Preparation and Characterization of Magnetic Iron-Cobalt/Silica Nanoadsorbent and its Application for Preconcentration of Arsenic (III) in Water

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Introduction

In South Africa, arsenic based compounds like herbicides, fertilizers, wood preservatives, animal feed additives, paint pigments and metal alloys are the sources of arsenic that is found in ground and surface environmental water.[1] Exposure to high levels of arsenic (> 50µg/L) causes skin disorder, cancer of kidneys, gangrene and it is lethal.[2] A novel, low cost $CoFe_2O_4/SiO_2$ (cobalt-iron/silica) nanoadsorbent with selectivity towards arsenic was prepared. The nanoadsorbent was used to preconcentrate trace arsenic (III) from different water sources prior to inductively coupled plasma optical emission spectroscopic analysis.

Materials and Methods

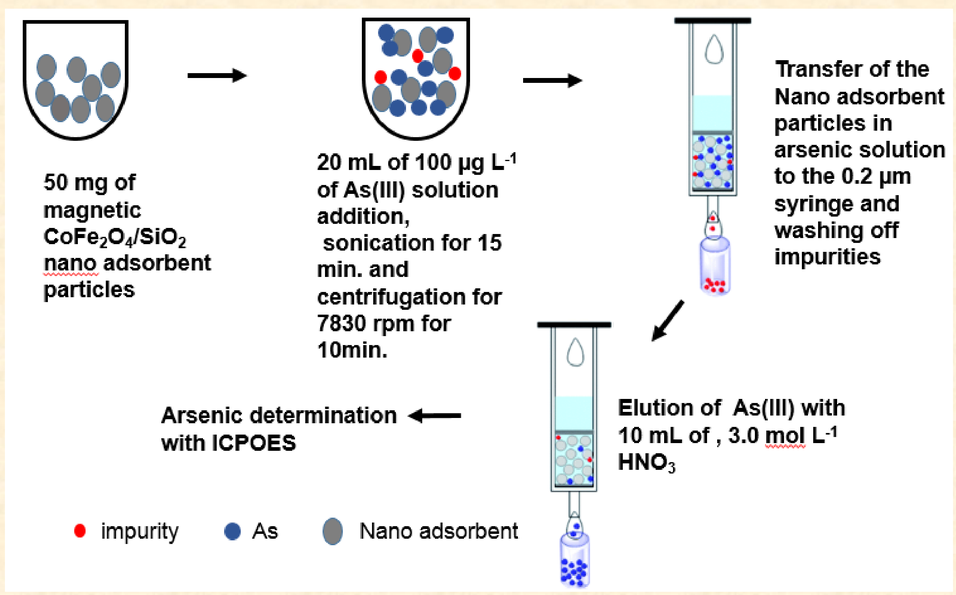

Fig.1 The particles synthesis of $CoFe_2O_4$/ SiO_2 nanoadsorbent

Results

Characterization

- **SEM-EDS :**confirmed the presence and distribution of Si, Co, Fe and O elements in the $CoFe_2O_4$/ SiO_2 nanocomposite. The percentage compositions for Si, Co, Fe and O were 2.0%, 38.6%, 8.41% and 51%, respectively.

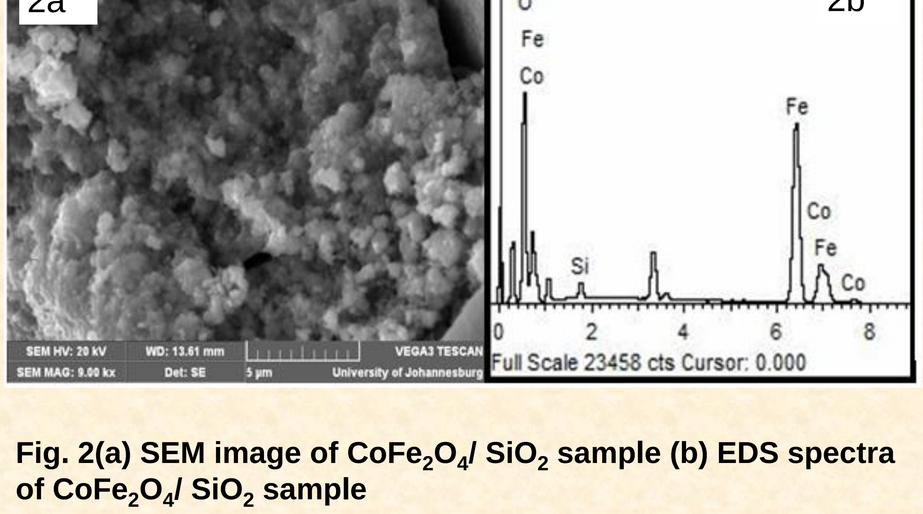

Fig. 2(a) SEM image of $CoFe_2O_4$/ SiO_2 sample (b) EDS spectra of $CoFe_2O_4$/ SiO_2 sample

- **XRD:** indicated the crystalline nature of the nanocomposite material. The observed peaks from 18 θ to 35 θ shows the crystalline form of SiO_2 formation around the $CoFe_2O_4$ cores and the peak originating from 39.1 θ with decreased intensity is due to $CoFe_2O_4$ particles (**see figure 3**).

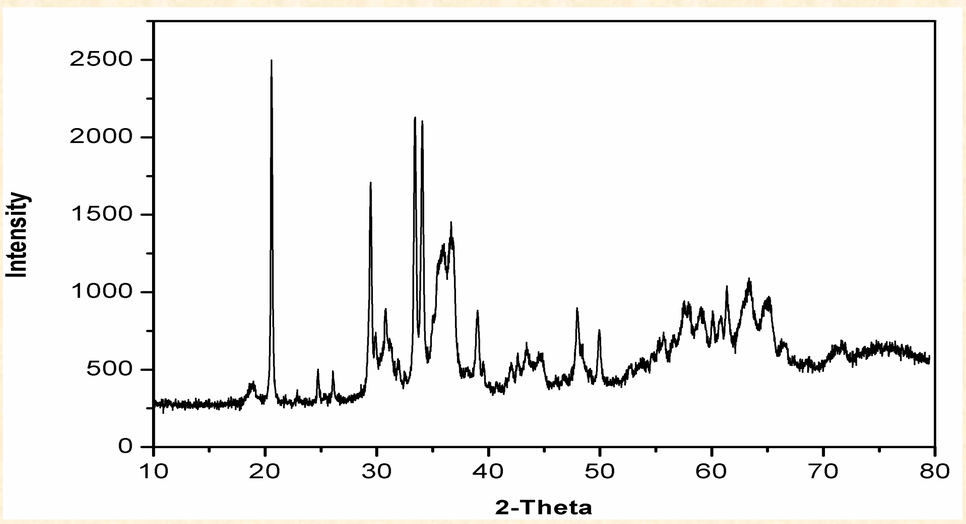

Fig.3 :XRD pattern of magnetic $CoFe_2O_4/SiO_2$

Influential Parameters on As(III) Adsorption on the nanoadsorbent

❖ **Effect of pH**

At pH 3.0 – 3.5 quantitative (higher) recovery for As(III) was achieved. Hence, pH 3 was selected as an optimum pH for solid phase extraction of the As(III) for subsequent experiments.

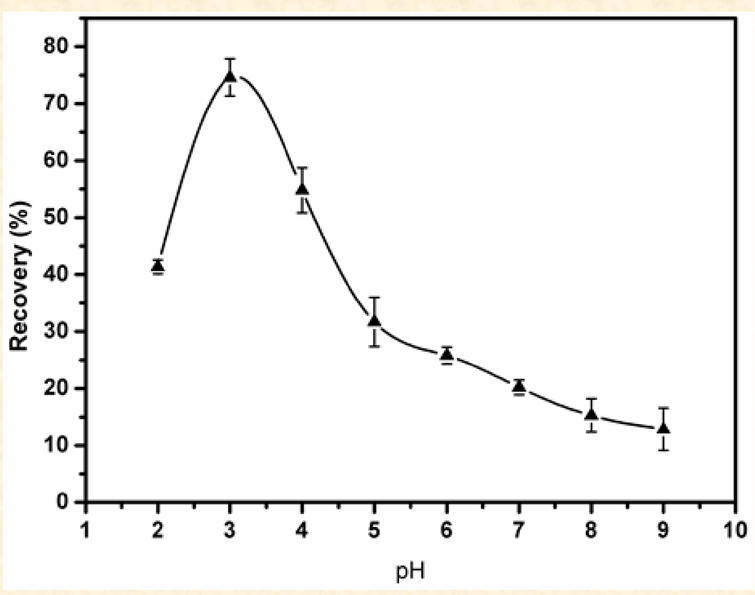

Fig. 4. Effect of pH on preconcentration of arsenic (III): Experimental conditions: analyte concetration -100 µg.L^{-1}, adsorbent mass - 50 mg, sample volume - 20 mL, eluent volume - 10 mL; eluent concentration - 3 mol. L^{-1} HNO_3

❖ **Effect of mass of the nanoadsorbent**

Higher recoveries (up to 75.5%) were obtained when $CoFe_2O_4$/ SiO_2 amount was 100 mg . Accordingly, 100 mg of $CoFe_2O_4$/ SiO_2 was used in all subsequent experiments.

Table 1:Effect of nanoadsorbent mass

Mass (mg)	% Recovery
10	20.9± 5.2
30	35.7± 2.4
50	48.0± 1.3
70	53.9± 2.7
100	75.5± 2.0

❖ **Effect of type and eluent concentration**

5 mol L^{-1} HCl solution gave the maximum recovery of the analyte hence it was found to be an optimal eluent solution for desorption of As(III) from the magnetic nano-sorbent.

Table 2: Effect of type and eluent concentration

Concentration	HCl (%Recovery)	HNO_3 (%Recovery)
1	6.0	11.4
2	42.9	23.6
3	55.9	37.8
4	61.1	55.9
5	100	68.2

❖ **Capacity and Reusability of the Nanoadsorbent**

The capacity of the nanoadsorbent was 8.5 mg g^{-1}
and it can be used 15 times without affecting As (III) recoveries.

❖ **Method Validation (ICPOES):**

- **LOD and LOQ** values were 0.126 and 0.420 µg L^{-1}
- **RSD** ranged from 0.339-1.08% for 4 different concentrations with a replicate of 3 measurements.
- Certified Reference material containing 250 mg L^{-1} of As(III) (CWWTM-D, water) yield ≥ 90% of As(III).

Conclusion

- $CoFe_2O_4/SiO_2$ were successfully applied in preconcentrating and adsorbing trace amounts of As(III).
- Optimized parameters like pH, mass of the nanoadsorbent, eluent type and concentration are crucial for As (III) adsorption from water.

References

1. Farrel J.W (2009) Nanomagnetite Enhances Sand Filtration For Removal of Arsenic From Drinking Water, Houston, Texas (MSc Thesis)

2. Ramudzuli M.R, Horn A.C (2014). Arsenic residues in soil at cattle dip tanks in the Vhembe district, Limpopo Province, South Africa, South African Journal of Science. 110:64-70

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