



Synthesis of Fe_3O_4 magnetic nano and micro-particles coated with chitosan and polyacrylic acid

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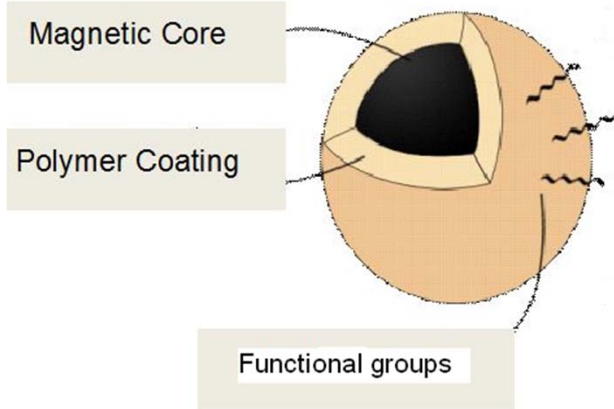
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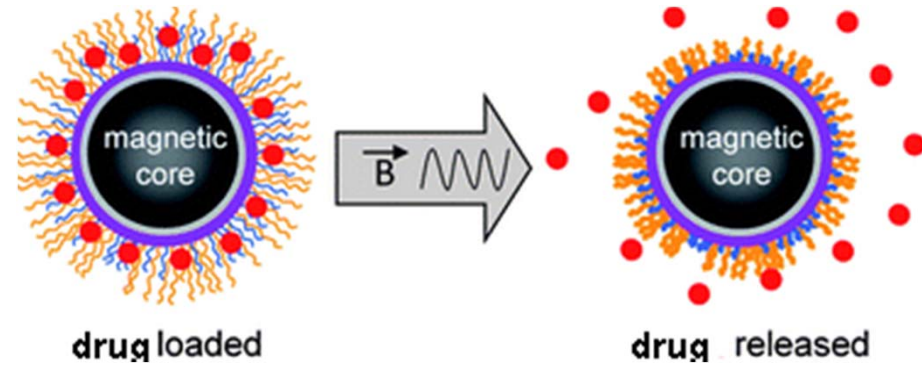
OVERVIEW

- ▶ **Preparation and characterization of Fe_3O_4 chitosan coated nanoparticles**
- ▶ **Formation and characterization of chitosan- polyacrylic acid Fe_3O_4 coated microparticles; their magnetic properties**
- ▶ **Adsorption properties of the polymer coated microparticles - their use for the retention of reactive dyes from the aqueous solutions**

Polymer coated magnetic particles



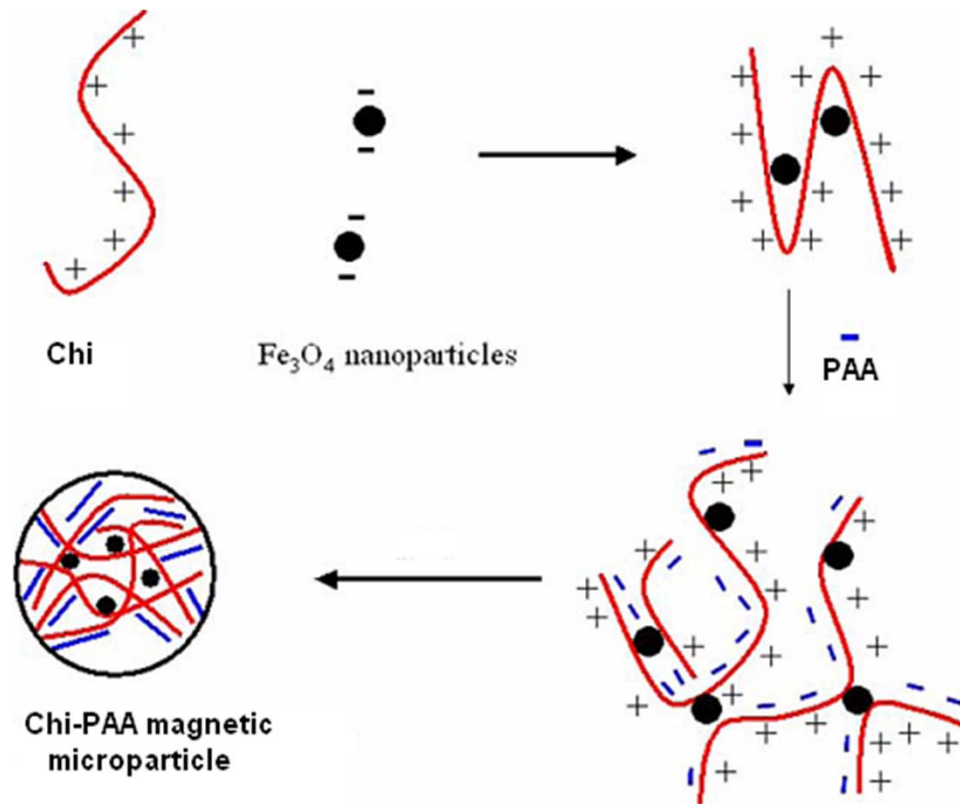
Drug carriers



Sorption of pollutants from aqueous medium

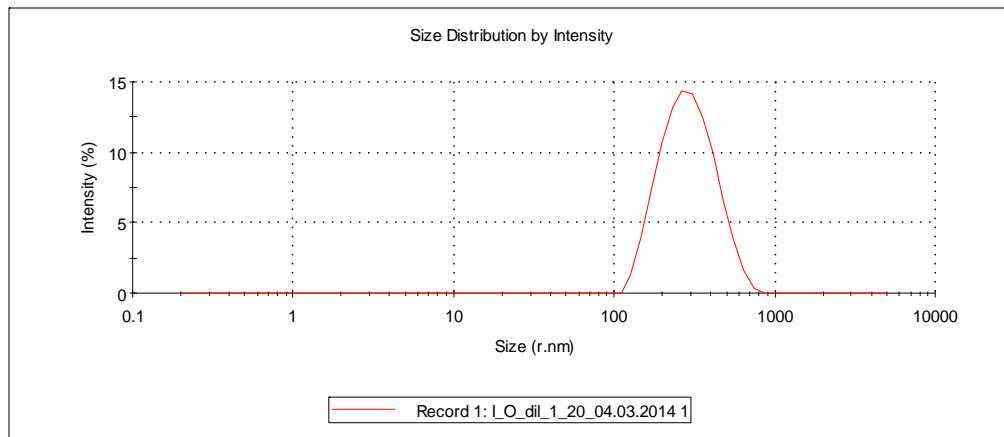


Preparation of polymer coated microparticles

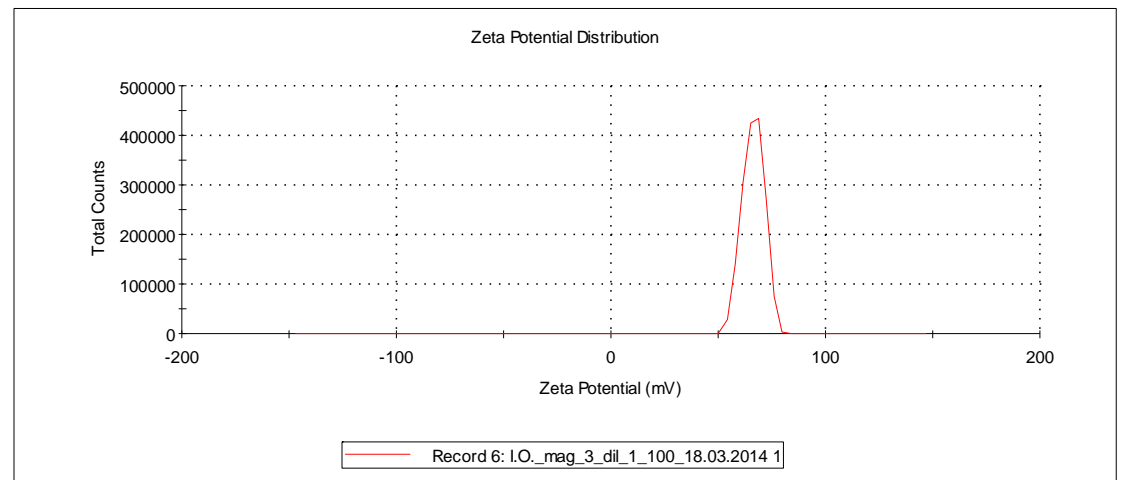


Characterization of Fe_3O_4 chitosan coated nanoparticles

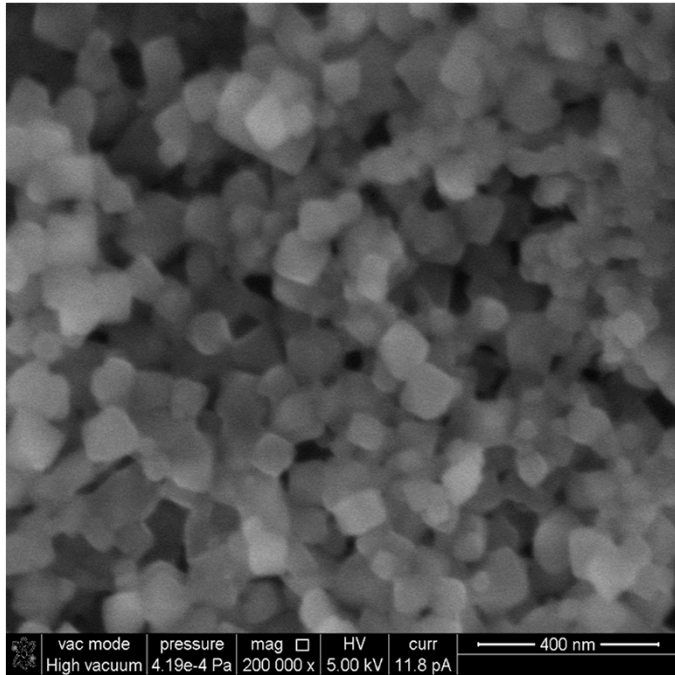
Size distribution



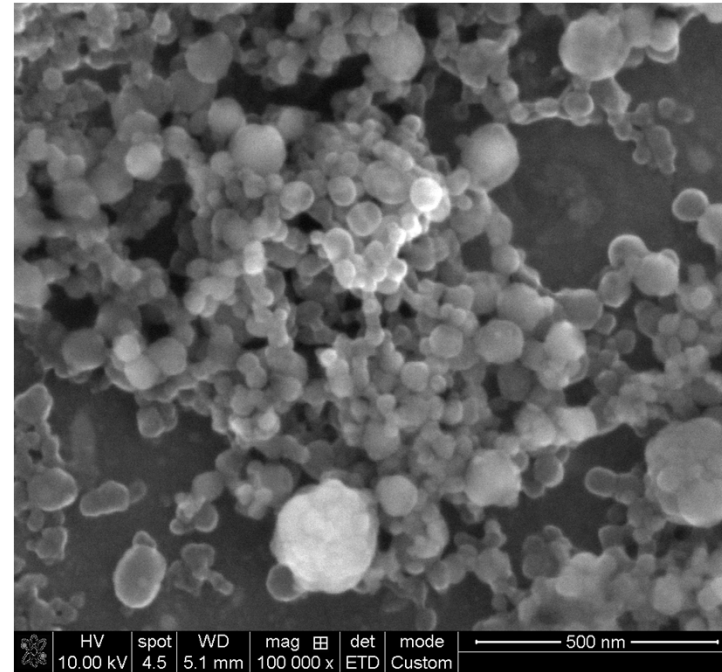
Zeta Potential



SEM images of chitosan coated iron oxide nanoparticles

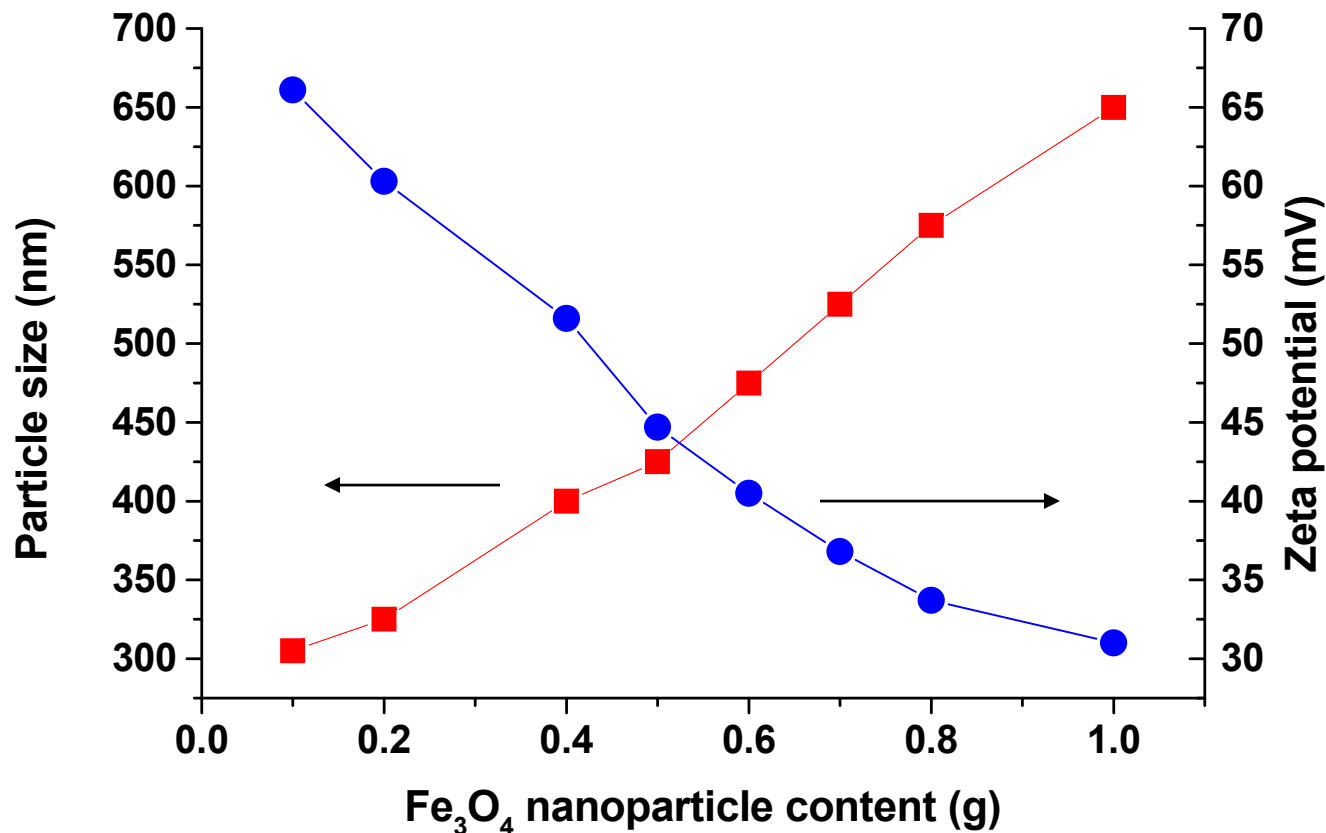


Iron oxide nanocrystallites

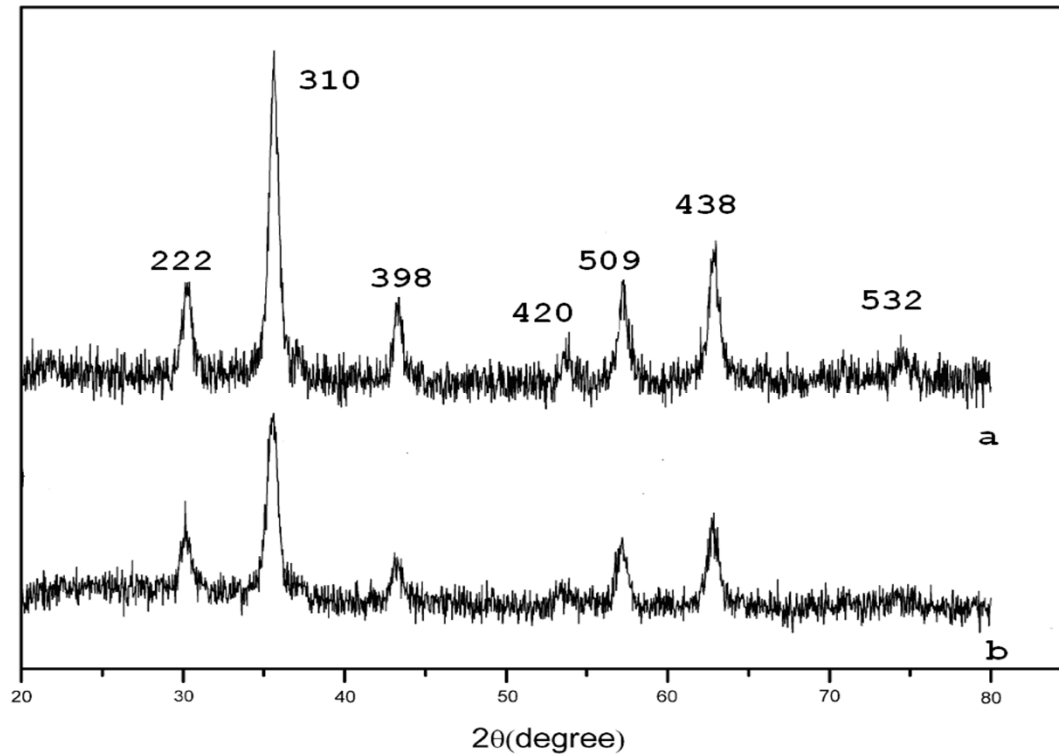


Iron oxide chitosan coated nanoparticles

Effect of Fe_3O_4 content on the particle properties



XRD patterns



Fe_3O_4 nanoparticles (a); Chi coated Fe_3O_4 nanoparticles (b)

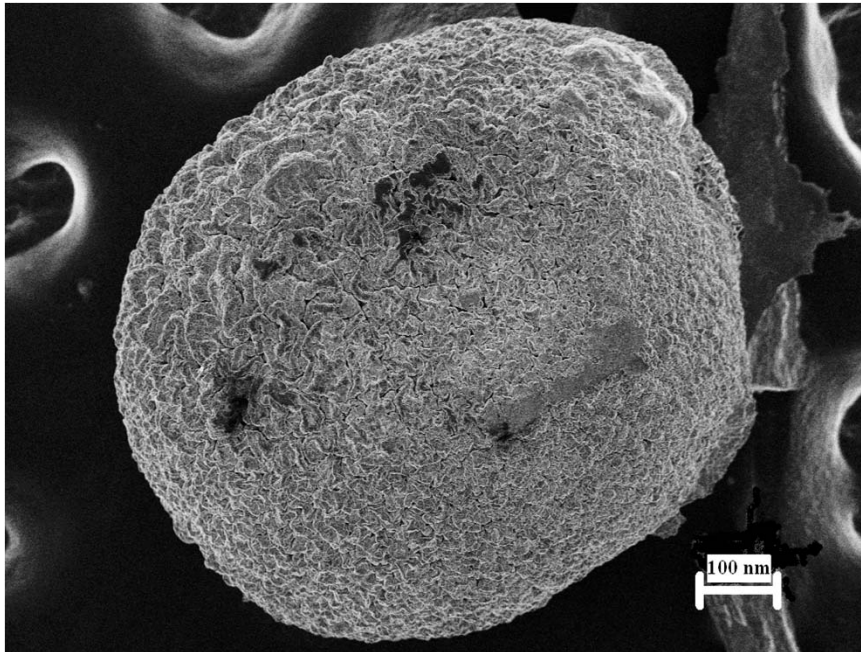
Coated nanoparticles have a core of pure Fe_3O_4 with a cubic inverse spinel structure

Microparticles porosity and surface area – BET method

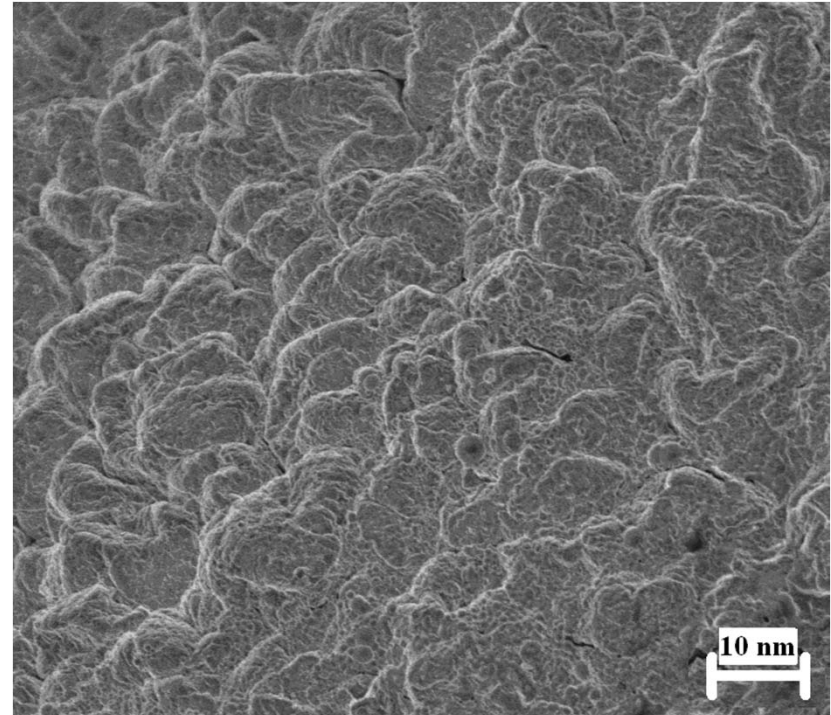
Particle type	Mean pore radius Broekhoff- de Boer (nm)	Surface area BET method (m²/g)
Fe₃O₄ –Chi coated (aggregated)	25	248
Fe₃O₄ –Chi coated (simple)	15	285
Fe₃O₄ –Chi-PAA coated (PCM)	9	355

Analysis of the particles have been made by the nitrogen adsorption method

SEM microscopy of polymer coated microparticles (PCM)



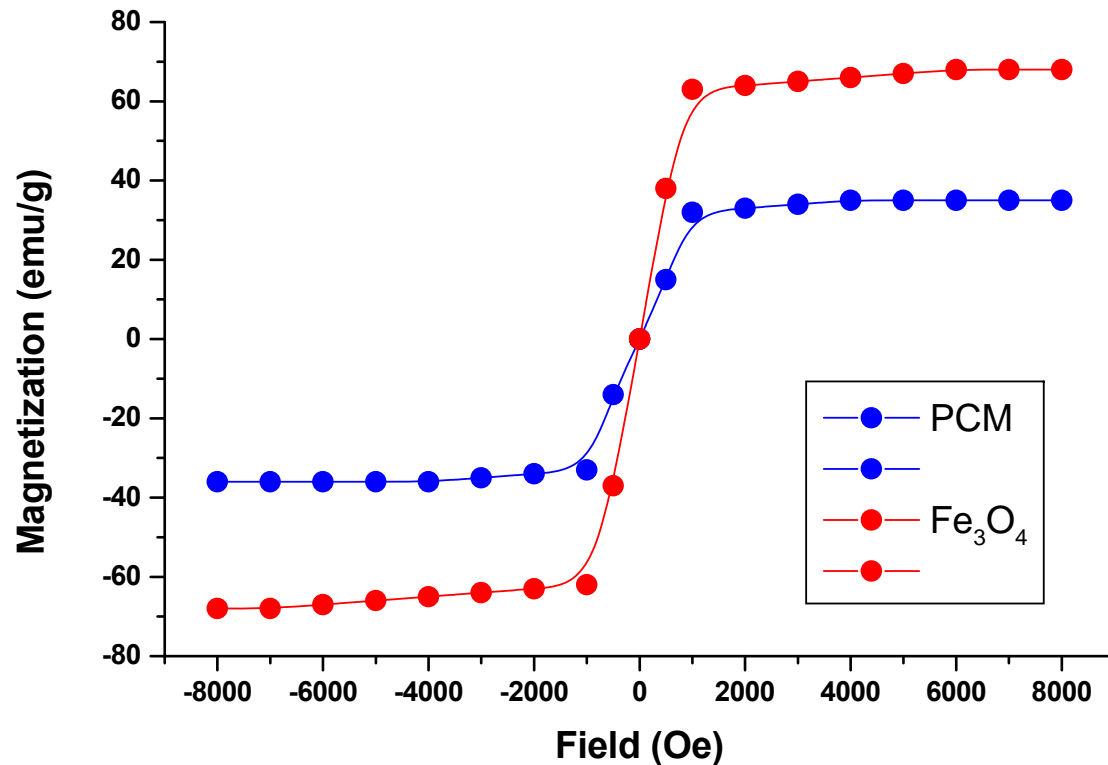
Chi-PAA microparticles



Surface of PCM particles

Magnetic properties

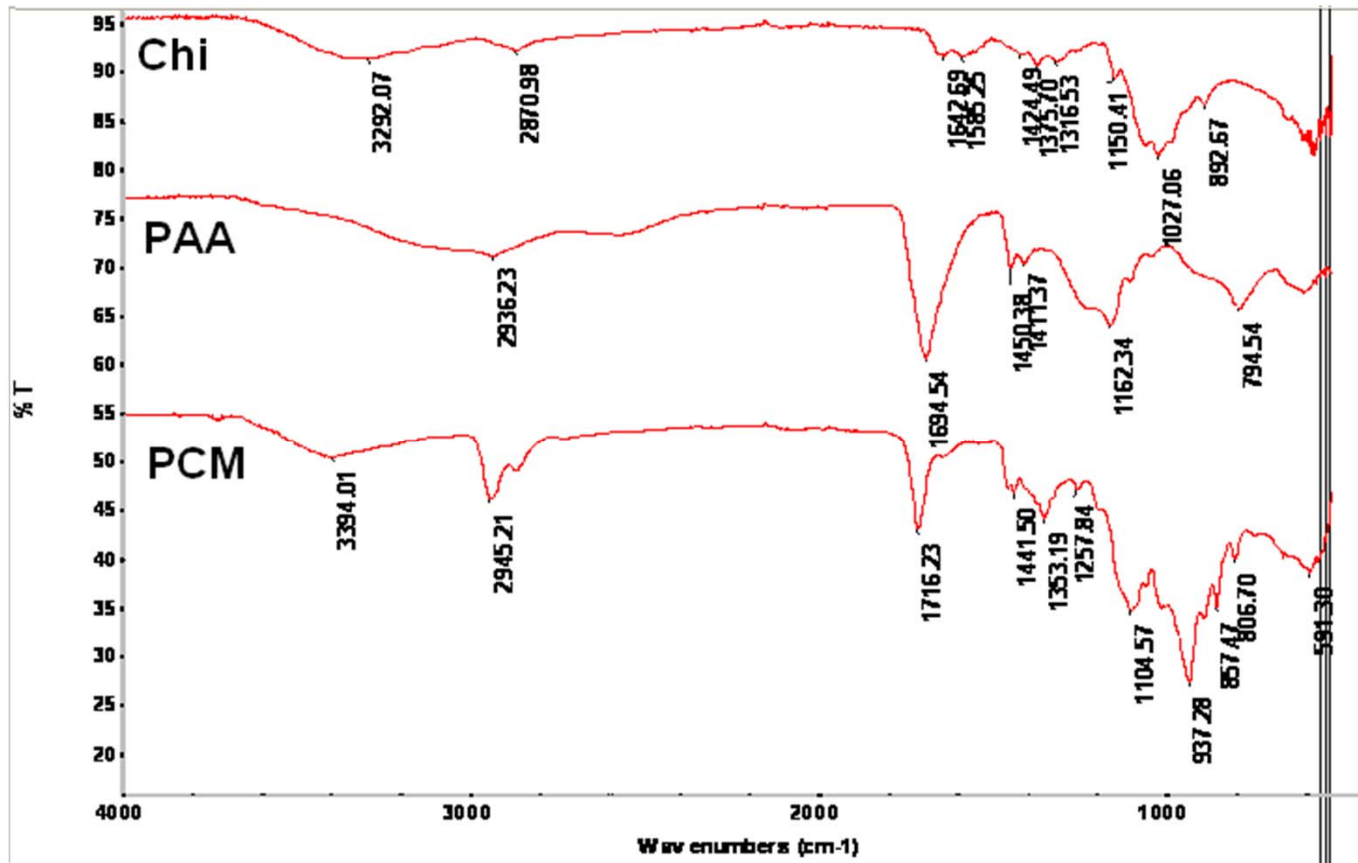
- ▶ Magnetization curve for pure magnetite nanoparticles, and for Fe_3O_4 magnetic nanoparticles, at room temperature



Saturation magnetization showed good superparamagnetic properties for the polymer coated microparticles.

Formation of the coated layers

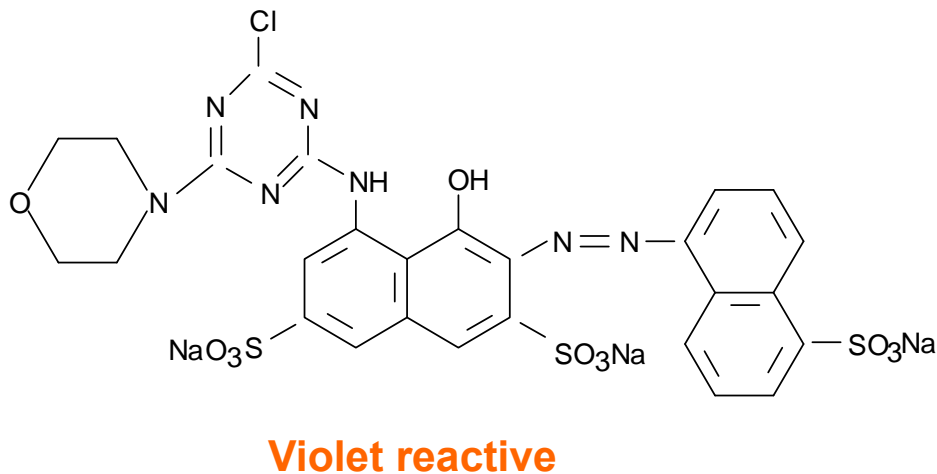
Interaction between chitosan and PAA on iron oxide microparticles - the FTIR analysis



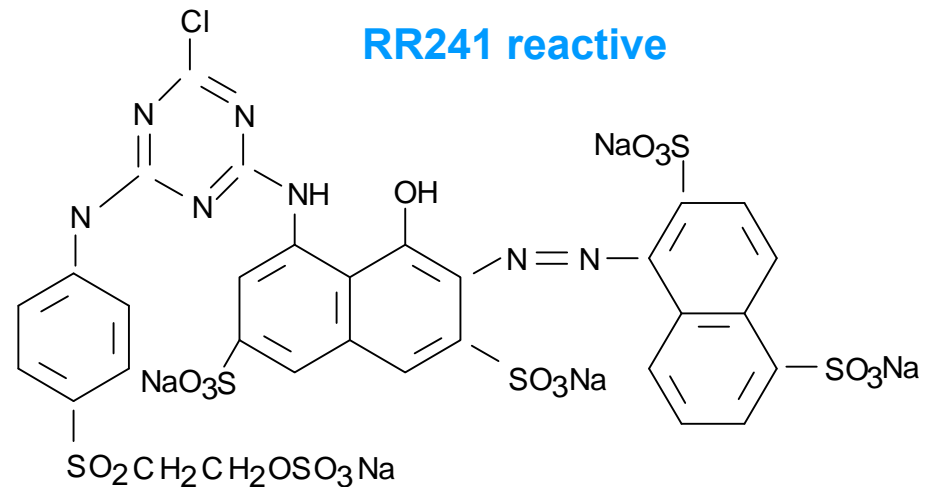
NH₂ - 1424 and 1375 cm⁻¹ → 1441 and 1353 cm⁻¹
COO⁻ 1694 cm⁻¹ → 1716 cm⁻¹

Adsorption properties of polymer coated microparticles

Iron oxide magnetic polymer coated microparticles (PCM) have potential applications for the retention of organic substances (like reactive dyes) from aqueous solutions, because we assume that they exhibit good adsorption properties and can be handled externally to be easily separated from the environment.

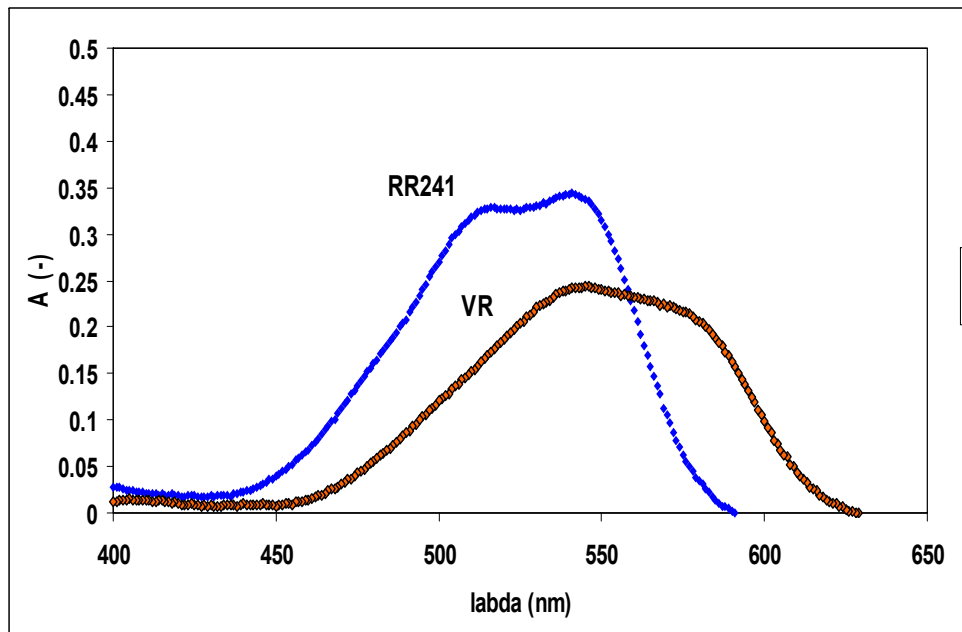


Structure of reactive dyes



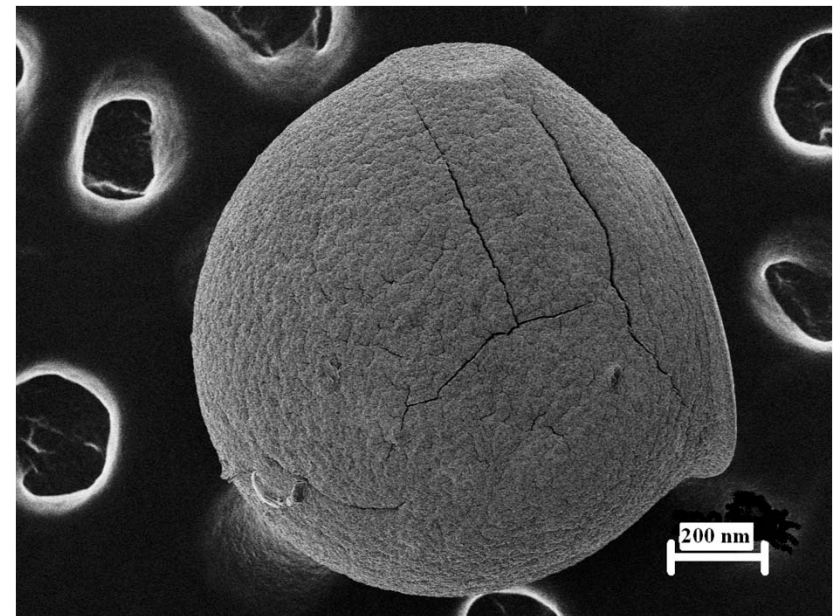
The adsorption of dyes on PCM particles

Characterization of reactive dyes –
UV-Vis spectroscopy

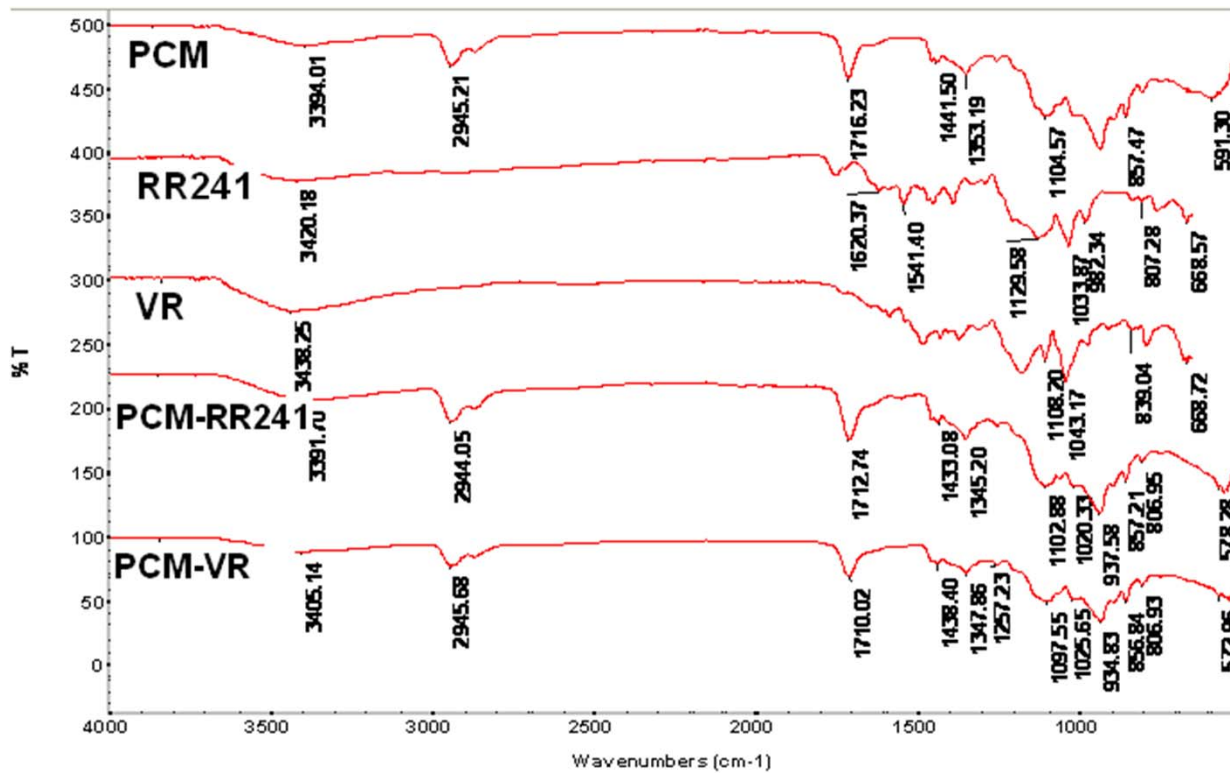


VR $\lambda_{\max} = 543 \text{ nm}$;
RR241 $\lambda_{\max} = 540 \text{ nm}$

PCM particle after the dye
adsorption



Interaction of PCM particles with RR241 and VR dyes



SO_3^- - 1129 and 1033 cm^{-1} → 1102 and 1020 cm^{-1} (RR241)

1108 and 1043 cm^{-1} → 1097 and 1025 cm^{-1} (VR)

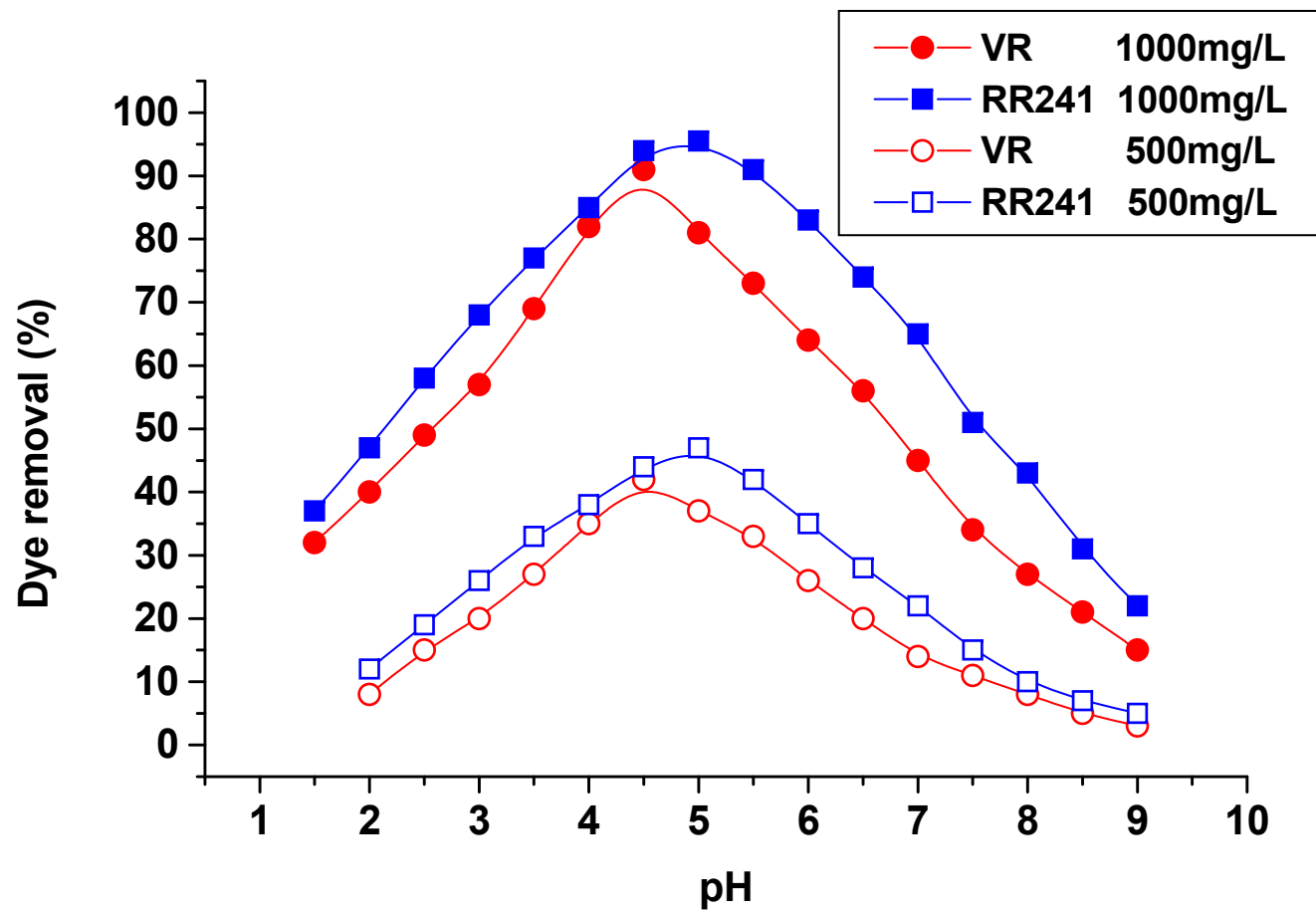
NH_2 - 1441 and 1353 cm^{-1} → 1433 and 1345 cm^{-1} (PCM-RR 241)

→ 1438 and 1347 cm^{-1} (PCM-VR)

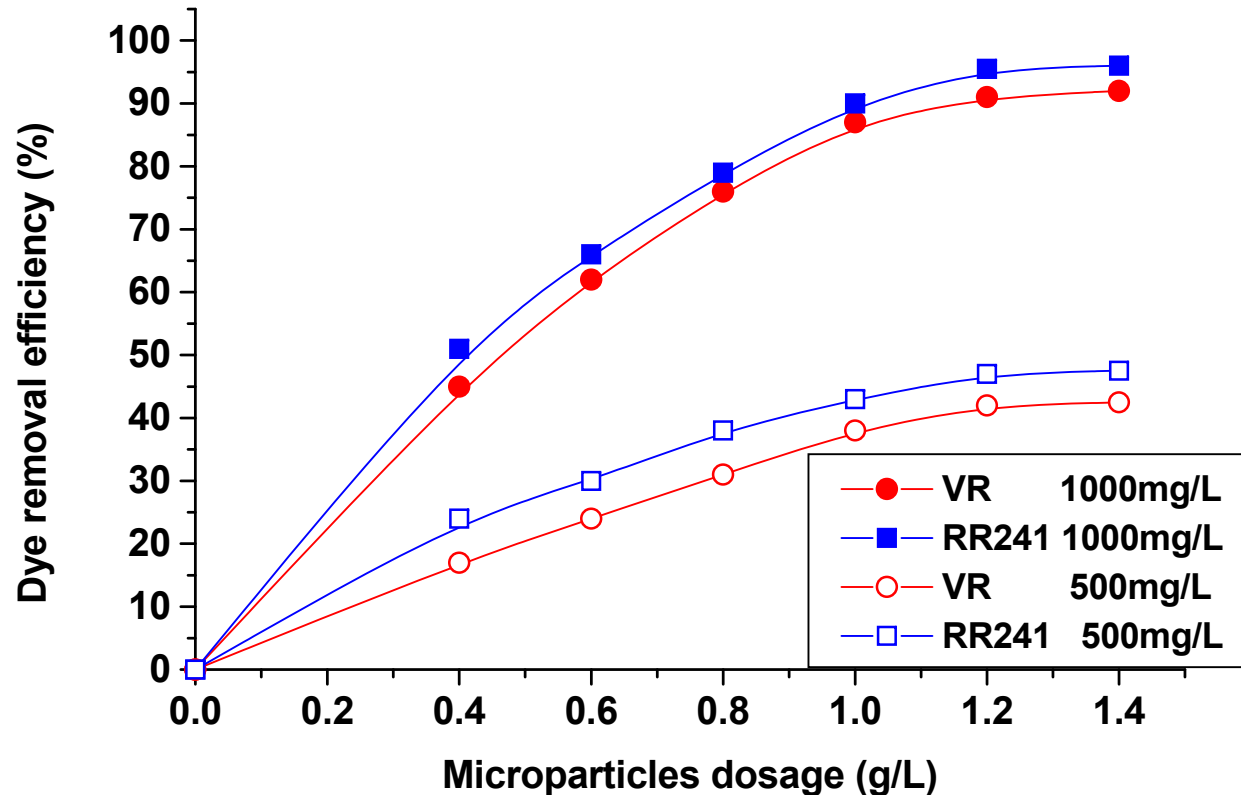
Fe-O-Fe - 591 cm^{-1} → 578 cm^{-1} (PCM-RR 241)

→ 572 cm^{-1} (PCM-VR)

Effect of pH on dyes adsorption

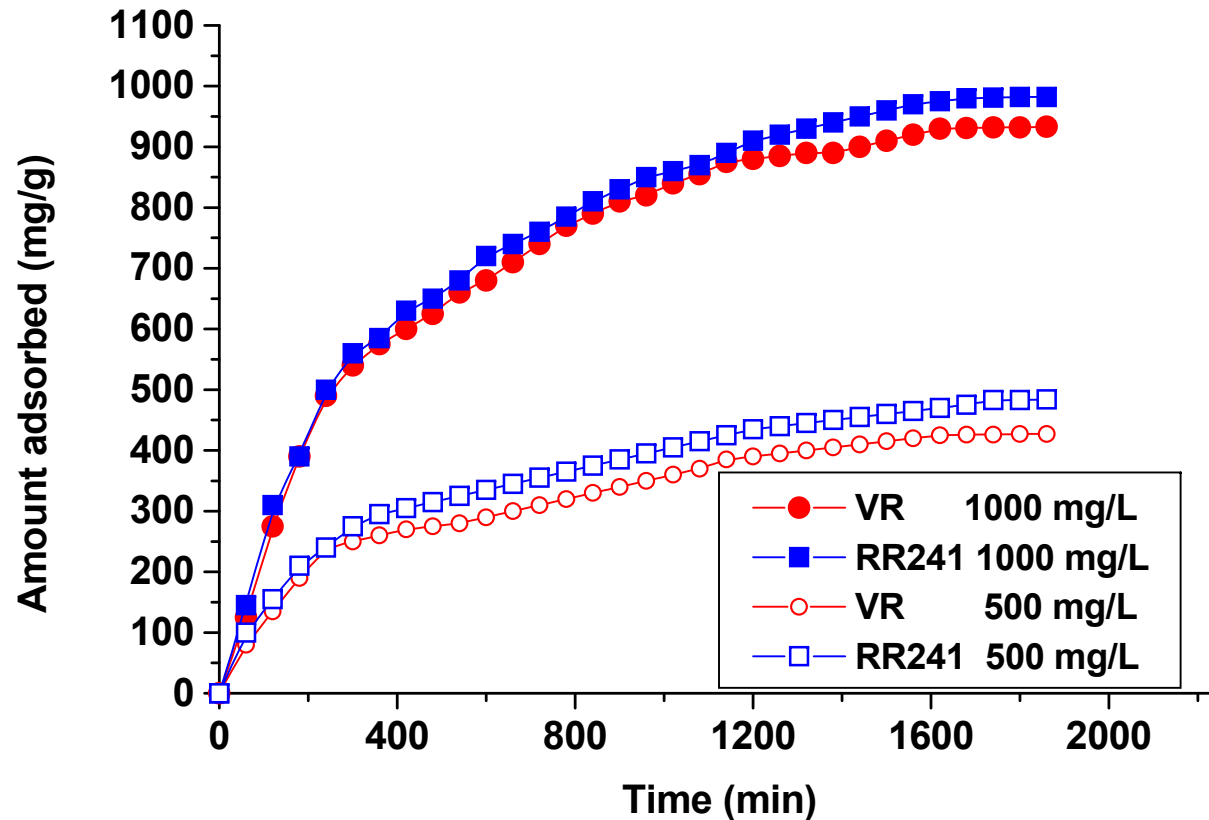


Dyes removal efficiency versus adsorbent dosage



Dye removal efficiency increases with the quantity of used microparticles, and it is dependent of initial concentrations.

Kinetics of dyes adsorption



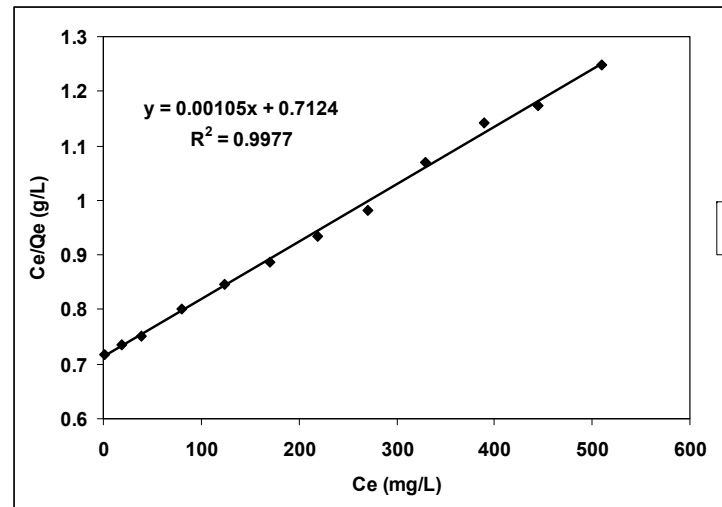
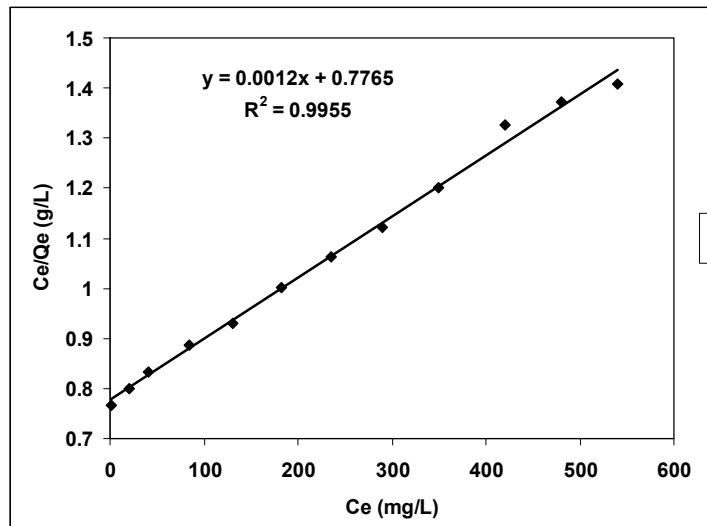
Effect of contact time and initial dye concentration, on the adsorbed amount of dyes at 25⁰ C and PCM dosage of 1.2 g/L

Adsorption model

Langmuir isotherm

$$q_e = \frac{K_L q_m C_e}{1 + K_L C_e} \quad (1)$$

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (2)$$



Isotherm constants and correlation coefficients for reactive dyes adsorbed on PCM

VR			RR241		
Q_m (mg/g)	K_L (L/mg)	R^2	Q_m (mg/g)	K_L (mg/L)	R^2
833.33	1.55	0.9955	952.38	1.47	0.9977

CONCLUSIONS

- ▶ **Iron oxide microparticles were coated with complex layers of polymers from interaction of chitosan with PAA.**
- ▶ **The obtained polymer coated microparticles have dimensions between 700-800 nm, rough surface with many pores and present free, unbound amino groups.**
- ▶ **The PCM magnetic microparticles showed pH-dependent behaviours and superparamagnetic properties.**
- ▶ **The PCM microparticles have been shown to be an efficient solution for reactive dyes adsorption from aqueous solution in a concentrations range of 5-1000 mg/L.**
- ▶ **The FTIR spectra reveal that the dye adsorption occurs through an interaction between positively charged amino groups of coated microparticles and negatively charged sulfate groups of reactive dyes.**
- ▶ **The dyes removal efficiency increases with increasing the dose of PCM microparticles, and with the initial contaminant concentration.**
- ▶ **The experimental data show that the adsorption of reactive dyes on polymer coated microparticles takes place in accordance with the Langmuir model.**

ACKNOWLEDGMENTS

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Va multumesc!