

A PROJECT REPORT ON

**BIOSORBENT SYNTHESIS USING LOW-COST SUBSTRATE
AND THEIR APPLICATIONS FOR POLLUTANT BEARING
WASTEWATER TREATMENT**

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BY

Mr. Dinesh Pramod Katyare (71904172E)
Mr. Shreyas Digambar Jate (71904122J)

UNDER THE GUIDANCE OF
Prof. Dr. S.N. Jain



DEPARTMENT OF CHEMICAL ENGINEERING
K.K. WAGH INSTITUTE OF ENGINEERING EDUCATION AND RESEARCH, NASHIK
SAVITRIBAI PHULE PUNE UNIVERSITY

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CERTIFICATE

This is to certify that the project report entitled

BIOSORBENT SYNTHESIS USING LOW-COST SUBSTRATE AND THEIR APPLICATIONS FOR POLLUTANT BEARING WASTEWATER TREATMENT

Submitted by

Mr. Dinesh Pramod Katyare (71904172E)

Mr. Shreyas Digambar Jate (71904122J)

is bonafide work carried out by them under the supervision of Prof. Dr. S.N. Jain and it is approved for the partial fulfillment of the requirement of Savitribai Phule Pune University for the award of the Degree of Bachelor of Engineering (Chemical Engg.)

This project report has not been earlier submitted to any other Institute or University for the award of any Degree or Diploma.

Prof. Dr. S. N. Jain

Internal Guide

Department of Chemical Engg.

Prof. Dr. V. S. Mane

Head

Department of Chemical Engg.

External Examiner

Date:

Place: Nashik

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Mr. Dinesh Pramod Katyare (71904172E)

Mr. Shreyas Digambar Jate (71904122J)

Abstract

Main contributing factor to colour pollution is the discharge of dyes into the water from various industries such as textile, electroplating, paper, food, plastic, and tanneries. The adsorption process using commercial-activated carbons is very effective for the removal of dyes from wastewater, but its high cost has provoked the search for alternatives and low-cost adsorbents. In the present work we have synthesized biosorbent from waste petals of chrysanthemum flower. Chemical treatment using potassium hydroxide (KOH) was applied to the raw powder of chrysanthemum to improve surface characteristics for better adsorption. The synthesized biosorbent was then applied for the removal of Acid Blue 113 dye. Biosorbents were characterized using SEM and BET techniques. Surface area of raw biosorbent was obtained as $7.8 \text{ m}^2/\text{g}$, which was increased to $11.3 \text{ m}^2/\text{g}$ for KOH activated biosorbent, establishing the importance of given KOH activation treatment. The batch experiments were conducted in orbital shaking incubator to study the effect of biosorbent dose from 0.1, 0.2, 0.3, 0.4, and 0.5 gm for contact time (0-240 min) at temperature (20-60°C). The study indicated that maximum removal of 80.13% was found to be maximum at 0.4 gm for activated biosorbent. The further experiments were conducted to study the effect of contact time, and temperature on the removal of Acid Blue 113 dye. The maximum dye adsorption took place at 150 min. The temperature study was performed for high and low temperature. We kept high temperature up to 60 °C and low temperature as 20 °C. The maximum dye adsorption occurred at lower temperature, which showed that studied adsorption is exothermic in nature. Dye removal values were obtained as 80.13%, 77.27%, 73.82%, 68.36% and 58.43% from 1st to 5th cycle respectively. Thus, biosorbent synthesized in the present study can be reused for first 4 cycles, which has established the importance of the present study.

Keywords: Acid Blue 113; Adsorption; Dye removal; Batch study; Kinetics; Reusability; KOH

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Chapter 1

Introduction

Chapter 1

Introduction

The availability of sufficient quantity and good quality of water is a major challenge faced nowadays all over the world and it is of utmost importance to protect this scarce good from the pollution which will be caused by different pollutants. Color is the most visible form in which pollution is noticed, and the main contributing factor to this type of pollution is the discharge of dyes into the water from various industries like textile, electroplating, paper, food, plastic, and tanneries. Dyes are one among the main pollutants found in industrial effluents and causing significant pollution. Dyes are available in natural and artificial forms. The growing population and specific demands of customers have almost replaced natural dyes with synthetic ones. Annually, 1 million tons of dyes are being manufactured worldwide to meet the demand of the industries [1].

The textile dyeing industry consumes large quantities of water and produces large volumes of wastewater from different steps within the dyeing and finishing processes. The total dye consumption of the textile industry worldwide is quite 107 kg/year. However, approximately 1 million kg/ year of dyes are discharged into water streams by the textile industry. A dye is a colored substance on applying gets chemically bonded with the substrate. It is difficult to treat wastewater-containing dyes since dyes are optically and thermally stable thanks to the complex aromatic structure. Dyes offer resistance to chemical, biochemical and photochemical degradation, and hence, it is important to develop improved treatment processes. The dye is usually applied in an solution and should require a mordant to enhance the fastness of the dye on the fiber [2].

Effluent derived from the textile and dyestuff activities can provoke serious environmental impact in the neighbouring receptor water bodies because of the presence of toxic reactive dyes, chlorolignin residues, and dark coloration. Dyes are harmful to human and aquatic life. The presence of dyes in water imparts colour to water and reduces the penetration of sunlight to the interior of the water body and causes a reduction in the photosynthesis of plants affecting their growth and generally the whole aquatic life. The pollution due to the dyes may also cause cancer (Carcinogenic) and mutation in mammals. Exposure to dyes can also result in irritation to the skin, eyes, and respiratory tract in human beings and animals [3].

Overall, the discharge of dye-bearing wastewater into river streams poses severe issues, and recent

studies revealed the mutagenic and genotoxic effects on a living organism, which have been studied for different models. The study conducted on wastewater as obtained from the dye manufacturing industry revealed the presence of 314 chemical compounds, out of which, 32 compounds were reported to have mutagenic activity. Rahman analyzed effluents from the silk dyeing industry to check their genotoxic potential and reported that dye effluents are toxic to eukaryotic cells and have genotoxic properties, which can lead to cancer. Considering all these negative effects, it is imperative to develop effective treatment schemes for the removal of dyes from wastewater before they can pollute the natural sources of water and create significant environmental issues. Color is that the first contaminant to be recognized in wastewater. Even small amount of dye present in water (less than 1 ppm for some dyes) is highly visible and unacceptable. Over 100,000 commercially available dyes exist and more than 7 million tons per year are produced annually. Due to their good solubility, synthetic dyes are common water pollutants and that they may frequently be found in trace quantities in industrial wastewater. An indication of the size of the matter is given by the very fact that two percent of dyes that are produced are discharged directly in aqueous effluent. Due to increasingly stringent restrictions on the organic content of industrial effluents, it is necessary to eliminate dyes from wastewater before it is discharged. However, wastewater containing dyes is very difficult to treat, since the dyes are recalcitrant organic molecules, resistant to aerobic digestion, and are stable to light, heat, and oxidizing agents [4].

Dye molecules comprise two key components: the chromophores, responsible for producing the color, and the auxochromes, which can not only supplement the chromophore but also render the molecule soluble in water and provides enhanced affinity (to attach) toward the fibers. Dyes exhibit structural diversity and are classified in different ways. These are often classified both by their chemical structure and their application to the fiber type. Dyes can also be classified on the idea of their solubility: soluble dyes which include acid, mordant, metal complex, direct, basic, and reactive dyes; and insoluble dyes including azoic, sulfur, vat, and disperse dyes. Besides this, either a serious azo linkage or an anthraquinone unit also characterizes dyes chemically. It is worthwhile noting that azo dyes are the most widely used and accounts for 65–70% of the total dyes produced. The classification of dyes on basis of structure is an appropriate system and has many advantages, like it readily identifies dyes as belonging to a group and having characteristic properties, e.g., azo dyes (strong, good all-round properties, cost-effective) and anthraquinone dyes (weak, expensive),

there are a manageable number of chemical groups (about a dozen). Besides these, both the synthetic dye chemist and therefore the dye technologist use this classification most generally. However, the classification supported application is advantageous before considering chemical structures intimately due to the complexities of the dye nomenclature from this sort of system. It is also a worth point that classification by application is the principal system adopted by the Color Index (C.I.). In the present review, we'll attempt to use the dye names supported their application or their C.I. name/number [3].

Different chemical, biological and physical processes are used for the treatment of wastewater to get rid from toxic pollutants. Some of the physico-chemical water treatment methods like flocculation, precipitation, membrane filtration, coagulation, ultrafiltration, reverse osmosis, nanofiltration, photocatalytic oxidation, electrochemical oxidation, flotation, ion-exchange and adsorption techniques are extensively used for removal of pollutants from wastewater. Some of the treatment techniques, though beneficial but suffer from certain drawbacks, for instance, coagulation technique is economically feasible but results in formation of sludge. Photo-degradation is also one of the most widely used methods for the removal of different dyes and advantageous technique as no sludge is formed but associated with the drawback as the process leads to formation of byproducts. Among wastewater treatment techniques, the adsorption is one among the promising techniques thanks to low cost, simplicity and straightforward processing at commercial scale and has gained significant attention and activated carbon prepared from biomass proved to be highly efficient during this regard. Activated carbons as derived from non-renewable sources like petroleum residues, coal, lignite, bitumen, etc. are widely applied as adsorbents but are quite expensive. Hence possibilities using low- cost alternates as adsorbents are investigated in various studies. The adsorbent material to be applied for adsorption should also be non-toxic, eco-friendly and cheap. Numerous studies supported the technique of adsorption are performed by the researchers using various adsorbents but the search of adsorbent from a sustainable point of view is till the thrust area of research [4].

Recently, numerous approaches are studied for the event of cheaper and effective adsorbents. Many non-conventional low-cost adsorbents, including natural materials, biosorbents, and waste materials from industry and agriculture, are proposed by several workers. These materials might be used as adsorbents for the removal of dyes from solution. Some of the reported adsorbents include pine tree leaves, sugarcane bagasse lignin, cattail root, CaCl_2 modified bentonite, rice

straw, bagasse fly ash, rice husk ash, agricultural waste and timber industry waste carbons, ZnCl_2 activated kiwi peels, KOH activated sunflower Piths [2].

1.1 Classification of dyes and their usage:

There are several ways for classification of dyes. Each class of dye features a very unique chemistry, structure and particular way of bonding. While some dyes can react chemically with the substrates forming strong bonds within the process, others are held by physical forces. Some of the ways of classification are:

1.1.1 Anionic (Acid) Dyes: It is a sodium salt of a sulfonic, carboxylic or phenol organic acid. It is soluble in water and possesses affinity for amphoteric fiber. azo (including premetallized), anthraquinone, triphenylmethane, azine, xanthene, nitro, and nitroso are the main chemical families of these dyes. It is used for nylon, wool, silk, modified acrylics, and also to some extent for paper, leather, ink-jet printing, food, and cosmetics. Most synthetic food colors fall here in this category.

1.1.2 Cationic (Basic) Dyes: These water-soluble dyes yield-colored cations in solution and that's why are called as cationic dyes. The principal chemical classes includes diazahemicyanine, triarylmethane, cyanine, hemicyanine, thiazine, oxazine and acridine. It is used for paper, polyacrylonitrile, modified nylons, modified polyesters and to some extent in medicine too. Originally, these were used for silk, wool, and tannin-mordanted cotton.

1.1.3 Disperse Dyes: These are substantially water-insoluble non-ionic dyes used mainly on polyester and to some extent on nylon, cellulose, cellulose acetate, and acrylic fibers also used for hydrophobic fibers from aqueous dispersion. They generally contain azo, anthraquinone, styryl, nitro, and benzodifuran one groups.

1.1.4 Direct Dyes: They are water-soluble anionic dyes. When dyed from aqueous solution in the presence of electrolytes, it has a strong affinity for cellulosic fibres when dyed in a neutral or slightly alkaline dye bath or at near boiling point with addition of either sodium chloride, sodium sulphate, or sodium carbonate. It is used in the dyeing of cotton and rayon, paper, leather, and, to some extent to nylon. Poly-azo compounds, as well as certain stilbenes, phthalocyanines, and oxazines, are common colours in this category.

1.1.5 Reactive Dyes: They have a chromophore attached to a substituent that is capable of directly reacting with the fiber substrate. These dyes contain chromophoric groups such as azo,

anthraquinone, triarylmethane, phthalocyanine, formazan, oxazine, and others that establish a chemical bond with the fibre. Their chemical structures are simpler, absorption spectra show narrower absorption bands, and the dyeing's are brighter making them advantageous over direct dyes. Generally, used for cotton and other cellulosic, but are also used to a small extent on wool and nylon.

1.1.6 Solvent Dyes: These dyes are solvent soluble (water insoluble) and generally non-polar or little polar, i.e., lacking polar solubilizing groups such as sulfonic acid, carboxylic acid, or quaternary ammonium. azo and anthraquinone are the most common chemical classes, but phthalocyanine and triarylmethane are also employed. It is used for plastics, gasoline, lubricants, oils, and waxes.

1.1.7 Sulfur Dyes: They have intermediate structures and though they form a relatively small group of dyes the low cost and good wash fastness properties make this class important from an economic point of view. It is used for cotton and rayon and has limited use with polyamide fibers, silk, leather, paper, and wood.

1.1.8 Mordant dyes: These dyes use a mordant, which improves the fastness of the dye against water, light and perspiration. The choice of mordant is extremely important as different mordants can change the ultimate color significantly. These are basically used for wool.

1.1.9 Vat Dyes: This type of dye is water insoluble and incapable of dyeing fibers directly. The water-soluble alkali metal salt of the dye is produced by reduction in alkaline liquor, and it has an affinity for the textile fibre in this leuco form. The principal chemical class containing anthraquinone (including polycyclic quinones) and indigoids. These are used for cotton mainly to cellulosic fibers as soluble leuco salts and for rayon and wool too.

Chapter 2

Literature Survey

Chapter2

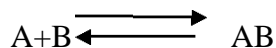
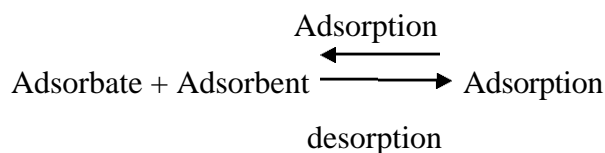
Literature Survey

2.1 Adsorption

Adsorption is a adhesion of atoms, ions and molecules from a gas, liquid or dissolved solid to a surface. This process generates a film of the adsorbate on a surface of the adsorbent. This process is different from absorption, in which a fluid i.e. absorbate is dissolved by a liquid or solid (the absorbent), respectively.

Adsorption is a surface phenomenon, and absorption involves the whole volume of the material. The term sorption surrounds both processes, and desorption is the reverse of it. Adsorption is a surface-based phenomenon. Similar to surface tension, adsorption is a result of surface energy. In the bulk material, all bonding requirements (be they ionic, covalent or metallic) of the constituent atoms of the material are filled by other atoms in the material.

Though, atoms on the surface of adsorbent are not fully surrounded by other adsorbent atoms and hence can attract adsorbates. The exact nature of the bonding depends on the details of species involved, still the adsorption process is generally classified as physisorption (characteristic of weak van der Waals forces) or chemisorption (characteristic of covalent bonding). It may also occur due to the electrostatic attraction. In this process of adsorption, the adsorbate gets adsorbed on the adsorbent.



Adsorption is present in many natural, physical, biological and chemical systems and is widely used in industrial applications such as heterogeneous catalysts, activated charcoal, capturing and using waste heat to provide the cold water for air conditioning and other process Requirements like (adsorption chillers), synthetic resins, increasing storage capacity of carbide-derived carbons and water purification.

As per the Le-Chatelier principle, the direction of equilibrium would shift in the direction where

the stress can be relieved. In case of application of excess of pressure to the equilibrium system, the equilibrium will shift in that direction where the number of molecules decreases. As number of molecules decreases in the forward direction, with the increases in pressure, forward direction of equilibrium will be attained. After saturation pressure P_s , adsorption does not occur further. This can be explained using the fact that there are limited numbers of vacancies on the surface of the adsorbent. At high pressure a stage is attained when all the sites are occupied and further increase in the pressure does not cause any difference in adsorption process. At high pressure, Adsorption does not depend on pressure.

2.1.1 Types of Adsorption

Adsorption can be physical or chemical in nature. The Force of attraction exists between the adsorbate and adsorbent. These forces of attraction occur due to Van der waal forces of attraction which are weak forces and due to chemical bond, which are strong forces of attraction. In accordance with the type of forces of attraction existing between adsorbate and adsorbent, adsorption can be classified into two types: Physical Adsorption or Chemical Adsorption.

- **Physical Adsorption or Physisorption**

Physical adsorption looks like the condensation of gases to liquids and depends on the physical, or van der Waals, force of attraction between the solid adsorbent and the adsorbate molecules. There is no chemical specificity in physical adsorption, any gas which tends to be adsorbed on any solid if temperature is sufficiently low or the pressure of the gas is sufficiently high. Physical Adsorption occur with the formation of multilayer of adsorbate on adsorbent. With the increase in temperature, process of Physisorption decreases.

- **Chemical Adsorption or Chemisorption**

Gases are held to solid surface in chemical adsorption using chemical forces that are specific for every surface and each gas. Chemical adsorption happens usually at higher temperatures than those at which physical adsorption; furthermore, chemical adsorption is ordinarily a slower process than physical adsorption and, like most chemical reactions, frequently involves an energy of activation. Chemisorption occurs with formation of single layer of adsorbate on adsorbent. With the increases in temperature, Chemisorption first increases and then decreases.

2.1.2 Isotherms

Adsorption isotherms are very important for the researchers dealing with environmental protection and adsorption techniques. An adsorption isotherm is a graph which represents the change in the amount of adsorbate(x) adsorbed on the surface of the adsorbent with the change in pressure by keeping temperature constant. The Freundlich and Langmuir isotherms these are two methods used for predicting the adsorption capacity of a given material.

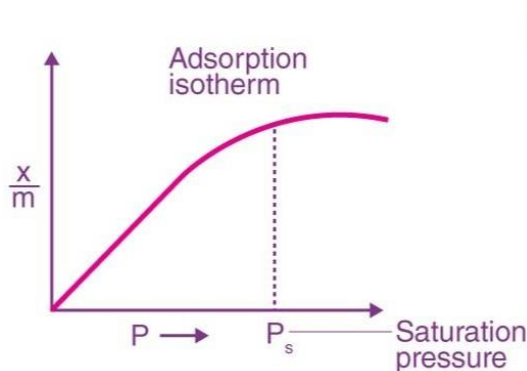


Fig. 2.1 – Adsorption Isotherm

According to Le Chatelier's principle, the direction of equilibrium in a reaction shifts in the direction in which stress is made free. So, on the application of excess pressure on the system, the equilibrium will shift in the direction where the number of molecules will decrease so that the pressure in the system decreases.

From the graph, we observe that after reaching a pressure P_s , which is the saturation pressure, the change in the amount of adsorbent adhering to the adsorbate becomes zero. This occurs because the surface area available for adsorption is limited and as all the sites are occupied, a further increase in pressure does not cause any difference.

The Freundlich and Langmuir isotherms these are two methods used for predicting the adsorption capacity of a given material.

Different adsorption isotherms have been proposed by different scientists namely, Langmuir Isotherm, Freundlich isotherm, and BET theory.

- **Freundlich Adsorption Isotherm:**

Freundlich adsorption gives the changes in the quantity of gas adsorbed by a unit mass of solid adsorbent with the change in pressure of the system for a given temperature. The expression for the Freundlich isotherm can be given using the following equation:

$$\frac{x}{m} = kP^{\frac{1}{n}}$$

where $n > 1$

Where x = the mass of the gas adsorbed, m = mass of the adsorbent, P = pressure and n is a constant depending upon the nature of adsorbent and the gas at a given temperature. Take logarithm on both the sides. We get,

$$\log \frac{x}{m} = \log k + \frac{1}{n} \log P$$

The plot of the given equation is a straight line. It can be shown by the following curve.

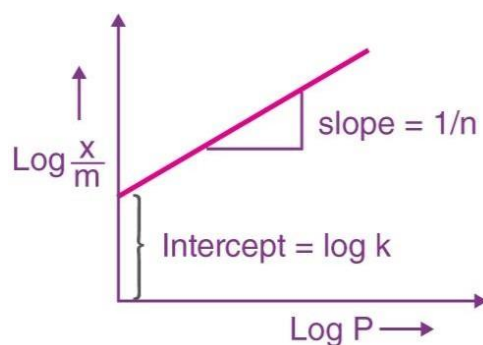


Fig. 2.2 – Freundlich Isotherm

- **Langmuir Adsorption Isotherm:**

The Freundlich adsorption isotherm is followed by two more isotherms i.e., Langmuir adsorption isotherms and BET theory. The Langmuir adsorption isotherms shows linear adsorption at low densities and a maximum surface coverage at higher solute metal concentrations.

The Langmuir adsorption isotherm has the form:

$$\frac{X}{M} = \frac{abc}{1 + ac}$$

where

- X is the weight of a solute sorbed by M grams of solid,
- C is the equilibrium solute concentration,
- a and b are both constants 1/a is equal to the concentration when 1/2 of the available adsorption sites.

The Langmuir adsorption is applicable to a monolayer adsorption on a homogeneous surface when no interaction takes place between adsorbed species.

- **BET Adsorption Isotherm:**

The Brunauer, Emmett and Teller proposed the theory of multilayer adsorption in 1938 (BET Theory) which assumes that physisorption results in the formation of multilayer adsorption. According to this theory solid surface has uniform sites of adsorption and that adsorption at one site does not affect adsorption at neighbouring sites. After the formation of the monolayer, the adsorption process continues with the formation of multilayer which involves the second layer, third layer and so on. The equation for BET is

$$\frac{P}{V_{total}(P - P_0)} = \frac{1}{V_{mono}C} + \frac{C - 1}{V_{mono}C} \left[\frac{P}{P_0} \right]$$

$$V_{total} = \frac{V_{mono}C \left[\frac{P}{P_0} \right]}{\left[P - \frac{P}{P_0} \right] \left[1 + C \left[\frac{P}{P_0} \right] - \frac{P}{P_0} \right]}$$

2.2 Reviews on Some Recent Works on Adsorbent:

Various researchers work regarding synthesis of low-cost adsorbent are discussed below.

Jain et al. reported the application of waste tea residue (WTR) for adsorption of anionic dye (Acid Blue 25) in batch and continuous operation. The insight of functional groups, surface charge, morphology, composition, surface area and particle size of waste tea residue was obtained by the characterization techniques of FTIR, zeta potential, SEM-EDX, BET, and DLS analysis.

They did batch study to check influence of operating pH, adsorbent loading, influent concentration, contact duration of adsorption and temperature on dye remediation. The kinetic data showed the pseudo 2nd order model whereas equilibrium data showed the Redlich Peterson model. It showed maximum dye uptake as 127.14 mg g^{-1} at optimized pH of 1, loading of 3.5 g L^{-1} and higher temperature as 318 K. Adsorption process was found to be spontaneous, physical and favoured with the rise in temperature. Reusability of WTR in multiple cycles showed a slight drop in dye uptake from $27.95 \pm 0.26 \text{ mg g}^{-1}$ at 1st cycle to $26.24 \pm 0.21 \text{ mg g}^{-1}$ at 3rd cycle. Continuous studies were also conducted in packed column and influence of column operating parameters as packing height (36 cm), concentration ($50\text{--}200 \text{ mg L}^{-1}$) and the flow rate of influent ($5\text{--}9 \text{ mL min}^{-1}$) on the efficacy of dye removal were investigated. The obtained results of batch and continuous studies depicted that WTR could be used effectively for remediation of targeted anionic dye from the aqueous phase [1].

Mane and Babu reported the application of Eucalyptus wood (Eucalyptus globulus) saw dust (SD) treated using sodium hydroxide to improve adsorption capacity for the removal of Congo red from aqueous solutions. The treated saw dust using sodium hydroxide was analysed using SEM, FTIR, TGA, and XRD. The Brunauer–Emmett–Teller (BET) surface area, pore-volume, and average pore diameter of SD were $0.3742 \text{ m}^2/\text{g}$, $0.00836 \text{ cm}^3/\text{g}$, and 893.6 \AA . The average particle size of SD was $182.8 \text{ }\mu\text{m}$. The bulk density of SD was 212 kg/m^3 . The porosity of SD is 0.3. Equilibrium and kinetic adsorption studies were carried using saw dust. They studied the effect of various operating parameters like initial pH, contact time, adsorbent dose, initial concentration, and temperature on the removal of Congo red. The Congo red adsorption data were fitted to different isotherm models. They found that Redlich and Peterson model fitted well here. They calculated the thermodynamic parameters such as Gibbs free energy, enthalpy, and entropy change. This adsorbent was found to be both effective and economically viable [2].

Hameed investigated the use of Papaya Seeds (PS) for the adsorption of cationic dye (methylene blue). They conducted batch studies to study the effects of contact time, initial concentration ($50\text{--}360 \text{ mg/L}$), pH ($3\text{--}10$), and adsorbent dose ($0.05\text{--}1.00 \text{ g}$) on the removal of methylene blue (MB) at a temperature of $30 \text{ }^\circ\text{C}$. By using the Langmuir, the Freundlich, and the Temkin isotherms the equilibrium was analyzed. It showed that Langmuir model fits well with a maximum adsorption capacity of 555.557 mg/g . The pseudo-second-order kinetics was the best for the adsorption of methylene blue by PS with a good correlation. The results demonstrated that the PS is very

effective to remove methylene blue from aqueous solutions [3].

Kooli et al. synthesized adsorbent from, local clay mineral from the khulais area with activation at different acid to clay mineral ratios (in mass) at 90 °C. Smectite was a prominent phase in the local clay mineral, which also contained kaolinite and quartz as impurities. The smectite phase of the treated clay minerals underwent structural modifications after acid activation. Powder x-ray diffraction and Fourier transform infrared revealed that the other phases were unaffected. There was a drop in Al_2O_3 , MgO , Fe_2O_3 , and Na_2O concentration, followed by a relative increase in SiO_2 . The acid-activated clays exhibited lower cation exchange capacity and higher specific surface areas. The acidity of local clay minerals was increased up to a point of activation, then gradually decreased. At a 0.5 acid to clay mineral ratio, a maximum specific surface area of 330 m^2/g was attained. The raw clay mineral's elimination capability increased from 50 mg/g to 73 mg/g after acid activation. After six recycle runs, about 85% of the dye removal may still be retained [5].

Jain and Gogate reported the synthesis of a novel biosorbent from *Ficus racemosa* leaves based on the treatment using NaOH were applied for removal of Acid Blue 25 from aqueous solution. SEM, Fourier transform infrared spectroscopy, and Brunauer–Emmett–Teller analyses were used to characterise the synthesised biosorbent. The use of NaOH to remove lignin from biomass and cause the formation of substantial pores was proven. Batch experiments were conducted to determine the effect of important operating parameters on the extent of Acid Blue 25 removal, including pH (range of 2–10), biosorbent dose (range of 1–10 g/L), contact time (range of 0–5 h), initial dye concentration (range of 50–400 mg/L), and temperature (range of 293–323 K). The best settings were pH 2, 4 g/L biosorbent dose, 3 h contact duration, and 323 K temperature, which resulted in maximal dye elimination. Langmuir and Temkin isotherm models were found to best fit the equilibrium data. Desorption-adsorption studies conducted for seven cycles indicated the potential reusability of synthesized biosorbent for the treatment of dye effluents [6].

Kumar and Barakat studied the use of binary oxidized cactus fruit peel (CFP) as an adsorbent for the removal of brilliant green (BG). Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and energy dispersive X-ray spectroscopy (EDX) were used to evaluate the produced adsorbent (EDAX). Batch adsorption experiments were carried out to see how contact time, initial solution pH, initial BG concentration, and temperature affected adsorption. The initial pH of the solution affects the adsorbent surface charge and BG structure, with pH 3 being the best for adsorption. The pseudo-second order model best fit the adsorption

experimental data, according to kinetic analysis. The Langmuir isotherm model was found to fit the equilibrium adsorption data, with a maximum monolayer capacity of 166.66 mg g^{-1} at 20°C . The thermodynamics of BG adsorption revealed that it was an exothermic and spontaneous process. Physical forces were involved in the adsorption of BG onto CFP, as measured by the magnitude of enthalpy change (H^0) of 25.43 kJ/mol [7].

Mokri et al. investigated the adsorption of C.I. Acid Red 97 (AR97) on walnut shell (WS) to find out the desired conditions. In batch mode, the effects of operational parameters such as adsorbent dosage, agitation speed, contact time, pH, and temperature were investigated. At 60 minutes of contact time, agitation rate of 140 rpm, and 12 g/L of adsorbent at 291 K, 73.33 percent of AR97 (50 mg/L) was decolorized. With a strong correlation coefficient of more than 0.99, the rate of adsorption follows pseudo-second-order kinetics. By using the BET technique, the specific surface area of WS was estimated to be $1.28 \text{ m}^2/\text{g}$. The values of thermodynamics parameters such Gibbs free energy (G^0), enthalpy (H^0), and entropy (S^0) revealed that the adsorption process is spontaneous, exothermic, and mostly physical. The Langmuir, Freundlich, and Redlich–Peterson isotherms were used to describe equilibrium data mathematically, and the results showed that they correspond well with the Freundlich model [8].

Aragaw and Angerasa used mechanical, wet, and thermal procedures to create kaolin adsorbents (beneficiated, raw powder, and calcined) from Ethiopian natural kaolin. FTIR, SEM/EDS, XRD, and XRF were used to characterise the geochemical and surface properties of kaolin adsorbent. Basic operating parameters (starting dye concentrations, pH, temperature, contact time, and adsorbent dosage) were investigated in the batch experiment. At an initial dye concentration of 20 mg/L, solution pH of 9, temperature of 30°C , contact time of 60 minutes, and adsorbent dosage of 1g/100 L, percentage removal efficiency of basic yellow 28 (BY28) dye was recorded as 94.79 percent, 92.08 percent, and 87.08 percent onto beneficiated, raw, and calcined kaolin adsorbents, respectively. The molar ratio of $\text{SiO}_2/\text{Al}_2\text{O}_3$ in Ethiopian kaolin was found to be 2.911 percent mass composition, which is greater than the expected pure kaolinite norm, allowing us to categorise the kaolin clay as siliceous. The calculated values of G^0 for beneficiated adsorbent are -1.243, 1.576, and 4.396 kJ/mol at 303.15, 323.15, and 343.15 K, respectively, for 20 mg/L dye concentration and solution pH of 9, implying that the thermodynamic behaviour at the lowest temperature is more feasible and spontaneous than the higher temperature behaviour. For the BY 28 dye adsorption in the tested concentration range, the pseudo-second-order model was fitted.

For raw and calcined adsorbents, the Langmuir isotherm is used, while the Freundlich isotherm is used for the beneficiated adsorbent. At a contact period of 1.0 h, an adsorbent dosage of 1.0 g, an initial dye concentration of 20 mg/L, and a solution pH of 9 at 30 °C, the amount of dye taken up by beneficiated, raw, and calcined kaolin adsorbents was found to be 1.896, 1.842, and 1.742 mg/g, respectively. The findings revealed that these raw and produced local kaolin adsorbents can be used as low-cost dye removal options in industrial wastewater [9].

Ahmad and Ansari reported the use of low cost and eco-friendly chemically treated lawsonia inermis seed powder (CTLISP) as adsorbent for the removal of brilliant green (BG) dye (93%) from aqueous solution. FTIR, EDX, SEM, and TEM methods were used to characterise the adsorbent. It was investigated the effects of several parameters such as concentration, pH, dosages, and contact time. At 50 °C, the maximum adsorption capacity of 34.96 mg g⁻¹ was discovered. The equilibrium was reached in 180 mins, and the experimental data for isotherms and kinetic investigations were clarified. The data was best suited for the Langmuir isotherm with a maximum (R²) value of 0.990 and pseudo-second order kinetics with a maximum (R²) value of 0.998, according to the results. The thermodynamic parameters H⁰, G⁰, and S⁰ were also examined, and the results revealed that the process was endothermic and spontaneous. Using 1 M oxalic acid as an eluting agent, the desorption investigation revealed that 84 percent of the adsorbed BG could be desorbed. The breakthrough and exhausting capacities, respectively, were found to be 12.5 and 45.0 mg g⁻¹. Regeneration experiments were also conducted in order to make the procedure more cost-effective and practicable, and the adsorbent was successfully reused up to the fifth cycle [10].

Lu Et al. investigated Ipomoea aquatica root (“kangkong” root or KR) as a plausible environmentally friendly adsorbent for Auramine O (AO) dye. The pseudo second order and Elovich models best described adsorption kinetics, indicating that chemisorption of two reactive moieties may be involved. The use of experimentally obtained adsorption equilibrium data to investigate the conformity of many adsorption isotherm models, including Langmuir, Freundlich, Temkin, Redlich-Peterson, and Sips, as well as corresponding error analyses, revealed that the Sips model was the best-fit model, resulting in an appealing maximum adsorption capacity of 455.74 mg g⁻¹. Furthermore, when applying base treatment, regeneration and reuse of the spent adsorbent would be possible with 75 percent AO dye adsorbed even at the 5th cycle [11].

Aksu reported the application of dried activated sludge for the adsorption of Reactive Blue 2, and Reactive Yellow 2 dye. The micro-organism was grown in an agitated and aerated enrichment

medium containing the following ingredients (amounts given per dm³): glucose 5 g, yeast extract 1 g, Na₂SO₄ 120 mg, KH₂PO₄ 40 mg, NH₄Cl 42 mg, ZnCl₂ 0.21 mg, H₃BO₃ 0.10 mg CH₃COONa 400 mg, CaCl₂.2H₂O 50 mg, NiCl₂.6H₂O 0.08 mg, FeSO₄.7H₂O 5 mg and MgSO₄.7H₂O 150 mg at pH 6.5. After 4–5 days of growth, the cells were centrifuged at 4000 rpm and then washed twice with double-distilled water and dried at 60°C for 24 h. For biosorption studies, 5 g of dried biomass was suspended in 100 cm³ of distilled water and homogenized in a homogenizer at 8000 rpm and then stored in the refrigerator. 10 cm³ dried activated sludge suspension was contacted with 90 cm³ of solution containing a known concentration of dye. All the final solutions contained 0.5 g dm⁻³ of biosorbent. The test solutions containing RB2 or RY2 were prepared by diluting 1.0 g dm⁻³ of stock solutions of dyes obtained by dissolving the exact quantities of RB2 (CI 61211, 60% purity Aldrich) and RY2 (CI 18972, 50% purity Aldrich) in 1 dm³ of double-distilled water, respectively. The experiments were conducted in Erlenmeyer flasks containing 100 cm³ of dye synthetic solutions. The flasks were agitated at 150 rpm constant shaking rate for 240 min to ensure equilibrium was reached. Samples (5 cm³) were taken before mixing the biosorbent with dye bearing solutions and at predetermined time intervals for the residual dye concentration in the solution. Before analysis the samples were centrifuged at 4000 rpm and the supernatant fraction was analyzed for the remaining dye. All of the experiments were conducted in pairs. The experiments were carried out at a temperature of 25°C to mimic ambient conditions. Colorimetric analysis was used to determine the amounts of unadsorbed RB2 and RY2 in the biosorption medium. Yellow and blue absorbance were measured at 404 and 607 nm, respectively [12].

Akar and et al. studied dye biosorption potential of chemically modified non-conventional biomass obtained from *Pyracantha coccinea*. The modifying agent was hegzadecylethyldimethylammonium bromide (HDEDMABr), a cationic surfactant. Batch mode equilibrium experiments, zeta potential measurements, and FTIR studies were used to investigate the dye biosorption characteristics of modified biomass. Modified biomass has a high biosorption yield for Acid Red 44 (AR44) dye when compared to dried natural biomass. Biosorption equilibrium was reached in around 40 minutes of contact time, according to kinetic data. The pseudo-second-order kinetic model could explain the biosorption process, and it also followed the intraparticle diffusion model up to 40 minutes, but diffusion is not the only rate-controlling phase. A comparison of the several isotherms revealed that the Langmuir isotherm model best characterised dye biosorption utilising modified biomass, with a maximum monolayer capacity of

105.0 mg dye g⁻¹ biosorbent. Biosorption thermodynamic characteristics calculated revealed an exothermic and spontaneous reaction. In the presence of various salt concentrations in the biosorption medium, good biosorption yields ranged from 73.32 to 87.44 percent. [13].

Al Ghouti et al. reported the use of diatomaceous earth for the removal of Methylene blue (MB), Cibacron reactive black C-NN (RB) and reactive golden yellow MI-2RN (RY) dye. The reactive dyes were supplied in liquid form from Ciba specialty chemicals, Basel, Switzerland. Methylene blue is a basic dye, whereas Cibacron black and yellow are reactive dyes that are distinguished by the presence of nitrogen—nitrogen azo linkages. A standard stock solution of these dyes was generated by dilution with deionized water to a final concentration of 1000 mg/dm³ according to the chemical structure of methylene blue. Methylene blue solutions were buffered with 1 mM potassium phosphate solution due to its acid–base nature (pH 9.2). The hydrolysis of reactive black and yellow dyes was carried out according to Laszlo's instructions. Dyes were hydrolyzed by soaking 0.5 g in 100 ml of 100 mM Na₂CO₃ for 1 hour at 50 °C. It was then diluted to 0.5 l with deionized water, yielding a solution containing 1 mM hydrolyzed dye in 20 mM Na₂CO₃. The dye solutions' maximum wavelengths, λ_{max} , were 663, 597, and 430 nm for methylene blue, reactive black, and yellow, respectively. Throughout the experiment, a PerkiElmer spectrophotometer was employed [14].

Nassar and El-Geundi reported the application of natural adsorbents like natural clay, bagasse pith and maize cob for the removal of Astrazon Blue, Maxilon Red, and Telon Blue from aqueous solution. Gabal El-Mohasham, El-sheikh Fadl Village, El-Minia Governorate, Egypt, provided natural clay. It was sieved and ground into various particle sizes. The clay's moisture content was measured to be 7.70.2 percent. Abou-Korkas sugar mill at El-Minia, Egypt, provided bagasse pith. The depithing procedure was carried out at the sugar mill. There was 14.50.5 percent moisture content. It had been sieved into several particle size ranges. Maize cob was harvested in Egypt's governorate of El-Minia. Maize cob was cut to the minimum possible size and was left to equilibrate to a fixed moisture content ($14 \pm 0.3\%$) prior to sieving and the experimental study. Before being used, none of the adsorbents were pretreated in any way. As adsorbates, three dyestuffs were utilised. The dyestuffs that were employed in this research are given below. Commercial salts were made from dyestuffs. A spectrophotometer, model Spectro-Plus MK1A, with a 1 cm light-path cell was used to detect the concentration of colouring matter. When the absorbance reached 0.6, dilutions were performed. For each dyestuff, the wavelength was chosen

to yield the highest absorbance, A. The bottle-point approach was used to determine adsorption isotherms. A fixed quantity of adsorbent was agitated for a sufficient amount of time with dye solutions of varying concentrations to guarantee equilibrium. This method could be used to determine the adsorbents' equilibrium adsorption capacity. All of the trials were carried out at room temperature, which varied between 23 and 27°C on different days of the experiment [15].

Chikri et al. studied the efficiency of sawdust for the application of dye removal. Adsorption is the deposition of adsorbate at the interface between two phases (liquid-solid interface or gas-solid interface), in general. The numerous sawdusts utilised as a precursor for the manufacture of activated carbons are the subject of this review. A review of the literature on sawdust, its components, activation methods, dye removal efficacy, and ambient conditions impacts was conducted. It has also been reported on the applicability of several adsorption kinetic models and adsorption isotherm models for dye removal by sawdust-derived activated carbons. Finally, the utilisation of sawdust as a basic material for various composites and mixtures that can be used as granular activated carbon is highlighted in this research. [16].

Inbaraj and Sulochana reported the application of fruit shell of *Terminalia catappa* for removal of mercury (II) from aqueous solution. The removal of mercury (II) from aqueous solution was accomplished using a carbonaceous sorbent made from the fruit shell of the Indian almond (*Terminalia catappa*) after sulfuric acid treatment. Mercury uptake is influenced by the pH of the aqueous solution, with maximum uptake occurring in the 5–6 pH range. Among the other kinetic models evaluated (pseudo first order and pseudo second order), the sorption kinetics conformed well to the modified second order model. When compared to the Freundlich model, the Langmuir and Redlich–Peterson isotherm models accurately described the equilibrium data, and the monolayer sorption capacity obtained was 94.43 mg/g. Temperature enhanced sorption capacity, and the thermodynamic parameters H^0 , S^0 , and G^0 revealed that Hg (II) sorption was endothermic and spontaneous, with greater unpredictability at the solid–solution interface. The maximal uptake of Hg (II) from 30 mg/l required an optimum carbon dose of 4 g/l, and the mathematical formula generated revealed a 0.94 correlation between experimental and computed percentage removals for every carbon dose investigated. At pH 1.0, almost 60% of the Hg (II) adsorbed from the wasted carbon was recovered, whereas 94 percent was desorbed using a 1.0 percent KI solution [17].

Al-qodah reported the application of Shale Oil Ash (SOA) for the adsorption of reactive dyes. The SOA used in this study was obtained from the Authority of Natural Resources-Jordan. Samples of

this material were burned at 800°C for 1 h. After cooling, the residual ash was ground and sieved into different fractions. Three fractions with diameters of 53-15, 150-250, and 250-355 μm were employed. The ash particles have a bulk density of 1450 kg/m³ and a porosity of 0.42. To dissolve the soluble fraction of the ash, the SOA was mixed with warm water and agitated for 15 minutes. After that, the ash was changed and dried to determine its density and voidage. The adsorption capability of SOA was investigated by contacting single component dye solutions of various concentrations with mixed amounts of the ash at 20, 40, 60, 80, 100, 150, 200, 250, 300, and 340 ppm. These tests were carried out in a shaker bath with a constant temperature. In each experiment, 100 ml from the dye solution was placed in a 500-ml conical flask containing 0.17 g of the ash. After closing the flasks, they were mounted in the orbital shaker for 24 h. The solutions were then altered and analyzed. This procedure was repeated at four different temperatures of 20, 25, 35 and 45°C [18].

Ajami et al. investigated the application of orange peel for the adsorption of Direct Red 23, and Direct Red 80. In jars holding 250 ml of a dye solution (50 mg/l) at various pHs (2–10), various volumes of orange peel (0.5–2 g) for DR23 and (0.2–1 g) for DR80 were mixed. For each of the two dyes, pH experiments were done to find the optimum pH at which maximal colour removal could be obtained with orange peel. To achieve equilibrium conditions, experiments were carried out at doses of 25, 50, 75, 100, and 125 mg/l using 2 g adsorbent for DR23 and 1 g adsorbent for DR80 at pH 2 and 25°C for 24 hours. Equilibrium was established after 15 min. There was no significant difference between stirring rates of 30–200 rpm when different agitation rates were used. After the experiments, the samples were centrifuged with a Hettich EBA20 to determine the dye concentration. The results were fitted to the Langmuir adsorption isotherm with correlation coefficients of 0.9762 and 0.9997 DR23 and DR80, respectively [19].

Wang and Wu reported the application of fly ash for the adsorption of pollutant dye in aqueous solution. They investigated the effect of different concentrations and pH on victoria blue adsorption and found that adsorption increased with dye concentration and that the maximum removal was achieved at pH 8. The uptake of Victoria blue by fly ash is diffusion regulated, and the process follows the first-order adsorption rate expression. The equilibrium data suit the Langmuir adsorption model nicely. Gupta et al. studied the ability of fly ash to remove Omega Chrome Red ME from aqueous solutions and discovered that low adsorbate concentrations, small adsorbent particle size, low temperature, and an acidic pH of the medium all favoured chrome dye

removal. Peat, steel plant slag, bentonite clay, and fly ash, all low-cost adsorbents, were investigated for dye removal by Ramakrishna and Virarag havan. The results showed that fly ash and slag removed a lot of acid dyes, while peat and bentonite removed a lot of basic colours. The removals of acid and basic dyes were equivalent to that of granular activated carbon, while the performance of disperse dyes was far superior to that of granular activated carbon. Coconut shell, groundnut shell, and rice husk were used to test their ability to remove Basic Red 09 colour from wastewater. When operating expenses such as production, maintenance, feedstock, transportation, labour, and distribution costs are factored in, the cost of 1 g of adsorbent needed to remove 4.54, 0.91, and 0.97g is disclosed. The maximum adsorption capacity (46.3 mg/g) and lowest cost-per-unit in grammes of Basic Red 09 dye removal were found in groundnut shell-based biochar (0.91). For the elimination of RhB dye, a phosphoric acid functionalized locust bean pod adsorbent was created, and the starting cost of this adsorbent was established. They revealed that the activated carbon generated by these plant sources was roughly six times less costly than conventional activated carbon. The expense is mostly borne by phosphoric acid and deionized water [20].

Tukaram bai et al. studied the biosorption of Eosin yellow using Sugarcane Bagasse. Batch operations were used to see how different experimental parameters including agitation duration (5–120 minutes), pH (2–9), Biosorbent dosage (1–4 g/L), Biosorbent size (100, 120, 150 mm), Initial concentration (25–150 mg/L), and Temperature (283–323 K) affected the results. The best fitting models for rate kinetics and isotherms were found after studying biosorption kinetics and isotherms. The pseudo first order equation and pseudo second order equation were used to examine the kinetic experimental data. To match the equilibrium data, the Langmuir, Freundlich, Redlich-peter son, Temkin, and Harkin-Jura isotherms were utilised. The equilibrium agitation time for Eosin yellow dye was 90 minutes, and the pH value was 6. Increases in biosorbent dosage from 0.05 to 0.25 g and biosorbent diameters from 100 to 150 mm reduced absorption while increasing percentage elimination. The % biosorption of Eosin yellow was increased with an increase in the temperature. The dye removal data of Eosin yellow follows the Langmuir model with the best fit and dye uptake obtained was 58.47 mg/g. The results indicated that sugarcane bagasse can be used as a good low-cost alternative biosorbent for treatment of effluents containing eosin yellow [21].

Bhattacharjee et al. reported the application of watermelon rind for adsorption of Methylene Blue. Chemical activation using various chemicals has been attempted to manufacture activated carbon from low-cost agricultural waste products. The high surface area (500–1500 m²/g), variety of

surface functional groups, and well-developed interior microporosity are all advantages of activated carbon. Uner et al. employed zinc chloride to chemically activate watermelon rind to make activated carbon (WRAC). The author conducted a thorough investigation of methylene blue biosorption utilising watermelon rind activated carbon and obtained positive findings. They found that increasing the adsorption temperature had a positive effect on MB adsorption and the maximum adsorption capacity (gm) was found to be 231.48 mg/g. The equilibrium and kinetic data were best described by Langmuir isotherm model and pseudo second order model. The researchers looked at numerous models for determining the diffusion mechanism for MB adsorption into WRAC, including the intraparticle diffusion model, the Bangham model, and the Boyd model, and found that intraparticle, film, and pore diffusion all played a role in the adsorption process. Jawad et al. looked into methylene blue (MB) adsorption utilising watermelon rind as a biosorbent. While Jawad et al. employed standard oven dried WMR, they used sulfuric acid as a chemical activator to chemically activate the dried WMR for MB biosorption. The biosorption using chemically activated watermelon rind had a higher adsorption capacity (200 mg/g) than the normal WMR which was 188.68 mg/g, both at 303 K. Both biosorption process exhibited similar results with regards to kinetic and equilibrium data as both sets of data fitted the pseudo second order model and Langmuir isotherm model [22].

Munagapati and Kim studied and investigated the adsorption of Congo Red (CR), an anionic azo dye, from aqueous solution by using Cationic Modified Orange Peel Powder (CMOPP). The influences of pH, contact time, initial dye concentration, and temperature were investigated to find the best circumstances. FTIR analysis was used to characterise the biosorbent. Langmuir and Freundlich isotherm models were used to examine the equilibrium data. At different temperatures, the maximum adsorption capacity of CR on CMOPP was determined to be 107, 144, and 163 mg/g, respectively (298, 308 and 318 K). The Freundlich isotherm did not fit the equilibrium data as well as the Langmuir model. The kinetic results showed that a pseudo-second-order kinetic model well characterised the adsorption of CR onto CMOPP [23].

Monteiro et al. studied the efficiency of two lignocellulosic waste materials, wood residues and coconut mesocarp, as biosorbents towards two representative textile dyes (Remazol Red, RR and Remazol Brilliant Violet, RBV). Both natural matrices had their moisture, carbohydrate, protein, lipid, ash, and fibre contents measured. Infrared spectroscopy, X-ray diffractometry, scanning electron microscopy, specific surface area analysis, and thermogravimetry were also used to

analyse the materials. UV-Visible spectrophotometry was used to track dye desorption. Both coconut mesocarp (CM) and wood residues were shown to be effective biosorbents for the dyes under study. The Langmuir model describes the adsorption of violet dye onto coconut mesocarp, while the Freundlich equation accurately describes all Remazol red. [24].

Crominski da Silva et al. studied residual chia-seed-oil-extraction biomass as a biosorbent for removal of Reactive Yellow B2R textile dye from aqueous solutions in batch system to suggest an appropriate and eco-friendly application, other than incineration or landfill. The biosorption technique performed well at 303 K and for up to 60 minutes, indicating that it has a wide range of applications in textile wastewater treatment. Kinetic, thermodynamic, and equilibrium experiments proved that the biosorption process was effective. At pH 2 and 150 rpm of agitation speed, efficiency was at its peak. The best fit was pseudo-second order, which had an equilibrium period of and nearly 92 percent dye removal from aqueous solution [25].

Morosanu et al. studied and investigated the simultaneous biosorption of a binary metal-dye mixture onto rapeseed waste (RS) resulted from oil press. Because of their low biodegradability and harmful effects even at low concentrations, lead (Pb) and Reactive Blue 19 (Rb19) dye were employed as model pollutants. The biosorbent was minimally processed before usage, and infrared spectroscopy was used to describe it before and after simultaneous biosorption. Kinetic studies were used to assess the biosorption rates of each sorbate at three different Pb: Rb19 molar ratios. The process was better defined by a pseudo-second order rate of reaction, according to the kinetic modelling. Freundlich isotherm model could explain the equilibrium data for the dye uptake, while Langmuir isotherm, followed by Freundlich model, could describe the biosorption of lead. At varied initial concentrations of contaminants in the aqueous phase, biosorption selectivity was measured. The presence of Pb (II) ions increased the absorption of Rb19 in small to medium concentration ranges, compared to its individual biosorption. The rate governing stage of the simultaneous biosorption process, according to Boyd's model, is film diffusion. Desorption was carried out separately for each contaminant and was based on the eluting pH. The practical consequences of simultaneous biosorption with RS waste (reactor configuration, scale-up approach, and exhausted biosorbent management) were also examined [26].

Inbaraj and Sulochana investigated the Jackfruit peel carbon (JPC) for adsorption of rhodamine-B. For batch tests, the JPC was ground and the part retained between 44 and 89 m sieves was employed. SEM micrographs and physico-chemical properties of JPC were also described.

Physico-chemical characteristics and scanning electron micrographs of JPC were also reported. Rhodamine-B dye was procured from Ciba-Geigy. The dye stock solution (1000 mg L⁻¹) was made by dissolving the required amount of dye in distilled water and increasing the volume to the necessary level. Dilutions were used to provide different beginning dye concentrations for batch tests. Batch experiments were carried out by agitating a known quantity of JPC with 100 mL of dye solution of desired concentration at pH 6.0 and at room temperature ($32 \pm 0.5^\circ\text{C}$) in an electronic orbital shaker (250 rpm) equipped with incubation hood. After reaching equilibrium, samples were removed from the shaker, centrifuged, and the supernatant solution was tested for residual dye concentration with a Jasco Double Beam Spectrophotometer (UVIDEC-430B) at a wavelength of 553.8 nm [27].

Ahmad and Alrozi studied the adsorption of malachite green (MG) dye onto rambutan peel-based activated carbon (RPAC). The effects of different parameters such as the initial MG concentration, contact time, solution temperature and initial pH on MG adsorption were studied. The absorption of MG was observed to increase when the initial concentration, contact time, and solution temperature increased. The Freundlich model best reflected the adsorption equilibrium data. The adsorption kinetic model was discovered to be pseudo-second-order. The intraparticle diffusion model was used to determine the mechanism of the adsorption process [28].

Lebron et al. studied and evaluated three algae species, *Fucus vesiculosus* (F. vesiculosus), *Spirulina maxima* (S. maxima) and *Chlorella pyrenoidosa* (C. pyrenoidosa), for their ability to remove methylene blue from aqueous solutions. These samples were chemically modified with orthophosphoric acid (H₃PO₄) and zinc chloride (ZnCl₂) and compared with their respective unmodified samples in terms of equilibrium, kinetic and thermodynamic parameters. All of the samples showed a change in the biosorbent surface after the modification process. The oxygen content of F. vesiculosus and C. pyrenoidosa rose, as measured by an energy x-ray spectrometer (EDS), indicating that their surfaces had been modified by the addition of carboxylic groups. In comparison to normal algae, transformed algae had better heat stability. The samples F. vesiculosus modified with H₃PO₄ (1162.9 mg g⁻¹) and C. pyrenoidosa modified with ZnCl₂ (212.0 mg g⁻¹) achieved the greatest results in terms of maximum biosorption capacity after chemical modification, with increases of 39.9% and 85.9%, respectively. For the green algae S. maxima, a reduction of 40.6% and 22.2% was observed after the activation process with H₃PO₄ and ZnCl₂, however in either case the variation observed was considered non-significant ($p > 0.05$). After 40

minutes, the equilibrium was reached in a modified *F. vesiculosus* sample. After 15 and 30 minutes, respectively, equilibrium was reached for *C. pyrenoidosa* and *S. maxima* treated with ZnCl_2 . The biosorption process presented an exothermic nature and the predominance of physical interactions between the biosorbent and adsorbate. In general, the modified samples presented a superior performance, therefore being considered an efficient alternative to the conventional treatment forms [29].

Sivaraj et al. studied the effectiveness of orange peel in adsorbing Acid violet 17 from aqueous solutions as a function of agitation time, adsorbent dosage, initial dye concentration and pH. The adsorption follows both Langmuir and Freundlich isotherms. The adsorption capacity Q_0 was 19.88 mg/g at initial pH 6.3. The equilibrium time was found to be 80 min for 10, 20, 30 and 40 mg/L, dye concentration respectively. At pH 2.0, an adsorbent dosage of 600 mg/50 ml of 10 mg/L dye concentration resulted in a maximum clearance of 87 percent. Adsorption increases with increase in pH. Maximum desorption of 60% was achieved in water medium at pH 10 [30].

2.3 Environmental effects of Dyes

Dyes cause severe problems in the environment. The problems which are caused in environment by dyes are given below:

- Depending on the exposure time and dye concentration, dyes can have acute and/or chronic effects on exposed organisms.
- They have been reported to cause carcinogenesis, mutagenesis, chromosomal fractures, teratogenicity, and respiratory toxicity.
- The presence of dyes in water gives it colour and reduces the amount of sunlight that reaches the inside of the water body, reducing plant photosynthesis and impacting plant growth and, in general, aquatic life.
- Exposure to dyes can also result in irritation to the skin, eyes, and respiratory tract in human beings and animals.

Dyes are non-biodegradable and stable due to their synthetic origin and complicated structure, making dye-containing wastewater difficult to manage. Even at very low concentrations, dyes decrease water clarity and hence are undesirable.

2.4 Dye removal techniques

Treatment processes for contaminated waste streams include chemical precipitation, membrane filtration, ion exchange, carbon adsorption and co-precipitation/ adsorption. Adsorption has proven to be a good approach for treating industrial waste effluents, with considerable cost, availability, profitability, ease of operation, and efficiency advantages over traditional processes, particularly from an economic and environmental standpoint. Low-cost adsorbents are by-products or waste materials from other processes that require little processing and are abundant in nature. Agricultural by-products are available in large quantities and constitute one of the most abundant renewable resources in the world. The abundance and availability of agricultural byproducts make them good sources of raw materials for activated carbons. Due to their low cost, after being expended, these materials can be disposed of without expensive regeneration. Many biomass material such as bark, coconut shells, and wood are used in the production of commercial activated carbon.

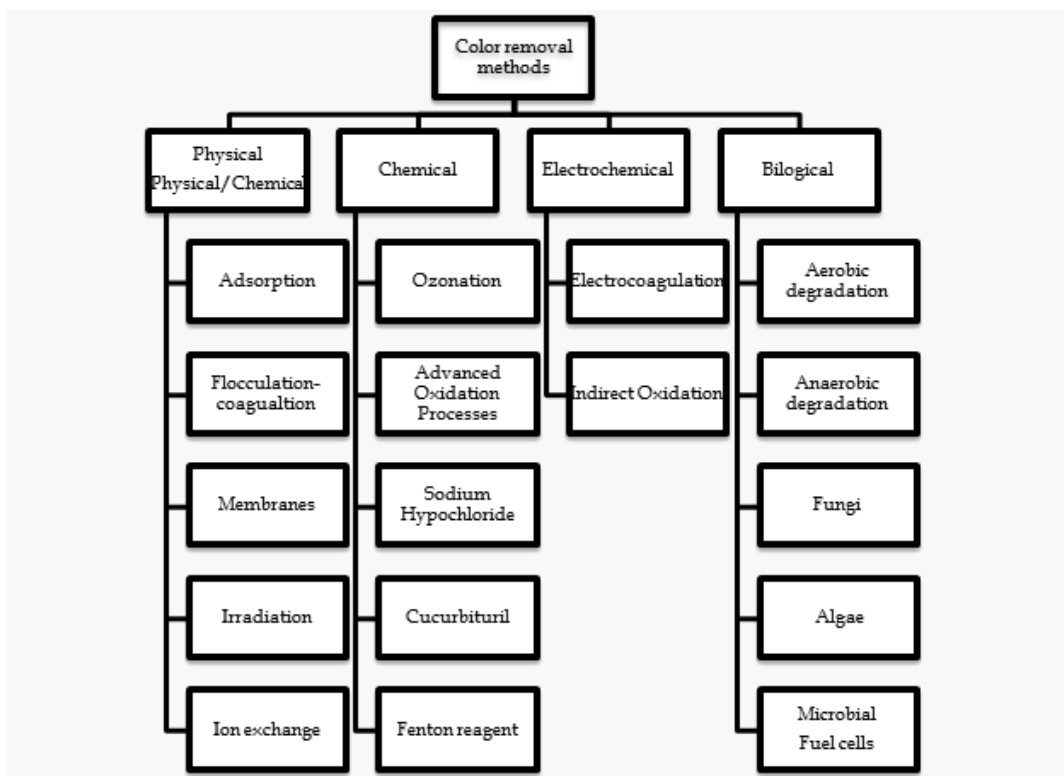


Fig. 2.3 Methods of dye removal from wastewater

2.4.1 Dye removal by biological, physical and chemical methods:

There are different methods to achieve Dye removal, such as: biological, physical, chemical and electrochemical because of the high cost and disposal problems, many of these conventional methods for treating dye wastewater have not been widely applied at large scale in the textile and paper industries. Because of the complicated structure of the effluents, there is currently no single procedure capable of providing effective treatment. Biological treatment is often the most economical alternative when compared with other physical and chemical processes. However, due to technical limitations, their use is frequently limited. Biological treatment necessitates a vast land area and is limited by diurnal variable sensitivity, toxicity of particular chemicals, and design and operation flexibility. Different physical methods are widely used, such as membrane filtration processes such as Nano-filtration, reverse osmosis, electro- dialysis and adsorption techniques.

2.4.2 Physical Methods

Adsorption is one of the most frequently used methods in textile industry as tertiary treatment. Although activated carbon is a good adsorbent for a wide spectrum of colours, its high cost and complexity of regeneration limit its use in decolorization. Some researchers have used low-cost adsorbent materials such as peat, clay, fly ash, granulated ferric hydroxide, zeolites, bentonite clay, agriculture wastes, wood chip, pumice, silica gel, and palygorskite clay to identify an economically viable application for the adsorption process. The coagulation- flocculation technique is a frequently used treatment method in textile wastewater treatment plants to discolor textile effluents and reduce the total load of suspended solids and organic pollutants, however, the additional chemical load on the effluent increases the sludge production and leads to the uncompleted dye removal. Membrane filtration has the ability to clarify, concentrate and separate dyes from the effluent. In textile industry, membranes represent an exciting potential for the recycling of hydrolyzed reactive dyes. Initial investment expense; probable fouling and the formation of various wastes comprising insoluble colours (e.g., indigo); and starch that requires further treatment are some of the considerations for membrane-based treatments.

2.4.3 Chemical Methods

Ozonation is one of the most effective means of decolorization of dye-laden wastewater and has demonstrated to be able to achieve high color and residual COD removal with improved biodegradability. One significant benefit is that ozone can be applied in a gaseous condition, which means it does not add to the volume of wastewater or sludge. The downside of ozonation is that it has a limited half-life (usually 20 minutes), which necessitates continual administration and makes it a costly technique. Advanced Oxidation Processes are one of the traditional methods used for the removal of inorganic/organic compounds from wastewater. The efficiency of advanced oxidation processes is based on the synthesis of oxidising reagent ($\bullet\text{OH}$) radicals, which attack the chromogenic groups, resulting in the production of organic peroxide radicals, which are then converted to CO_2 , H_2O , and inorganic salts. Ozonation, photocatalysis, electrochemical oxidation, Fenton and Fenton-like processes are all examples of advanced oxidation processes. Combining O_3 , H_2O_2 , TiO_2 , UV radiation, electron-beam irradiation, and ultrasound is a typical way to speed up the production of $\bullet\text{OH}$. The oxidation potential of $\text{O}_3/\text{H}_2\text{O}_2$, O_3/UV , and $\text{H}_2\text{O}_2/\text{UV}$ is the largest among them. Fenton's reagent is a chemical that can be used to treat wastewater that is resistant to biological treatment. Advantages of this process include COD, color and toxicity reduction. Sludge formation due to the flocculation of the reagent and dye molecules is a major downside of this approach. In South Africa, several full-scale Fenton's reagent plants have been erected to treat textile mill effluents. This method was discovered to be effective at decolorizing a variety of colours.

2.4.4 Biological Methods

In textile wastewater, the biological process eliminates dissolved organic materials. The removal efficiency is influenced by the ratio of organic load/dye and the microorganisms load, its temperature, as well as the oxygen concentration present in the system. According to the oxygen requirements, biological methods can be classified into aerobic, anaerobic and anoxic or facultative or a combination of these. Aerobic treatment, anaerobic treatment, and combination anaerobic–aerobic treatment are the three types of biological dye removal used in the textile and dyestuff production industries. Biological processes alone may or may not be adequate, depending on local requirements. The process could require the involvement of other physical, chemical, or

physicochemical operations. Most dyes are resistant to aerobic degradation, but can be partially decolorized in anaerobic circumstances, according to popular belief. For coloured wastewater from the textile and dyestuff industries, anaerobic decolorization followed by aerobic post-treatment is commonly advised. This condition can be implemented either by spatial separation of the anaerobic and aerobic sludge using a sequential anaerobic– aerobic reactor system or within one reactor, commonly termed an integrated anaerobic–aerobic reactor system. Fungal methods for the decolorization of azo dyes have been studied in detail. Microorganisms do not readily destroy azo dyes, however *P. chrysosporium* can degrade them. Other fungi have been found to decolorize dye-containing effluents, including *Hirschioporus larincinus*, *Inonotus hispidus*, *Phlebia tremellosa*, and *Coriolus versicolor*. Many chromophore groups of dyes have been decolorized and degraded using white-rot fungus such as *Dichomitus*, *squalens*, *Daedalea avida*, *Irpex avus*, and *Polyporus sanguineus*. Industrial application of fungal methods is not yet feasible for actual textile wastewater effluents because of the slow process kinetics.

2.4.5 Color removal using commercial activated carbon

Solid sorbent adsorption techniques are commonly employed to remove certain types of chemical contaminants from water, particularly those that are almost unaffected by standard biological wastewater treatment. Activated carbon, however, has been shown to be an excellent adsorbent for the removal of a wide variety of organic and inorganic contaminants dissolved in aqueous media or from the gaseous environment, among all the sorbent materials proposed. In actuality, activated carbon is a rudimentary form of graphite with a random or amorphous structure that is very porous, with apparent fractures, fissures, and slits of molecular dimensions. The wide usefulness of activated carbon is a result of its chemical and mechanical stability, high adsorption capacity and high degree of surface reactivity. These unique characteristics are usually related to its internal pore structure, surface characteristics, pore volume, porosity, chemical nature of the source materials, presence of functional group on pore surface and the activation process. The most important structures determining surface features of activated carbon are surface structures of carbon oxygen (functional groups).

However, activated carbon presents several disadvantages. It is quite expensive, the higher the quality, the greater the cost, non-selective and ineffective against disperse and vat dyes. Saturated carbon regeneration is also costly, time-consuming, and results in a loss of adsorbent. For most

pollution control applications, the usage of carbons derived from highly expensive starting materials is likewise unjustified. As a result, many workers are looking for more cost-effective adsorbents.

Chapter 3

Objectives

Chapter 3

Objectives

Based on the conducted literature survey, we have finalized following objectives of the proposed work:

- Replacement of activated carbon using low-cost biosorbents.
- Synthesis of cost-effective biosorbent using flowers waste.
- Effect of biosorbent dose on AB 113 removal.
- Effect of contact time on AB 113 removal.
- Effect of temperature on AB 113 removal
- Reusability study of biosorbent

Chapter 4

Raw Materials, Methodology and Characterization

Chapter 4

Raw Materials, Methodology and Characterization

4.1 Basic Materials (Chrysanthemum)

4.1.1 Plant specification

- **Chrysanthemum**

Chrysanthemum, (genus *Chrysanthemum*), is a genus of about 40 species of flowering plants in the aster family (Asteraceae), it is native primarily to subtropical and temperate areas of the Old World. The Chrysanthemums are specially found in East Asia, they are often depicted in art. Cultivated species, known as mums, are grown as fall-blooming ornamentals and are very important in floral industry. Flower chrysanthemum (*Chrysanthemum morifolium*) has more than 100 cultivars, which includes button, pompon, daisy, and spider forms. Many plants of this genus are perennial herbs or subshrubs. Most of have simple aromatic leaves that alternate along the stem. Some have disk and ray flowers in heads, but others lack ray or disk flowers. The Cultivated species and hybrids usually have large flower heads, those of wild species are much smaller



Fig. 4.1 – Petals of Chrysanthemum

4.2 Adsorbent preparation method

The petals of Chrysanthemum are obtained by the waste generated from temples. The waste petals then allowed for sun drying. The dried petals were crushed into a powder and sieved to get proper mesh size. The dried powder is then used for the further several experimentations.

4.3 Carbonization and Activation Process

4.3.1 KOH Process

- The KOH solution was 1% v/v. (1ml KOH in 100 ml distilled water)
- The dried sample of raw CS powder was soaked well with KOH solution in oven at 120°C for 2 hours.
- The adsorbent and KOH solution ratios were 1:8.
- The dried sample was washed off by hot water to remove the lignin and free the pores using vacuum filtration.
- The washed sample was then allowed to get dry in oven for 5 hours at 60 °C.
- The dried sample was crushed for attaining equal size particles and stored in air-tight container for further experimental study.



Fig. 4.2 – Overall process of biosorbent synthesis



Fig. 4.3 Experimental image of lignin removal from biosorbent



Fig. 4.4 Experimental image of vacuum filtration for biosorbent activation

4.4 Batch Operation:

Batch operation were carried out to observe the effect of various experimental parameters such as:

- Biosorbent dosage
- Contact time
- Temperature
- Regeneration.

4.5 Pollutant Dye

Acid Blue 113

Name: C. I. Acid Blue 113, C.I.26360

Molecular Structure: Double azo class

Molecular Formula: $C_{32}H_{21}N_5Na_2O_6S_2$

Molecular Weight: 681.65

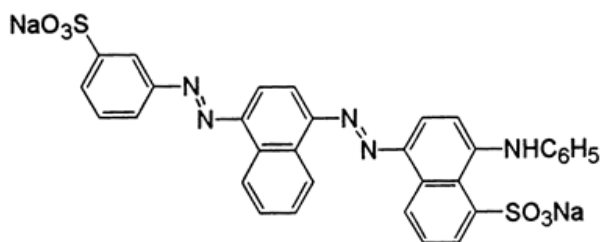


Fig. 4.5 - Chemical Structure of Acid Blue 113



Fig. 4.6 – Physical appearance of Acid Blue 113

Chapter 5

Experimentation

Chapter 5

Experimentation

5.1 Trials:

We had applied the synthesized biosorbent on removal of different dyes from wastewater as Basic Violet 14, Acid Blue 9, Acid Blue 158, Acid Blue 113 and Acid Orange 10. Instead of distilled water wash, we did hot water wash. We didn't get the satisfactory results for these dyes except Acid Blue 113. So, we selected the Acid Blue 113 for our further project experimentation.

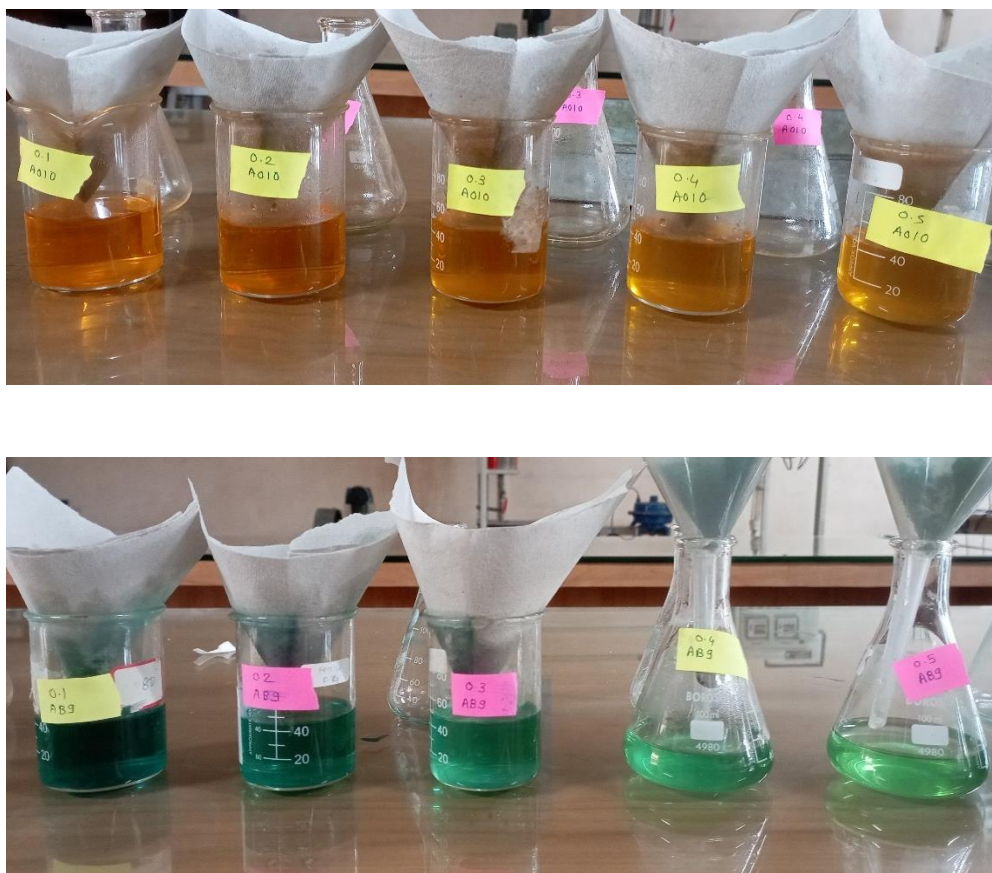


Fig. 5.1 Trials of synthesized biosorbent on different dyes

5.2 Analysis of Acid Blue 113 dye and characterization studies:

Spectrum of Acid Blue 113 dye was established using UV-visible spectrophotometer (UV 1800, Shimadzu Japan). Maximum wavelength of UV absorption, λ_{\max} of dye was found as 566 nm. The quantification of dye in the residual solutions was performed using UV-visible

spectrophotometer by recording the data at of 602 nm. Initially calibration chart was prepared based on the analysis of dye solutions of unknown concentrations.

Biosorbents were characterized using SEM and BET techniques. Surface area of raw adsorbent was obtained as $7.8 \text{ m}^2/\text{g}$, which was increased to $11.3 \text{ m}^2/\text{g}$ for KOH activated biosorbent, establishing the importance of given KOH activation treatment. The obtained SEM images are depicted in Fig. 5.2a and 5.2b for activated biosorbent before and after adsorption respectively. It can be seen from the figures that significant pores are there in Fig. 5.2a, which seems to be blocked after dye adsorption as can be seen from Fig.5.2b.

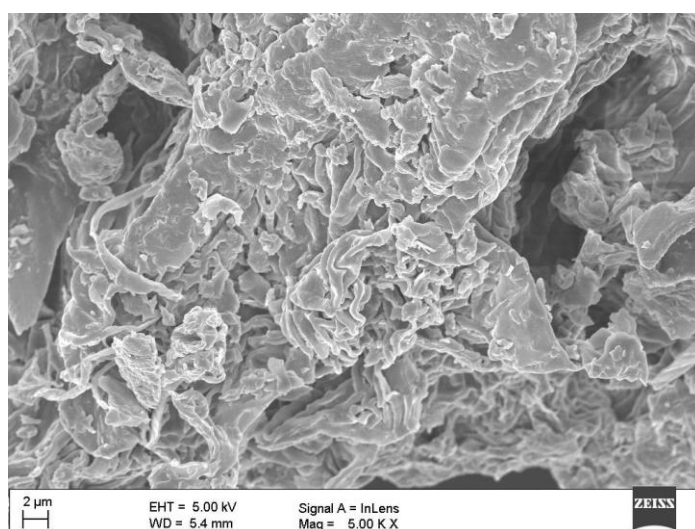


Fig. 5.2a SEM image before dye adsorption

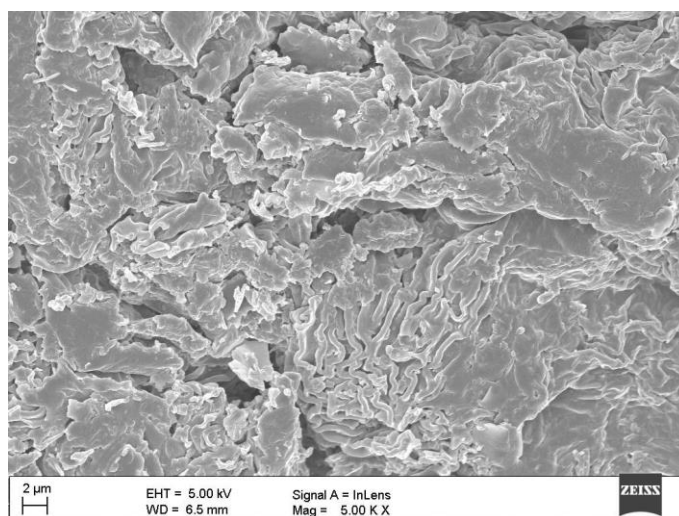


Fig. 5.2b SEM image after dye adsorption

5.3 Effect of Adsorbent Dosage:

To find the effect of adsorbent dosage on dye removal, a fixed adsorbate (dye) concentration of 100 mg/L and a constant volume (50 mL) is taken, keeping all other experimental conditions constant. The experiments were conducted at different values of activated biosorbent from 0.1, 0.2, 0.3, 0.4, and 0.5 gm and also for raw biosorbent. The obtained results are depicted in Fig. 5.3. Dye removal for the activated biosorbent is found to increase significantly from 26.21% at the activated biosorbent dose of 0.1 gm to 80.13% at the dose of 0.4 gm. Dye removal was remained almost unchanged with increase in the further dose hence dose of 0.4 gm for activated biosorbent was finalized for the further experimentation.



Fig. 5.3a Biosorbent dosage experimentation image showing removal of AB113

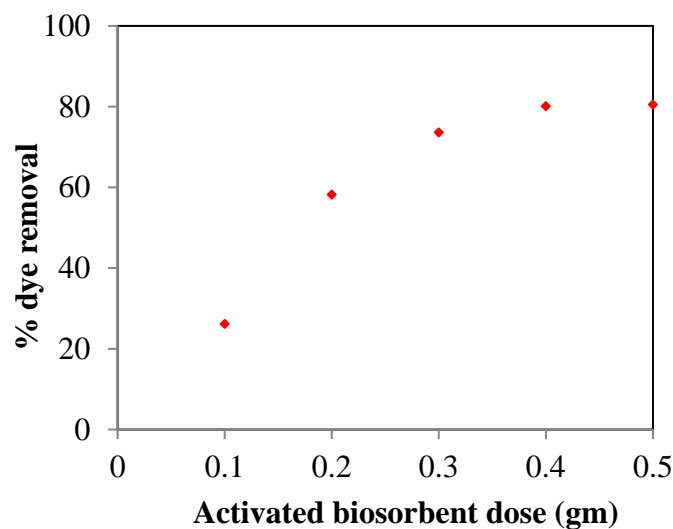


Fig 5.3b Effect of biosorbent dose on dye removal

5.4 Effect of contact time:

A fixed concentration of adsorbate of 100 ppm at constant volume 50 mL, was taken into consideration to find the effect of contact time for adsorption, keeping all other parameters constant. The experiments were carried out at various agitation time from 30 mins to 240 mins. It was investigated that dye removal was found to increase significantly from 10.27% at 30 minute of contact time to 80.13% at 150 minute of contact time. No further significant removal was noted when contact time was further increased from 150 to 240 min. Hence contact time of 150 minute was finalized for the further studies.



Fig. 5.4 Experimentation image of effect of contact time

5.5 Effect of Temperature:

A various concentration of adsorbate from 50 ppm, 100 ppm, 200 ppm, 300 ppm, 400 ppm, 500 ppm at constant volume 50 mL was taken into consideration to find the effect of temperature on adsorption, keeping all other parameters constant. The study was done at two different temperatures, at high and low temperature. We kept high temperature as 60 °C and low temperature to 20 °C. Maximum dye removal was observed at lower temperature of 20 °C, which has confirmed exothermic nature of adsorption.



Fig. 5.5a Temperature experimentation Image (at 60 °C)

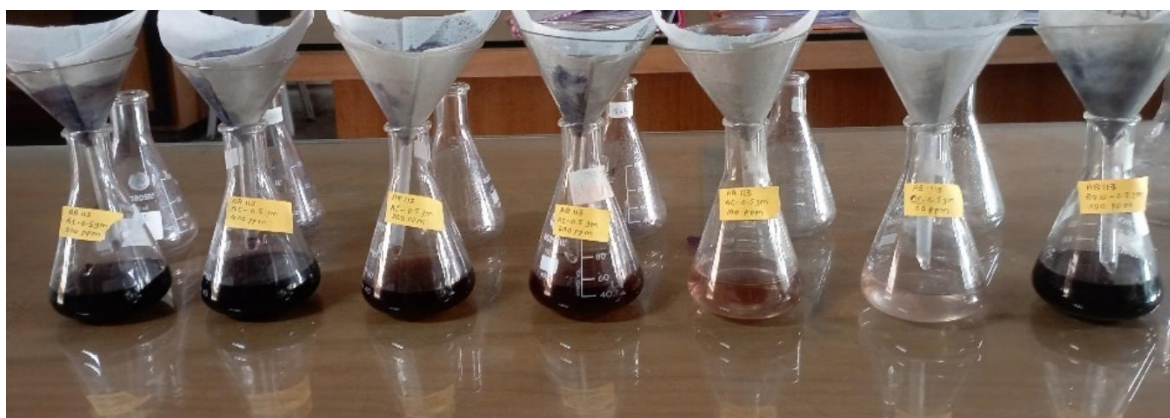


Fig. 5.5b Temperature experimentation Image (at 20 °C)

5.6 Regeneration Study:

A fixed concentration of adsorbate of 100 ppm at constant volume 50 mL was taken into consideration to do regeneration study, keeping all other parameters constant. After first cycle dye adsorbed was desorbed using ethanol and it's been observed that almost all dye was removed which means this is a physical adsorption and we can reuse that adsorbent again. We performed the reusability study till 5 cycles, and it's been investigated that the adsorbent can be reused till 4 cycles. Dye removal values were obtained as 80.13%, 77.27%, 73.82%, 68.36% and 58.43% from 1st to 5th cycle respectively. Thus, biosorbent synthesized in the present study can be reused for first 4 cycles, which has established the importance of the present study.



Fig. 5.6a Desorption study using ethanol as desorbing agent



Fig. 5.6b Reusability study Image till 5 cycles

Chapter 6

Conclusion and Future Plan

Chapter 6

Conclusion and Future Plan

In the present work, we have synthesized biosorbent from waste petals of Chrysanthemum. The raw biosorbent is activated using KOH treatment and then applied for the removal of Acid Blue 113 dye from synthetic wastewater. Biosorbents were characterized using SEM and BET techniques. Surface area of raw biosorbent was obtained as $7.8 \text{ m}^2/\text{g}$, which was increased to $11.3 \text{ m}^2/\text{g}$ for KOH activated biosorbent, establishing the importance of given KOH activation treatment. We have performed batch experiments to study the effect of biosorbent dosage, effect of contact time, effect of temperature and reusability study. Better removal was established for the KOH activated adsorbent in comparison with the raw adsorbent. The maximum dye removal of 80.13% took place at 0.4 gm of activated biosorbent, at 150 min, and at 20°C . The maximum dye removal at less temperature established exothermic nature of present adsorption process. The reusability study showed that the adsorbent can be reused efficiently up to 4 cycles. Dye removal values were obtained as 80.13%, 77.27%, 73.82%, 68.36% and 58.43% from 1st to 5th cycle respectively. Thus, biosorbent synthesized in the present study can be reused efficiently for first 4 cycles, which has established the importance of the present study. The obtained results of batch study can be fitted to different kinetic and isotherm models. The present study can be extended for continuous study in a column operation, which is industrially important.

Chapter 7

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Chapter 7

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Annexure (Plagiarism Report)



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