

**BATCH STUDIES IN REMOVAL OF FUCHSINE DYE USING AGRO WASTE
BIOMASS**

**Sathyabama Institute of Science and Technology
(Deemed to be University)**

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Technology in Chemical Engineering.

By

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INSTITUTE OF SCIENCE AND TECHNOLOGY
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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **S ANTO FROILIN (40190003)** who carried out the project entitled "**BATCH STUDIES IN REMOVAL OF FUCHSINE DYE USING AGRO WASTE BIOMASS**" under my supervision from November 2023 to April 2024.

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ABSTRACT

The research delves into the effective removal of Fuch sine dye, a common pollutant in wastewater, by employing agro waste biomass as a sustainable and cost-efficient adsorbent. Through a series of batch experiments, several key parameters were systematically investigated and optimized to enhance the removal efficiency. These parameters included the duration of contact between the dye solution and the adsorbent material (30 minutes), the temperature of the system (50°C), the initial concentration of the dye in the solution (200 ppm), and the dosage of the adsorbent material (20 g/L). The optimization parameters, including adsorption time (15 mins) dye removal% peaks at 68.31, adsorbent volume (50 ml) dye removal % peaks at 78.70, initial dye concentration (200 mg/l) dye removal % peaks at 71.89, adsorbent dosage (20g/l)

To better understand the adsorption process, advanced mathematical models such as the Freundlich isotherm and pseudo-second-order kinetic model were employed, showcasing a robust agreement with the experimental data. Additionally, the adsorbent material underwent comprehensive characterization using sophisticated techniques including Brunauer-Emmett-Teller (BET) surface area analysis, Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and Field Emission Scanning Electron Microscopy (FESEM). These analyses provided valuable insights into the surface morphology, structural properties, and chemical composition of the adsorbent material, elucidating its mechanisms and efficacy in removing Fuch sine dye from aqueous solutions.

The findings of this study underscore the promising potential of utilizing agro waste biomass as a sustainable and eco-friendly solution for wastewater treatment. Not only does this approach offer significant environmental benefits by repurposing agricultural waste materials, but it also presents practical applications in addressing water pollution challenges, contributing to the advancement of sustainable practices in environmental management and wastewater treatment processes.

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LIST OF ABBREVIATIONS

1. ppm: Parts Per Million
2. UV-Vis: Ultraviolet-Visible
3. FTIR: Fourier Transformation Infrared Spectroscopy
4. SEM: Scanning Electron Microscopy
5. EDAX: Energy Dispersive X-ray Analysis
6. XRD: X-ray Diffraction
7. FESEM: Field Emission Scanning Electron Microscopy
8. EDS: Ehlers- Danlos syndrome

CHAPTER - 1

INTRODUCTION

1.1 BACKGROUND.

Water is an important component as all the living organisms on earth need water for their daily vital activities. With the growth of population and urbanization, the need for water gets intensified. However, the various man-made activities allow the pollutants like dye molecules to make their way into natural water bodies, rendering the water dangerous for consumption. The polluted water bodies deeply affect the health of humans and many other living organisms consuming it. Water is an important component as all the living organisms on earth need water for their daily vital activities. With the growth of population and urbanization, the need for water gets intensified. However, the various man-made activities allow the pollutants like dye molecules to make their way into natural water bodies, rendering the water dangerous for consumption. The polluted water bodies deeply affect the health of humans and many other living organisms consuming it. Thus, it becomes essential to treat water in a large quantity, with the fast and straightforward process to make it safe for consumption of not only humans but also of the other living organism living among us.

Fuchsin (sometimes spelled fuchsine) or rosaniline hydrochloride is a magenta dye with chemical formula $C_{20}H_{19}N_3 \cdot HCl$. It becomes magenta when dissolved in water; as a solid, it forms dark green crystals.

Basic fuchsin's effects are multifaceted, with both advantageous and disadvantageous aspects depending on the context. Let's delve deeper into each area:

Positive Effects:

- **Specificity:** Techniques can be tailored to target specific molecules. For instance, the Ziehl-Neelsen stain cleverly exploits the unique cell wall composition of Mycobacteria, allowing for specific diagnosis of tuberculosis and leprosy.
- **Visualization Powerhouse:** It highlights vital cellular structures like the nucleus (DNA-rich) and cytoplasm (ribosome-rich), enabling researchers to

study cell function, identify abnormalities, and understand cell cycle progression with the Feulgen stain.

- Versatility in Microscopy: Beyond routine staining, basic fuchsin can be employed in specialized techniques like the PAS stain to pinpoint carbohydrates in cell walls, fungi, and basement membranes.
- Textile Dyeing: Basic fuchsin imparts a beautiful red or pink color to textiles like cotton and wool. However, its susceptibility to fading under sunlight limits its widespread use in commercial textiles where colorfastness is paramount.

Other Applications:

- pH Indicator: Basic fuchsin acts as a readily available colorimetric indicator. Its color shift from red (acidic) to violet/blue (basic) provides a simple way to assess solution acidity/alkalinity.
- Culture Media Differentiation: By incorporating basic fuchsin into specific culture media, scientists can differentiate between bacterial strains based on their sugar fermentation abilities. Fermenting bacteria alter the media's pH, causing a color change due to basic fuchsin's pH sensitivity.
- Mucin Staining: This dye's ability to target mucins proves valuable in studying these essential components of mucus and other secretions, aiding in research on respiratory and gastrointestinal health.

Negative Effects:

- Skin Irritation: Direct contact with basic fuchsin can cause skin irritation or allergic reactions.
- Eye Irritation/Damage: Contact with eyes can lead to irritation or even damage the delicate ocular tissues.
- Environmental Impact: Improper disposal of basic fuchsin poses a threat to water sources. Since biodegradation can be slow, the dye may persist in the environment, potentially impacting aquatic life.

Additional Considerations:

- Light Sensitivity: As mentioned earlier, basic fuchsin's susceptibility to fading under sunlight restricts its use in applications requiring colorfastness.

- **Synthetic Dye:** Being a synthetic dye, concerns regarding potential long-term health effects linger. While research is ongoing, it's essential to handle basic fuchsin with proper safety measures.

In conclusion, basic fuchsin's effects present a balancing act. It offers significant benefits in various fields, particularly biological staining and diagnostics. However, responsible handling practices are crucial to minimize potential health and environmental risks.

The adsorption of Fuchsine dye plays a crucial role across a spectrum of applications, ranging from wastewater treatment to analytical chemistry, where the binding of crystal violet molecules onto surfaces, predominantly solid materials like activated carbon or other adsorbents, is pivotal. This process serves as the linchpin in removing crystal violet from solutions, making it imperative to comprehend thoroughly to optimize removal techniques and boost efficiency.

Understanding the intricacies of fuchsine adsorption entails delving into a myriad of factors, each exerting a significant influence on the process. Among these factors, pH stands out as a key determinant, as variations in pH levels can profoundly affect the surface charge of both the dye molecules and the adsorbent material, consequently altering their interaction and adsorption capacity. Similarly, temperature plays a critical role, influencing the kinetic energy of the system and thus impacting the rate and extent of adsorption.

Moreover, the properties of the adsorbent material itself, such as surface area, pore size distribution, and surface chemistry, significantly influence Fuchsine adsorption. For instance, materials with higher surface areas and tailored surface chemistries often exhibit enhanced adsorption capacities due to increased active sites available for interaction with dye molecules.

Beyond these factors, the presence of other ions or organic matter in the solution can also influence crystal violet adsorption by competing for adsorption sites or forming complexes with the dye molecules, thereby altering their availability for adsorption.

By unraveling the complex interplay of these factors, researchers can gain valuable insights into the mechanisms governing crystal violet adsorption, paving the way for the development of more efficient and sustainable removal techniques. This exploration not only enhances our understanding of dye removal processes but also

holds promise for addressing pressing environmental challenges associated with dye pollution. Thus, this introduction serves as a springboard for a deeper dive into the nuanced dynamics of crystal violet adsorption, highlighting its significance and potential implications across various scientific and environmental domains.

The biomass employed for the adsorption operation in this study was sourced from *Chromolaena odorata*, a local invasive plant commonly known as Siam Weed. Originally indigenous to South America, *C. odorata* has successfully spread to various regions of India. This plant species possesses distinct growth characteristics that contribute to its invasive nature. Despite having a woody base, *C. odorata* exhibits a flexible and soft stem structure, allowing it to thrive as a creeper, often sprawling over other vegetation and reaching impressive heights of up to 10 meters in favorable conditions. Notably, each plant produces a prolific number of seeds, estimated to range between 80,000 to 90,000 per individual, equipped with mechanisms for attachment to various surfaces like cloth, fur, and machinery, facilitating its widespread dispersal over long distances.

Another remarkable aspect of *C. odorata*'s biology is its rapid regenerative ability. Capable of sprouting from its roots and exhibiting vigorous growth rates of up to 3 centimeters per day under optimal environmental conditions, this plant demonstrates resilience and adaptability in diverse habitats. Moreover, the leaves of *C. odorata* are valued for their medicinal properties, particularly for their antimicrobial qualities, making them traditionally sought-after for healing wounds and other ailments. By harnessing the biomass derived from *C. odorata* for the adsorption process, this study not only addresses environmental concerns associated with invasive species but also capitalizes on the unique properties of this plant for sustainable applications in wastewater treatment and other environmental remediation efforts.

In the present study, we embarked on a novel endeavor by utilizing the stem of *Chromolaena odorata*, an invasive plant notorious for its disruptive effects on local vegetation, for the adsorption of CV dye. To the best of our knowledge, this represents the inaugural application of *C. odorata* biomass in such adsorption operations, marking a significant breakthrough in the field. Our approach

commenced with a systematic exploration, initially employing a one-variable-at-a-time methodology to conduct adsorption studies. Subsequently, we adopted a more sophisticated statistical approach, namely response surface methodology (RSM), to systematically investigate the optimal conditions of various variables aimed at maximizing dye removal percentage.

Furthermore, our investigation extended to assessing the reusability of the adsorbent, a critical aspect for evaluating its long-term efficacy and sustainability in practical applications. Through a series of experiments, we aimed to ascertain the feasibility of utilizing the *C. odorata* stem biomass as a reusable adsorbent for prolonged and efficient dye removal from aqueous solutions.

In addition to these fundamental studies, we designed and implemented a single-stage batch adsorber, envisioning its potential for large-scale dye removal operations. This innovative design not only demonstrates the scalability of our approach but also lays the groundwork for the practical implementation of our findings in real-world settings.

By repurposing biomass from an invasive plant species like *C. odorata* for dye adsorption, our research not only addresses environmental concerns associated with invasive flora but also presents a sustainable solution for wastewater treatment. Through this interdisciplinary endeavor, we contribute to both environmental conservation efforts and the advancement of efficient water treatment technologies, paving the way for a more sustainable future.

CHAPTER - 2

LITERATURE SURVEY

Chauhan P et al. (2021) High selectivity and adsorption proficiency of surfactant-coated selenium nanoparticles for dye removal application. The rate of environmental pollution augmenting at an alarming rate due to the continuous disposal of toxic dyes directly into the environment and water streams. The direct contact of dyes with water resources directly affects the living beings. The identification of superior methods for the treatment of water pollution caused due to effluent dyes needs higher consideration among researchers for the well-being of living flora and fauna. The available methods for controlling the decontamination of water through toxic dyes have various drawbacks. So, it is highly significant to develop such materials which can easily adsorb the dyes without causing any toxic effect on the environment and living beings. While keeping all the facts in mind, the current work highlights the comparative enhancement in adsorption capacity and selectivity of Brij-58-coated selenium nanoparticles (Brij-58@Se NPs) towards the removal of bromophenol blue (BB) dye from series of chosen dyes in aqueous media. The fabricated Se NPs were methodically characterized and the adsorption behaviour displayed fast adsorption efficiency (98% within 6 min) for BB dye out of series of chosen dyes. The efficacy of the adsorbent was further tested in six different water resources and displayed 95% adsorption efficiency for BB dye in different wastewater samples. Therefore, Brij-58@Se NP is expected as a potential adsorbent for the adsorption of organic dyes from wastewater samples.

Anyanwu S et al. (2017) Effect of ethanolic extract of *Chromolaena odorata* on the kidneys and intestines of healthy albino rats. The use of plants and plant products for medicinal purposes is an age-long practice in traditional communities and is becoming prominent globally. This study was performed to evaluate the effect of ethanolic extract of *Chromolaena odorata* on the kidney and intestine of albino rats.

Marques BS et al. (2019) Adsorption of a textile dye onto piacava fibres: kinetic, equilibrium, thermodynamics, and application in simulated effluent. This research was conducted to evaluate the methylene blue dye adsorption by piacava fibers. The effects of adsorbent amount, pH, kinetics, equilibrium, and thermodynamics were analysed, as well as the adsorbent performance in the treatment of synthetic textile effluents. The adsorbent characterization was also performed. Experimental kinetic data were fitted with pseudo-first-order, pseudo-second-order, and Elovich models. The equilibrium tests were done at 298, 308, and 318 K, and the models of Freundlich, Langmuir, Redlich–Peterson, and Sips were used. The adsorption was favoured using 0.025 g of adsorbent, pH 10, and 318 K. The Elovich model provided better fit to kinetic data. The equilibrium experimental points were well represented by the Sips model. The maximum experimental adsorption capacity of methylene blue dye was 427.3 mg g⁻¹. It was verified a spontaneous, favourable, and endothermic adsorption. Piaçava fibre was a promising low-cost material to be used for colour removal in effluents containing methylene blue.

Dahunsi SO et al. (2018) Anaerobic conversion of *Chromolaena odorata* This study evaluated the anaerobic mono-digestion of two different samples of *Chromolaena odorata*. Combinations of mechanical and thermo-alkaline pretreatments were applied to one of the two samples and labelled as “X” while the second had no thermo-alkaline treatment and was labelled as “Y”. The Central Composite Design was used to design the pretreatments. The physicochemical characteristics of the substrates were carried out using standard methods after appropriate pretreatments. From the experimental set-ups, the most probable actual Combination of different pretreatment methods enhanced enormous biogas yield from the digested substrates. Optimization of the generated biogas data was carried out using the Response Surface Methodology (RSM) and the Artificial Neural Networks (ANNs). The coefficient of determination (R²) for RSM was lower compared to that of ANN. This shows that ANNs model gives higher accuracy than RSM model. Further utilization of this weed for biogas production is encouraged by the results from this study.

Rahul et al.(2022) Use of activated *Chromolaena odorata* biomass for the removal of crystal violet from aqueous solution: Kinetic, Equilibrium and Thermodynamic study In the present study, biomass from the *Chromolaena odorata* plant's stem was activated using sulfuric acid to absorb crystal violet (CV) dye. The adsorption operation of CV dye was studied considering the effect of variables like pH, initial dye concentration, time, adsorbent dosage, and temperature. The pseudo-second-order equation best fitted the kinetic study, and thermodynamic parameters calculated were activation energy (9.56 kJ/mol), change in Gibbs energy (81.43 to 96.7 kJ/mol), enthalpy change (6.89 kJ/mol), and entropy change [-254.4 J/ (mol K)]. Response surface methodology (RSM) estimated at pH (4.902), adsorbent dosage (8.33 g/L), dye concentration (82.30 ppm), temperature (300.13 K) dye removal of 97.53% is possible. FTIR, SEM, XRD, BJH, and BET confirmed adsorption operation. The adsorbent can be reused for 5 cycles effectively. Langmuir isotherm best fitted the adsorption operation was used for designing a theoretical single-stage batch adsorber for large-scale operation.

Efeovbokhan et al.(2016) Synergy of Siam weed (*Chromolaena odorata*) and poultry manure for energy generation: Effects of pretreatment methods, modelling and process optimization. The co-digestion of *Chromolaena odorata* with poultry manure was evaluated in this study. Two samples of the weed: (A: which was pre-treated with mechanical, chemical and thermal methods) and (B: which was pretreated using mechanical and chemical methods only) were separately digested with poultry manure. Biogas generation started from the 2nd to 4th and 4th to 7th day for samples 'A' and 'B' respectively. The most desired actual biogas yield from samples 'A' and 'B' were 3884.20 and 2544.70 (10-4m³/kg VS) respectively and the gas composition was 68±2% Methane and 20±2% Carbon dioxide for sample A while it was 62±3% Methane and 22±2% Carbon dioxide for sample B. In all, there was a 38.06% increase in gas generation in 'A' over 'B'. (0.9009) was high suggesting high accuracy in the modelling and prediction. The worldwide usage of *C. odorata* is encouraged.

Soraysa Zahedi et al. Anaerobic conversion of *Chromolaena odorata* (Siam weed) to biogas. This study evaluated the anaerobic mono-digestion of two different samples of *Chromolaena odorata*. Combinations of mechanical and thermo-alkaline pretreatments were applied to one of the two samples and labelled as "X" while the second had no thermo-alkaline treatment and was labelled as "Y". The Central Composite Design was used to design the pretreatments. The physicochemical characteristics of the substrates were carried out using standard methods after appropriate pretreatments. From the experimental set-ups, the most probable actual biogas yields in experiments "X" and "Y" were 0.3554 m³/kg Total Solid (TS) fed and 0.1803 m³/kg TS fed with the desirability of 99 and 100%, respectively. Further shown in the result is a 49.2% higher experimental (actual) biogas yield in experiment "X" over "Y". Gas chromatographic analysis revealed the CH₄ and CO₂ content of both experiments to be 65 ± 1.5%; 21 ± 3% and 53.5 ± 2.5%; 26 ± 3%, respectively. Combination of different pretreatment methods enhanced enormous biogas yield from the digested substrates. Optimization of the generated biogas data was carried out using the Response Surface Methodology (RSM) and the Artificial Neural Networks (ANNs). The coefficient of determination (R²) for RSM was lower compared to that of ANN. This shows that ANNs model gives higher accuracy than RSM model. Further utilization of this weed for biogas production is encouraged by the results from this study.

CHAPTER – 3

AIM AND SCOPE

3.1 AIM

- To prepare adsorbent from Siam weed leaves and stem.
- To characterize the adsorbent
- Batch studies in adsorber
- Use of batch studies in waste water treatment process
- Optimization studies in batch adsorbent

3.2 OBJECTIVE

1. Determining the adsorption capacity: Batch adsorption studies aim to quantify the amount of adsorbate (such as dyes, heavy metals, or organic compounds) that can be removed by a specific adsorbent material under controlled conditions. This helps in understanding the maximum adsorption capacity of the material and its efficiency in removing pollutants from a solution.
2. Evaluating adsorption kinetics: Batch studies help in analyzing the rate at which adsorption occurs over time. By monitoring the adsorption process at different time intervals, researchers can determine the kinetics of adsorption, including the initial adsorption rate, equilibrium time, and overall adsorption efficiency.
3. Assessing equilibrium behavior: Batch adsorption studies aim to reach equilibrium between the adsorbate and adsorbent, where the rate of adsorption equals the rate of desorption. By studying the equilibrium behavior, researchers can determine the optimal contact time required for maximum adsorption efficiency.
4. Optimizing process parameters: Batch studies allow for the investigation of various process parameters, such as initial concentration of the adsorbate, pH of the solution, temperature, and adsorbent dosage. By systematically varying these parameters, researchers can optimize the conditions for achieving the highest adsorption efficiency.
5. Studying adsorption isotherms: Batch adsorption studies help in constructing adsorption isotherms, which describe the relationship between the amount of

adsorbate adsorbed onto the adsorbent surface at equilibrium and the concentration of the adsorbate in the solution. Understanding the adsorption isotherms can provide insights into the adsorption mechanism and surface properties of the adsorbent material.

Overall, batch adsorption studies play a crucial role in characterizing the adsorption behavior of different materials and optimizing the adsorption process for various environmental and industrial applications.

3.3 SCOPE

Exploration of Agro Waste Biomass: Batch studies offer the opportunity to explore different types of agro waste biomass, such as rice husk, coconut shell, sawdust, and others, as potential adsorbents for fuchsine dye removal. This involves assessing the availability, cost-effectiveness, and adsorption capacity of these materials.

Optimization of Adsorption Process: Batch studies allow for the optimization of the adsorption process by investigating the influence of parameters like pH, initial dye concentration, contact time, adsorbent dosage, and temperature. This optimization ensures efficient dye removal and helps in determining the most favorable conditions for adsorption.

Kinetics and Thermodynamics Studies: Batch studies enable the evaluation of adsorption kinetics and thermodynamics to understand the mechanisms involved in the dye removal process. This includes determining the rate of adsorption, equilibrium adsorption capacity, and the effect of temperature on adsorption efficiency.

Characterization of Adsorbent Material: Batch studies involve the characterization of agro waste biomass before and after the adsorption process using techniques such as FTIR, SEM, and XRD. This characterization helps in identifying any structural changes in the adsorbent material and confirming the adsorption mechanism.

Reusability and Sustainability: Batch studies assess the reusability of agro waste biomass as adsorbents for multiple cycles, evaluating their sustainability and economic viability. This involves studying the regeneration process and determining any loss of adsorption capacity over successive cycles.

Development of Adsorption Models: Batch studies contribute to the development of adsorption models based on isotherm equations (e.g., Langmuir or Freundlich) to predict adsorption behavior and design practical batch adsorption systems for large-scale applications.

Application in Wastewater Treatment: Ultimately, the scope of batch studies extends to the practical application of agro waste biomass in wastewater treatment for the removal of fuchsine dye, addressing environmental concerns and contributing to sustainable solutions.

In summary, batch studies in the removal of fuchsine dye using agro waste biomass offer a comprehensive exploration of adsorption processes, from material selection and optimization to characterization, reusability, and application in wastewater treatment.

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS REQUIRED

The following materials were used for the synthesis Chromolaena :

- Chromolaena(Siam weed)
- Hot air oven
- sulfuric acid (H_2SO_4)
- Magnetic pellet.
- water
- Sodium Bicarbonate($NaHCO_3$)
- 1-liter beakers
- Filter papers
- Stirrer

4.2 PREPARATION OF ADSORBENT

1. Drying:

- The stems and leaves of Chromolaena odorata are spread out in a thin layer and exposed to sunlight.
- Sunlight helps to evaporate moisture from the plant material, reducing its water content. This step is crucial because excessive moisture can affect subsequent processing steps and the quality of the final product.

2. Grinding and Sieving:

- Once the plant material is adequately dried, it is ground into a fine powder using a grinder or mill.
- After grinding, the powdered material is sieved to achieve a uniform particle size. Sieving helps to remove any larger particles and ensures consistency in the size of the final product.
- The target particle size range of 75 to 100 μm is chosen based on the specific requirements of the intended application, such as maximizing surface area for adsorption.

3. Activation with Acid:

- In this step, a portion of the powdered *Chromolaena odorata* sample is mixed with a specific volume (500 mL) of 0.1 N H₂SO₄ (sulfuric acid) solution.
- The mixture is stirred for 18 hours. This duration allows the acid to interact with the plant material, potentially breaking down organic components and creating surface functional groups that can enhance adsorption properties.
- Acid activation is a common technique used to modify the surface chemistry and increase the porosity of adsorbent materials, improving their capacity to adsorb target substances from solution.

4. Neutralization:

- After the acid activation step, the material obtained is soaked or rinsed in a solution of 0.1 N NaHCO₃ (sodium bicarbonate).
- Sodium bicarbonate is a mild base and is used to neutralize any residual acid present in the material. This step helps to prevent the acid from interfering with subsequent processes or applications and ensures the material is safe to handle.
- Neutralization may also help to stabilize the pH of the material, making it more suitable for certain adsorption applications.

5. Drying:

- Following neutralization, the material is dried to remove excess moisture and prepare it for storage or further processing.
- Drying is typically carried out using an oven set to a specific temperature (353 K or approximately 80°C in this case) for a defined duration (6 hours).
- Proper drying helps to prevent microbial growth, maintain the stability of the material, and ensure consistent performance in subsequent application.

4.3 METHODOLOGY

Adsorbent Preparation

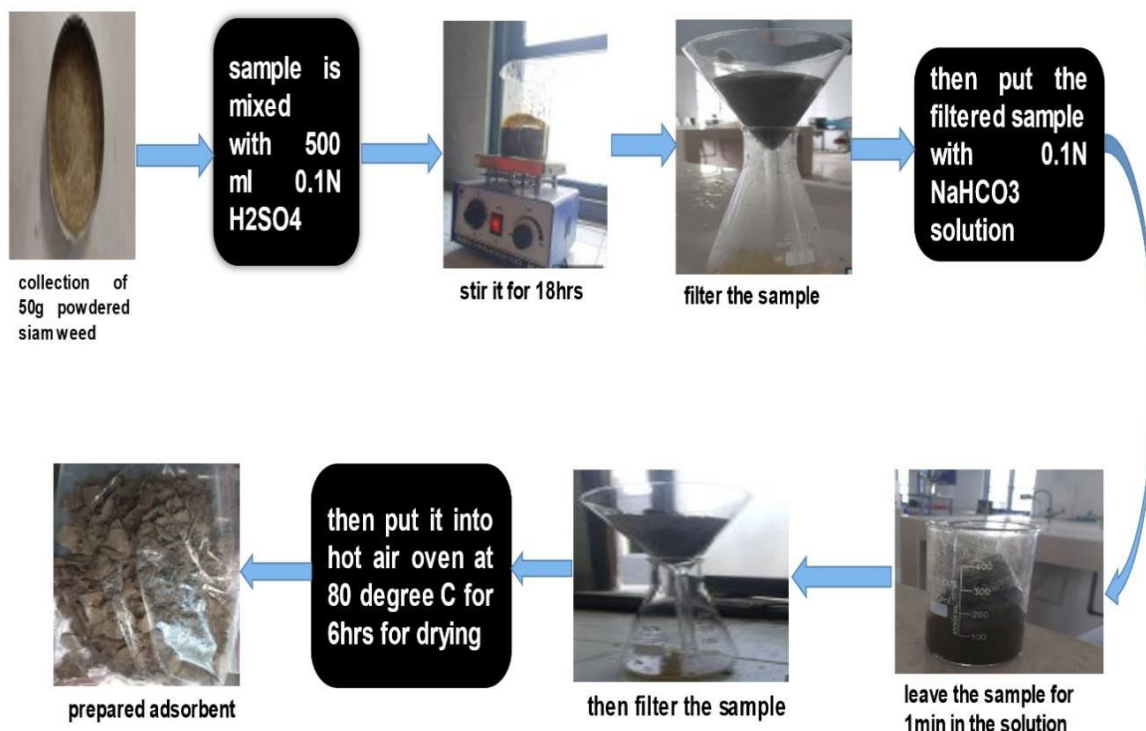


FIG 4.1: PREPARATION OF ADSORPTION

4.4 CHARACTERISATION OF ADSORBENTS

Fourier Transform Infrared (FTIR) Spectroscopy was employed to analyze surface functional groups of *Chromolaena odorata*. The spectrum was obtained by recording wavelengths ranging from 3300 cm⁻¹ to 1519 cm⁻¹ in transmission mode. X-Ray Diffraction (XRD) was utilized to examine structural variations in the prepared adsorbents. Field-emission scanning electron microscopy (FESEM) with energy dispersive X-ray spectroscopy (EDX) was done to study the morphological structure of the adsorbents.

4.5 ADSORPTION EXPERIMENT

The experimental investigation delved deeply into exploring the adsorption potential of *Chromolaena odorata*'s acid-activated stem (AASCO) towards Fuchsin dye, employing a meticulously Batch studies as the experimental platform. Within this setup, meticulous attention was paid to maintaining experimental integrity and accuracy. The setup's total volume of 1000mL provided ample space for conducting the experiments, ensuring sufficient interaction between the AASCO adsorbent and the Fuchsin dye solution. Moreover, the inclusion of 50 mL of the prepared dye solution within this setup facilitated precise control over the experimental conditions, allowing for accurate measurement and analysis.

To establish the desired concentrations for experimentation, the Fuchsin dye solution was meticulously derived from a stock solution of 1000 ppm. This process involved careful dilution to achieve the specific concentrations required for testing. Such meticulous preparation ensured that the experimental conditions accurately reflected real-world scenarios, enhancing the reliability and applicability of the study's findings.

In initiating the adsorption process, great care was taken to introduce a precise quantity of the AASCO adsorbent into the Fuchsin dye solution. This step was critical in ensuring consistent and reproducible results across experiments. The subsequent exposure of this mixture to a batch studies, with a magnetic stirrer of maintained for a duration of 15 minutes, provided optimal conditions for adsorption to occur. This carefully controlled environment promoted efficient interaction between the adsorbent and the dye molecules, facilitating the adsorption process.

Throughout the experimentation, a systematic approach was adopted to investigate key variables such as the dosage of AASCO adsorbent, initial Fuchsin dye concentrations, and the duration of exposure. By systematically varying these parameters, the study aimed to elucidate their influence on the adsorption process comprehensively. This meticulous exploration allowed for a nuanced understanding of the factors affecting adsorption efficiency, enabling the identification of optimal conditions for maximum adsorption capacity.

Upon completion of the adsorption operation, stringent filtration procedures were employed using Whatman number 1 filter paper to separate the adsorbent-dye complex from the solution. This step was crucial in isolating the adsorbed dye molecules for subsequent analysis. The subsequent analysis of the filtrate using a UV-vis spectrophotometer, with a focus on determining the residual CV dye concentration at its maximum absorption wavelength ($\lambda_{\text{max}}=590 \text{ nm}$), provided quantitative insights into the extent of adsorption achieved.

In conclusion, the experimental investigation represented a meticulous and thorough exploration of the adsorption potential of AASCO towards CV dye, conducted with a high degree of precision and attention to detail. By employing a systematic approach and stringent experimental protocols, valuable insights were gained into the factors influencing adsorption efficiency, paving the way for further advancements in the field of adsorption science and environmental remediation strategies.

$$\text{Dye Removal\%} = \frac{(C_o - C_t)}{C_o} \times 100 \quad (1)$$

$$\text{Biosorption Capacity (mg/g)} = \frac{(C_o - C_t) V}{m} \quad (2)$$

where C_o is the concentration of Fuchsine dye initially present in aqueous solution (mg/L), C_t is the Fuchsine dye concentration at time t (mg/L), m is the mass of the biomass (g), and V is the volume of the solution

CHAPTER – 5

RESULTS AND CHARACTERISATION

5.1 FTIR(Fourier Transform Infrared)

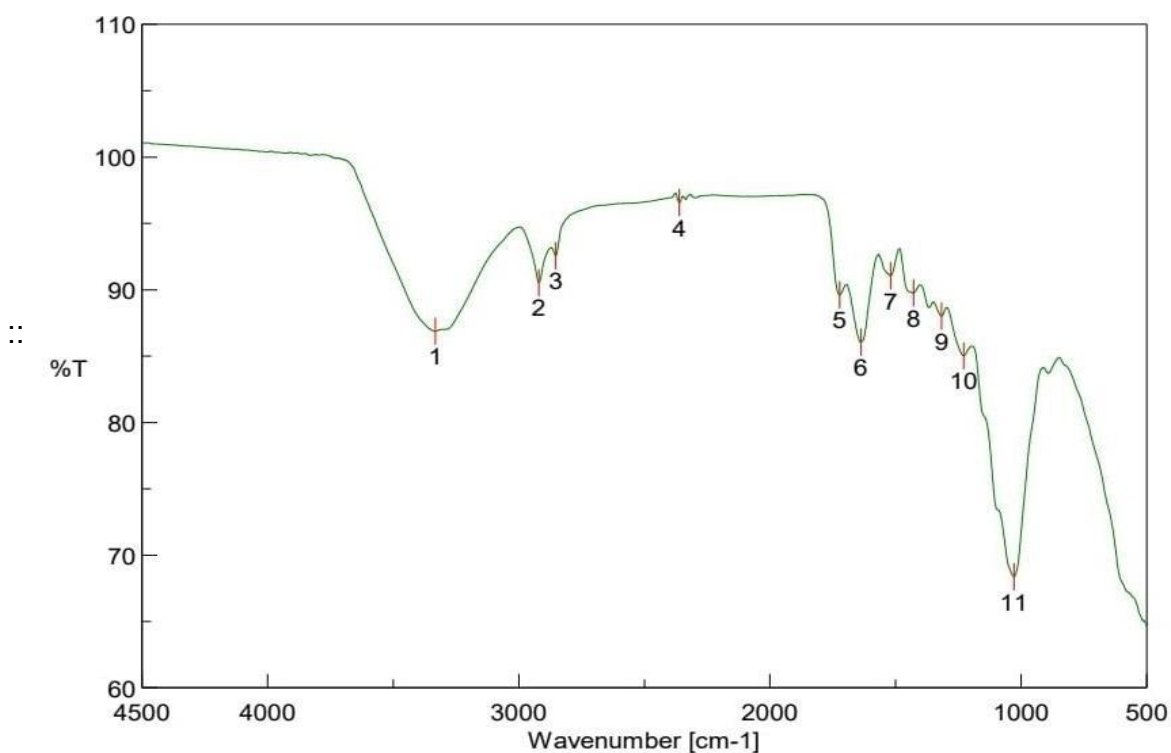


Fig .5.1: FTIR Spectroscopy results for synthesis of Chromolaena

Wavenumbers around 3300 cm⁻¹ indicate an O-H stretch, suggesting the presence of alcohols. C-H stretches are observed between 2800-3000 cm⁻¹, shows the presence of alkanes. The C=O stretch around 1723 cm⁻¹ points towards ketones. Similarly, the peak at 1519 cm⁻¹ corresponds to a C=C stretch, indicating alkenes or aromatics.

5.2 XRD (X-Ray diffraction).

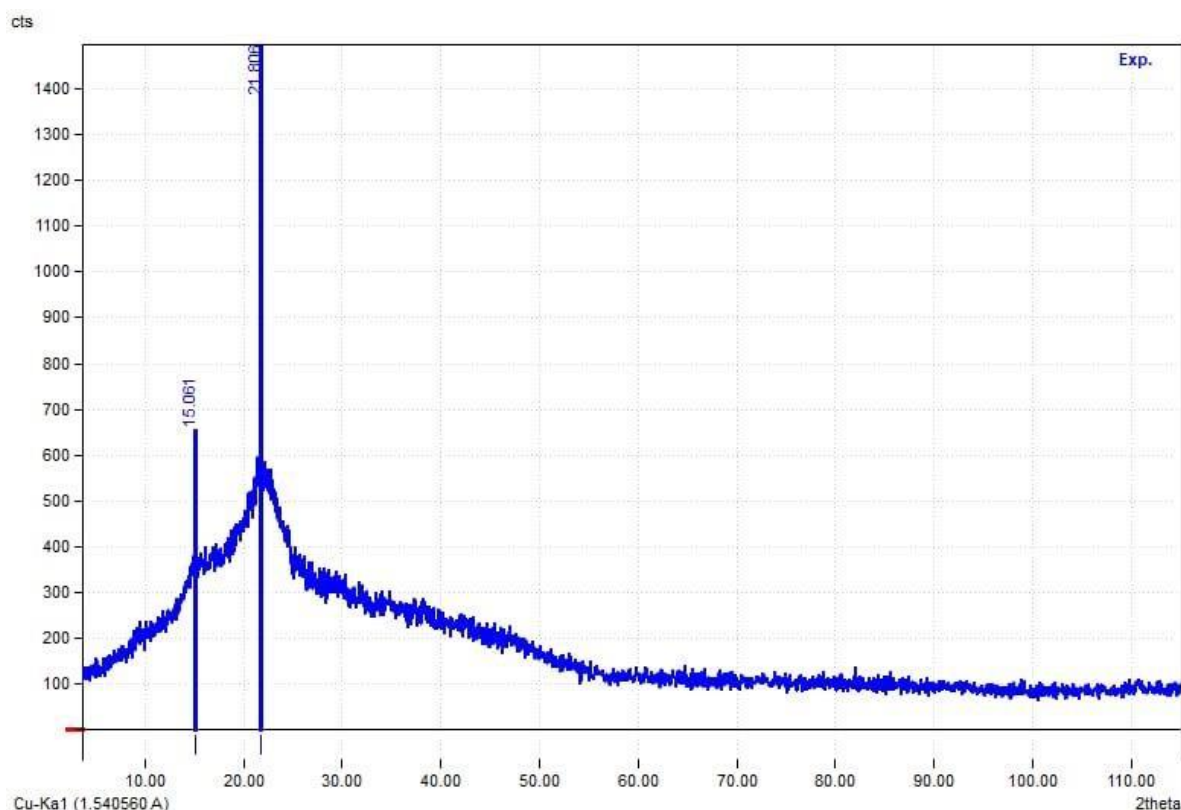


Fig .5.2: X-Ray diffraction spectroscopy

- XRD spectrum is indicated in Fig. 5.1.2, which shows a well-defined and intense peak at 2 (degree of 21.956), indicating the presence of crystalline and well-defined structures present in *C. odorata* biomass.
- Furthermore, the intensity of the well-defined peak does not reduce after the biomass is activated with acid or after the adsorption operation.
- Therefore, Fig. 5.1.2 indicates that the adsorption of fuchsine dye on AASCO will probably take place on the upper part of the crystalline surface

5.3 FESEM

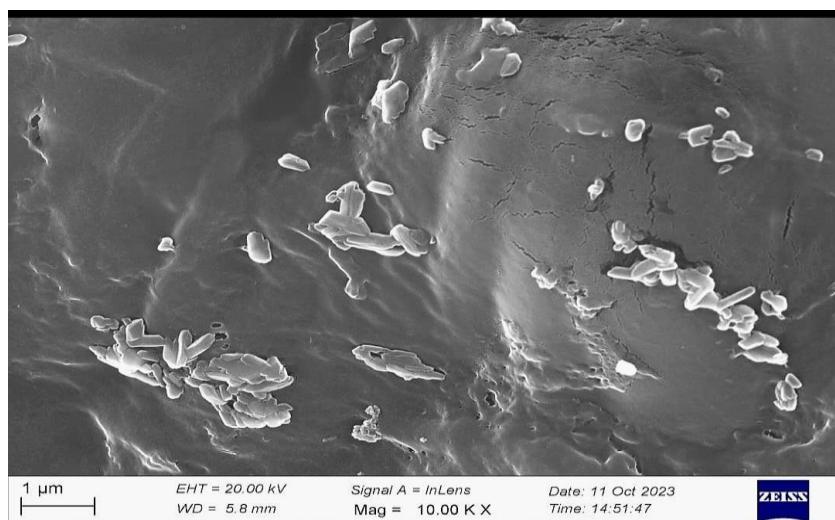


Fig 5.3: FESEM Image under 10.00 KX magnification (before adsorption)

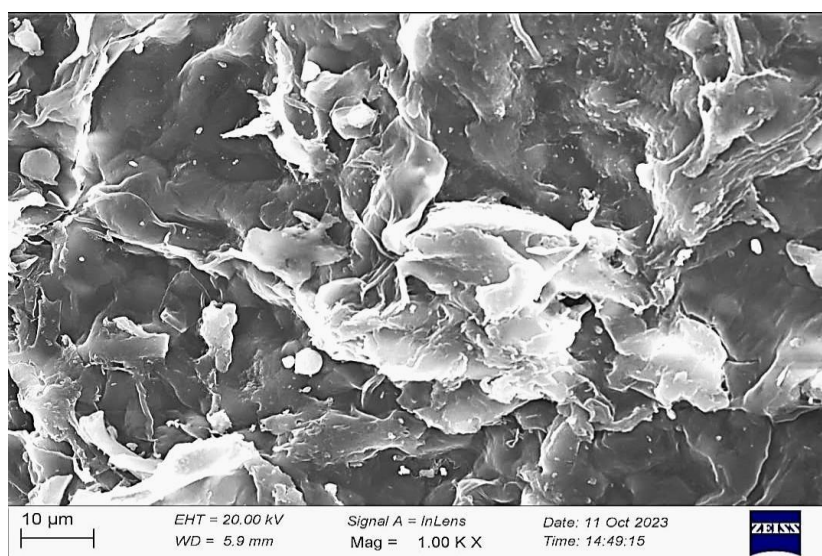


Fig.5.4: FESEM Image under 1.00 KX magnification (before adsorption)

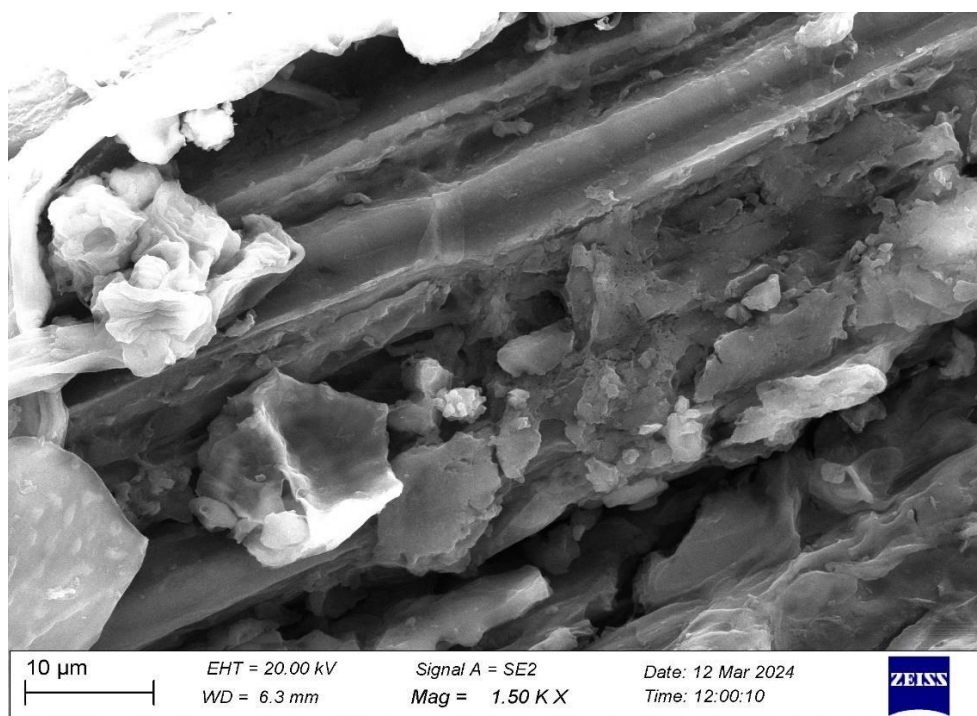


Fig 5.5: FESEM Image under 1.50 KX magnification (after adsorption)

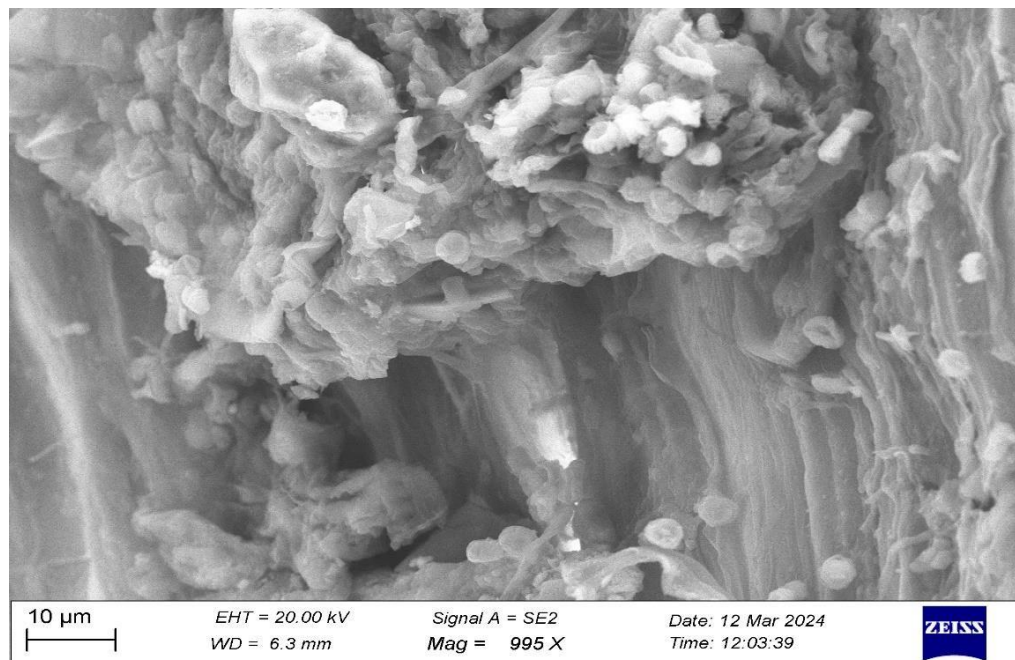


Fig 5.6:: FESEM Image under 995 X magnification (after adsorption)

5.4 Electron Dispersive X-Ray (EDX)

The EDAX mapping shows the composition of the adsorbent sample. The carbon and oxygen peaks obtained are due to the large amounts of C-H, C-O and O-H bonds present in Siam weed. The presence of sodium and sulfur is due to the addition of sodium bicarbonate and sulfuric acid during the activation process.

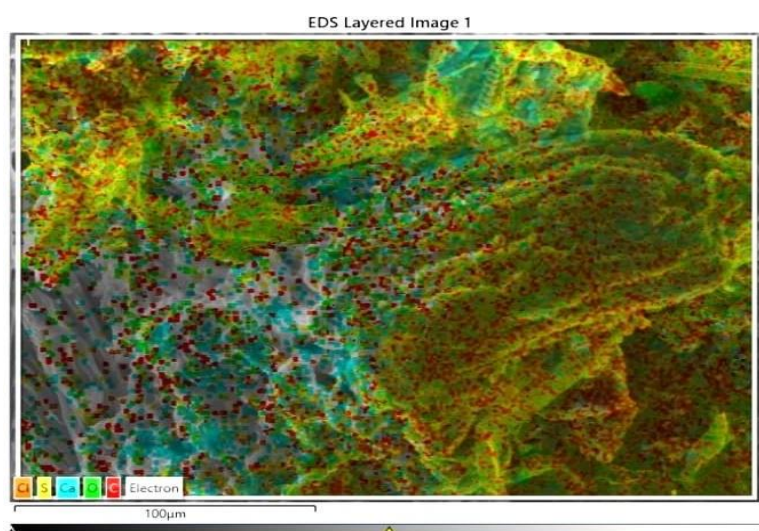


Fig 5.7: EDS layered image of siam weed before adsorption

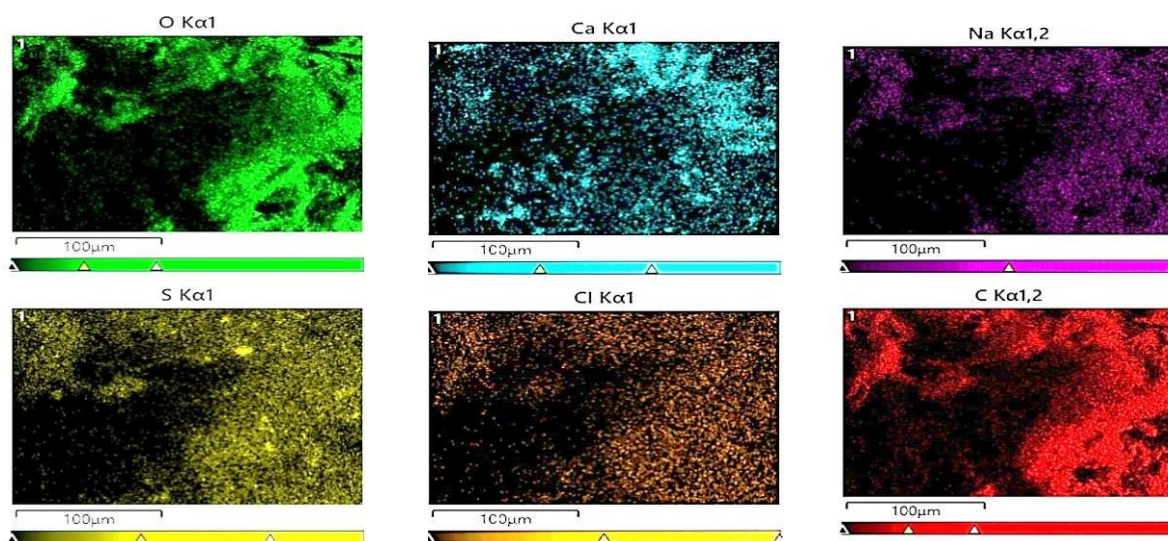


Fig 5.8: EDS layered image of O, S, Na, C, Ca, Cl before adsorption

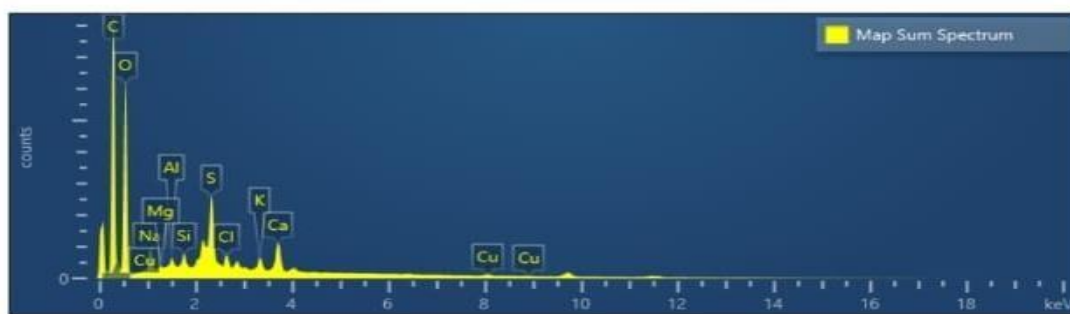


FIG 5.9: EDX(energy dispersive x-ray analysis) SPECTRA OF SEAM WEED

TABLE 5.1 EDAX (Energy dispersive X ray Analysis) before adsorption

Element	Line Type	Weight %	Weight Sigma	%	atomic %
C	k series	56.58	0.38		65.31
O	k series	36.73	0.34		31.83
Na	k series	1.14	0.04		0.69
Mg	k series	0.08	0.02		0.04
Al	k Series	0.25	0.02		0.13
Si	k series	0.36	0.02		0.18
S	k series	1.89	0.04		0.82
Cl	k series	0.55	0.02		0.22
K	k series	0.55	0.02		0.2
Ca	k series	1.44	0.03		0.5
Cu	k series	0.43	0.06		0.09
Total		100			100

The EDAX mapping shows the composition of the adsorbent sample. The carbon and oxygen peaks obtained are due to the large amounts of C-H, C-O and O-H bonds present in Siam weed. The presence of sodium and sulfur is due to the addition of sodium bicarbonate and sulfuric acid during the activation process.

5.5 OPTIMIZATION PARAMETERS

5.5.1 Contact time

The collected samples are tested with UV spectrophotometer, and the removal percentage of the adsorbent can be found by calculating the absorbance value obtained

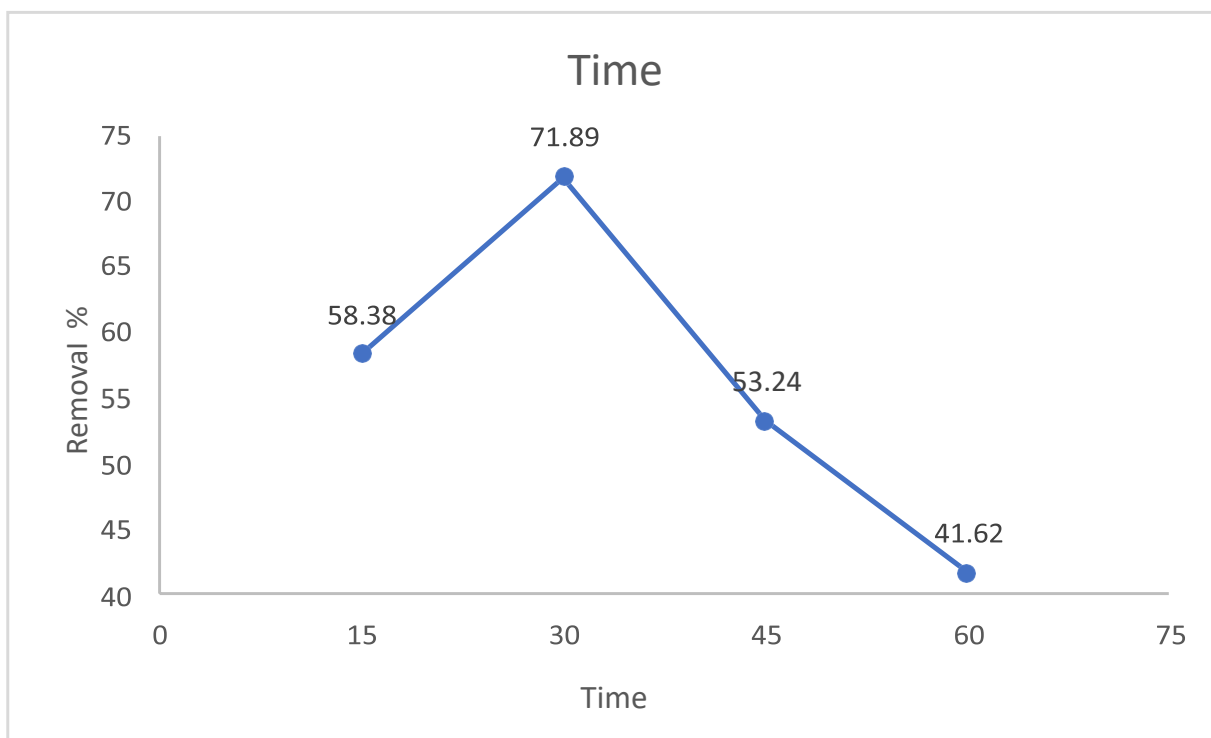


FIG 5.10: OPTIMIZATION OF TIME

From the data present, it can be concluded that the optimum contact time for the maximum removal of dye particles is approximately 15 minutes. One possible explanation for this observation is that after 15 minutes, most of the active sites of the adsorbent are likely occupied by dye molecules, leading to saturation of the adsorption capacity. Additionally, it is plausible that after 15 minutes, the rate of desorption becomes slightly higher than the rate of adsorption. This means that while some dye molecules continue to be adsorbed onto the active sites of the adsorbent, there is also a concurrent process of dye molecules being released from the adsorbent

surface back into the solution. This equilibrium between adsorption and desorption processes could result in a plateau in the removal efficiency observed around 15 minutes. Therefore, the observed optimal contact time of 15 minutes likely represents a balance between adsorption and desorption kinetics, where the adsorption capacity of the adsorbent is maximized before desorption processes start to dominate.

5.5.2 Temperature

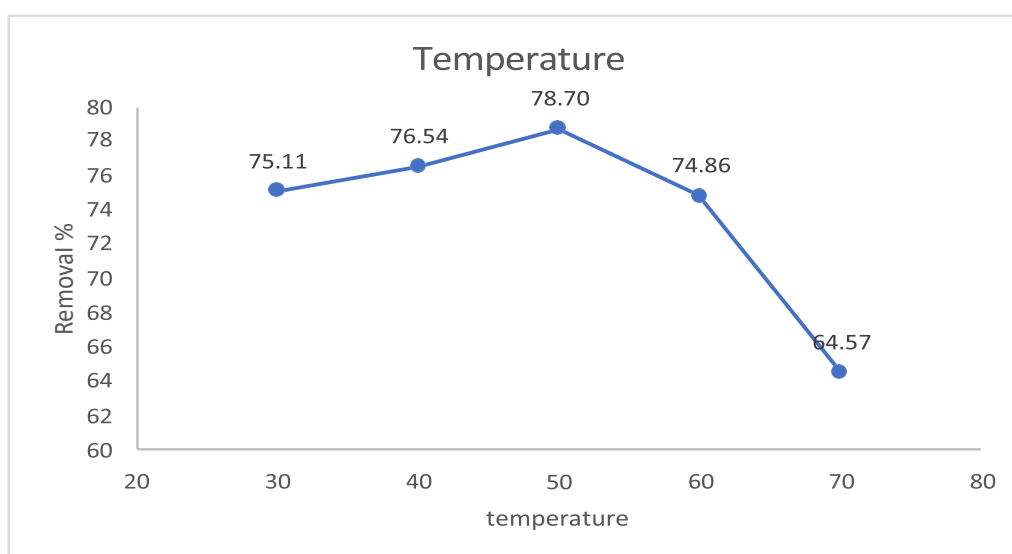


FIG 5.11: OPTIMIZATION OF TEMPERATURE

The removal of adsorption was studied by varying the temperature (30,40,50,60,70) while keeping the dosage initial concentration and contact time 15 mins. Removal was highest when temperature was 50 degree (78.70) so 50 degree was chosen as the optimum temperature

5.5.3 CONCENTRATION

UV spectrophotometer is used to evaluate the collected samples, and the absorbance value obtained can be used to calculate the removal percentage of the adsorbent.

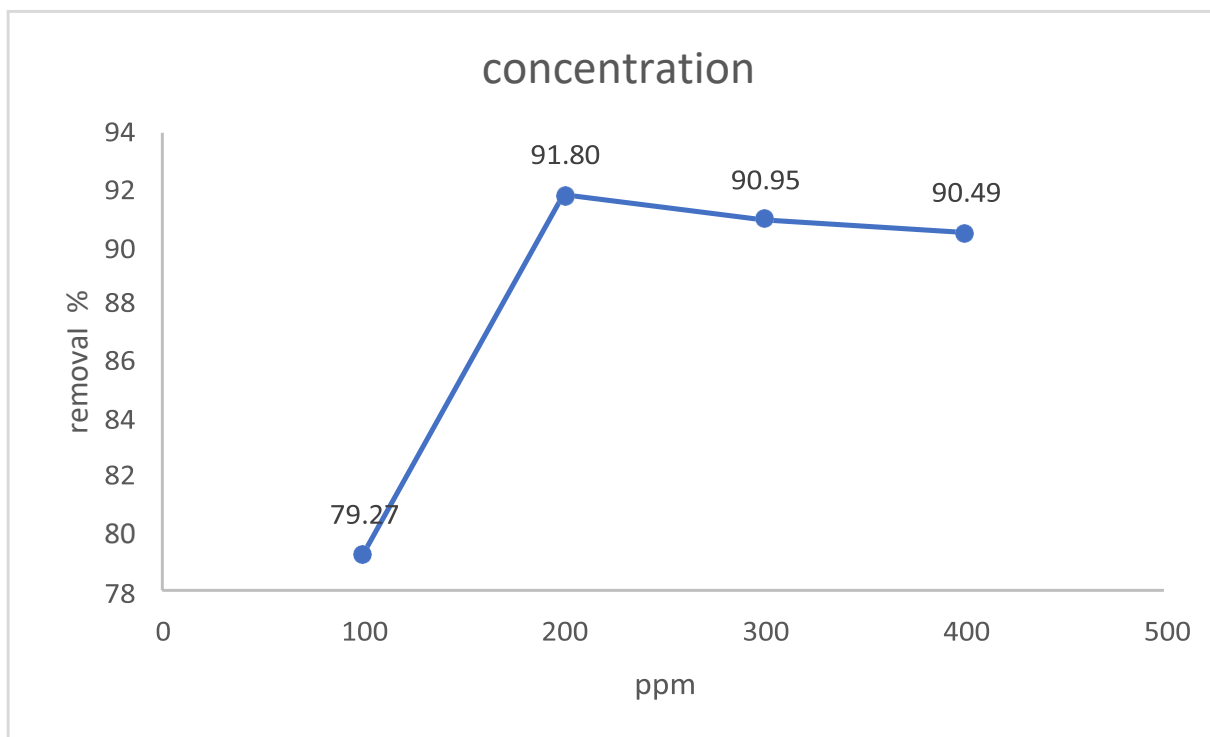


FIG 5.12: OPTIMIZATION OF CONCENTRATION

Based on the study where the removal of crystal violet dye was investigated by varying the initial concentration (100, 200, 300, 400, 500 ppm) while keeping the adsorbent dosage constant at 0.2 g and a contact time of 15 minutes, it can be concluded that the adsorption of 20 ppm is the most efficient for the maximum removal of dye particles. This observation aligns with the principle that at lower concentrations of the dye solution, there are numerous accessible adsorption sites available on the surface of the adsorbent. As a result, more dye molecules have the opportunity to interact with and be adsorbed onto these accessible sites, leading to a higher adsorption rate.

5.5.4 ADSORBENT DOSAGE

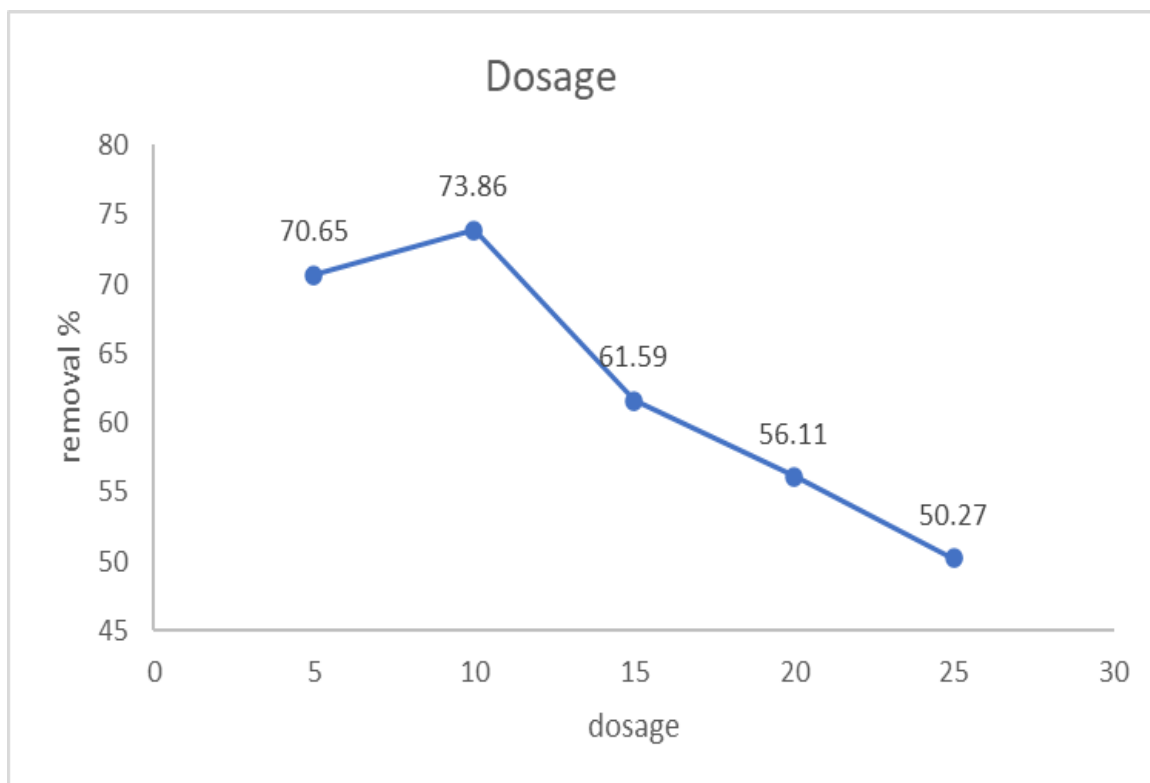


FIG 5.13: ADSORBENT DOSAGE

The collected samples are tested with UV spectrophotometer, and the removal percentage of the adsorbent can be found by calculating the absorbance value obtained. The data suggests that the optimum dosage for the maximum removal of dye particles is at 20 g/L of adsorbent dosage. One possible explanation for this finding is related to the availability of adsorption sites on the surface of the adsorbent material. At low dosages of the adsorbent, there are plenty of available adsorption sites on the surface. As more adsorbate (dye particles) is introduced into the system, these sites become progressively filled. This leads to an increase in the adsorption rate as more dye particles are captured by the available sites. However, as the dosage of the adsorbent continues to increase, the available sites become gradually saturated. Once the maximum capacity of the adsorbent is reached, further increases in dosage lead to fewer additional adsorption sites being available for dye molecules. Consequently, the adsorption rate starts to decrease because there are fewer sites available for additional adsorption, despite the higher dosage of the adsorbent.

5.6 KINETIC AND ISOTHERM STUDY

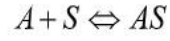
The study investigated the kinetic mechanism involved in the adsorption process of Fuchsin dye with AASCO adsorbent using intraparticle diffusion, also known as the Weber and Morris kinetic model. Additionally, three kinetic models were employed: pseudo-first-order kinetic equation, pseudo-second-order kinetic equation, and the Elovich model. For interpretation of the adsorption data, four isotherm models were utilized: Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich. These models were employed to understand the equilibrium adsorption behaviour. The kinetic model that best fitted the adsorption operation was utilized alongside Arrhenius and Eyring equations to calculate various thermodynamic parameters such as activation energy, change in Gibbs free energy, change in enthalpy, and change in entropy. These parameters provide insights into the energetics and spontaneity of the adsorption process. Furthermore, the isotherm model that best fitted the adsorption data was employed in designing a single-stage batch adsorber. This design aspect is crucial for scaling up the process for practical applications.

5.7 ADSORPTION KINETICS

5.7.1 PSEUDO FIRST ORDER

TABLE 5.2: PSEUDO FIRST ORDER KINETICS

ppm	R^2	K	q _e
400	0.975	0.045	488.8228
300	0.932	0.037	292.6566
200	0.905	0.054	604.2572
100	0.895	0.036	264.542



$$\frac{q_t}{q_e} = 1 - \exp\left[-\left(k_A C_S + \frac{k_A}{k_S}\right)t\right]$$

$$\log(q_e - q_t) = \log q_e - \frac{k_{ad}}{2.303}t$$

$$k_{ad} = \left[\left(k_A C_S + \frac{k_A}{k_S} \right) \right]$$

$$q_t = X_{At} \quad \text{and} \quad q_e = X_{Ae}$$

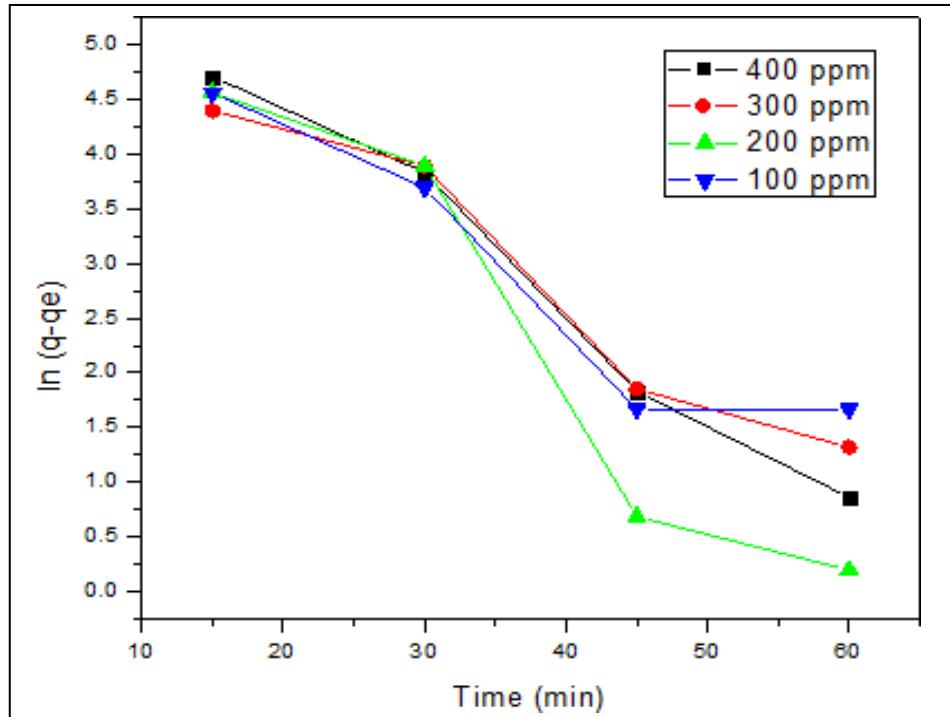


FIG 5.14: PSEUDO FIRST ORDER KINETICS

5.7.2 PSEUDO SECOND ORDER

TABLE 5.3: PSEUDO SECOND ORDER

	400 ppm	300 ppm	200 ppm	100 ppm
R2	0.999	0.999	0.999	0.999
k2	0.002	0.002	0.001	0.001
qe	500	500	1000	1000
h	500	500	1000	1000

$$\frac{dq_t}{dt} = k(q_e - q_t)^2$$

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_q} + kt$$

$$\frac{t}{q_t} = \frac{1}{h} + \frac{1}{q_e} t$$

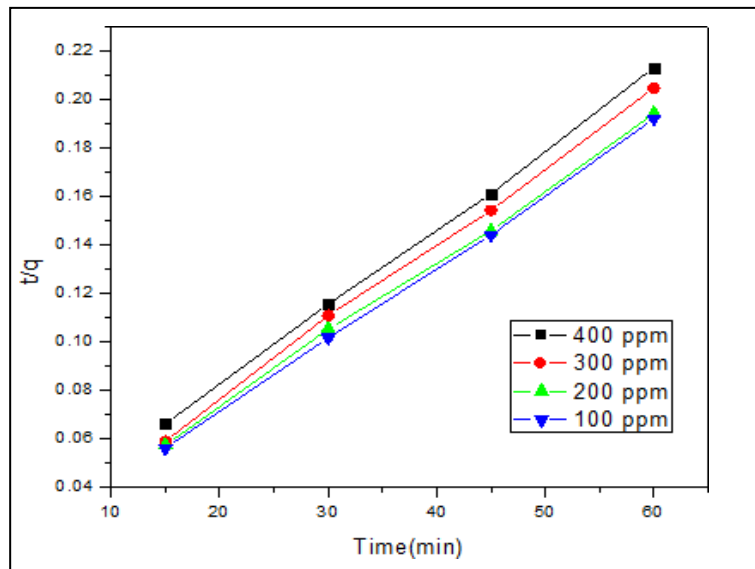


FIG 5.15: PSEUDO SECOND ORDER KINETICS

5.7.3 WEBER MORRIS

TABLE 5.4: WEBER MORRIS KINETIC

	R2	kid	C
1500 ppm	0.866	380.2	16.5
1200 ppm	0.939	439.1	13.56
900 ppm	0.875	453.1	14.86
600 ppm	0.92	461.6	15.43

$$q_t = K_{id}t^{0.5} + C$$

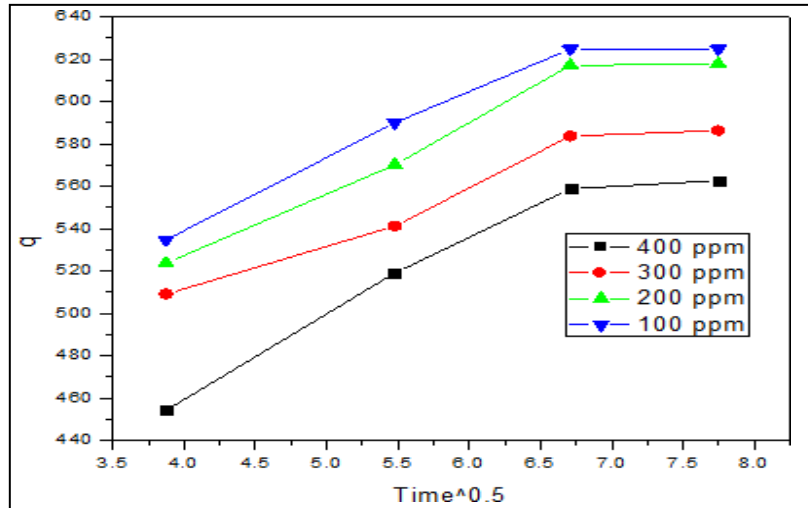


FIG 5.16: WEBER MORRIS KINETICS

In conclusion, the adsorption process under investigation predominantly adheres to the kinetics of pseudo-first order rather than pseudo-second order. This determination is supported by the superior fit of experimental data to the pseudo-first order model, as evidenced by the highest R-squared (R2) value attained. The observed adsorption behaviour suggests that the rate-limiting step in the adsorption process likely involves the surface interaction between the adsorbate and the adsorbent, a characteristic commonly associated with pseudo-first order kinetics. As such, this finding underscores the importance of considering kinetic models in understanding and optimizing adsorption processes for practical applications.

5.8 ADSORPTION ISOTHERM MODELS

5.8.1 LANGUMIUR ISOTHERM

TABLE 5.5 LANGUMIUR ISOTHERM

R^2	0.93
Qm	0.104932
KL	0.020672

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

$$R_L = \frac{1}{1 + K_L C_0}$$

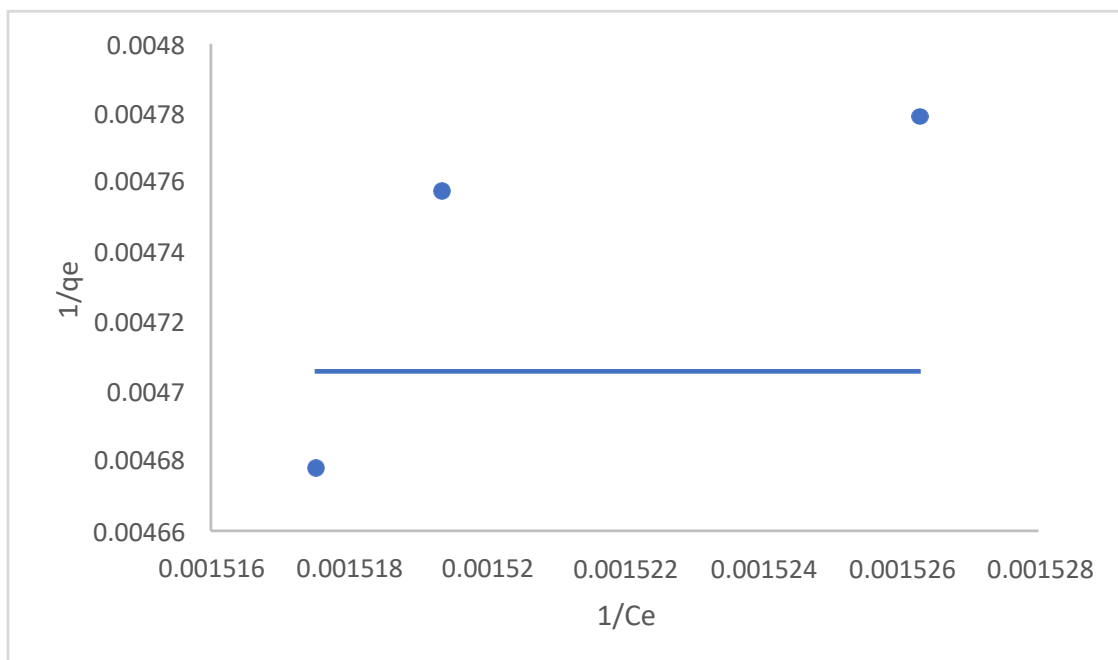


FIG 5.17: LANGUMUIR ISOTHERM

5.8.2 FREUNDLINCH ISOTHERM

TABLE 5.6: FREUNDLINCH ISOTHERM

R^2	0.996
kf	57.16833
n	2.380952

$$q_e = K_F C_e^{1/n}$$

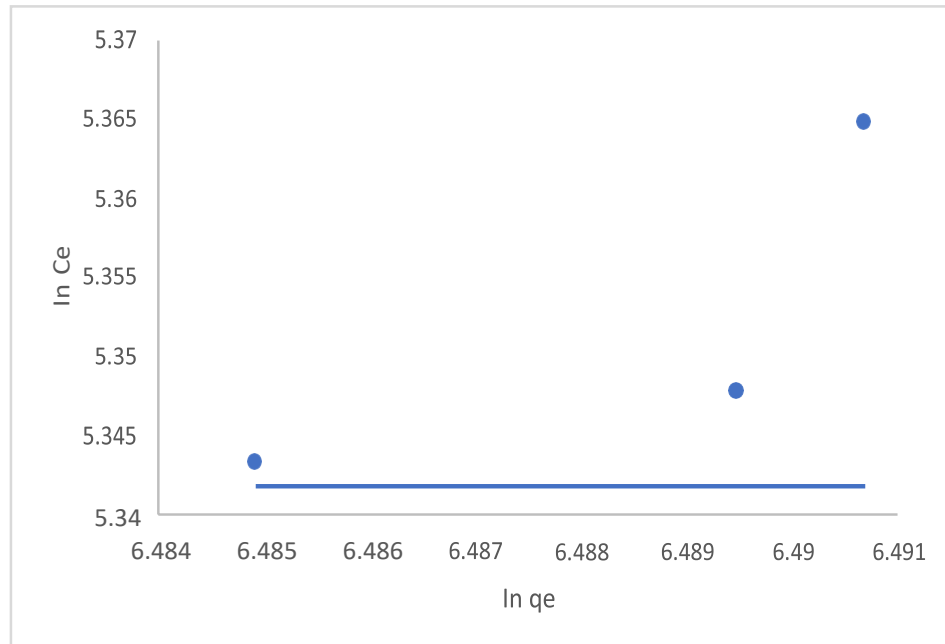


FIG 5.18: FREUNDLINCH ISOTHERM

5.8.3 TEMKIN ISOTHERM

TABLE 5.7: TEMKIN ISOTHERM

R^2	0.966
A	0.0397
B1	-250

$$q_e = B \ln (A C_e)$$

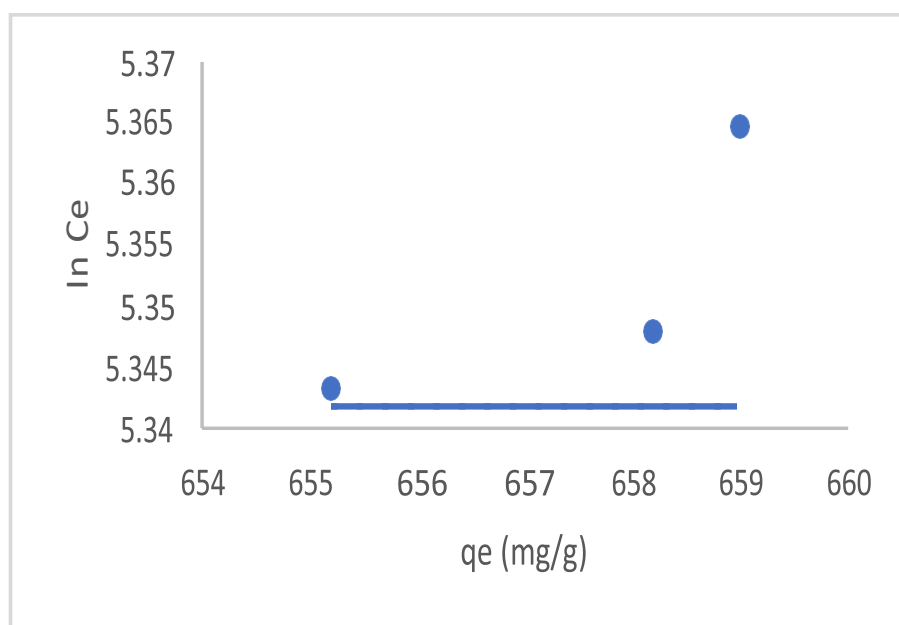


FIG 5.19: TEMPKIN ISOTHERM

5.8.4 DUBININ RADUSHKEVICH

TABLE 5.8: DUBININ RADUSHKEVICH

R^2	0.938
β	9.26
qmD	3.18E+28

$$\ln q_e = \ln q_{mD} - \beta \left[RT \left(1 + \frac{1}{C_e} \right) \right]^2$$

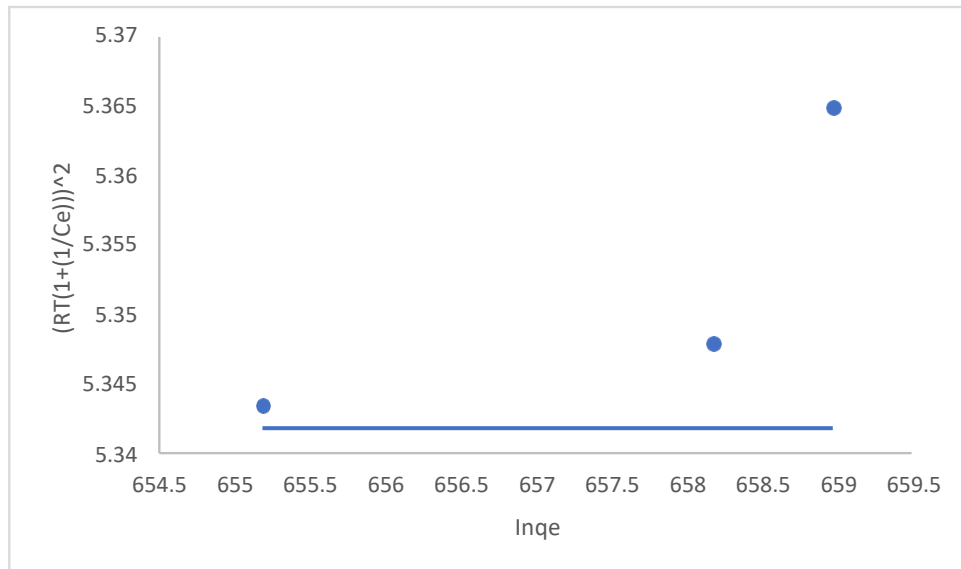


FIG 5.19: DUBININ RADUSHKEVICH (FOR ADSORPTION)

Based on the experimental data and analysis, it can be concluded that the adsorption process follows the Freundlich isotherm model most closely among the Langmuir, Freundlich, and Temkin isotherms. This conclusion is supported by the highest R² value obtained for the Freundlich isotherm model. The Freundlich model describes multilayer adsorption onto heterogeneous surfaces, which suggests that the adsorbate molecules exhibit non-ideal behaviour and form a heterogeneous distribution of adsorption sites on the adsorbent surface. into the adsorption mechanism and surface properties of the adsorbent under investigation.

CHAPTER – 6

CONCLUSION AND SUMMARY

The present adsorption study has shown the feasibility of Fuchsine dye adsorption on the AASCO adsorbent. The batch adsorption experiment data was used in the study of adsorption kinetics and isotherm. In conclusion, the adsorption of Fuchsine dye demonstrates a promising method for wastewater treatment or dye removal processes. The efficient adsorption capacity observed in this study suggests the potential application of the adsorbent material in real-world scenarios, contributing to environmental remediation efforts. Further research and optimization are recommended to enhance the efficacy and scalability of this adsorption process. The adsorption-desorption reusability study indicated that the AASCO adsorbent could be reused for 3 cycles with higher Fuchsine dye removal efficiency. This study primarily suggests that the adsorption operation of CV dye molecules by AASCO adsorbent is a thermodynamically feasible process. A scale-up of the adsorption operation was theoretically investigated by designing a single-stage adsorber. Adsorption of Fuchsine dye involves the binding of the dye molecules to a solid surface, typically through physical or chemical interactions. This process is often used in water treatment to remove dye pollutants. The efficiency of adsorption depends on factors like surface area, material characteristics, and dye concentration. Studies focus on optimizing conditions for maximum adsorption capacity and understanding the mechanisms involved, which can include electrostatic interactions, Van der Waals forces, or chemical bonding. Overall, the goal is to develop effective and sustainable methods for removing crystal violet dye from aqueous solutions.

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LIST OF ABBREVIATIONS

1. ppm: Parts Per Million
2. UV-Vis: Ultraviolet-Visible
3. FTIR: Fourier Transformation Infrared Spectroscopy
4. SEM: Scanning Electron Microscopy
5. EDAX: Energy Dispersive X-ray Analysis
6. XRD: X-ray Diffraction
7. FESEM: Field Emission Scanning Electron Microscopy
8. EDS: Ehlers- Danlos syndrome

CHAPTER - 1 INTRODUCTION

1.1 BACKGROUND.

Water is an important component as all the living organisms on earth need water for their daily vital activities. With the growth of population and urbanization, the need for water gets intensified. However, the various man-made activities allow the pollutants like dye molecules to make their way into natural water bodies, rendering the water dangerous for consumption. The polluted water bodies deeply affect the health of humans and many other living organisms consuming it. Water is an important component as all the living organisms on earth need water for their daily vital activities. With the growth of population and urbanization, the need for water gets intensified. However, the various man-made activities allow the pollutants like dye molecules to make their way into natural water bodies, rendering the water dangerous for consumption. The polluted water bodies deeply affect the health of humans and many other living organisms consuming it. Thus, it becomes essential to treat water in a large quantity, with the fast and straightforward process to make it safe for consumption of not only humans but also of the other living organism living among us.

Fuchsine (sometimes spelled fuchsin) or rosaniline hydrochloride is a magenta dye with chemical formula $C_{20}H_{19}N_3 \cdot HCl$. It becomes magenta when dissolved in water; as a solid, it forms dark green crystals.

Basic fuchsin's effects are multifaceted, with both advantageous and disadvantageous aspects depending on the context. Let's delve deeper into each area:

Positive Effects:

- **Specificity:** Techniques can be tailored to target specific molecules. For instance, the Ziehl-Neelsen stain cleverly exploits the unique cell wall composition of Mycobacteria, allowing for specific diagnosis of tuberculosis and

leprosy.

- **Visualization Powerhouse:** It highlights vital cellular structures like the nucleus (DNA-rich) and cytoplasm (ribosome-rich), enabling researchers to study cell function, identify abnormalities, and understand cell cycle progression with the Feulgen stain.
- **Versatility in Microscopy:** Beyond routine staining, basic fuchsin can be employed in specialized techniques like the PAS stain to pinpoint carbohydrates in cell walls, fungi, and basement membranes.
- **Textile Dyeing:** Basic fuchsin imparts a beautiful red or pink color to textiles like cotton and wool. However, its susceptibility to fading under sunlight limits its widespread use in commercial textiles where colorfastness is paramount.

Other Applications:

- **pH Indicator:** Basic fuchsin acts as a readily available colorimetric indicator. Its color shift from red (acidic) to violet/blue (basic) provides a simple way to assess solution acidity/alkalinity.
- **Culture Media Differentiation:** By incorporating basic fuchsin into specific culture media, scientists can differentiate between bacterial strains based on their sugar fermentation abilities. Fermenting bacteria alter the media's pH, causing a color change due to basic fuchsin's pH sensitivity.
- **Mucin Staining:** This dye's ability to target mucins proves valuable in studying these essential components of mucus and other secretions, aiding in research on respiratory and gastrointestinal health.

Negative Effects:

- **Skin Irritation:** Direct contact with basic fuchsin can cause skin irritation or allergic reactions.
- **Eye Irritation/Damage:** Contact with eyes can lead to irritation or even damage the delicate ocular tissues.
- **Environmental Impact:** Improper disposal of basic fuchsin poses a threat to water sources. Since biodegradation can be slow, the dye may persist in the environment, potentially impacting aquatic life.

Additional Considerations:

- **Light Sensitivity:** As mentioned earlier, basic fuchsin's susceptibility to fading under sunlight restricts its use in applications requiring colorfastness.
- **Synthetic Dye:** Being a synthetic dye, concerns regarding potential long-term health effects linger. While research is ongoing, it's essential to handle basic fuchsin with proper safety measures.

In conclusion, basic fuchsin's effects present a balancing act. It offers significant benefits in various fields, particularly biological staining and diagnostics. However, responsible handling practices are crucial to minimize potential health and environmental risks.

The adsorption of Fuchsine dye plays a crucial role across a spectrum of applications, ranging from wastewater treatment to analytical chemistry, where the binding of crystal violet molecules onto surfaces, predominantly solid materials like activated carbon or other adsorbents, is pivotal. This process serves as the linchpin in removing crystal violet from solutions, making it imperative to comprehend thoroughly to optimize removal techniques and boost efficiency.

Understanding the intricacies of fuchsine adsorption entails delving into a myriad of factors, each exerting a significant influence on the process. Among these factors, pH stands out as a key determinant, as variations in pH levels can profoundly affect the surface charge of both the dye molecules and the adsorbent material, consequently altering their interaction and adsorption capacity. Similarly, temperature plays a critical role, influencing the kinetic energy of the system and thus impacting the rate and extent of adsorption.

Moreover, the properties of the adsorbent material itself, such as surface area, pore size distribution, and surface chemistry, significantly influence Fuchsine adsorption. For instance, materials with higher surface areas and tailored surface chemistries often exhibit enhanced adsorption capacities due to increased active sites available for interaction with dye molecules.

Beyond these factors, the presence of other ions or organic matter in the solution can also influence crystal violet adsorption by competing for adsorption sites or forming complexes with the dye molecules, thereby altering their availability for adsorption. By unraveling the complex interplay of these factors, researchers can gain valuable insights into the mechanisms governing crystal violet adsorption, paving the way for the development of more efficient and sustainable removal techniques. This exploration not only enhances our understanding of dye removal processes but also holds promise for addressing pressing environmental challenges associated with dye pollution. Thus, this introduction serves as a springboard for a deeper dive into the nuanced dynamics of crystal violet adsorption, highlighting its significance and potential implications across various scientific and environmental domains.

The biomass employed for the adsorption operation in this study was sourced from *Chromolaena odorata*, a local invasive plant commonly known as Siam Weed. Originally indigenous to South America, *C. odorata* has successfully spread to various regions of India. This plant species possesses distinct growth characteristics that contribute to its invasive nature. Despite having a woody base, *C. odorata* exhibits a flexible and soft stem structure, allowing it to thrive as a creeper, often sprawling over other vegetation and reaching impressive heights of up to 10 meters in favorable conditions. Notably, each plant produces a prolific number of seeds, estimated to range between 80,000 to 90,000 per individual, equipped with mechanisms for attachment to various surfaces like cloth, fur, and machinery, facilitating its widespread dispersal over long distances.

Another remarkable aspect of *C. odorata*'s biology is its rapid regenerative ability. Capable of sprouting from its roots and exhibiting vigorous growth rates of up to 3 centimeters per day under optimal environmental conditions, this plant demonstrates resilience and adaptability in diverse habitats. Moreover, the leaves of *C. odorata* are valued for their medicinal properties, particularly for their antimicrobial qualities, making them traditionally sought-after for healing wounds and other ailments. By harnessing the biomass derived from *C. odorata* for the adsorption process, this study not only addresses environmental concerns associated with invasive species but also capitalizes on the unique properties of this plant for sustainable applications in wastewater treatment and other environmental remediation efforts.

In the present study, we embarked on a novel endeavor by utilizing the stem of *Chromolaena odorata*, an invasive plant notorious for its disruptive effects on local vegetation, for the adsorption of CV dye. To the best of our knowledge, this represents the inaugural application of *C. odorata* biomass in such adsorption operations, marking a significant breakthrough in the field. Our approach commenced with a systematic exploration, initially employing a one-variable-at-a-time methodology to conduct adsorption studies. Subsequently, we adopted a more sophisticated statistical approach, namely response surface methodology (RSM), to systematically investigate the optimal conditions of various variables aimed at maximizing dye removal percentage.

Furthermore, our investigation extended to assessing the reusability of the adsorbent, a critical aspect for evaluating its long-term efficacy and sustainability in practical applications. Through a series of experiments, we aimed to ascertain the feasibility of utilizing the *C. odorata* stem biomass as a reusable adsorbent for prolonged and efficient dye removal from aqueous solutions.

In addition to these fundamental studies, we designed and implemented a single-stage batch adsorber, envisioning its potential for large-scale dye removal operations. This innovative design not only demonstrates the scalability of our approach but also lays the groundwork for the practical implementation of our findings in real-world settings.

By repurposing biomass from an invasive plant species like *C. odorata* for dye adsorption, our research not only addresses environmental concerns associated with invasive flora but also presents a sustainable solution for wastewater treatment. Through this interdisciplinary endeavor, we contribute to both environmental conservation efforts and the advancement of efficient water treatment technologies, paving the way for a more sustainable future.

CHAPTER - 2

LITERATURE SURVEY

Chauhan P, Bhasin KK, Chaudhary S (2021) High selectivity and adsorption proficiency of surfactant-coated selenium nanoparticles for dye removal application. The rate of environmental pollution augmenting at an alarming rate due to the continuous disposal of toxic dyes directly into the environment and water streams. The direct contact of dyes with water resources directly affects the living beings. The identification of superior methods for the treatment of water pollution caused due to effluent dyes needs higher consideration among researchers for the well-being of living flora and fauna. The available methods for controlling the decontamination of water through toxic dyes have various drawbacks. So, it is highly significant to develop such materials which can easily adsorb the dyes without causing any toxic effect on the environment and living beings. While keeping all the facts in mind, the current work highlights the comparative enhancement in adsorption capacity and selectivity of Brij-58-coated selenium nanoparticles (Brij-58@Se NPs) towards the removal of bromophenol blue (BB) dye from series of chosen dyes in aqueous media. The fabricated Se NPs were methodically characterized and the adsorption behaviour displayed fast adsorption efficiency (98% within 6 min) for BB dye out of series of chosen dyes. The efficacy of the adsorbent was further tested in six different water resources and displayed 95% adsorption efficiency for BB dye in different wastewater samples. Therefore, Brij-58@Se NP is expected as a potential adsorbent for the adsorption of organic dyes from wastewater samples.

Anyanwu S, Inyang IJ, Asemota EA, Obioma OO, Okpokam DC, Agu VO (2017) Effect of ethanolic extract of *Chromolaena odorata* on the kidneys and intestines of healthy albino rats. The use of plants and plant products for medicinal purposes is an age-long practice in traditional communities and is becoming prominent globally. This study was performed to evaluate the effect of ethanolic extract of *Chromolaena odorata* on the kidney and intestine of albino rats.

CHAPTER – 3

AIM AND SCOPE

3.1 AIM

- To prepare adsorbent from Siam weed leaves and stem.
- To characterize the adsorbent
- Batch studies in adsorber
- Use of batch studies in waste water treatment process
- Optimization studies in batch adsorbent

3.2 OBJECTIVE

1. Determining the adsorption capacity: Batch adsorption studies aim to quantify the amount of adsorbate (such as dyes, heavy metals, or organic compounds) that can be removed by a specific adsorbent material under controlled conditions. This helps in understanding the maximum adsorption capacity of the material and its efficiency in removing pollutants from a solution.

2. Evaluating adsorption kinetics: Batch studies help in analyzing the rate at which adsorption occurs over time. By monitoring the adsorption process at different time intervals, researchers can determine the kinetics of adsorption, including the initial adsorption rate, equilibrium time, and overall adsorption efficiency.

3. Assessing equilibrium behavior: Batch adsorption studies aim to reach equilibrium between the adsorbate and adsorbent, where the rate of adsorption equals the rate of desorption. By studying the equilibrium behavior, researchers can determine the optimal contact time required for maximum adsorption efficiency.

4. Optimizing process parameters: Batch studies allow for the investigation of various process parameters, such as initial concentration of the adsorbate, pH of the solution, temperature, and adsorbent dosage. By systematically varying these parameters, researchers can optimize the conditions for achieving the highest adsorption efficiency.

5. Studying adsorption isotherms: Batch adsorption studies help in constructing adsorption isotherms, which describe the relationship between the amount of adsorbate adsorbed onto the adsorbent surface at equilibrium and the concentration of the adsorbate in the solution. Understanding the adsorption isotherms can provide insights into the adsorption mechanism and surface properties of the adsorbent material.

Overall, batch adsorption studies play a crucial role in characterizing the adsorption behavior of different materials and optimizing the adsorption process for various environmental and industrial applications.

3.3 SCOPE

Exploration of Agro Waste Biomass: Batch studies offer the opportunity to explore different types of agro waste biomass, such as rice husk, coconut shell, sawdust, and others, as potential adsorbents for fuchsin dye removal. This involves assessing the availability, cost-effectiveness, and adsorption capacity of these materials.

Optimization of Adsorption Process: Batch studies allow for the optimization of the adsorption process by investigating the influence of parameters like pH, initial dye concentration, contact time, adsorbent dosage, and temperature. This optimization ensures efficient dye removal and helps in determining the most favorable conditions for adsorption.

Kinetics and Thermodynamics Studies: Batch studies enable the evaluation of adsorption kinetics and thermodynamics to understand the mechanisms involved in the dye removal process. This includes determining the rate of adsorption, equilibrium adsorption capacity, and the effect of temperature on adsorption efficiency.

Characterization of Adsorbent Material: Batch studies involve the characterization of agro waste biomass before and after the adsorption process using techniques such as FTIR, SEM, and XRD. This characterization helps in identifying any structural changes in the adsorbent material and confirming the adsorption mechanism.

Reusability and Sustainability: Batch studies assess the reusability of agro waste biomass as adsorbents for multiple cycles, evaluating their sustainability and economic viability. This involves studying the regeneration process and determining any loss of adsorption capacity over successive cycles.

Development of Adsorption Models: Batch studies contribute to the development of adsorption models based on isotherm equations (e.g., Langmuir or Freundlich) to predict adsorption behavior and design practical batch adsorption systems for large-scale applications.

Application in Wastewater Treatment: Ultimately, the scope of batch studies extends to the practical application of agro waste biomass in wastewater treatment for the removal of fuchsine dye, addressing environmental concerns and contributing to sustainable solutions.

In summary, batch studies in the removal of fuchsine dye using agro waste biomass offer a comprehensive exploration of adsorption processes, from material selection and optimization to characterization, reusability, and application in wastewater treatment.

CHAPTER 4

MATERIALS AND METHODOLOG

4.1 MATERIALS REQUIRED

The following materials were used for the synthesis Chromolaena :

- Chromolaena(Siam weed)
- Hot air oven
- sulfuric acid (H_2SO_4)
- Magnetic pellet.
- water
- Sodium Bicarbonate(NaHCO_3)
- 1-liter beakers
- Filter papers
- Stirrer

4.2 STOCK SOLUTION PREPARATION

To prepare a 1000 mL solution containing 1000 ppm of crystal violet dye, the process began with accurately weighing 1 gram of crystal violet dye using a digital balance. The dye was then dissolved in a small volume of an appropriate solvent within a clean 1000 mL volumetric flask, ensuring complete dissolution. Subsequently, the flask was filled to the mark with the solvent, and thorough mixing was conducted to achieve uniformity. The concentration was then verified and adjusted if necessary. Finally, the flask was labeled with the solution details and stored appropriately. This meticulous procedure guaranteed the creation of a precise solution suitable for various applications.

4.3 PREPARATION OF ADSORBENT

1. Drying:

- The stems and leaves of *Chromolaena odorata* are spread out in a thin layer and exposed to sunlight.
- Sunlight helps to evaporate moisture from the plant material, reducing its water content. This step is crucial because excessive moisture can affect subsequent processing steps and the quality of the final product.

2. Grinding and Sieving:

- Once the plant material is adequately dried, it is ground into a fine powder using a grinder or mill.
- After grinding, the powdered material is sieved to achieve a uniform particle size. Sieving helps to remove any larger particles and ensures consistency in the size of the final product.
- The target particle size range of 75 to 100 μm is chosen based on the specific requirements of the intended application, such as maximizing surface area for adsorption.

3. Activation with Acid:

- In this step, a portion of the powdered *Chromolaena odorata* sample is mixed with a specific volume (500 mL) of 0.1 N H_2SO_4 (sulfuric acid) solution.
- The mixture is stirred for 18 hours. This duration allows the acid to interact with the plant material, potentially breaking down organic components and creating surface functional groups that can enhance adsorption properties.
- Acid activation is a common technique used to modify the surface chemistry and increase the porosity of adsorbent materials, improving their capacity to adsorb target substances from solution.

4. Neutralization:

- After the acid activation step, the material obtained is soaked or rinsed in a solution of 0.1 N NaHCO_3 (sodium bicarbonate).
- Sodium bicarbonate is a mild base and is used to neutralize any residual acid present in the material. This step helps to prevent the acid from interfering with subsequent processes or applications and ensures the material is safe to handle.
- Neutralization may also help to stabilize the pH of the material, making it more suitable for certain adsorption applications.

5. Drying:

- Following neutralization, the material is dried to remove excess moisture and prepare it for storage or further processing.
- Drying is typically carried out using an oven set to a specific temperature (353 K or approximately 80°C in this case) for a defined duration (6 hours).
- Proper drying helps to prevent microbial growth, maintain the stability of the material, and ensure consistent performance in subsequent application.

4.4 METHODOLOGY

Adsorbent Preparation

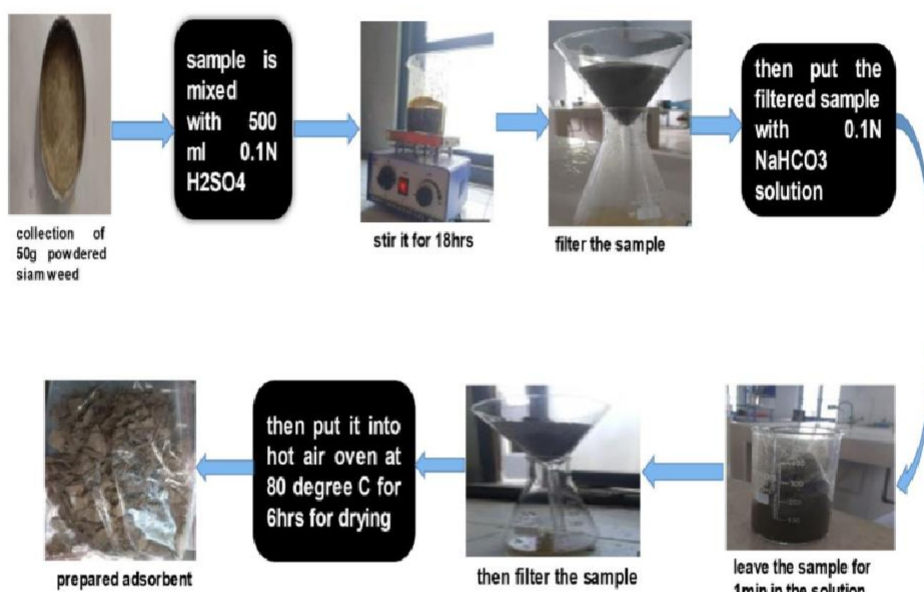


FIG 4.1 PREPARATION OF ADSORPTION

CHAPTER – 5 RESULTS AND DISCUSSION

5.1 CHARACTERISATION

FTIR(Fourier Transform Infrared)

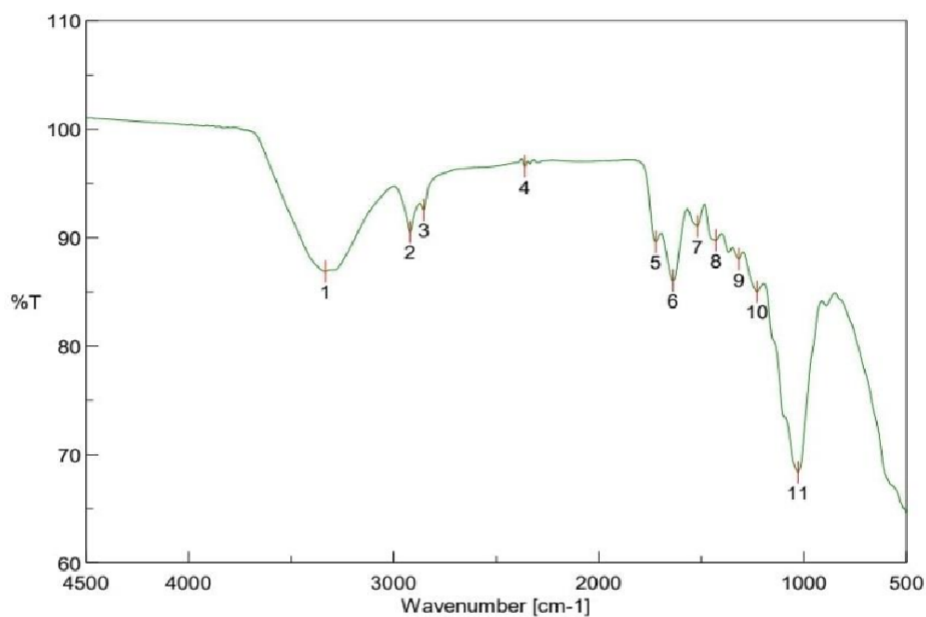


Fig .5.1 FTIR Spectroscopy results for synthesis of Chromolaena

Wavenumbers around 3300 cm^{-1} indicate an O-H stretch, suggesting the presence of alcohols. C-H stretches are observed between 2800-3000 cm^{-1} , shows the presence of alkanes. The C=O stretch around 1723 cm^{-1} points towards ketones. Similarly, the peak at 1519 cm^{-1} corresponds to a C=C stretch, indicating alkenes or aromatics.

XRD (X-Ray diffraction).

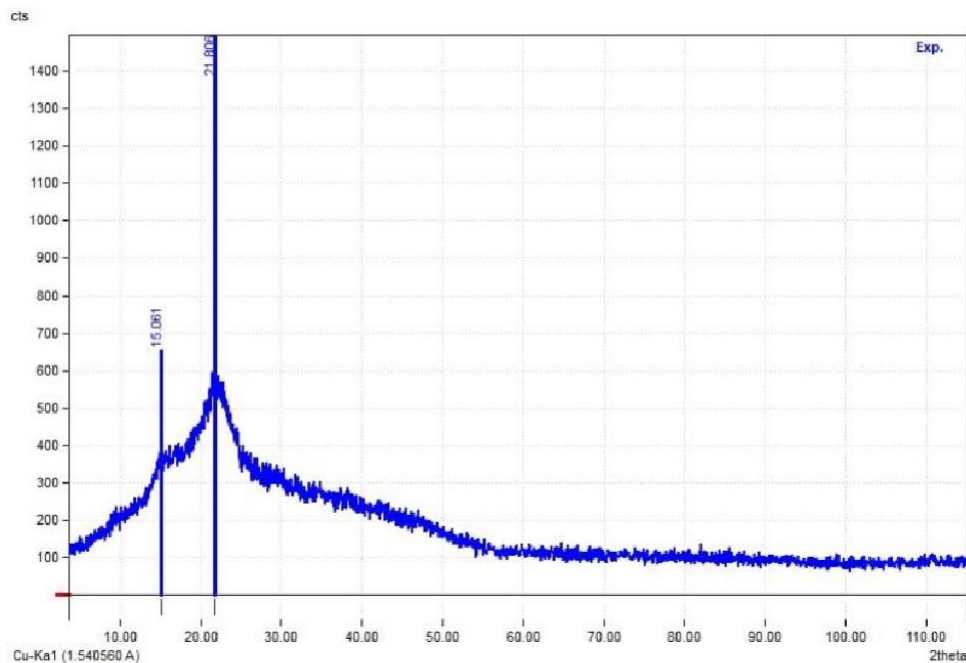


Fig .5.2 X-Ray diffraction spectroscopy

- XRD spectrum is indicated in Fig. 5.1.2, which shows a well-defined and intense peak at 2 (degree of 21.956), indicating the presence of crystalline and well-defined structures present in *C. odorata* biomass.
- Furthermore, the intensity of the well-defined peak does not reduce after the biomass is activated with acid or after the adsorption operation.
- Therefore, Fig. 5.1.2 indicates that the adsorption of fuchsine dye on AASCO will probably take place on the upper part of the crystalline surface

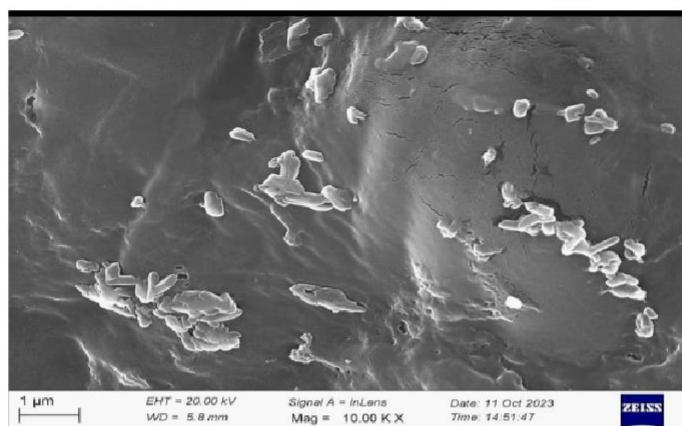


Fig 5.3 SEM Image under 10.00 KX magnification (before adsorption)

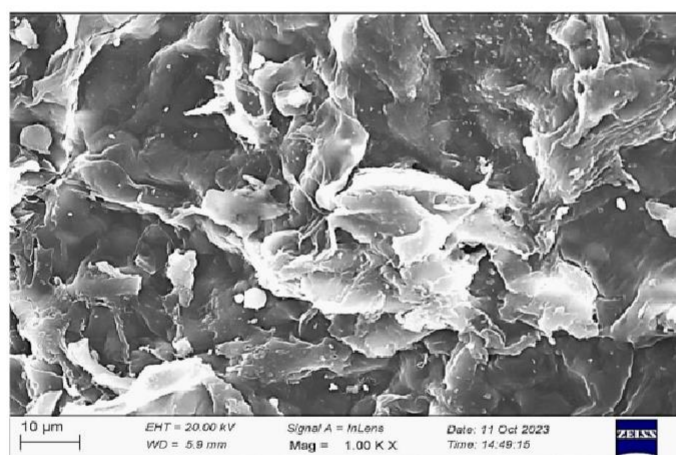


Fig.5.4 SEM Image under 1.00 KX magnification (before adsorption)

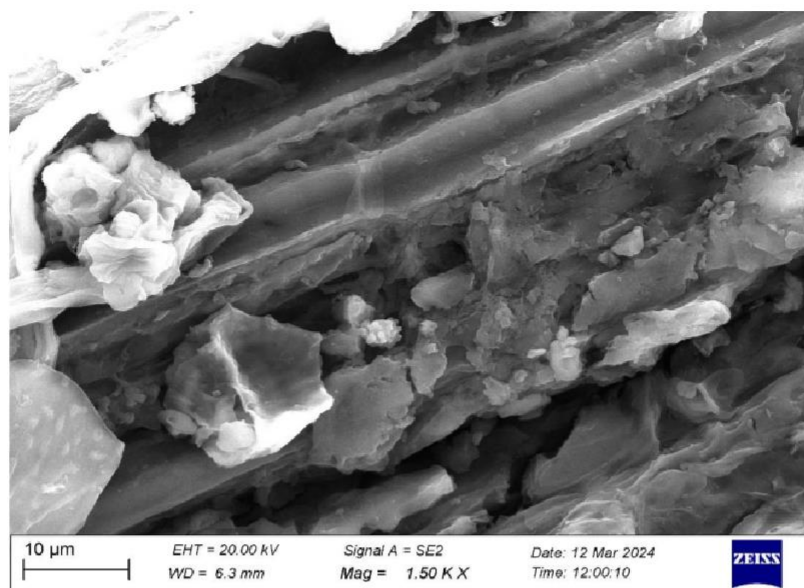


Fig 5.5 SEM Image under 1.50 KX magnification (after adsorption)

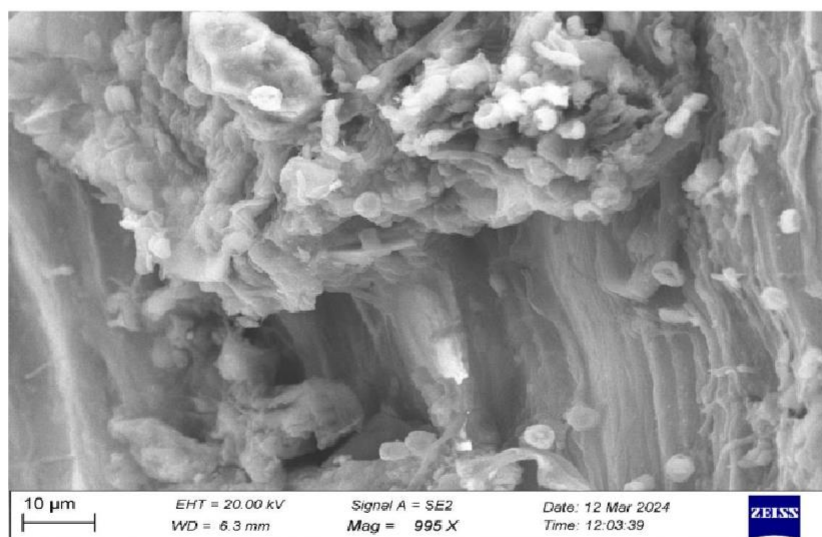


Fig 5.6 SEM Image under 995 X magnification (after adsorption)

5.2 Electron Dispersive X-Ray (EDX)

The EDAX mapping shows the composition of the adsorbent sample. The carbon and oxygen peaks obtained is due to the large amounts of C-H, C-O and O-H bonds present in siam weed. The presence of sodium and sulfur is due to the addition of sodium bi carbonate and sulfuric acid the during activation process

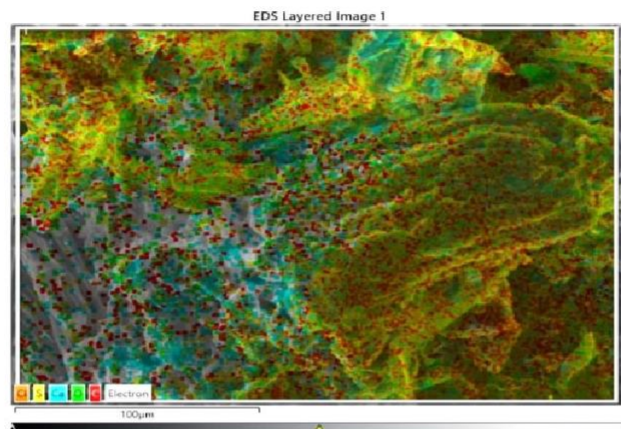


Fig 5.7 EDS layered image of siam weed before adsorption

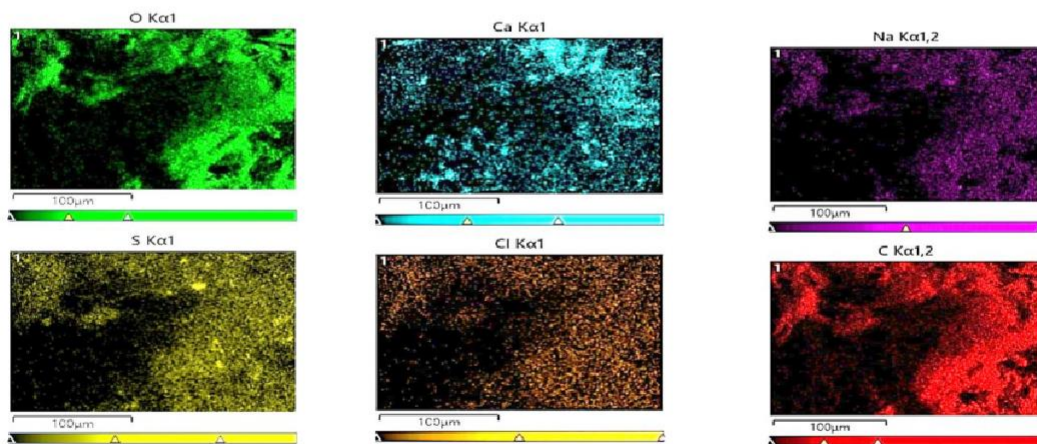


Fig 5.8 EDS layered image of o,s,na,c,ca,cl before adsorption

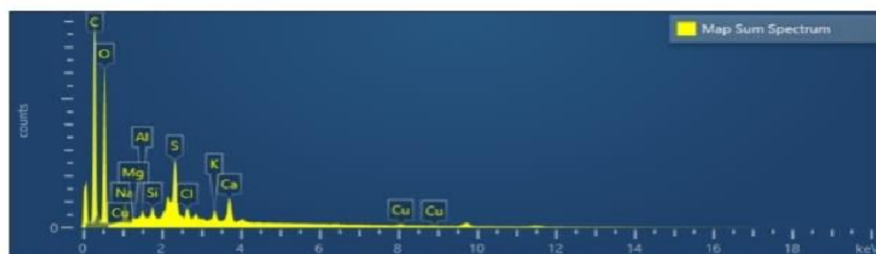


FIG 5.9 EDX(energy dispersive x-ray analysis) SPECTRA OF SEAM WEED

TABLE 5.1 EDAX (Energy dispersive X ray Analysis) before adsorption

Element	Line Type	Weight %	Weight Sigma	% atomic
C	κ series	56.58	0.38	65.31
O	k series	36.73		31.83
Na	κ series	1.14		0.69
Mg	κ series	0.08		0.04
Al	κ Series	0.25	0.02	0.13
Si	κ series	0.36		0.18
S	κ series	1.89		0.82
Cl	κ series	0.55	0.02	0.22
K	κ series	0.55	0.02	0.2
Ca	κ series	1.44	0.03	0.5
Cu	κ series	0.43	0.06	0.09
Total		100		100

5.3 FACTORS OF ADSORPTION

- **Nature of Adsorbate:** The chemical composition, size, shape, and polarity of the adsorbate molecules or particles significantly affect their interaction with the adsorbent surface.
- **Concentration of Adsorbate:** Higher concentrations of adsorbate molecules in the solution generally lead to increased adsorption onto the adsorbent surface until saturation is reached.
- **pH:** The pH of the solution can influence the charge of both the adsorbate and the adsorbent surface, affecting their interaction.

- **Properties of Adsorbent:** Adsorbate properties, such as molecular structure, polarity, and reactivity, profoundly impact adsorption processes. Factors like temperature and pressure further influence adsorption behaviour
- **Surface Area:** Greater surface area provides more sites for adsorption to occur, increasing the adsorption capacity of the material.
- **Surface Chemistry:** The chemical composition, functional groups, and surface charge of the adsorbent influence its affinity for specific adsorbates.
- **Porosity:** Porous materials have a higher internal surface area and can adsorb more molecules, especially those with smaller sizes.
- **Operating Conditions:** encompassing factors like temperature, pressure, and flow rate, play a crucial role in determining the efficiency and effectiveness of adsorption processes
- **Temperature:** Higher temperatures can enhance adsorption by increasing the kinetic energy of molecules, leading to more frequent collisions with the adsorbent surface.
- **Pressure:** In gas-phase adsorption, higher pressure can increase adsorption by providing more gas molecules to interact with the adsorbent surface.
- **Contact Time:** Longer contact times between the adsorbate and adsorbent allow for more adsorption to occur until equilibrium is reached.
- **Agitation:** Agitation or stirring promotes better contact between the adsorbate and adsorbent, enhancing the adsorption rate.

5.4 OPTIMIZATION PARAMETERS

5.4.1 Contact time

The collected samples are tested with UV spectrophotometer, and the removal percentage of the adsorbent can be found by calculating the absorbance value

obtained

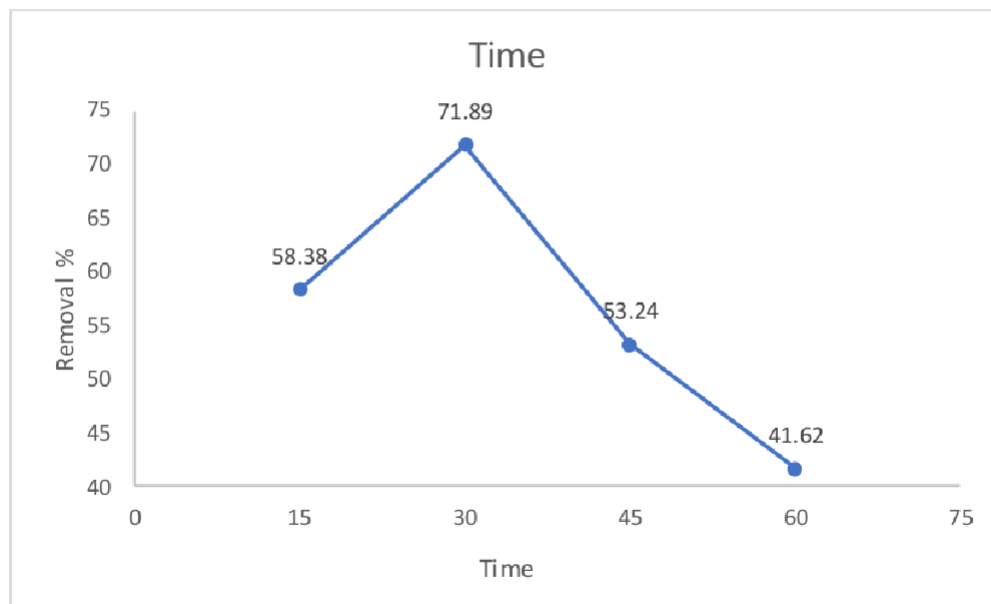


FIG 5.10 OPTIMIZATION OF TIME

From the data present, it can be concluded that the optimum contact time for the maximum removal of dye particles is approximately 15 minutes. One possible explanation for this observation is that after 15 minutes, most of the active sites of the adsorbent are likely occupied by dye molecules, leading to saturation of the adsorption capacity. Additionally, it is plausible that after 15 minutes, the rate of desorption becomes slightly higher than the rate of adsorption. This means that while some dye molecules continue to be adsorbed onto the active sites of the adsorbent, there is also a concurrent process of dye molecules being released from the adsorbent surface back into the solution. This equilibrium between adsorption and desorption processes could result in a plateau in the removal efficiency observed around 15 minutes. Therefore, the observed optimal contact time of 15 minutes likely represents a balance between adsorption and desorption kinetics, where the adsorption capacity of the adsorbent is maximized before desorption processes start to dominate.

5.4.2 Temperature

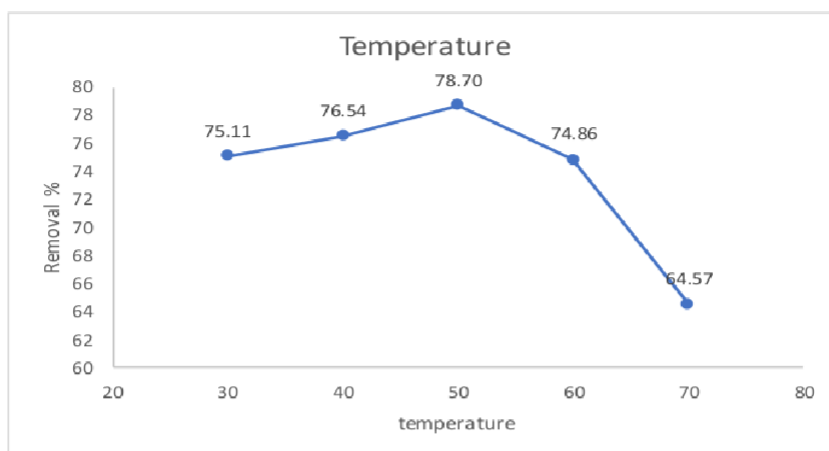


FIG 5.11 OPTIMIZATION OF TEMPERATURE

The removal of adsorption was studied by varying the temperature (30,40,50,60,70) while keeping the dosage initial concentration and contact time 15 mins. Removal was highest when temperature was 50 degree (78.70) so 50 degree was chosen as the optimum temperature

5.4.3 CONCENTRATION

UV spectrophotometer is used to evaluate the collected samples, and the absorbance value obtained can be used to calculate the removal percentage of the adsorbent.

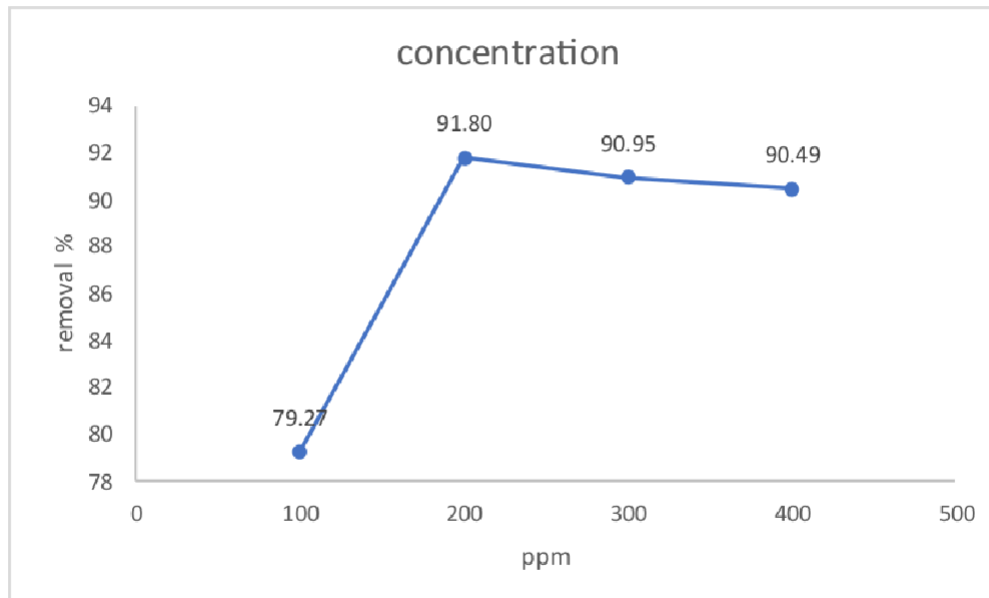


FIG 5.12 OPTIMIZATION OF CONCENTRATION

Based on the study where the removal of crystal violet dye was investigated by varying the initial concentration (100, 200, 300, 400, 500 ppm) while keeping the adsorbent dosage constant at 0.2 g and a contact time of 15 minutes, it can be concluded that the adsorption of 20 ppm is the most efficient for the maximum removal of dye particles. This observation aligns with the principle that at lower concentrations of the dye solution, there are numerous accessible adsorption sites available on the surface of the adsorbent. As a result, more dye molecules have the opportunity to interact with and be adsorbed onto these accessible sites, leading to a higher adsorption rate.

5.4.4 Adsorbent dosage

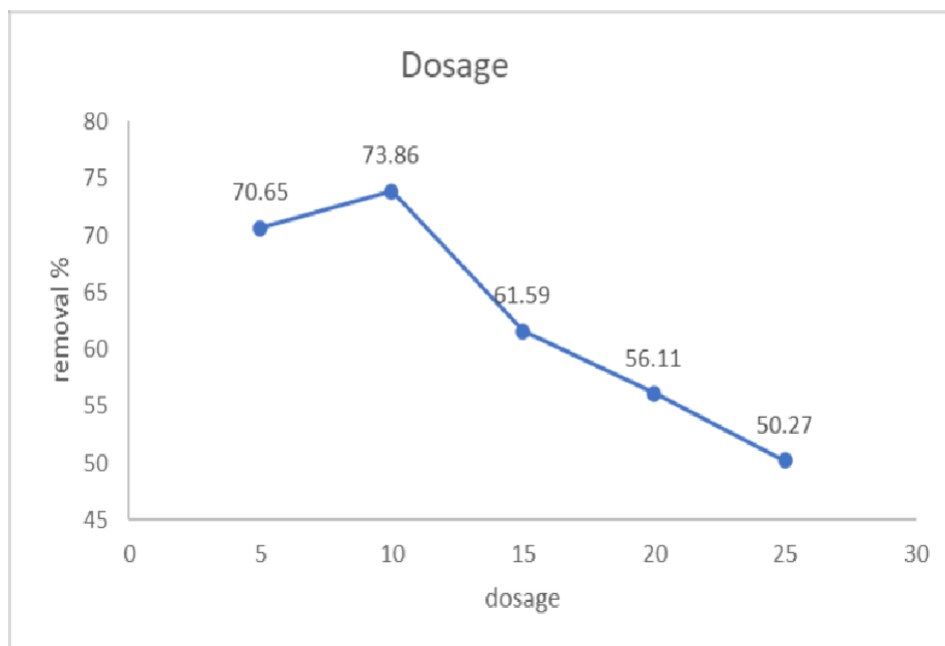


FIG 5.13 ADSORBENT DOSAGE

The collected samples are tested with UV spectrophotometer, and the removal percentage of the adsorbent can be found by calculating the absorbance value obtained. The data suggests that the optimum dosage for the maximum removal of dye particles is at 20 g/L of adsorbent dosage. One possible explanation for this finding is related to the availability of adsorption sites on the surface of the adsorbent material. At low dosages of the adsorbent, there are plenty of available adsorption sites on the surface. As more adsorbate (dye particles) is introduced into the system, these sites become progressively filled. This leads to an increase in the adsorption rate as more dye particles are captured by the available sites. However, as the dosage of the adsorbent continues to increase, the available sites become gradually saturated. Once the maximum capacity of the adsorbent is reached, further increases in dosage lead to fewer additional adsorption sites being available for dye molecules. Consequently, the adsorption rate starts to decrease because there are fewer sites available for additional adsorption, despite the higher dosage of the adsorbent.

5.5 KINETIC AND ISOTHERM STUDY

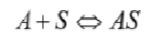
The study investigated the kinetic mechanism involved in the adsorption process of Fuchsine dye with AASCO adsorbent using intraparticle diffusion, also known as the Weber and Morris kinetic model. Additionally, three kinetic models were employed: pseudo-first-order kinetic equation, pseudo-second-order kinetic equation, and the Elovich model. For interpretation of the adsorption data, four isotherm models were utilized: Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich. These models were employed to understand the equilibrium adsorption behaviour. The kinetic model that best fitted the adsorption operation was utilized alongside Arrhenius and Eyring equations to calculate various thermodynamic parameters such as activation energy, change in Gibbs free energy, change in enthalpy, and change in entropy. These parameters provide insights into the energetics and spontaneity of the adsorption process. Furthermore, the isotherm model that best fitted the adsorption data was employed in designing a single-stage batch adsorber. This design aspect is crucial for scaling up the process for practical applications.

5.6 ADSORPTION KINETICS

5.6.1 PSEUDO FIRST ORDER

TABLE 5.2 PSEUDO FIRST ORDER KINETICS

ppm	R ²	K	qe
400	0.975	0.045	488.8228
300	0.932	0.037	292.6566
200	0.905	0.054	604.2572
100	0.895	0.036	264.542



$$\frac{q_t}{q_e} = 1 - \exp\left[-\left(k_A C_S + \frac{k_A}{k_S}\right)t\right]$$

$$\log(q_e - q_t) = \log q_e - \frac{k_{ad}}{2.303}t$$

$$k_{ad} = \left[\left(k_A C_S + \frac{k_A}{k_S} \right) \right]$$

$$q_t = X_{At} \quad \text{and} \quad q_e = X_{Ae}$$

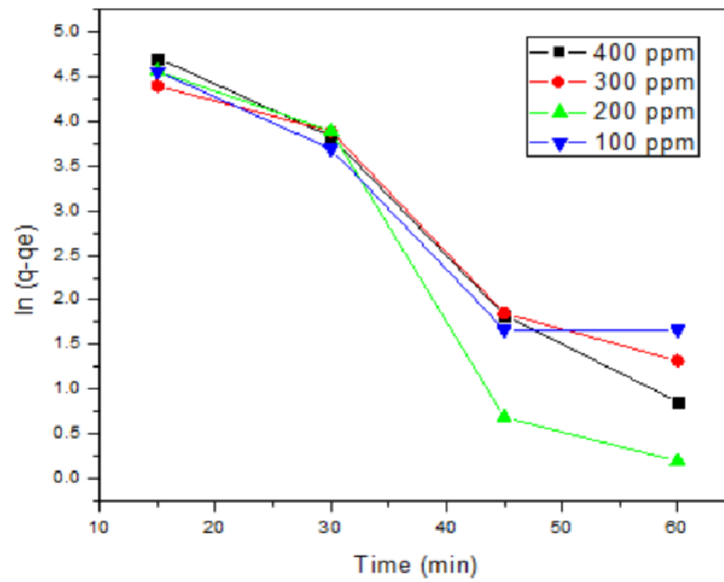


FIG 5.14 PSEUDO FIRST ORDER KINETICS

5.6.2 PSEUDO SECOND ORDER

TABLE 5.3 PSEUDO SECOND ORDER

	400 ppm	300 ppm	200 ppm	100 ppm
R ²	0.999	0.999	0.999	0.999
k ₂	0.002	0.002	0.001	0.001
q _e	500	500	1000	1000
h	500	500	1000	1000

$$\frac{dq_t}{dt} = k(q_e - q_t)^2$$

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + kt$$

$$\frac{t}{q_t} = \frac{1}{h} + \frac{1}{q_e} t$$

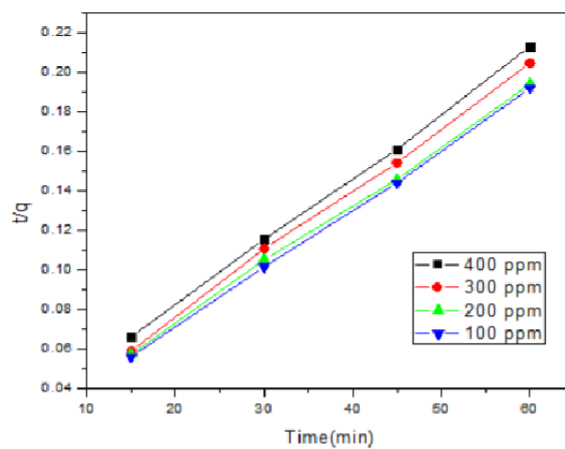


FIG 5.15 PSEUDO SECOND ORDER KINETICS

5.6.3 WEBER MORRIS

TABLE 5.4 WEBER MORRIS KINETIC

	R2	kid	C
1500 ppm	0.866	380.2	16.5
1200 ppm	0.939	439.1	13.56
900 ppm	0.875	453.1	14.86
600 ppm	0.92	461.6	15.43

$$q_t = K_{id}t^{0.5} + C$$

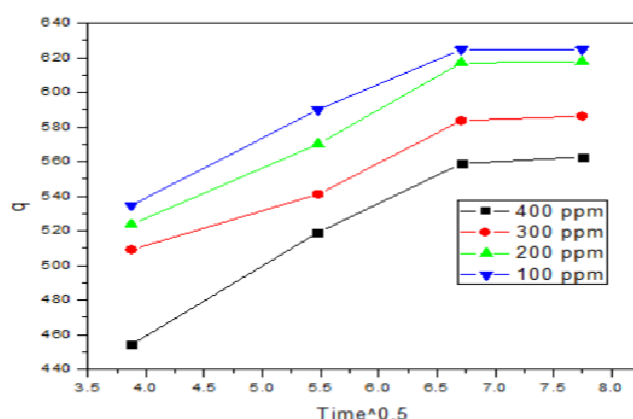


FIG 5.16 WEBER MORRIS KINETICS

In conclusion, the adsorption process under investigation predominantly adheres to the kinetics of **pseudo-first order** rather than **pseudo-second order**. This determination is supported by the superior fit of experimental data to the **pseudo- first order model**, as evidenced by the highest R-squared (R2) value attained. The observed adsorption behaviour suggests that the rate-limiting step in the adsorption process likely involves the surface interaction between the adsorbate and the adsorbent, a characteristic commonly associated with pseudo-first order kinetics. As such, this finding underscores the importance of considering kinetic models in understanding and optimizing adsorption processes for practical applications.

5.7 ADSORPTION ISOTHERM MODELS

5.7.1 LANGUMIUR ISOTHERM

TABLE 5.5 LANGUMIUR ISOTHERM

R^2	0.93
Qm	0.104932
KL	0.020672

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

$$R_L = \frac{1}{1 + K_L C_0}$$

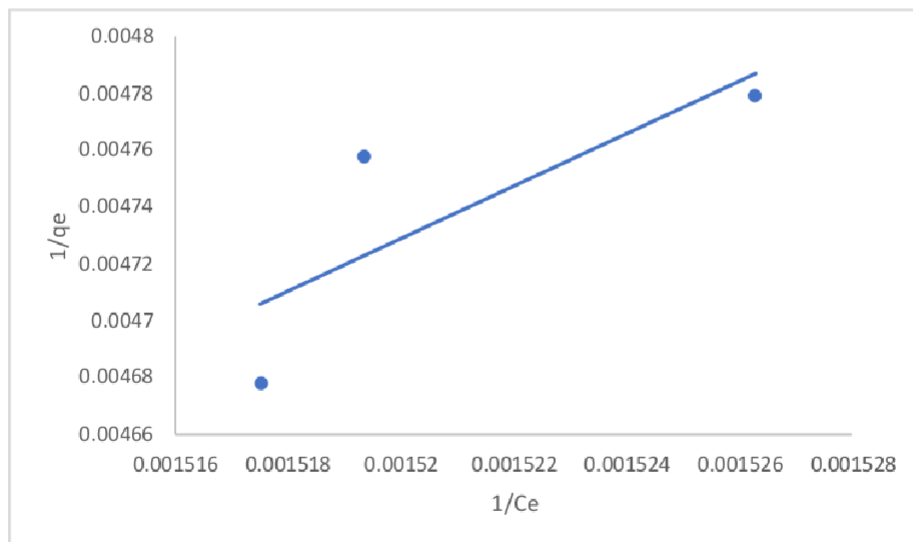


FIG 5.17 LANGUMUIR ISOTHERM

5.7.2 FREUNDLINCH ISOTHERM

TABLE 5.6 FREUNDLINCH ISOTHERM

R^2	0.996
kf	57.16833
n	2.380952

$$q_e = K_F C_e^{1/n}$$

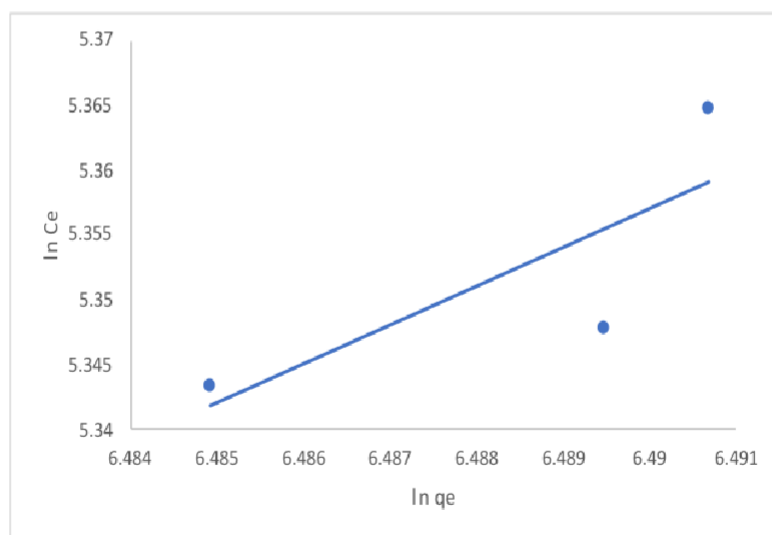


FIG 5.18 FREUNDLINCH ISOTHERM

5.7.3 TEMKIN ISOTHERM

TABLE 5.7 TEMKIN ISOTHERM

R^2	0.966
A	0.0397
B1	-250

$$q_e = B \ln (A C_e)$$

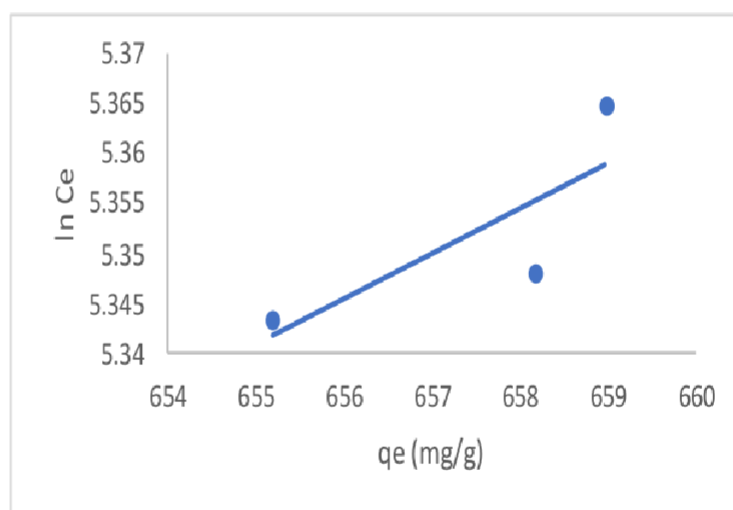


FIG 5.19 TEMPKIN ISOTHERM

5.7.4 DUBININ RADUSHKEVICH

TABLE 5.8 DUBININ RADUSHKEVICH

R^2	0.938
β	9.26
qmD	3.18E+28

$$\ln q_e = \ln q_{mD} - \beta \left[RT \left(1 + \frac{1}{C_e} \right) \right]^2$$

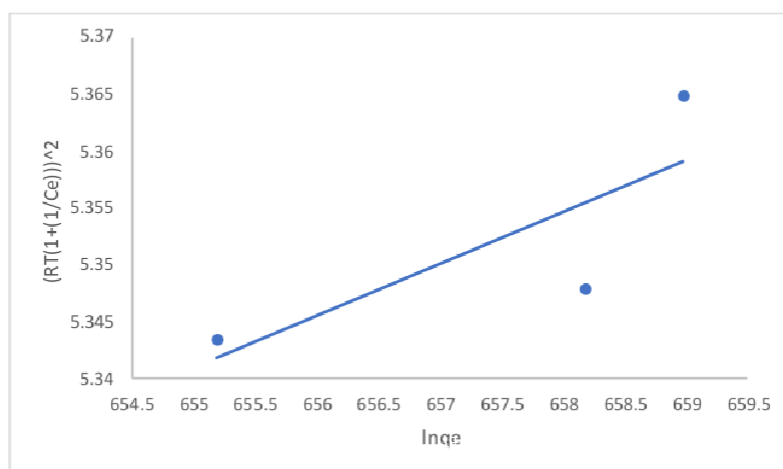


FIG 5. DUBININ RADUSHKEVICH (FOR ADSORPTION)

Based on the experimental data and analysis, it can be concluded that the adsorption process follows the Freundlich isotherm model most closely among the Langmuir, Freundlich, and Temkin isotherms. This conclusion is supported by the highest R^2 value obtained for the Freundlich isotherm model. The Freundlich model describes multilayer adsorption onto heterogeneous surfaces, which suggests that the adsorbate molecules exhibit non-ideal behaviour and form a heterogeneous distribution of adsorption sites on the adsorbent surface. Therefore, the Freundlich isotherm provides a better fit to the experimental data and offers valuable insights into the adsorption mechanism and surface properties of the adsorbent under investigation.

CHAPTER – 6

CONCLUSION AND SUMMARY

The present adsorption study has shown the feasibility of Fuchisine dye adsorption on the AASCO adsorbent. The batch adsorption experiment data was used in the study of adsorption kinetics and isotherm. In conclusion, the adsorption of Fuchisine dye demonstrates a promising method for wastewater treatment or dye removal processes. The efficient adsorption capacity observed in this study suggests the potential application of the adsorbent material in real-world scenarios, contributing to environmental remediation efforts. Further research and optimization are recommended to enhance the efficacy and scalability of this adsorption process. The adsorption-desorption reusability study indicated that the AASCO adsorbent could be reused for 3 cycles with higher Fuchisine dye removal efficiency. This study primarily suggests that the adsorption operation of CV dye molecules by AASCO adsorbent is a thermodynamically feasible process. A scale-up of the adsorption operation was theoretically investigated by designing a single-stage adsorber. Adsorption of Fuchisine dye involves the binding of the dye molecules to a solid surface, typically through physical or chemical interactions. This process is often used in water treatment to remove dye pollutants. The efficiency of adsorption depends on factors like surface area, material characteristics, and dye concentration. Studies focus on optimizing conditions for maximum adsorption capacity and understanding the mechanisms involved, which can include electrostatic interactions, Van der Waals forces, or chemical bonding. Overall, the goal is to develop effective and sustainable methods for removing crystal violet dye from aqueous solutions.

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