

SUGARCANE BAGASSE AS NANOMAGNETIC BIOSORBENT (SB-NMB) COMPOSITE FOR THE REMOVAL OF ACID BLUE 9 FROM AQUEOUS SOLUTIONS

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

By

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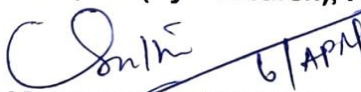
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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the Bonafide work of **ANKIT MYLARAPU (40190002), KALLA GIREESH VENKATA RAMANA (40190033)** who carried out the project entitled "**SUGARCANE BAGASSE AS NANOMAGNETIC BIOSORBENT (SB-NMB) COMPOSITE FOR THE REMOVAL OF ACID BLUE 9 FROM AQUEOUS SOLUTIONS**" under my supervision from NOVEMBER 2023 TO APRIL 2024.


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We, **ANKIT MYLARAPU** and **KALLA GIREESH VENKATA RAMANA** hereby declare that the Project Report entitled "**SUGARCANE BAGASSE AS NANOMAGNETIC BIOSORBENT (SB-NMB) COMPOSITE FOR THE REMOVAL OF ACID BLUE 9 FROM AQUEOUS SOLUTIONS**" done by us under the guidance of **Dr. D. Prabu, M.S (By Research), Ph.D.**, at Sathyabama Institute of Science and Technology is submitted in partial fulfilment of the requirements for the award of Bachelor of Technology degree in **CHEMICAL ENGINEERING**.

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ABSTRACT

Water pollution, which has become a major issue, is primarily caused by pollutants such as dyes, and water treatment techniques have become critical for addressing this issue. Adsorption was chosen among the several removal techniques as it is inexpensive, environmentally friendly, composed of renewable biodegradable materials, and offers a variety of adsorbent alternatives. In this study, Sugarcane Bagasse was impregnated with Nano Magnetic Particles (NMPs) utilising the Co-precipitation process to produce a Sugarcane Bagasse – Nanomagnetic Biosorbent (SB-NMB) composite to remove Acid Blue 9 which is a synthetic organic compound utilised as a blue colourant in pharmaceuticals and many other industries. Sugarcane Bagasse is a waste product with little economic value and has demonstrated excellent potential as a biosorbent due to its superparamagnetic properties. The characteristics of the biosorbent were investigated in this study using various characterisation techniques like FTIR which analysed the functional groups. SEM with EDX analysed the porous nature and the weight percentage of the particle. XRD showed the crystalline structure, VSM exhibited a Ferromagnetic signal with a rather dominating paramagnetic signal and BET showed the surface area to be 57.988 m²/g. Adsorption studies to remove acid blue 9 were conducted utilising a range of synthetic dye solution concentrations (20-100 mg/L), pH (2-7), adsorbent dosage (0.1-0.6g/L), and contact time (0-90 min). The adsorption isotherm and kinetics show that the Freundlich isotherm and Pseudo-second-order concepts offered the greatest fitting.

Thermodynamic studies prove that the adsorption mechanism was spontaneous and this investigation yielded removal % efficiency and capacity (mg/g) of 92.05% and 9.205 mg/g, respectively, therefore the study's findings offer a lot of promise for tackling water pollution concerns.

Keywords- Adsorption, water pollution, Acid Blue 9, SB-NMB.

TABLE OF CONTENTS

CHAPTER NUMBER	CONTENTS	PAGE NUMBER
	ABSTRACT	I
	TABLE OF CONTENTS	ii
	LIST OF ABBREVIATIONS	iv
	LIST OF FIGURES	v
	LIST OF TABLES	Viii
1	INTRODUCTION	1
	1.1 TYPES OF DYES	5
	1.1.1 Azo dyes	5
	1.1.2 Reactive dyes	6
	1.1.3 Metal complex dyes	6
	1.1.4 Sulphur dyes	7
	1.2 DIFFERENT TYPES OF ADSORBENTS	8
	1.2.1 Pomegranate peels	8
	1.2.2 Prawn Shells	9
	1.2.3 Eggshells	10
	1.2.4 Banana peels	11
	1.3. APPLICATIONS OF SB-NMB	12
2	LITERATURE SURVEY	13
3	AIM AND SCOPE	18
	3.1 AIM	18
	3.2 SCOPE	18
4	MATERIALS AND METHODOLOGY	19
	4.1 MATERIALS	19
	4.1.1 Chemicals Required	19
	4.1.2 Equipment Required	19
	4.2 METHODOLOGY	20
	4.2.1 Drying of Sugarcane Bagasse	20
	4.2.2 Preparation of Sugarcane Bagasse	20
	Activated Carbon	
	4.2.3 Impregnation process of SB-NMB	23

	4.2.4 Co-precipitation Process of SB-NMB	24
	4.2.5 Filtration and Drying of SB-NMB	25
	4.2.6 Magnetic Separation of SB-NMB	26
	4.3 PREPARATION OF A BLUE 9 ION SOLUTION	27
	4.4 ADSORPTION EXPERIMENT	27
	4.4.1 Adsorption Isotherm Experiments	28
	4.4.2 Adsorption Kinetic and Mechanism Experiments	31
	4.4.3 Adsorption Thermodynamic Experiments	32
5	RESULTS AND DISCUSSION	34
	5.1 CHARACTERIZATION OF SB-NMB	34
	5.1.1 Fourier Transform Infrared Spectroscopy	34
	5.1.2 Scanning Electron Microscopy	35
	5.1.3 Energy Dispersive X-Ray	37
	5.1.4 X-ray diffraction	39
	5.1.5 Vibrating Sample Magnetometry	40
	5.1.6 Brunauer-Emmett-Teller	41
	5.2 OPTIMIZATION PARAMETERS	42
	5.2.1 Effect of pH of SB-NMB	43
	5.2.2 Effect of Contact Time of SB-NMB	43
	5.2.3 Effect of Concentration of SB-NMB	45
	5.2.4 Effect of Temperature of SB-NMB	46
	5.2.5 Effect of Dosage of SB-NMB	47
	5.3 ADSORPTION ISOTHERM	48
	5.4 ADSORPTION KINETICS AND MECHANISM	50
	5.5 THERMODYNAMIC PARAMETERS	52
	5.6 CHARACTERIZATION AFTER ADSORPTION	55
	5.6.1 Fourier Transform Infrared Spectroscopy	55
	5.6.2 Scanning Electron Microscopy	56
	5.6.3 Energy Dispersive X-Ray	58
6	SUMMARY AND CONCLUSION	60
	REFERENCES	61

LIST OF ABBREVIATIONS

ABBREVIATIONS	ACRONYMS
SB	Sugarcane Bagasse
SB-AC	Sugarcane Bagasse - Activated Carbon
RR120	Reactive Red 120
SB-NMB	Sugarcane Bagasse - Nanomagnetic Biosorbent
NMB	Nanomagnetic Biosorbent
SEM	Scanning Electron Microscopy
FTIR	Fourier Transform-Infrared Spectroscopy
XRD	X-ray diffraction
EDX	Energy dispersive X-ray
VSM	Vibrating Sample Magnetometer
BET	Brunauer-Emmett-Teller

LIST OF FIGURES

FIGURE NUMBER	FIGURE NAME	PAGE NUMBER
1.1	Chemical Structure of Acid Blue 9	1
1.2	Dye wastewater released into nearby water bodies	2
1.3	Chemical Structure of Sugarcane Bagasse	3
1.4	Chemical structure of an orange-coloured azo dye	5
1.5	Chemical structure of Reactive Red 120 dye	6
1.6	Chemical structure of Metal Complex dye	6
1.7	Chemical structure of Sulfur dye	7
1.8	Pomegranate peels	8
1.9	Prawn shells	9
1.10	Chicken Eggshells	10
1.11	Cavendish Banana Peels	11
4.1	Drying of Sugarcane Bagasse	20
4.2	Sugarcane Bagasse kept in a clay pot	21
4.3	sieving of sugarcane Bagasse powder	21
4.4	Soaked Sugarcane Bagasse	22
4.5	Filtering the Sugarcane Bagasse powder	22
4.6	Drying of Sugarcane Bagasse powder	23
4.7	Activated Carbon	23
4.8	Impregnation Process	24
4.9	Co-Precipitation Process	24
4.10	Filtration Process	25
4.11	Drying Process	25
4.12	SB-NMB	26

4.13	Schematic Representation of Preparation of SB-NMB	26
4.14	Acid Blue 9 with pH apparatus	27
5.1	FTIR spectrum of sugarcane bagasse Powder	34
5.2	Sem Image (a) of SB-NMB	35
5.2	Sem Image (b) of SB-NMB	36
5.3	EDX Image (a) of SB-NMB	37
5.3	EDX Image (b) of SB-NMB	37
5.4	XRD image of SB-NMB	39
5.5	VSM Image of SB-NMB	40
5.6	BET Image (a) of SB-NMB	41
5.6	BET Image (b) of SB-NMB	42
5.7	Lambda max (λ_{max}) of Acid Blue 9 from ultraviolet-visible spectroscopy	42
5.8	Effect of pH on Acid Blue 9 ion removal by SB-NMB	43
5.9	Effect of Contact Time on Acid Blue 9 ion by SB-NMB	44
5.10	Effect of initial Acid Blue 9 ion concentration for the adsorption onto SB-NMB	45
5.11	Effect of temperature on Acid Blue 9 ion removal by SB-NMB	46
5.12	Effect of adsorbent dose on Acid Blue 9 ions removal by SB-NMB	57
5.13	(a) Langmuir and Freundlich Adsorption isotherm for the removal of Acid Blue 9 ions by SB-NMB	49
5.13	(b) Temkin Adsorption isotherm for the removal of Acid Blue 9 ions by SB-NMB	49

5.14	Adsorption kinetic for eradicating Acid Blue 9 ions by Sb-NMB	51
5.15	Thermodynamic analysis for the adsorption of Acid Blue 9 by SB-NMB	53
5.16	Fourier-transform infrared (FTIR) after adsorption	55
5.17	Sem Image (a) of SB-NMB after adsorption	56
5.17	Sem Image (b) of SB-NMB after adsorption	57
5.18	EDX Image (a) of SB-NMB after adsorption	58
5.18	EDX Image (b) of SB-NMB after adsorption	58

LIST OF TABLES

TABLE NUMBER	TABLE NAME	PAGE NUMBER
5.1	EDX Map Sum Spectrum of SB-NMB	38
5.2	XRD Peak List	39
5.3	Adsorption isotherm parameters for the removal of Acid Blue 9 ions by SB-NMB	50
5.4	Adsorption Kinetic value for elimination of Acid Blue 9 ions by SB-NMB	52
5.5	Thermodynamic parameters for the adsorption of Acid Blue 9 ions onto SB-NMB	54
5.6	EDX Map Sum Spectrum of SB-NMB	59

CHAPTER-1 INTRODUCTION

Rapid worldwide industrialization in the Textile, Cosmetics, Metal processing, fertilizer, pulp and paper, rubber-plastic, batteries, mining, electroplating, pharmaceutical and metallurgical industries has increased the water resources pollution(Izydorczyk et al., 2021). Color-rendering compounds, such as dyes and pigments, if released untreated can increase the biochemical oxygen demand in rivers, blocking light transmission into the water and as a result, interfering with aquatic natural processes that depend on light(Subbareddy et al., 2020). The organic azo dye Acid Blue 9, usually referred to as FD&C Blue No. 1, is one of the most significant dyes. Its molecular formula is $C_{37}H_{39}N_3O_9S_3$, and its molecular weight is 765.91.

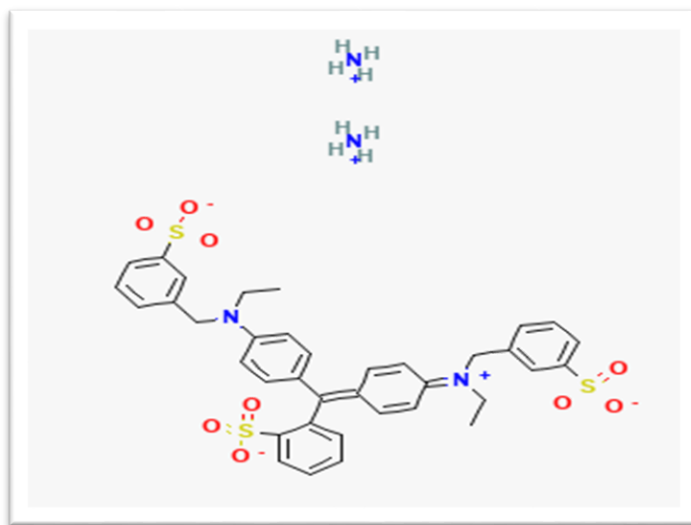


Fig. 1.1 Chemical Structure of Acid Blue 9.

This water-soluble dye is widely utilised in a variety of applications, including dyeing fabrics such as wool, polyamide fibre, and silk and is also utilised as an analyte in the development of azo-dye removal methods. When humans come into direct contact with Acid Blue 9, can cause eye and skin irritation. Inhaling its fumes or dust particles might irritate the respiratory system, causing trouble breathing. Prolonged or recurrent exposure might have more serious health consequences, such as allergic reactions and organ damage. The manufacture and disposal of Acid Blue 9 can pollute the air, and water and generate hazardous waste. Improper handling and disposal of this dye

can lead to soil pollution and worsening environmental degradation. It is considered a contaminant because of its water solubility, and its presence in sewage poses a threat to natural water reserves and living species, harming water quality even at low levels(Neetha et al., 2018).



Fig. 1.2 Dye wastewater released into nearby water bodies.

Runoff water from industrial operations contains dyes that harm nearby water sources. To avoid health hazards, removing these dangerous pigments before disposing of wastewater is essential. Hazardous compounds that have been released into the environment in large quantities have caused several ecological concerns due to their persistence and non-biodegradability(Andrade Siqueira et al., 2020). Traditional techniques, like chemical coagulation, liquid-liquid exchangers, ion exchanges, membrane filtration, electrical, chemical removal, and coagulation, have downsides such as insufficient removal, high energy requirements, hazardous sludge, limited efficiency, delicate working conditions, and expensive disposal(Bhattacharjee et al., 2020).

Adsorption is the promising technological method for removing dyes from wastewater. It has several benefits, including low energy consumption, the ability to use adsorbent

materials made from waste or byproducts, and cost-effectiveness in terms of both initial investment and ongoing operations(Shokri and Fard, 2022). Adsorption has received a lot of attention recently and is currently an appealing choice for treating wastewater. This method is preferred over other removal methods due to the low amount of chemicals it uses and the variety of available adsorbents. Dyes and heavy metals can be removed from wastewater using this simple, manageable technology that is safe and effective even at deficient pollution concentrations(Türkmen et al., 2022). sugarcane bagasse, Banana peels, potatoes, apples, lemons, coffee grounds, decaf coffee grounds, grape seeds, papaya seeds and orange peels have all been utilised as inexpensive adsorbents(Frezzini et al., 2019).

Agricultural waste was chosen because it is readily available, easy to procure, and potentially solves the agricultural waste disposal problem. Sugarcane (*Saccharum officinarum*) is crushed for juice extraction, and bagasse, which amounts to about 25% of the overall weight and consists 60% to 80% of carbohydrates, is discarded as agricultural waste(Rezende et al., 2011). Sugarcane bagasse (SB) is one of the major agro-industrial wastes that consists of lignin (5-20%), cellulose (30-80%), and hemicellulose (5-40%)(Nunes et al., 2020).

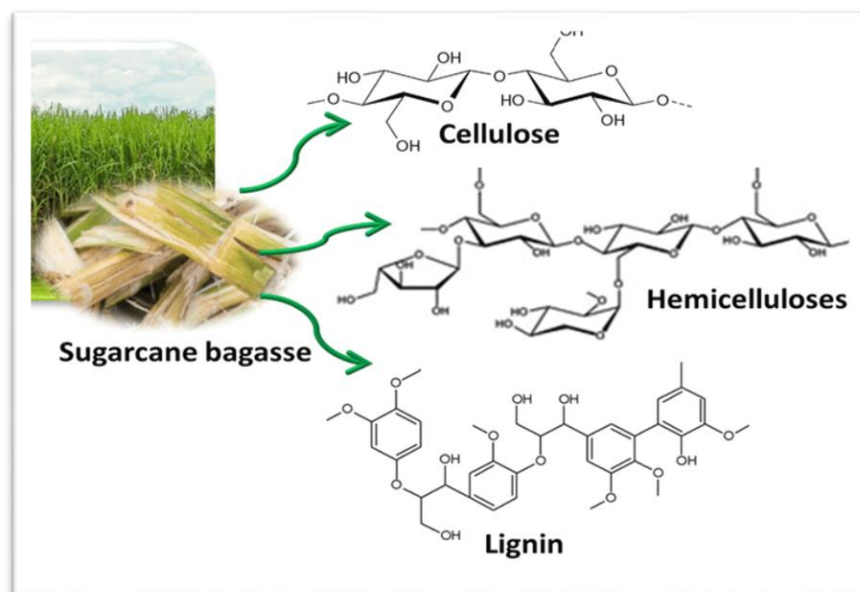


Fig. 1.3 Chemical Structure of Sugarcane Bagasse.

Sugarcane bagasse's been investigated as a potential biosorbent for dye removal, especially when converted into nanomagnetic forms(Aruna et al., 2021). Sugarcane bagasse was employed to remove safranin O, with adsorption capacities of 62.884 mg/g for base-treated SB, 58.853 mg/g for raw SB, and 54.822 mg/g for acid-treated Sygarcane Bagasse, respectively. It was known as an efficient adsorbent for Congo-red adsorption, with the adsorption capacity of 38.2 mg/g and a pseudo-second order kinetic model. According to the study, the adsorption capability for methylene blue and acid orange II elimination using sugarcane bagasse treated with propionic acid was 59.5 and 25.5 mg/g respectively(Ahmad et al., 2018).

Objectives of the study are

- (i) To Synthesize sugarcane bagasse-derived nanomagnetic biosorbent composite (SB-NMB) to remove Acid Blue 9 from aqueous solutions.
- (ii) To characterize and analyze the properties and performance of the synthesized SB-NMB, such as FTIR, SEM, EDX, XRD, VSM, BET adsorption capacity, and kinetics surface properties.
- (iii) To obtain conclusive findings and data regarding the performance of the SB-NMB in removing Acid Blue 9 from aqueous solutions.

The batch studies were evaluated, including the solution's pH, contact time, dye concentration, temperature, and adsorbent dosage. To further comprehend the experimental data, isotherm, kinetic, and thermodynamic parameters were examined as well.

1.1 TYPES OF DYES

1.1.1 Azo dyes

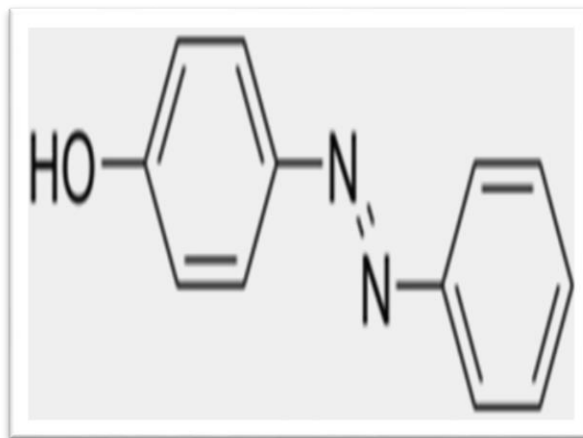


Fig. 1.4 Chemical structure of an orange-coloured azo dye.

Synthetic organic compounds with the azo bond ($-N=N-$) are azo dyes. They consist of an azo link connecting two aromatic amines. Azo dyes are utilized in several industries, such as leather, textiles, cosmetics, food, and pharmaceuticals. Azo dyes are large synthetic organic dyes that make up more than half of the dye produced annually worldwide. An azo dye's colour is influenced by the rest of the molecule's structure, including the presence of functional groups (Vaiano and De Marco, 2023).

Azo dyes, which contain the nitro group known for its mutagenicity and can yield harmful byproducts like 1,4-phenylenediamine and o-tolidine upon degradation, are still used and produced worldwide due to their economic value and advantageous characteristics, despite being banned in certain parts of the world due to their toxicity and carcinogenic potential, as well as their capacity to induce allergic reactions and mutagenic activity (Gupta et al., 2006).

1.1.2 Reactive dyes

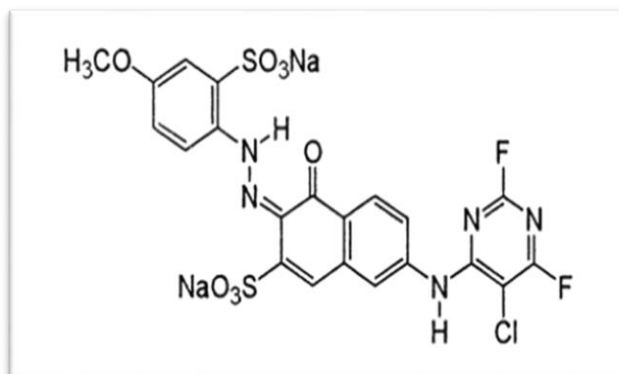


Fig. 1.5 Reactive Red 120 dye's Chemical structure.

Reactive dyes are chemicals that combine with other molecules to form new compounds. They are employed in a wide range of chemical processes, including food colouring, textile dyeing, and pharmaceutical manufacturing. The majority of the time, reactive dyes are used to colour cellulose fibres like viscose and cotton. Additionally, they have evolved into more and more significant for wool and polyamide. Reactive dyes are mostly used for printing cellulosic fibres like cotton and linen because of their broad colour spectrum(Ahmad et al., 2018).

Reactive dyes can sometimes result in hypersensitivity and allergic skin reactions. Based on the chemical benzidine, a few dyes are believed to be carcinogenic.

1.1.3 Metal Complex dyes

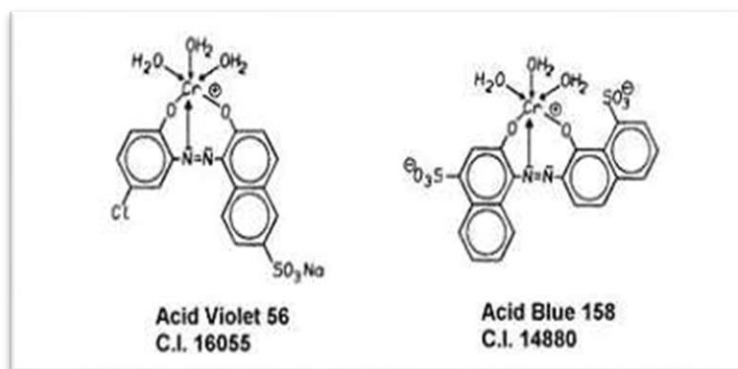


Fig. 1.6 Metal Complex dye's Chemical structure.

Metal complex dyes are a class of dyes that contain a central metal ion coordinated to organic molecules called ligands. These ligands are typically azo dyes, which means they contain an azo group ($-N=N-$). The metal ion and the ligands interact with each other through coordination bonds. The colour, stability, solubility, and substrate affinity of a metal complex dye are all significantly influenced by metal ions and ligands selection (Kolya and Kang, 2024).

Metal ions commonly utilised in metal complex dyes include copper, chromium, nickel and cobalt. Adverse health effects, including skin sensitivity, lung cancer, liver and kidney damage, and respiratory irritation, can result from exposure to metal complex dyes. Extended exposure to these metals can cause systemic toxicity as well as long-term health issues.

1.1.4 Sulphur dyes

Sulfur-containing dyes are a class of dyes with complex molecular structures. They are produced by heating organic substances with sulphur or sodium polysulfide, such as aromatic amines and aminophenols. Their unique characteristics are typically derived from heterocyclic rings containing sulphur, such as derivatives of thioxanthene or dithioxanthene.

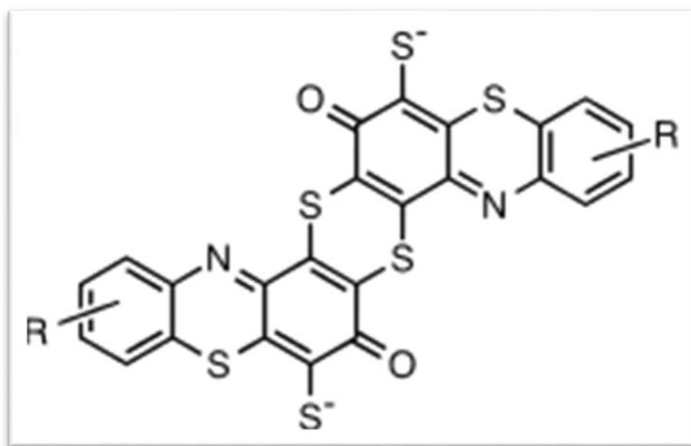


Fig. 1.7 Chemical structure of Sulfur dye.

The denim business uses dyes that contain sulphur. They are often easy to apply,

affordable, and have good wash-fastness. The most common colours for sulphur dyes are dark blue, brown, and black. Although a pink or softer scarlet colour is obtainable, red sulphur dyes are unknown. The most common black dye used worldwide to colour cellulosic materials is Sulphur Black 1.

Humans as well as animals may be intoxicated by sulfur dyes. Respiratory issues, skin, and eye irritation can result from sulfur dye exposure. Considerable DNA damage has also been observed in the tadpoles that were dyed.

Regular sulphur dyes are difficult to dissipate have poor storage stability, are highly alkaline and smell bad, and are prone to contamination because of their affinity for objects.

1.2 DIFFERENT TYPES OF ADSORBENTS

1.2.1 Pomegranate peels



Fig. 1.8 Pomegranate peels.

Pomegranate peels have been investigated as a natural and sustainable adsorbent for a variety of environmental applications due to their widespread availability, low cost, and high adsorption capacity. Due to their high surface area and porous nature, pomegranate peels offer a large number of active adsorption sites. Their shape is porous, which increases their ability to adsorb a variety of contaminants. It has been shown that pomegranate peels may efficiently adsorb a variety of contaminants, such

as organic compounds, dyes, heavy metals, and nutrients(Hashem et al., 2023). Research has demonstrated their effectiveness in air purification, soil remediation, and wastewater treatment. Pomegranate peel-based adsorbents have prospective uses in a range of industries, such as industrial facilities, agricultural settings, and wastewater treatment plants. By using them, green technologies for environmental remediation are developed.

1.2.2 Prawn shells



Fig. 1.9 Prawn shells.

Prawn shells are converted into several forms, such as chitin and chitosan, and then used as an adsorbent or biosorbent. Seafood is regarded as a staple meal and a key component of the food chain system in many regions of the world(Liu et al., 2021).

Chitosan finds use in the paper, textile, pharmaceutical, cosmetic, healthcare, agricultural, biotech, and food sectors. Chitosan that is sourced from prawn shells has a great potential for adsorbing many types of pollutants because of its porous structure and functional groups, which include hydroxyl and amino groups. These groups facilitate the formation of robust connections between chitosan and pollutants by mechanisms such as chemical complexation, hydrogen bonding, and electrostatic attraction.

Adsorbents based on prawn shells have been used in a variety of environmental

remediation applications, such as soil stabilisation, wastewater treatment, groundwater remediation, and air purification. These adsorbents are especially helpful in sectors where pollution management is a top concern, like aquaculture, food processing, and pharmaceutical manufacturing (Ramadhani et al., 2020).

1.2.3 Eggshells



Fig. 1.10 Chicken Eggshells.

Eggshells, which are typically thought of as food waste, have shown promise as an adsorbent for a range of environmental uses. They are mostly composed of calcium carbonate (CaCO_3), containing trace amounts of other chemical substances and proteins. Potential adsorption sites are provided by the pore structure of eggshells, which is facilitated by calcium carbonate (Abatan et al., 2020).

Eggshells are an affordable and easily accessible adsorbent choice, particularly in areas where eggs are extensively consumed. Reusing eggshells as adsorbents can encourage sustainable habits and help reduce trash.

Eggshell-based adsorbents were investigated for wastewater treatment, heavy metal removal, dye adsorption, and nutrient recovery, among other environmental remediation applications. They can be applied to the removal of pollutants from waste water (Badrealam et al., 2018).

1.2.4 Banana peels



Fig. 1.11 Canvendish Banana Peels.

The vast surface area and porous structure of banana peels increase their adsorption capability. The abundance of active sites is available for the adsorption of impurities from waste water due to the presence of pores.

Both cellulose and hemicellulose, which are polysaccharides with hydroxyl (-OH) groups, are abundant in banana peels. These functional groups use van der Waals forces and hydrogen bonds to enhance interactions with contaminants(Kok Seng, 2018).

It has been shown that banana peels can absorb pigments from wastewater effluents. Dye adsorption on banana peels is thought to be caused by interactions between functional groups on the peel's surface and dye molecules. Because they are affordable and easily obtainable, banana peels are a financially advantageous adsorbent choice, especially in areas where banana consumption is high. Banana peels can be used as adsorbents to reduce waste and encourage environmentally friendly behaviours(Abdulfatai et al., 2013).

1.3 APPLICATIONS OF SB-NMB

- Removal of dyes.
- Removal of Heavy Metals.
- Removal of Personal Care and Pharmaceutical Products
- Water Purification
- Environmental Restoration
- Resource recovery
- Agricultural effluent treatment
- Biosensing
- Medical and Biomedical applications
- Catalysis

CHAPTER-2 LITERATURE SURVEY

- **Azaritorbat et al. (2023)** have published a scientific article regarding the effectiveness of o-toluidine removal by adsorption and biodegradation techniques compared in this study. The adsorption process involved the use of sugarcane bagasse (SCB) as an adsorbent, while the biodegradation process involved using white-rot fungi, more precisely *Phanerochaete chrysosporium*. The purpose of the study was to investigate how effectively both of these methods could remove o-toluidine while also assessing the adsorbent capability of SCB and the biodegradation efficiency of *Phanerochaete chrysosporium*. Adsorption kinetics, isotherms, and biodegradation assays were among the techniques used in the study to evaluate the extent to which SCB and *Phanerochaete chrysosporium* removed o-toluidine from aqueous solutions. The results of the study could assist in creating economical and environmentally friendly techniques for treating wastewater contaminated with o-toluidine by employing sugarcane bagasse as an adsorbent and white-rot fungi for biodegradation.

- **Munilakshmi et al. (2023)** have published a scientific paper focusing on adsorption investigations involving the use of new adsorbents consisting of prefabricated flocs of aluminium sulphate, ferric chloride, and other materials to decolourize Acid Blue dye. This study looks into the kinetics and equilibrium of the Acid Blue dye adsorption process in aqueous solutions. The study examines the extent to which flocs perform as adsorbents to remove Acid Blue dye, emphasising the potential of chemicals like ferric chloride and aluminium sulphate to decolourize wastewater. The rate and level of dye removal are revealed by the kinetic and equilibrium studies which also highlight the impact of these adsorbents in water treatment procedures. Overall, this study adds to our insight into the kinetics and equilibrium of the Acid Blue dye adsorption process and helps to explain the behaviour of the dye on preformed flocs. The promise of new adsorbents in environmental rehabilitation

efforts is highlighted by their use in the removal of dyes, such as aluminium sulphate and ferric chloride.

- **Elshabrawy et al. (2023)** have published a scientific article focusing on using sugarcane bagasse pulp as a powerful adsorbent material for treating wastewater, specifically to remove dyes from textile waste water. The research emphasises how sugarcane bagasse pulp can be used to mitigate wastewater dye pollution, an important environmental issue because of the toxicity of dyes even in small amounts. This study uses chemically modified sugarcane bagasse to facilitate the biosorption process of dye removal from actual textile effluent. The experimental data and the adsorption process findings indicate that sugarcane bagasse pulp is a useful tool for removing dyes from wastewater. The use of sugarcane bagasse pulp as an adsorbent material provides a low-cost, ecologically acceptable approach for wastewater treatment, particularly for dye removal effluent.

The presence of artificial neural network (ANN) results in the research suggested that the study also investigates the usage of ANN to predict the adsorption process. With this method, the process may be optimised and the adsorption behaviour can be better understood.

- **Vasuki et al. (2023)** have published a scientific article investigating the adsorption process highlighting the effectiveness of activated carbon derived from coconut shells and palmyra fruit nutshell in removing Acid Blue 9 and Brilliant Green.

This study compares the effectiveness of these two forms of activated carbon in adsorbing Acid Blue 9 and Brilliant Green from aqueous solutions with a focus on dye removal and also examines the adsorption mechanism of these dyes, emphasising the efficiency of activated carbon made from the shells of coconuts and palmyra fruit nuts in removing Acid Blue 9 and Brilliant Green. This work attempts to shed light on the possible use of materials obtained from agricultural

waste for applications involving the removal of dyes by comparing the adsorption capabilities of these two forms of activated carbon.

This comparative study advances knowledge on the application of activated carbon made from coconut and palmyra fruit nut shells for environmental remediation, particularly concerning the removal of dyes from waste water sources. The results of this study can provide important insights into long-term and efficient dye-contaminated wastewater treatment strategies.

- **Yaseen et al. (2020)** published a scientific article that reported that modified sugarcane bagasse had a maximum removal efficiency of 99.1% for methylene blue.

The isotherm models Langmuir and Freundlich were used to analyze the adsorption data, and the Langmuir isotherm model was found to fit the data better than the Freundlich isotherm model, indicating that the adsorption process followed a monolayer adsorption mechanism. This study also examines sugarcane bagasse's adsorption capacities in removing methylene blue from aqueous solutions both before and after alkaline treatment. The researchers concluded that modified sugarcane bagasse could be used as a highly effective adsorbent for the removal of methylene blue from textile industry wastewater.

Overall, the study by Yaseen et al. (2020) provides valuable insights into the potential use of modified SB for the removal of methylene blue from textile industry wastewater.

- **Elgarahy et al. (2021)** published a review article discussing the biosorption process in a critical evaluation as a successful and environmentally friendly method to remove metalloids, heavy metals, and dyes from wastewater. The review explores recent advances in biomaterials-based biosorbents, providing insights into their application in environmental remediation processes.

The paper highlights the efficacy of the several biosorbents utilised in the procedure for adsorbing contaminants from wastewater. Potential possibilities for removing hazardous chemicals from aqueous solutions include biosorbents

such as sugarcane bagasse, bacteria, cyanobacteria, yeasts, algae, fungi, and waste items.

The mechanics underlying the biosorption process are also examined. These mechanisms are essential to the adsorption of contaminants by biosorbents and enhance the treatment process' overall efficacy.

Overall, the study offers a thorough review of biosorption as an environmentally friendly and effective way to remove metalloids, heavy metals, and dyes from wastewater. It highlights the possibilities of different biosorbents in tackling environmental pollution issues and emphasises the significance of using biosorption technology as a sustainable solution to water treatment.

- **Siqueira et al. (2020)** published a scientific article that investigated the adsorption of methylene blue dye employing sugarcane bagasse as a biosorbent, highlighting the material's sustainable and economical potential for dye removal applications.

To comprehend the adsorption process, several characteristics were examined and the biomass was characterised. Batch adsorption investigations were carried out together with SEM to characterise the biomass. The efficiency of SCB as a biosorbent for the removal of methylene blue was assessed through the analysis of kinetics, adsorption isotherms, and thermodynamics.

The findings shown that after 24 hours of contact time, SB showed a maximum adsorption capacity of 9.41 mg/g at 45°C. The pseudo-second order model was supported by the adsorption kinetics data, indicating a chemical process was in effect. The heterogeneous adsorption process might be adequately described by the three-parameter isotherm model proposed by Sips.

Overall, this study demonstrates the extent to which sugarcane bagasse works as a biosorbent for removing methylene blue dye from aqueous solutions, providing wastewater treatment operations with an economical and sustainable alternative.

- **Ahmad et al. (2018)** published a scientific about Reactive Red 120 (RR120) which can be removed from aqueous solutions using sugarcane bagasse powder as a biosorbent. The focus of the study is on the batch treatment of textile dye RR120 and the purpose is to determine how different adsorption factors affect the removal of RR120 from aqueous solutions. The study's findings indicated that sugarcane bagasse powder is a very effective biosorbent for removing RR120. The potential of this agricultural waste product as an effective and economical method for removing dye from aqueous solutions is demonstrated by the notable removal percentages that result from the optimised circumstances.

Overall, studies on the biosorbent use of sugarcane bagasse powder to remove RR120 from aqueous solutions indicate the technology's effectiveness, affordability, and environmental advantages for wastewater treatment applications.

CHAPTER-3 AIM AND SCOPE

3.1 AIM

- To **Synthesize** sugarcane bagasse-derived nanomagnetic biosorbent (SB-NMB) composite for removing Acid Blue 9 from aqueous solutions.
- To **characterize and analyze** the properties and performance of the synthesized SB-NMB, such as FTIR, SEM, EDX, XRD, VSM, BET adsorption capacity, and kinetics surface properties.
- To **obtain conclusive findings** and data regarding the performance of the SB-NMB in removing Acid Blue 9 from aqueous solutions.
- To **assess the potential application** of the SB-NMB as a biosorbent.

3.2 SCOPE

- **A sustainable approach** to wastewater treatment and environmental remediation.
- **cost-effective solution** for the removal of Dyes and Heavy metals compared to other conventional methods.
- Conduct adsorption studies to **investigate the effectiveness** of SB-NMB in removing Acid Blue 9 from aqueous solutions

CHAPTER-4 MATERIALS AND METHODOLOGY

The following materials were used to synthesize the SB-NMB composite:

4.1 MATERIALS

Sugarcane bagasse has been gathered from a roadside juice shop in Sholinganallur and the Acid Blue 9 was acquired from Subha Chemicals in Tambaram.

4.1.1 Chemicals Required

- Hydrochloric Acid (HCl)
- Ferric Sulphate ($\text{Fe}_2(\text{SO}_4)_3$)
- Ferric Chloride (FeCl_3)
- Ammonium Hydroxide (NH_4OH)
- Ethanol ($\text{C}_2\text{H}_5\text{OH}$)
- Distilled Water
- ACID BLUE 9.

4.1.2 Equipment Required

- Muffle Furnace
- Hot Air Oven
- Burette
- Hot Plate Magnetic Stirrer
- Rotary Shaker
- Ultraviolet-visible spectroscopy
- pH meter
- Weighing balance

4.2 METHODOLOGY

4.2.1 Drying of Sugarcane Bagasse

Sugarcane bagasse was collected, and rinsed twice with tap water, then with deionized water, to remove dust and particles that could interfere with the analytical technique. The wet SB was then sun-dried for 10 to 14 days.



Fig. 4.1 Drying of Sugarcane Bagasse.

4.2.2 Preparation of Sugarcane Bagasse Activated Carbon

The sun-dried sugarcane bagasse was chopped into little pieces (approximately 3 cm) and placed in a small mud pot, sealed with clay and placed in a Muffle furnace for an hour at 600-800°C.



Fig. 4.2 Sugarcane Bagasse kept in a clay pot.

The sugarcane bagasse from the clay was ground into powder, sieved at 85 mesh number and kept in jars for experimental usage.



Fig. 4.3 Sieving of sugarcane Bagasse powder.

The Sugarcane Bagasse powder is activated by soaking it in distilled water and hydrochloric acid (HCl) for about 24 hours.

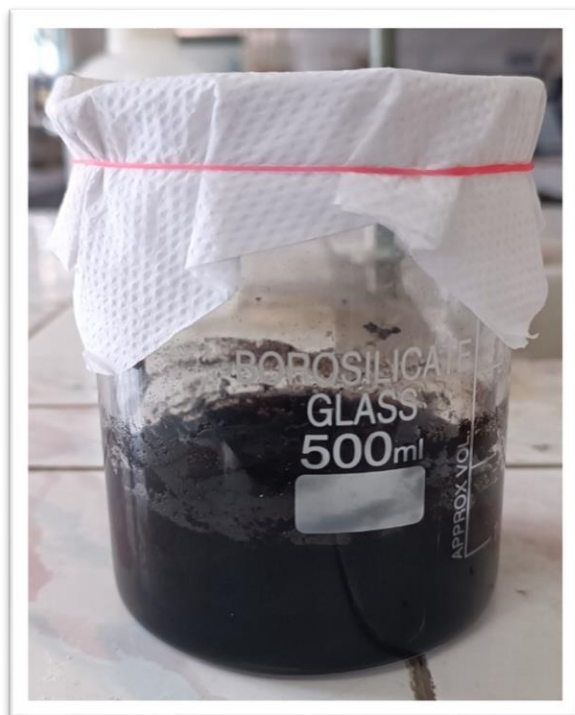


Fig. 4.4 Soaked Sugarcane Bagasse.

After filtering the solution, the solid powder is thoroughly washed with distilled water.



Fig. 4.5 Filtering the Sugarcane Bagasse powder.

The filtered product is then dried in a muffle oven for one hour at 150°C.



Fig. 4.6 Drying of Sugarcane Bagasse powder.

The dried product is collected and called sugarcane bagasse activated carbon (SB-AC).



Fig. 4.7 Activated Carbon

4.2.3 Impregnation Process of SB-NMB

The Activated Carbon is then impregnated with 2g of Ferric Chloride (FeCl_3), 1g of Ferric Sulphate ($\text{Fe}_2(\text{SO}_4)_3$) and 1g of the SB-AC.

The conical flask was filled with all the solid components and placed inside a hot plate

magnetic stirrer and 50 ml of distilled water was added. The mixture is then allowed to stir constantly for 15 minutes at a temperature of 40°C. (Sugarcane Bagasse – Nanomagnetic Biosorbent)

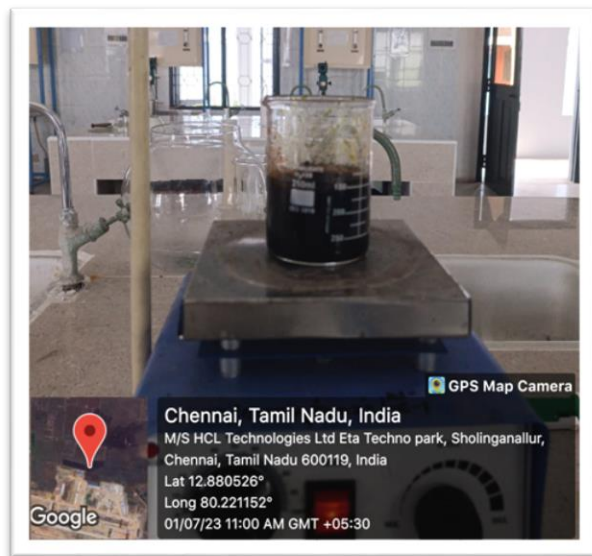


Fig. 4.8 Impregnation Process

4.2.4 Co-precipitation Process of SB-NMB

For the co-precipitation process, 50ml of ammonium hydroxide (NH_4OH) is measured and added to the solution drop by drop using a burette retained in the magnetic stirrer.



Fig. 4.9 Co-Precipitation Process

4.2.5 Filtration and Drying of SB-NMB

After the co-precipitation, give it 15 minutes to cool. filter the sample now and then wash it with distilled water and Ethanol to prevent rusting.



Fig. 4.10 Filtration Process

The Filtrate is then dried in Muffle Oven at 150°C for 30 minutes. The powdered particle is the Sugarcane Bagasse impregnated with a Magnetic Nanoparticle (SB-NMB).



Fig. 4.11 Drying Process

4.2.6 Magnetic Separation of SB-NMB



Fig. 4.12 SB-NMB

Magnetic Attraction was checked using a Magnet.

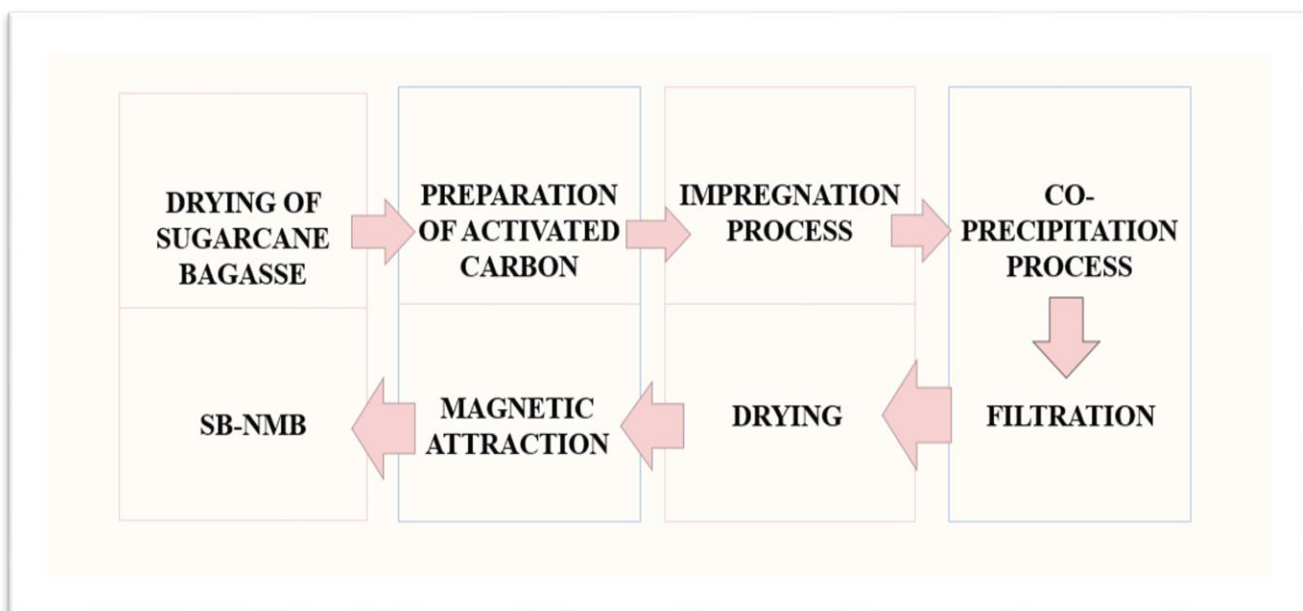


Fig. 4.13 Schematic Representation of Preparation of SB-NMB

4.3 PREPARATION OF ACID BLUE 9 SOLUTION

The Analytical Reagent Grades (AR) served as the standard way to handle all chemicals. To Prepare the stock solution with an Acid blue 9 concentration of 1000 mg/L, 1g of dye has been measured and dissolved in 1000 ml of distilled water. Diluting the stock solution in the proper order, dye levels were adjusted to reach the necessary test concentrations (ranging between 20 to 100 mg/L). Prior to adding the adsorbent to the aqueous solution, the pH of each standard solution was adjusted to the appropriate consistency using 0.1M NaOH and 0.1M HCl. A pH meter was used to measure each solution's pH.

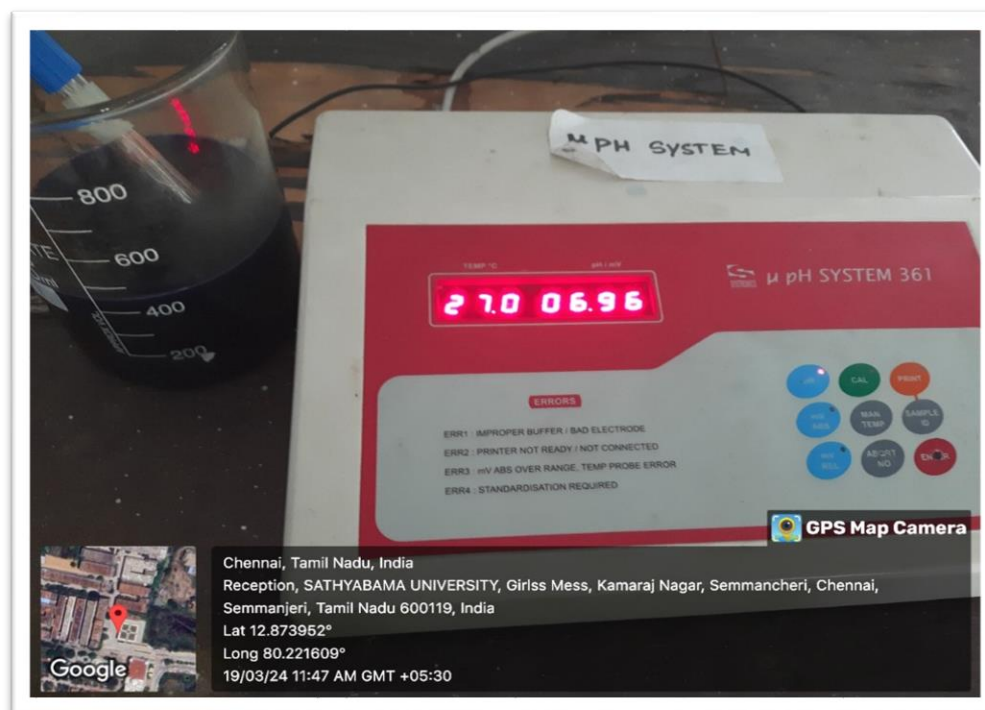


Fig. 4.14 Acid Blue 9 with pH apparatus

4.4 ADSORPTION EXPERIMENT

- The effectiveness of SB-NMB composite for removing Acid Blue 9 ions from Aqueous solutions is being investigated by studying the effects of different parameters.

- Batch adsorption studies were conducted to optimize various parameters affecting the adsorption process. Parameters investigated include:
 1. Solution pH (2.0 - 7.0)
 2. Contact Time (0 - 90 Minutes)
 3. Initial Acid Blue 9 ion concentration (20 - 100 mg/L)
 4. Temperature (305 – 335 K)
 5. Adsorbent dosage (SB-NMB) (0.1 – 0.6 g/L)
- Fixed-volume (100 mL) experiments were performed using flasks and the mixture was agitated using a rotary shaker.
- The percentage removal of Acid Blue 9 ions was calculated using the provided equation:

$$\%R = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (1)$$

Where,

C_0 (mg/L) = Initial Acid Blue 9 ion concentration

C_e (mg/L) = Equilibrium Acid Blue 9 ion concentration

- The experiment employed several models to comprehend the adsorption process, which included:
 1. Adsorption isotherms
 2. Adsorption thermodynamics
 3. Adsorption kinetics
 4. Adsorption mechanism

4.4.1 Adsorption Isotherm Experiments

Adsorption isotherm measurements were performed at an ideal pH of 5 by adding a set quantity of SB-NMB (0.2 g) to 100 mL of Acid Blue 9 solution at different initial concentrations (20-100 mg/L).

In a rotating shaker, the mixture was agitated until equilibrium was achieved and the final Acid Blue 9 concentration in the solution was measured.

The three isotherm models developed to describe the adsorption of Acid Blue 9 onto SB-NMB were Langmuir, Freundlich, and Temkin models.

The equilibrium concentration of Acid Blue 9's in the solution (C_e) is related to the quantity of Acid Blue 9 adsorbed per gram of SB-NMB (q_e) in each model equation.

To match the experimental data to the model equations, several constants need to be determined.

1. Langmuir Isotherm Model:

Langmuir Isotherm Model estimates the number of active sites undergoing pressure and adsorption.

The Linear form is-

$$q_e = \frac{bq_{max}}{1+bc_e} \quad (2)$$

The above equation is rearranged for plotting.

$$\frac{1}{q_e} = \frac{1}{bq_{max}} \cdot \frac{1}{C_e} + \frac{1}{q_{max}} \quad (3)$$

Where,

q_e = Quantity of adsorbate adsorbed per unit weight of adsorbent at equilibrium (mg/g)

C_e = Concentration of adsorbate at equilibrium in solution after adsorption (mg/l)

q_{max} = Maximum adsorption capacity (mg/g)

b = Langmuir adsorption equilibrium constant (l/mg)

2. Freundlich Isotherm Model:

Freundlich Isotherm Model estimates the quantity of gas adsorbed by unit mass of solid adsorbent with pressure.

The Linear form is-

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (4)$$

Where,

q_e = Extent of adsorbate adsorbed per unit weight of adsorbent at equilibrium (mg/g)

C_e = Equilibrium concentration of adsorbent in solution after adsorption (mg/l)

K_f = Freundlich constant indicating the adsorption capacity

n = Empirical constant.

3. Temkin Isotherm Model:

The adsorption's heat of the molecules decreases linearly with the increase in coverage of the adsorbent surface.

The Temkin Model equation-

$$q_e = B_T \ln K_T + B_T \ln C_e \quad (5)$$

Where,

K_T = Equilibrium binding constant (lm/g)

B_T = Heat of adsorption (Tempkin constant) (J/mol)

b_T = Tempkin isotherm constant related to the variation of adsorption energy (J/mol)

$$B_T = \frac{RT}{b_T} \quad (6)$$

R is the gas constant (8.314 J/Kmol)

T = temperature (K)

4.4.2 Adsorption Kinetics and Mechanism Experiments

Acid Blue 9 ions were removed successfully by adding 0.2g of SB-NMB to 100 mL of the solution (20–100 mg/L) in a series of conical flasks containing the dye. Samples were taken out of the conical flask from the shaking incubator for a predetermined period (0-90min).

For a given time t and q_t (mg/g), the following formulae were used to calculate the amount of Acid Blue 9 ions adsorbed onto SB-NMB.

$$q_t = \frac{(C_0 - C_t)V}{m} \quad (7)$$

Where,

V = density of the Acid Blue 9 ion solution (L)

m = mass of SB-NMB (g)

C_t = concentrated level of Acid Blue 9 ions present within the mixture via period t (mg/L).

The equilibrium adsorption models that characterise the adsorption properties, such as pseudo first order, pseudo second order, and Elovich Kinetic parameters, were examined using the statistics on kinetic models.

The following are kinetic models:

1. Pseudo-first order Kinetic Model:

$$\ln(q_e - q_t) = \ln q_e - K_1 t \quad (8)$$

Where,

q_e = equilibrium adsorption capacity (mg/g)

q_t = Adsorption capacity at time t (mg/g)

K_1 = pseudo-first-order rate constant (min^{-1})

2. Pseudo-second order Kinetic Model:

$$\frac{t}{q_e} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} \quad (9)$$

Where,

q_e = equilibrium adsorption capacity (mg/g)

K_2 = pseudo-Second-order rate constant (min^{-1})

3. Elovich Kinetic Model:

$$q_t = \frac{1}{\alpha} \ln(\alpha\beta) + \frac{1}{\alpha} \ln t \quad (10)$$

Where,

q_t = Adsorption capacity at time t (mg/g)

α = Initial adsorption rate

β = Desorption constant during each experiment

4.4.3 Adsorption Thermodynamic Experiments

To conduct the sorption investigation, 0.2g of SB-NMB was infused into 100 ml of Acid Blue 9 solution contained in a series of conical flasks with initial Acid Blue 9 ion levels ranging from 20 to 100 mg/L. An incubation shaker was used to store the adsorption apparatus, which was satisfactorily maintained at temperatures ranging from 305 to 335 K.

The following set of formulas was used to determine values

$$K_c = \frac{q_e}{c_e} \quad (11)$$

$$\Delta G^0 = -RT \ln K_c \quad (12)$$

$$\ln K_c = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (13)$$

$$\Delta G = \Delta H - T\Delta S \quad (14)$$

$$\Delta S = C \times R \quad (15)$$

$$\Delta H = \text{slope} \times R \quad (16)$$

Where,

ΔG = variation in surface energy

R = Gas Constant (8.314J/Kmol)

T = Temperature (k)

K_c = Equilibrium constant

q_e = equilibrium adsorption capacity (mg/g)

C_e = Equilibrium concentration of adsorbent in solution after adsorption (mg/l)

ΔS = variation in entropy

ΔH = variation in enthalpy

CHAPTER-5 RESULTS AND DISCUSSIONS

5.1 CHARACTERIZATION OF SB-NMB

5.1.1 Fourier Transform Infrared (FTIR) Spectroscopy.

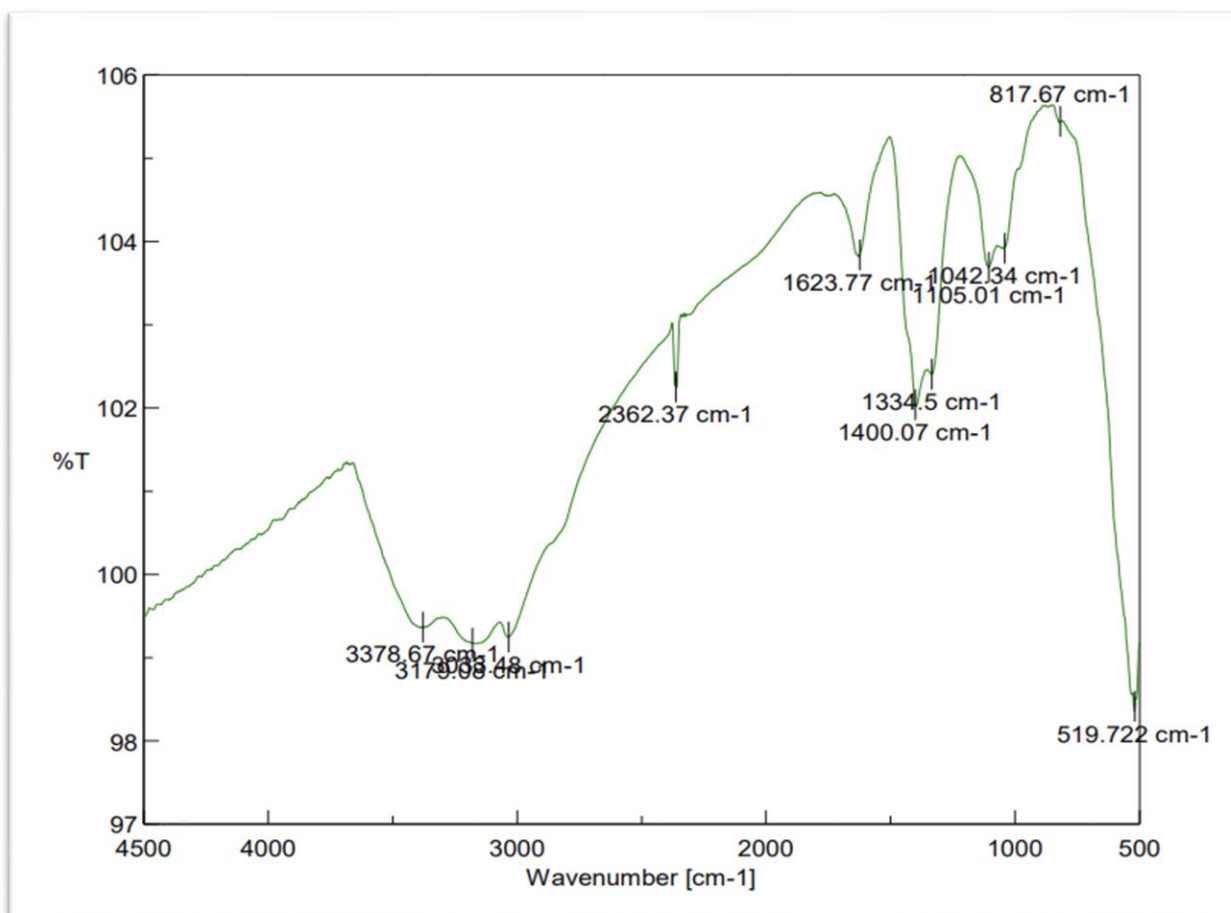


Fig.5.1 FTIR spectrum of sugarcane bagasse powder.

The Fourier-transform infrared (FTIR) spectrum shown in Figure 5.1 shows several unique peaks, each of which corresponds to a particular chemical bond or functional group. At 3378.67 cm⁻¹, a wide band was found, indicating the presence of hydroxyl groups. Stretching C-H bonds were indicated by the peaks at 3179.08 cm⁻¹ and 3033.48 cm⁻¹. The presence of C-C bonds was revealed by the occurrence of peaks at 2362.37 cm⁻¹, and at 1623.77 cm⁻¹ indicating the presence of C=C bonds. C-F bonds were present, as evidenced by a peak at 1400.07 cm⁻¹. A peak at 1334.5 cm⁻¹ represented

C-N Amine bonds. The presence of C-O bonds was suggested by a peak at 1105.01 cm^{-1} . At 1042.34 cm^{-1} , the C-N bond of the amine group was found. The spectrum displayed C-H bond aromatic out-of-plane bending at 817.67 cm^{-1} . At 519.722 cm^{-1} , a peak that may have been caused by bromide or iodide C-X bonds was also seen. The synthesis of SB-NMB adsorbent is validated by comparing the FTIR spectrum.

5.1.2. Scanning Electron Microscopy (SEM).

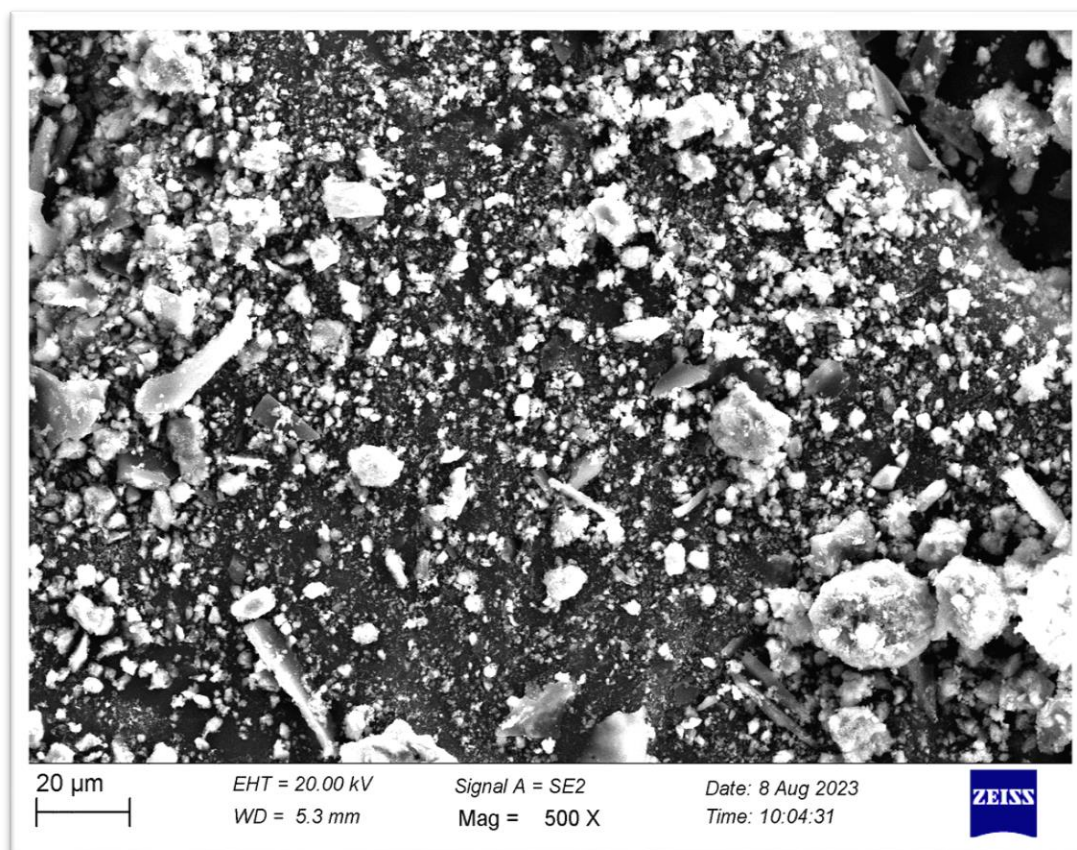


Fig.5.2 Sem Image (a) of SB-NMB.

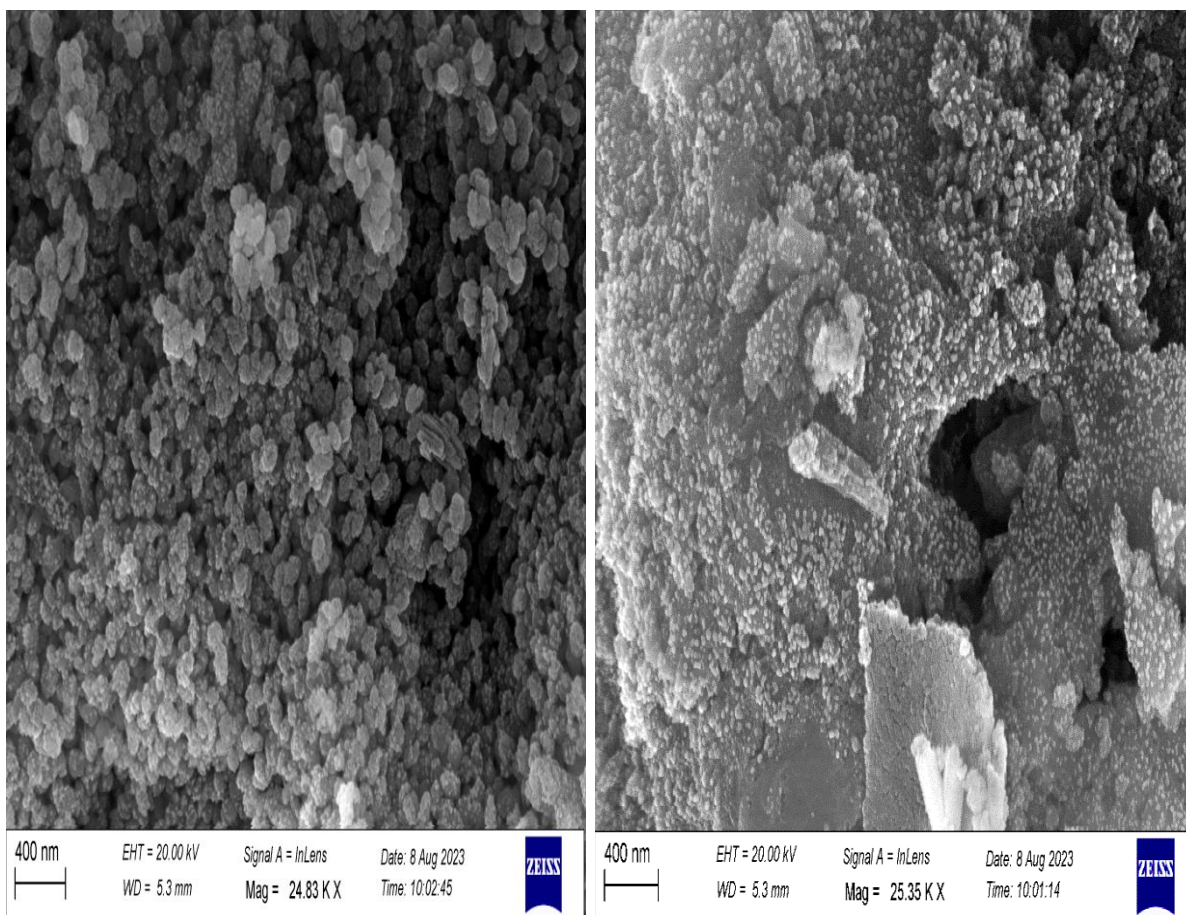


Fig.5.2 Sem Image (b) of SB-NMB.

Fig.5.2 (a) and (b) refers to the SEM image. The Scanning Electron Microscope (SEM) is one of the most widely used experimental methods for the research and analysis of micro and nanoparticle imaging characterization of solid materials. SEM is utilized for particle size characterization primarily because of its 10nm resolution. SEM is used to examine the morphology of the powder before consolidation. The methods are simple: a two-sided carbon tape is applied to an SEM sample stub, and then powder is sprinkled on top. SEM analyses were used to examine the morphology of the sample. The SB-NMB sample's porous, uneven, and amorphous surface is visible in SEM images.

5.1.3 Energy Dispersive X-Ray (EDX).

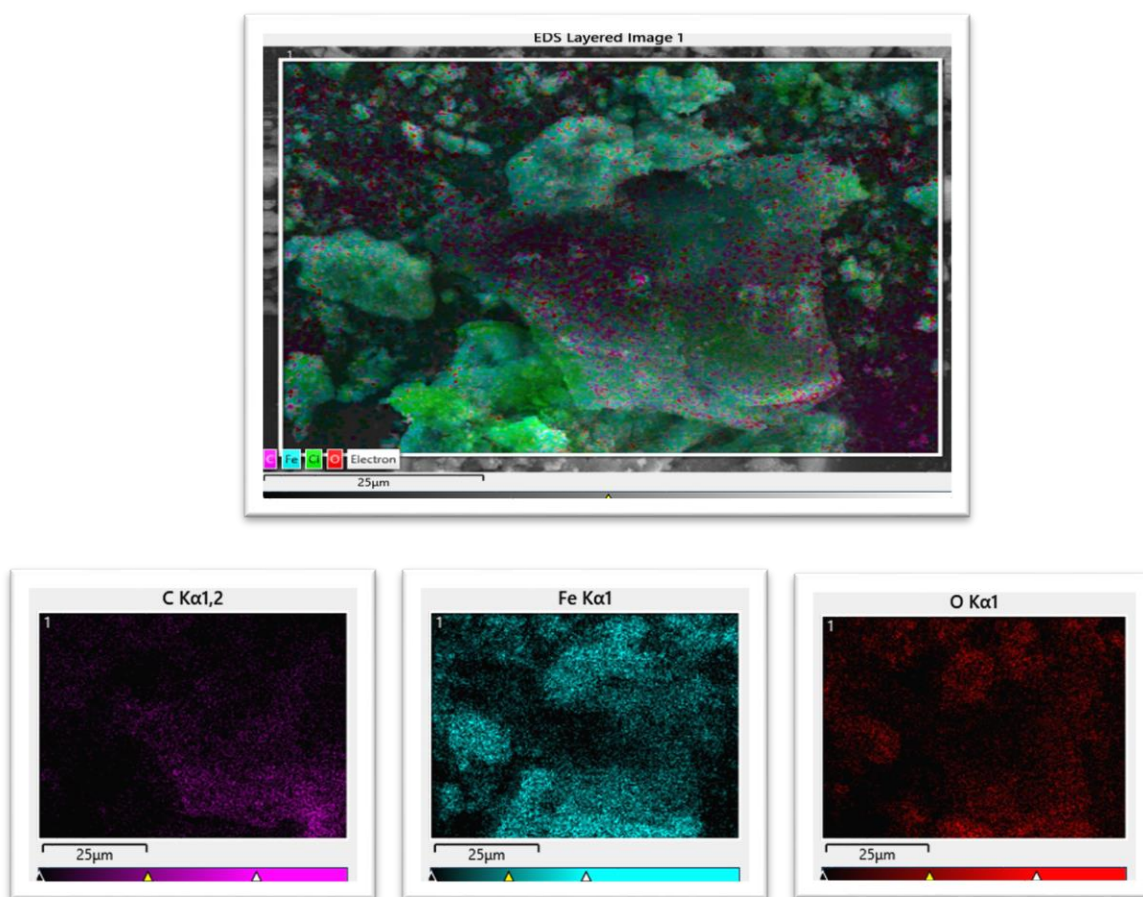


Fig.5.3 EDX Image (a) of SB-NMB.

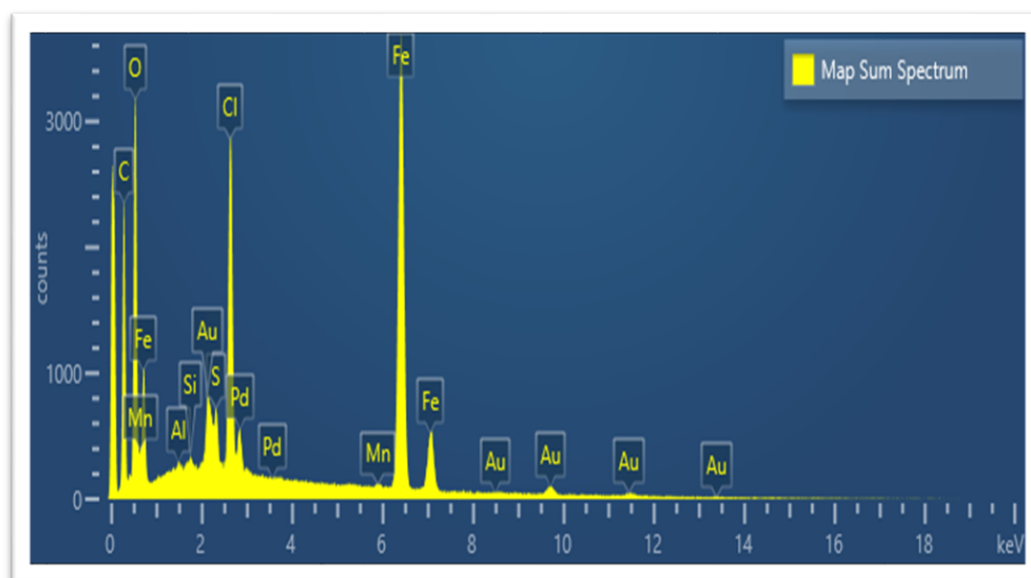


Fig.5.3 EDX Image (b) of SB-NMB.

Table 5.1 EDX Map Sum Spectrum of SB-NMB

Map Sum Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	41.70	1.54	62.26
O	K series	23.26	0.65	26.07
Al	K series	0.11	0.03	0.07
Si	K series	0.10	0.02	0.07
S	K series	0.76	0.04	0.42
Cl	K series	4.91	0.14	2.49
Mn	K series	0.17	0.05	0.06
Fe	K series	25.57	0.69	8.21
Pd	L series	0.58	0.10	0.10
Au	M series	2.84	0.16	0.26
Total		100.00		100.00

Figures 5.3 (a) and (b), Table 5.1 show the EDX pattern with the SB-NMB's Elemental weight percentage. An X-ray method called Energy Dispersive X-ray analysis, often known as EDS or EDX, is utilised to ascertain a substance's elemental composition, SEM with EDX determines the types and amounts of elements present at the nanomaterial surface.

The elemental makeup of the SB-NMB's sample was examined using EDX analysis. According to the EDX pattern, the synthesised SB-NMB sample is mostly made up of Carbon (41.70%), Iron (25.57%), oxygen (23.26%) and chlorine (4.91%).

5.1.4. X-ray diffraction (XRD).

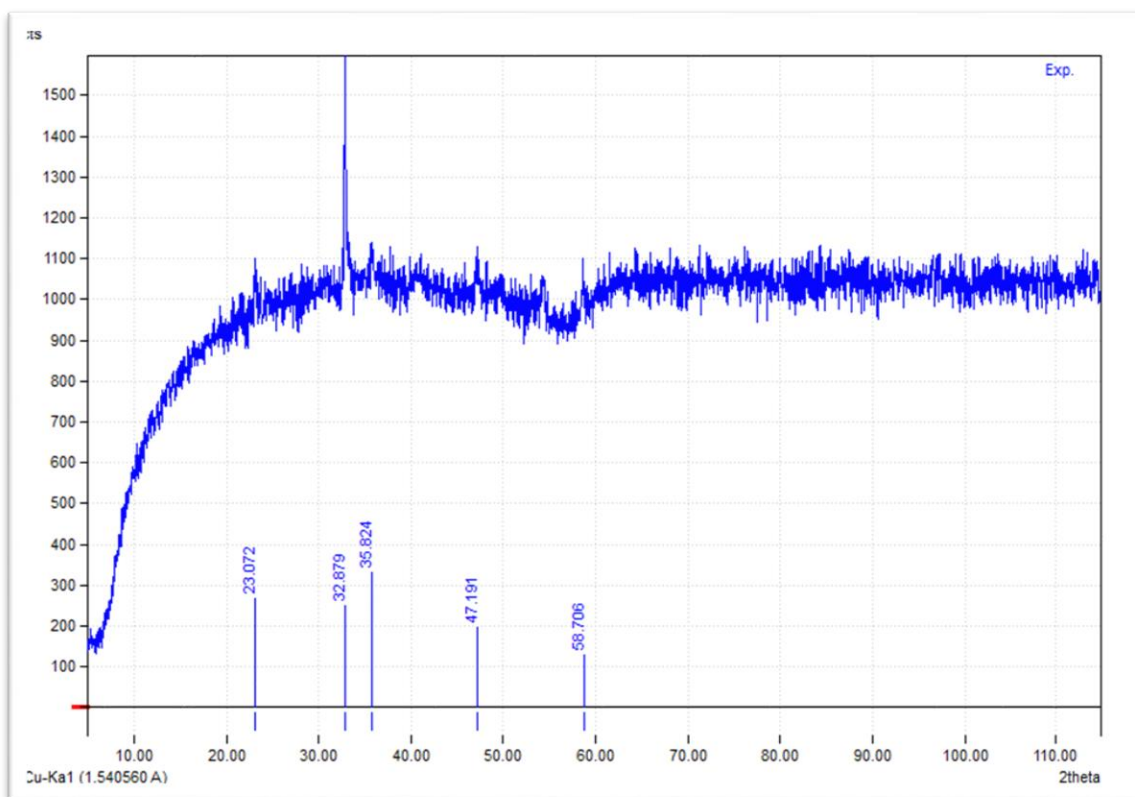


Fig.5.4 XRD Image of SB-NMB.

Table 5.2 XRD Peak List

No.	2theta [°]	d [Å]	I/I0 (peak height)	Counts (peak area)	FWHM
1	23.07	3.8517	430.36	268.46	0.7359
2	32.88	2.7218	1000.00	249.75	0.2946
3	35.82	2.5045	245.79	330.42	1.5859
4	47.19	1.9244	157.56	197.20	1.4765
5	58.71	1.5714	128.32	129.95	1.1947

The X-ray diffraction (XRD) pattern of activated carbon produced from SB and impregnated with iron oxide is shown in Fig.5.4 and the peak list in table 5.2. XRD is a

method for figuring out the underlying crystal structure of a material. Constructive interference between monochromatic X-rays and a crystalline substance is the basic tenet of xrd. These X-rays are produced by a cathode ray tube and collimated before being aimed at the substance to concentrate them. The XRD pattern can be used to determine the locations of magnetic diffraction peaks with miller indices by matching two theta values, which are 23.07, 32.88, 35.82, 47.09, and 58.71 degrees and the strongest peak with a 2theta value of 32.88 degrees, a d-spacing of 2.7218 Å, and a relative intensity (I/I_0) of 1000.00.

5.1.5 Vibrating Sample Magnetometry (VSM).

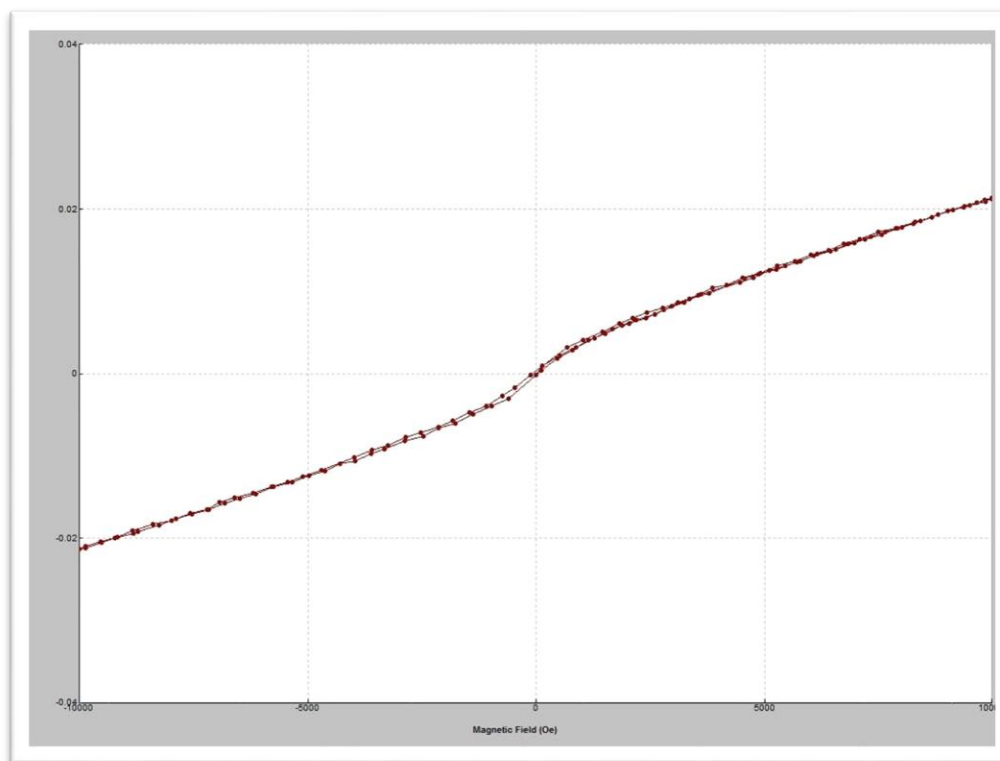


Fig.5.5 VSM image of SB-NMB.

A vibrating sample magnetometer is a common tool for analysing the magnetic characteristics of a variety of materials, including diamagnetic, paramagnetic, ferromagnetic, and anti-ferromagnetic ones. The magnetometer detects magnetic moment directly from an applied magnetic field. According to Faraday's Law of

Induction, VSM works by generating an electric field from a changing magnetic field. The hysteresis loop in Fig.5.5 exhibits a Ferromagnetic signal with a rather dominating paramagnetic signal. Key aspects of the VSM result include: The coercivity is roughly 0.004 T, the saturation magnetization is about 0.04 emu/g, and the remanent magnetization is about 0.02 emu/g. Overall, the VSM finding is consistent with a ferromagnetic material with a predominately paramagnetic signal.

5.1.6 Brunauer–Emmett–Teller (BET)

The BET method is used to determine the specific surface area of porous materials, such as adsorbents and nanoparticles. Its technique is based on measurements of gas adsorption, often nitrogen adsorption at low temperatures, and provides data on the porosity and surface area of materials.

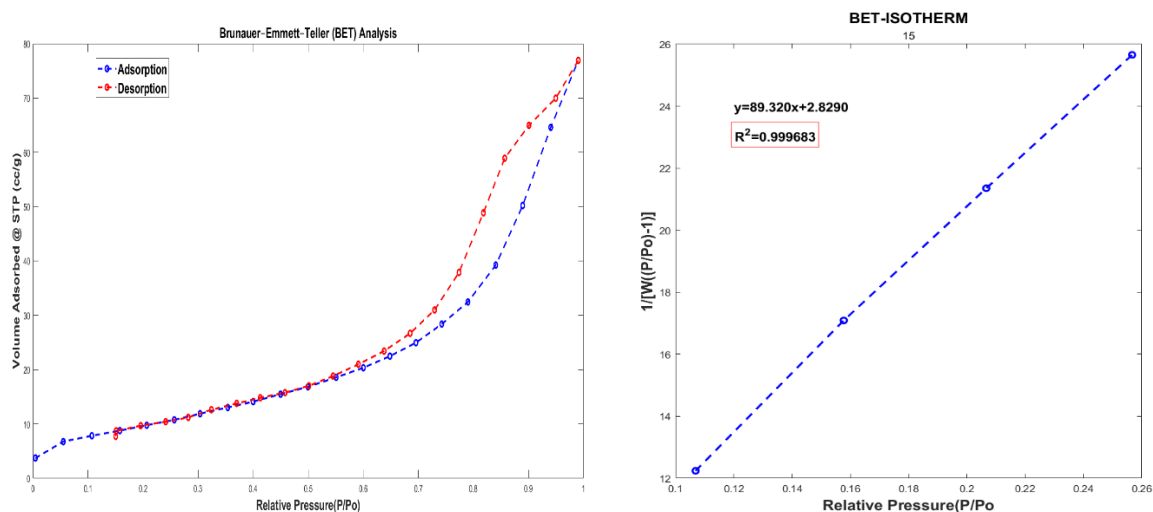


Fig.5.6 BET Image (a) of SB-NMB.

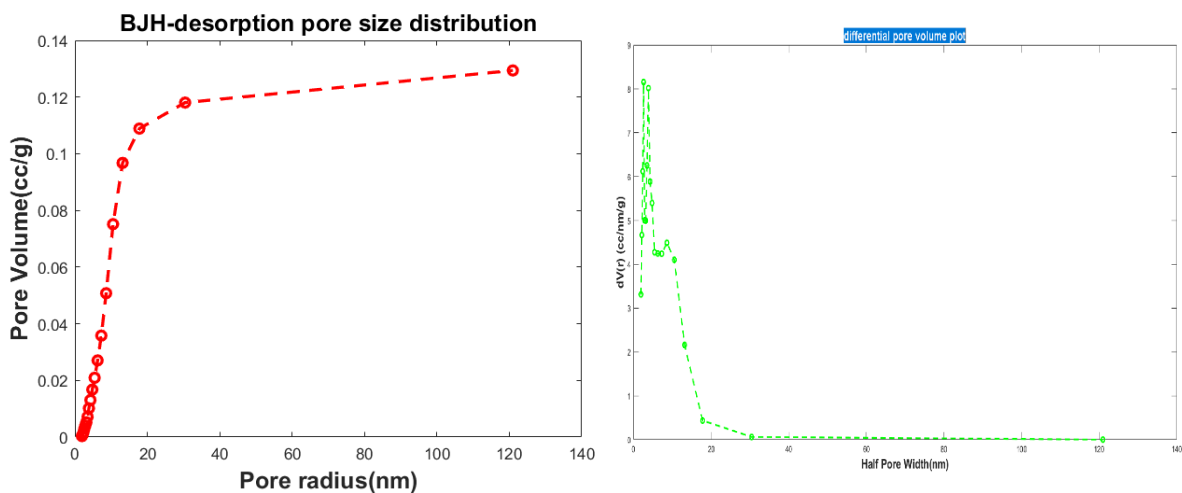


Fig.5.6 BET Image (b) of SB-NMB

Nitrogen gas adsorption-desorption was used to examine the SB-NMB sample's specific surface area and pore size distribution (Fig. 5.6a). The SB-specific NMB surface area in (Fig. 5.6b) is approximately 57.988 m²/g. The BJH method's desorption branch is used to examine the pore-size distribution. The SB-NMB has a pore diameter of 10.530 nm and a pore volume of 10.530 cc/g (Fig. 5.6c).

5.2 OPTIMIZATION PARAMETERS

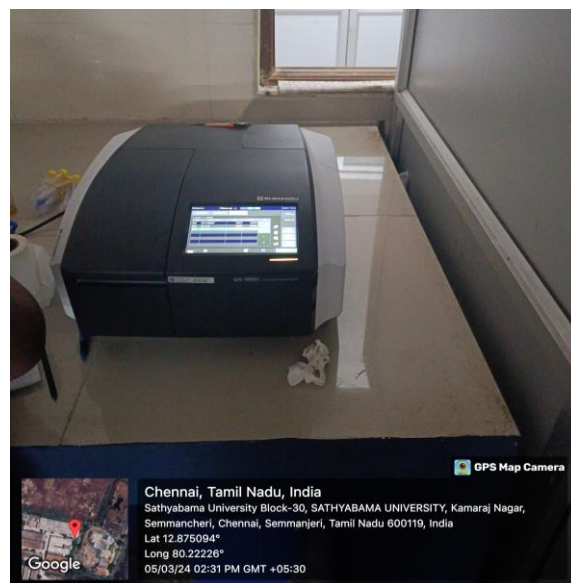
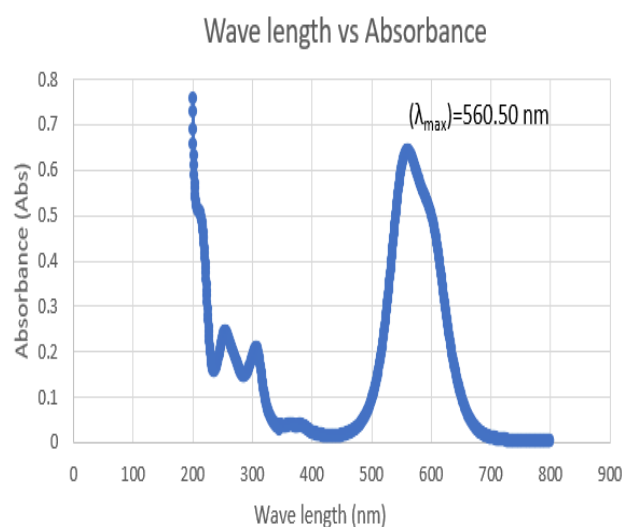


Fig.5.7 Lambda max (λ_{max}) of Acid Blue 9 from ultraviolet-visible spectroscopy

5.2.1 Effect of pH of SB-NMB.

The adsorption onto the SB-NMB was investigated in the pH range of 2–7. Figure 5.8 shows that Acid Blue 9 ions decrease as pH rises. Lower pH values, such as pH 7, which is neutral, allow H⁺ ions to occupy the majority of the adsorption sites on the SB-NMB surface, while Acid Blue 9 ions adsorbed less due to the surface's electrostatic repulsion with H⁺ ions. According to the graph, the disposal percentage falls as the pH range rises, and the ideal pH for 92.05% removal is 5.0.

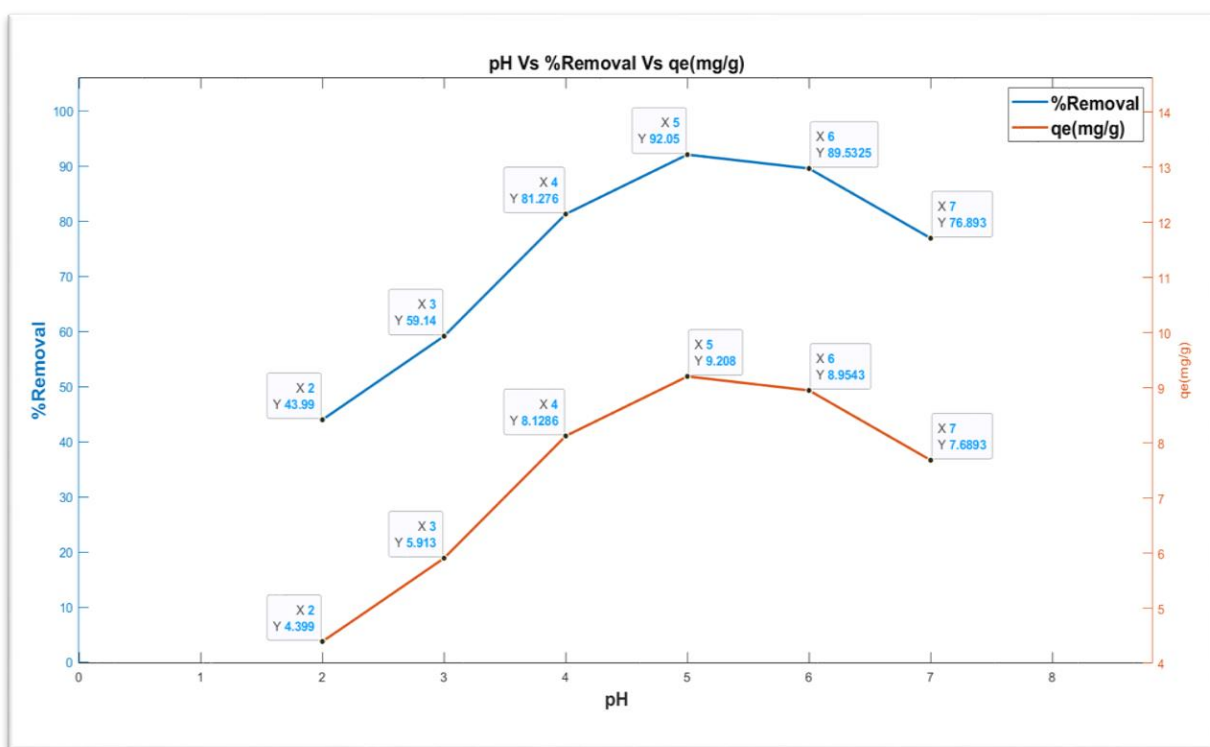


Fig.5.8 Effect of pH on Acid Blue 9 ion removal by SB-NMB (Volume = 100ml, Time = 45 min, Dosage = 0.2 g/l, Conc = 20 mg/l or 20PPM and Temp = 305.15 K and the optimum pH is 5 for 92.05 % Removal and $q_e = 9.208$ mg/g)

5.2.2 Effect of Contact Time of SB-NMB.

A comparison of operation times (0–90 minutes) for the particular removal of Acid Blue 9 using adsorptive SB-NMB is shown in Fig. 5.9. This graph shows that the amount of

Acid Blue 9 ions eliminated increases with time, peaking at 45 minutes. The highest percentage of Acid Blue 9 ion removal can be observed whenever the residential time for Acid Blue 9 ion adsorption over onto adsorbent is long. The study found that the percentage of Acid Blue 9 ions removed increased with contact time, with balancing achieved at 45 minutes for SB-NMB for 5 different starting concentrations.

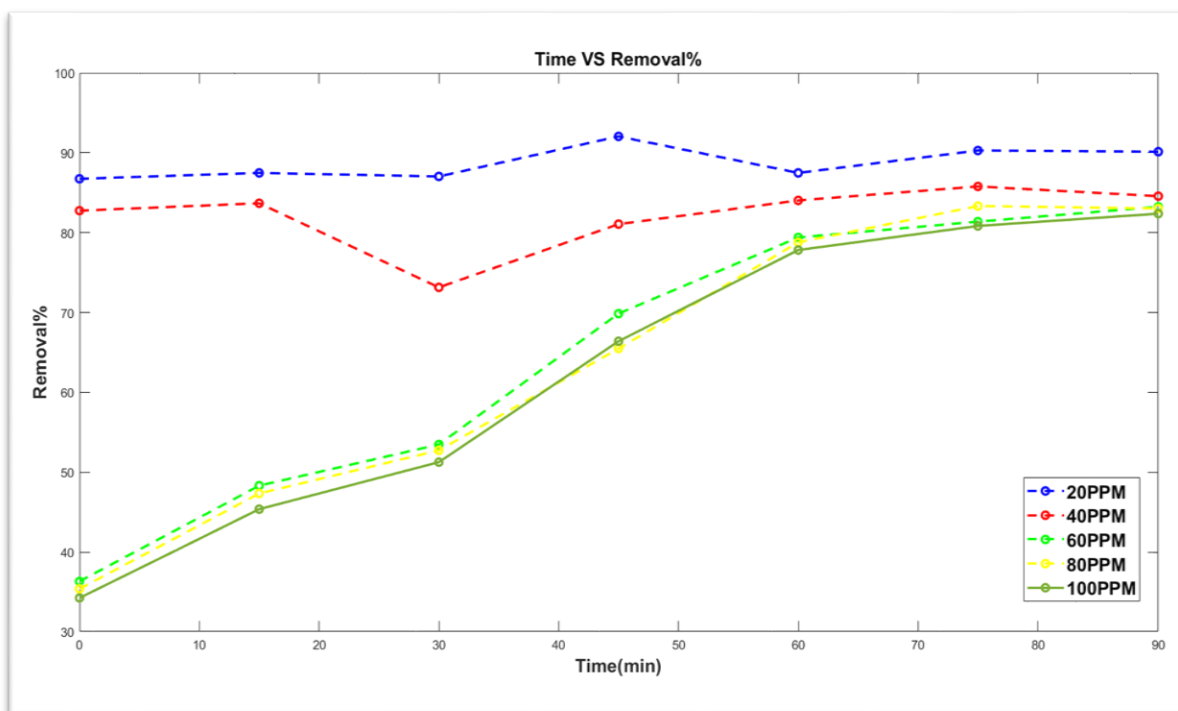


Fig.5.9 Effect of Contact Time on Acid Blue 9 ion by SB-NMB (Volume = 100ml, Dosage = 0.2 g/l, Conc = 20 mg/l, Temp = 305.15 K, pH = 5 and the Optimum Time is 45 min for 92.05 %)

The single, uneven, and continuous adsorption curves that led to saturation suggest that the ion molecules may have covered the adsorbent surface in a monolayer. Given the small difference between the adsorption values at 60 and 90 minutes, a quasi-equilibrium scenario was assumed coupled with a steady-state approximation for the 60-minute contact.

5.2.3 Effect of Concentration of SB-NMB.

An initial ion concentration in the 20–100 mg/L range was investigated, and the effects are shown in Fig. 5.10. The image clearly shows that as the starting ion concentration increased, the proportion of Acid blue 9 ions removed decreased.

The initial ion concentration provides the driving force required to get over the obstacle preventing Acid blue 9 mass transfer between the aqueous phase and solid phase.

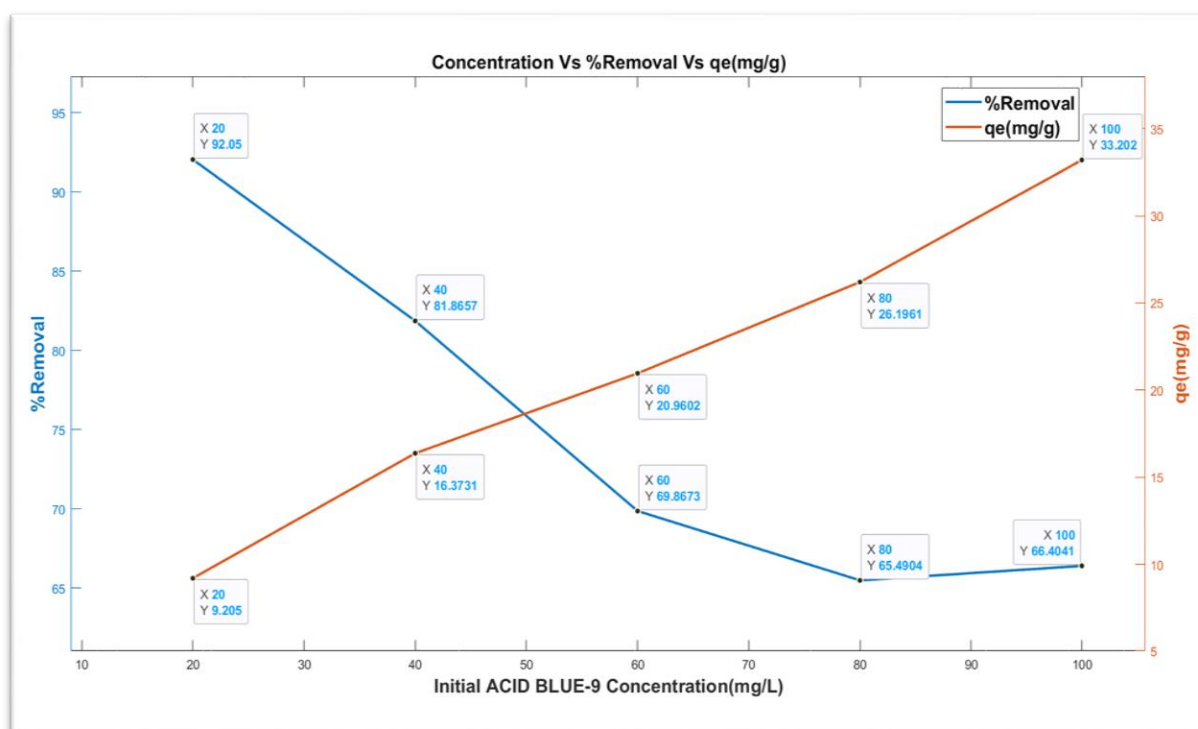


Fig.5.10 Effect of initial Acid Blue 9 ion concentration for the adsorption onto SB-NMB (Volume = 100ml, Time = 45 min, Dosage = 0.2 g/l, Temp = 305.15 K, pH = 5, and the optimum conc is 20 mg/l for 92.05 % Removal, and $q_e = 9.205$)

At pH 5.0, the impact of the initial Acid blue 9 ion concentration (20–100 mg/L) was examined in the presence of the intended adsorbent. The graphs showed that the percentage of Acid Blue 9 ion removal decreased from 92.05% to 66.4% with an increase in Acid Blue 9 ion concentration from 20–100 mg/L.

The drop in the percentage of Acid Blue 9 ion clearance is the primary cause of the decline in catalytic activity reported on SB-NMB beyond a specific concentration of Acid Blue 9 ions. The substantial drop in sorption efficiency is inferred to be caused by the saturation of active sites that are open to sorption at greater concentrations of Acid Blue 9 ions.

5.2.4 Effect of Temperature of SB-NMB.

The ideal temperature conditions are shown in the above figure 5.11. The temperature ranges from 305K to 335K. The maximum percentage removal occurs at 305.15K, which is the ideal temperature. For the sample, this measurement is made at several doses, which range from 20 to 100 mg/L. The value of % removal steadily drops as the temperature rises.

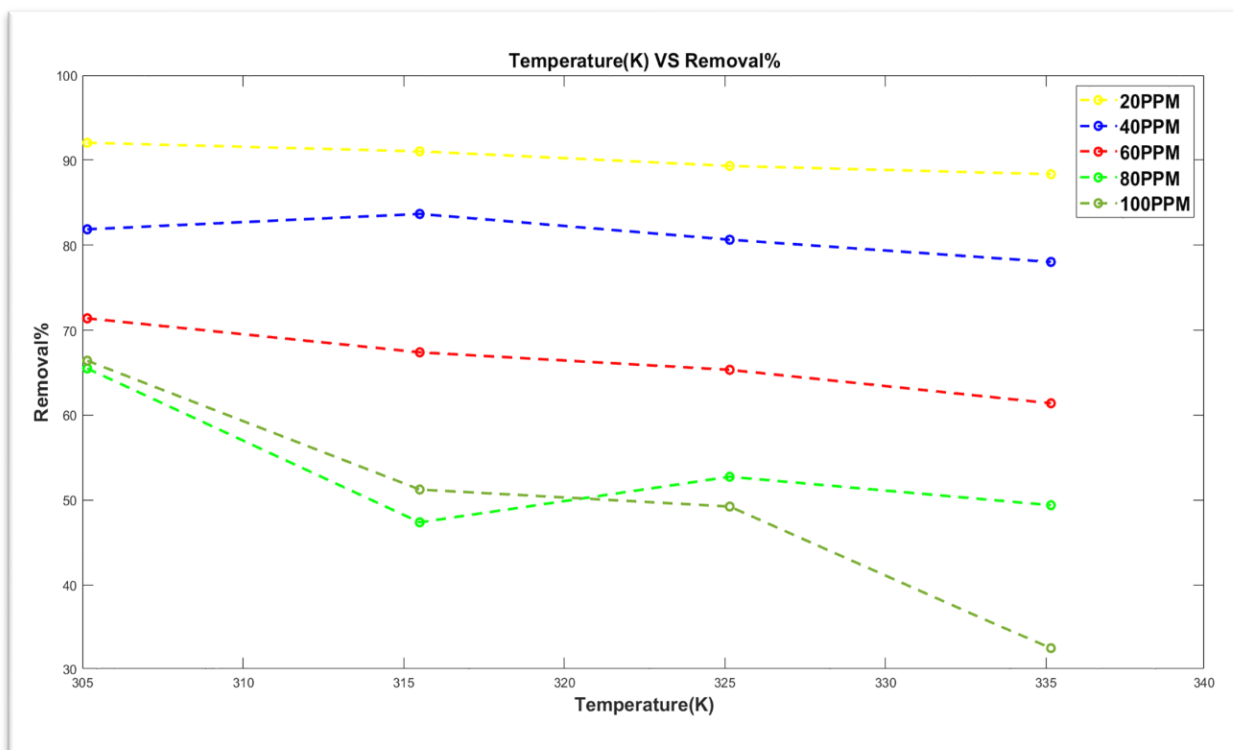


Fig.5.11 Effect of temperature on Acid Blue 9 ion removal by SB-NMB (Volume = 100ml, Time = 45 min, Dosage = 20mg/l, Conc = 20 mg/l, pH = 5 and the optimum Temp = 305.15 K for 92.05 % Removal, and $q_e = 9.205$)

This is primarily because, as Fig. 5.11 illustrates, the decrease in surface activity suggests that the adsorption process between the adsorbents and Acid Blue 9 ion is exothermic.

5.2.5 Effect of Dosage of SB-NMB.

A important factor in determining the adsorption potential of a given starting concentration of Acid Blue 9 ions is the dosage of SB-NMB.

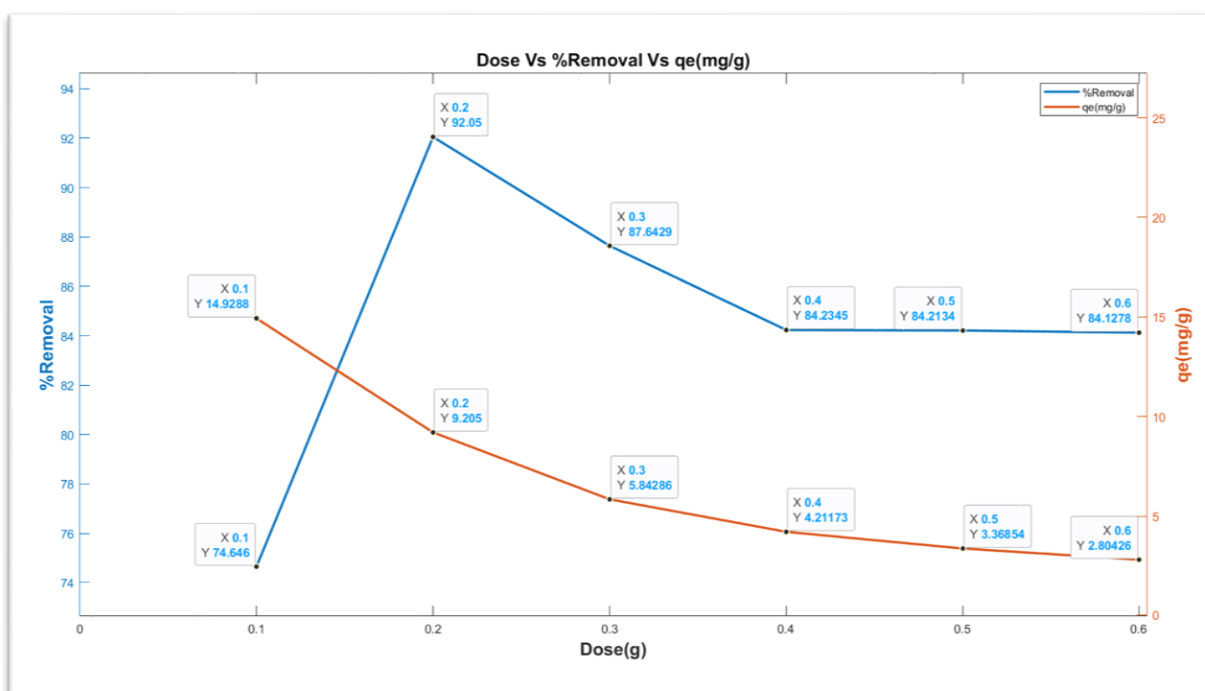


Fig.5.12 Effect of adsorbent dose on Acid Blue 9 ions removal by SB-NMB (Volume = 100ml, Time = 45 min, Conc = 20 mg/l, Temp = 305.15 K, pH = 5 and the optimum Dosage is 0.2 g/l for 92.05 % Removal and $q_e = 9.025$)

The plot of the adsorbent dose against the percentage removal (%) is shown in Fig. 5.12. It proves that the percentage of Acid Blue 9 ions removed changes as the adsorbent mass and the adsorbent dose change. The elimination percentage increases from 74.6% to 92.05% and then gradually declines with an increase in adsorbent dosage of between 0.1 and 0.6g/L. As indicated in Fig. 5.12, the ideal dosage is 0.2 g/L.

5.3 ADSORPTION ISOTHERMS

The adsorption isotherm shows the relationship between the adsorbate on the adsorbent and the adsorbate in the fluid medium in an intermediate state. To evaluate adsorption isotherm data, it is necessary to compare it to several isotherm models that can be used for the adsorption design phase. This improves the building of an adsorption process for the absorption of adsorbate.

The Langmuir, Freundlich, and Temkin models are used in this study to assess the adsorption equilibrium between Acid Blue 9 ions and SB-NMB. Using MATLAB R2023b, the graphs are plotted and these isotherms' validity was investigated. The solutions are displayed in Fig. 5.13. (a) and (b), Table 5.3 shows the coefficient of determination and the relevance of the adsorption isotherm variables.

The equilibrium adsorption statistics per R^2 standard may be adequately described by the Freundlich adsorption isotherm theory, based on the data in Table 5.3. In addition, the Freundlich model outperforms the Langmuir, and Temkin theories in terms of fit to the experimental data. The Freundlich adsorption isotherm, which would have been appropriate to either a sole exposure adsorbed species each of one limited concentration scope, is in concept a functional application if an adsorb besides a highly incongruous high adsorption layer. The implications of the value of n , that supplies an indication of regarding pseudo-linearity's extent here among the intensity on the remedy with adsorption, to be as follows:

linearly ($n = 1$),

chemical ($n < 1$),

physical ($n > 1$).

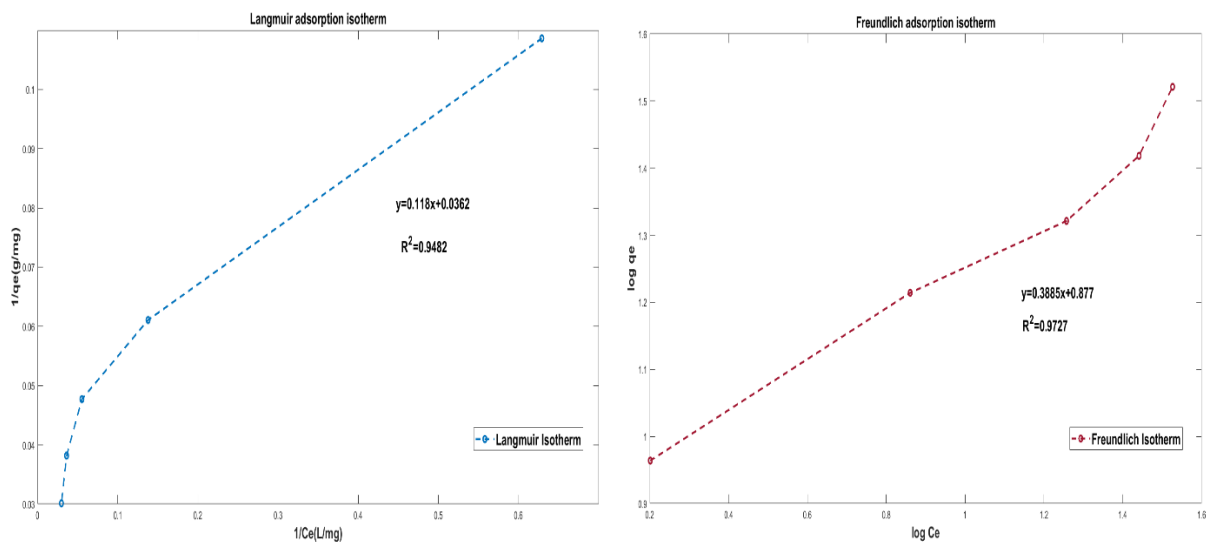


Fig.5.13 (a) Langmuir and Freundlich Adsorption isotherm for the removal of Acid Blue 9 ions by SB-NMB

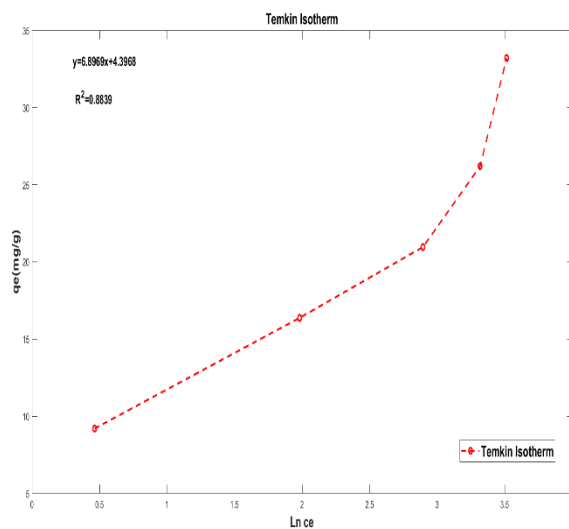


Fig.5.13 (b) Temkin Adsorption isotherm for the removal of Acid Blue 9 ions by SB-NMB

According to Table 5.3, the n amount was calculated to be 2.574. This a n -value corresponds to existing adsorption structure anywhere around 1 and 10, implying believed adsorption was indeed a physical entity one. An SB-NMB was determined to have a highest monolayer adsorption capacity (q_{max}) for metal adsorption of 27.6243 mg/g.

The weak interaction between Acid Blue 9 and SB-NMB suggests that there may be difficulties during the adsorption process. However, an evolving adsorption strategy and thorough statistical measure evaluation have led to the identification of a particular combination of parameters or algorithms, which is the most successful in forecasting the adsorption of Acid Blue 9 onto SB-NMB. The above demonstrates that perhaps the current approach of adsorption had been an evolving method. Based on R^2 and error values, the accompanying is really the finest combination for the algorithms for isotopes of adsorption examined for extraction Acid Blue 9 particle in SB-NMB.

Freundlich > Langmuir > Temkin isotherms

The results of the experiment show a significant complementarity between empirical data and theoretical expectations, with the experimental findings closely matching the predictions of the Freundlich model. This validation reaffirms that the Freundlich theory can be applied to the ion elimination of Acid Blue 9 by SB-NMB.

Table 5.3 Adsorption isotherm parameters for removing Acid Blue 9 ions by SB-NMB

ADSORPTION ISOTHERM MODELS	DIMENSIONS	VALUES	R^2
Langmuir	$q_{\max}$ (mg/g)	27.6243	0.9482
	B (lm/g)	0.30672	
Freundlich	K_f	7.5335	0.9727
	n (g/L)	2.574	
Temkin	B_T (J/mol)	6.8969	0.8839
	K_T (l/mg)	1.89	

5.4 ADSORPTION KINETICS AND MECHANISM

The ratio and sorption mechanism involving Acid Blue 9 ions and SB-NMB were then predicted using the equilibrium adsorption data. Three kinetic models are investigated

in this work. The correct solutions are shown in Fig. 5.14. These are the pseudo-first-order, pseudo-second-order, and Elovich kinetic models that assisted in understanding sorption kinetics for removing Acid Blue 9.

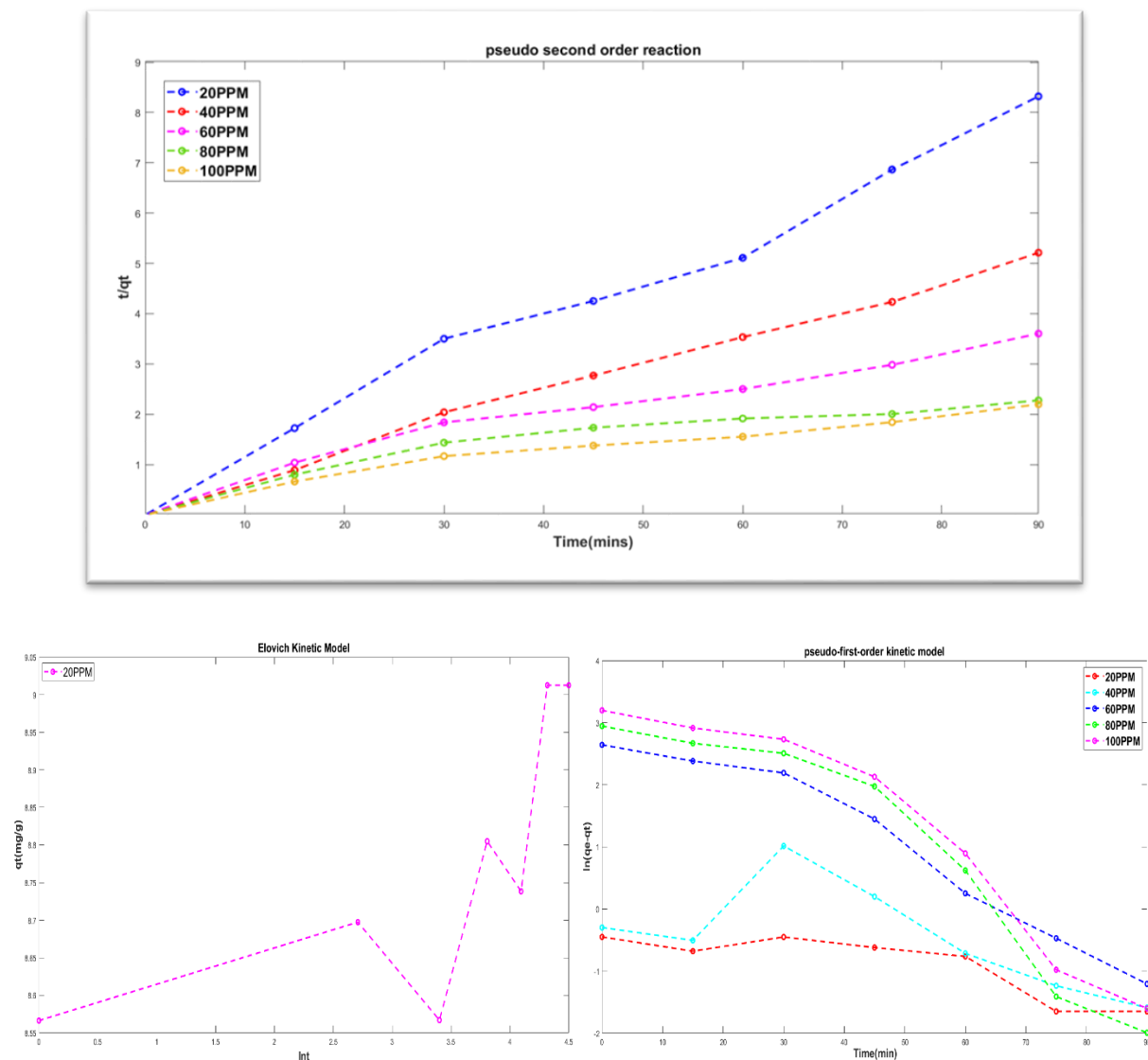


Fig.5.14 Adsorption kinetic for eradicating Acid Blue 9 ions by Sb-NMB (Acid Blue 9 concentration = 20 mg/L, solution pH = 5.0, adsorbent dose = 0.2 mg/L, volume of sample= 100mL, temperature = 303.15 K)

Predictions for a range of configurations are shown in Table 5.4, along with pseudo-first-order kinetic constant (k_1) and pseudo-second-order kinetic constant (k_2), as well as equilibrium adsorption capacity (q_e).

The coefficient of determination (R^2) for the pseudo-second-order kinetic model was shown to be greater when compared to the pseudo-first-order model. This implies that the observed adsorption rates are better described by the pseudo-second-order model. The physical adsorption phase %, which is obtained by fitting sorption kinetics, shows that a pseudo-second-order kinetic model describes the removal of Acid Blue 9 onto SB-NMB. Determining the process's rate-limiting stage requires an understanding of the adsorption mechanism.

Table 5.4 Adsorption Kinetic value for elimination of Acid Blue 9 ions by SB-NMB

Kinetic Model	Parameters	Concentration of Acid Blue 9 ion solution (mg/L)				
		20	40	60	80	100
Pseudo-first-order equation	K_1 (min^{-1})	-0.000154	-0.0001655	-0.0004977	-0.0009033	-0.0005833
	q_e (mg/g)	0.73425	1.32644	21.70174	36.00291	43.94764
	R^2	0.7715	0.3033	0.9434	0.8308	0.8528
Pseudo-second-order equation	K_2 (min^{-1})	0.03125	0.0323	0.00392	0.00150	0.001158
	q_e (mg/g)	11.389	17.3611	26.7379	40.98	51.54
	R^2	0.9868	0.9959	0.9601	0.8812	0.8821

The equilibrium adsorption capacity (q_e), which is derived using pseudo-second-order kinetic parameters, and the experimental q_e values show a significant agreement, according to the observations. This agreement confirms that the pseudo-second-order kinetic model is applicable in real-world scenarios.

5.5 EVALUATION OF THERMODYNAMIC PARAMETERS

A thermodynamic principle provides structure to the underlying enthalpy change associated with the adsorption process. The process was defined using the

thermodynamic parameters change in free energy (ΔG°), change in enthalpy (ΔH°), and change in entropy (ΔS°) rather than SB-NMB.

As shown in Figure 5.15, the values of ΔH° and ΔS° were obtained from the intercept and gradient, respectively, of a linear plot of $\log K_c$ against $1/T$. The equation can be used to calculate thermodynamic parameters using this graphical form.

The values of the estimated thermodynamic parameters are shown in Table 5.5. The table illustrates the spontaneous character of the reactions and the stability of the adsorbate at varying temperatures through negative values of ΔG° . Furthermore, it appears that the adsorption process is advantageous within a specific temperature range based on the growing negativity of ΔG° as temperature drops. Increased temperature may make previously deposited Acid Blue 9 ions more mobile, which could result in desorption from SB-NMB's inner and exterior surfaces.

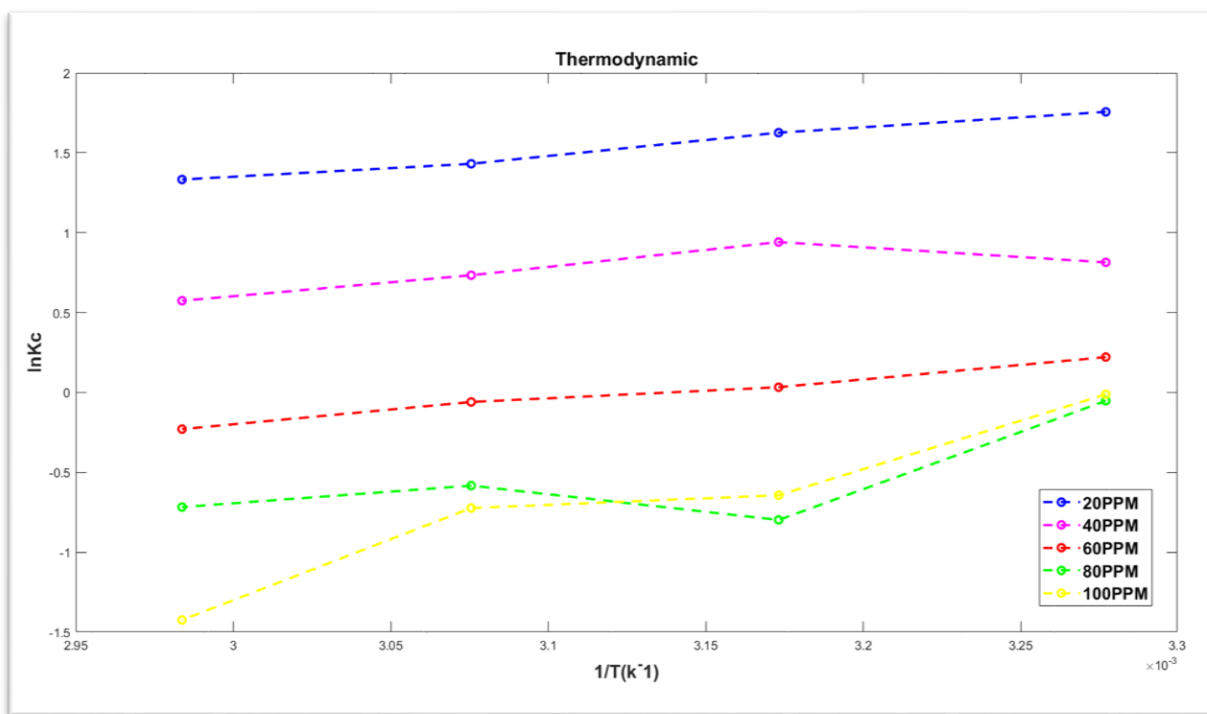


Fig 5.15 Thermodynamic analysis for the adsorption of Acid Blue 9 by SB-NMB

A negative value of ΔG° denotes spontaneity and offers insights into the adsorption process. Because of the weak interaction between Acid Blue 9 ions and SB-NMB,

physical adsorption is confirmed in this instance by the negative ΔH° , which indicates an exothermic sorption process. Decreased unpredictability at the surface is indicated by the negative ΔS° , which is probably caused by the gain of particle motion after adsorption i.e, the system becomes more ordered. Furthermore, enthalpy changes have a stronger effect on adsorption than entropy changes, as indicated by larger ΔH° , highlighting their importance in the process.

Table 5.5 Thermodynamic parameters for the adsorption of Acid Blue 9 ions onto SB-NMB

Initial Acid Blue 9 ion Conc. (mg/L)	ΔH° (kJ mol ⁻¹)	ΔS° (Jmol ⁻¹ k ⁻¹)	ΔG° (kJ mol ⁻¹)			
			305.15K	315.15K	325.15K	335.15K
20	-12.4619	-26.19	-4.46998	-4.20808	-3.94618	-3.68428
40	-7.73293	-17.8177	-2.2985	-2.11768	-1.9395	-1.76132
60	-12.2864	-38.4963	-0.53927	-0.154307	-0.230656	-0.61561
80	-15.4566	-52.8097	-0.658322	-1.186418	-1.714515	-2.24261
100	-36.663	-120.486	-0.10254	-1.307408	-2.512273	-3.717138

5.6 CHARACTERIZATION AFTER ACID BLUE 9 ADSORPTION

5.6.1 Fourier Transform Infrared Spectroscopy (FTIR).

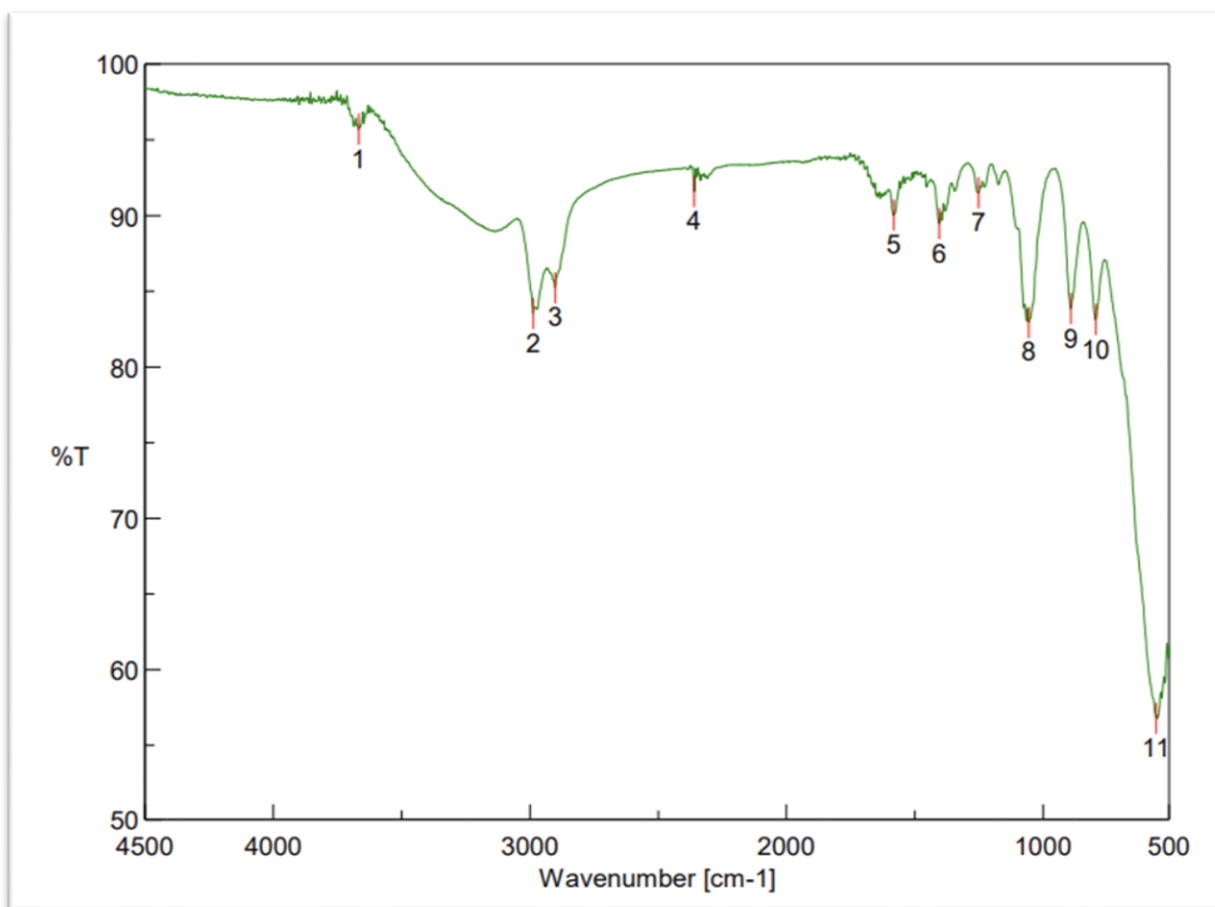


Fig. 5.16 Fourier-transform infrared (FTIR) after adsorption

The Fourier-transform infrared (FTIR) spectrum shown in Figure 5.16 shows several unique peaks, each of which corresponds to a particular chemical bond or functional group. At 3666.98 cm⁻¹, a band was found, indicating the presence of hydroxyl groups. Stretching C-H bonds were indicated by the peaks at 2987.2 cm⁻¹ and 2901.38 cm⁻¹. The presence of C≡N bonds was revealed by the occurrence of peaks at 2359.48 cm⁻¹, and at 1582.31 cm⁻¹ indicating the presence of N-H bond. N=O bonds were present, as evidenced by a peak at 1404.89 cm⁻¹. A peak at 1251.58 cm⁻¹ represented C-N Amine bonds. The presence of C-X bonds was suggested by a peak at 1055.84

cm^{-1} . At 890.952 cm^{-1} , the C-H bond was found. The spectrum displayed C-H bond aromatic out-of-plane bending at 792.6 cm^{-1} . At 551.542 cm^{-1} , a peak that may have been caused by bromide, iodide or chloride C-X bonds was also seen. The synthesis of SB-NMB adsorbent is validated by comparing the FTIR spectrum.

5.6.2 Scanning Electron Microscopy (SEM).

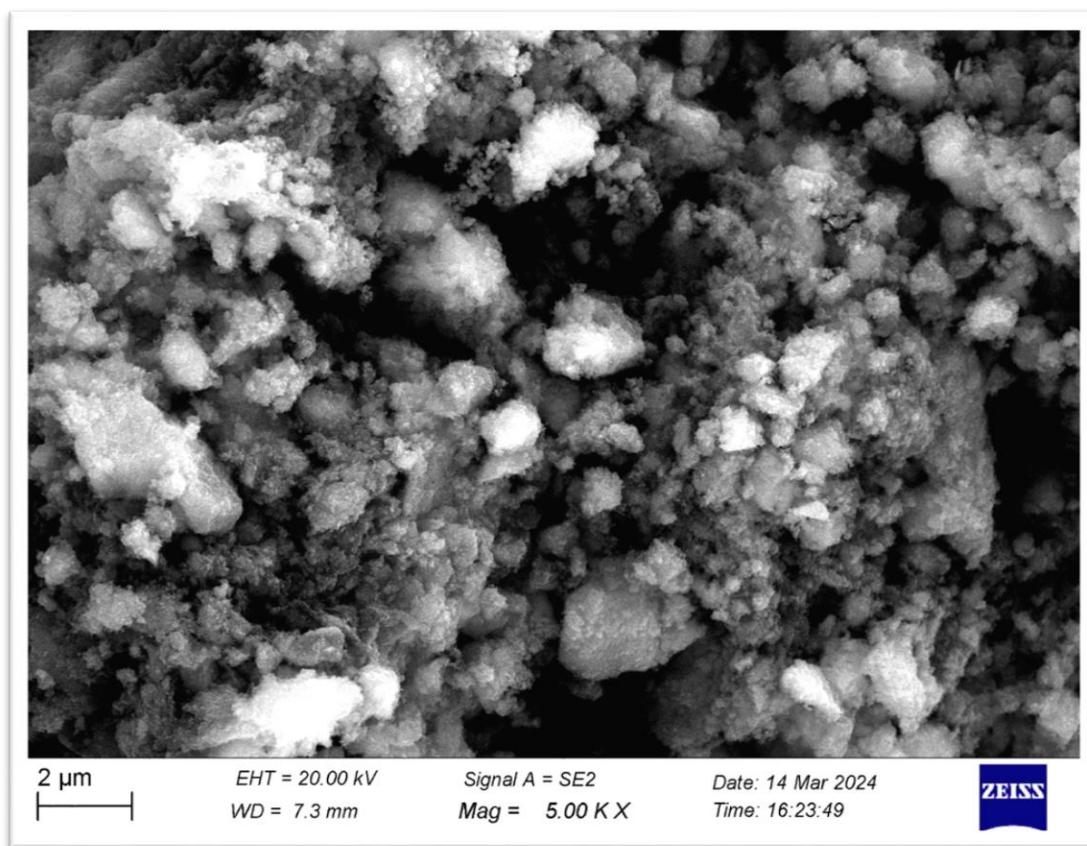


Fig.5.17 Sem Image (a) of SB-NMB after adsorption.

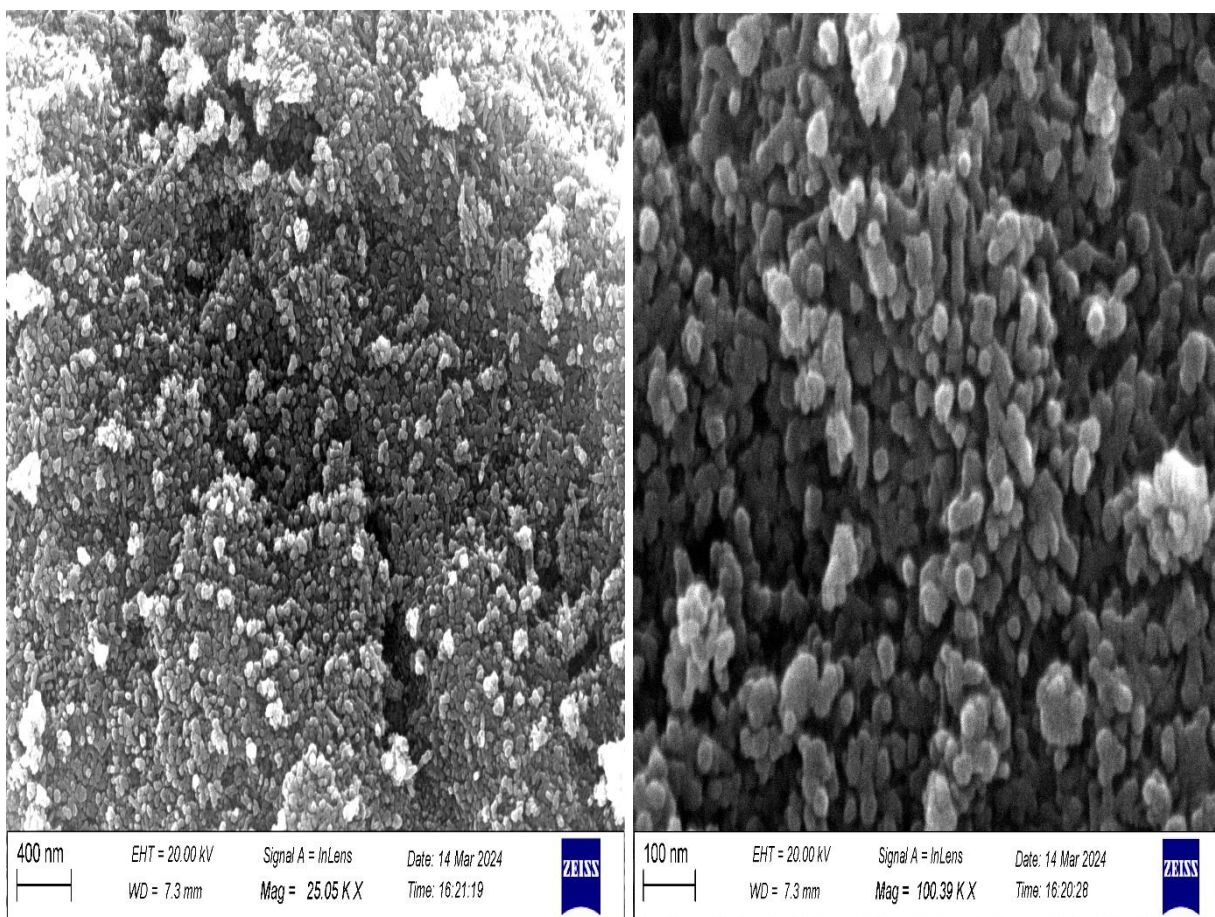


Fig.5.17 Sem Image (b) of SB-NMB after adsorption.

Following dye uptake, SB-NMB was subjected to scanning electron microscopy to look for any potential alterations in the surface morphology (Fig. 5.17 (a) and (b)). The surface morphology demonstrated that sugarcane bagasse after dye uptake had few pores, but SB-NMB had greater pore diameters before dye uptake. This could be the result of the dye and other produced tiny complex molecules occupying the pores.

5.6.3 Energy Dispersive X-Ray (EDX).

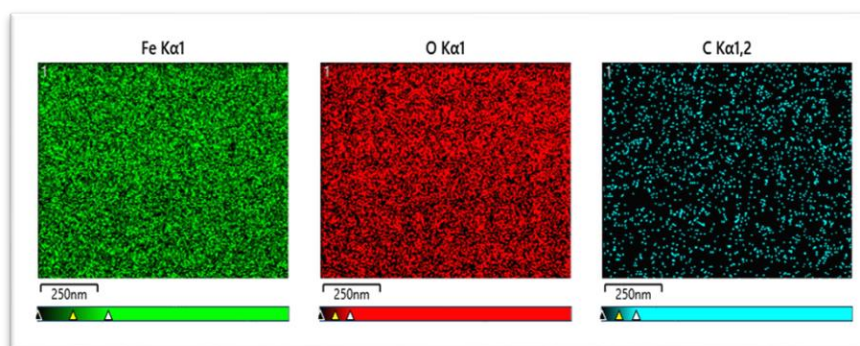
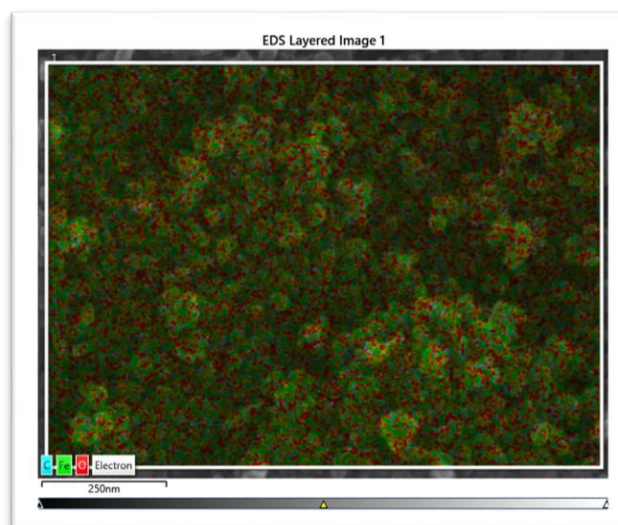


Fig.5.18 EDX Image (a) of SB-NMB after adsorption.

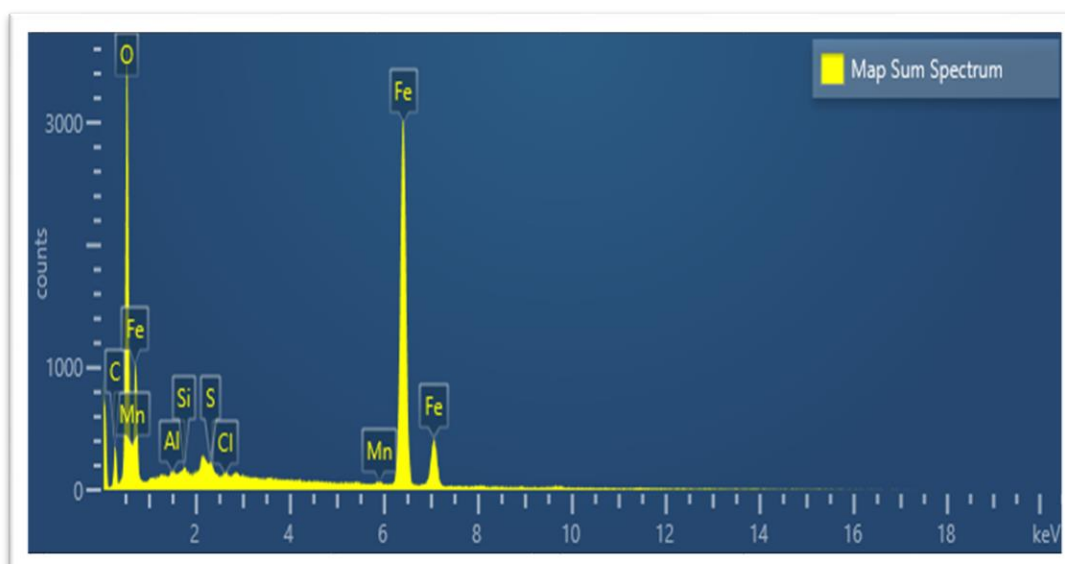


Fig.5.18 EDX Image (b) of SB-NMB after adsorption.

Table 5.6 EDX Map Sum Spectrum of SB-NMB

Map Sum Spectrum Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	15.17	0.70	29.62
O	K series	32.98	0.39	48.34
Al	K series	0.21	0.06	0.19
Si	K series	0.18	0.05	0.15
S	K series	0.18	0.05	0.13
Cl	K series	0.21	0.04	0.14
Mn	K series	0.29	0.08	0.12
Fe	K series	50.77	0.50	21.32
Total		100.00		100.00

Figures 5.18 (a) and (b), Table 5.6 show the EDX pattern with the SB-NMB weight percentage.

The elemental makeup of the SB-NMB sample was examined using EDX analysis. According to the EDX pattern, the synthesised SB-NMB sample is mostly made up of Carbon (15.17%), Iron (50.77%), and oxygen (32.98%).

CHAPTER-6

6. SUMMARY AND CONCLUSION

Adsorption technology is widely acknowledged as being extremely beneficial in removing dyes, heavy metals, and other harmful effluents from water. This work primarily focuses on the recently developed SB-NMB for the removal of Acid Blue 9 contaminants from water. Analytical methods were used to characterize SB-NMB and due to their exceptional adsorption effectiveness, the use of magnetic nano-based adsorbents offers tremendous promise. Using this method, dye removal can be accomplished more affordably than with more expensive adsorbents now in use. The study also highlights the possibility for other adsorbents including Banana peels, Pomegranate peels, Eggshells and prawn shells, which can be recycled from biowaste items that would otherwise be worthless. According to the research results, the optimum readings are pH 5, contact time 45 minutes, concentration 20 mg/l, temperature 305.15 K and the dosage is 0.2g/l. To evaluate adsorption isotherm data, the Langmuir, Freundlich, and Temkin models are used and the Freundlich model outperforms with an R^2 value of 0.9727 and n as 2.574 g/L. The $q_{\max}$ of Langmuir is 27.6243 mgg⁻¹. pseudo-first-order, pseudo-second-order, and Elovich kinetic models have assisted in understanding sorption kinetics and based on R^2 the adsorption rates are better described by the pseudo-second-order model. Thermodynamic characteristics which include Gibbs free energy, enthalpy, and entropy estimates demonstrate that the adsorption process was spontaneous and Exothermic. The characterization was done after adsorption with FTIR which indicated the broadening of peaks due to the adsorbate molecules interaction, SEM indicated the reduction in pores due to the dye uptake and EDX detected the changes in Elemental Composition. In conclusion, research into using magnetic nanoparticles as adsorbents for dye removal from wastewater shows compelling advantages over conventional adsorbents like activated carbon, including affordability, reusability, and improved selectivity.

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ABSTRACT

Water pollution, which has become a major issue, is primarily caused by pollutants such as dyes, and water treatment techniques have become critical for addressing this issue. Adsorption was chosen among the several removal techniques as it is inexpensive, environmentally friendly, composed of renewable biodegradable materials, and offers a variety of adsorbent alternatives. In this study, Sugarcane Bagasse was impregnated with Nano Magnetic Particles (NMPs) utilising the Co-precipitation process to produce a Sugarcane Bagasse – Nanomagnetic Biosorbent (SB-NMB) composite to remove Acid Blue 9 which is a synthetic organic compound utilised as a blue colourant in pharmaceuticals and many other industries. Sugarcane Bagasse is a waste product with little economic value and has demonstrated excellent potential as a biosorbent due to its superparamagnetic properties. The characteristics of the biosorbent were investigated in this study using various characterisation techniques like FTIR which analysed the functional groups. SEM with EDX analysed the porous nature and the weight percentage of the particle. XRD showed the crystalline structure, VSM exhibited a Ferromagnetic signal with a rather dominating paramagnetic signal and BET showed the surface area to be 57.988 m²/g. Adsorption studies to remove acid blue 9 were conducted utilising a range of synthetic dye solution concentrations (20-100 mg/L), pH (2-7), adsorbent dosage (0.1-0.6g/L), and contact time (0-90 min). The adsorption isotherm and kinetics show that the Freundlich isotherm and Pseudo-second-order concepts offered the greatest fitting. Thermodynamic studies prove that the adsorption mechanism was spontaneous and this investigation yielded removal % efficiency and capacity (mg/g) of 92.05% and 9.205 mg/g, respectively, therefore the study's findings offer a lot of promise for tackling water pollution concerns.

Keywords- Adsorption, water pollution, Acid Blue 9, SB-NMB.

CHAPTER-1 INTRODUCTION

Rapid worldwide industrialization in the Textile, Cosmetics, Metal processing, fertilizer, pulp and paper, rubber-plastic, batteries, mining, electroplating, pharmaceutical and metallurgical industries has increased the water resources pollution(Izydorczyk et al., 2021). Color-rendering compounds, such as dyes and pigments, if released untreated can increase the biochemical oxygen demand in rivers, blocking light transmission into the water and as a result, interfering with aquatic natural processes that depend on light(Subbareddy et al., 2020). The organic azo dye Acid Blue 9, usually referred to as FD&C Blue No. 1, is one of the most significant dyes. Its molecular formula is $C_{37}H_{39}N_3O_9S_3$, and its molecular weight is 765.91.

This water-soluble dye is widely utilised in a variety of applications, including dyeing fabrics such as wool, polyamide fibre, and silk and is also utilised as an analyte in the development of azo-dye removal methods. When humans come into direct contact with Acid Blue 9, can cause eye and skin irritation. Inhaling its fumes or dust particles might irritate the respiratory system, causing trouble breathing. Prolonged or recurrent exposure might have more serious health consequences, such as allergic reactions and organ damage. The manufacture and disposal of Acid Blue 9 can pollute the air, and water and generate hazardous waste. Improper handling and disposal of this dye can lead to soil pollution and worsening environmental degradation. It is considered a contaminant because of its water solubility, and its presence in sewage poses a threat to natural water reserves and living species, harming water quality even at low levels(Neetha et al., 2018).

Runoff water from industrial operations contains dyes that harm nearby water sources. To avoid health hazards, removing these dangerous pigments before disposing of wastewater is essential. Hazardous compounds that have been released into the environment in large quantities have caused several ecological concerns due to their persistence and non-biodegradability(Andrade Siqueira et al., 2020). Traditional techniques, like chemical coagulation, liquid-liquid exchangers, ion exchanges,

membrane filtration, electrical, chemical removal, and coagulation, have downsides such as insufficient removal, high energy requirements, hazardous sludge, limited efficiency, delicate working conditions, and expensive disposal(Bhattacharjee et al., 2020).

Adsorption is the promising technological method for removing dyes from wastewater. It has several benefits, including low energy consumption, the ability to use adsorbent materials made from waste or byproducts, and cost-effectiveness in terms of both initial investment and ongoing operations(Shokri and Fard, 2022). Adsorption has received a lot of attention recently and is currently an appealing choice for treating wastewater. This method is preferred over other removal methods due to the low amount of chemicals it uses and the variety of available adsorbents. Dyes and heavy metals can be removed from wastewater using this simple, manageable technology that is safe and effective even at deficient pollution concentrations(Türkmen et al., 2022). sugarcane bagasse, Banana peels, potatoes, apples, lemons, coffee grounds, decaf coffee grounds, grape seeds, papaya seeds and orange peels have all been utilised as inexpensive adsorbents(Frezzini et al., 2019).

Agricultural waste was chosen because it is readily available, easy to procure, and potentially solves the agricultural waste disposal problem. Sugarcane (*Saccharum officinarum*) is crushed for juice extraction, and bagasse, which amounts to about 25% of the overall weight and consists 60% to 80% of carbohydrates, is discarded as agricultural waste(Rezende et al., 2011). Sugarcane bagasse (SB) is one of the major agro-industrial wastes that consists of lignin (5-20%), cellulose (30-80%), and hemicellulose (5-40%)(Nunes et al., 2020).

Sugarcane bagasse's been investigated as a potential biosorbent for dye removal, especially when converted into nanomagnetic forms(Aruna et al., 2021). Sugarcane bagasse was employed to remove safranin O, with adsorption capacities of 62.884 mg/g for base-treated SB, 58.853 mg/g for raw SB, and 54.822 mg/g for acid-treated Sygarcane Bagasse, respectively. It was known as an efficient adsorbent for Congo-red adsorption, with the adsorption capacity of 38.2 mg/g and a pseudo-second order kinetic model. According to the study, the adsorption capability for methylene blue and

acid orange II elimination using sugarcane bagasse treated with propionic acid was 59.5 and 25.5 mg/g respectively (Ahmad et al., 2018).

Objectives of the study are

- (i) To Synthesize sugarcane bagasse-derived nanomagnetic biosorbent composite (SB-NMB) to remove Acid Blue 9 from aqueous solutions.
- (ii) To characterize and analyze the properties and performance of the synthesized SB-NMB, such as FTIR, SEM, EDX, XRD, VSM, BET adsorption capacity, and kinetics surface properties.
- (iii) To obtain conclusive findings and data regarding the performance of the SB-NMB in removing Acid Blue 9 from aqueous solutions.

The batch studies were evaluated, including the solution's pH, contact time, dye concentration, temperature, and adsorbent dosage. To further comprehend the experimental data, isotherm, kinetic, and thermodynamic parameters were examined as well.

1.1 TYPES OF DYES

1.1.1 Azo dyes

Synthetic organic compounds with the azo bond ($-N=N-$) are azo dyes. They consist of an azo link connecting two aromatic amines. Azo dyes are utilized in several industries, such as leather, textiles, cosmetics, food, and pharmaceuticals. Azo dyes are large synthetic organic dyes that make up more than half of the dye produced annually worldwide. An azo dye's colour is influenced by the rest of the molecule's structure, including the presence of functional groups (Vaiano and De Marco, 2023).

Azo dyes, which contain the nitro group known for its mutagenicity and can yield harmful byproducts like 1,4-phenylenediamine and o-tolidine upon degradation, are still used and produced worldwide due to their economic value and advantageous characteristics, despite being banned in certain parts of the world due to their toxicity

and carcinogenic potential, as well as their capacity to induce allergic reactions and mutagenic activity(Gupta et al., 2006).

1.1.2 Reactive dyes

Reactive dyes are chemicals that combine with other molecules to form new compounds. They are employed in a wide range of chemical processes, including food colouring, textile dyeing, and pharmaceutical manufacturing. The majority of the time, reactive dyes are used to colour cellulose fibres like viscose and cotton. Additionally, they have evolved into more and more significant for wool and polyamide. Reactive dyes are mostly used for printing cellulosic fibres like cotton and linen because of their broad colour spectrum(Ahmad et al., 2018).

Reactive dyes can sometimes result in hypersensitivity and allergic skin reactions. Based on the chemical benzidine, a few dyes are believed to be carcinogenic.

1.1.3 Metal Complex dyes

Metal complex dyes are a class of dyes that contain a central metal ion coordinated to organic molecules called ligands. These ligands are typically azo dyes, which means they contain an azo group ($-N=N-$). The metal ion and the ligands interact with each other through coordination bonds. The colour, stability, solubility, and substrate affinity of a metal complex dye are all significantly influenced by metal ions and ligands selection(Kolya and Kang, 2024).

Metal ions commonly utilised in metal complex dyes include copper, chromium, nickel and cobalt. Adverse health effects, including skin sensitivity, lung cancer, liver and kidney damage, and respiratory irritation, can result from exposure to metal complex dyes. Extended exposure to these metals can cause systemic toxicity as well as long-term health issues.

1.1.4 Sulphur dyes

Sulfur-containing dyes are a class of dyes with complex molecular structures. They are produced by heating organic substances with sulphur or sodium polysulfide, such as aromatic amines and aminophenols. Their unique characteristics are typically derived from heterocyclic rings containing sulphur, such as derivatives of thioxanthene or dithioxanthene.

The denim business uses dyes that contain sulphur. They are often easy to apply, affordable, and have good wash-fastness. The most common colours for sulphur dyes are dark blue, brown, and black. Although a pink or softer scarlet colour is obtainable, red sulphur dyes are unknown. The most common black dye used worldwide to colour cellulosic materials is Sulphur Black 1.

Humans as well as animals may be intoxicated by sulfur dyes. Respiratory issues, skin, and eye irritation can result from sulfur dye exposure. Considerable DNA damage has also been observed in the tadpoles that were dyed.

Regular sulphur dyes are difficult to dissipate have poor storage stability, are highly alkaline and smell bad, and are prone to contamination because of their affinity for objects.

1.2 DIFFERENT TYPES OF ADSORBENTS

1.2.1 Pomegranate peels

Pomegranate peels have been investigated as a natural and sustainable adsorbent for a variety of environmental applications due to their widespread availability, low cost, and high adsorption capacity. Due to their high surface area and porous nature, pomegranate peels offer a large number of active adsorption sites. Their shape is porous, which increases their ability to adsorb a variety of contaminants. It has been shown that pomegranate peels may efficiently adsorb a variety of contaminants, such as organic compounds, dyes, heavy metals, and nutrients (Hashem et al., 2023).

Research has demonstrated their effectiveness in air purification, soil remediation, and wastewater treatment. Pomegranate peel-based adsorbents have prospective uses in a range of industries, such as industrial facilities, agricultural settings, and wastewater treatment plants. By using them, green technologies for environmental remediation are developed.

1.2.2 Prawn shells

Prawn shells are converted into several forms, such as chitin and chitosan, and then used as an adsorbent or biosorbent. Seafood is regarded as a staple meal and a key component of the food chain system in many regions of the world(Liu et al., 2021).

Chitosan finds use in the paper, textile, pharmaceutical, cosmetic, healthcare, agricultural, biotech, and food sectors. Chitosan that is sourced from prawn shells has a great potential for adsorbing many types of pollutants because of its porous structure and functional groups, which include hydroxyl and amino groups. These groups facilitate the formation of robust connections between chitosan and pollutants by mechanisms such as chemical complexation, hydrogen bonding, and electrostatic attraction.

Adsorbents based on prawn shells have been used ⁵⁶ in a variety of environmental remediation applications, such as soil stabilisation, wastewater treatment, groundwater remediation, and air purification. These adsorbents are especially helpful in sectors where pollution management is a top concern, like aquaculture, food processing, and pharmaceutical manufacturing (Ramadhani et al., 2020).

1.2.3 Eggshells

Eggshells, which are typically thought of as food waste, have shown promise as an adsorbent for a range of environmental uses. They are mostly composed of calcium carbonate (CaCO_3), containing trace amounts of other chemical substances and proteins. Potential adsorption sites are provided by the pore structure of eggshells, which is facilitated by calcium carbonate(Abatan et al., 2020).

Eggshells are an affordable and easily accessible adsorbent choice, particularly in areas where eggs are extensively consumed. Reusing eggshells as adsorbents can encourage sustainable habits and help reduce trash.

Eggshell-based adsorbents were investigated for wastewater treatment, heavy metal removal, dye adsorption, and nutrient recovery, among other environmental remediation applications. They can be applied to the removal of pollutants from waste water (Badrealam et al., 2018).

1.2.4 Banana peels

The vast surface area and porous structure of banana peels increase their adsorption capability. The abundance of active sites is available for the adsorption of impurities from waste water due to the presence of pores.

Both cellulose and hemicellulose, which are polysaccharides with hydroxyl (-OH) groups, are abundant in banana peels. These functional groups use van der Waals forces and hydrogen bonds to enhance interactions with contaminants (Kok Seng, 2018).

It has been shown that banana peels can absorb pigments from wastewater effluents. Dye adsorption on banana peels is thought to be caused by interactions between functional groups on the peel's surface and dye molecules. Because they are affordable and easily obtainable, banana peels are a financially advantageous adsorbent choice, especially in areas where banana consumption is high. Banana peels can be used as adsorbents to reduce waste and encourage environmentally friendly behaviours (Abdulfatai et al., 2013).

1.3 APPLICATIONS OF SB-NMB

- Removal of dyes.
- Removal of Heavy Metals.
- Removal of Personal Care and Pharmaceutical Products
- Water Purification
- Environmental Restoration
- Resource recovery
- Agricultural effluent treatment
- Biosensing
- Medical and Biomedical applications
- Catalysis

CHAPTER-2 LITERATURE SURVEY

- **Azaritorbat et al. (2023)** have published a scientific article regarding the effectiveness of o-toluidine removal by adsorption and biodegradation techniques compared in this study. The adsorption process involved the use of sugarcane bagasse (SCB) as an adsorbent, while the biodegradation process involved using white-rot fungi, more precisely *Phanerochaete chrysosporium*. The purpose of the study was to investigate how effectively both of these methods could remove o-toluidine while also assessing the adsorbent capability of SCB and the biodegradation efficiency of *Phanerochaete chrysosporium*. Adsorption kinetics, isotherms, and biodegradation assays were among the techniques used in the study to evaluate the extent to which SCB and *Phanerochaete chrysosporium* removed o-toluidine from aqueous solutions. The results of the study could assist in creating economical and environmentally friendly techniques for treating wastewater contaminated with o-toluidine by employing sugarcane bagasse as an adsorbent and white-rot fungi for biodegradation.
- **Munilakshmi et al. (2023)** have published a scientific paper focusing on adsorption investigations involving the use of new adsorbents consisting of prefabricated flocs of aluminium sulphate, ferric chloride, and other materials to decolourize Acid Blue dye. This study looks into the kinetics and equilibrium of the Acid Blue dye adsorption process in aqueous solutions. The study examines the extent to which flocs perform as adsorbents to remove Acid Blue dye, emphasising the potential of chemicals like ferric chloride and aluminium sulphate to decolourize wastewater. The rate and level of dye removal are revealed by the kinetic and equilibrium studies which also highlight the impact of these adsorbents in water treatment procedures. Overall, this study adds to our insight into the kinetics and equilibrium of the Acid Blue dye adsorption process and helps to explain the behaviour of the dye on preformed flocs. The promise of new adsorbents in environmental rehabilitation

efforts is highlighted by their use in the removal of dyes, such as aluminium sulphate and ferric chloride.

- **Elshabrawy et al. (2023)** have published a scientific article focusing on using sugarcane bagasse pulp as a powerful adsorbent material for treating wastewater, specifically to remove dyes from textile waste water. The research emphasises how sugarcane bagasse pulp can be used to mitigate wastewater dye pollution, an important environmental issue because of the toxicity of dyes even in small amounts. This study uses chemically modified sugarcane bagasse to facilitate the biosorption process of dye removal from actual textile effluent. The experimental data and the adsorption process findings indicate that sugarcane bagasse pulp is a useful tool for removing dyes from wastewater. The use of sugarcane bagasse pulp as an adsorbent material provides a low-cost, ecologically acceptable approach for wastewater treatment, particularly for dye removal effluent.

The presence of artificial neural network (ANN) results in the research suggested that the study also investigates the usage of ANN to predict the adsorption process. With this method, the process may be optimised and the adsorption behaviour can be better understood.

- **Vasuki et al. (2023)** have published a scientific article investigating the adsorption process highlighting the effectiveness of activated carbon derived from coconut shells and palmyra fruit nutshell in removing Acid Blue 9 and Brilliant Green.

This study compares the effectiveness of these two forms of activated carbon in adsorbing Acid Blue 9 and Brilliant Green from aqueous solutions with a focus on dye removal and also examines the adsorption mechanism of these dyes, emphasising the efficiency of activated carbon made from the shells of coconuts and palmyra fruit nuts in removing Acid Blue 9 and Brilliant Green. This work attempts to shed light on the possible use of materials obtained from agricultural

waste for applications involving the removal of dyes by comparing the adsorption capabilities of these two forms of activated carbon.

This comparative study advances knowledge on the application of activated carbon made from coconut and palmyra fruit nut shells for environmental remediation, particularly concerning the removal of dyes from waste water sources. The results of this study can provide important insights into long-term and efficient dye-contaminated wastewater treatment strategies.

- **Yaseen et al. (2020)** published a scientific article that reported that modified sugarcane bagasse had a maximum removal efficiency of 99.1% for methylene blue.

The isotherm models Langmuir and Freundlich were used to analyze the adsorption data, and the Langmuir isotherm model was found to fit the data better than the Freundlich isotherm model, indicating that the adsorption process followed a monolayer adsorption mechanism. This study also examines sugarcane bagasse's adsorption capacities in removing methylene blue from aqueous solutions both before and after alkaline treatment. The researchers concluded that modified sugarcane bagasse could be used as a highly effective adsorbent for the removal of methylene blue from textile industry wastewater.

Overall, the study by Yaseen et al. (2020) provides valuable insights into the potential use of modified SB for the removal of methylene blue from textile industry wastewater.

- **Elgarahy et al. (2021)** published a review article discussing the biosorption process in a critical evaluation as a successful and environmentally friendly method to remove metalloids, heavy metals, and dyes from wastewater. The review explores recent advances in biomaterials-based biosorbents, providing insights into their application in environmental remediation processes.

The paper highlights the efficacy of the several biosorbents utilised in the procedure for adsorbing contaminants from wastewater. Potential possibilities for removing hazardous chemicals from aqueous solutions include biosorbents

such as sugarcane bagasse, bacteria, cyanobacteria, yeasts, algae, fungi, and waste items.

The mechanics underlying the biosorption process are also examined. These mechanisms are essential to the adsorption of contaminants by biosorbents and enhance the treatment process' overall efficacy.

Overall, the study offers a thorough review of biosorption as an environmentally friendly and effective way to remove metalloids, heavy metals, and dyes from wastewater. It highlights the possibilities of different biosorbents in tackling environmental pollution issues and emphasises the significance of using biosorption technology as a sustainable solution to water treatment.

- **Siqueira et al. (2020)** published a scientific article that investigated the adsorption of methylene blue dye employing sugarcane bagasse as a biosorbent, highlighting the material's sustainable and economical potential for dye removal applications.

To comprehend the adsorption process, several characteristics were examined and the biomass was characterised. Batch adsorption investigations were carried out together with SEM to characterise the biomass. The efficiency of SCB as a biosorbent for the removal of methylene blue was assessed through the analysis of kinetics, adsorption isotherms, and thermodynamics.

The findings shown that after 24 hours of contact time, SB showed a maximum adsorption capacity of 9.41 mg/g at 45°C. The pseudo-second order model was supported by the adsorption kinetics data, indicating a chemical process was in effect. The heterogeneous adsorption process might be adequately described by the three-parameter isotherm model proposed by Sips.

Overall, this study demonstrates the extent to which sugarcane bagasse works as a biosorbent for removing methylene blue dye from aqueous solutions, providing wastewater treatment operations with an economical and sustainable alternative.

- **Ahmad et al. (2018)** published a scientific about Reactive Red 120 (RR120) which can be removed from aqueous solutions using sugarcane bagasse powder as a biosorbent. The focus of the study is on the batch treatment of textile dye RR120 and the purpose is to determine how different adsorption factors affect ¹the removal of RR120 from aqueous solutions. The study's findings indicated that sugarcane bagasse powder is a very effective biosorbent for removing RR120. The potential of this agricultural waste product as an ⁶⁷effective and economical method for removing dye from aqueous solutions is demonstrated by the notable removal percentages that result from the optimised circumstances.

Overall, studies on the biosorbent use of sugarcane bagasse powder to remove RR120 from aqueous solutions indicate the technology's effectiveness, affordability, and environmental advantages for wastewater treatment applications.

CHAPTER-3 AIM AND SCOPE

3.1 AIM

- To **Synthesize** sugarcane bagasse-derived nanomagnetic biosorbent (SB-NMB) composite for removing Acid Blue 9 from aqueous solutions.
- To **characterize and analyze the** properties and performance of the synthesized SB-NMB, such as FTIR, SEM, EDX, XRD, VSM, BET adsorption capacity, and kinetics surface properties.
- To **obtain conclusive findings** and data regarding the performance of the SB-NMB in removing Acid Blue 9 from aqueous solutions.
- To **assess the potential application** of the SB-NMB as a biosorbent.

3.2 SCOPE

- **A sustainable approach** to wastewater treatment and environmental remediation.
- **we are optimizing** the synthesis process and experimental conditions to **enhance the adsorption efficiency and capacity** of SB-NMB.
- We are conducting adsorption studies to **investigate the effectiveness** of SB-NMB in removing Acid Blue 9 from aqueous solutions.

CHAPTER-4 MATERIALS AND METHODOLOGY

The following materials were used to synthesize the SB-NMB composite:

4.1 MATERIALS

Sugarcane bagasse has been gathered from a roadside juice shop in Sholinganallur and the Acid Blue 9 was acquired from Subha Chemicals in Tambaram.

4.1.1 Chemicals Required

- Hydrochloric Acid (HCl)
- Ferric Sulphate ($\text{Fe}_2(\text{SO}_4)_3$)
- Ferric Chloride (FeCl_3)
- Ammonium Hydroxide (NH_4OH)
- Ethanol ($\text{C}_2\text{H}_5\text{OH}$)
- Distilled Water
- ACID BLUE 9.

4.1.2 Equipment Required

- Muffle Furnace
- Hot Air Oven
- Burette
- Hot Plate Magnetic Stirrer
- Rotary Shaker
- Ultraviolet-visible spectroscopy
- pH meter
- Weighing balance

4.2 METHODOLOGY

4.2.1 Drying of Sugarcane Bagasse

Sugarcane bagasse was collected, and rinsed twice with tap water, then with deionized water, to remove dust and particles that could interfere with the analytical technique. The wet SB was then sun-dried for 10 to 14 days.

4.2.2 Preparation of Sugarcane Bagasse Activated Carbon

The sun-dried sugarcane bagasse was chopped into little pieces (approximately 3 cm) and placed in a small mud pot, sealed with clay and placed in a Muffle furnace for an hour at 600-800°C.

The sugarcane bagasse from the clay was ground into powder, sieved at 85 mesh number and kept in jars for experimental usage.

The Sugarcane Bagasse powder is activated by soaking it in distilled water and hydrochloric acid (HCl) for about 24 hours.

After filtering the solution, the solid powder is thoroughly washed with distilled water.

The filtered product is then dried in a muffle oven for one hour at 150°C.

The dried product is collected and called sugarcane bagasse activated carbon (SB-AC).

4.2.3 Impregnation Process of SB-NMB

The Activated Carbon is then impregnated with 2g of Ferric Chloride (FeCl_3), 1g of Ferric Sulphate ($\text{Fe}_2(\text{SO}_4)_3$) and 1g of the SB-AC.

The conical flask was filled with all the solid components and placed inside a hot plate magnetic stirrer and 50 ml of distilled water was added. The mixture is then allowed to stir constantly for 15 minutes at a temperature of 40°C. (Sugarcane Bagasse – Nanomagnetic Biosorbent)

4.2.4 Co-precipitation Process of SB-NMB

For the co-precipitation process, 50ml of ammonium hydroxide (NH_4OH) is measured and added to the solution drop by drop using a burette retained in the magnetic stirrer.

4.2.5 Filtration and Drying of SB-NMB

After the co-precipitation, give it 15 minutes to cool. filter the sample now and then wash it with distilled water and Ethanol to prevent rusting.

The Filtrate is then dried in Muffle Oven at 150°C for 30 minutes. The powdered particle is the Sugarcane Bagasse impregnated with a Magnetic Nanoparticle (SB-NMB).

4.2.6 Magnetic Separation of SB-NMB

Magnetic Attraction was checked using a Magnet.

4.3 PREPARATION OF ACID BLUE 9 SOLUTION

The Analytical Reagent Grades (AR) served as the standard way to handle all chemicals. To Prepare the stock solution with an Acid blue 9 concentration of 1000 mg/L, 1g of dye has been measured and dissolved in 1000 ml of distilled water. Diluting the stock solution in the proper order, dye levels were adjusted to reach the necessary test concentrations (ranging between 20 to 100 mg/L). Prior to adding the adsorbent to the aqueous solution, the pH of each standard solution was adjusted to the appropriate consistency using 0.1M NaOH and 0.1M HCl. A pH meter was used to measure each solution's pH.

4.4 ADSORPTION EXPERIMENT

- The effectiveness of SB-NMB composite for removing Acid Blue 9 ions from Aqueous solutions is being investigated by studying the effects of different parameters.

- ¹ Batch adsorption studies were conducted to optimize various parameters affecting the adsorption process. Parameters investigated include:
 1. Solution pH (2.0 - 7.0)
 2. Contact Time (0 - 90 Minutes)
 3. Initial Acid Blue 9 ion concentration (20 - 100 mg/L)
 4. Temperature (305 – 335 K)
 5. Adsorbent dosage (SB-NMB) (0.1 – 0.6 g/L)
- Fixed-volume (100 mL) experiments were performed using flasks and the mixture was agitated using a rotary shaker.

- ³⁵ The percentage removal of Acid Blue 9 ions was calculated using the provided equation:

$$\%R = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (1)$$

Where,

⁴ C_0 (mg/L) = Initial Acid Blue 9 ion concentration

C_e (mg/L) = Equilibrium Acid Blue 9 ion concentration

- The experiment employed several models to comprehend the adsorption process, which included:
 1. Adsorption isotherms
 2. Adsorption thermodynamics
 3. Adsorption kinetics
 4. Adsorption mechanism

4.4.1 Adsorption Isotherm Experiments

Adsorption isotherm measurements were performed at an ideal pH of 5 by adding a set quantity of SB-NMB (0.2 g) ³² to 100 mL of Acid Blue 9 solution at different initial concentrations (20-100 mg/L).

In a rotating shaker, the mixture was agitated until equilibrium was achieved and the final Acid Blue 9 concentration in the solution was measured.

The four isotherm models developed to describe the adsorption of Acid Blue 9 onto SB-NMB were Langmuir, Freundlich, BET and Temkin models.

The equilibrium concentration of Acid Blue 9's in the solution (C_e) is related to the quantity of Acid Blue 9 adsorbed per gram of SB-NMB (q_e) in each model equation.

To match the experimental data to the model equations, several constants need to be determined.

1. Langmuir Isotherm Model:

Langmuir Isotherm Model estimates the number of active sites undergoing pressure and adsorption.

The Linear form is-

$$q_e = \frac{bq_{max}}{1+bc_e} \quad (2)$$

The above equation is rearranged for plotting.

$$\frac{1}{q_e} = \frac{1}{bq_{max}} \cdot \frac{1}{C_e} + \frac{1}{q_{max}} \quad (3)$$

2. Freundlich Isotherm Model:

Freundlich Isotherm Model estimates the quantity of gas adsorbed by unit mass of solid adsorbent with pressure.

The Linear form is-

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (4)$$

3. Temkin Isotherm Model:

The adsorption's heat of the molecules decreases linearly with the increase in coverage of the adsorbent surface.

The Temkin Model equation-

$$q_e = B_T \ln K_T + B_T \ln C_e \quad (5)$$

$$B_T = \frac{RT}{b_T} \quad (6)$$

3.4.2 Adsorption Kinetics and Mechanism Experiments

Acid Blue 9 ions were removed successfully by adding 0.2g of SB-NMB to 100 mL of the solution (20–100 mg/L) in a series of conical flasks containing the dye. Samples were taken out of the conical flask from the shaking incubator for a predetermined period (0-90min).

For a given time t and q_t (mg/g), the following formulae were used to calculate the amount of Acid Blue 9 ions adsorbed onto SB-NMB.

$$q_t = \frac{(C_0 - C_t)V}{m} \quad (7)$$

The equilibrium adsorption models that characterise the adsorption properties, such as pseudo first order, pseudo second order, and Elovich Kinetic parameters, were examined using the statistics on kinetic models.

The following are kinetic models:

1. Pseudo-first order Kinetic Model:

$$\ln(q_e - q_t) = \ln q_e - K_1 t \quad (8)$$

2. Pseudo-second order Kinetic Model:

$$\frac{t}{q_e} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} \quad (9)$$

3. Elovich Kinetic Model:

$$q_t = \frac{1}{\alpha} \ln(\alpha\beta) + \frac{1}{\alpha} \ln t \quad (10)$$

3.4.3 Adsorption Thermodynamic Experiments

To conduct the sorption investigation, 0.2g of SB-NMB was infused into 100 ml of Acid Blue 9 solution contained in a series of conical flasks with initial Acid Blue 9 ion levels ranging from 20 to 100 mg/L. An incubation shaker was used to store the adsorption apparatus, which was satisfactorily maintained at temperatures ranging from 305 to 335 K.

The following set of formulas was used to determine values

$$K_c = \frac{q_e}{c_e} \quad (11)$$

$$\Delta G^0 = -RT \ln K_c \quad (12)$$

$$\ln K_c = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (13)$$

$$\Delta G = \Delta H - T\Delta S \quad (14)$$

$$\Delta S = C \times R \quad (15)$$

$$\Delta H = \text{slope} \times R \quad (16)$$

CHAPTER-5 RESULTS AND DISCUSSIONS

5.1 CHARACTERIZATION OF SB-NMB

5.1.1 Fourier Transform Infrared (FTIR) Spectroscopy.

The Fourier-transform infrared (FTIR) spectrum shown in Figure 5.1 shows several unique peaks, each of which corresponds to a particular chemical bond or functional group. At 3378.67 cm^{-1} , a wide band was found, indicating the presence of hydroxyl groups. Stretching C-H bonds were indicated by the peaks at 3179.08 cm^{-1} and 3033.48 cm^{-1} . The presence of C-C bonds was revealed by the occurrence of peaks at 2362.37 cm^{-1} , and at 1623.77 cm^{-1} indicating the presence of C=C bonds. C-F bonds were present, as evidenced by a peak at 1400.07 cm^{-1} . A peak at 1334.5 cm^{-1} represented C-N Amine bonds. The presence of C-O bonds was suggested by a peak at 1105.01 cm^{-1} . At 1042.34 cm^{-1} , the C-N bond of the amine group was found. The spectrum displayed C-H bond aromatic out-of-plane bending at 817.67 cm^{-1} . At 519.722 cm^{-1} , a peak that may have been caused by bromide or iodide C-X bonds was also seen. The synthesis of SB-NMB adsorbent is validated by comparing the FTIR spectrum.

5.1.2. Scanning Electron Microscopy (SEM).

Fig.5.2 (a) and (b) refers to the SEM image. The Scanning Electron Microscope (SEM) is one of the most widely used experimental methods for the research and analysis of micro and nanoparticle imaging characterization of solid materials. SEM is utilized for particle size characterization primarily because of its 10nm resolution. SEM is used to examine the morphology of the powder before consolidation. The methods are simple: a two-sided carbon tape is applied to an SEM sample stub, and then powder is sprinkled on top. SEM analyses were used to examine the morphology of the sample. The SB-NMB sample's porous, uneven, and amorphous surface is visible in SEM images.

48

5.1.3 Energy Dispersive X-Ray (EDX).

Figures 5.3 (a) and (b), Table 5.1 show the EDX pattern with the SB-NMB's Elemental weight percentage. An X-ray method called Energy Dispersive X-ray analysis, often known as EDS or EDX, is utilised to ascertain a substance's elemental composition, SEM with EDX determines the types and amounts of elements present at the nanomaterial surface.

The elemental makeup of the SB-NMB's sample was examined using EDX analysis. According to the EDX pattern, the synthesised SB-NMB sample is mostly made up of Carbon (41.70%), Iron (25.57%), oxygen (23.26%) and chlorine (4.91%).

5.1.4. X-ray diffraction (XRD).

impregnated with iron oxide is shown in Fig.5.4 and the peak list in table 5.2. XRD is a method for figuring out the underlying crystal structure of a material. Constructive interference between monochromatic X-rays and a crystalline substance is the basic tenet of xrd. These X-rays are produced by a cathode ray tube and collimated before being aimed at the substance to concentrate them. The XRD pattern can be used to determine the locations of magnetic diffraction peaks with miller indices by matching two theta values, which are 23.07, 32.88, 35.82, 47.09, and 58.71 degrees and the strongest peak with a 2theta value of 32.88 degrees, a d-spacing of 2.7218 Å, and a relative intensity (I/I₀) of 1000.00.

20

5.1.5 Vibrating Sample Magnetometry (VSM).

A vibrating sample magnetometer is a common tool for analysing the magnetic characteristics of a variety of materials, including diamagnetic, paramagnetic, ferromagnetic, and anti-ferromagnetic ones. The magnetometer detects magnetic moment directly from an applied magnetic field. According to Faraday's Law of Induction, VSM works by generating an electric field from a changing magnetic field.

The hysteresis loop in Fig.5.5 exhibits a Ferromagnetic signal with a rather dominating

paramagnetic signal. Key aspects of the VSM result include: The coercivity is roughly 0.004 T, the saturation magnetization is about 0.04 emu/g, and the remanent magnetization is about 0.02 emu/g.

Overall, the VSM finding is consistent with a ferromagnetic material with a predominately paramagnetic signal.

²⁴ **5.1.6 Brunauer–Emmett–Teller (BET)**

The BET method is used to determine the specific surface area of porous materials, such as adsorbents and nanoparticles. Its technique is based on measurements of gas adsorption, often nitrogen adsorption at low temperatures, and provides data on the porosity and surface area of materials.

¹³ Nitrogen gas adsorption-desorption was used to examine the SB-NMB sample's specific surface area and pore size distribution (Fig. 5.6a). The SB-specific NMB surface area in (Fig. 5.6b) is approximately 57.988 m²/g. The BJH method's desorption branch is used to examine the pore-size distribution. The SB-NMB has a pore ³⁹diameter of 10.530 nm and a pore volume of 10.530 cc/g (Fig. 5.6c).

5.2 OPTIMIZATION PARAMETERS

5.2.1 Effect of pH of SB-NMB.

The adsorption onto the SB-NMB was investigated in the pH range of 2–7. Figure 5.8 shows that Acid Blue 9 ions decrease as pH rises. Lower pH values, such as pH 7, which is neutral, allow H⁺ ions to occupy the majority of the adsorption sites on the SB-NMB surface, while Acid Blue 9 ions adsorbed less due to the surface's electrostatic repulsion with H⁺ ions. According to the graph, the disposal percentage falls as the pH range rises, and the ideal pH for 92.05% removal is 5.0.

5.2.2 Effect of Contact Time of SB-NMB.

A comparison of operation times (0–90 minutes) for the particular removal of Acid Blue 9 using adsorptive SB-NMB is shown in Fig. 5.9. This graph shows that the amount of Acid Blue 9 ions eliminated increases with time, peaking at 45 minutes. The highest percentage of Acid Blue 9 ion removal can be observed whenever the residential time for Acid Blue 9 ion adsorption over onto adsorbent is long. The study found that the percentage of Acid Blue 9 ions removed increased with contact time, with balancing achieved at 45 minutes for SB-NMB for 5 different starting concentrations.

The single, uneven, and continuous adsorption curves that led to saturation suggest that the ion molecules may have covered the adsorbent surface in a monolayer. Given the small difference between the adsorption values at 60 and 90 minutes, a quasi-equilibrium scenario was assumed coupled with a steady-state approximation for the 60-minute contact.

5.2.3 Effect of Concentration of SB-NMB.

An initial ion concentration in the 20–100 mg/L range was investigated, and the effects are shown in Fig. 5.10. The image clearly shows that as the starting ion concentration increased, the proportion of Acid blue 9 ions removed decreased.

The initial ion concentration provides the driving force required to get over the obstacle preventing Acid blue 9 mass transfer between the aqueous phase and solid phase.

At pH 5.0, the impact of the initial Acid blue 9 ion concentration (20–100 mg/L) was examined in the presence of the intended adsorbent. The graphs showed that the percentage of Acid Blue 9 ion removal decreased from 92.05% to 66.4% with an increase in Acid Blue 9 ion concentration from 20–100 mg/L.

The drop in the percentage of Acid Blue 9 ion clearance is the primary cause of the decline in catalytic activity reported on SB-NMB beyond a specific concentration of Acid Blue 9 ions. The substantial drop in sorption efficiency is inferred to be caused by the saturation of active sites that are open to sorption at greater concentrations of Acid Blue 9 ions.

5.2.4 Effect of Temperature of SB-NMB.

The ideal temperature conditions are shown in the above figure 5.11. The temperature ranges from 305K to 335K. The maximum percentage removal occurs at 305.15K, which is the ideal temperature. For the sample, this measurement is made at several doses, which range from 20 to 100 mg/L. The value of % removal steadily drops as the temperature rises.

This is primarily because, as Fig. 5.11 illustrates, the decrease in surface activity suggests that the adsorption process between the adsorbents and Acid Blue 9 ion is exothermic.

5.2.5 Effect of Dosage of SB-NMB.

A important factor in determining the adsorption potential of a given starting concentration of Acid Blue 9 ions is the dosage of SB-NMB.

The plot of the adsorbent dose against the percentage removal (%) is shown in Fig. 5.12. It proves that the percentage of Acid Blue 9 ions removed changes as the adsorbent mass and the adsorbent dose change. The elimination percentage increases from 74.6% to 92.05% and then gradually declines with an increase in adsorbent dosage of between 0.1 and 0.6g/L. As indicated in Fig. 5.12, the ideal dosage is 0.2 g/L.

5.3 ADSORPTION ISOTHERMS

The ¹ adsorption isotherm shows the relationship between the adsorbate on the adsorbent and the adsorbate in the fluid medium in an intermediate state. To evaluate adsorption isotherm data, it is necessary to compare it to several ¹ isotherm models that can be used for the adsorption design phase. This improves the building of an adsorption process for the absorption of adsorbate.

³⁶ The Langmuir, Freundlich, and Temkin models are used in this study to assess the adsorption equilibrium between Acid Blue 9 ions and SB-NMB. Using MATLAB R2023b, the graphs are plotted and these isotherms' validity was investigated. The solutions are displayed in Fig. 5.13. (a) and (b), Table 5.3 shows the coefficient of determination and the relevance of the adsorption isotherm variables.

The equilibrium adsorption statistics per R^2 standard may be adequately described by the Freundlich adsorption isotherm theory, based on the data in Table 5.3. In addition, the Freundlich model outperforms the Langmuir, and Temkin theories in terms of ⁶¹ fit to the experimental data. The Freundlich adsorption isotherm, which would have been appropriate to either a sole exposure adsorbed species each of one limited concentration scope, is in concept a functional application if an adsorb besides a highly incongruous high adsorption layer. The implications of the value of n , that supplies an indication of regarding pseudo-linearity's extent here among the intensity on the remedy with adsorption, to be as follows:

⁶ linearly ($n = 1$),
chemical ($n < 1$),
physical ($n > 1$).

According to Table 5.3, the n amount was calculated to be 2.574. This a n -value corresponds to existing adsorption structure anywhere around 1 and 10, implying believed adsorption was indeed a physical entity one. An SB-NMB was determined to have a highest monolayer adsorption capacity (q_{max}) for metal adsorption of 27.6243 mg/g.

The weak interaction between Acid Blue 9 and SB-NMB suggests that there may be difficulties during the adsorption process. However, an evolving adsorption strategy and thorough statistical measure evaluation have led to the identification of a particular

combination of parameters or algorithms, which is the most successful in forecasting the adsorption of Acid Blue 9 onto SB-NMB. The above demonstrates that perhaps the current approach of adsorption had been an evolving method. Based on R^2 and error values, the accompanying is really the finest combination for the algorithms for isotopes of adsorption examined for extraction Acid Blue 9 particle in SB-NMB.

Freundlich > Langmuir > Temkin isotherms

The results of the experiment show a significant complementarity between empirical data and theoretical expectations, with the experimental findings closely matching the predictions of the Freundlich model. This validation reaffirms that the Freundlich theory can be applied to the ion elimination of Acid Blue 9 by SB-NMB.

5.4 ADSORPTION KINETICS AND MECHANISM

The ratio and sorption mechanism involving Acid Blue 9 ions and SB-NMB were then predicted using the equilibrium adsorption data. Three kinetic models are investigated in this work. The correct solutions are shown in Fig. 5.14. These are the pseudo-first-order, pseudo-second-order, and Elovich kinetic models that assisted in understanding sorption kinetics for removing Acid Blue 9.

Predictions for a range of configurations are shown in Table 5.4, along with pseudo-first-order kinetic constant (k_1) and pseudo-second-order kinetic constant (k_2), as well as equilibrium adsorption capacity (q_e).

The coefficient of determination (R^2) for the pseudo-second-order kinetic model was shown to be greater when compared to the pseudo-first-order model. This implies that the observed adsorption rates are better described by the pseudo-second-order model. The physical adsorption phase %, which is obtained by fitting sorption kinetics, shows that a pseudo-second-order kinetic model describes the removal of Acid Blue 9 onto SB-NMB. Determining the process's rate-limiting stage requires an understanding of the adsorption mechanism.

The equilibrium adsorption capacity (q_e), which is derived using pseudo-second-order kinetic parameters, and the experimental q_e values show a significant agreement, according to the observations. This agreement confirms that the pseudo-second-order kinetic model is applicable in real-world scenarios.

5.5 EVALUATION OF THERMODYNAMIC PARAMETERS

A thermodynamic principle provides structure to the underlying enthalpy change associated with the adsorption process. The process was defined using the thermodynamic parameters change in free energy (ΔG°), change in enthalpy (ΔH°), and change in entropy (ΔS°) rather than SB-NMB.

As shown in Figure 5.15, the values of ΔH° and ΔS° were obtained from the intercept and gradient, respectively, of a linear plot of $\log K_c$ against $1/T$. The equation can be used to calculate thermodynamic parameters using this graphical form.

The values of the estimated thermodynamic parameters are shown in Table 5.5. The table illustrates the spontaneous character of the reactions and the stability of the adsorbate at varying temperatures through negative values of ΔG° . Furthermore, it appears that the adsorption process is advantageous within a specific temperature range based on the growing negativity of ΔG° as temperature drops. Increased temperature may make previously deposited Acid Blue 9 ions more mobile, which could result in desorption from SB-NMB's inner and exterior surfaces.

A negative value of ΔG° denotes spontaneity and offers insights into the adsorption process. Because of the weak interaction between Acid Blue 9 ions and SB-NMB, physical adsorption is confirmed in this instance by the negative ΔH° , which indicates an exothermic sorption process. Decreased unpredictability at the surface is indicated by the negative ΔS° , which is probably caused by the gain of particle motion after adsorption i.e, the system becomes more ordered. Furthermore, enthalpy changes

have a stronger effect on adsorption than entropy changes, as indicated by larger ΔH° , highlighting their importance in the process.

5.6 CHARACTERIZATION AFTER ACID BLUE 9 ADSORPTION

5.6.1 Fourier Transform Infrared Spectroscopy (FTIR).

The Fourier-transform infrared (FTIR) spectrum shown in Figure 5.16 shows several unique peaks, each of which corresponds to a particular chemical bond or functional group. At 3666.98 cm^{-1} , a band was found, indicating the presence of hydroxyl groups. Stretching C-H bonds were indicated by the peaks at 2987.2 cm^{-1} and 2901.38 cm^{-1} . The presence of $\text{C}\equiv\text{N}$ bonds was revealed by the occurrence of peaks at 2359.48 cm^{-1} , and at 1582.31 cm^{-1} indicating the presence of N-H bond. N=O bonds were present, as evidenced by a peak at 1404.89 cm^{-1} . A peak at 1251.58 cm^{-1} represented C-N Amine bonds. The presence of C-X bonds was suggested by a peak at 1055.84 cm^{-1} . At 890.952 cm^{-1} , the C-H bond was found. The spectrum displayed C-H bond aromatic out-of-plane bending at 792.6 cm^{-1} . At 551.542 cm^{-1} , a peak that may have been caused by bromide, iodide or chloride C-X bonds was also seen. The synthesis of SB-NMB adsorbent is validated by comparing the FTIR spectrum.

5.6.2 Scanning Electron Microscopy (SEM).

Following dye uptake, SB-NMB was subjected to scanning electron microscopy to look for any potential alterations in the surface morphology (Fig. 5.17 (a) and (b)). The surface morphology demonstrated that sugarcane bagasse after dye uptake had few pores, but SB-NMB had greater pore diameters before dye uptake. This could be the result of the dye and other produced tiny complex molecules occupying the pores.

5.6.3 Energy Dispersive X-Ray (EDX).

Figures 5.18 (a) and (b), Table 5.6 show the EDX pattern with the SB-NMB weight percentage.

The elemental makeup of the SB-NMB sample was examined using EDX analysis. According to the EDX pattern, the synthesised SB-NMB sample is mostly made up of Carbon (15.17%), Iron (50.77%), and oxygen (32.98%).

CHAPTER-6

6. SUMMARY AND CONCLUSION

Adsorption technology is widely acknowledged as being extremely beneficial in removing dyes, heavy metals, and other harmful effluents from water. This work primarily focuses on the recently developed SB-NMB for the removal of Acid Blue 9 contaminants from water. Analytical methods were used to characterize SB-NMB and due to their exceptional adsorption effectiveness, the use of magnetic nano-based adsorbents offers tremendous promise. Using this method, dye removal can be accomplished more affordably than with more expensive adsorbents now in use. The study also highlights the possibility for other adsorbents including Banana peels, Pomegranate peels, Eggshells and prawn shells, which can be recycled from biowaste items that would otherwise be worthless. According to the research results, the optimum readings are pH 5, contact time 45 minutes, concentration 20 mg/l, temperature 305.15 K and the dosage is 0.2g/l. To evaluate adsorption isotherm data, the Langmuir, Freundlich, and Temkin models are used and the Freundlich model outperforms with an R^2 value of 0.9727 and n as 2.574 g/L. The $q_{\max}$ of Langmuir is 27.6243 mg g^{-1} . pseudo-first-order, pseudo-second-order, and Elovich kinetic models have assisted in understanding sorption kinetics and based on R^2 the adsorption rates are better described by the pseudo-second-order model. Thermodynamic characteristics which include Gibbs free energy, enthalpy, and entropy estimates demonstrate that the adsorption process was spontaneous and Exothermic. The characterization was done after adsorption with FTIR which indicated the broadening of peaks due to the adsorbate molecules interaction, SEM indicated the reduction in pores due to the dye uptake and EDX detected the changes in Elemental Composition. In conclusion, research into using magnetic nanoparticles as adsorbents for dye removal from wastewater shows compelling advantages over conventional adsorbents like activated carbon, including affordability, reusability, and improved selectivity.

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PAGE 12

PAGE 13

PAGE 14

PAGE 15

PAGE 16

PAGE 17

PAGE 18

PAGE 19

PAGE 20

PAGE 21

PAGE 22

PAGE 23

PAGE 24

PAGE 25

PAGE 26

PAGE 27

PAGE 28

PAGE 29

PAGE 30

PAGE 31

PAGE 32

PAGE 33

PAGE 34

PAGE 35

PAGE 36
