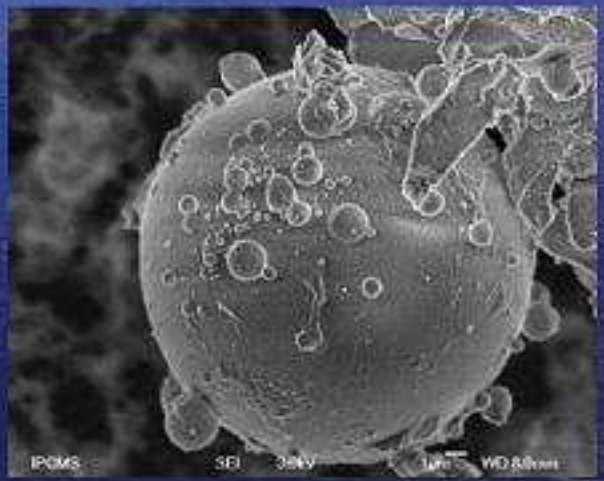
2. SiO_2 spheres – with size about 3μm - preparation



- Size of apprx. 3 μm
- Wide size distribution
- Gelation by ammonia addition

3 µm SiO_2 spheres– SEM



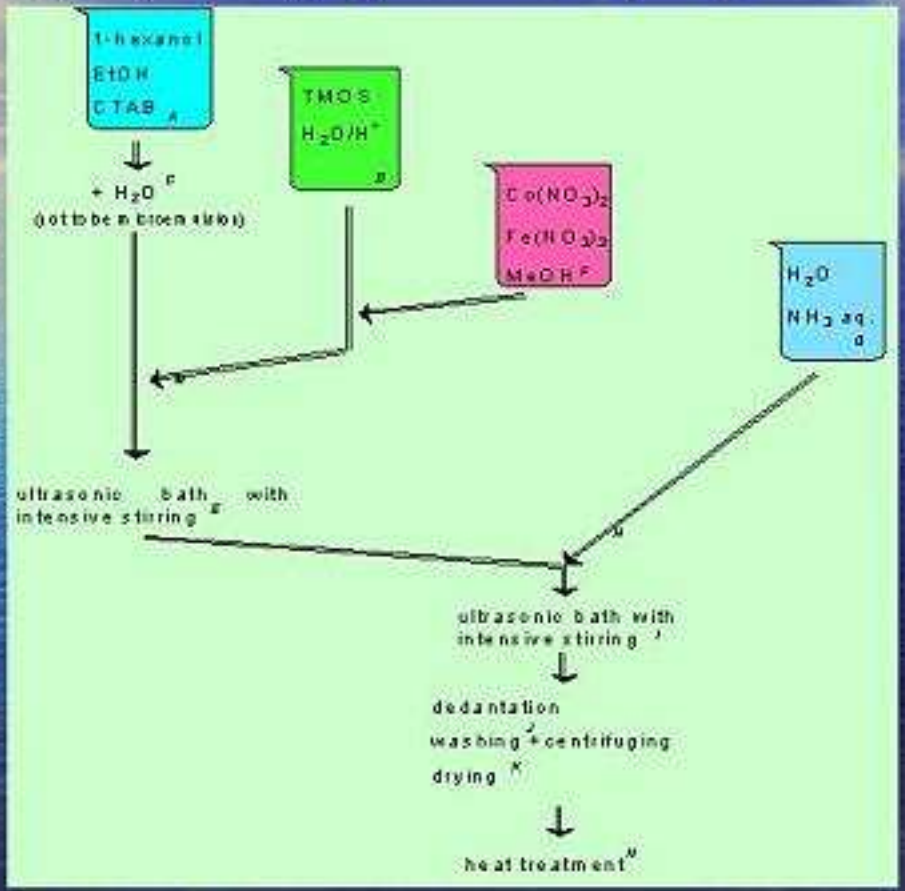


3μm spheres – results and problems

- Large size distribution
- Agregation (both in suspension and in "dry" state)

- Cracking of balls
- Presence of unregular bulk material

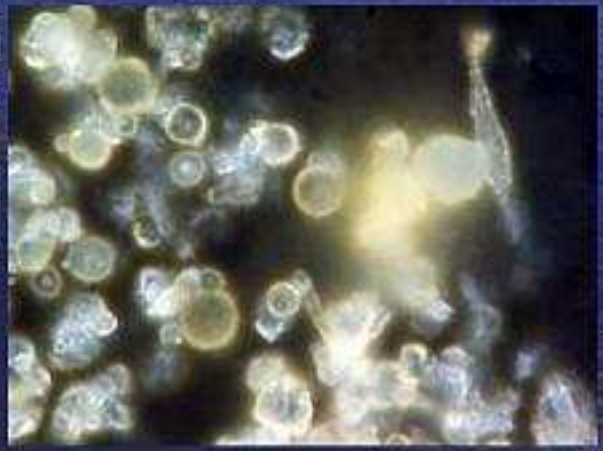
$CoFe_2O_4/SiO_2$ spheres – preparation



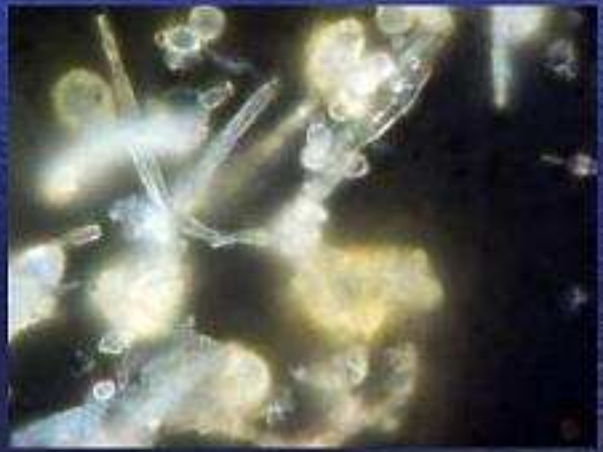
$CoFe_2O_4/SiO_2$ spheres - News

- Gelation works succesfully also in quite acidic media!
- Nitrates introduction does not change too much relations in the emulsion system!

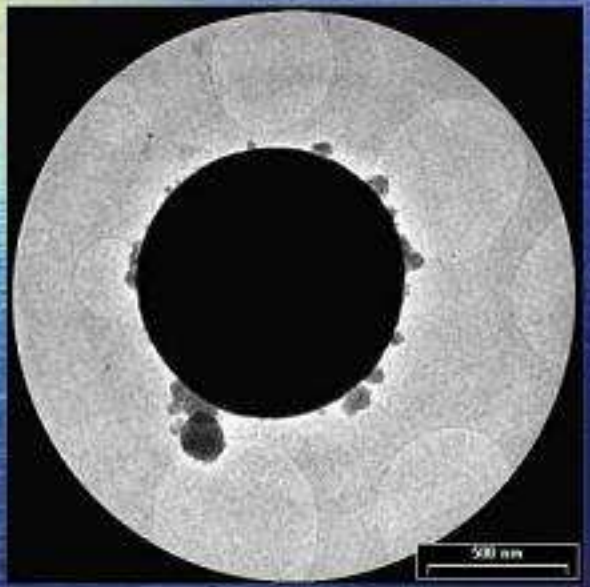
$CoFe_2O_4/SiO_2$ spheres and rods optical microskope

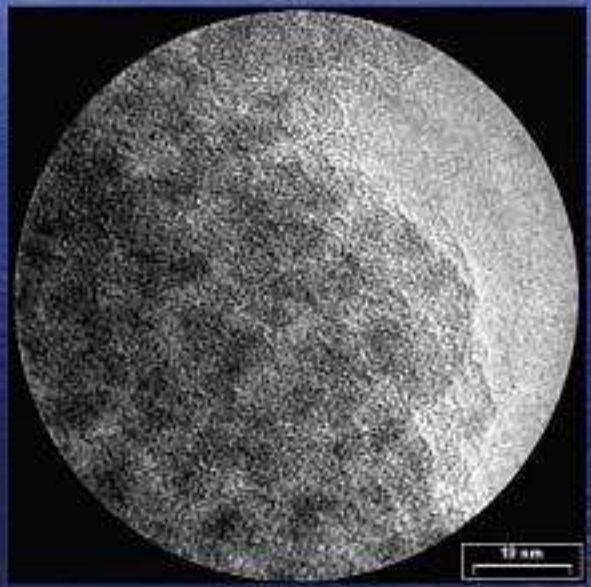


$CoFe_2O_4/SiO_2$ spheres and rods optical microskope

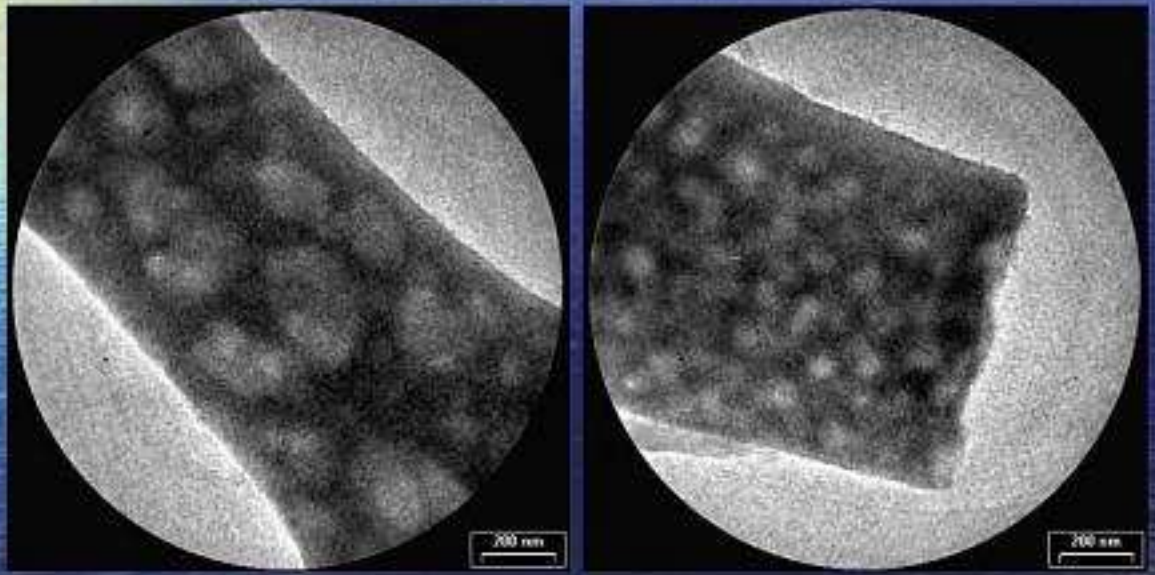


$CoFe_2O_4/SiO_2$ spheres– HRTEM



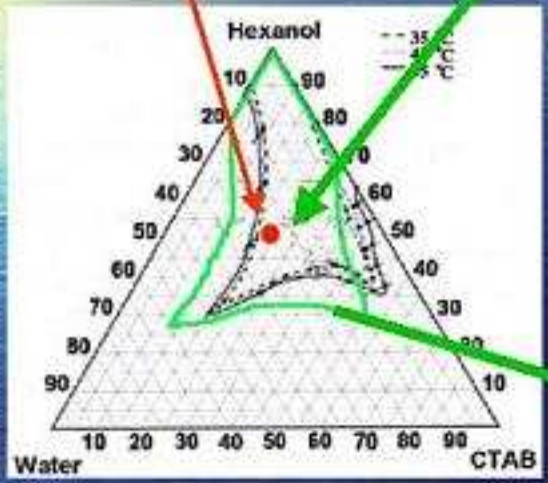


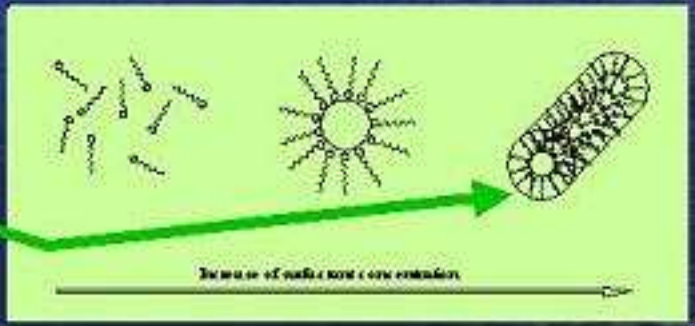
$CoFe_2O_4/SiO_2$ needles –HRTEM



Phase diagram water-hexanol-CTAB

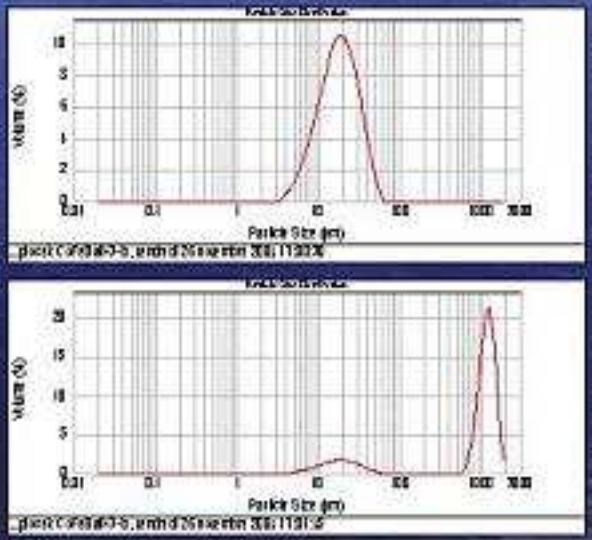
CTAB + $NO_3^{(1-)}$ ⇔ [illegible] + $Br^{(1-)}$





$CoFe_2O_4/SiO_2$ spheres– results

- Measured magnetization = 2.6 emu/$g_{(nanocomposite)}$ = 16 emu/$g_{(spinell)}$ (sample was not saturated)
- Particle size 1-10 μm (problem with particle agregation)



Conclusion

- It was found the way to prepare silica spheres with size about 1 μm
- It is possible to prepare particles with different shape, i.e. spheres , rods, needles
- It is possible to prepare macrospheres of magnetic nanocomposites

To do:

- Optimizing the preparation to have narrower size distribution
- Prepare "spherical" nanocomposites with other magnetic materials
- Enhance macroporosity of prepared materials

Thanks to all coworkers,
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Thank you