Natural Fiber-Based Biosorbents for Methylene Blue Dye Removal Using *Parthenium hysterophorus* and *Lantana camara*

Thesis submitted in the fulfilment of the requirement for the degree of

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Master of Technology

In

Biotechnology

By

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Under the supervision of

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Candidate's Declaration

I hereby declare that work represented in this report is entitled **"Natural Fiber-Based Biosorbents for Methylene Blue Dye Removal Using *Parthenium hysterophorus* and *Lantana camara* "** in partial fulfilment of the requirement of the award of the degree of **Master of technology** in Biotechnology submitted to the Department of Biotechnology & Bioinformatics, Jaypee university of Information technology, Waknaghat is an authentic record of my own work carried out over the period from July 2024 to May 2025 under the supervision of **Dr. Abhishek Chaudhary , Assistant professor, Professor of Department of Biotechnology and Bioinformatics** Jaypee University of Information Technology, Waknaghat, Solan, India.

I also authentic that I have carried out the above-mentioned project work under the proficiency stream Biotechnology.

This work has not been submitted previously for any degree, diploma, or other academic purposes. I have ensured that all sources of information used in this project have been duly acknowledged.

The matter embodied in the report has not been submitted for the award of any other degree or diploma.

Robin Singh Rangra, 235011003

Supervisor’s Certificate

This is to certify that the work presented in the project report titled “**Natural Fiber-Based Biosorbents for Methylene Blue Dye Removal Using *Parthenium hysterophorus* and *Lantana camara***” in partial fulfilment of the requirement for the award of degree of **Masters of Technology** in Biotechnology submitted to the department of Biotechnology and Bioinformatics, Jaypee University of Information Technology Waknaghat. Is an authentic record of work carried out during the period of July 2024 to May 2025 under the supervision of **Dr. Abhishek Chaudhary**, Assistant Professor, Department of Biotechnology and Bioinformatics.

This is to certify that the above statement made is correct to the best of my knowledge.

(Supervisor)

Dr. Abhishek Chaudhary

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Dated:

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Robin Singh Rangra (235011003)

LIST OF ABBREVIATION

%	Percentage
MB	Methylene Blue
Fig.	Figure
Conc.	Concentration
P H	*Parthenium hysterophorus*
L C	*Lantana camara*
FTIR	Fourier Transformed Infrared Spectroscopy
EDTA	Ethylene diamine tetra acetate
NaOH	Sodium Hydroxide
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
EDS	Energy Dispersive X-ray Spectroscopy
µl	Micro mol
mM	Mili mol
AOP	Advanced Oxidation Process
ph	Power of Hydrogen Ion
H_2SO_4	Sulfuric Acid
Distilled Water	D W
H_2O_2	Hydrogen Peroxide
ºC	Degree Celsius
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetry
VSM	Vibrating Sample Magnetometry
Vol.	Volume

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ABSTRACT

The discharge of synthetic dyes, e.g., methylene blue (MB), from the textile sector into water bodies has serious environmental and health implications. Due to the chemical stability and non-biodegradability of MB, efficient and environmentally friendly remediation strategies are needed. In this research, the possibility of utilizing natural fibres from invasive plant species (*Parthenium hysterophorus* and *Lantana camara*) as efficient biosorbents in MB removal from wastewater was investigated. The plant material was Soxhlet extracted in ethanol to purify the material, and followed by sodium hydroxide alkali hydrolysis to improve fibre accessibility. The modified fibres were subsequently mechanically processed to sheet form for adsorption experiments. The functional groups and surface morphology of the resultant fibres were investigated using techniques like Fourier-transform infrared spectroscopy (FTIR) and inverted microscopic analysis. Batch adsorption experiments were performed with MB working concentrations of 40 microlitres. The Langmuir isotherm model was used to model the adsorption data, which manifested high adsorption capacities due to surface hydroxyl and carboxyl groups that improve high adsorption affinity with MB molecules. The findings illustrate the efficacy of *Parthenium hysterophorus* and *Lantana camara* fibres as low-cost, renewable, and biodegradable biosorbents in the removal of dye wastewater. The method provides a sustainable strategy in wastewater treatment and offers a utilizable application of invasive plant biomass, thereby promoting environmental conservation and resource management.

Chapter- 1

Introduction:

Water pollution has become a worldwide issue with the increase in environmental problems related to development. Among the wide range of pollutants, industrially employed synthetic dyes are problematic due to their potential toxicity and persistency. Discharge of dyes into water bodies without treatment causes water discoloration, inhibition of light penetration, disturbance in aquatic life, and hazards to human and wildlife health. With these conditions, sustainable and environmentally friendly approaches to wastewater treatment are receiving more attention. Plant-based natural plant fibers have been considered as potential dye adsorbents because of their abundance, biodegradation, and accessibility of functional groups to adsorb dye molecules. This study investigates the potential of invasive weed plants *Parthenium hysterophorus* and *Lantana camara* as natural adsorbents for the removal of methylene blue from water-polluting sources. [1]

1.1 Background and Problem Statement

Water pollution has become a serious issue worldwide with the increasing environmental issues related to development. Synthetic dyes, widely applied in the textile industry, are of special interest among all the pollutants for their toxicity and persistence. If released into water bodies without proper treatment, these dyes not only give colour but also inhibit light penetration, interfere with aquatic life, and are harmful to wildlife and human health. To counteract these issues, green and sustainable wastewater treatment processes are receiving greater interests. Plant-derived natural fibres have been found to be promising candidates for dye adsorption due to their abundance, biodegradability, and the availability of functional groups for binding dye molecules. This work discusses the applicability of using invasive plant species, namely P H and L C, as natural adsorbent for the removal of methylene blue dye from polluted water sources. [1]

Table 1: *Examples of reported health and environmental impacts of Methylene Blue (MB) dye* [3]

Parameters	Details	References
Toxicity to aquatic life	Highly toxic to fish and microorganisms; reduces biodiversity in water bodies.	[3]
Health risks to humans	Can cause nausea, vomiting, anaemia, increased heart rate, and methemoglobinemia upon exposure.	[3]
Inhibition of photosynthesis	MB in water absorbs light, preventing sunlight from reaching aquatic plants and algae, thus inhibiting photosynthesis.	[3]
Aesthetic and oxygen depletion	Imparts blue colour to water, causing aesthetic pollution; decomposition consumes oxygen, leading to lower DO levels.	[1]

Given the persistent and hazardous nature of dyes like MB, regulatory limits on dye effluent discharge are strict. Developing efficient and affordable remediation technologies is essential to protect environmental health.

1.2 Treatment Techniques for Dye Removal

Treatment Techniques for Dye Removal Over the years, a variety of physical, chemical, and biological methods have been employed to treat dye-contaminated wastewater [1][4]. Common techniques include coagulation–flocculation, membrane filtration, advanced oxidation processes (AOPs), electrochemical degradation, biodegradation, and adsorption using activated carbon or other sorbents [1][2]. While many of these methods can achieve significant dye removal, each has its drawbacks. Chemical methods, such as oxidation or ozonation, often incur high reagent costs and may generate toxic by-products. Membrane processes and ion exchange can be effective but are hindered by membrane fouling and high operational costs. Biological treatments using microbes can degrade certain dyes but are generally slow and ineffective for recalcitrant dyes like MB, which have complex aromatic structures [1]. In contrast, adsorption has emerged as a cost-effective and versatile method for dye removal [1][4]. Adsorption processes can remove dyes even at low concentrations and are relatively simple to design and operate. Activated carbon is the most commonly used adsorbent due to its high surface area and affinity for organic pollutants. However, the expense of commercial activated carbon has spurred research into low-cost alternative adsorbents derived from waste materials [4]. Recently, a wide range of agricultural and biomass wastes have been explored as inexpensive adsorbents, often referred to as “biosorbents,” for dye removal. These include sawdust, fruit peels, crop residues, and other plant fibres, which are abundant, renewable, and require minimal processing. By converting waste materials into useful adsorbents, adsorption technology can become more sustainable and economical. Among physical methods, adsorption stands out for its ease of operation and high removal efficiencies for various dyes [1]. Therefore, developing natural fibre-based adsorbents is a promising strategy to address dye pollution in an eco-friendly manner.

1.3 Natural Fiber-Based Biosorbents

Plant biomass-based natural fibers are particularly prospective biosorbents as they have a high percentage of cellulose, hemicellulose, and lignin, which bear functional groups (hydroxyl and carboxyl) that can interact with dye molecules [5]. Rice husk, coconut coir, jute, agricultural residues, and sawdust have been found effective in the removal of dyes from aqueous solutions [6]. Jute fiber-activated carbon, for instance, has indicated an adsorption capacity of around

225.6 mg/g for methylene blue (MB), with good kinetic behavior [7]. Coconut coir dust, even without proper chemical activation, has high adsorption affinity for MB, with removal efficiencies of over 90% under optimal conditions [1]. Banana peel and sugarcane bagasse have also showed MB adsorption capacities of 20 to 35 mgr/g under batch experimentation [6]. Evidence from these findings confirms that lignocellulosic fibers, even considered as waste by-products, can be converted into effective sorbents for dye pollutants.

Table 2: *Adsorption capacities of various natural biosorbents for Methylene Blue dye (monolayer Langmuir capacity, q_{max})*[1]

Biosorbent	**Adsorption Capacity Of MB**	**Reference**
Jute fibre activated carbon	225.6 mg/g	[7]
Coconut coir dust (raw)	*93–98% removal* (at 50–200 mg/L initial) *~50–80 mg/g (est.)*	[1]
Banana pseudo-stem fibre	42.3 mg/g	[8]
Sawdust (Indian Rosewood)	30.7 mg/g	[7]
Sugarcane bagasse (raw)	34.2 mg/g	[7]
Sunflower seed hull activated carbon	150–200 mg/g (range)	[1]
Parthenium hysterophorus weed (raw)	18–20 mg/g	[7]

Biosorbent	Adsorption Capacity Of MB	Reference
Lantana camara stem activated carbon	19.8 mg/g	[7]

Note: Capacities are as reported in referenced studies under particular experimental conditions (solution pH, temperature, etc.). "Removal" percentages are provided where capacity in mg/g was not explicitly reported.

As can be observed from Table 2, for the vast majority of untreated or poorly treated biomass adsorbents, the adsorption capacities for the dye are relatively moderate (tens of mg per gram), whereas some chemically or heat-activated (e.g., jute AC or sunflower hull carbon) ones possess much greater capacities. Even invasive weed materials – Parthenium and Lantana – proved to possess competitive performance (almost 20 mg/g for MB) in initial tests [7]. This indicates that with appropriate surface modification or activation, even these weeds can prove efficient biosorbents.

1.4 *Parthenium hysterophorus* and *Lantana camara* as Target Biosorbents

Parthenium hysterophorus or Congress grass or feverfew and *Lantana camara* are weed species that are found invasive in most parts of the world. They are typically pests—Parthenium is allergenic and upsets ecological balance, Lantana is toxic to animals—leading to large-scale campaigns to control them. The application of this unwanted biomass to a valuable adsorbent for wastewater treatment conforms to waste-to-resource principles and sustainable development. Earlier research has reported the potential of these plants as biosorbents. Lata et al. [19] used P H, a herbaceous weed, to adsorb a basic dye (MB) from water in batch experiments with acceptable removal efficiency. Mulugeta and Lelisa observed that the dose of P H biomass increased to significantly improve MB removal efficiency, indicating its high cationic dye affinity [9]. Likewise, LC, a flowering woody shrub, has been used as an adsorbent: Amuda et al. activated L C stems to produce activated carbon, which adsorbed more than 93% MB from textile wastewater [2]. A different study pretreated L C biomass with citric acid to increase surface functionality, with increased MB uptake capacity [10]. These encouraging findings suggest that invasive weeds can be converted to low-cost adsorbents. But there are gaps in knowledge and practical issues. The raw biomass of these weeds is likely to be waxy, oily, and contain other extractives that are detrimental to adsorption, and pretreatment procedures may have to be optimized. Besides, knowledge of the adsorption mechanism—how the dye molecules interact with the fibre surface—is essential to optimize performance further. This research fills such gaps by conducting systematic research on the preparation of P H and L C fibres and comparing their adsorption behaviour for MB removal.

1.5 Research Objectives and Scope

- The major aim of the present work is to develop effective biosorbents from P H and LC fibers for the removal of Methylene Blue dye from aqueous solutions. For the accomplishment of this purpose, the work is divided in some specific sub-objectives:
- **Fiber Preparation**: This includes the setup and assessment of multi-dimensional pretreatment methods on the weed fibers, i.e., alkali hydrolysis (NaOH treatment), Soxhlet solvent extraction, and mechanical beating/refining, with the objective of increasing their adsorption capacity. These treatments try to maximize surface area, remove natural inhibitors (such as oils and lignin), and develop additional binding sites on the fiber surface [5][11].
- **Characterization:** Treated and untreated fibers will be characterized using techniques such as Fourier Transform Infrared Spectroscopy (FTIR) to

identify functional groups, Scanning Electron Microscopy (SEM) to investigate surface morphology, and pHzpc (zero-point charge) measurement to establish surface charge characteristics [1]. The above analysis techniques will confirm changes in fiber chemistry (for instance, the elimination of hemicellulose/lignin due to alkali treatment) and morphological changes due to the treatments.

- **Batch Adsorption Studies:** The effectiveness of the biosorbents synthesized for the dye removal will be determined under batch adsorption studies. Among the most critical parameters to be studied are solution pH, initial dye concentration, contact time, and temperature. The effects of these parameters on MB adsorption will be evaluated for determining the optimal adsorption conditions for each biosorbent.
- **Adsorption Isotherm and Kinetics Modelling**: Equilibrium data will be interpreted through isotherm models, i.e., Freundlich and Langmuir, to determine maximum sorption capacity (q_{max}) and the corresponding affinity constants. Of significance, Langmuir model will define monolayer capacity and compare with the literature [1]. Kinetic data will be interpreted through models such as pseudo-second-order, pseudo-first-order, and intraparticle diffusion to describe the rate-controlling steps for the adsorption process [7]. A pseudo-second-order model, describing chemisorption, is predicted, based on similar studies, to most suitably describe the adsorption of MB onto these fibres [12].
- **Mechanistic Insights:** MB adsorption mechanism onto the biosorbents will be studied combining experimental data and analytical findings. FTIR spectra post-adsorption and pre-adsorption will be compared in order to establish peak shifts (e.g., –OH, C=O groups) to ascertain binding of the dye [22]. Electrostatic interaction role will be concluded from pH-dependent activity (as MB is a cationic dye, adsorption usually increases at higher pH when fibre surface takes on negative charge [1]). Other

potential interactions, e.g., π–π stacking between benzene rings of MB and any aromatic –group of the biosorbent, or n–π interaction between donor atom lone-pair electrons and the π-system of the dye, will be examined based on literature [13].

The scope of this research is experimentation and analysis at the laboratory scale. Even though the main focus is on basic understanding and comparison of evaluation, the findings will be presented in the context of practical implementation, for example, room for scaling up operations, regeneration and reuse of biosorbents, as well as spent adsorbent disposal. The following chapter presents an overview of the literature to put the research strategy in context of recent scientific knowledge.

Chapter 2: Literature Review

2.1 Methylene Blue: Structure and Environmental Impact

Methylene Blue (MB) is a heterocyclic aromatic dye of the thiazine class, with molecular formula $C_{16}H_{18}N_3SCl$ 319.85 g/mol [2]. In aqueous solution it appears as a bright blue cationic dye that readily binds to negatively charged surfaces.

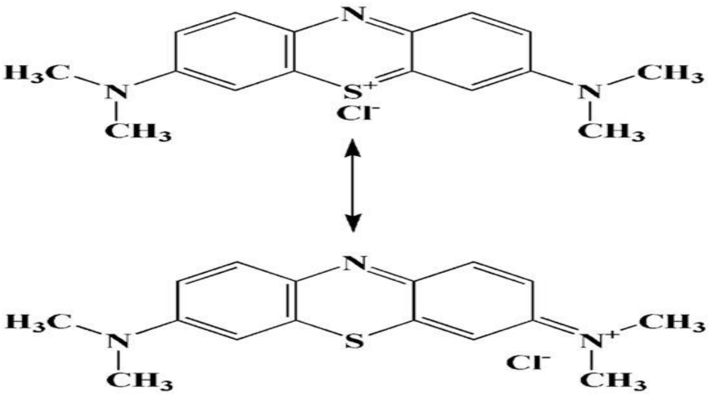


Fig. 1: Illustrates the chemical structure of the MB cation [3]·

The molecular structure of methylene blue (MB) contains a delocalized π-electron system across its phenothiazine ring, with the charge being largely localized on the central nitrogen atom. This molecular structure is the reason behind MB's intense coloration and tendency to bind to oppositely charged sites. When in salt form, methylthioninium chloride, the MB cation is usually accompanied by a chloride anion (Cl−) to balance the charge. MB is one of the most widely used basic dyes used in industries with extensive applications across industries for the dyeing of natural fibers like cotton, wool, and silk, in printing inks, in paper production, and as a biological stain [3][2]. Its extensive use is one of the contributing factors to its frequent occurrence in industrial effluent. From an environmental perspective, MB poses enormous environmental challenges due to its toxicity, environmental persistence, and resistance to biodegradation. Extensive reviews of the toxicity of MB have highlighted its ability to cause severe health complications, such as respiratory distress, vomiting, cyanosis, and tachycardia, in human beings exposed to high levels. Moreover, it is considered to be potentially carcinogenic and mutagenic. In aquatic ecosystems, even trace levels of MB, in terms of parts per million, make water greatly discolored and is toxic to fish and microorganisms [3]. MB also inhibits the growth of aquatic plants and algae by inhibiting light penetration. Moreover, as an organic pollutant, MB consumes dissolved oxygen during degradation, which results in anoxic conditions fatal to aquatic organisms [1][3]. As a result, regulatory limits tend to restrict dye levels in wastewater to a few mg/L or even µg/L. As such, it is imperative that MB is removed or neutralized in wastewater before discharge. Traditional biological treatment operations in wastewater treatment facilities are very ineffective as regards methylene blue (MB) due to the complexity of the aromatic character of the dye, which makes it resistant to microbial degradation. Such inherent resistance is the major reason why adsorption is the preferred supplementary operation for MB removal after primary treatment. Even though adsorption does not allow for the destruction of the dye, it precipitates the dye onto a solid phase from which it can be recovered or disposed without any risk. In the case of MB, a suitable adsorbent should have a high concentration of anionic or aromatic functional sites to effectively trap the cationic MB molecules. Most natural fibers meet the requirement with minimal processing because they have surface functional groups (e.g., deprotonated carboxylates or phenolic –OH groups) onto which the cationic dyes are adsorbed by mechanisms of electrostatic attraction or charge-transfer interactions [13].

2.2 Adsorption Mechanisms for Dye Removal

Adsorption is a phenomenon where molecules, called adsorbates, collect on the surface of a solid, called the adsorbent, to form an adsorbed film. In dye adsorption systems, there can be more than one mechanism working in parallel: Electrostatic Attraction: This mechanism is usually dominant for cationic dyes like methylene blue (MB) when the adsorbent surface carries negative charges. Natural fibres, if treated with an alkaline solution, are often found to have deprotonated acidic groups (–COO—, OH) on their surfaces [5]. MB cations are drawn to these negatively charged sites by Coulombic forces, leading to adsorption. The role of electrostatic interactions is usually demonstrated by the strong pH dependency of adsorption; for instance, MB removal improves at elevated pH values (above the pH_{zpc} of the adsorbent) because the surface becomes increasingly negatively charged [1]. π–π Interactions: MB has an extended π-conjugation within aromatic rings. If the adsorbent too has aromatic structures, as is the case with activated carbon or charred biomass, π–π stacking is possible between MB and the adsorbent surface [13]. Basically, the aromatic rings stack and are attracted by van der Waals forces. This mechanism is important for carbonaceous adsorbents where surface graphitic domains can interact with MB's phenothiazine ring. Amuda et al. (2014) found that even after activation, *Lantana camara* carbon still had some functional groups and π-electron-rich domains that would have contributed to MB uptake through means other than purely electrostatic [12]. n–π Interaction and Hydrogen Bonding: Functional groups on biomass like –OH, –NH, and –C=O are capable of acting as electron donors. An "n–π" interaction is a lone pair (n) on a donor atom (e.g., O or N in the fibre) interacting with the π system of the dye or vice versa [13]. MB's π system can accept electrons from donor sites, creating a weak complex. Hydrogen bonding between MB (which in solution can also be in protonated forms or with water of hydration) and polar adsorbent groups can also exist. Though MB is not a good hydrogen bond donor, some interactions through its amine groups or associated water molecules can create hydrogen bonds with surface –OH or –COOH groups of the fibre. Physical Adsorption (Dispersion Forces): Similar to any adsorption process, nonspecific van der Waals forces are responsible for MB adsorption. High surface area adsorbents offer extensive surfaces for these interactions. Porous adsorbents can also confine MB in pore spaces (pore diffusion), especially if pore diameter is similar to the size of MB, which is a relatively large molecule. Intraparticle diffusion into pores can often control the adsorption rate at later stages of the process [1]. These mechanisms can occur in parallel, with the predominance of one over another depending on the surface chemistry of the adsorbent, solution pH, and the presence of any functional group alterations. In biosorbents from plants, researchers usually find that chemical modification can increase some mechanisms. For example, citric acid modification of Lantana biomass (introducing more carboxyl groups) would increase electrostatic and hydrogen-bonding interactions with MB [10], while high-temperature activation (char formation) would increase π–π interactions. Quantifying adsorption capacity and determining the mechanism, adsorption isotherm models are utilized. The Langmuir isotherm is particularly important in monolayer adsorption of the dye molecules. This suggests a homogeneous surface comprising equivalent sites in a limited number, without any interactions between the adsorbed species. The Langmuir model can be primarily expressed as:

$$Q_e = \frac{Q_o K_L C_e}{1 + K_L C_e}$$

where qe is the amount of dye adsorbed at equilibrium (mg/g), Ce is the equilibrium dye concentration (mg/L), Q_0 is the monolayer adsorption capacity (mg/g), and KL is the Langmuir affinity constant (L/mg). A linear form often used is

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{Q_0 K_L C_e}$$

The Langmuir parameters allow calculation of RL, a dimensionless constant that represents the favorability of adsorption. In this case, 0 and 1 represent favorable adsorption [8]. Most dye adsorption processes, like methylene blue (MB) on different biosorbents, precisely follow the Langmuir model, which represents monolayer coverage on a similarly homogenous surface [1]. For example, MB adsorption on treated banana fiber follows the Langmuir model, which shows maximum adsorption capacity (Q) of 42.28 mg/g and very good correlation coefficient (R^2≈0.998) [8], which favors homogeneous binding energy on the surface in this system. Freundlich isotherm, an empirical model, is often applied to dye adsorption on heterogeneous surfaces. It can be represented as

$$q_e = K_f (C_e)^{\frac{1}{n}}$$

In the context of adsorption studies, Kf and n are Freundlich constants that correspond to adsorption capacity and intensity, respectively. A larger value of n (or a smaller value of 1/n) indicates more favourable adsorption, characterized by stronger bonding at lower concentrations. Biosorbents frequently exhibit a good fit to the Freundlich model, which reflects surface heterogeneity, or the presence of different types of binding sites [8]. In numerous studies, both the Langmuir and Freundlich models are capable of describing adsorption equilibrium; however, the Langmuir model is preferred when a distinct plateau in adsorption is observed at high concentrations, signifying site saturation. Regarding adsorption kinetics, the two most commonly employed models are the pseudo-first-order model (Lagergren rate equation) and the pseudo-second-order model (Ho and McKay model). The pseudo-first-order model posits that the rate of occupation of adsorption sites is proportional to the number of unoccupied sites, and it is expressed in linear form as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t$$

where q_t is the amount adsorbed at time t, and $k_1$is the first-order rate constant (min^{-1}). This model often fits the initial stages of adsorption but not the entire time range for dye systems.

The pseudo-second-order model is:

$$\frac{t}{q_1} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$

The k2 notation is the second-order rate constant (g mg−1 min−1). A straight line in a t/q1 vs. t plot indicates whether this model is valid or not [8]. For most materials, the pseudo-second-order model is valid to desorb MB. This means that chemisorption (chemical bonding with valence forces or electron sharing) is the rate-determining step [7][12]. Al Ashik et al. (2023) described that MB adsorption onto coir dust followed the pseudo-second-order model with $R^2 > 0.99$, but the pseudo-first-order model was not well-fitted [1]. This means that the rate of adsorption is a function of the number of available sites and is accompanied by surface reactions like ion exchange. Sometimes the process is more than a single step: rapid adsorption on the surface, followed by slower adsorption within pores (intraparticle diffusion). Intraparticle diffusion can be confirmed with the Weber-Morris plot qt vs. t1/2. If the plot is a straight line through the origin, intraparticle diffusion is the only rate-determining step; otherwise, other processes occur. Coir dust showed a multi-step plot, which means both film diffusion and intraparticle diffusion [1]. In short, MB adsorption onto biosorbents is a multi-faceted process with surface chemistry and dye characteristics. Ionic interactions and physicochemical adsorption allow natural fibres to adsorb MB from water. Understanding these basics facilitates studies on Parthenium and Lantana fibres.

2.3 Preparation and Modification of Natural Fibers for Adsorption

Raw plant fibres typically necessitate pretreatment to enhance their adsorption capabilities. The literature outlines several treatment methodologies designed to augment surface area, porosity, and the number of active binding sites on biomass adsorbents.

- **Alkaline Treatment (Alkali Hydrolysis):** boiling or soaking the fibre in a basic solution, typically sodium hydroxide (NaOH). The treatment allows saponification of the fatty acids and removal of natural waxes, oils, lignin, and hemicellulose from the fibre surface, exposing the crystalline cellulose structure and producing additional hydroxyl (–OH) and carboxylate (–COO−) groups on the surface. This increases the roughness and porosity of the fibres due to the loss of amorphous content [5]. For instance, treatment of banana fibre with 5% NaOH was found to enhance its methylene blue (MB) adsorption due to increased surface roughness and charge density, as evidenced by the alteration of the fibre's point of zero charge (pH_{zpc}), leading to a more charged surface at neutral pH [8]. Alkali treatment conditions should be controlled precisely as harsh or prolonged treatment might result in cellulose degradation and loss of structural integrity of the biosorbent.

- **Soxhlet Extraction:** utilizes a Soxhlet apparatus to extract soluble constituents from plant biomass using organic solvents. Ethanol or a toluene-ethanol mixture is typically used to extract pigments, resins, and fats from fibers. Researchers typically subject weed biomass to Soxhlet extraction before activation for the purpose of purifying natural compounds that do not adsorb. In Parthenium or Lantana, Soxhlet extraction can eliminate allelopathic chemicals or essential oils contained in these weeds, yielding clean surface chemistry and active sites free from extraneous material. Soxhlet extraction has been effectively used on cotton fibers to eliminate waxy materials, greatly improving the following dye uptake of the fibers [11].
- **Mechanical Beating/Fibrillation** Mechanical grinding or agitation of fibers to break them down to smaller fibrils. In paper production, the process of beating is used to increase fiber flexibility and surface area. In adsorbent treatment, grinding or high-shear blending of fibers can result in particle size diminution and partial breakdown of the fiber cell walls with the finer fragments produced having greater exposure of internal surfaces [12]. For example, beating of Parthenium weed to fine powder has been utilized in several studies to allow sieving to provide uniform small particle size (e.g., <300 μm) [4]. Having finer particles has a greater surface area-to-volume ratio, which can enhance adsorption capacity, such as in the case of coir dust, where the finer particle fractions showed better methylene blue

adsorption than the coarse ones [1]. Over-grinding, however, can introduce problems with solids-liquid separation after treatment and pressure drop issues in flow systems, though otherwise it improves removal efficiency in batch treatment.

- **Chemical Activation and Grafting:** Apart from mere alkali treatment, more advanced higher-order chemical modifications have been investigated to improve the characteristics of biosorbents. Acid treatments, especially with H2SO4, can create sulfonated surfaces or enhance porosity by dehydration and carbonization of the biomass. For example, studies demonstrated that Parthenium biomass treated with concentrated H2SO4 led to activated carbon with improved adsorption capacities for methylene blue (MB) [19]. Another approach to modification is grafting functional monomers onto fiber surfaces, such as the addition of acrylic acid to introduce additional –COOH groups. Although these modifications significantly enhance adsorption capacities, they fall outside the scope of "low-cost" preparation techniques and were not the focus of interest for most studies on natural biosorbents. A less severe approach, as practiced by Banerjee et al. (2014), was the citric acid treatment of *Lantana camara*, which serves as a crosslinker to introduce carboxylic functionality to the surface [10]. The Lantana treated with citric acid had higher MB removal than the untreated biomass, thus demonstrating the value of introducing functional groups that show strong interaction with dyes by ionic or hydrogen bond interactions.

In summary, the preparation of plant-based biosorbents typically involves a cleaning step (washing, solvent extraction), a chemical treatment (alkali or acid) to enhance surface chemistry, and size reduction (milling) to increase surface area. Each of these steps has been reported in the literature to significantly improve dye adsorption performance. For P H and L C, studies have employed some of these techniques. Mulugeta & Lelisa (2014) utilized raw Parthenium powder (after drying and grinding) as an adsorbent and still achieved notable dye removal [9]; this suggests that even untreated weed possesses inherent adsorption capability, likely due to its natural porosity and functional groups. Nonetheless, subsequent studies have applied alkali-treated Parthenium adsorbents, which demonstrated higher capacities, attributed to the deprotonation of its phenolic groups and increased surface area [15]. For *Lantana camara*, Amuda et al. (2014) initially carbonized the stems at 300 °C and then steam-activated them to develop porosity [12]. The resulting activated carbon had an average pore size of approximately 20 µm and efficiently adsorbed MB, with a capacity near 20 mg/g [12]. This indicates that thermal activation can create a network of micropores and mesopores in Lantana, augmenting van der Waals and π–π interactions in addition to any surface functional group attractions. Importantly, the choice of preparation method can influence not only capacity but also kinetics. A highly porous, small-particle adsorbent is likely to reach equilibrium faster due to shorter diffusion paths but may be more challenging to separate from the solution. Researchers must balance these considerations depending on the intended application (batch treatment vs. column filters). The literature suggests that for invasive weed biosorbents, relatively simple treatments like alkali washing can yield a material that is both high-performing and still low-cost, as these chemicals are inexpensive and the processes are straightforward.

2.4 Adsorption Studies on *Parthenium hysterophorus*

Several researchers have explored the use of *Parthenium hysterophorus* (commonly known as congress grass) as a biosorbent for pollutants, leveraging its status as agricultural waste. Hem Lata and colleagues (2007) were among the first to study dye adsorption using P H [12]. They tested white P H plant powder for the removal of MB from aqueous solutions. The weed was simply dried, ground, and sieved to produce a fine powder. Their findings indicated that P H biomass could remove a basic dye with efficiency comparable to other agricultural wastes. Key findings included an optimal pH of around 8–9 for MB adsorption, consistent with the behaviour of cationic dyes, a rapid initial uptake within the first 30–60 minutes, and an equilibrium capacity of approximately 10–20 mg/g for the untreated biomass. They also noted that the performance of P H as an adsorbent was influenced by the pretreatment of the plant—washing with deionized water and mild drying improved consistency in results, suggesting the removal of soluble impurities that might otherwise leach out and interfere [9]. In 2014, Mulugeta and Lelisa expanded on this by examining untreated P H weed for MB removal under various conditions. They observed that increasing the adsorbent dosage led to higher overall MB removal (%), although the adsorption capacity per unit mass decreased at very high dosages due to site aggregation and interference [9]. This is a common trend, as more adsorbent provides more sites to capture dye but can also cause partial overlap or crowding of sites. Mulugeta and Lelisa reported an optimal dosage beyond which removal efficiency plateaued, and they also optimized agitation speed and contact time. Their study confirmed that P H weed, without any chemical activation, could achieve over 80% removal of MB from a 25 mg/L solution in a matter of hours [16]. While they did not report a precise qmax from an isotherm, the data correspond to an estimated capacity of around 18–20 mg/g (as also listed in Table 2). This value is significant considering no costly treatment was applied. It underscores the natural adsorption potential of P H, likely due to its porous structure and active phytochemicals (the plant contains compounds like sesquiterpene lactones which might interact with dyes). Further studies on P H -based adsorbents have also explored modifications. For instance, P H was used to prepare activated carbon via acid treatment and pyrolysis for the removal of another dye, Rhodamine-B [17]. Although that study targeted a different dye, the resulting activated carbon had a much higher surface area and showed excellent adsorption, suggesting that a similar approach could greatly enhance MB uptake as well. Another innovative approach was the conversion of P H to biochar and magnetizing it (loading with Fe_3O_4) to create a magnetically separable adsorbent [18]. Meseret et al. (2020) developed magnetite-doped P H biochar, which could be easily retrieved with a magnet after treating dye solutions [19]. They found that the magnetic biochar maintained high MB removal efficiency (around 95%) and could be reused multiple times with only a slight loss of capacity. Although magnetization is an extra step, it addresses a practical aspect: the separation of spent biosorbent from treated water.

According to the literature, one notable advantage of *Parthenium hysterophorus* is its high cellulose content (exceeding 30–40%) and significant lignin content (~10–15%), which give it a rigid, porous structure [20]. When treated with alkali, much of the hemicellulose (and some lignin) is removed, resulting in a structure akin to cellulosic gels that can swell in water and offer numerous binding sites. P H also naturally contains surface hydroxyl and carbonyl groups (due to its plant cell wall composition and residual tannins) that aid in dye binding. FTIR analyses in chromium adsorption studies have demonstrated that P H activated carbon shows peaks corresponding to O–H, C=O, and aromatic C=C groups, which shift after metal uptake [21]. By analogy, similar shifts are anticipated when MB is adsorbed, indicating the involvement of these groups in binding. In conclusion, *Parthenium hysterophorus* has evolved from being tested in its raw form to more advanced forms (such as activated carbon and magnetic composite) as an adsorbent. Even in its raw form, it can effectively remove MB from water, although its capacity might be moderate. Treatments like alkali activation significantly enhance its performance. The weed's invasiveness ensures it is inexpensive and widely available, making it an excellent candidate for development into a commercial biosorbent if consistent performance can be achieved. The literature confirms that utilizing P H not only aids in dye removal but also helps mitigate the environmental threat posed by the weed itself, creating a win-win situation.

2.5 Adsorption Studies on *Lantana camara*

Lantana camara, a plant traditionally regarded as invasive, has garnered attention for its potential in biosorption applications. This woody shrub is characterized by a complex matrix of cellulose in its stems and leaves, along with various organic compounds. Historically considered a pest, *Lantana camara* is now being explored for innovative uses, such as a source of activated carbon and adsorbents. Omotayo Amuda and colleagues (2014) conducted a significant study in which *Lantana camara* stems were transformed into activated carbon for the removal of methylene blue (MB) [22]. In their methodology, dried Lantana stems underwent carbonization at 300 °C, followed by steam activation at elevated temperatures. The resultant steam-activated *Lantana camara* carbon (SALC) exhibited a well-developed porous structure, with a BET surface area comparable to that of commercial activated carbons. Scanning electron microscopy (SEM) images revealed a network of macropores and mesopores approximately 20 µm in size. This activated carbon demonstrated rapid adsorption kinetics for MB, achieving equilibrium within approximately 60 minutes, with a removal efficiency exceeding 90% for initial MB concentrations ranging from 50 to 200 mg/L [22]. The maximum adsorption capacity (qmax) derived from the Freundlich isotherm was approximately 19.84 mg/g [22], which the authors noted is comparable to other low-cost adsorbents and could be enhanced with further activation. The study also found that adsorption was favoured at higher pH levels and temperatures, suggesting an endothermic process potentially driven by chemical interactions or increased pore diffusion at elevated temperatures [22]. The MB adsorption on Lantana carbon conformed well to a Freundlich isotherm ($R^2 \approx 0.99$) and pseudo-second-order kinetics ($R^2 \approx 0.99$) [22]. The high R^2 values for the Langmuir isotherm (0.995–0.999) indicated that monolayer coverage was a reasonable assumption, despite the heterogeneous surface [1]. The authors concluded that *Lantana camara* stems, typically considered a useless weed, can be effectively utilized to produce activated carbon for the treatment of dye-laden wastewater [22]. Another study by S. Banerjee et al. (2014) adopted a different approach by employing *Lantana camara* in a less energy-intensive manner [10]. They modified raw Lantana biomass with citric acid under heat, a process that attaches citrate molecules to the fibre, introducing multiple carboxylic acid groups. This "green" modification circumvents the need for high-temperature activation. The citric acid-modified Lantana (referred to as CLB in their study) was evaluated for MB removal in batch mode, demonstrating improved performance over unmodified Lantana, particularly at neutral to alkaline pH levels [10]. Fourier-transform infrared (FTIR) spectra revealed new peaks corresponding to ester linkages from citric acid, and a shift in –OH stretching frequency following MB adsorption, indicating MB binding at those sites [10]. Kinetic analysis indicated pseudo-second-order behaviour, and thermodynamic parameters suggested the process was endothermic and spontaneous at higher temperatures [10]. Although the Langmuir isotherm capacity for the modified Lantana was not explicitly stated in the abstract, the enhanced removal suggests it was likely in the range of 25–30 mg/g. This value surpasses many raw biosorbents, underscoring that even without producing activated carbon, chemical modification of Lantana fibre can yield an effective sorbent.

Beyond its application in dye adsorption, *Lantana camara* has been investigated for its efficacy in heavy metal removal, such as Cr (VI) adsorption following acid treatment of Lantana leaves [23]. Research consistently indicates that the adsorption capacity of Lantana can be significantly enhanced through treatments that either increase its surface area or introduce functional groups. The stems of Lantana contain lignin, which can form char upon heating; this char provides a π-electron system conducive to dye uptake. Conversely, Lantana leaves are rich in polyphenols and terpenoids, which may contribute to adsorption through specific chemical interactions, although leaves are less frequently utilized due to their propensity to leach organics. Comparatively, *Lantana camara* and *Parthenium hysterophorus* exhibit similarities as adsorbents: both are invasive weeds, lignocellulosic in nature, and have demonstrated methylene blue (MB) removal capabilities in the tens of mg/g range in their untreated forms. However, the woody stems of Lantana facilitate its conversion into activated carbon more readily, as it can withstand carbonization. In contrast, Parthenium, being more herbaceous, may yield less char upon pyrolysis. Consequently, Parthenium is often employed in its raw or simply treated form, whereas Lantana has been explored both in its raw state and as activated carbon. The literature on Lantana biosorbents is still developing, yet the findings are promising. A notable advantage is that *Lantana camara* is abundantly available in tropical regions where it is an invasive species; utilizing it as an adsorbent could provide a cost-effective material while promoting its removal from natural ecosystems. The overarching conclusion from previous studies is that *Lantana camara* can serve as a viable biosorbent for MB, particularly when subjected to basic pretreatments or activation. This exemplifies the broader principle that nuisance plants can be transformed into valuable tools for pollution control.

2.6 Other Natural Fiber Adsorbents: A Comparative Context

To contextualize the performance of Parthenium and Lantana, it is useful to compare them with other natural fibre-based biosorbents studied for MB removal. Numerous agricultural by-products have been tested over the past decade, each with varying degrees of success. We will briefly review a few representative examples: jute fibre, coconut coir, banana fibre, sawdust, and others (some of which were listed in Table 2 earlier).

- **Jute Fiber:** Jute is a long bast fibre derived from the jute plant, rich in cellulose (≈60%) and hemicellulose (≈20%). Senthilkumaar et al. (2005)

produced activated carbon from jute fibre through phosphoric acid activation. The resulting jute fibre carbon exhibited an exceptionally high Langmuir capacity of 225.64 mg/g for MB, ranking among the highest reported for a biosorbent. This impressive capacity is attributed to the high surface area created by chemical activation and the graphitic nature of the carbon formed. The study also noted that MB adsorption on jute carbon was optimal at pH levels of 5–10, following the Langmuir isotherm and intraparticle diffusion kinetics [7]. While this capacity significantly surpasses those of Parthenium or Lantana adsorbents, it is crucial to recognize the difference in preparation: activated carbon versus raw/modified fibre. This highlights the upper limit of what a lignocellulosic precursor can achieve when thoroughly activated. Additionally, raw jute fibre (non-carbonized) has been tested in composite forms, such as being embedded in hydrogels, for MB removal, demonstrating capacities around 30–50 mg/g in those forms [8].

- **Coconut Coir Pith:** Coir pith, a corky particulate material derived from coconut husk, is often regarded as waste in the coir fibre processing industry. Abdullah Al Ashik et al. (2023) investigated the use of raw coconut coir dust as a cost-effective adsorbent. The coir dust was utilized in its natural form, merely sieved into different particle sizes. It achieved approximately 88–93% removal of MB from a ~32 mg/L solution at pH 9, with smaller particles demonstrating superior performance due to their increased surface area. When the adsorption capacity of coir dust was modelled using Langmuir, it was found to be around 80 mg/g for the smallest particle fraction at 30 °C. This is notably impressive for an untreated material. FTIR analysis revealed that lignin and cellulose functional groups participated in binding MB, as evidenced by shifts in –OH and –C–O bands post-adsorption [1]. Coir contains lignin (≈45%), which likely provides aromatic sites for π interactions and also features exchangeable cation sites. The study on coir underscores that even without chemical activation, certain fibres can inherently exhibit a high affinity for MB, potentially due to their microstructural characteristics. It also reinforces the earlier point that reducing the adsorbent particle size can significantly enhance performance.

- **Banana Fiber and Peel:** Banana pseudo-stem fibre and banana peels, both rich in cellulose (and starches in the peel), have been extensively studied. Md. Wasikur Rahman et al. (2022) explored the use of banana pseudo-stem fibres for MB adsorption. They treated the fibres with alkali, achieving a capacity of approximately 42 mg/g (Langmuir), as previously noted [8]. Kinetic studies revealed a two-stage adsorption process: an initial rapid phase (surface adsorption) followed by a slower intra-fibre diffusion phase [8]. Conversely, banana peel contains pectin and some proteins, offering different functional groups. One study reported that banana peel powder could remove MB up to about 20 mg/g [22]. While the performance of banana-derived adsorbents is moderate, their abundant availability as food industry waste makes them appealing. Researchers often enhance banana fibres by converting them to activated carbon or modifying them with acid to improve capacity beyond 100 mg/g, but in their raw form, they align with the capacity range of P H and L C.
- **Sawdust (Wood Waste):** Sawdust from various woods (teak, oak, pine, etc.) has long been tested for dye removal. Garg et al. (2004) used Indian rosewood sawdust to remove MB and got about 30 mg/g capacity for the raw sawdust. They improved this by treating the sawdust with formaldehyde (to prevent it from leaching organic matter) and with sulfuric acid (to increase surface area), which nearly doubled the adsorption capacity [24]. Sawdust primarily acts through surface adsorption and some ion-exchange if tannins are present. It’s an inexpensive adsorbent but usually requires modification to be highly effective. Nonetheless, its

performance in raw form still provides a useful benchmark: raw Parthenium (15–20 mg/g) and Lantana AC (~20 mg/g) are in the same league as many raw sawdust adsorbents.

- **Other Notable Materials:** Various other agricultural wastes have been documented, such as sunflower seed hull activated carbon (Hameed, 2008), which has a capacity of approximately 150 mg/g as previously mentioned [1]. Sugarcane bagasse in its raw form (Zhang et al., 2011) shows a capacity of about 34 mg/g for MB [22]. Luffa cylindrica (loofah sponge) fibres (Kesraoui et al., 2016) achieved around 88 mg/g for MB after slight modification. Pineapple leaf powder (Rahmat et al., 2016) demonstrates moderate MB sorption. Rice husk and its ash typically exhibit lower MB affinity (~5–10 mg/g for untreated husk), but this increases significantly for rice husk ash or activated carbon. Each material's unique composition affects its interaction with MB. For example, rice husk contains silica, which does not significantly aid in MB adsorption, resulting in its lower capacity in raw form [25]. In contrast, loofah fibre, being almost pure cellulose, can swell in water, exposing numerous –OH groups, which can lead to substantial MB uptake.

From this comparative perspective, we deduce that Parthenium and Lantana biosorbents, particularly when modestly treated, stand out among the array of natural adsorbents. While they may not surpass activated carbons derived from specialized precursors, they offer practical advantages, such as being cost-free raw materials, readily available, and providing the dual benefit of weed management. Furthermore, both can potentially be enhanced (through activation or chemical modification) to outperform many conventional biosorbents. The literature frequently highlights that although the initial capacities of raw materials might appear modest, the true benefit of biosorbents lies in their cost-effectiveness and abundance. In a full-scale application, it is feasible to use larger quantities of an inexpensive biosorbent to achieve the desired removal, a strategy not viable with costly commercial carbons.

2.7 Characterization and Analytical Techniques in Biosorption Studies

Literature on biosorbents uniformly shows the importance of material characterization in explaining adsorption results. Key techniques and their typical findings are:

- **FTIR (Fourier Transform Infrared Spectroscopy Fourier Transform Infrared Spectroscopy:** is employed to identify functional groups present on the biosorbent and to detect alterations following adsorption. For instance, in the cases of Parthenium and Lantana, FTIR spectra typically display broad bands around 3200–3400 cm−1 (O–H/N–H stretching), 1700–1740 cm−1 (C=O stretching of carboxyl or ester), 1600 cm−1 (aromatic C=C or conjugated C=O), and 1050–1150 cm−1 (C–O stretching in alcohols or ethers) [22][23]. Subsequent to methylene blue (MB) adsorption, shifts in these peaks or changes in intensity indicate interactions. Amuda et al. (2014) observed shifts in the O–H and C≡N (from lignin aromatic rings) bands of Lantana carbon post-MB adsorption, indicating the involvement of these groups in binding MB [22]. Similarly, it is anticipated that Parthenium would exhibit a decrease in the intensity of –OH bands and a shift in the C=O band after MB loading, as MB may form hydrogen bonds or electrostatic interactions with these groups. FTIR thus provides molecular-level evidence of adsorption mechanisms; for example, the emergence of new peaks could suggest the retention of MB molecules (or functional moieties of MB) on the surface, although typically, one looks for shifts rather than new peaks for physisorption.

- **Surface Morphology (SEM/TEM):** Scanning Electron Microscopy (SEM) provides detailed images of the adsorbent surface. Research frequently reveals a rough, porous surface in plant-derived adsorbents. In the study by Angassa et al. (2020) on Parthenium activated carbon, SEM images prior to adsorption displayed an irregular, pitted surface, whereas post-Cr (VI) adsorption, the pores appeared obstructed or covered, corroborating the material's uptake onto the surface. For dyes, SEM can similarly illustrate the deposition of dye molecules, which may manifest as a coating or altered texture on the biosorbent. Energy Dispersive X-ray Spectroscopy (EDS) coupled with SEM can be employed to detect elements such as sulphur or nitrogen from MB on the surface, further substantiating dye adsorption. In the case of Lantana carbon, SEM was instrumental in confirming its pore structure (notably, ~20 µm pores) that facilitated MB diffusion [23].
- **Surface Area and Porosity (BET Analysis):** Scanning Electron Microscopy (SEM) provides detailed images of the adsorbent surface. Research frequently reveals a rough, porous surface in plant-derived adsorbents. In the study by Angassa et al. (2020) on Parthenium activated carbon, SEM images prior to adsorption displayed an irregular, pitted surface, whereas post-Cr (VI) adsorption, the pores appeared obstructed or covered, corroborating the material's uptake onto the surface. For dyes, SEM can similarly illustrate the deposition of dye molecules, which may manifest as a coating or altered texture on the biosorbent. Energy Dispersive X-ray Spectroscopy (EDS) coupled with SEM can be employed to detect elements such as sulphur or nitrogen from MB on the surface, further substantiating dye adsorption. In the case of Lantana carbon, SEM was instrumental in confirming its pore structure (notably, ~20 µm pores) that facilitated MB diffusion [23].

- **Point of Zero Charge (pH_{zpc}):** • The pH_{zpc} the pH at which the adsorbent's net surface charge is zero. Alkaline treatments often raise the pHzpc of biomass by deprotonating acidic groups. Coir dust was found to have pH_{zpc} ~7.65[1], meaning at pH >7.65 the surface is negative (favouring MB adsorption), and at pH <7.65 it is positive (MB adsorption less favoured due to electrostatic repulsion). Knowing pH_{zpc} helps explain why an adsorbent works best at certain pH values. If Parthenium or Lantana fibre has, say, pH_{zpc} ~6 (typical for many plant materials), experiments show MB uptake is much greater above that pH [1]. Researchers thus often adjust solution pH to around 8 in batch tests to maximize dye removal, as noted across many studies.
- **UV-Visible Spectrophotometry**: The primary method for determining methylene blue (MB) concentration in solution, both prior to and following adsorption, involves spectrophotometric analysis. MB exhibits a pronounced absorption peak in the visible spectrum, approximately between 664 and 668 nm. By observing the reduction in absorbance at $\lambda \approx 665$ nm, the extent of MB removal can be quantified. Numerous studies, including those focusing on Parthenium and Lantana, present spectra illustrating the reduction of the characteristic MB peak subsequent to treatment with the biosorbent [9][4]. Occasionally, a minor shift in the peak wavelength is noted, which may occur due to the stacking (dimerization) of MB molecules or alterations in the local microenvironment; however, Beer's law is generally applicable for quantification. UV-Vis analysis is a straightforward technique and has been employed in all referenced studies to calculate adsorption capacities using calibration curves.
- **Other techniques**: In advanced research, X-ray Photoelectron Spectroscopy (XPS) is frequently employed to examine surface elemental states, thereby confirming the binding of methylene blue (MB), as evidenced by the detection of the N1s peak of MB on the adsorbent.

Thermal analysis techniques, such as Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC), are utilized to assess changes in thermal stability following dye adsorption, with the potential for additional weight loss steps due to the loaded dye. Furthermore, Vibrating Sample Magnetometry (VSM) is applicable for magnetic biosorbents, although this is typically beyond the scope of foundational studies.

In summary, the literature offers a comprehensive set of tools for characterizing biosorbents and elucidating adsorption mechanisms. These methodologies facilitate the interpretation of interactions between Parthenium and Lantana fibres and MB. For instance, in our forthcoming experiments, we anticipate that Fourier Transform Infrared Spectroscopy (FTIR) will confirm the presence of functional groups such as –OH and –COOH on treated Parthenium/Lantana and reveal changes post-MB binding, potentially indicated by a new peak around 1580 cm−1, suggesting aromatic ring interactions. Scanning Electron Microscopy (SEM) is expected to display the porous, irregular structure of the fibres and any alterations following alkaline treatment, such as increased fibrillation and reduced surface smoothness. The characterization results from existing literature will inform the analysis of our experimental data.

2.8 Summary of Literature Findings

The literature reviewed indicates that natural fibre biosorbents are a viable solution for dye removal, offering a combination of efficacy and low cost. *Parthenium hysterophorus* and *Lantana camara* specifically have demonstrated the following in prior studies:

- **Adequate Baseline Performance**: Both weed biomasses, even in their untreated powder form, demonstrate a notable capacity to adsorb MB from aqueous solutions, with adsorption capacities ranging from 10 to 20 mg/g and achieving over 80% removal in low-concentration solutions [9][2]. This suggests that these biomasses possess inherent adsorption potential that can be further developed.

- **Enhancement via Treatment:** Simple chemical treatments, such as alkaline hydrolysis for Parthenium and citric acid modification for Lantana, along with thermal activation, can significantly enhance their adsorption capacities, in some instances by a factor of 2–3[22] [10]. Consequently, the literature indicates a clear strategy for optimizing these biosorbents, primarily through the augmentation of accessible surface area and functional group density.
- **Comparative Analysis with Other Biosorbents:** The performance of P H and L C is comparable to that of numerous other agricultural waste materials. While they may not yet achieve the exceptionally high adsorption capacities characteristic of specialized activated carbons, their effectiveness is adequate for practical applications in the treatment of moderately concentrated dye effluents. Furthermore, in contexts where biomass is abundantly available, the absolute capacity can be compensated by utilizing a greater quantity of material.
- **Mechanistic Understanding:** Previous research employing spectroscopy and isotherm/kinetic modelling has elucidated the mechanisms by which these biosorbents function. The adsorption of MB onto these fibres is characterized by a combination of electrostatic interactions and surface adsorption. The application of Langmuir and Freundlich models has been successful, indicating predominantly monolayer coverage on heterogeneous sites [1][8]. The predominance of pseudo-second-order kinetics suggests that chemisorption is a significant factor [22]. These findings are consistent with observations for similar biosorbents and will guide the experimental design and analysis of this thesis research.

- **Gaps and Considerations:** Although Parthenium and Lantana have been individually studied, there is a notable absence of direct comparative research evaluating them under identical conditions. The literature also lacks comprehensive data on the regeneration of these specific biosorbents, specifically regarding how many adsorption-desorption cycles they can endure. Some studies suggest that desorption of MB from biosorbents can be achieved using dilute acid or organic solvents, but this requires confirmation for weed-based adsorbents. Another gap lies in scaling: most experiments have been conducted at a batch scale in flasks. There is limited information on how Parthenium or Lantana adsorbents perform in continuous flow systems or packed columns, which would be crucial for real wastewater treatment applications. These gaps highlight areas for future research beyond the scope of initial MTech studies, but they are noted as potential extensions of the knowledge from these studies.

In conclusion, the literature presents an encouraging outlook for using *Parthenium hysterophorus* and *Lantana camara* as biosorbents. They are effective, particularly when properly prepared, and they convert environmental liabilities (invasive weeds) into assets for pollution remediation. Building on these findings, the experimental phase of this thesis will focus on optimizing the preparation of these biosorbents and quantifying their performance in MB removal, while employing the discussed analytical techniques to uncover the underlying adsorption mechanisms. The following chapters will detail the materials and methods adopted for this research, followed by results and discussions that compare with the literature benchmarks summarized here.

Chapter 3: Materials And Methods

3.1 Materials

The materials used in this study include plant biomass, laboratory chemicals, and equipment for fibre preparation and sheet formation. The two invasive plant species selected for this work were *Parthenium hysterophorus* and *Lantana camara*, sourced locally from roadside areas of Masyana and Dumehar, Himachal Pradesh. These plants were chosen due to their high cellulose content and wide availability.

Fig. 2: *Lantana camara* and *Parthenium hysterophorus* weed

Chemicals and Reagents:

- Ethanol (Absolute)
- Sodium hydroxide (NaOH)
- Hydrogen peroxide (H_2O_2)
- Ethylene diamine tetraacetate (EDTA)
- Distilled water (DW)

Apparatus and Equipment:

- Grinder
- Soxhlet extractor and thimble
- Wooden frame with muslin cloth
- Beakers and glassware

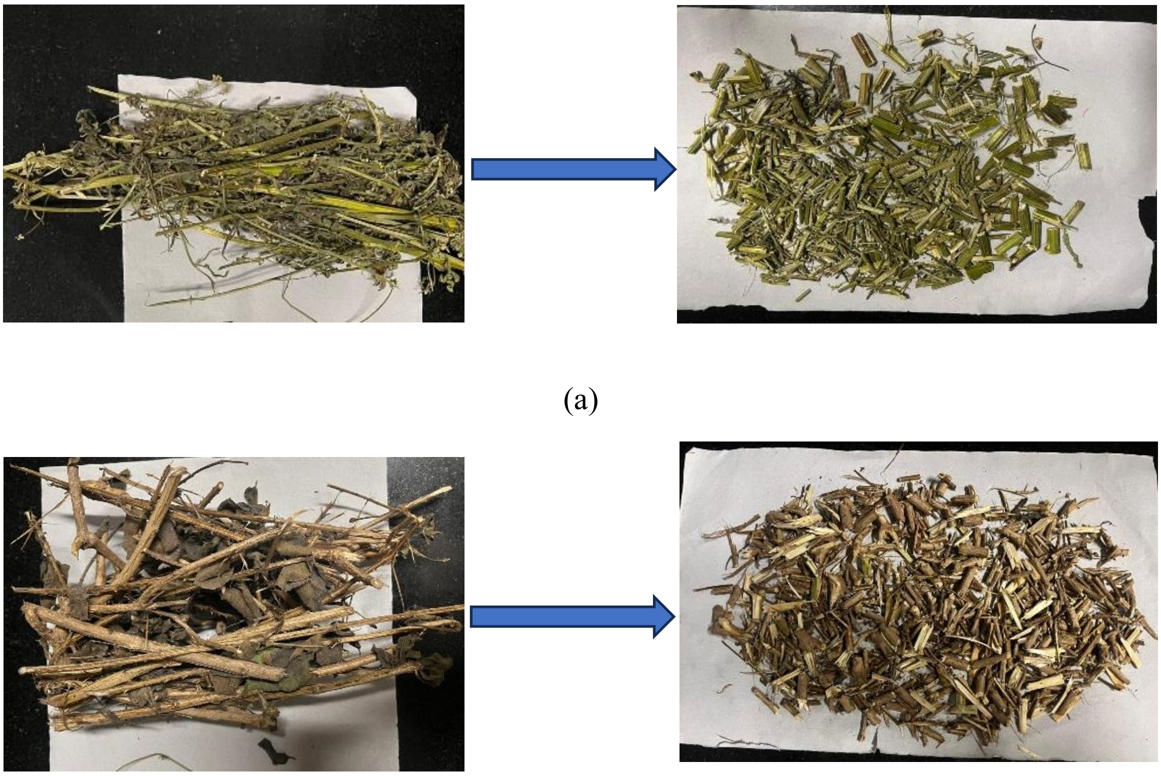
- Oven
- Pestle and mortar
- UV-Vis spectrophotometer
- FTIR spectrometer
- Inverted microscope

3.2 Methods:

3.2.1 Sample Preparation

Drying and Cutting

The collected plants were washed and shade-dried for 2 days, followed by overnight drying at 37°C in an oven. Once dried, the plant material was cut into 1–2 cm long pieces.

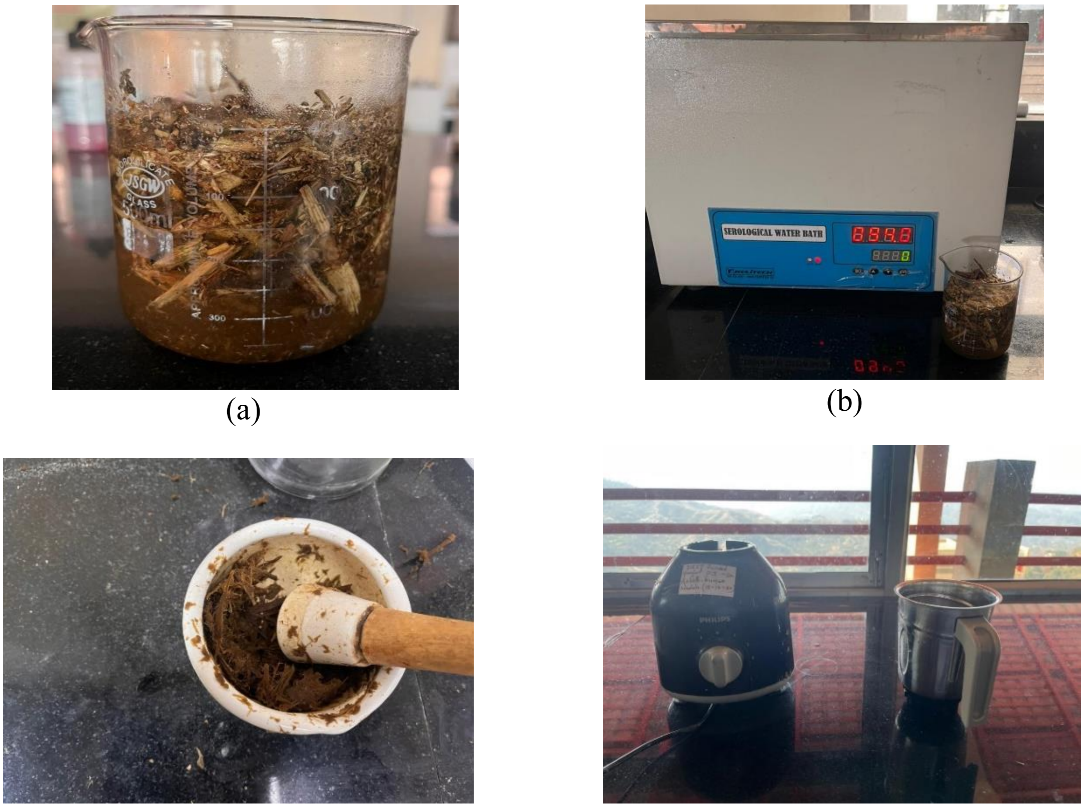

(a)

(b)

Fig. 3: Drying and cutting of (a) *Parthenium* and (b) *Lantana*

3.2.2 Pulp Extraction: Method 1 —Beating assisted Pulping

1. EDTA Pre-treatment: The chopped fibres were cooked in 0.5% EDTA at pH 5 and 95°C for 3 hours.
2. Alkali and Peroxide Treatment: The washed biomass was then treated with 8% NaOH and 2% H_2O_2 at 95°C for another 3 hours in a 1:8 solid-to-liquid ratio.
3. Beating and Grinding: The treated pulp was manually beaten using a pestle and mortar and further processed for 10–15 minutes in a grinder.
4. Washing: The pulp was thoroughly washed and suspended in water for sheet formation.


(a) (b)

(c) (d)

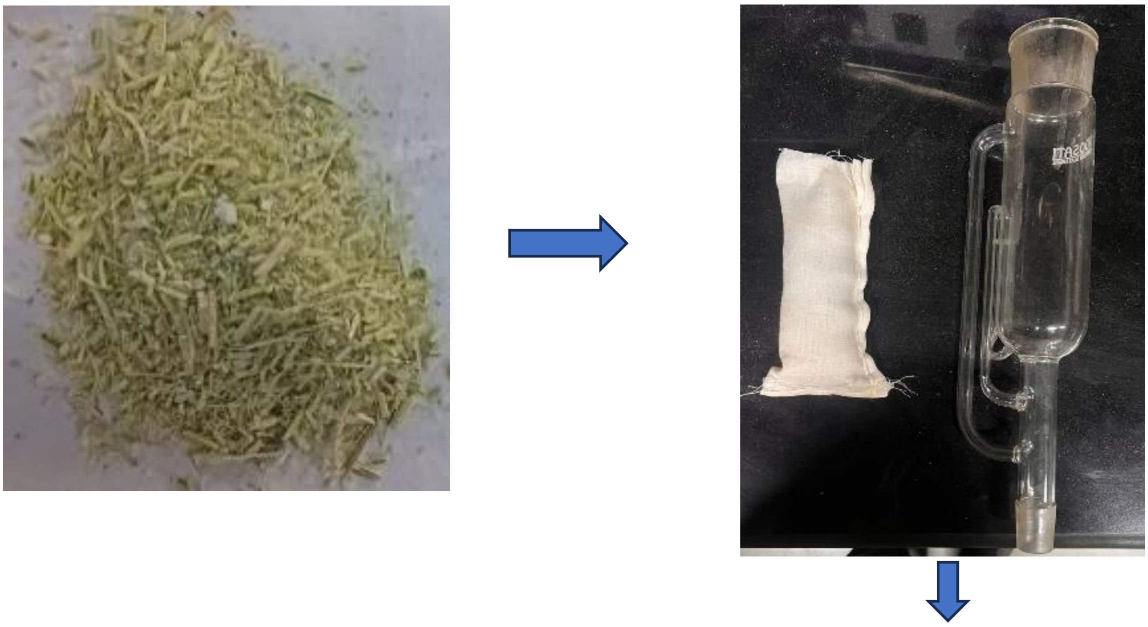
Fig. 4: (a) Pretreatment were given to the fibres, (b) Cooking of fibres are done with water bath at 95 ºC, (c) Beating is done manually by pestle mortar, (d) Grinding is performed using Philips grinder.

3.2.3 Pulp Extraction: Method 2 — Soxhlet-Assisted Pulping

In this method, dried and ground plant material was subjected to Soxhlet extraction with absolute ethanol for 6–7 hours to remove waxes, oils, and other extractives. The residual biomass was then treated using the same alkaline peroxide pulping process described above.

Before

Extractor Chamber and Thimble



After

Soxhlet Assembly

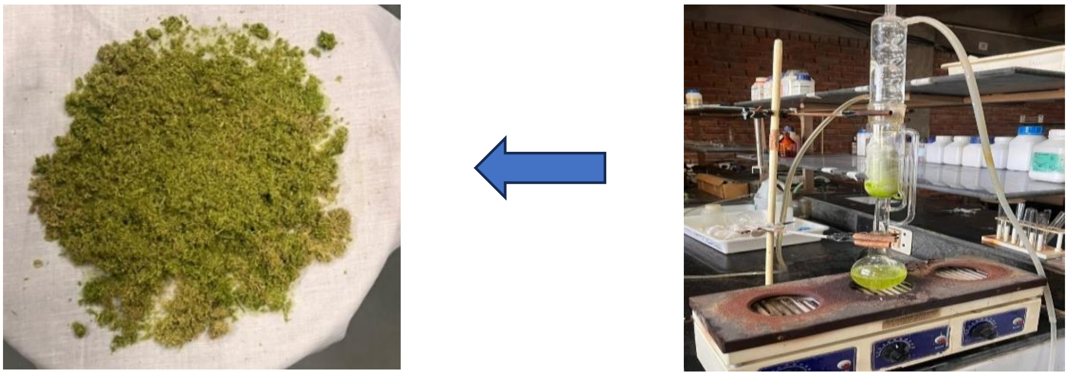


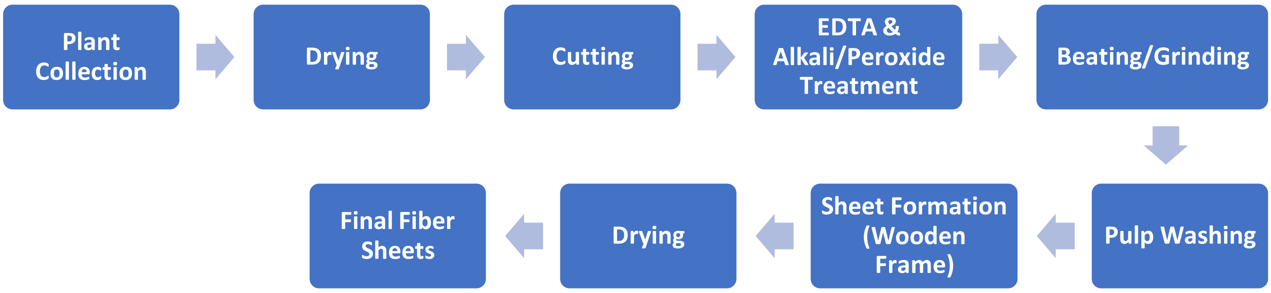
Fig. 5: Parts of Soxhlet assembly, before and after images of fibre

3.2.4 Sheet Formation

The pulp obtained from either method was mixed in water, and fibres were extracted using a wooden frame covered with muslin cloth. The frame acted as a sieve to capture the dispersed fibres.

1. Drying: The collected fibre mat was transferred to dry muslin cloth, pat-dried using blotting paper, and pressed overnight underweight.
2. Final Drying: The sheets were then air-dried for an additional day in shade.

Fig. 6: (a) Appearance of fibre initially, (b) Process of making sheets from fibres using water and wooden frame, (c) Photo of Dried sheet



Flowchart: Fiber Sheet Preparation Process

3.2.5 Adsorption Experiment

Dye Solution Preparation: Methylene blue (MB) stock solution of 0.01 M was prepared and diluted to achieve a working concentration of 12 mg/L (corresponding to 40 µL in 10 mL).

Batch Adsorption Tests: Paper strips (2 × 2 cm) were immersed in 20 mL distilled water. All experiments were conducted at room temperature for a constant contact time of 20 minutes with gentle agitation.

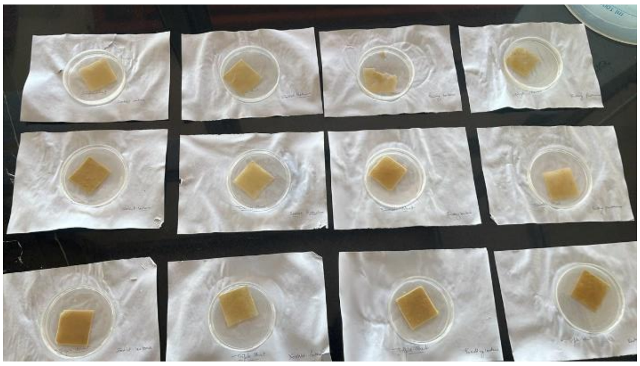


Fig. 7: Batch absorption test with distilled water

$$q_e = (C_o - C_e)\, V/W$$

Determination of Ce and Qe:

- Ce (equilibrium concentration) was determined using a UV-Vis spectrophotometer at $\lambda = 665$ nm.
- Qe (adsorbed amount) was calculated by: $Q_e = (Ci - Ce) \times V / m$ Where: Ci: Initial MB concentration (mg/L) Ce: Equilibrium concentration (mg/L) V: Volume of solution (L) m: Mass of the adsorbent (g)

3.2.6 Characterization Techniques

- FTIR (Fourier Transform Infrared Spectroscopy): Used to identify the functional groups (–OH, –COOH, etc.) on the fibre surface and confirm interaction with MB.
- Inverted Microscopy: Used to analyse the microstructure and surface morphology of the fibre sheets before and after dye adsorption.

3.2.7 Additional Measurements

- **pH Measurement:** The pH of fibres was determined using pH paper and a digital pH meter.
- **Absorption Capacity:** Determined by measuring the volume of dye solution absorbed per unit area of paper strip.
- **Wicking Test:** Strips were tested for capillary movement of dye solution (3 cm height strip test).

Chapter 4: Results and Discussion

4.1 Overview of Adsorption Data

Twelve samples derived from *Parthenium hysterophorus* and *Lantana camara* fibres were tested for their methylene blue adsorption performance. Sample sets were derived from two treatment methods and analysed for dye uptake over 20 minutes. The following key variables were determined:

- Initial dye concentration ($C_i = 12$ mg/L)
- Equilibrium concentration (C_e)
- Adsorption capacity (Q_e)
- Langmuir parameters (Q_{max}, KL)

4.1.1 Water absorption:

Water Absorption (%) = $[(W_1 - W_0) / W_0] \times 100\}$

Where:

- **W_0** = Initial dry weight of the sample (before water exposure)
- **W_1** = Final weight after absorbing water (typically after a set immersion time)

4.1.2 Percentage MB absorption:

% MB Removal = $[(C_o - C_e) / C_o] \times 100$

Where:

- C_i is the initial concentration of methylene blue in mg/L,
- C_e is the equilibrium concentration of methylene blue in mg/L.

Fig. 8: Colour intensity of MB increases on the strip as we move towards right we perform double, triple absorption

4.1.3 Adsorption capacity (Q_e):

$$Q_e = \frac{(C_o - C_e) \times Vol. of\ sol.}{Mass\ of\ strip}$$

4.1.4 Maximum monolayer adsorption capacity (Q_{max}):

The formula to calculate Q_{max} (maximum monolayer adsorption capacity) is derived from the Langmuir isotherm equation:

$$Q_e = \frac{Q_{max} \times K_L \times C_e}{1 + K_L C_e}$$

Where:

- Q_e = amount of adsorbate adsorbed at equilibrium (mg/g)
- Q_{max} = maximum adsorption capacity (mg/g)
- Ce = equilibrium concentration of the adsorbate in solution (mg/L)
- K_L = Langmuir constant (L/mg)

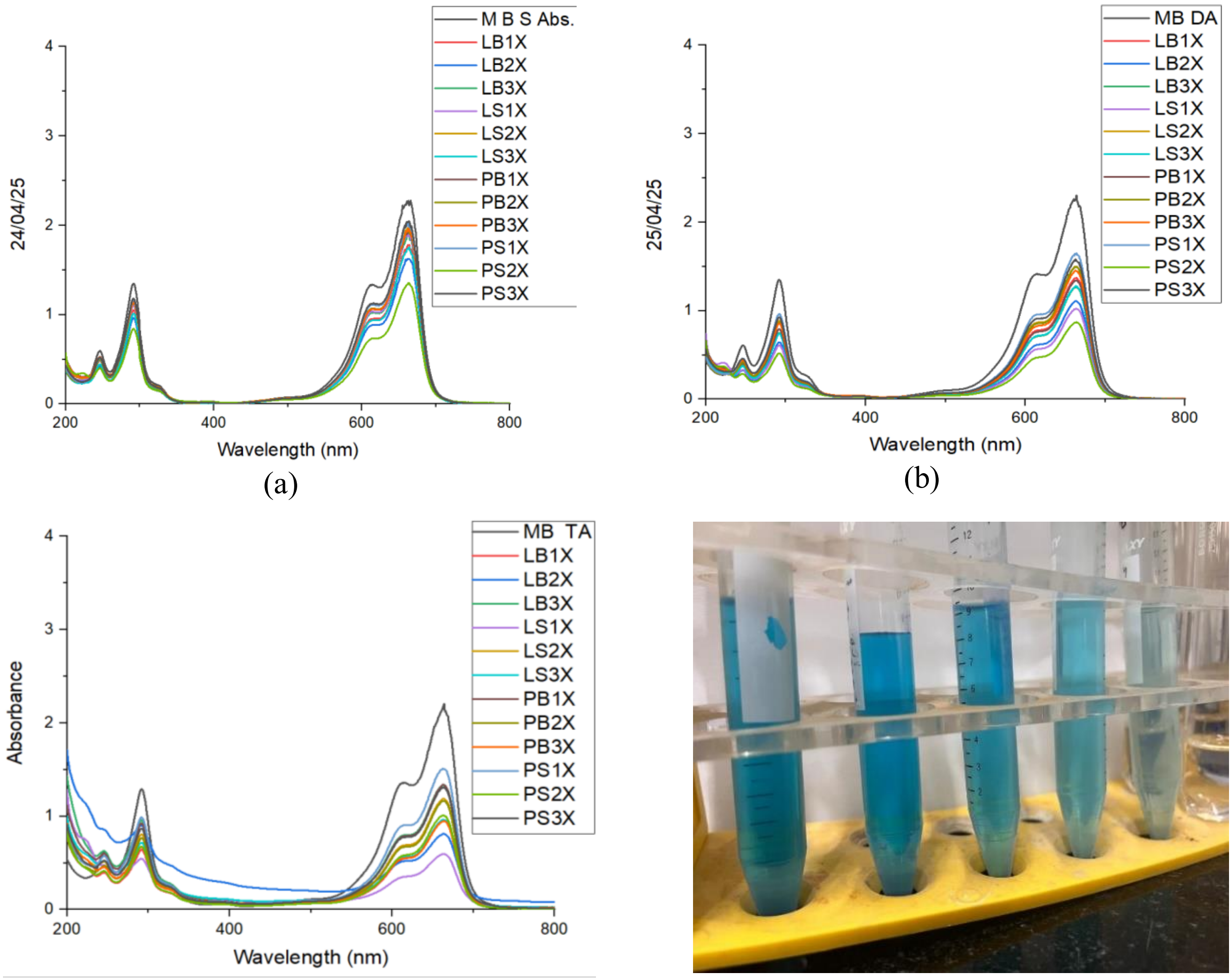
Note: Higher KL_{Value} indicates higher binding affinity

Table 3. Calculated values for MB %age Q_e ,

Samples	Co (M)	Ce(M)	(Co-Ce)	MB %age	Qe (mM/g)
PS1X	2.3747E-05	1.73E-05	6.45E-06	27.17199	0.001613
PS3X	2.3747E-05	1.65E-05	7.25E-06	30.54078	0.000558
LB2X	2.3747E-05	1.59E-05	7.89E-06	33.24468	0.001128
PB2X	2.3747E-05	1.57E-05	8.04E-06	33.86525	0.001149
PB3X	2.3747E-05	1.53E-05	8.47E-06	35.68262	0.000652
LB1X	2.3747E-05	1.44E-05	9.34E-06	39.31738	0.002334
PB1X	2.3747E-05	1.41E-05	9.63E-06	40.55851	0.002408
LS3X	2.3747E-05	1.34E-05	1.04E-05	43.70567	0.000798
LB3X	2.3747E-05	1.33E-05	1.05E-05	44.19326	0.000807
LB2X	2.3747E-05	1.17E-05	1.2E-05	50.57624	0.001716
LS1X	2.3747E-05	1.08E-05	1.29E-05	54.52128	0.003237
PS2X	2.3747E-05	9.14E-06	1.46E-05	61.52482	0.002087

4.2 UV-Vis Spectrophotometry Analysis

UV-Vis absorbance spectrum (200-800nm) was observed before and after adsorption and MB peak was considered corresponding to 665 nm. These values were converted to concentrations using a standard calibration curve. The percentage removal of methylene blue was calculated. Maximum MB removal efficiency for Parthenium fibres reached up to **61.5%**, confirming their functional adsorption capacity under test conditions.



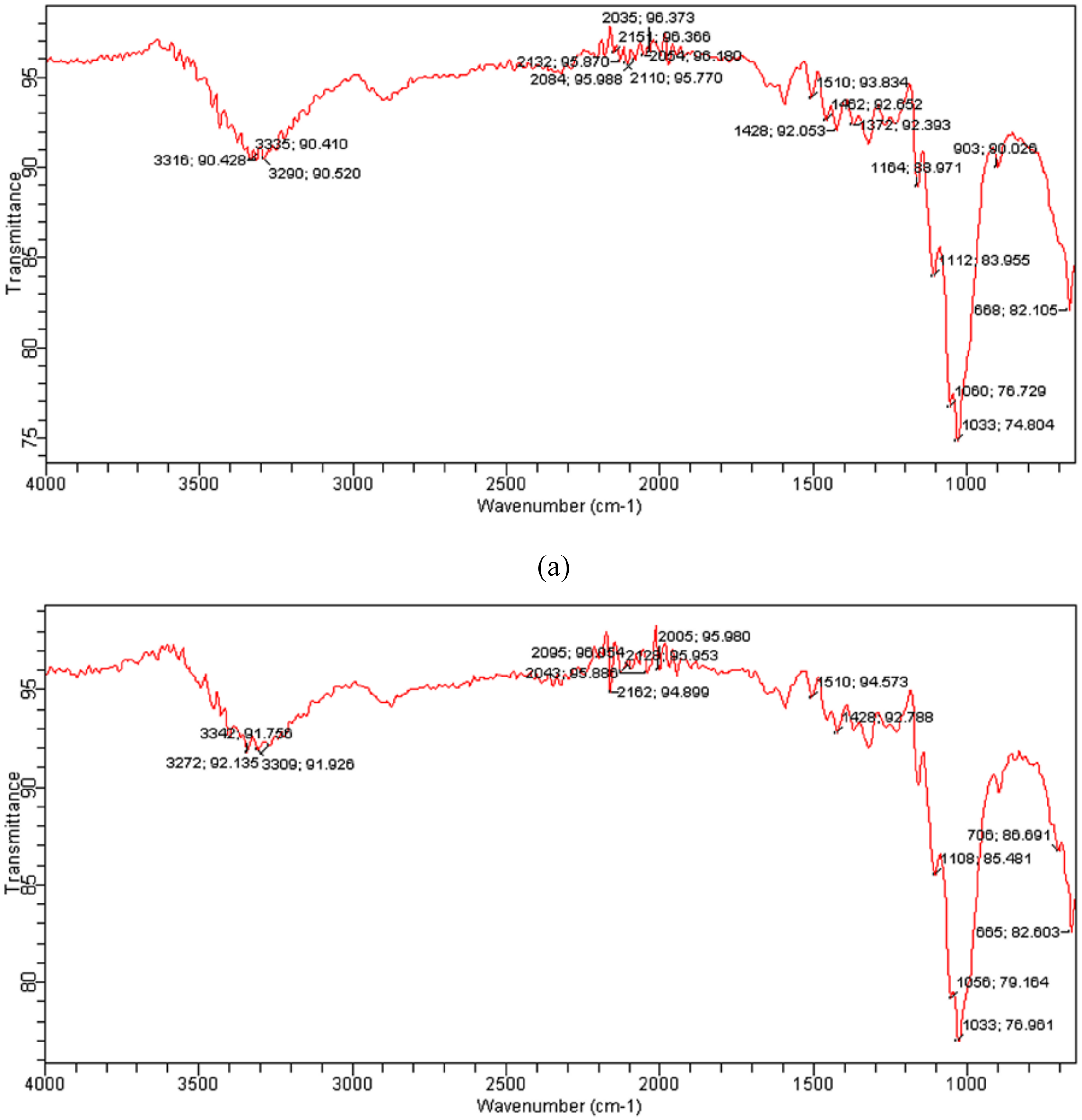
(a) (b)

(c) (d)

Fig. 9: (a)Uv-vis absorbance spectrum for single absorption of MB on fibers (b) Double absorption of MB on fibers (c) Triple absorption of MB on fibers and maximum MB removal efficiency for Parthenium fibres reached up to **61.5%** (d) Colour intensity of dye decreases from left to right as absorption on fibers increases

4.4 FTIR Spectroscopy Results

FTIR spectra identified functional groups on the fibre surface, notably –OH, –COOH, and C=O, all of which showed minor shifts after dye adsorption, suggesting chemical interaction with MB molecules.



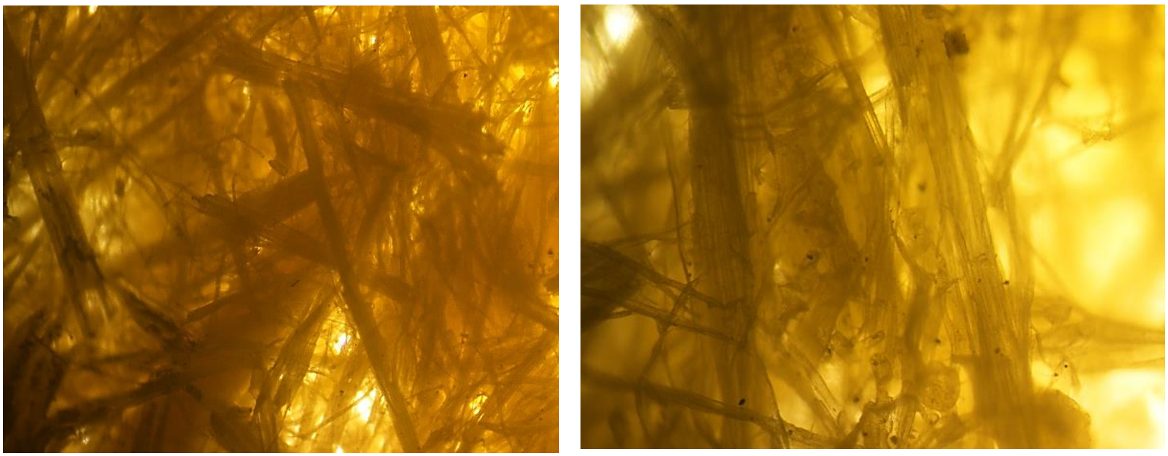
(a)

(b)

Fig. 10: FTIR plot representing stretching and bending of –OH, –COOH, and C=O functional groups in (a) Lantana, (b) Parthenium sheet

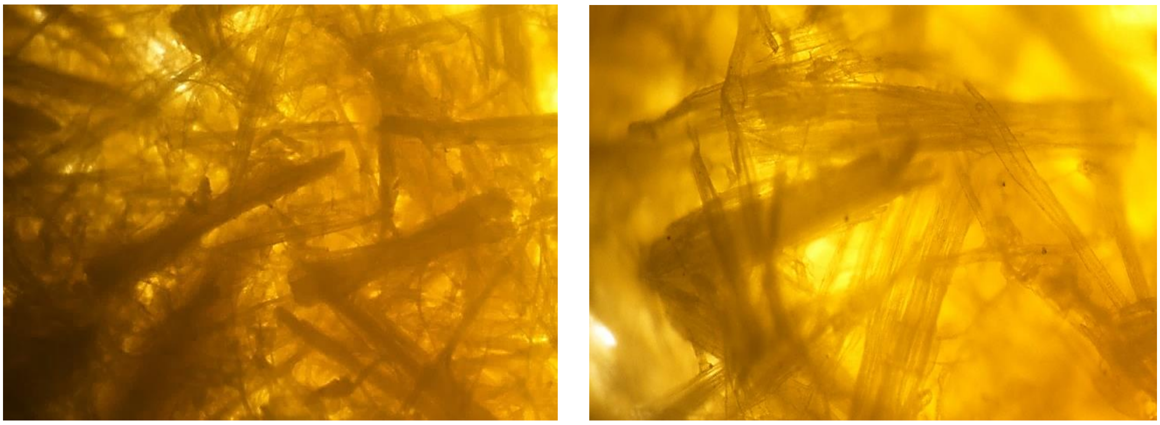
4.5 Microscopy Observations

Microscopy images (4× and 10×) before adsorption showed clear fibres distribution (random) and void between them.



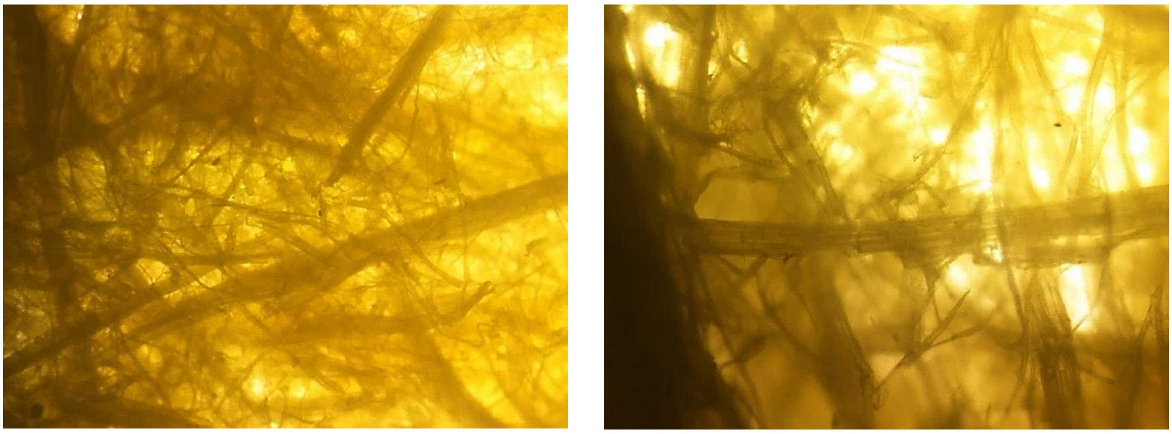
(4X) (10X)

(a) Lantana Beating Method



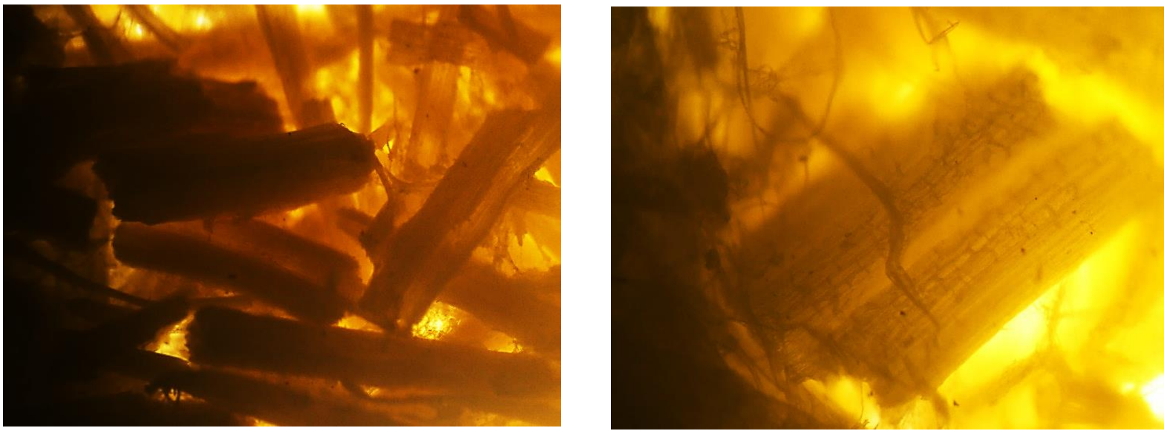
(4X) (10X)

(b) Lantana Soxhlet Method



(4X) (10X)

(c) Parthenium Beating Method



(4X) **(10X)**

(d) Parthenium Soxhlet Methods

Fig. 11: Inverted microscopic imaging of various samples under 4X and 10X magnification

4.6 Effect of pH, Absorption, and Wicking Behaviour

- **pH:** Adsorption was more effective at neutral to basic pH of Fibers.
- **Absorption Capacity:** Lantana fibres absorbed more solution but showed slightly lower dye retention.
- **Wicking:** Lantana strips showed faster capillary rise; however, Parthenium had better dye saturation.

4.7 Comparative Analysis of Fiber Types

- **Parthenium** fibres performed better in MB removal (up to 61.5% efficiency).
- **Lantana** fibres showed better structure and reusability potential.

4.8 Summary

The study confirms that both fibres act as viable biosorbents for cationic dye MB. Langmuir modelling supports monolayer adsorption behaviour.

Chapter 5: Conclusions and Future Work

Key Findings

- Parthenium fibre sheets achieved up to **61.5% MB removal**.
- FTIR confirmed involvement of hydroxyl and carboxyl groups.
- Microscopy image exhibited fibers voids and their arrangement on the sheet.
- Lantana absorbed more fluid but had slightly lower dye uptake.
- Eosin trials were excluded due to poor performance.
- Reuse capacity of fibers was evaluated by performing multiple absorption.

Limitations

- Small-scale batch setup may not reflect real wastewater behaviour.
- Only MB was effectively adsorbed; no performance against anionic dyes.
- Adsorption assumptions may not fully reflect real binding dynamics.

Future Work

- Modify fibres chemically (e.g., amination or crosslinking) to improve binding.
- Test reusability and regeneration with appropriate desorption agents.
- Expand to other dye types and pollutants, including heavy metals.
- Pilot-scale implementation with flow systems.
- Perform life-cycle assessment to evaluate sustainability.

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