KWAME NKRUMAH UNIVERSITY OF SCIENCE AND TECHNOLOGY, KUMASI,GHANA
UNIVERSITY OF ENERGY AND NATURAL RESOURCES, SUNYANI,GHANA

IRON SULPHIDE FUNCTIONALIZED POLYANILINE NANOCOMPOSITE FOR THE REMOVAL OF EOSIN Y FROM WATER: EQUILIBRIUM AND KINETIC STUDIES

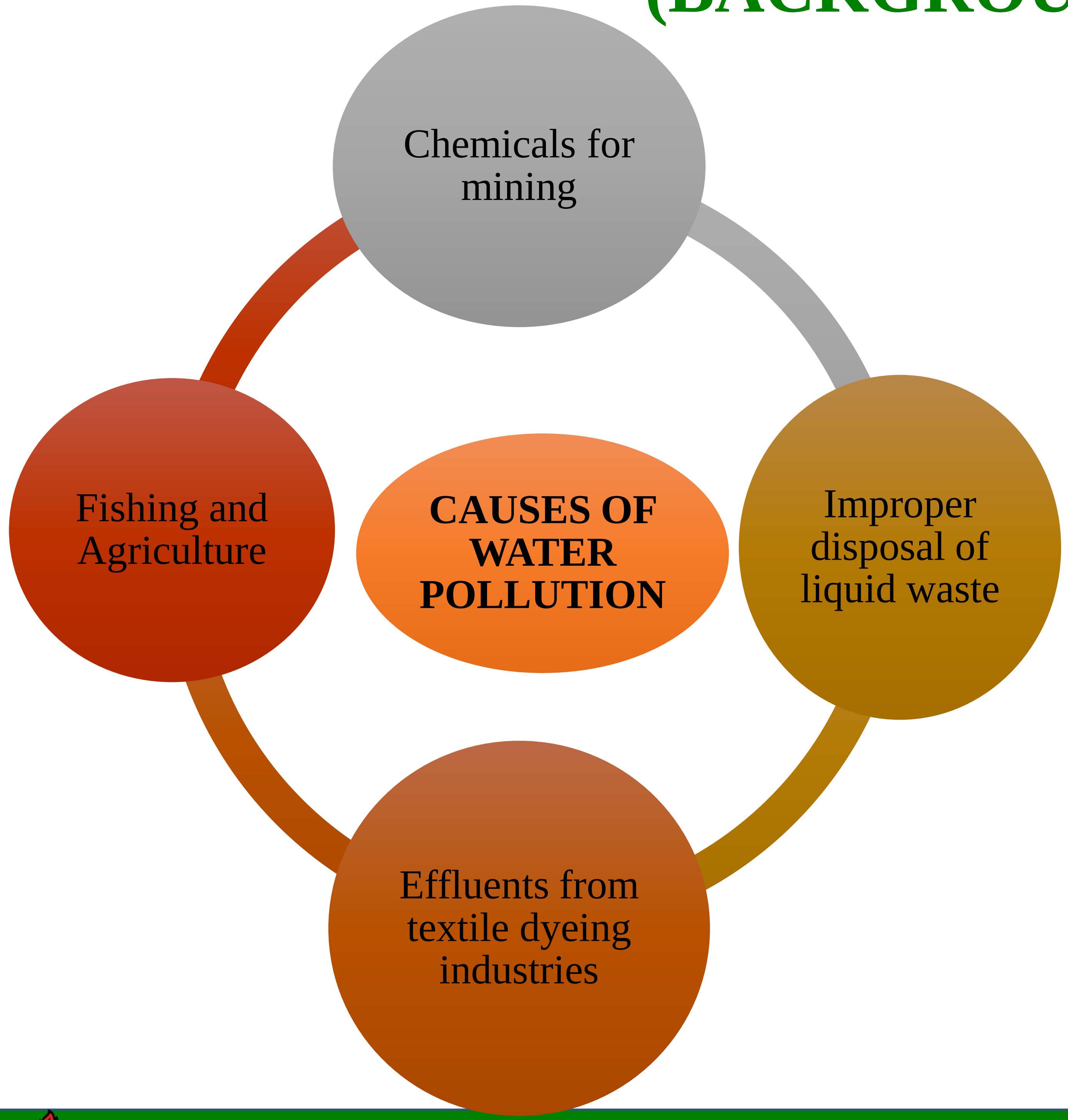
By

Bernice Yram Danu, Ph.D

OUTLINE

- ❑ **INTRODUCTION**
  - ❖ Background and Motivation
  - ❖ Problem Statement
  - ❖ Justification
  - ❖ Aims and Objectives
  - ❖ Novelty Statement
- ❑ **METHODOLOGY**
- ❑ **RESULTS AND DISCUSSION**
- ❑ **CONCLUSION AND RECOMMENDATION**
- ❑ **REFRENCES**
- ❑ **ACKNOWLEDGEMENT**

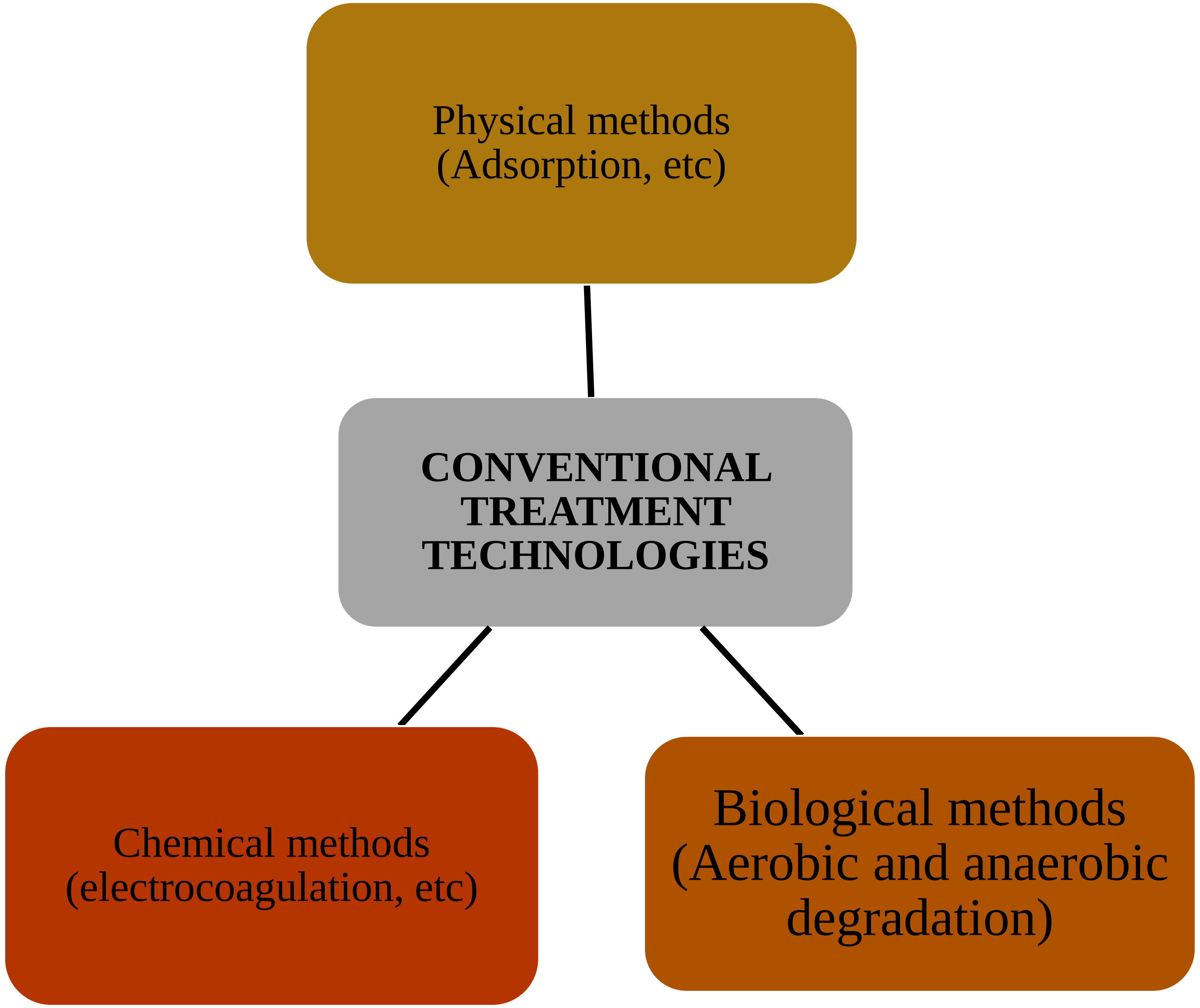
INTRODUCTION:
WATER POLLUTION AS AN ENVIRONMENTAL HAZARD (BACKGROUND AND MOTIVATION)



- Among these various causes of water pollution, textile industries form one of the major contributors of pollutant release into clean water, after agriculture and mining

- Constituents of these effluents , collectively make it highly toxic, carcinogenic and damaging to fauna, flora and humankind.

PROBLEM STATEMENT



SETBACKS

- Sludge generation
- High maintenance cost
- Toxicity of dyes to microorganism
- Clogging of surface membranes, frequent formation of scales, etc.

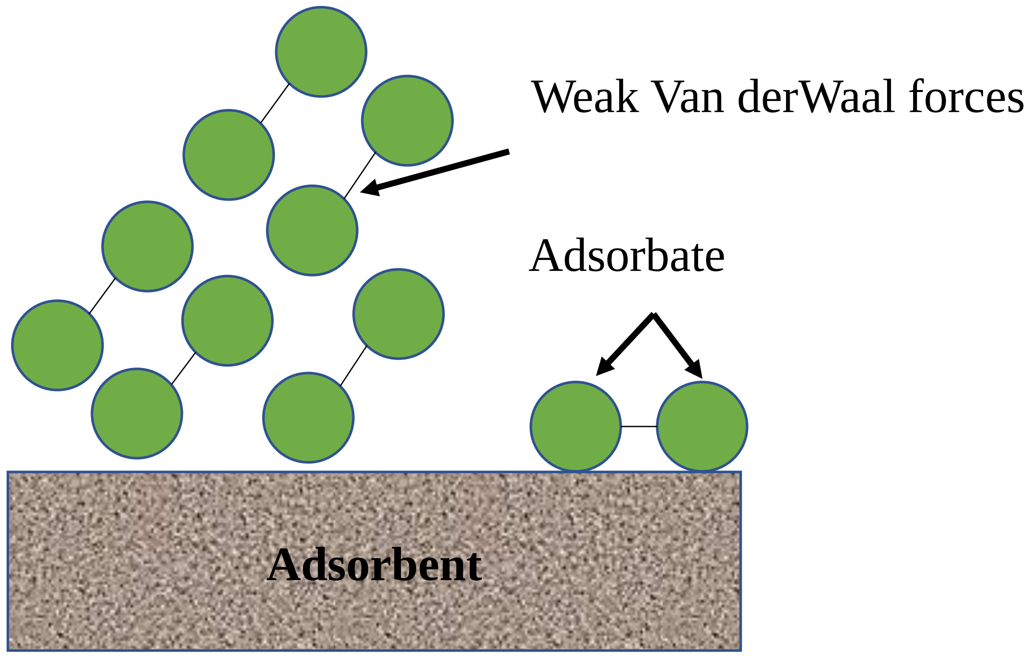
ADSORPTION

- flexible and simple in design
- low cost and ease of operation
- does not result in the formation of harmful substances.

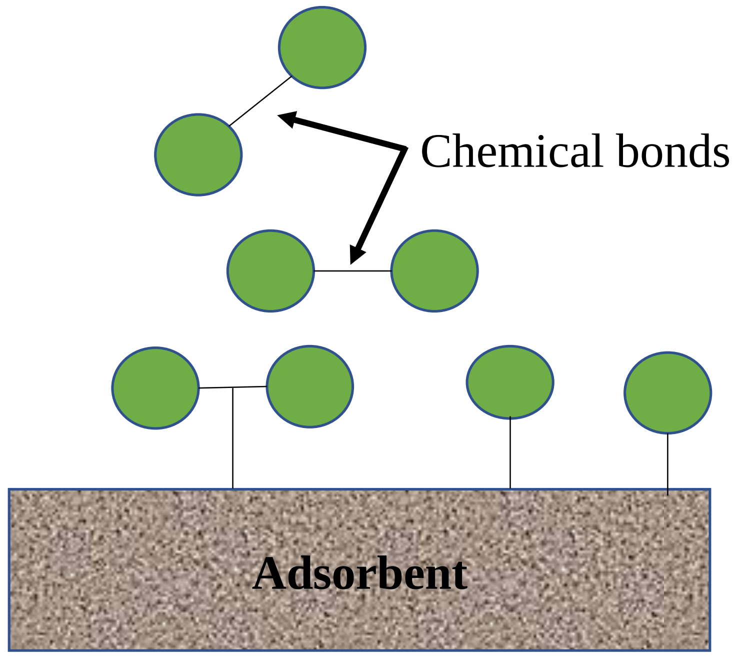
PROBLEM STATEMENT CONT'D

- **Adsorption**: adhesion of molecules, ions or atoms from, liquids and gas onto a surface.

- **Adsorbent types**
  - Agricultural byproducts
  - Nanoparticles
  - Polymeric adsorbents
  - Composite



Physical adsorption



Chemical adsorption

JUSTIFICATION

- Nanoparticles: nanometer range(1 nm-100 nm)
- Unique properties due to high surface to volume ratio: high chemical reactivity, manipulation of optical and electrical properties
- Advent of nanotechnology can provide a solution in water remediation

Lack of total recovery

Surface modification by incorporating into polymers

Do we have the proposed material?

Characterize

- FTIR: Confirm the formation of the nanocomposite
- XRD: crystal structure and crystalline size
- SEM and TEM: Morphology
- SAED: nature of material
- BET: Surface area and bioavailability

Efficacy of material for dye removal

UV-Vis Spectroscopy: Adsorption removal experiments

- Adsorption isotherms
- Adsorption kinetics
- Thermodynamic properties

AIM AND OBJECTIVES OF THE STUDY

AIM

The overarching aim of this research was to synthesise and disperse iron sulphide nanoparticles into polyaniline for the removal of eosin Y in water.

OBJECTIVE

❑To synthesise FeS nanoparticles and FeS functionalized polyaniline nanocomposites by co-precipitation method and in-situ polymerization technique, respectively.

❖Characterize the materials using advanced analytical techniques such as FTIR, XRD, TEM, SEM, BET and UV/Visible spectroscopy.

❖Perform adsorption and kinetics studies on the adsorbents using eosin yellow (EY) dye.

NOVELTY OF RESEARCH

To the best of my knowledge, there is no work on FeS functionalized PANI for the removal of dyes in water.

METHODOLOGY

Preparation of FeS nanoparticles and FeS/PANI nanocomposite

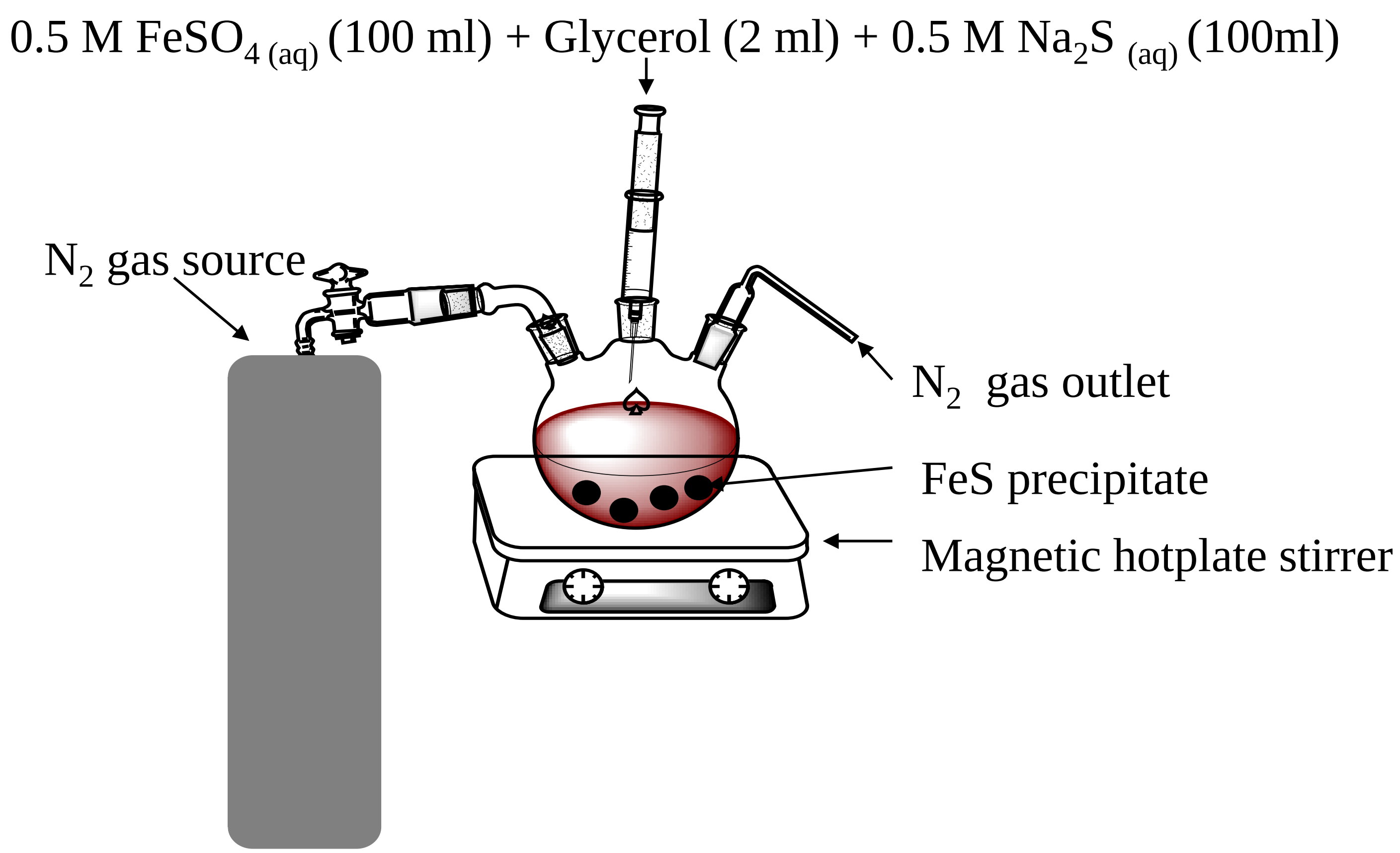


Figure 1a. Schematic of the synthesis of FeS nanoparticles

- Stirred 30 min
- Washed with D.W and ethanol copiously.
- Oven dried at 60 °C and pulverized

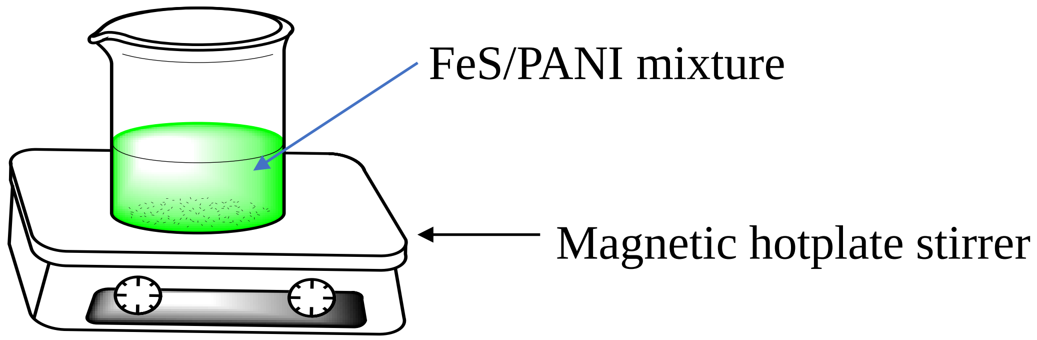
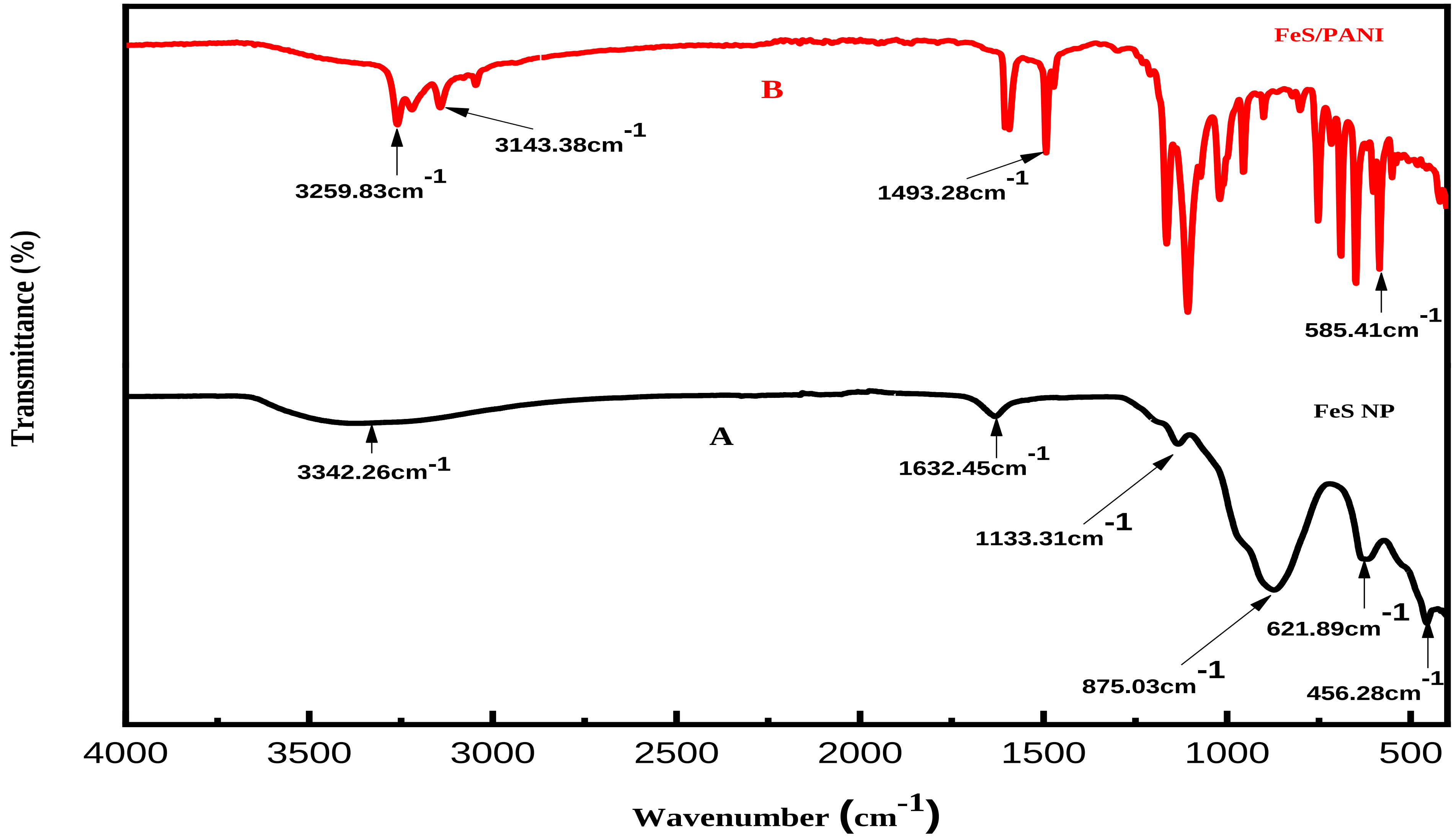


Figure 1b. Schematic of the synthesis of FeS/PANI

- FeS (0.9 g) + D.W (20 ml)
- Aniline (2 ml) + methanol (5 ml)
- Aniline (5 ml) + methanol (25 ml)
- $CuSO_{4\,(aq)}$ (16 ml) dropwise
- Stirred 30 min
- Filtered by D.W addition
- Oven dried at 60 °C and pulverized

RESULTS AND DISCUSSION

FTIR Analysis

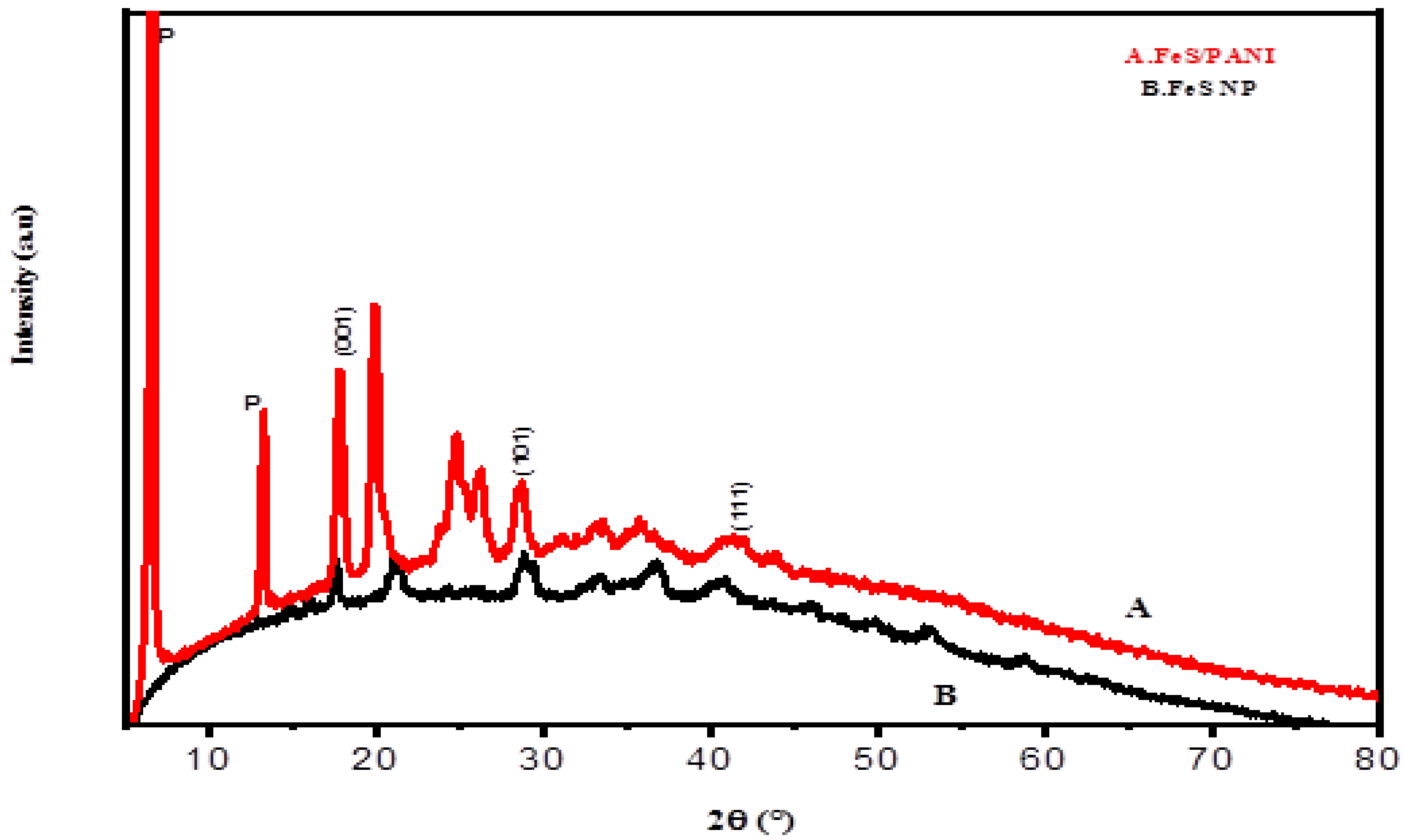


- **3342 cm^{-1}: O-H vibrations**
- **1632 cm^{-1} and 1133 cm^{-1} : S-S and S-O stretch**
- **621 cm^{-1} and 456 cm^{-1} : Fe-S stretching**
- **3259 cm^{-1} :N-H stretch**
- **3143 cm^{-1} and 1493 cm^{-1} : C-H and C-C aromatic stretch**
- **585 cm^{-1} : Fe-S stretch**

Figure 2. FTIR spectra of FeS (A) nanoparticles and FeS/PANI (B) composite

RESULTS AND DISCUSSION CONT'D

XRD Analysis



- **Peak positions at 17.56^o, 28.83^o and 40.75^o correspond to (001), (101) and (111) planes : tetragonal mackinawite phase**
- **Peak positions at 6.48°, 19°-20°:Interplanar spacing in PANI particles**

Figure 3. XRD spectra of FeS/PANI (A) nanocomposite and FeS nanoparticles (B)

RESULTS AND DISCUSSION CONT'D

SEM, TEM AND SAED Analyses

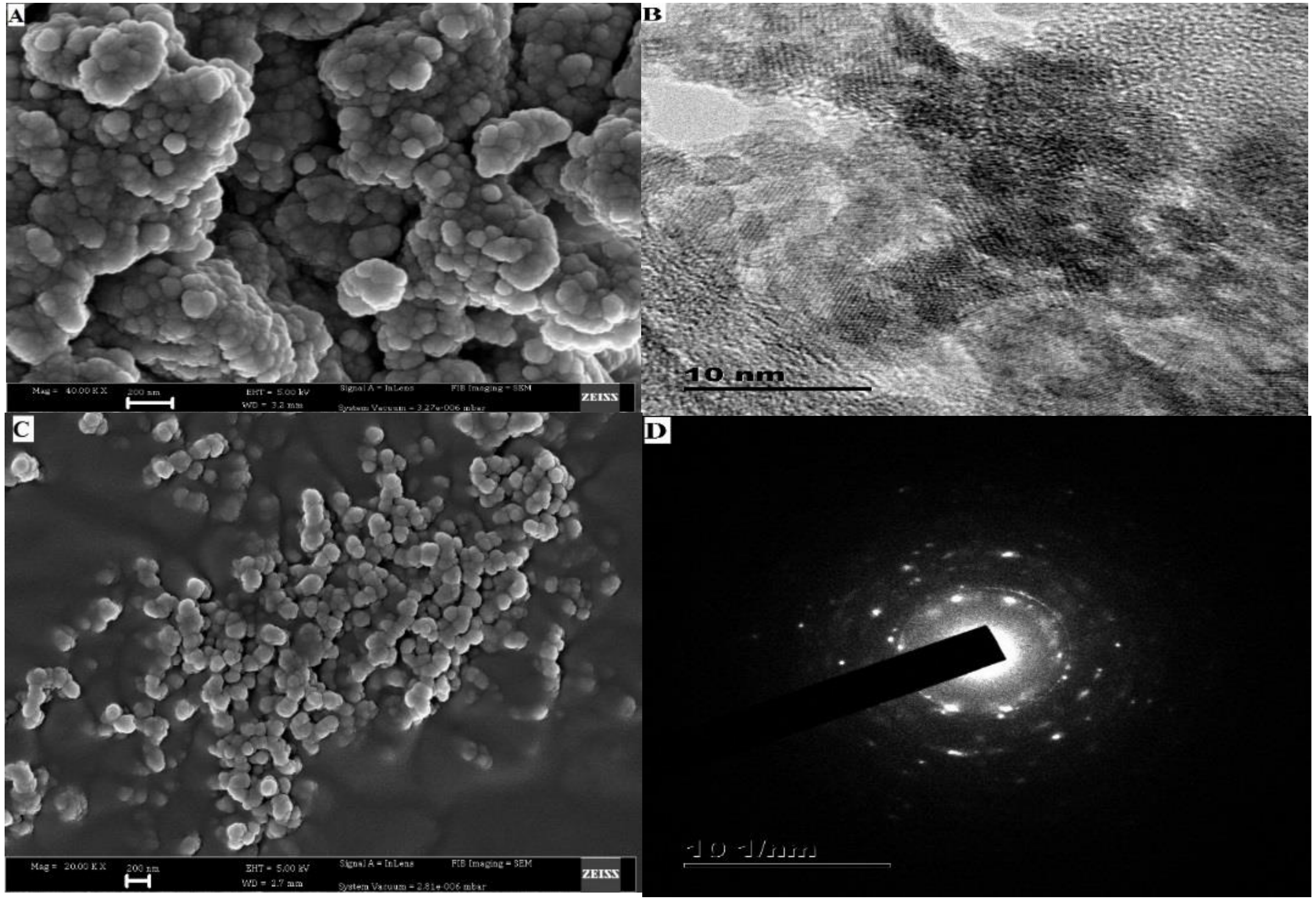


Figure 4. SEM image of FeS NP (A), TEM image of FeS NP (B), SEM image of FeS/PANI nanocomposite (C) and SAED pattern of FeS NP (D)

BET Analysis

Table 1. BET surface area, pore volume and pore size value of FeS/PANI nanocomposite

Sample ID	BET Surface Area (m^2/g)	Pore Volume (cm^3/g)	Pore Size (Ǻ)
FeS/PANI nanocomposite	18.2	0.0342	75.1699

Adsorption Removal Experiments

Effect of EY concentration and FeS/PANI dosage

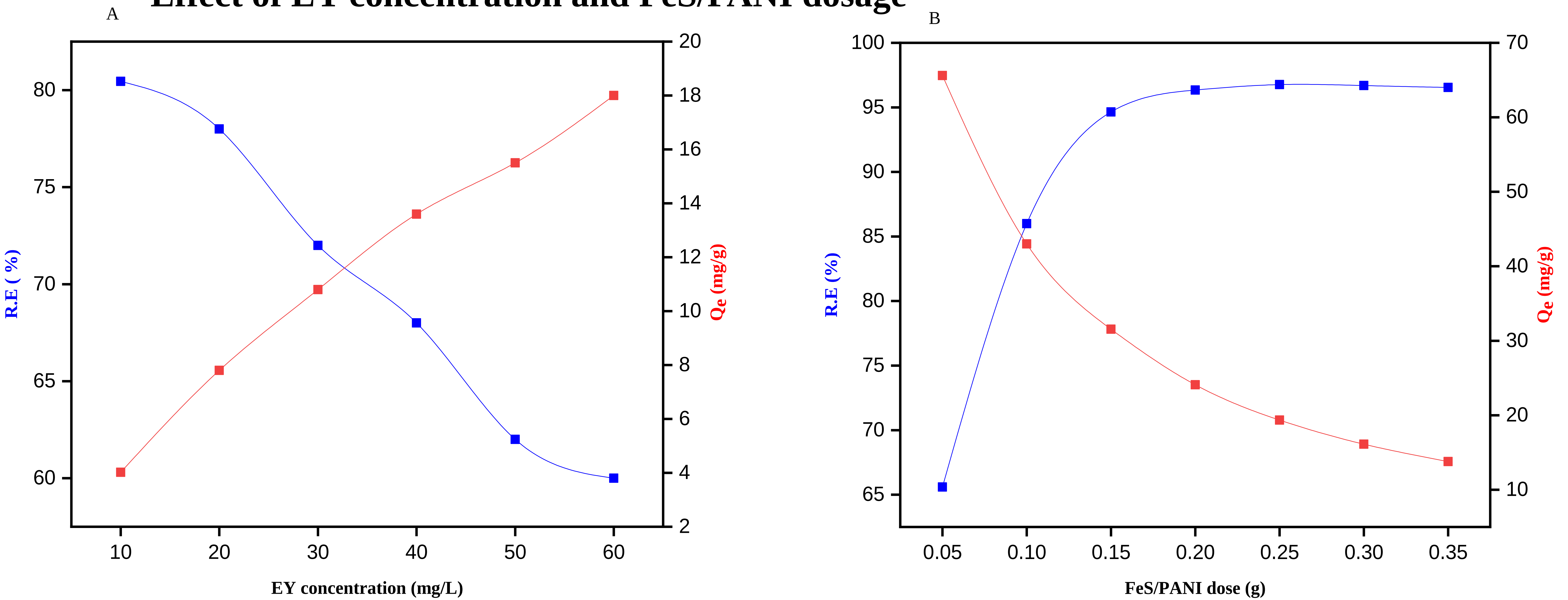


Figure 5. Effect of initial concentration (A) and adsorbent dosage (B) on EY adsorption onto FeS/PANI nanocomposite

RESULTS AND DISCUSSION CONT'D

Effect of pH and contact time

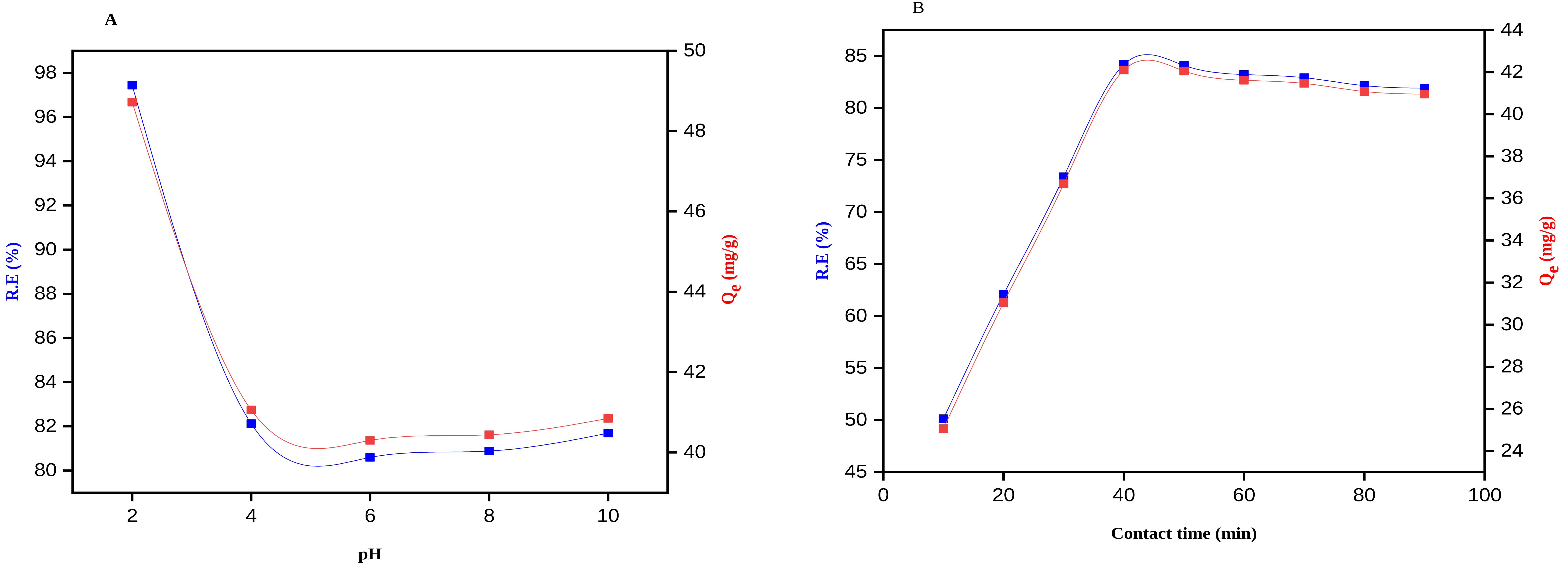


Figure 6. Effect of pH (A) and contact time (B) on EY adsorption onto FeS/PANI nanocomposite

RESULTS AND DISCUSSION CONT'D

Effect of temperature

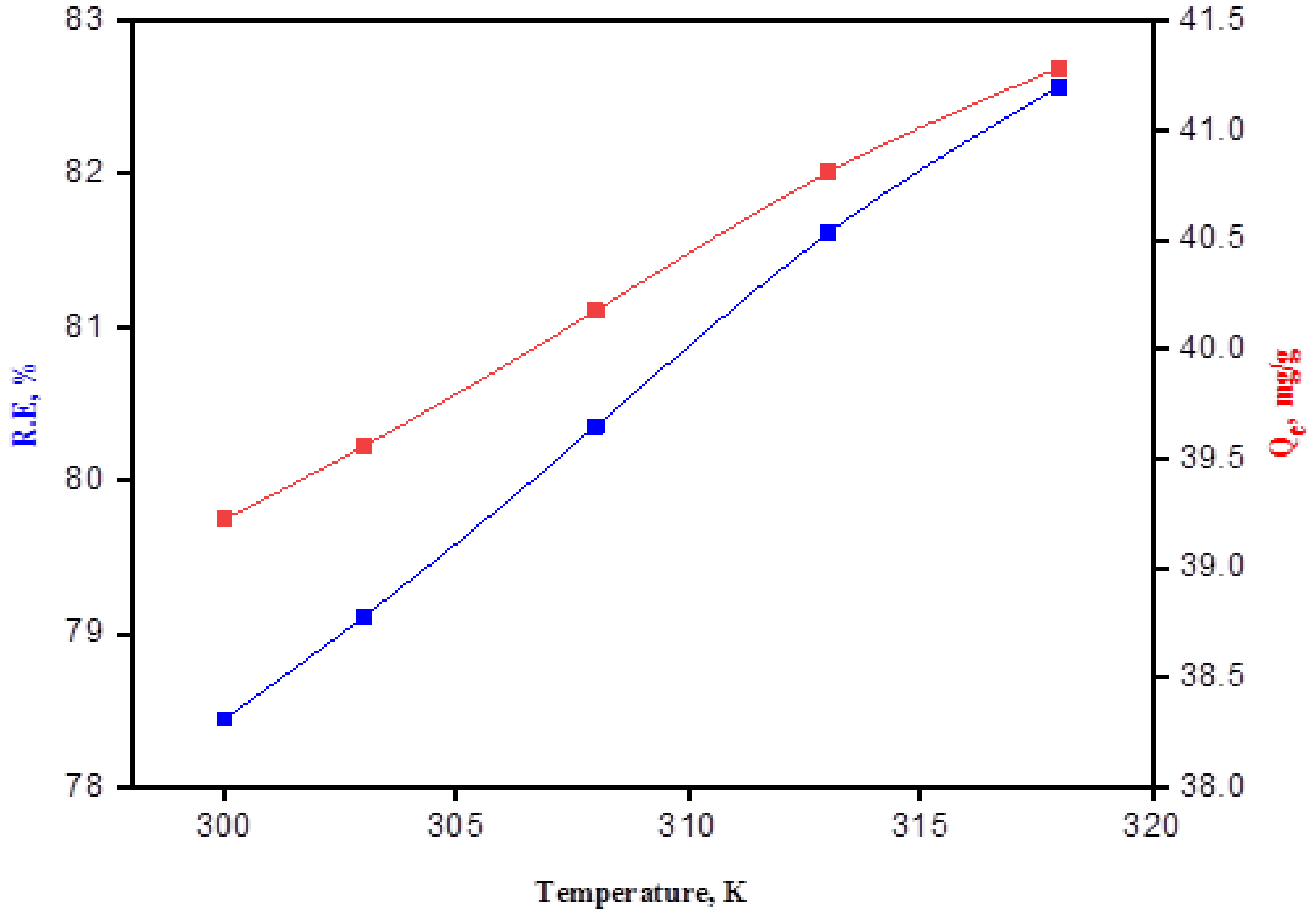


Figure 7. Effect of temperature on EY adsorption onto FeS/PANI nanocomposite

Adsorption Isotherms

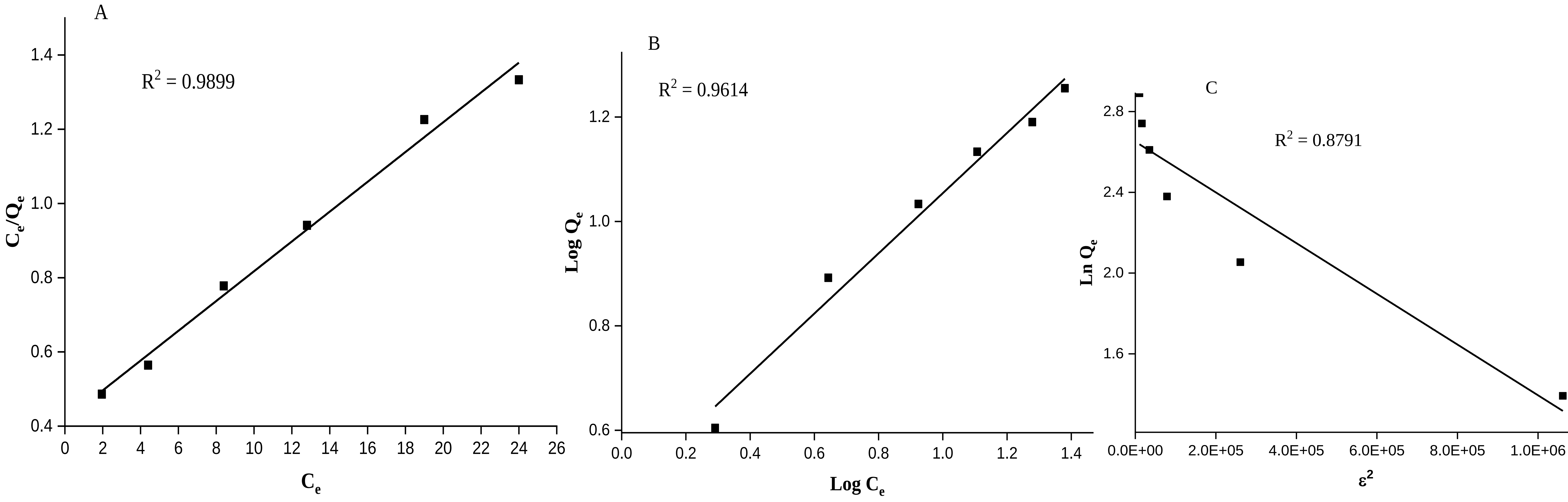


Figure 8. Langmuir (A), Freundlich (B) and Dubinin-Radushkevich (C) plots of EY adsorption onto FeS/PANI nanocomposite

RESULTS AND DISCUSSION CONT'D

Adsorption Kinetics

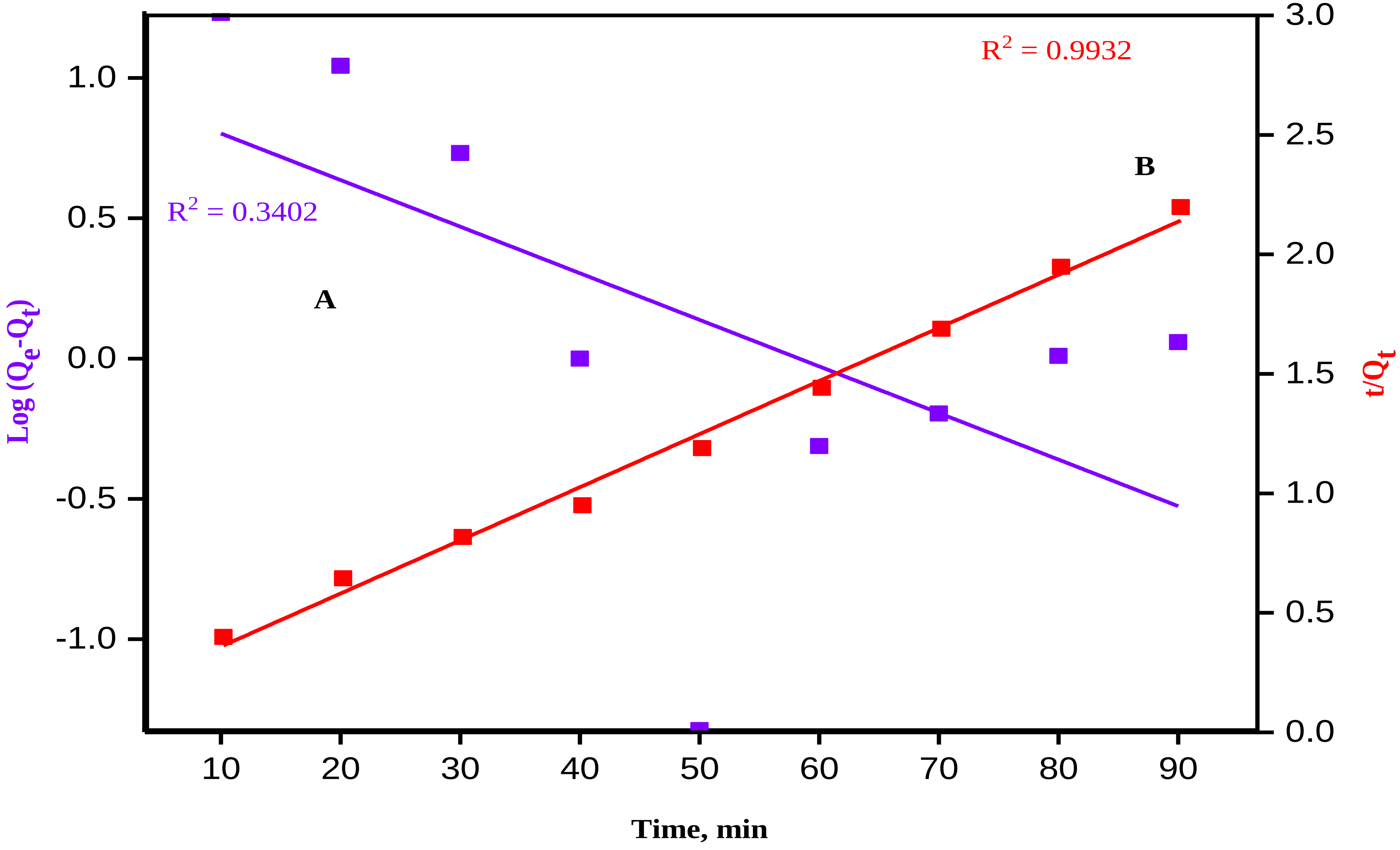


Figure 9. A pseudo first order (A) and pseudo- second-order (B) kinetic plot of EY adsorption onto FeS/PANI nanocomposite

DETERMINATION OF THE THERMODYNAMIC PARAMETERS

- $\Delta G° = \Delta H° - T\Delta S°$ (1)

- $\Delta G° = -RT \ln k_d$ (2)

- $k_d = \frac{q_e}{c_e}$ (3)

Equating eq (1) and (2) gives

- $\ln k_d = \frac{\Delta S°}{R} - \frac{\Delta H°}{RT}$ (4)

- Eq (2) can be used to determine the values of ΔG° at various temperatures. The values of ΔH° and ΔS° can be determined from the slope and intercept respectively of a linear plot of $\ln k_d$ against $\frac{1}{T}$.

Adsorption Thermodynamics

Table 2. Thermodynamic parameters for EY adsorption onto FeS/PANI nanocomposite

ΔH° (kJ/mol)	ΔS° (kJ/mol)	ΔG° (kJ/mol.K)				
		300 K	303 K	308 K	313 K	318 K
11.84	0.044	-1.49	-1.60	-1.83	-2.07	-2.28

CONCLUSION

- FeS nanoparticles were successfully prepared using the co-precipitation method.
- FTIR and XRD analyses revealed the incorporation of the nanoparticles in the polymer matrix.
- The nanocomposites produced, showed high adsorption activity towards organic dye (eosin yellow) in aqueous solutions during the batch adsorption experiments (i.e. R.E > 70% in all adsorption experiments).
- The optimum dosage for eosin removal for FeS/PANI was found to 0.15 g, lower adsorbate concentrations were most favourable for adsorption and acidic pH(FeS/PANI) was favourable.
- The pseudo second kinetic model was favourable during the adsorption process. This was also reported in literature for the adsorption of eosin yellow dye by various adsorbents.

RECOMMENDATION

- An investigation may be conducted to determine the extent of reusability of the nanomaterials.
  - This will help determine the number of cycles that the adsorbents can be used for in water treatment.
- These nanomaterials can be explored in the fabrication of filters for water purification.
- FeS/PANI can be employed for the treatment of effluents from the textile industries in the country.
- Perform zeta potential analysis to determine the overall charge on the surface of the nanocomposites.
- FeS/PANI nanocomposites can be explored for antimicrobial, antifungal and pharmaceutical study.

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ACKNOWLEDGEMENT

- Royal Society- Department for International Development (DFID) Africa Capacity Building Initiative (ACBI), UK.

- Prof Ampong, Prof Awudza and Dr. Agorku (Supervisors) and the late Paul O’Brien for granting me the opportunity to be part of the DFID initiative.

- DST-CSIR National Center for Nanostructured Materials, CSIR, South Africa, for providing technical support in characterizing the materials.

- Staff and technicians of the Physics, Chemistry, Chemical engineering, Theoretical and Applied Biology and Pharmacy departments, KNUST

THANK YOU

Danu, B. Y., et al. "Iron Sulfide Functionalized Polyaniline Nanocomposite for the Removal of Eosin Y from Water: Equilibrium and Kinetic Studies." *Polymer Science, Series B* 63.3 (2021): 304-313.

DOI: https://doi.org/10.1134/S1560090421030040