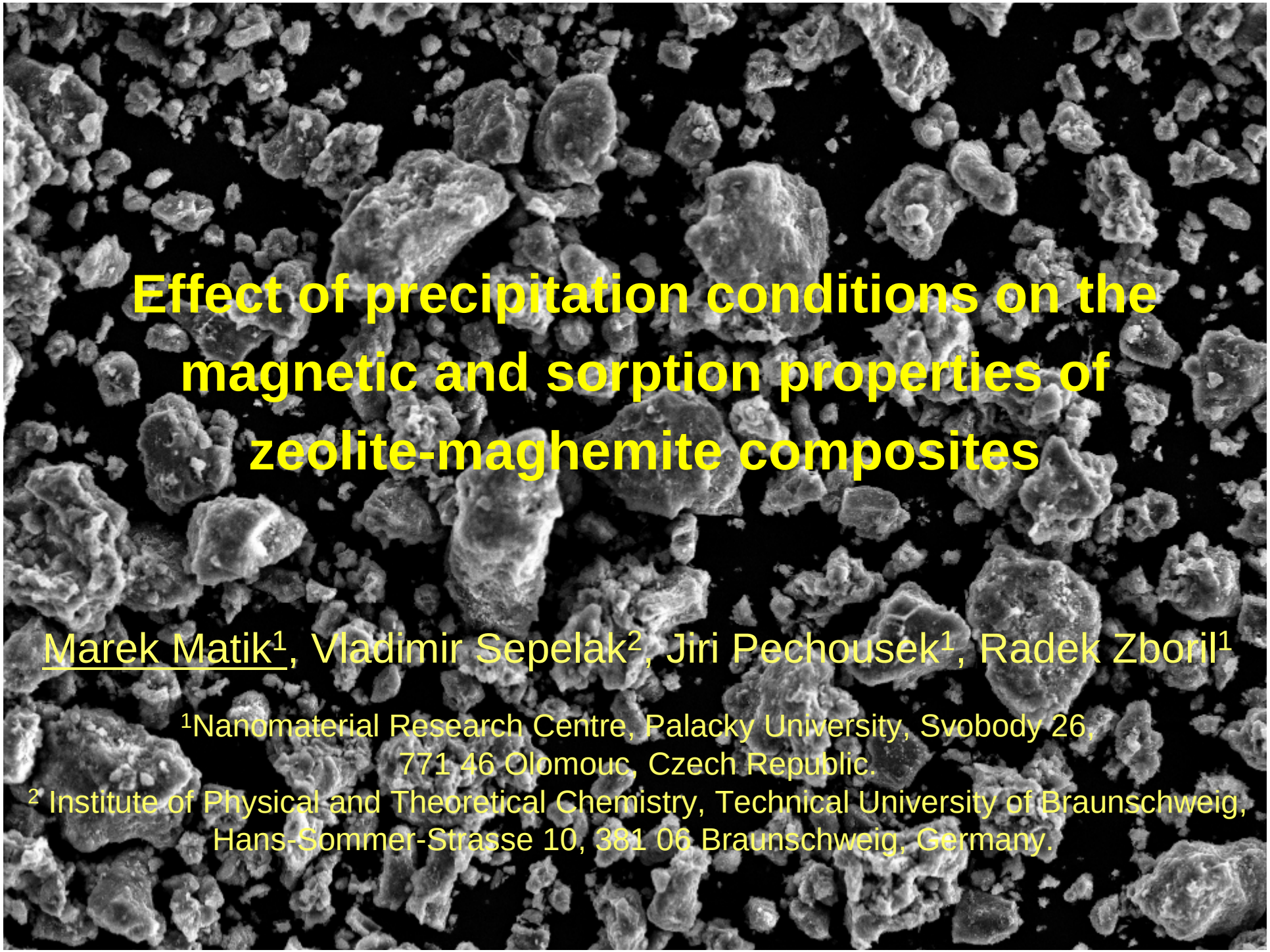

Effect of precipitation conditions on the magnetic and sorption properties of zeolite-maghemite composites

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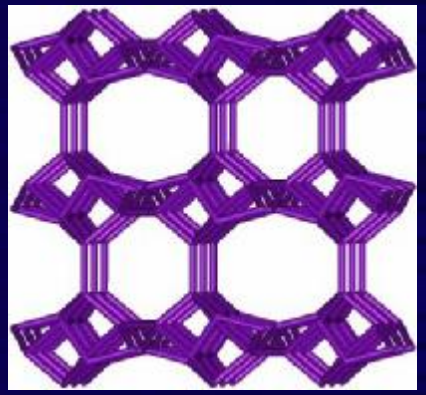
MAGNETIC ZEOLITE

WHY ZEOLITE ?

good adsorbent of cations

cheap material (Nižný Hrabovec deposit)



WHY MAGNETIC MODIFICATION ?

magnetic separation

maghemite – adsorbent

(cations and anions)

ZEOLITE

General formula of zeolite:

Me2/m.Al2O3.nSiO2.pH2O,

where

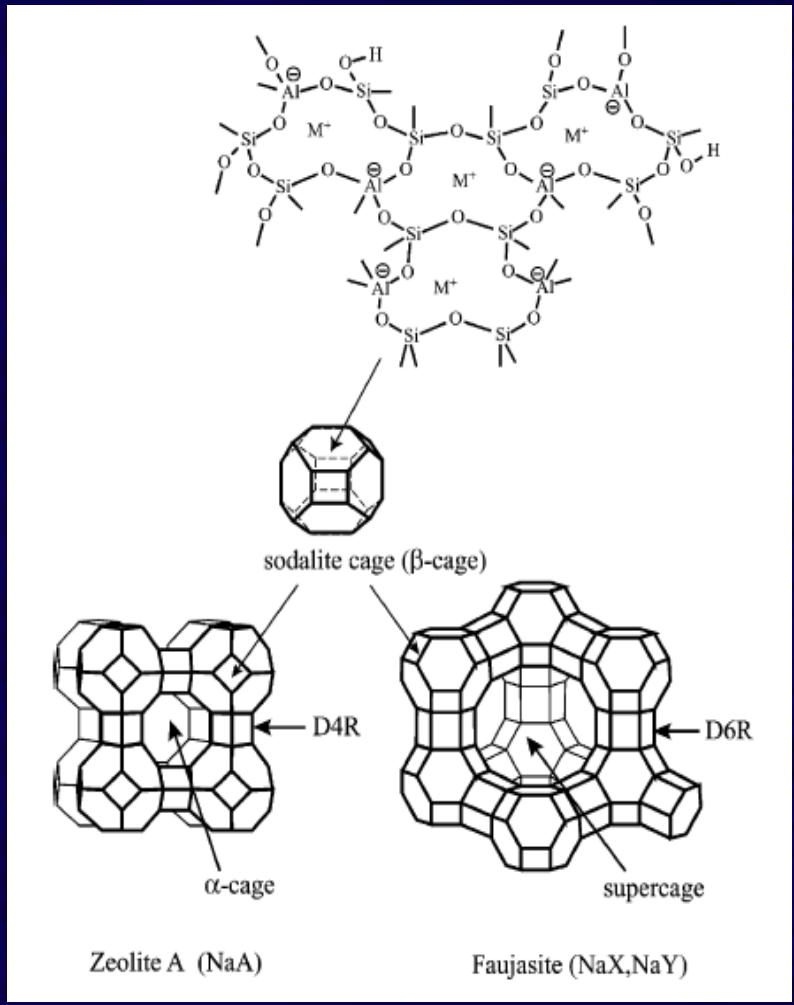
Me – cation of alkali (m=1), and
alkali earth metal (m=2)

n - SiO_2/Al_2O_3 molar ratio

p – water molecules

Classification:
Natural
Modificated : cation changes
: ratio Al/Si changes
: surface changes
Synthetic

Usage: catalysis, adsorption



MODIFICATION WITH Fe PARTICLES

- Modification of natural zeolite with magnetite and maghemite particles
- Coverage of zeolite surface by magnetic metal nanoparticles (Fe, Co, Ni),
- Incorporation of Fe ions into zeolite lattice

SYNTHESIS

$$\mathbf{ZEOLIT + Fe^{2+} + Fe^{3+} + OH^{-} \rightarrow ZEOLIT - \gamma\text{-}Fe_2O_3}$$

CONDITIONS OF SYNTHESIS

Zeolite : Maghemite ratio :

2:1
5:1
10:1

Interaction time after precipitation:

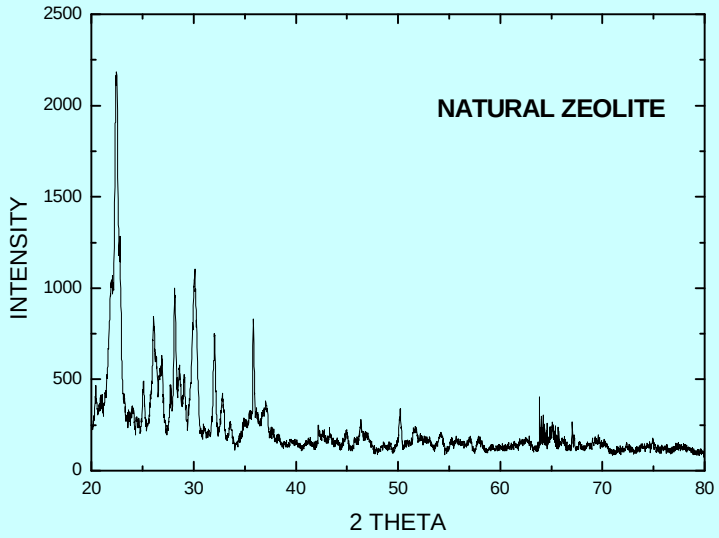
0 min.
30 min.
90 min.
24 hod.

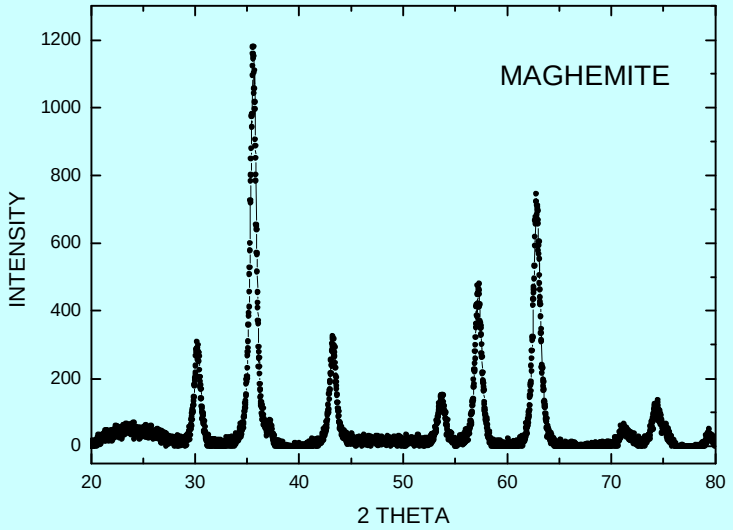
Temperature of precipitation :

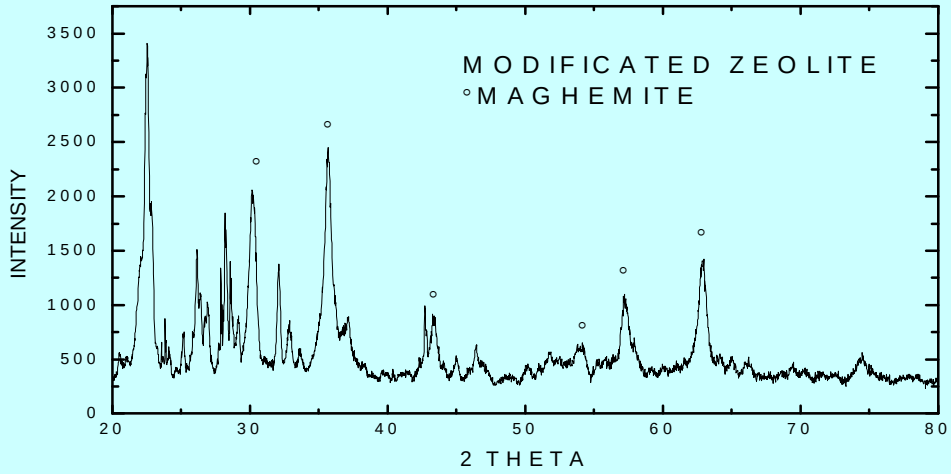
20 °C
50 °C
85 °C

Identification: XRD, Mössbauer spectroscopy, SEM, TEM, VSM.

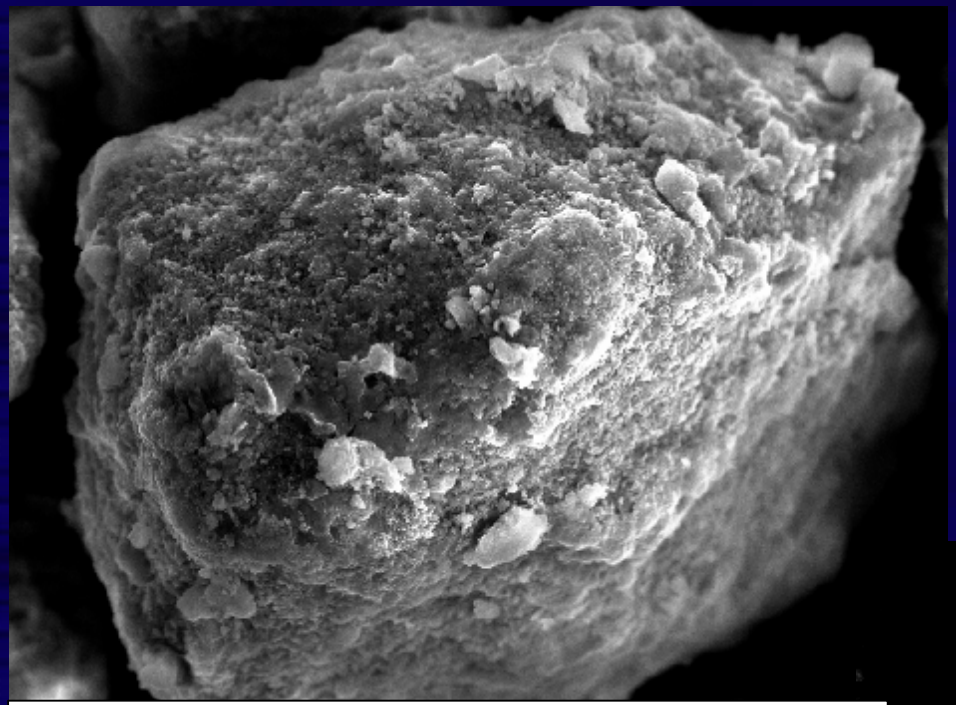
XRD-ANALYSIS

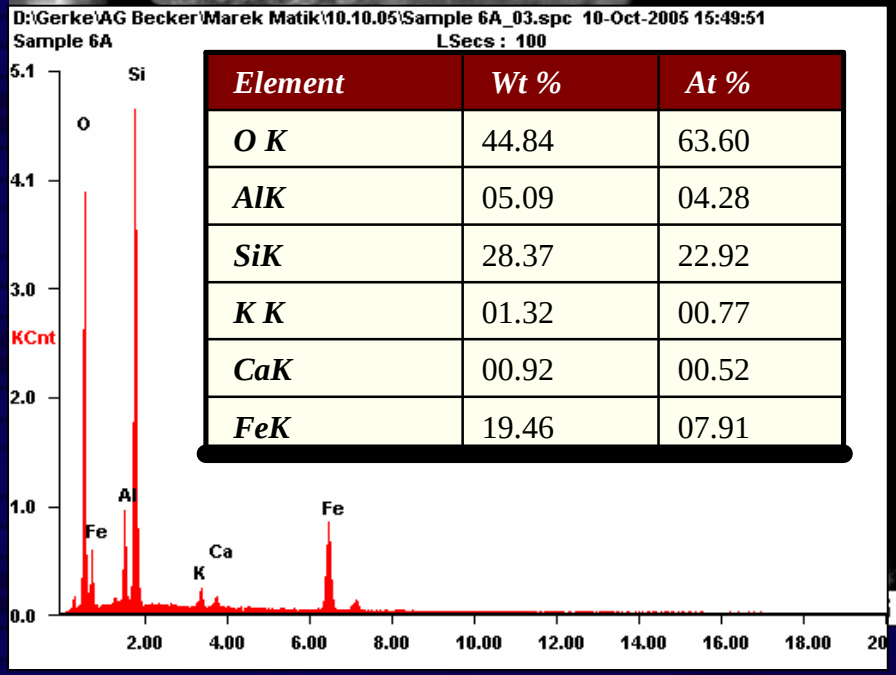




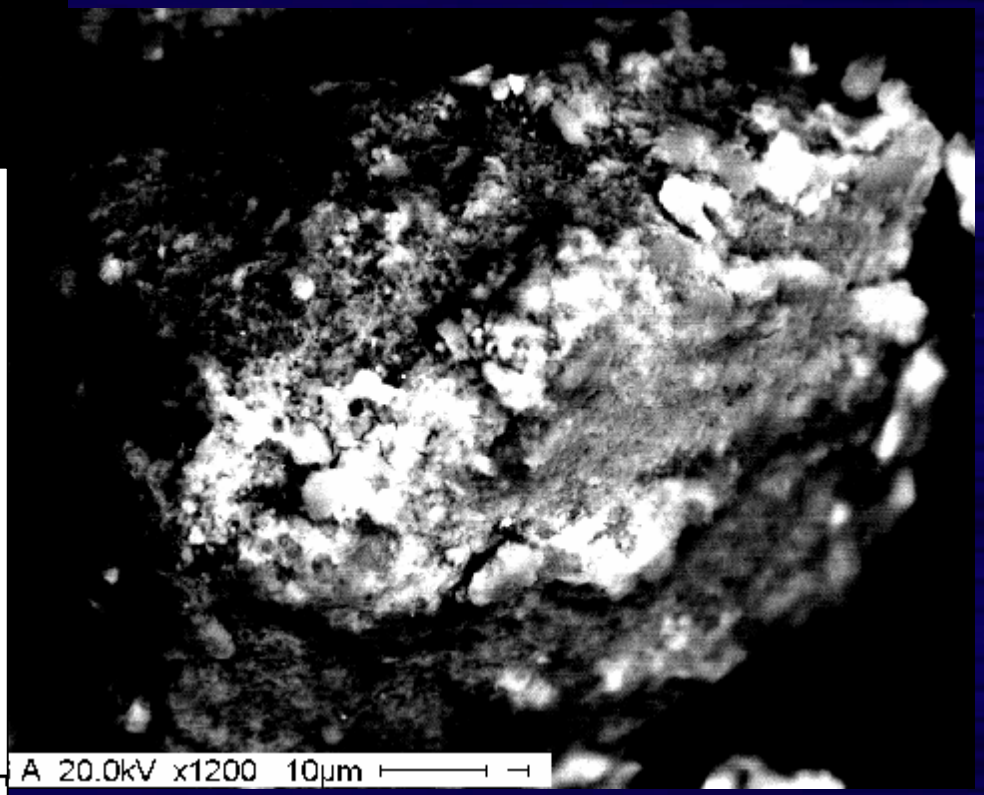


SEM





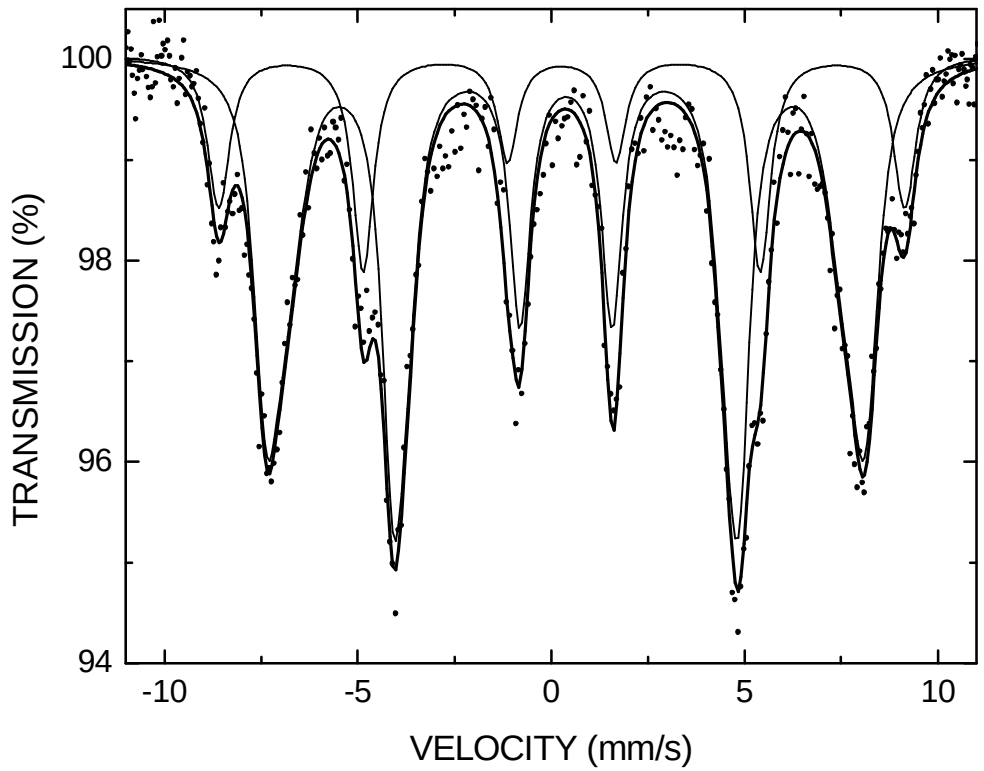
Element	*Wt %*	*At %*
O K	44.84	63.60
AlK	05.09	04.28
SiK	28.37	22.92
K K	01.32	00.77
CaK	00.92	00.52
FeK	19.46	07.91



MOSSBAUER

T = 10K, $H_{ext.}$ = 5T
$H_{ext.} \perp \gamma$-rays

$$H_{obs(A)} = H_{(A)} + H_{ext}$$
$$H_{obs[B]} = H_{[B]} - H_{ext}$$



$(Fe^{3+})[\ Fe^{3+}_{5/3}\ [\ ]_{1/3}]O_4$ 3 : 5
1 : 3

	state	IS (mm/s)	B (T)		I (p) (%)	σ_B (T)
A	Fe^{3+}	0.38	55.00		24.77[a]	0.56
	A_1/A_3			1.54		
	A_2/A_3			2.10		
	Fe^{3+}	0.49			75.23	
B	Komp. 1		48.00[a]		(0.55)	0.93
	Komp. 2		45.00[a]		(0.45)	1.89
	A_1/A_3			2.30		
	A_2/A_3			2.24		

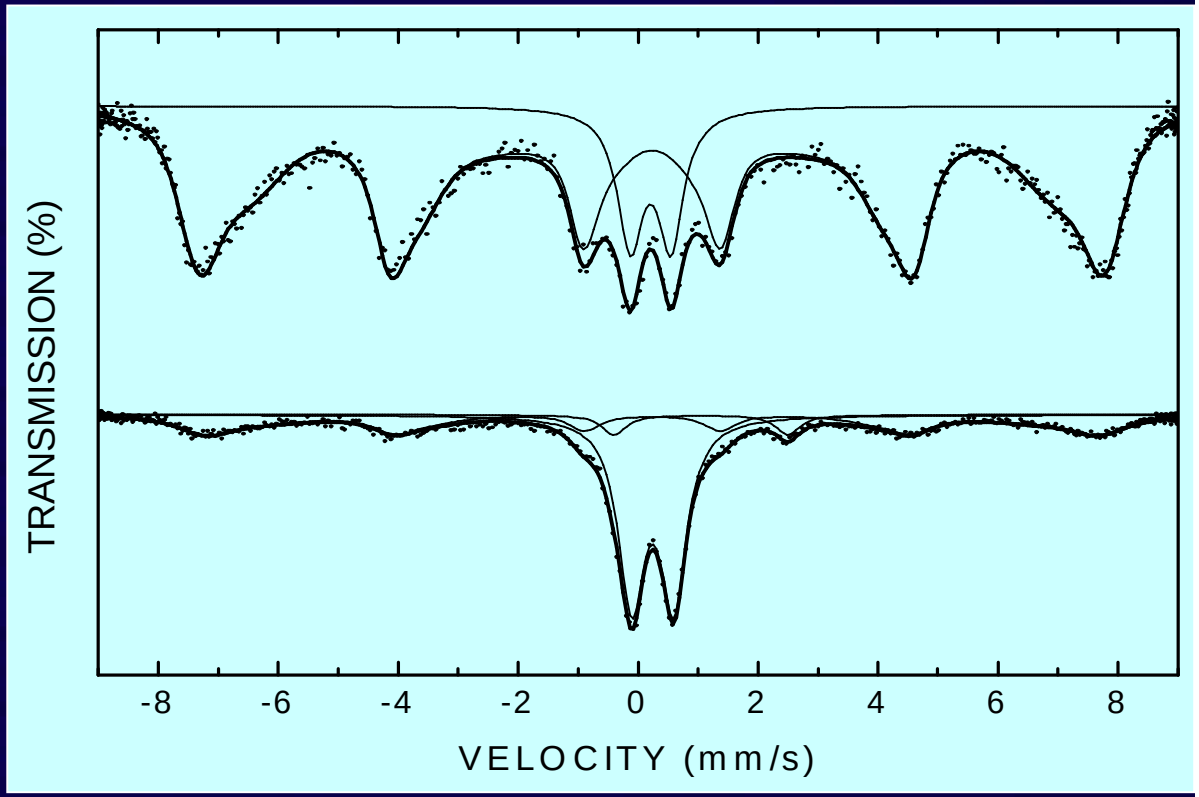
[a] fixed parameters

MOSSBAUER

Weight ratio

Zeolite/Fe

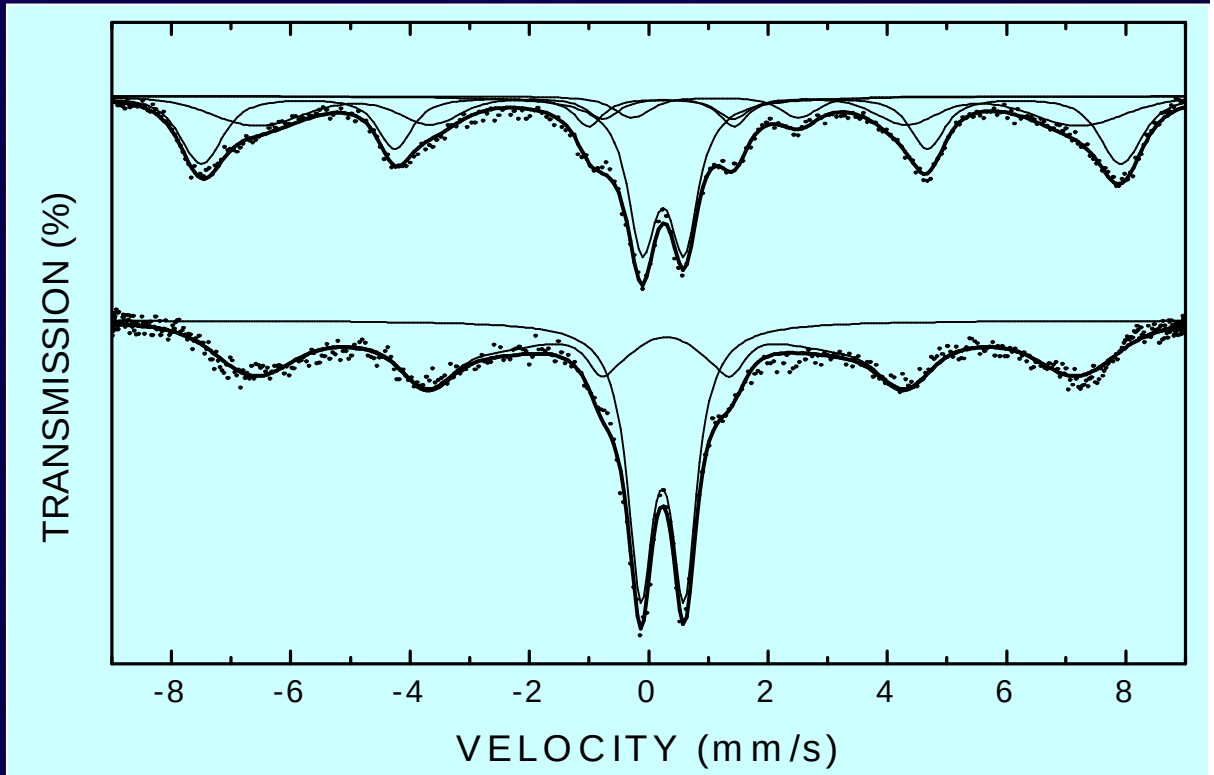
2:1, 10:1



Weight ratio zeolit : Fe	state	IS (mm/s)	QS / B (mm/s) / (T)	*I* / (p) (%)	σ_B (T)
2 : 1	$Fe^{3+}_{sup.}$	0,32	0,66	12,45	0
	Fe^{3+}	0,34		87,55	
	Komp 1		46,88	(35,42)	1,23
	Komp 2		42,21	(33,93)	3.20
	Komp 3		27,81	(30,65)	12,96
10 : 1	$Fe^{3+}_{sup.}$	0,35	0,70	59,45	0.11
	Fe^{2+}	1,15	2,90	6,21	0
	Fe^{3+}	0,35		34,34	
	Komp 1		46,17	(32)	1,52
	Komp 2		43,00	(68)	5,95

MOSSBAUER

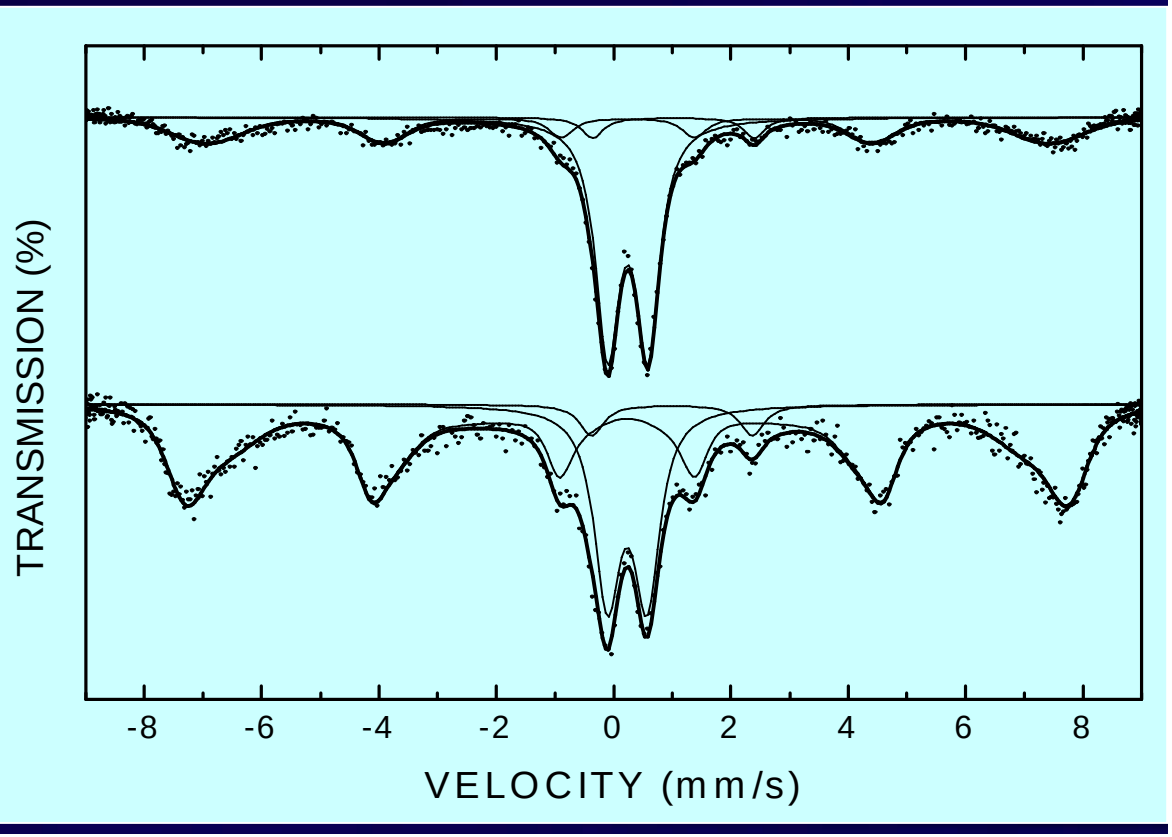
Temperature
of precipitation
20°C, 85°C



Temperature (°C)	state	IS (mm/s)	QS / B (mm/s) / (T)	*I*/ (p) (%)	σ_B (T)
20	$Fe^{3+}_{sup.}$	0,35	0,70	28,97	0
	Fe^{2+}	1,23	2,79	5,27	0,30
	Fe^{3+}	0,32	47,80	36,40	1,27
	Fe^{3+}	0,44	42,73	29,36	3,93
85	$Fe^{3+}_{sup.}$	0,34	0,73	39.16	0.01
	Fe^{3+}	0.40		60.84	
	Komp 1		42.89	(51)	3.16
	Komp 2		30.59	(49)	10.34

MOSSBAUER

Interaction time
after precipitation
0, 24 hours.



Time (hour)	state	IS (mm/s)	QS / B (mm/s) / (T)	*I*/ (p) (%)	σ_B (T)
0	$Fe^{3+}_{sup.}$	0,35	0,68	61.75	0.13
	Fe^{2+}	1,13	2,75	5,26	0
	Fe^{3+}	0,35	44.54	32.99	3.17
24	$Fe^{3+}_{sup.}$	0,34	0,67	28.04	0.13
	Fe^{2+}	1.10	2.74	4.30	0
	Fe^{3+}	0.34		67.65	
	Komp 1		46.72	(44)	1.24
	Komp 2		42.69	(36)	2.98
	Komp. 3		27.20	(20)	10.08

VSM

ZFC-FC

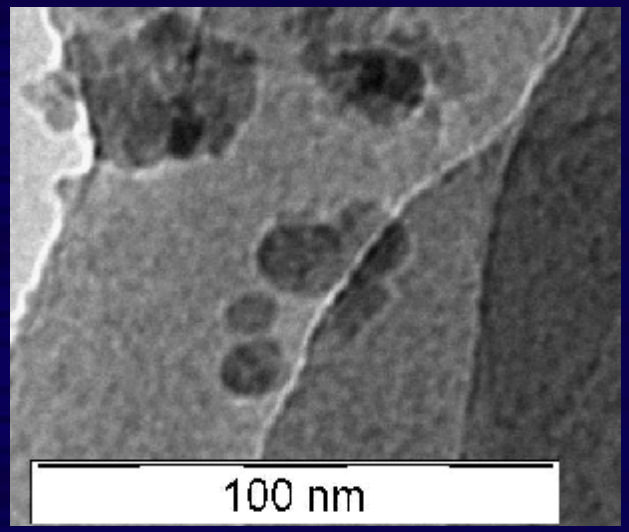
10-240 K H=0.01 T

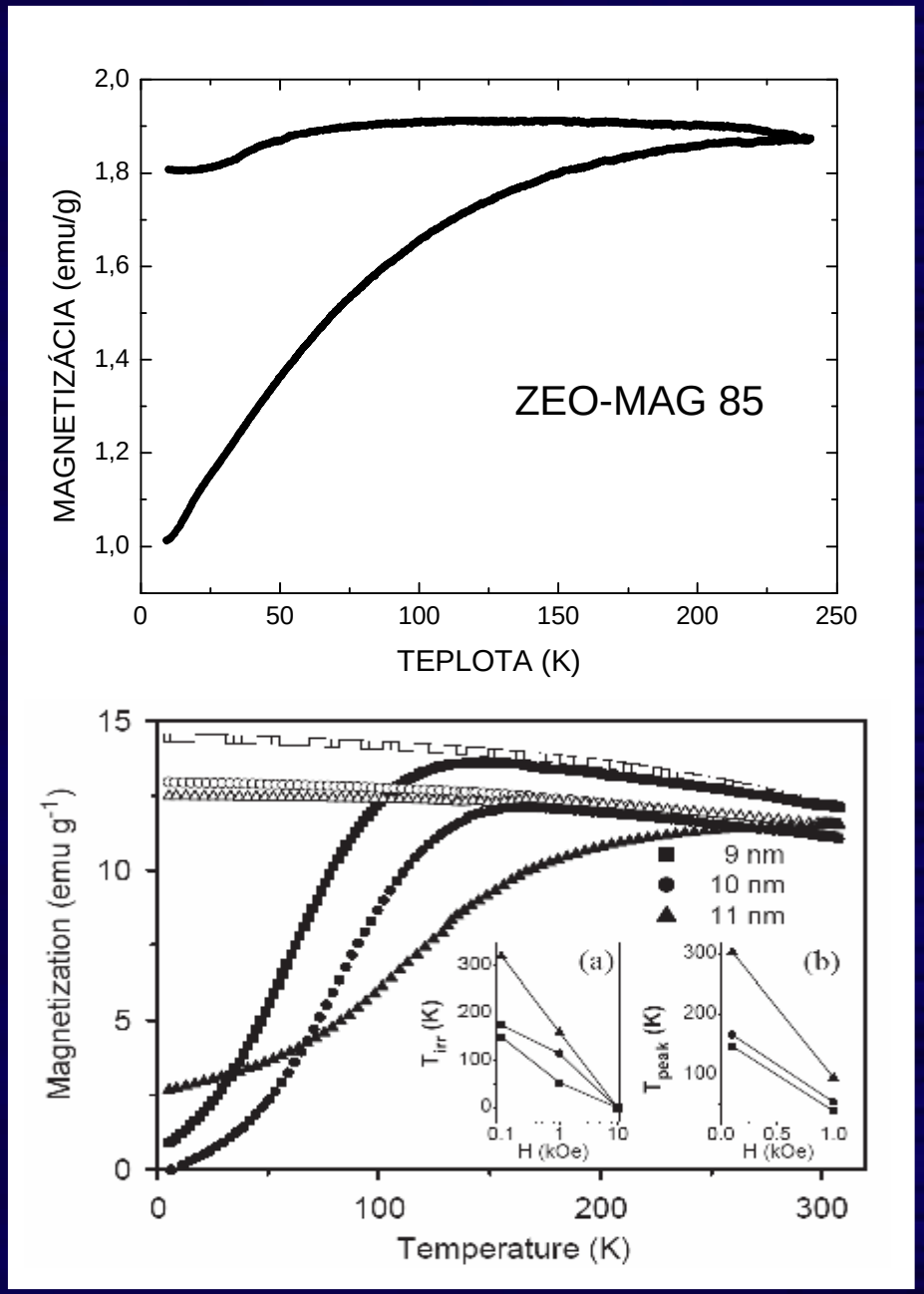
zero-field-cooling (ZFC)

and field-cooling (FC) procedures

spin-glass systems

collective blocking of particle moments





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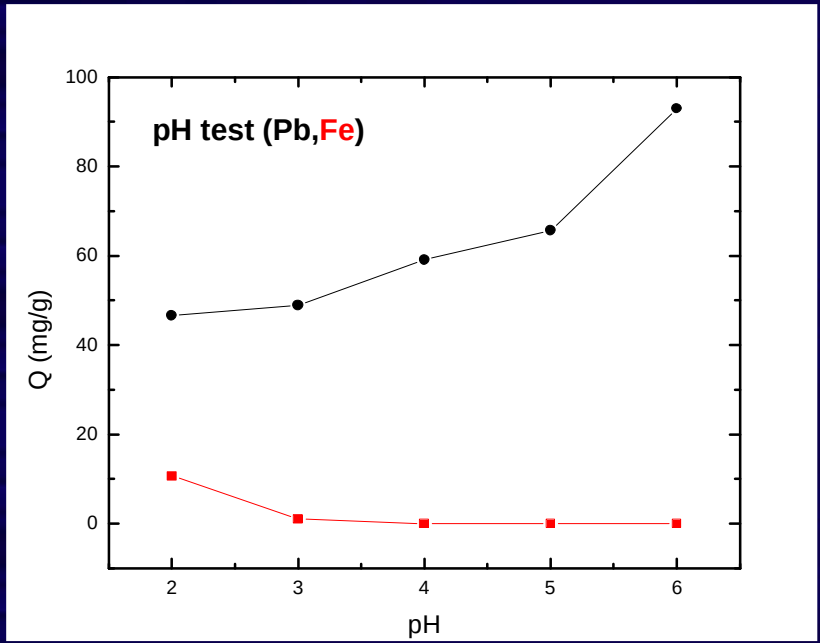
SURFACE AND MAGNETISM

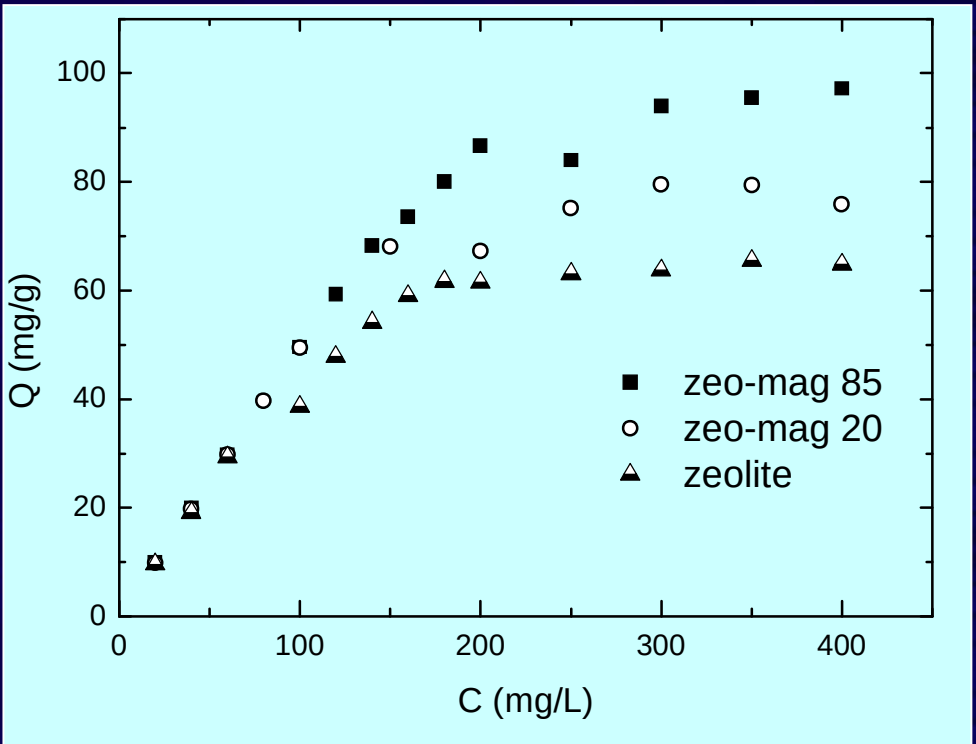
- Indirect identification of maghemite content with volume susceptibility
- Comparison of surface area and pore volume with magnetic properties of modified zeolite

Surface and magnetic characterization of magnetic composites

	Surface area [m^2/g]	Pore volume [cm^3/g]	Susceptibility [SI units]
Natural zeolite	20.85	0.067	270
Temperature 20°C	25.78	0.099	107 287
Temperature 85°C	33.90	0.117	158 582
Zeolite/Fe 2	49.72	0.14	254 765
Zeolite/Fe 10	31.53	0.084	73 258
Time interact. 0 hours.	34.19	0.07	96 847
Time interact. 24 hours.	27.57	0.102	114 723

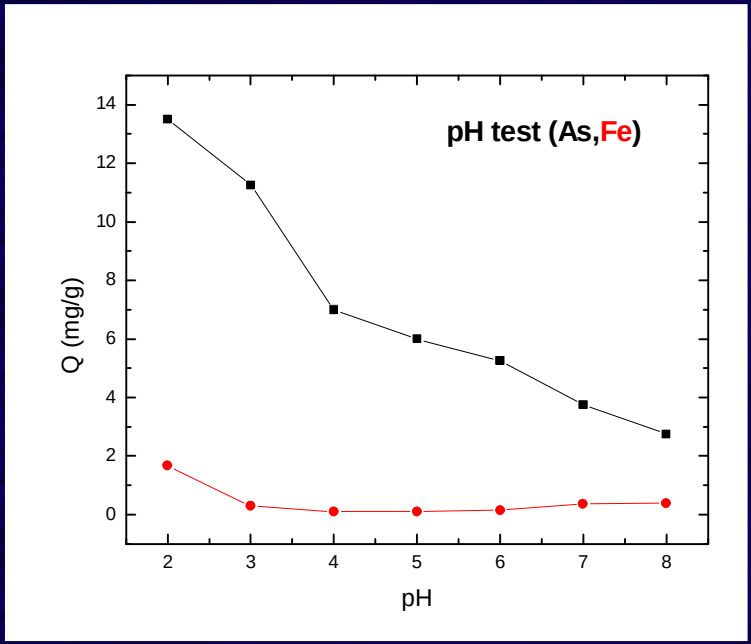
ADSORPTION of Pb

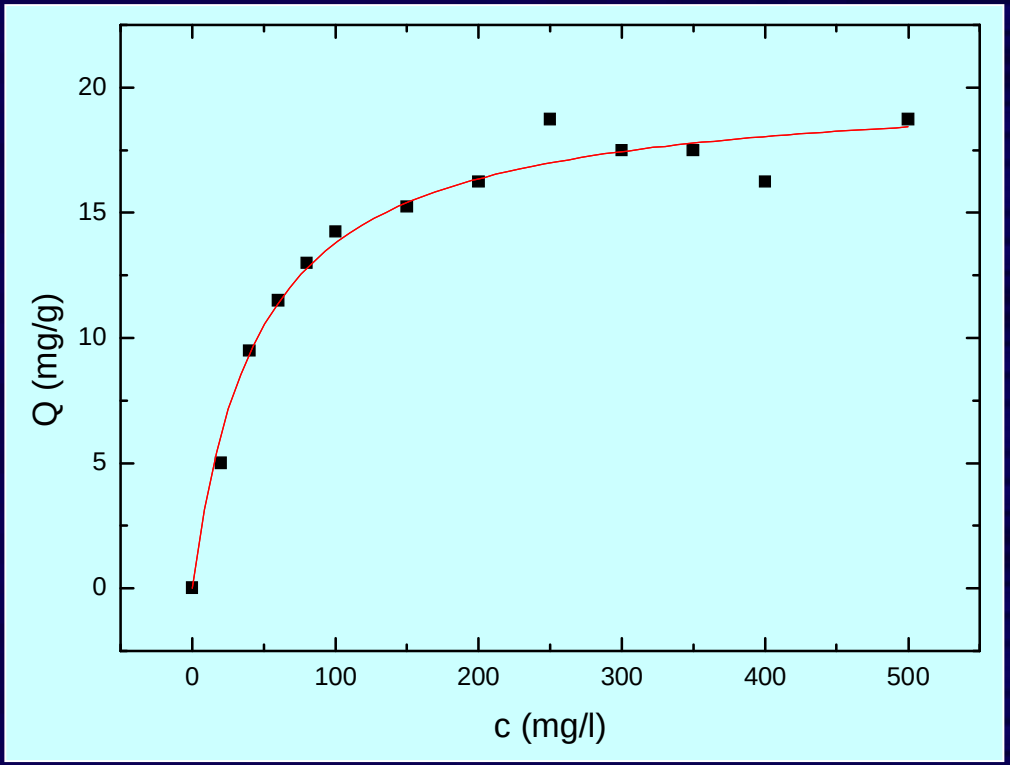




Composite conc. .....2 g/L
Starting conc. Pb^{2+} .....20 – 400 mg/L
Adsorption time..... 24 hours
Temperature (const.) ..... 25°C
Ionic strength.....I = 0

ADSORPTION of As





Composite conc. .....2 g/L
Starting conc. AsO_4^{3-} .....20 – 500 mg/L
Adsorption time..... 24 hours
Temperature (const.) ..... 25°C
Ionic strength.....I = 0

CONCLUSIONS

- **Particle size dependence on reaction conditions**
- **Increasing of composite surface**
- **Ability of cation and anion adsorption**
- **Magnetic properties**