



# COMPARATIVE ADSORPTION STUDIES OF NI (II) IONS ON MAGNETIC-CHITOSAN GRAFTED (ALKYL ACRYLATE) COMPOSITE PARTICLES

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# Introduction/motivation

## *Heavy metals*

- are toxic to human health;
- mainly produced by industrial activities;

## Removal of heavy metals

- chemical precipitation, membrane filtration, coagulation and flocculation, electrochemical methods, **adsorption**;

## Magnetic adsorbents

- magnetic iron oxide particles, magnetic nanocomposite adsorbents with polymeric supports, magnetic composites containing agricultural waste;



# Objectives and Outline

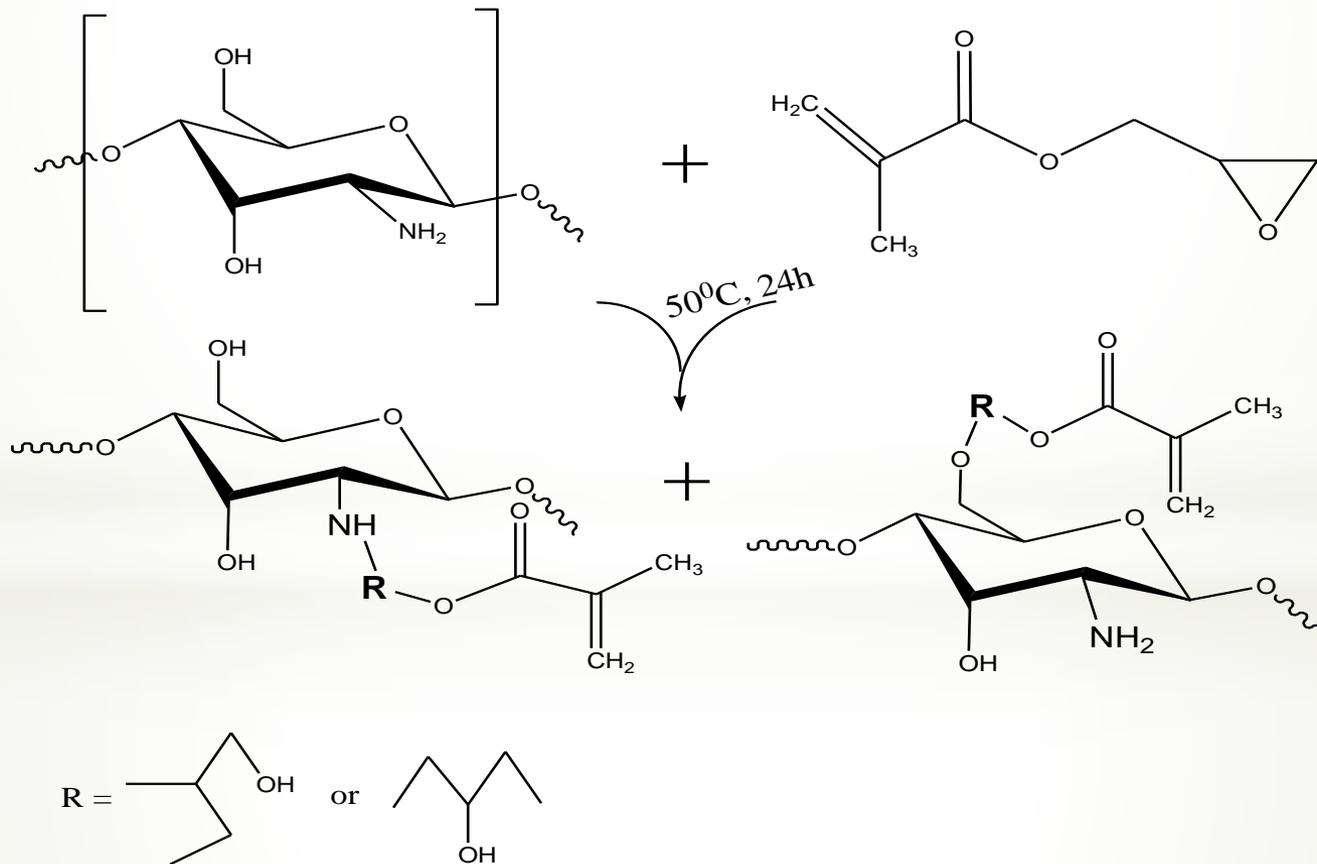
**Main objective:** Prepare magnetic chitosan grafted alkyl acrylate derivatives composite particles as a novel magnetic adsorbent material.

- Composite particles synthesis
- Characterization
- Sorption Experiments
- Desorption and Regeneration Studies
- Conclusions and future work

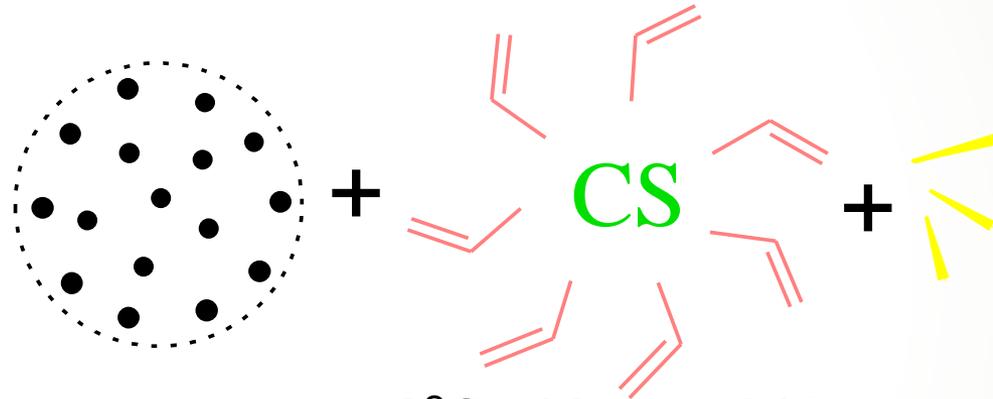
# Magnetite nanoparticles preparation



## Synthesis of chitosan grafted with GMA (CS)



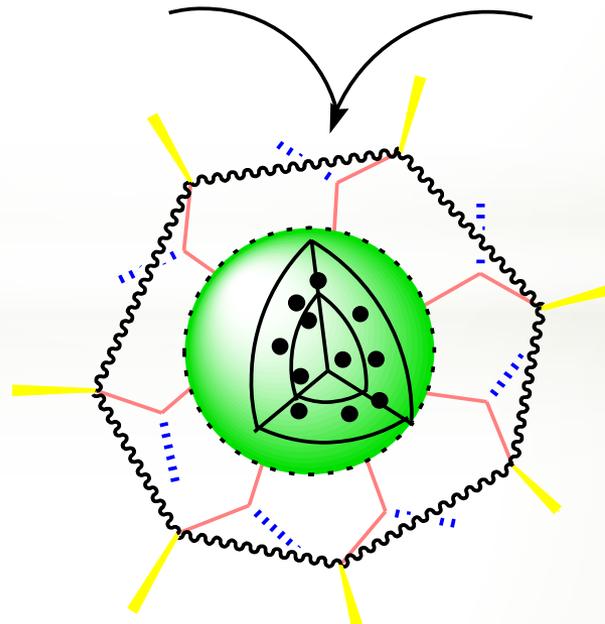
# Composite particles synthesis



$50^\circ\text{C}$ , 500 rpm, 24 h

EGDMA, AZO

-   $\text{Fe}_3\text{O}_4$
-  Cs-g-GMA
-  Monomer (BA, BMA, HA)
-  EGDMA
-  AZO



# TEM

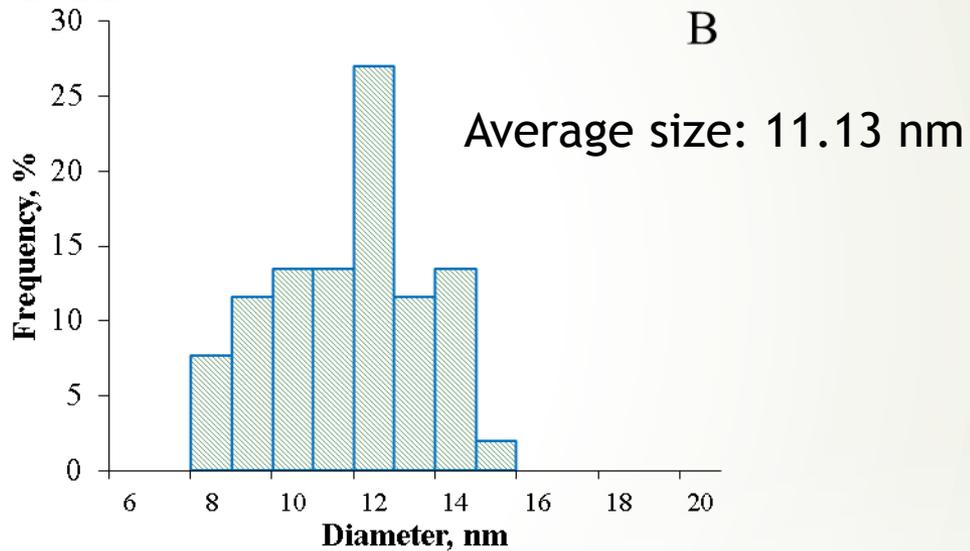
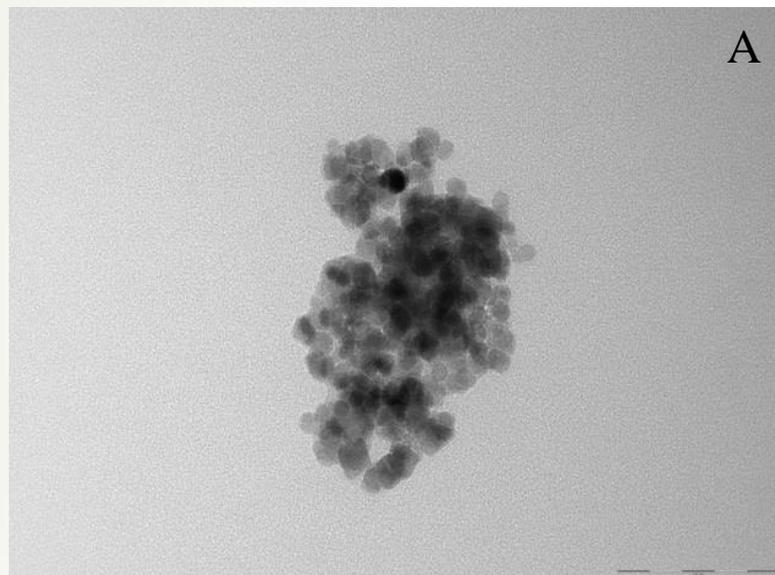


Figure 1. TEM picture (A) and size distribution histogram (B) of Fe<sub>3</sub>O<sub>4</sub>-CS-BMA

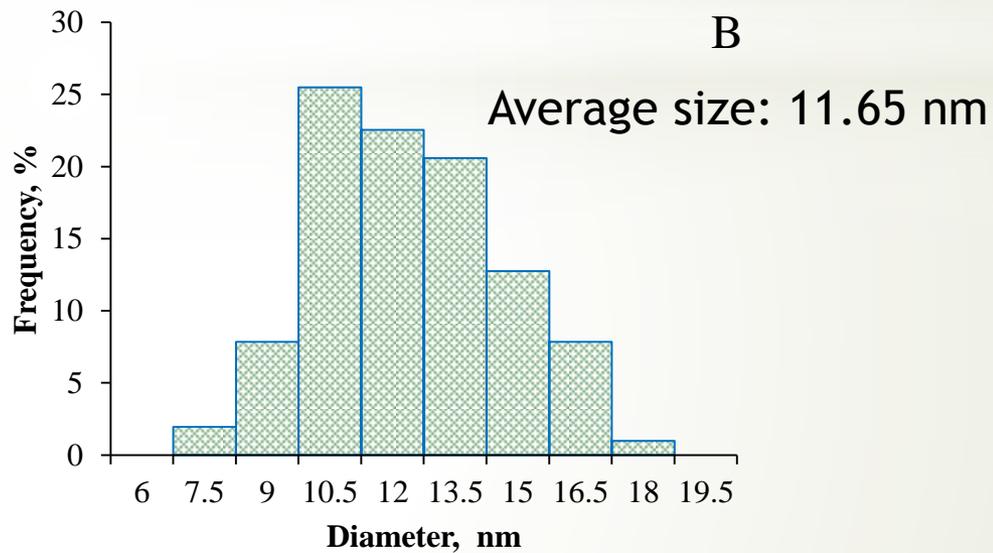
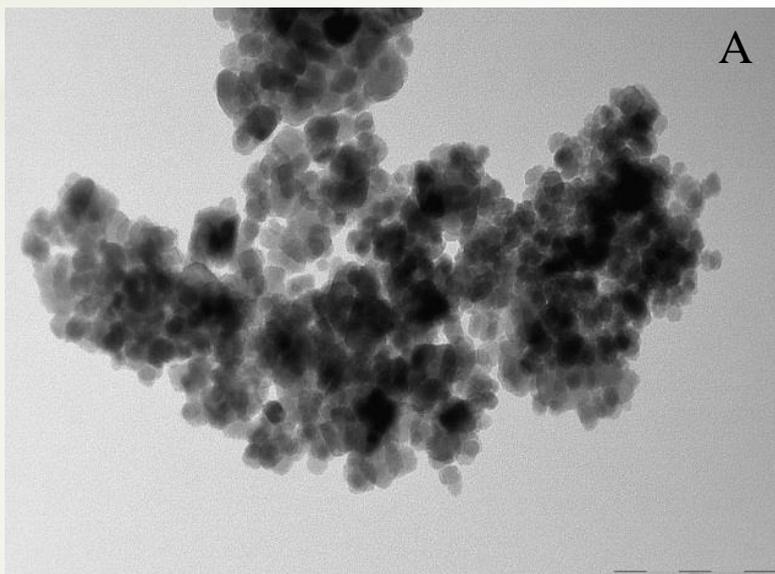


Figure 2. TEM picture (A) and size distribution histogram (B) of Fe<sub>3</sub>O<sub>4</sub>-CS-BA

# TEM

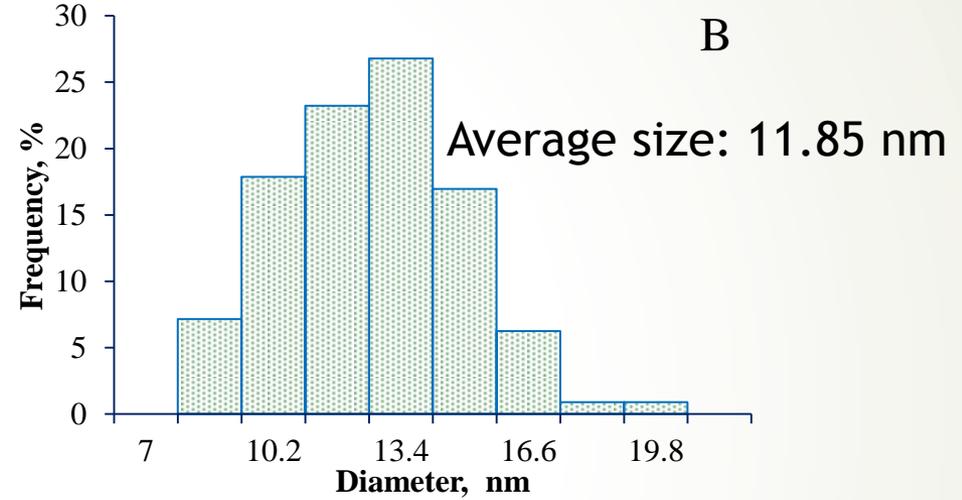
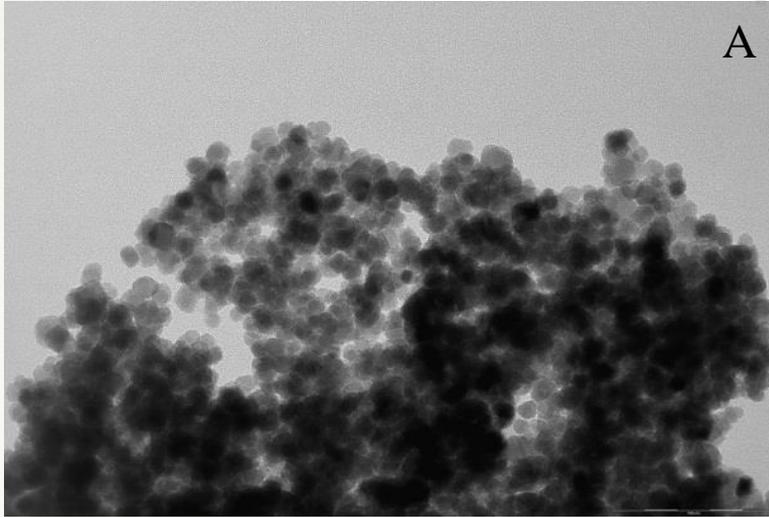
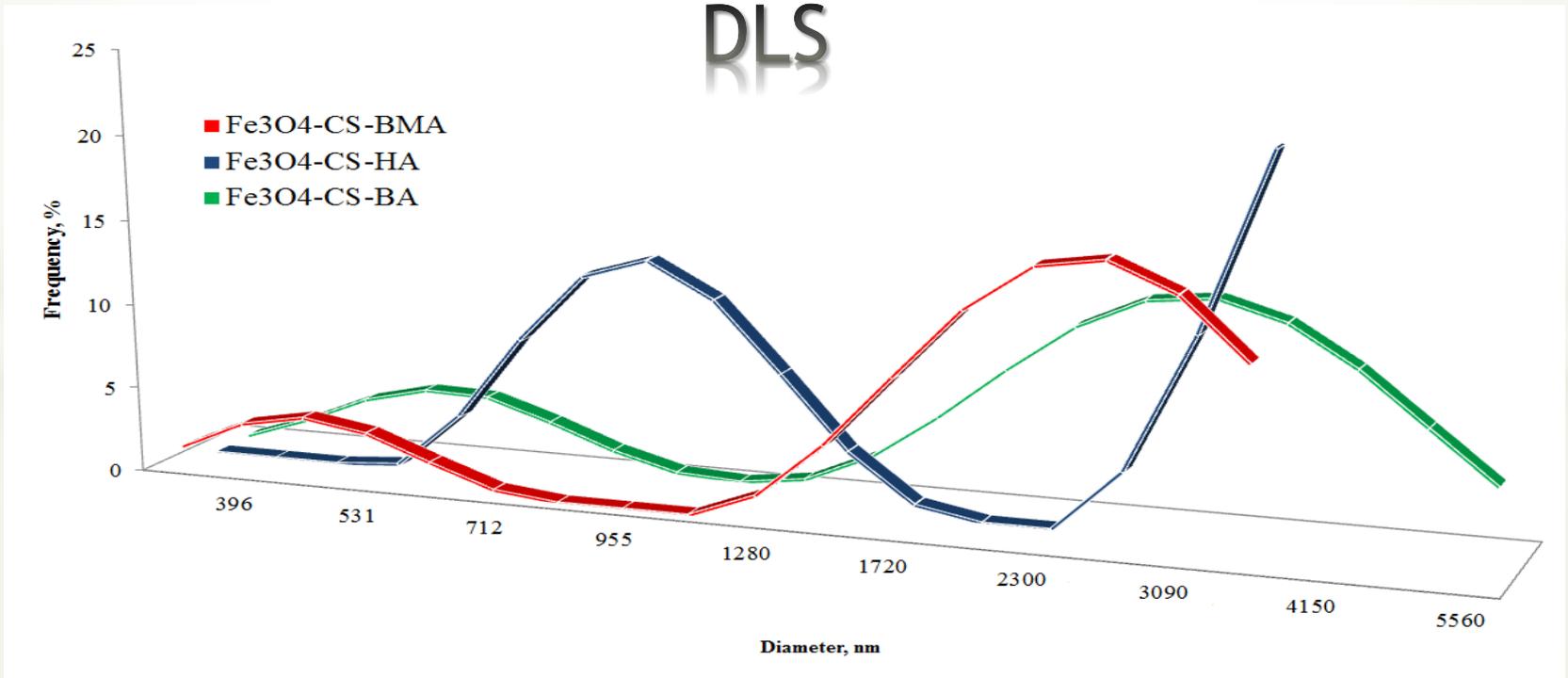
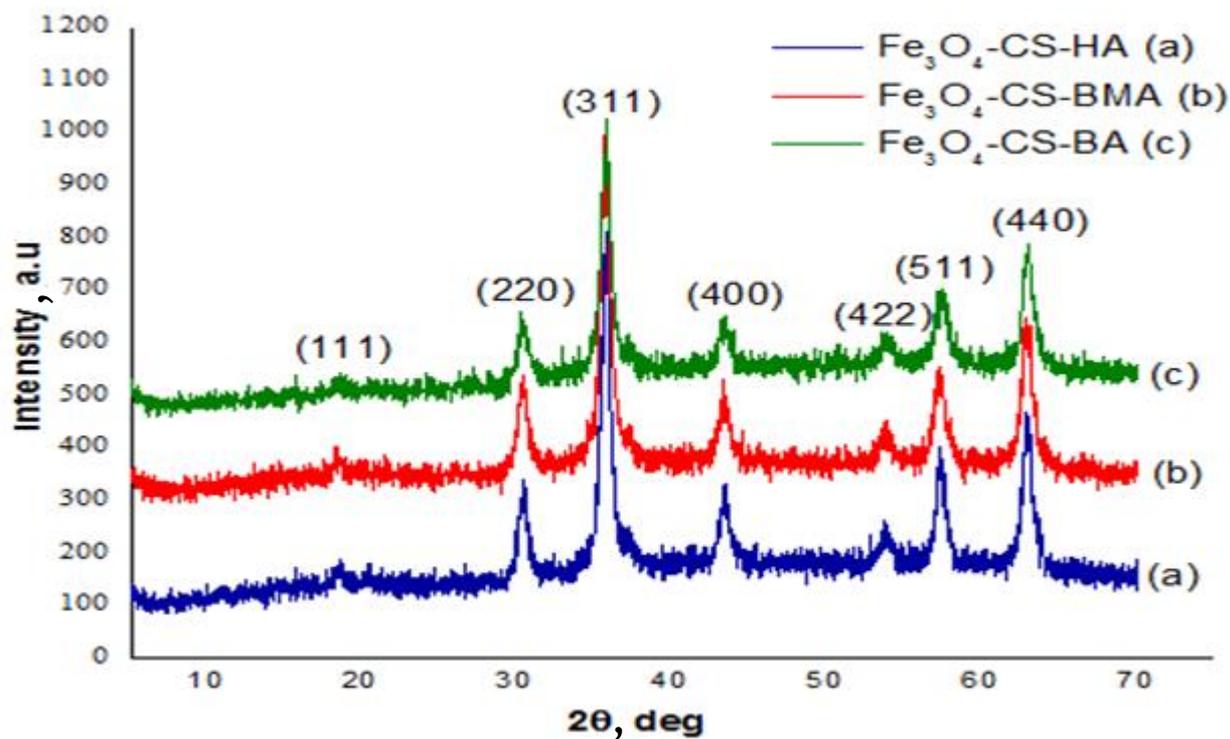


Figure 3. TEM picture (A) and size distribution histogram (B) of Fe<sub>3</sub>O<sub>4</sub>-CS-HA

# DLS

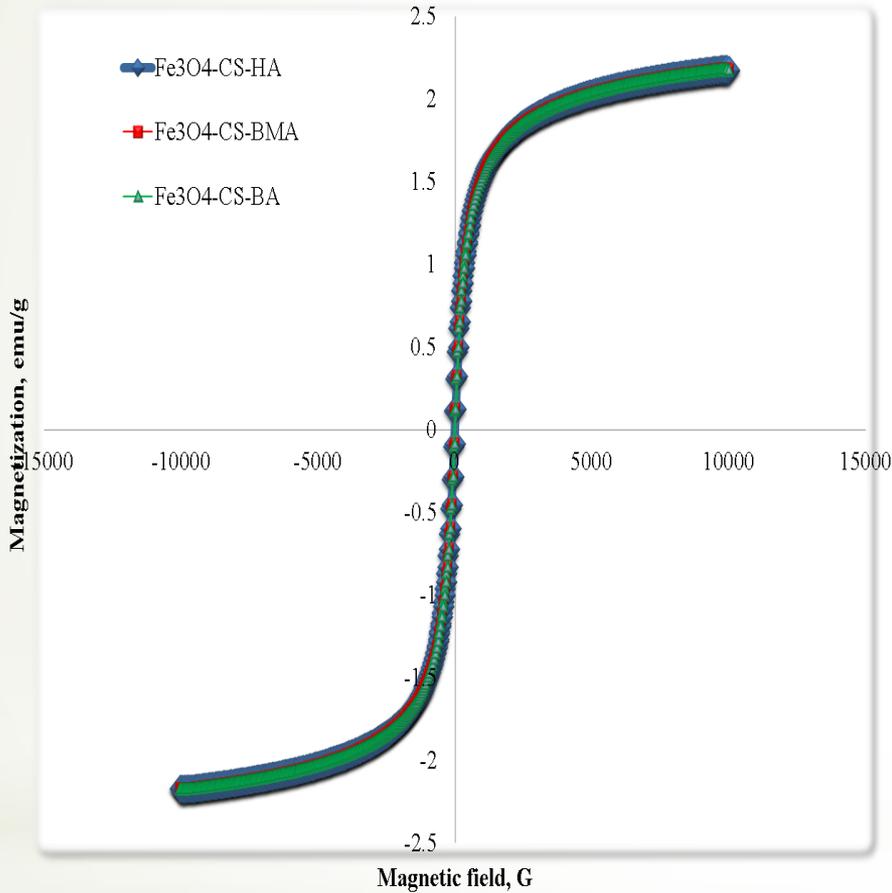


# XRD



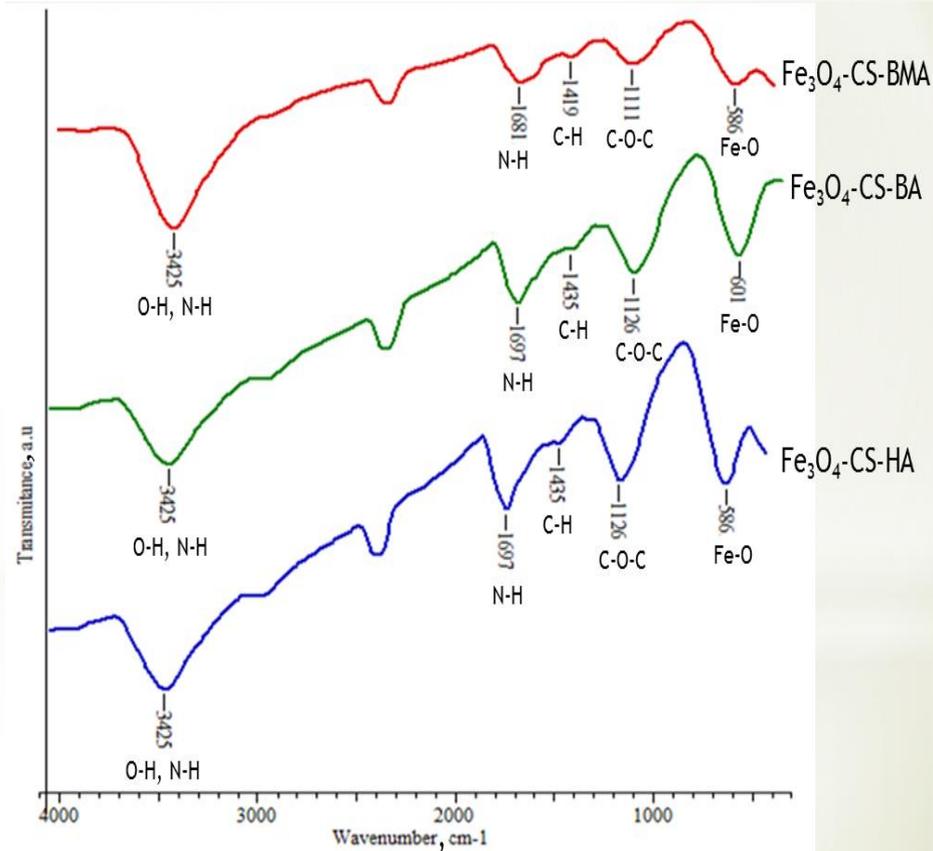
Material	Average size (TEM) (nm)	Size (XRD) (nm)
Fe <sub>3</sub> O <sub>4</sub> -CS-HA	11.84	10.8
Fe <sub>3</sub> O <sub>4</sub> -CS-BMA	9.84	11.3
Fe <sub>3</sub> O <sub>4</sub> -CS-BA	11.65	13.42

# Magnetization



Particles show remanent magnetization.

# FTIR



The chitosan and acrylates peaks are overlapped therefore, an exact estimation of chemical structure was not possible.

# Adsorption Experiments

We varied the following parameters:

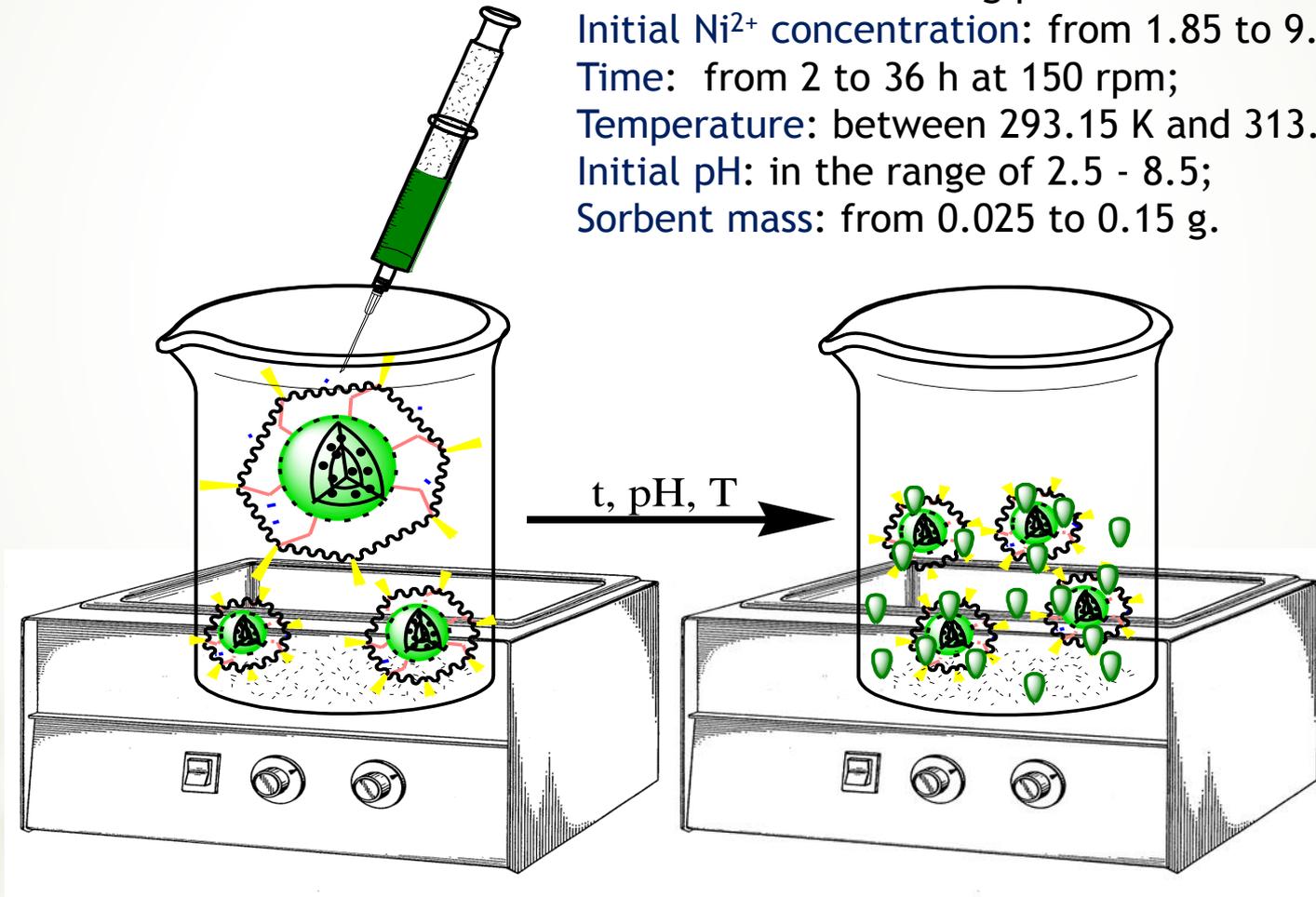
Initial  $\text{Ni}^{2+}$  concentration: from 1.85 to 9.6 mg/mL;

Time: from 2 to 36 h at 150 rpm;

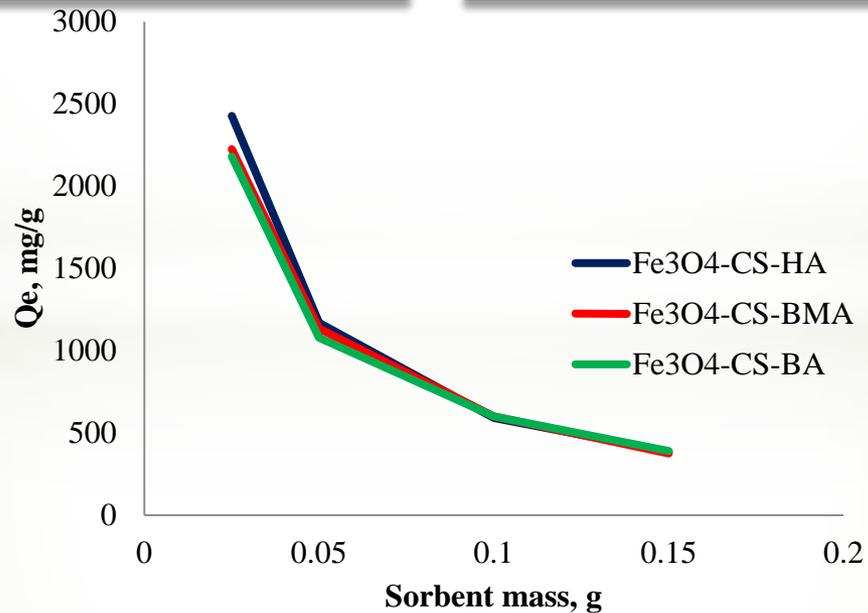
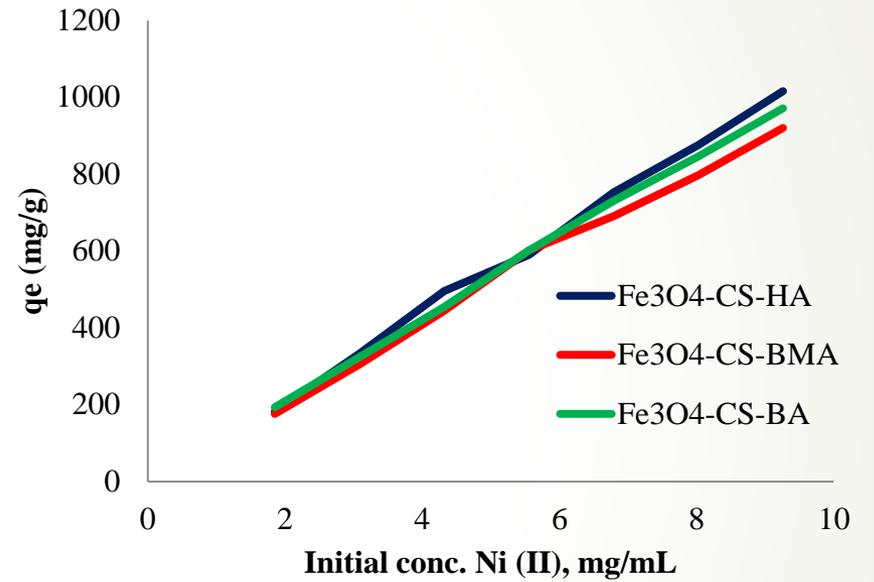
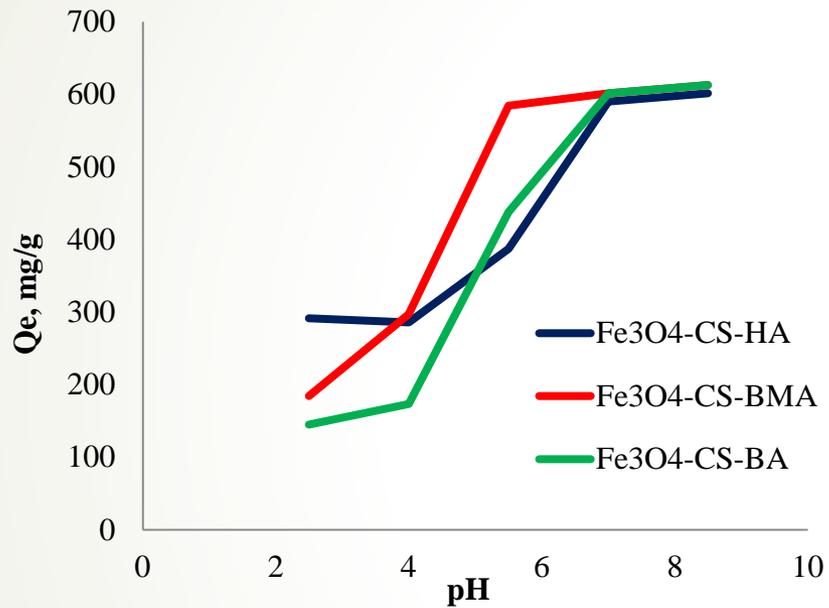
Temperature: between 293.15 K and 313.15 K;

Initial pH: in the range of 2.5 - 8.5;

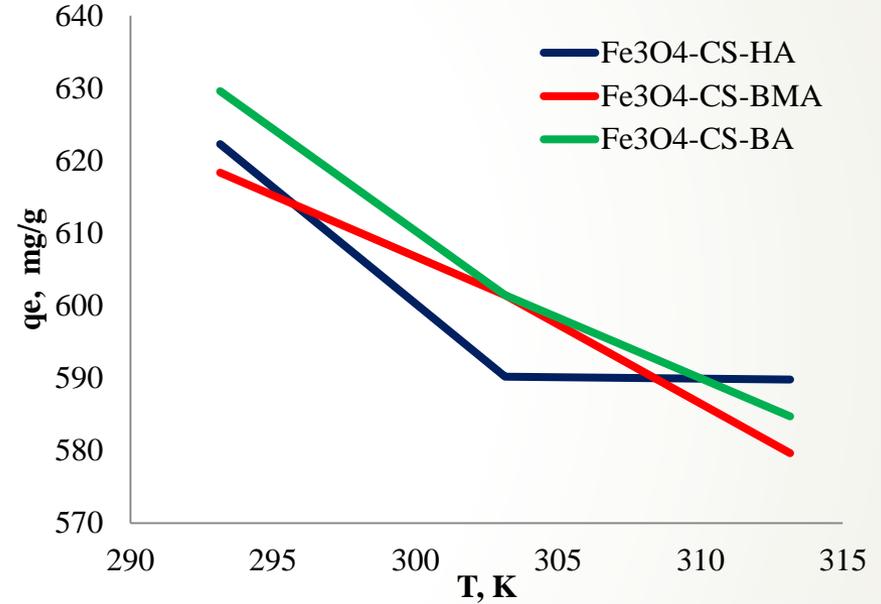
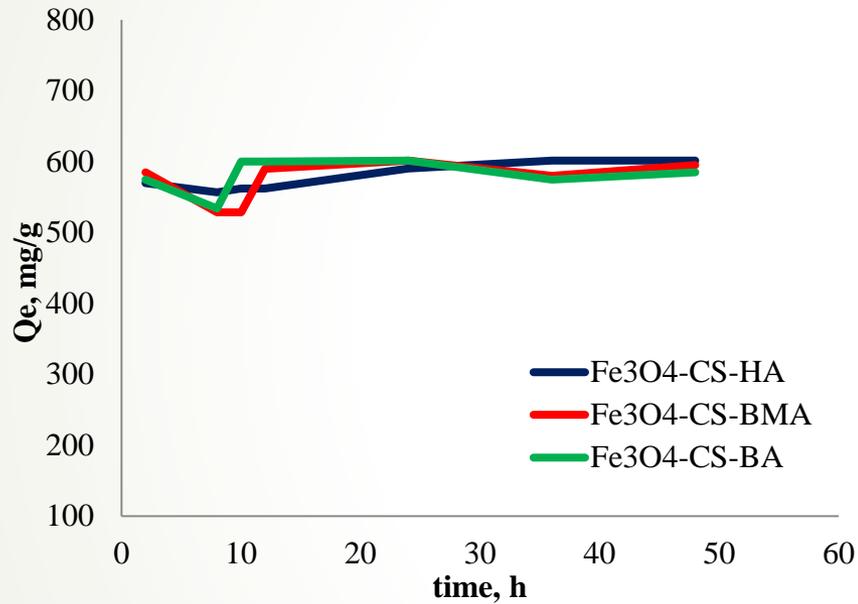
Sorbent mass: from 0.025 to 0.15 g.



# Sorption Experiments



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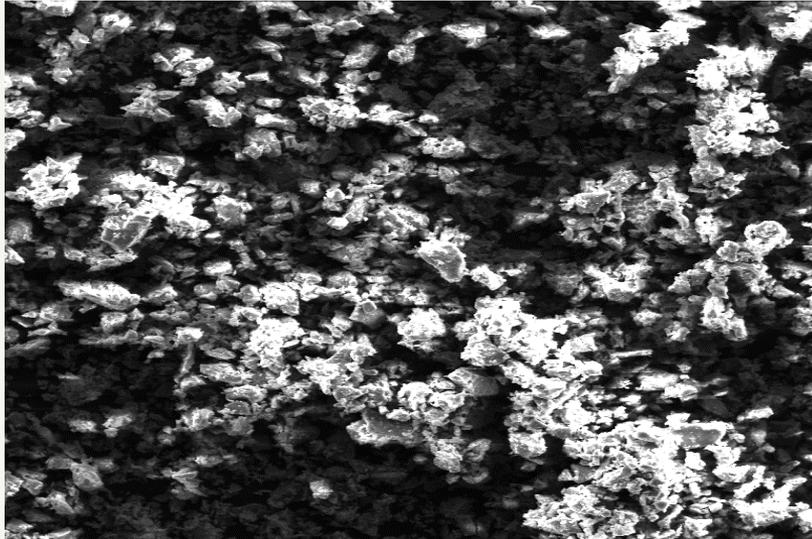
# Desorption and Regeneration Studies

Material	Desorption in 0.1 M HCl solution (%)
Fe <sub>3</sub> O <sub>4</sub> -CS-HA	40.5 - 97.5
Fe <sub>3</sub> O <sub>4</sub> -CS-BMA	34.8 - 73.81
Fe <sub>3</sub> O <sub>4</sub> -CS-BA	38.77 - 100

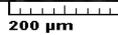
# Adsorption Isotherms

Material/Metal ion		HA/Ni <sup>2+</sup>	BMA/Ni <sup>2+</sup>	BA/Ni <sup>2+</sup>
<i>Langmuir constants</i>	R <sup>2</sup>	0.88	0.89	0.95
$\frac{C_e}{q_e} = \frac{1}{K_L \times q_m} + \frac{C_e}{q_m}$	q <sub>m</sub> (mg/g)	5000	5000	5000
	K <sub>L</sub> (mL/mg)	0.06	0.05	0.05
	R <sub>L</sub>	0.75	0.8	0.77
<i>Freundlich constants</i>	R <sup>2</sup>	0.98	0.98	0.99
$\log q_e = \log K_F + \frac{1}{n} \log C_e$	K <sub>F</sub>	215	194	220.7
	n	0.9	0.95	0.98
<i>Dubinin-Radushkevich constants</i>				
	R <sup>2</sup>	0.96	0.91	0.91
$\ln q_e = \ln X_m - K_{DR} \varepsilon^2$	X <sub>m</sub> (mg/g)	998.8	919.1	887.7
	K <sub>DR</sub> (mol <sup>2</sup> /kJ <sup>2</sup> )	0.56	0.58	0.48
	E <sub>S</sub>	0.94	0.93	1.02

# SEM

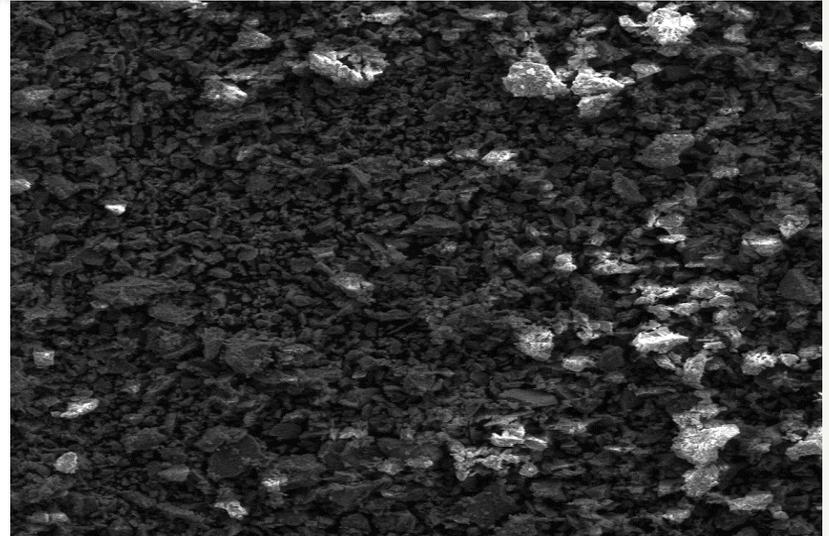


SEM HV: 30.00 kV WD: 14.25 mm  
View field: 1.44 mm Det: SE  
Date(m/d/y): 04/30/15 nicanorb

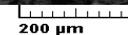


VEGA\\ TESCAN  
UTI-SIM

$\text{Fe}_3\text{O}_4\text{-CS-BMA}$

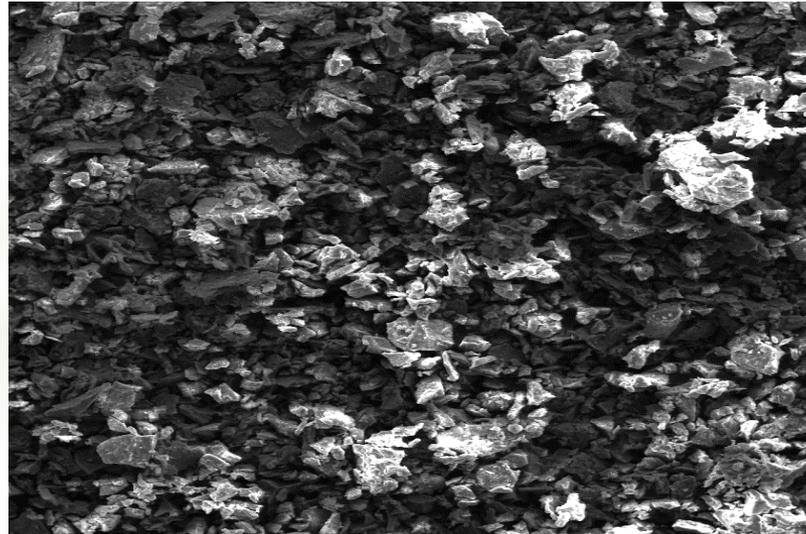


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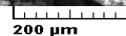


VEGA\\ TESCAN  
UTI-SIM

$\text{Fe}_3\text{O}_4\text{-CS-BA}$



SEM HV: 30.00 kV WD: 14.32 mm  
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Date(m/d/y): 04/30/15 nicanorb



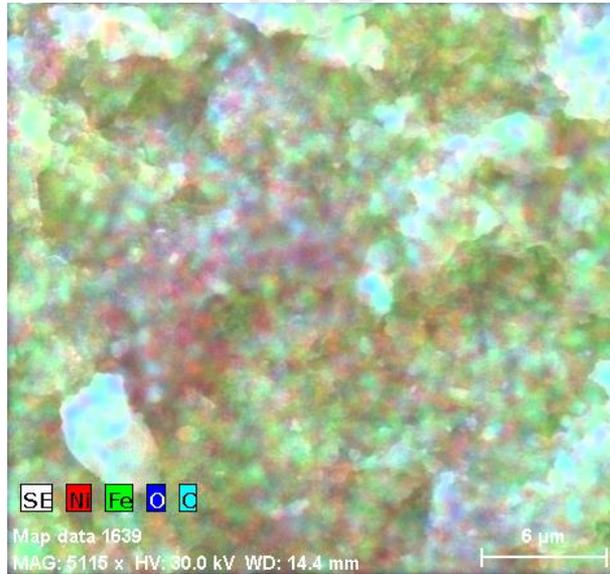
VEGA\\ TESCAN  
UTI-SIM

$\text{Fe}_3\text{O}_4\text{-CS-HA}$

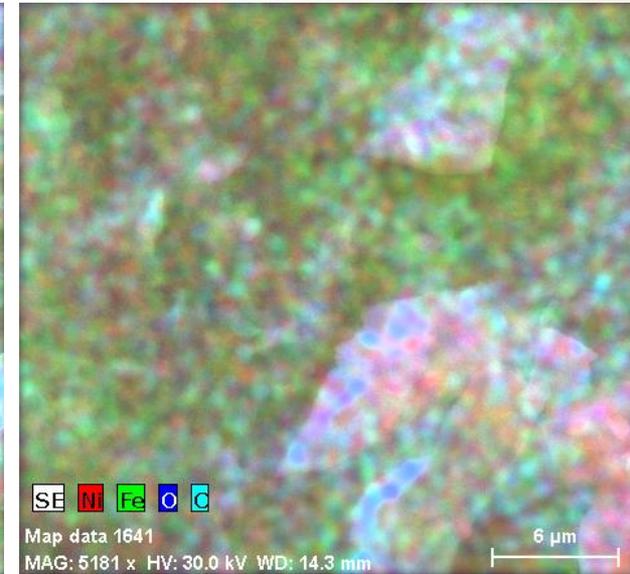
# EDX



$\text{Fe}_3\text{O}_4\text{-CS-BMA}$



$\text{Fe}_3\text{O}_4\text{-CS-BA}$



$\text{Fe}_3\text{O}_4\text{-CS-HA}$

Element	$\text{Fe}_3\text{O}_4\text{-CS-BMA}$		$\text{Fe}_3\text{O}_4\text{-CS-BA}$		$\text{Fe}_3\text{O}_4\text{-CS-HA}$	
	Atomic %	Wt. %	Atomic %	Wt. %	Atomic %	Wt. %
Oxygen	59.35	35.32	61.15	39.06	60.02	36.45
Iron	13.81	28.69	14.66	32.69	12.24	25.95
Carbon	13.02	5.81	15.24	7.31	13.65	6.22
Nickel	13.81	30.16	8.93	20.94	14.08	31.37

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**Future work:** Competitive adsorption of heavy metal ions.

# Acknowledgements

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**THANK YOU FOR YOUR ATTENTION!**