

Utilization of SPIONs coated Waste Derived Cellulosic Fiber for Bioremediation Applications

Submitted in partial fulfillment of the
requirements for the award of Bachelor of Engineering
degree in Biotechnology

By

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DEPARTMENT OF BIOTECHNOLOGY

**SCHOOL OF BIO AND
CHEMICAL ENGINEERING**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

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DECLARATION

We **D. Rajalakshmi (39230034), R. Sanjay Preeth (39230040), M. Sathiya Sree (39230041)** hereby declare that the Project Report entitled “**Utilization of SPIONs coated Waste Derived Cellulosic Fiber for Bioremediation Applications**” done by us under the guidance of Dr. M. Bavanilatha, Dr. D. Venkatesan (Internal) and Dr. Antony V Samrot (External) at Sathyabama Institute of Science and Technology, Chennai is submitted in partial fulfillment of the requirements for the award of Bachelor of Technology in Biotechnology.

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of D. Rajalakshmi (39230034), R. Sanjay Preeth (39230040), M. Sathiya Sree (39230041) who carried out the project entitled "**Utilization of SPIONs coated Waste Derived Cellulosic Fiber for Bioremediation Applications**" under our supervision from March 2022 to April 2022

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Abstract

Waste derived fibers are of great interest due to its application in various fields. Vegetable wastes can be used for fiber extraction as they are to be rich in cellulose content. In this project, fiber is extracted using vegetable wastes and further coated with Superparamagnetic Iron Oxide Nanoparticles (SPIONs) for the effective removal of contaminants in wastewater. It acts as an excellent candidate for the wastewater treatment due to its high removal efficiency and easy retrieval after treatment. The removal efficiency of SPIONs coated fiber have been examined through batch adsorption studies where the conditions such as absorbate concentration, absorbent concentration, pH, contact time were optimized

Keywords: fibers, nanoparticles, filtration, efficiency, treatment

CHAPTER - 1

INTRODUCTION

Plant wastes are can be utilized in making bioplastics, soil fertilizers, biofuels, animal feed etc. Recently, wastes are used in the extraction of fiber due to the higher cellulose content. Cellulose-based plant fibres are eco-friendly, biodegradable and renewable alternative to synthetic fibres due to their good tensile capabilities, lower density, low embodied energy and availability (Bousfield et al., 2018; Tamanna et al., 2021). Some of the cellulose derived products are cellulose acetate, cellulose acetate propionate, cellulose acetate butyrate is applied in many industries like automobile, coatings. Cellulose acetate is a natural polymer that has been modified to have a wide range of characteristics (Puls et al., 2011). These can be used in the making of cellulose acetate membrane which can be applied in the wastewater treatment, microfiltration, plastic films, depth filtration, and membrane chromatography, film packaging, cigarette filters in the textile industry, medical engineering, and in gas separation (Wang et al., 2022; Cindradewi et al., 2021). By the incorporation of nanotechnology, its application in wastewater treatment can be enhanced. In this study, SPIONs (Superparamagnetic Iron Oxide Nanoparticles) were used. SPIONs are widely used due to their size, magnetic property and high surface area (Mieloch et al., 2020; Smith et al., 2022). SPIONs are superparamagnetic iron-oxide nanoparticles with various chemical compositions, including magnetite hematite and maghemite and have a high magnetism effect in a magnetic field (Samrot et al., 2018). SPION can be used as an attractive alternative for separating chromium species easily with a magnet, making it for treating vast amounts of contaminated wastewater (Nogueira et al., 2019). In this work, reactor has been designed consisting of primary filtration with fibre extracted from vegetable wastes coated with SPIONs for the effective removal of chromium from wastewater and secondary filtration consisting of cellulose acetate membrane for the removal of other impurities.

CHAPTER - 2

LITERATURE SURVEY

Superparamagnetic Iron Oxide Nanoparticles (SPIONs) is commonly used nanoparticle with different applications. Synthesis of SPIONs was examined against *Vigna radiata* and *Eudrilus eugeniae*. Superparamagnetic Iron Oxide Nanoparticles was synthesized using chemical co-precipitation process by cobalt chloride. The synthesized SPIONs were categorized by UV-Visible Spectroscopy, SEM, EDX, XRD, TEM, AFM, XPS and Zeta potential. The synthesized SPIONs were found to be 15 nm and 20 nm respectively. The bioaccumulation of iron oxide in the treated plants was detected by Inductively coupled plasma - optical emission spectrometry analysis and Prussian blue staining. Cellular destruction and reduced reproduction rate were examined in SPIONs exposed *Eudrilus eugeniae* and ICP-OES analysis of earthworm samples confirms the bioaccumulation of SPIONs (Samrot et al, 2021)

Soils contaminated by virulent factor which is the major environment issue for human being. Sulfur (S), is a beneficial constituent for growth and for the plant development, especially it helps to reduce toxicity level. This study was to investigate different Sulfur methods (0, 50, 100 and 150 mg per kg) in reducing arsenic (As) toxicity using coriander (*Coriandrum sativum*) was examined. The results were found to be Sulfur approaches high activity of antioxidant enzymes and photosynthetic pigments, whereas low in arsenic induced oxidative stress. Shoot and root biomass takes place in the presence of sulfur in different regimes in reduction and arsenic in several concentrations. Sulfur additive in high arsenic concentration also improves protein level in shoot. Various Sulfur regimes showed development in both shoot and root, also in arsenic accumulation. The result shows that the phytoextraction is one among the effective process for toxic metals phytoremediation. Therefore, this current investigation is best evidence for phytoremediation capacity of plants for accumulation of toxic metals could be increased through exogenous sulfur process (Ghalehni and Poozesh, 2018)

An ancient water treatment process for *Moringa oleifera* seeds in water-soluble extract. Natural agent for high turbid and untreated pathogenic was effective for surface water. Reduction (80.0% to 99.5%) of high turbidity methods an appreciative clear supernatant, concurrently accompanied by 90.00% to 99.99% (1 to 4 log) bacterial reduction. Utilization of this low-cost *Moringa oleifera* process is suggested for simplified, point-of-use, low-risk water treatment, rural and peri-urban people living in extreme poverty are currently drinking best turbid and microbiologically polluted water (Lea, 2010)

Superparamagnetic iron oxide nanoparticle (SPIONs) has several methods in fields like drug

delivery, bio-sensing and bio-imaging. Furthermore, SPIONs have special surface chemistry for surface functionalization/coating with various organic or inorganic materials and thus making them suitable for different aspects. This review concluded with recent methods for synthesizing SPIONs suitable for various applications. Among, the effective activities of SPIONs are discussed herein. Hence, an overall about some of the recent process of SPIONs is introduced (Omar et al, 2019).

Chromium (Cr) is a toxic heavy metal can cause degenerative diseases on ingestion which is present in industrial effluent. Elimination of Cr from polluted water can become important to maintain and assist the nature of life. This study aimed to synthesis superparamagnetic iron oxide nanoparticles (SPIONs) to impregnate on initiate carbon and to check for increase in removal of Cr (VI). Characterization was done by UV-VIS spectrophotometer, FT-IR, SEM-EDX, XRD and VSM. Sp-GAC was carried out to possess superparamagnetic nature, and used in batch adsorption process by checking its pH, adsorbent concentration and optimization of adsorbent-adsorbate. Results have shown that Cr (VI) adsorption was dominant at acidic pH (pH 3) for both. In addition N-GAC showed effective adsorption. N-GAC and Sp-GAC adsorption were developed to be heeding to Langmuir (Aisha Farhana et al, 2022).

To improve the removal efficiency of crystal violet dye, this study uses nanoparticles in conjunction with the waste product. Orange peels are commonly discarded and considered waste, despite the fact that they are high in fibre and have potential applications. Citrus sinensis peels were collected, dried, pulverized, and analyzed using Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction Spectroscopy (XRD), Scanning Electron Microscope, and Energy Dispersive X-Ray analysis in this study (SEM- EDX). Peels were used to extract cellulose fibre, which was then characterized with FTIR, XRD, SEM-EDX, and Zeta potential before being coated with Superparamagnetic Iron Oxide Nanoparticles (SPIONs) and characterized with FTIR, XRD, SEM-EDX, and Zeta potential. In a batch adsorption investigation, crystal violet dye was removed by using peels, extracted cellulose fibre, and cellulose fibre coated SPIONs. Adsorption parameters such as adsorbate-adsorbent concentration, pH, and contact time were optimized, and it was discovered to obey the Langmuir isotherm (Samrot et al., 2022).

CHAPTER-3

AIM AND SCOPE

3.1 AIM

Current research is carried out to Utilization of *Coriandrum sativum* and its fibre coating on SPIONs for Bioremediation studies.

3.2 OBJECTIVES

- Collection of plant waste
- Extraction of Fibres from plant waste
- Synthesis of 7M SPIONs
- Coating the fibre with 7M SPIONs
- Characterization of Fibre, SPIONs and Coriander fibre with SPIONs
- Utilization in bioremediation as a new filtration system to remove heavy metals from wastewater

CHAPTER - 4

MATERIALS AND METHODS

4.1 COLLECTION OF SAMPLES

Vegetable waste were collected from grocery shop, Chennai.

4.3 EXTRACTION OF FIBRES:

Vegetable waste are mixed with aqueous 1M NaOH solution in the ratio powder/solution 1:30 (g/mL). After 24 hours at 70°C, the insoluble part is collected by filtration, washed several times with distilled water until it reaches neutral pH and then it is dried to get in a powdered form (a1)(Kouadri and Satha., 2018). Powdered form (a1) are mixed with a chloroform/ methanol solution (2:1;v/v) in the ratio powder/ solution 1:20(g/mL). After stirring 4hours at room temperature, the insoluble part is collected by filtration, need to wash with acetone for three times and finally dried to get in a powdered form(a2) (Supriya and Ramachandra, 2012).

Powdered form (a2) are mixed with 2% of Sodium hypochlorite solution in the ratio powder / solution 1/30(g/mL), at 80°C for 6 hours. The insoluble part is filtered , washed for several times with purified water until neutral pH and finally dried to get in a powdered form (a3) (de Rodriguez et al., 2006). 150mg of (a3) is mechanically stirred in 7.5mL of 40% (v/v) aqueous Sulfuric acid solution in the ratio powder / solution 20:1 (mg/mL), at room temperature for 30 minutes. The suspension is then subsequently diluted with purified water to stop acid hydrolysis. The insoluble part is then filtrated and finally dried to get in a powdered form (Morán et al.,2008).

4.6 SPIONs SYNTHESIS

4.6.1 PREPARATION OF PRECURSOR SALT

Ferric chloride salt and ferrous chloride salt was mixed together to get precursor salt

4.6.2 PREPARATION OF REDUCING AGENT

Mixture of NaOH and Ammonia (3:1)

- Mix the two-precursor solutions and kept in magnetic stirrer at 60°C for 10mins
- Once it got heated, magnetic bead was removed
- Add reducing agent drop by drop (NaOH and NH₃)
- Keep stirring using glass rod at 60°C

- Observe the colour changes to dark brown solution then it turns to viscous and then to light brown
- Finally, it turns to thick black solution. Allow it to cool for sometimes
- Cover with newspaper or with aluminum foil
- Keep aside for 24hours
- SPIONs formation occurs under the influence of magnetic field
- After 24hrs check the pH for supernatant and discard it
- Then add acid to the pellet and shake well to dissolve the pellet and check pH again
- Repeat until it becomes neutral to lyophilize

4.6.3 COATING OF SPIONs

A solution containing Aminopropyl triethoxyl silane (silane coupling agent) and ethanol in volume of the ratio 1:10 are prepared and mixed in ultrasonicator for 15 minutes, ensuring the homogeneous condition of the solution. Add 0.4 g of SPIONs to the solution and the sonicate for 30 minutes to attain uniform dispersion. Samples are then dip coated in the prepared solution for 30 minutes and later washed nitrogen purged water. The surface modified samples are dried to remove moisture content (Prasad et al., 2021).

4.7 CHARACTERIZATION OF FIBRE, SPIONS AND CORIANDER FIBRE WITH SPIONS:

The size and surface morphology of the synthesized Fibre, SPIONs and Coriander fibre with SPIONs was characterized using scanning electron microscopic micrographs JEOL-JSM-IT 200.

4.7 BATCH ADSORPTION STUDIES:

The purpose of the adsorption study was to see how much hexavalent chromium could be removed by the adsorbent.

4.7.1 PREPARATION OF ADSORBATE:

Different chromium concentrations in parts per million (ppm) were prepared for the study using potassium dichromate as the chromium salt. 500 ppm of potassium dichromate ($K_2Cr_2O_7$) in 100 mL MilliQ water was obtained by diluting 0.14 g of potassium dichromate ($K_2Cr_2O_7$) in 100 mL MilliQ water, and 100 ppm, 50 ppm, and 1 ppm of chromium solution were obtained by diluting 0.14 g of potassium dichromate ($K_2Cr_2O_7$) in 100 mL MilliQ water. To determine the best conditions, several concentrations of chromium solutions were tested against varying concentrations of adsorbents at

various time intervals and pH levels.

4.7.2 DETECTION TECHNIQUES OF CHROMIUM

The presence of chromium in the treated supernatants was determined by two methods: direct and indirect or DPC method.

4.7.3 DIRECT METHOD

The direct approach, which is commonly used for high chromium concentrations, is based on the UV-Vis absorbance maxima of chromium at 370 nm (above 1 ppm). The chromium in the treated supernatant was directly quantified using this method by measuring its absorbance at 370 nm.

4.7.4 DIPHENYL CARBAZIDE (DPC) METHOD OR INDIRECT METHOD

The DPC method can detect chromium concentrations of less than 1 ppm. 1 mL of the treated supernatant was mixed with 30 mL of 2 N H₂SO₄ to Cr⁶⁺ start dissociation, then 20 mL of DPC (250 mg DPC in 50 mL methanol/carbinol) was added to bond with Cr⁶⁺. The interaction with DPC resulted in a reddish-violet colour that was examined spectrophotometrically at 540 nm. 31,32

4.7.5 OPTIMIZATION OF ADSORBENT

Different concentrations of each adsorbent were determined in this experiment in the range of 0.5 g/L to 7 g/L in order to find the best adsorbent concentration. These doses were applied to 100 mL of 1 ppm chromium solution in various flasks. They were shaken for 2 hours to allow them to interact, and then the indirect method of chromium detection was used. The collected OD values were used to compute the final chromium concentration (using a standard graph) as well as the percentage elimination. The adsorbent concentration was displayed on the X-axis, and the percent removal was plotted on the Y-axis.

was plotted with concentration of adsorbent on X-axis and % removal on Y-axis.

$$\% \text{removal} = \frac{\text{final concentration} - \text{initial concentration}}{\text{Initial concentration}} \times 100$$

4.7.6 OPTIMIZATION OF ADSORBENT-ADSORBATE CONCENTRATION

In this stage, the best adsorbent was used and evaluated for the optimal dosage of adsorbent concentration for chromium removal. Along with the adsorbent, chromium concentration was also

optimized. Different concentrations of adsorbent to 10 g/L were taken as a set. Each set of adsorbent was added to different flasks containing varied chromium concentrations like 1 ppm, 50 ppm, 100 ppm, and 500 ppm. It was then left for interaction for 2 h under constant rotation, and later, the supernatant was analysed for chromium concentration. From the OD values, the final concentration of chromium (using standard graph) and the percentage removal was calculated, and a graph was plotted with the concentration of adsorbent on X-axis and % removal on Y-axis.

4.7.7 OPTIMIZATION OF PH

pH optimization was carried out to determine the adsorption at various pH levels and to determine whether acidic pH favours adsorption more. Different pH levels ranging from 1 to 6 were maintained in flasks containing 100 mL of 50 ppm chromium solution in this experiment. 0.5 M NaOH and 4 N HNO₃ were used to adjust the pH. Three sets of the pH ranges indicated above were taken. Adsorbents with concentrations of 1 g/L, 5 g/L, and 10 g/L were added to each of these sets and allowed to interact for 2 hours. At the end of the incubation, the supernatant was collected and tested using the direct method. The final chromium concentration (using standard graph) and percentage removal were estimated from the OD readings, and a graph was plotted with pH on the X-axis and percent removal on the Y-axis.

4.7.8. OPTIMIZATION OF TIME INTERVAL

The required dosages of adsorbent were taken and given to a 50 ppm chromium concentration in order to maximise the time required for full adsorption of chromium ions by adsorbent. The solution was constantly mixed, and the supernatant after centrifugation was collected every 15 minutes for 2 hours. The direct approach was used to examine the supernatant. The final chromium concentration (using standard graph) and percentage removal were determined from the OD values, and a graph was plotted with the time interval on the X-axis and percent removal on the Y-axis.

4.7.9 ADSORPTION ISOTHERM

After optimization of all the parameters such as adsorbent–adsorbate concentrations, time, and pH, the data were used to plot isotherms for the study. Three isotherms, namely, Langmuir ($1/q_e$ vs $1/C_e$), Freundlich ($\ln Q_e$ vs $\ln C_e$), and Temkin (q_e vs $\ln C_e$) isotherms, were plotted by calculating the C_e , Q_e , $1/C_e$, $\ln C_e$, $1/Q_e$, and $\ln Q_e$ values.

$Q_e = (C_i - C_e) V/m$; C_i = initial concentration; C_e = final concentration; V = volume; m = mass.

Characterization of Adsorbent

The pellet of adsorbed adsorbent obtained by centrifugation was dried after the adsorption studies were completed. After adsorption studies, a vibrating sample magnetometer was used to analyse

the magnetic characteristics of this adsorbent sample (VSM) (Samrot et al., 2018; Samrot et al., 2019).

4.8 DESIGN OF REACTOR FOR BIOREMEDIATION:

To remove chromium from waste water, a filtration system is designed. A glass reactor coiled with electromagnetic wire, where the coil is slightly lengthier. Super Paramagnetic Iron Oxide Nanoparticles (SPIONs) obey magnet. The electric current interacts with magnetic field. There is an inlet and outlet at the top and outlet valve at the bottom. Waste water containing chromium is poured in the inlet flow and SPIONs coated fiber is present in the surface of the reactor. After adding chromium water, it attaches to the adsorbent. Magnetic field is applied, SPIONs coated fiber gets attached to the corners to the reactor due to the magnetic field on the coil. Then the water will be free from chromium and it is further passed to cellulose acetate membrane which is actually made from the vegetable waste. Now SPIONs in the corner of the reactor will be collected at the bottom, as slurry, when we cut off the electricity to the coil. Further impurities are removed using cellulose acetate membrane. After the treatment, using suction pump, the purified water is drawn out and transferred to the tank

CHAPTER – 5

RESULTS AND DISCUSSION

5.1 SYNTHESIS OF FIBER, SPIONS AND SPIONS COATED FIBER:

The fiber extraction was done successfully (figure). SPIONs was synthesized by using chemical co-precipitation method (figure). It was further coated onto the fiber for the effective removal of chromium (figure)

5.2 SCANNING ELECTRON MICROSCOPY:

Scanning Electron Microscopy (SEM) is used for the analysis of morphology of the sample. From SEM analysis the size of the extracted fiber was analyzed and the size of synthesized SPIONs was found to be 25.09 nm in size (Figure 5.1-5.3). The size of the SPIONs coated fiber was also analyzed. Samrot et al., 2020 have reported that synthesized SPIONs of size 23 nm and 35 nm. Kavitha and Kala, 2016 have reported that, using SEM images of fibers industries can resolve contamination concerns, analyze component failure, find unknown particles, and research the interaction of chemicals with their substrates.

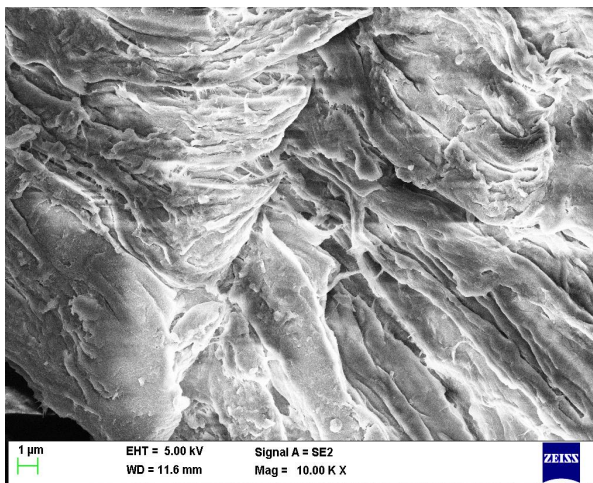


Figure 5.1 SEM Image of Fiber

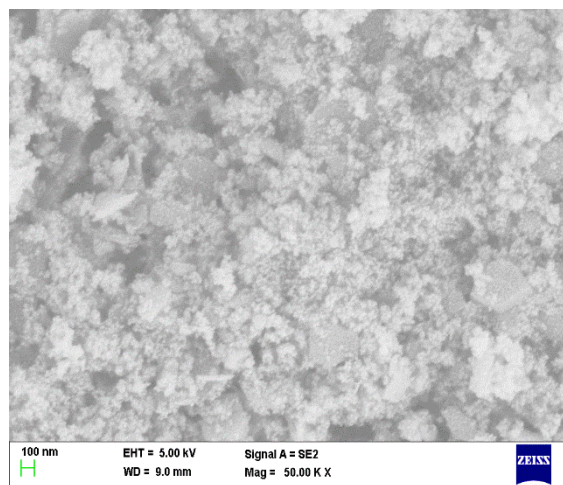


Figure 5.2 SEM Image of 7M SPIONs

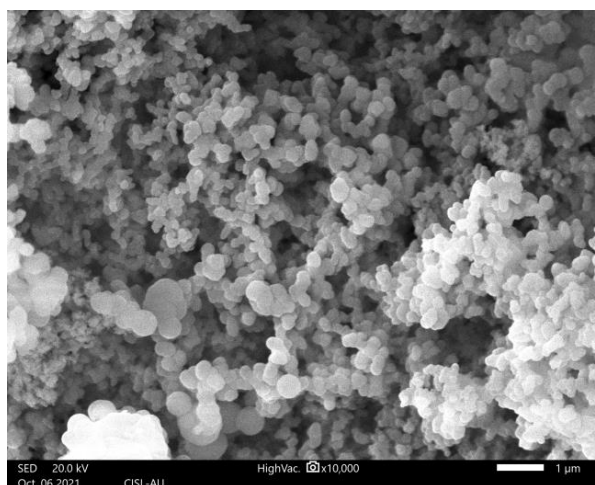


Figure 5.3 SEM Image of SPIONs coated fiber

5.3 BATCH ADSORPTION STUDIES:

The optimization condition such as adsorbate concentration, adsorbent concentration, pH, contact time were performed (Figure 5.4). The adsorbate concentration of 6 ppm was observed to be removed efficiently by the SPIONs coated fiber. Under the optimized adsorbate condition, the optimized adsorbent concentration was found to be 0.09 g. The pH at which highest removal of chromium was observed is determined to be 9. The contact time also plays an important role in batch

adsorption studies. The optimized contact time for the effective removal of chromium was observed to be 90 minutes. The adsorption pattern of the adsorbate on to the adsorption was studied using isotherm model. The R2 value was used for the determination of isotherm model. For. The adsorption pattern of chromium by SPION coated fiber was found to be tempkin model with the R2 value of 0.56.

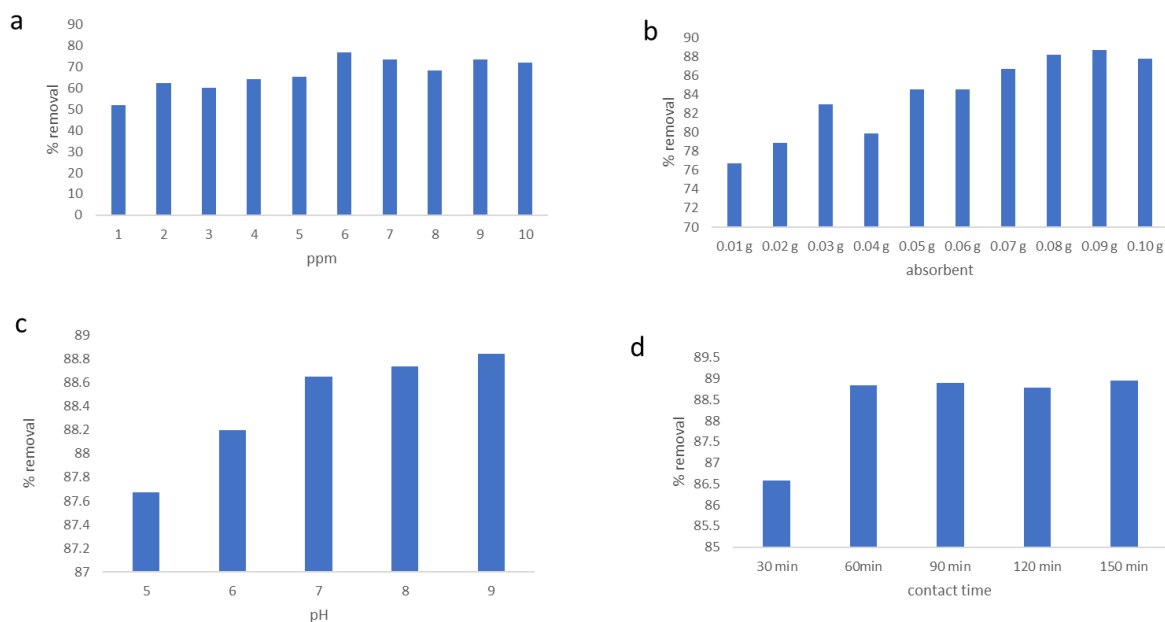


Figure 5.4 BATCH ADSORPTION STUDIES OF CHROMIUM

a) adsorbate optimization b) adsorbent optimization c) pH optimization d) contact time optimization

REACTOR:

It has been observed that the reactor has an excellent electromagnetic ability and helps in the easy retrieval of SPIONs after the treatment of chromium. Further purification of other large impurities was removed using the cellulose acetate membrane. This have given purified water and found to have removal efficiency of about 70-89%. The reaction setup was shown below (Figure 5.5-5.7)



FIGURE 5.5 REACTOR

CHAPTER – 6

SUMMARY AND CONCLUSIONS

The effective removal of chromium using SPIONS coated fiber was found to be 89%. A reactor and a setup were designed successfully for the effective removal of the chromium from contaminated water. Further, the other impurities present in the water is removed by the use of cellulose acetate membrane. This setup can be a one-step filtration process and will be more effective than traditional waste water treatment. This method will be easier, renewable and ecofriendly for the wastewater treatment

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Utilization of SPIONs coated Waste Derived Cellulosic Fiber for Bioremediation Applications

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Introduction

- Vegetable waste dumped on the ground are said to be biodegradable, emitting a foul odour while decaying. This attracts vectors of various diseases in birds, rats, and pigs leading to hazard (Singh et al., 2012)
- The current work aims to investigate the utilization of Fiber as phytostimulant and low cost bio-sorbent for the removal of dyes and antibiotics from aqueous medium (Sarimeseli et al., 2011).
- SPIONs were synthesized and was coated with the Fiber.
- Adsorption of dyes and antibiotics onto the SPIONs coated Fibers have been investigated after optimizing the experimental parameters namely concentration of adsorbent, adsorbate, pH and contact time (Bajpai and Jain, 2012).
- Hence the proposed method is a simple, low cost and eco friendly methodology for remediation and can be used as fertilizer (Shahwar et al., 2012)

AIM

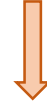
- Extraction of fiber from vegetable wastes and coating with Superparamagnetic Iron Oxide Nanoparticles (SPIONs) for bioremediation applications

OBJECTIVE

- The successful extraction, coating of nanoparticles and characterization of Fiber for bioremediation studies.

Workflow:

Collection of vegetable waste



Extraction of fiber

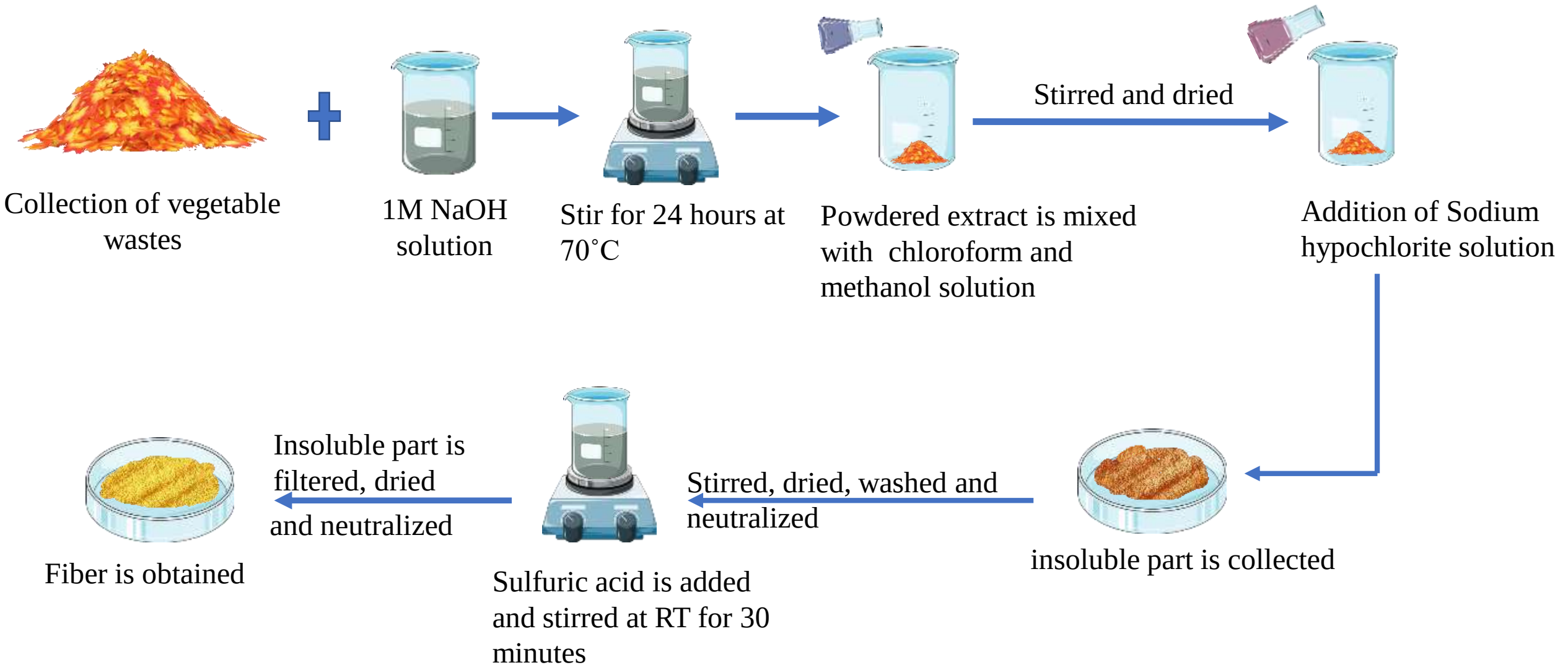


Coating of SPIONs onto fiber

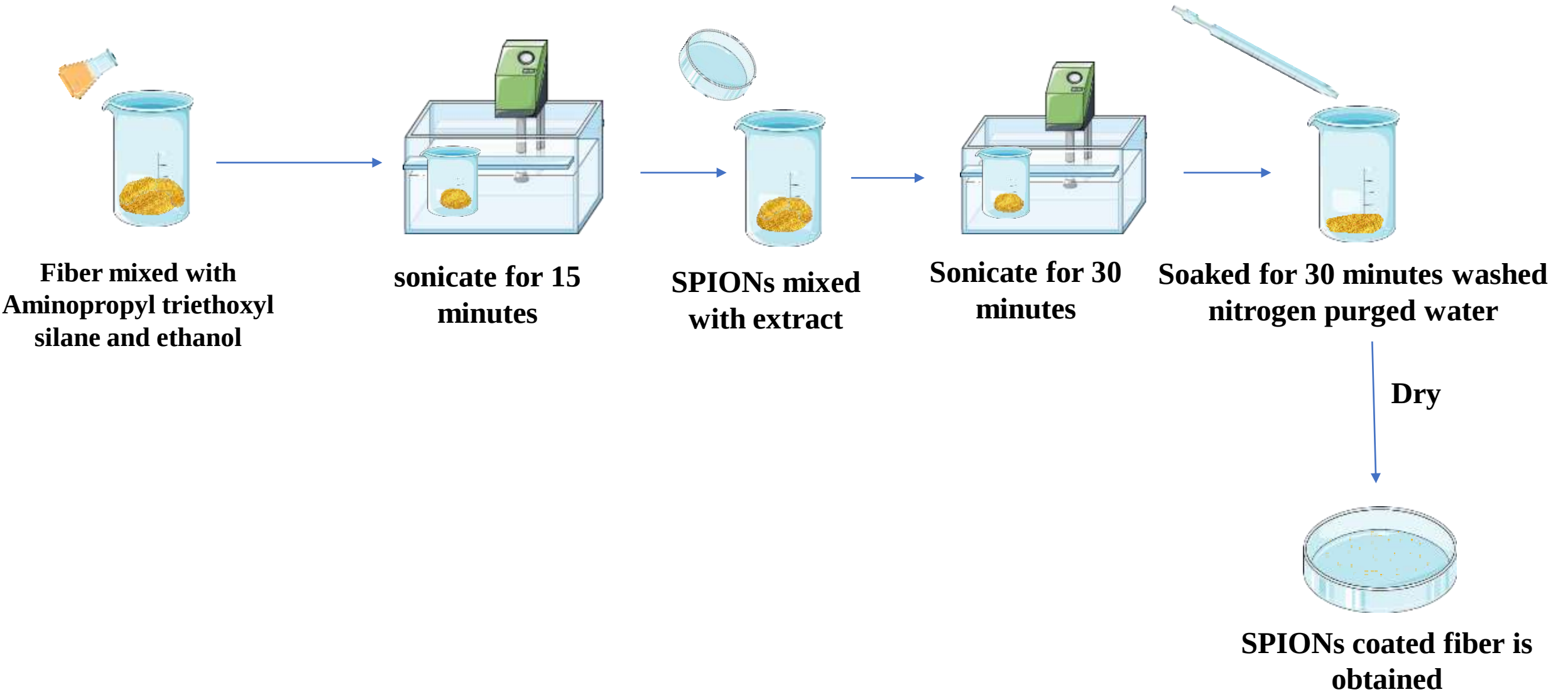


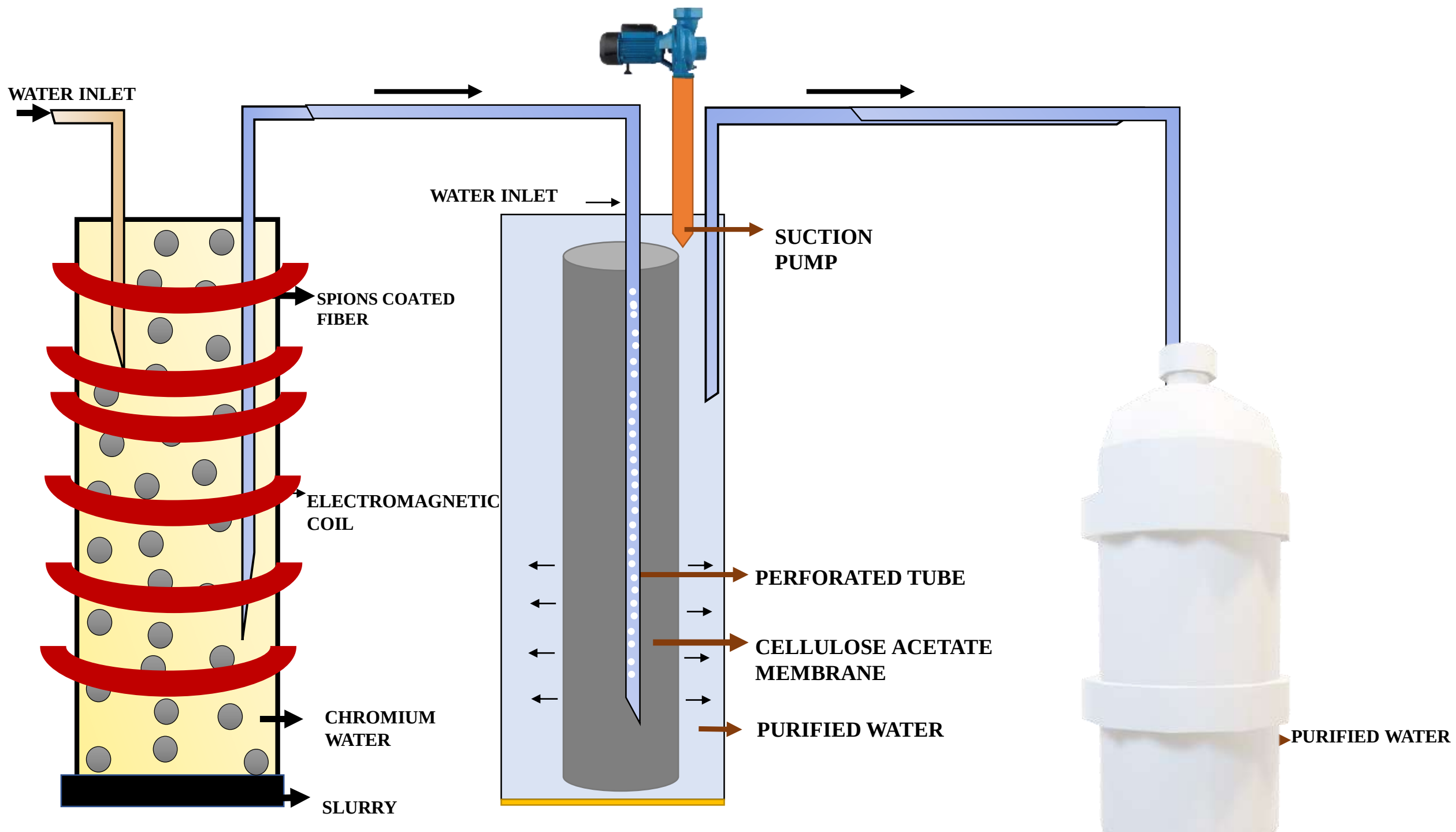
Utilization in bioremediation as a new filtration system to remove heavy metals from wastewater

EXTRACTION OF FIBER

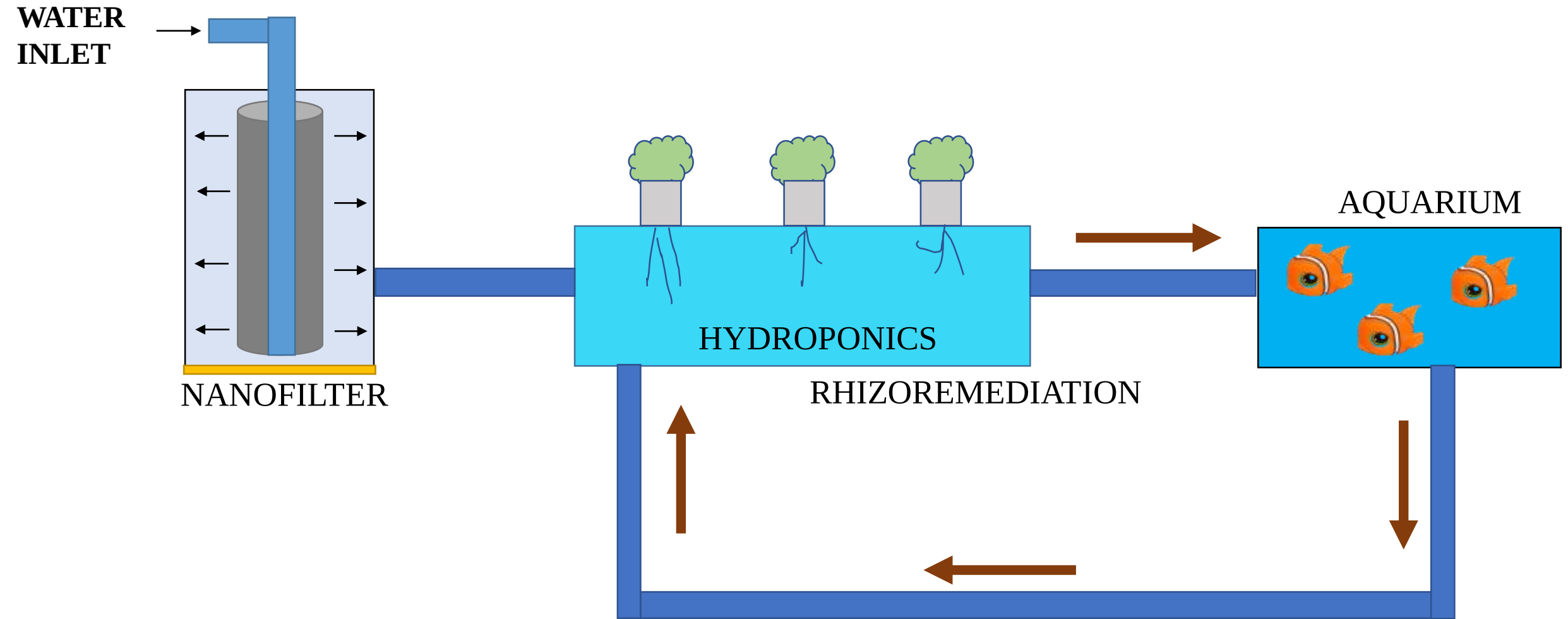


SYNTHESIS OF SPIONs COATED FIBER





Application of SPIONs coated Fiber as filter system

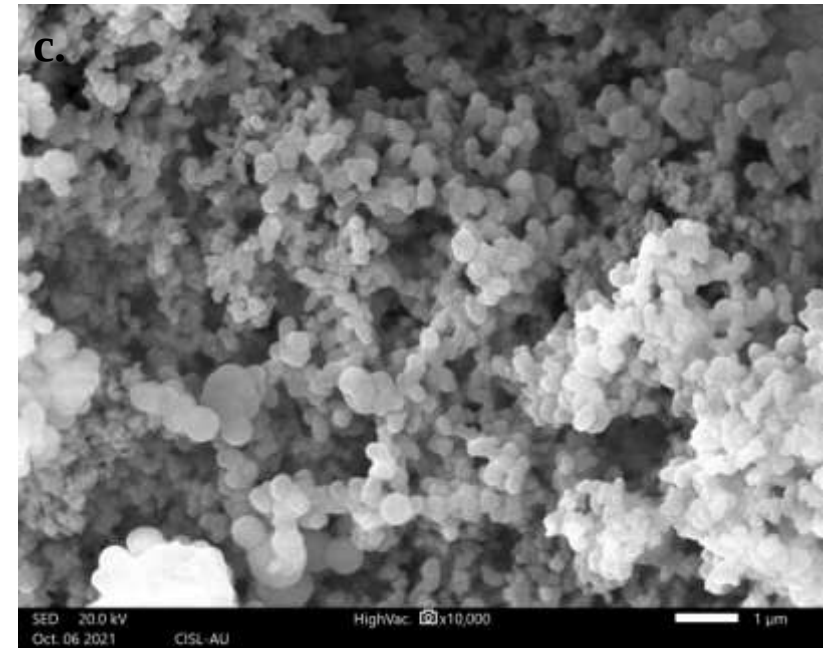
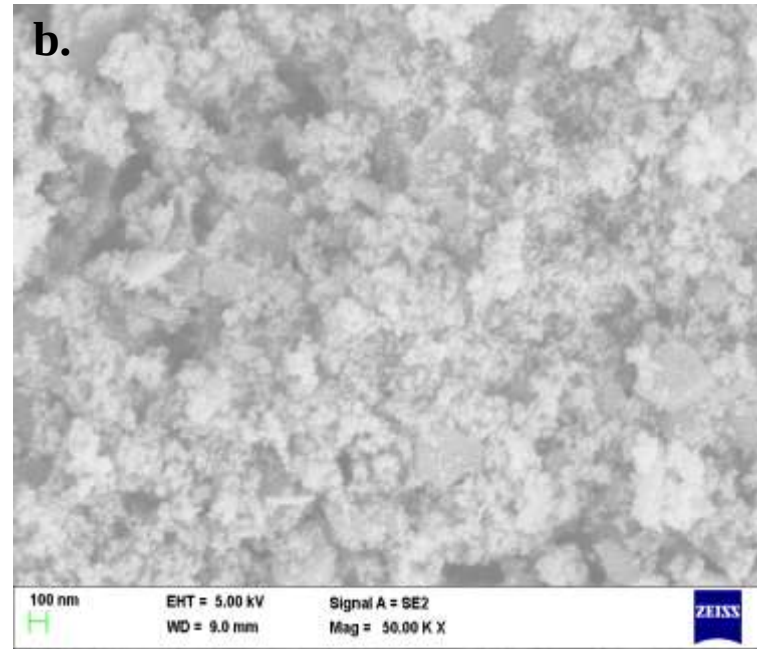
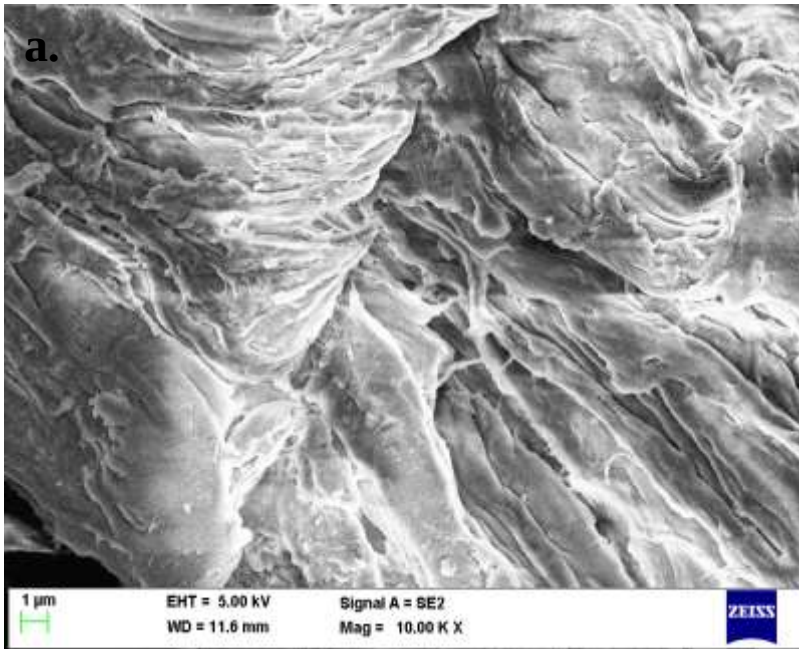


Extraction of Fiber and coating of SPIONs onto Fiber



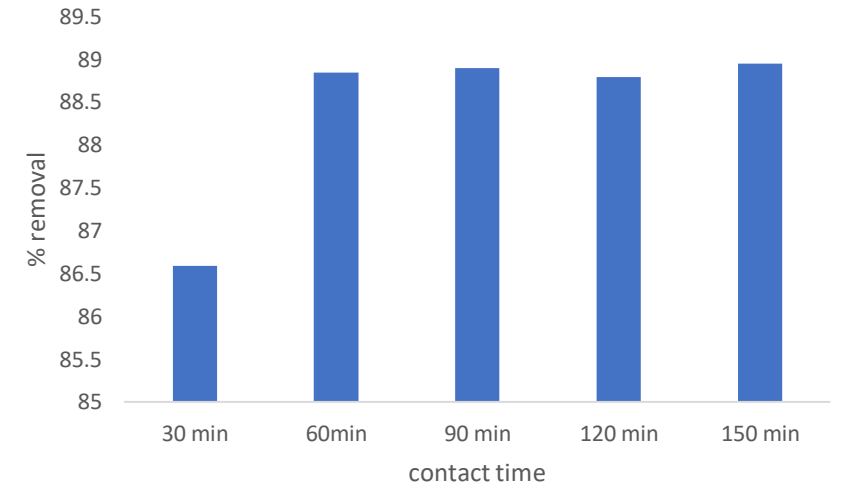
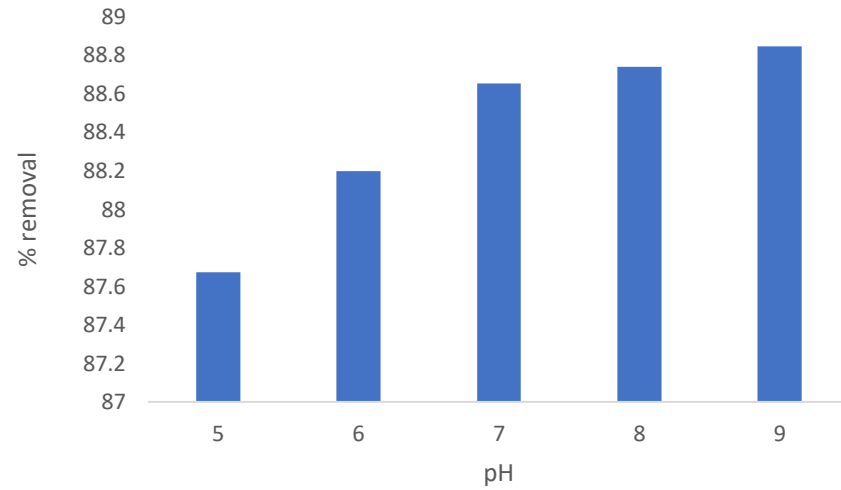
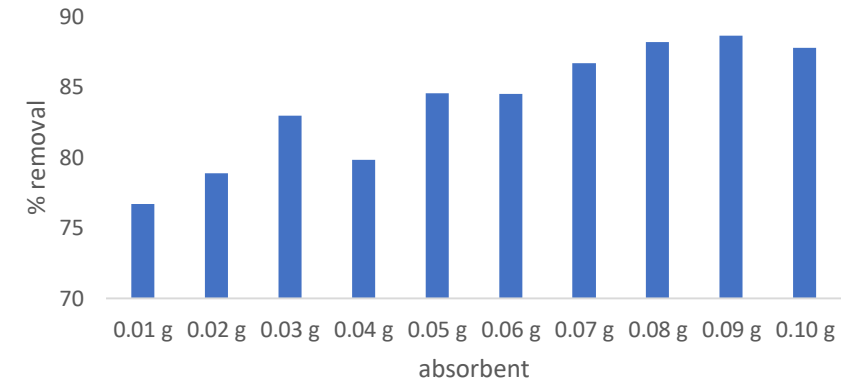
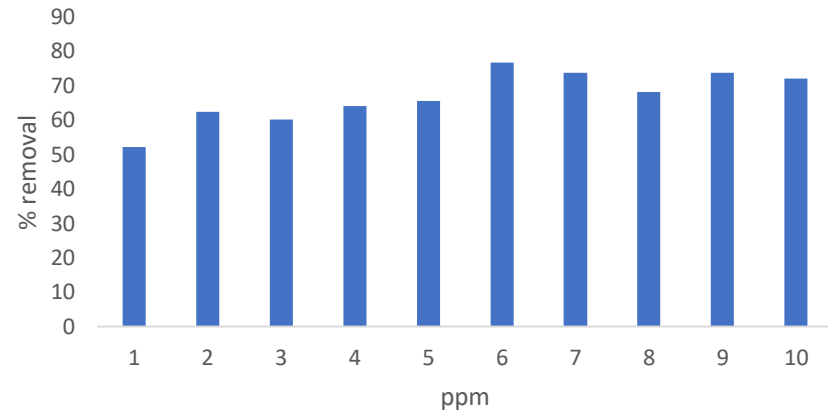
a. FIBER, b. SPIONs COATED POWDERED FIBER

Figure Characterization



a. Coriander fibre, b. 7M SPIONs, c. SPIONs coated powdered fibre

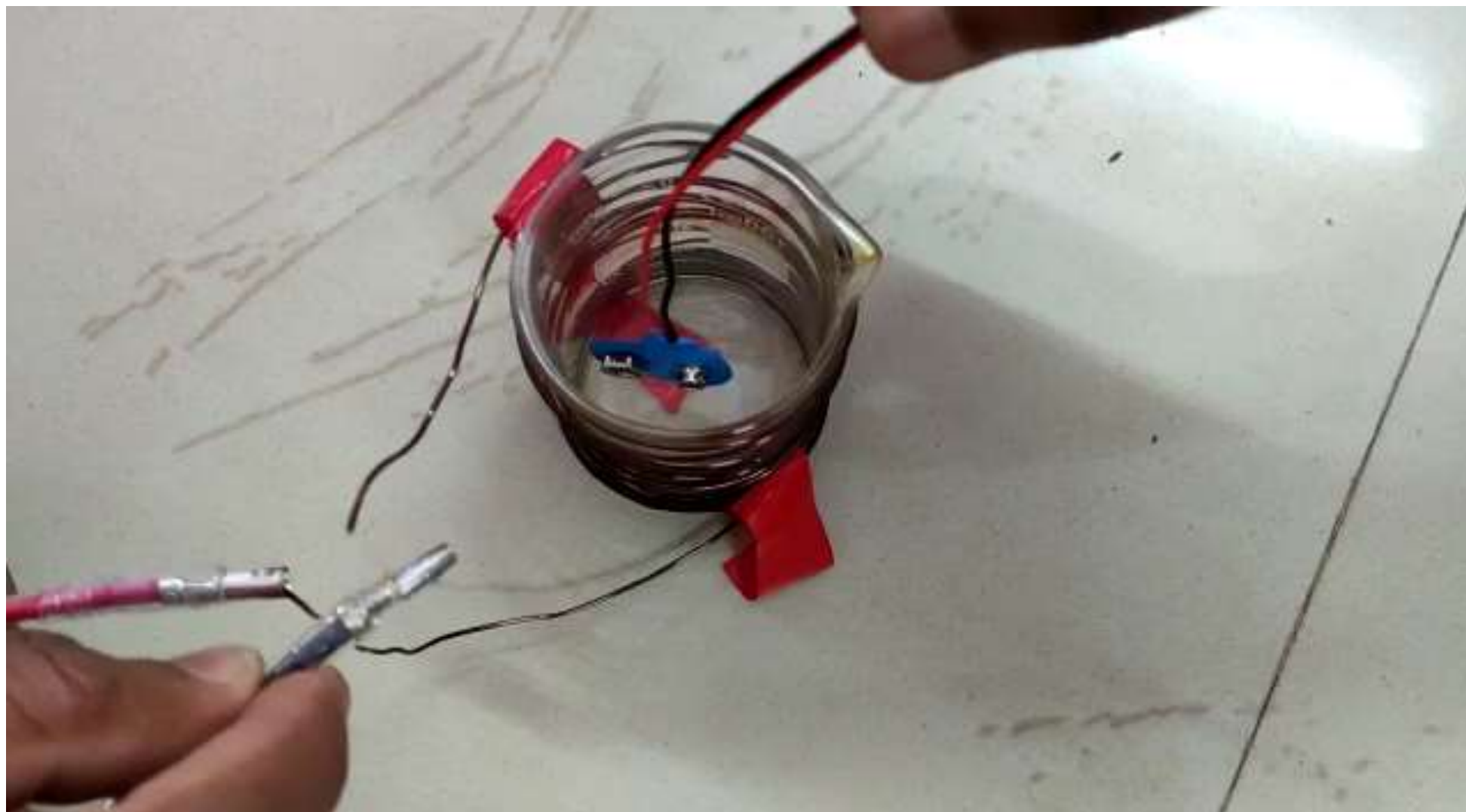
Adsorption study



Reactor



Trial Video



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THANK YOU!