Synthesis of iron oxide nanoparticles embedded biochar adsorbent for wastewater dye removal

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ABSTRACT

This study presents the synthesis of a novel magnetic biochar composite (MBC) for efficient removal of dye from aqueous solutions. Vegetable peels were washed several times using a distilled water to remove dirt and impurities which is present in the peels. Then the washed peels are dried in an open-air surface for about 1 week. The peels, after drying, were ground into a fine powder using a pulverizer and then sieved to obtain a uniform particles size. Once sieved the biomass was prepared then it undergoes pyrolysis at 550 °C for 20 minutes using a muffle furnace. Subsequently,9.5 grams of ferrous sulphate and 10.5 grams of ferric chloride is mixed with 200 mL of distilled water and stirred at 450 RPM for 1 hour. To facilitate the deposition of iron oxide nanoparticles a freshly prepared NaOH solution was added dropwise to achieve a pH 11. The resulting mixture was stirred for an additional hour, allowed to stand at room temperature for 30 minutes, filtered, washed with distilled water and then dried at 100 °C. Then the prepared biochar adsorbent and iron oxide nanoparticles are mix together for 1 hour for embedding them. Characterization was performed using scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR) and Ultra violet visible spectroscopy (UV-Vis) confirming the successful incorporation of iron nanoparticles onto the biochar surface. Adsorption studies revealed that product effectively removed over 78% of dyes from aqueous solutions. The composite exhibited superparamagnetic properties, enabling easy separation from the solution using a small bar magnet. These results indicate that the adsorbent embedded nanoparticles is a promising, sustainable adsorbent for wastewater treatment applications.

KEYWORDS:

Iron oxide nanoparticles, dye removal, wastewater treatment, water purification, regeneration of adsorbent.

1.INTRODUCTION:

One of the primary processes causing water contamination is wastewater production. Many facets of society use water, a natural resource, for a variety of purposes, including drinking, diluting of waste, manufacturing goods, food production, energy production and use, and so on to meet national needs (Sinitsyna et al., 2021). Wastewater generation cannot be completely avoided, and over the last three decades, its rates have multiplied. Higher water intake results in more wastewater being created, which lowers water quality and causes pollution (Rai et al., 2011). Wastewater is defined as the water used by humans for various reasons, including ground water, surface water, and storm water, as well as liquid or waterborne wastes from residential, commercial, or industrial facilities (Ibrahim et al., 2021). If and only if it is properly maintained, water, a natural resource, may be replenished. However, over time, all of these many activities affect the water's quality, resulting in pollution (Kadir et al., 2021). However, the use of wastewater poses larger dangers to human health and the environment, especially in underdeveloped countries where large volumes of untreated wastewater are used in agriculture and wastewater treatment is rarely practiced (Matseleng et al., 2021). Dyes are colorful chemicals that are frequently used to color things in the printing, food, leather, textile, and cosmetic sectors (Liugė & Paliulis, 2023). They often have properties like strong color strength and substrate-specific affinity and are soluble in water or other solvents. The fact that they pollute water is their biggest disadvantage (Elton & Spencer, 2021). Due to the chemical stability of many dye compounds, dye-waste water is usually very hazardous, non-biodegradable, and difficult to remediate. Prolonged exposure can affect the respiratory and neurological systems as well as key organs including the kidney and liver (Liu, 2020). Some countries have limited or outright banned the use of some artificial food coloring because of their links to childhood hyperactivity, allergic reactions, and potential cancer risks (Bekis Bozkurt & Özdemir, 2024). Biological oxygen demand (BOD) and chemical oxygen demand (COD), which indicate greater concentrations of organic matter to be broken down with oxygen, may be increased by the presence of dyes as pollutants (Muralidharan et al., 2013). Fish and other creatures are put in danger as a result of the water ecosystems decreased oxygen concentration. In wastewater treatment, conventional methods such as membrane filtration, electrochemical treatment, and chlorination have been extensively employed. These methods were discovered to be expensive and ineffective, nevertheless (Zhang et al., 2024). To address the drawbacks of the earlier techniques, more advanced techniques like adsorption, ultrafiltration, chemical precipitation, and coagulation were developed (Elma et al., 2021). Adsorbents are compounds that, by accumulating on their surface, remove materials such as gasses, liquids, or dissolved solids from another phase (usually a gas or liquid). The most often used adsorbents include silica gel, activated carbon, zeolites, clays, biochar, and biosorbents (such as plant peels, microbial biomass, and agricultural leftovers) (Osman et al., 2023). Thus, low-cost and environmentally beneficial alternatives such as bio-based adsorbents, such as vegetable peels and charcoal, are becoming more and more popular (Aina et al., 2025). Biochar is an excellent adsorbent material with a high specific surface area, rich surface functional groups, and a dense pore structure. Pyrolysis at different temperatures and contact periods is the usual method used to create biochar from any feedstock or biomass (Cai et al., 2020). In addition to a variety of natural polymers like cellulose, lignin, hemicellulose, and pectin, these peels—which are typically discarded—contain functional groups like -OH, -COOH, and $-NH_2$ that enable them to bind effectively with contaminants (Md Salim et al., 2021). Adsorbents made from vegetable peels are particularly good in removing organic pollutants, heavy metals, and colors from industrial wastewater. Because biochar-based adsorbents are regenerable and reusable, the color removal procedure is both economical and environmentally friendly over time (Ajala et al., 2023). Biochar is used in soil remediation and agriculture in addition to wastewater treatment. We can develop an environmentally friendly, affordable, and efficient method of water purification that complies with waste valorization and green technology regulations by introducing adsorbents made from vegetable peels into wastewater treatments, especially for decentralized or on-site applications (Muzammal et al., 2023). The source of biomass from which biochar is produced, as well as the preparation process, reaction circumstances, pollutant type, and mode of action, all have an impact on the biochar's potential (Kalina et al., 2022). Nanoparticles are incredibly small particles that range in size from one to one hundred nanometers. They have unique physical, chemical, and biological characteristics that set them apart from bulk particles of the same substance due to their small size and huge surface area (Bandion, 2024). There are several types of nanoparticles, such as metal-based nanoparticles, such as iron oxide, gold, and silver, as well as carbon-based, polymeric, and ceramic nanoparticles (Singh et al., 2024). The size of nanoparticles, which vary from one to one hundred nanometers, is extremely small. Because of their small size and large surface area, they exhibit distinct physical, chemical, and biological properties that distinguish them from bulk particles of the same material (Sune et al., 2024). Iron oxide, gold, and silver are examples of metal-based nanoparticles. Other forms of nanoparticles include carbon-based, polymeric, and ceramic nanoparticles. The kind of pollutant, reaction circumstances, production process, mode of action, and biomass supply all influence the potential of biochar (Dimulescu (Nica) et al., 2021). Nanoparticles are incredibly small, ranging in size from one to one hundred nanometers. They differ from bulk particles of the same material in their physical, chemical, and biological characteristics due to their small size and huge surface area. Metal-based nanoparticles include iron oxide, gold, and silver (Iriarte-Mesa et al., 2020). Carbon-based, polymeric, and ceramic nanoparticles are examples of further nanoparticle types. Because of their unique mix of advantageous qualities, iron oxide nanoparticles (IONPs) are chosen above other nanoparticles for use in wastewater treatment procedures including color removal. Carbon-based, polymeric, and ceramic nanoparticles are examples of further nanoparticle types (Abdulwahid et al., 2022). Iron oxide is safer for large-scale environmental applications since it is less poisonous and far more environmentally benign than other nanoparticles like silver, titanium dioxide, or gold. This results in a composite material that is easy to separate and repurpose in addition to being highly effective at capturing dye molecules (Mbanga et al., 2023). In contrast to precious or rare metals, iron is inexpensive and widely accessible, making large-scale IONP production more feasible (Besenhard et al., 2021). When iron oxide nanoparticles (IONPs) are added to charcoal, a highly efficient and multipurpose composite material is produced that can be used to remove dye from wastewater treatment (Salehirozveh et al., 2024). One of the main drivers behind adding IONPs to biochar is the introduction of magnetic functionality into the matrix. This makes it possible to easily recreate the entire composite using treated water (Ioncica et al., 2023). This facilitates adsorbent regeneration and reuse in addition to improving efficiency during operation. In wastewater treatment operations, immobilizing iron oxide nanoparticles in biochar generally improves the adsorbent's stability, performance, and reusability, making it a highly effective and environmentally beneficial method (Alsawy et al., 2022). The structural matrix of biochar prevents nanoparticle aggregation by incorporating IONPs into the material. The presence of diverse functional groups on both the nanoparticles and biochar contributes to stronger interactions with contaminants, ultimately making the embedded system a highly efficient and versatile adsorbent for dye removal from wastewater (Goswami et al., 2022). Embedding also increases the stability of the nanoparticles, minimizing their leaching into the treated water and thus making the process more environmentally safe (Geng et al., 2019).

2.MATERIALS AND METHODS:

2.1. Collection of raw materials:

The raw material for the adsorbent preparation is the vegetable peels which is collected from the houses and 98% concentrated sulfuric acid is bought from the market Karur, Tamilnadu. Then for the nanoparticle's preparation ferric chloride and ferrous sulphate and the chemical ammonia is bought from the nearby shop in Karur, Tamilnadu.

2.2. Preparation of biochar adsorbent:

Adsorbent was prepared using vegetable peels as a raw material. The peels were washed several times using a distilled water to remove dirt and impurities which is present in the peels. Then the washed peels are dried in an open-air surface for about 1 week. After drying, the peels were grind into a fine powder using a pulverizer and then sieved to obtain a uniform particles size. After sieving, 34.8 grams of biomass is taken and the biomass undergoes pyrolysis, which involves heating in a muffle furnace at a temperature of 600 °C for 25 mins. After pyrolysis process 11.7 grams of biochar is obtained. 11.7 grams of biochar is chemically activated by mixing with 11 ml of sulfuric acid (H_2SO_4) ie,. a ratio of 1:3. The prepared sample was washed with 250 ml of distilled water to neutralize (7) the pH and to reduce acidity. Then it is filtered and dried, 6 grams of biochar adsorbent is obtained.

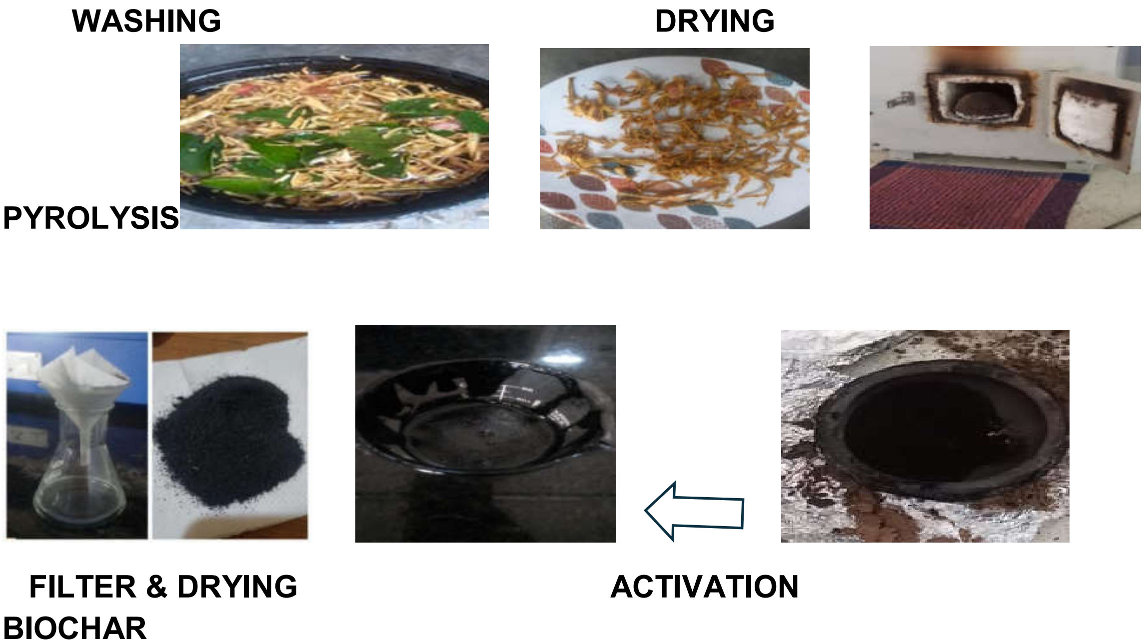


Fig 1: Flowsheet of preparation of biochar adsorbent.

2.3. Preparation of iron oxide nanoparticles:

Iron oxide nanoparticles are commonly synthesized using the co-precipitation method. In this process 9.5 grams of ferrous sulphate (Fe^{2+}) and 10.5 grams of ferric chloride (Fe^{3+}) salts are dissolved in 100 ml of deionized water under a RPM of 450 for about 15 mins. To this solution, 30 drops of ammonia (NaOH) is slowly added. The pH is adjusted to 10, the black precipitate is formed. Then the sample is washed with distilled water 5 times to remove impurities. Finally, the nanoparticles are dried in an oven for 20 mins and 8 grams of iron oxide nanoparticles is obtained.

FERROUS SULPHATE FERRIC CHLORIDE CO PRECIPITATION

NANOPARTICLES FILTER PRECIPITATED MIXTURE

Fig 2: Flowsheet of preparation of iron oxide nanoparticles.

2.4. Preparation of iron oxide nanoparticles embedded biochar adsorbent:

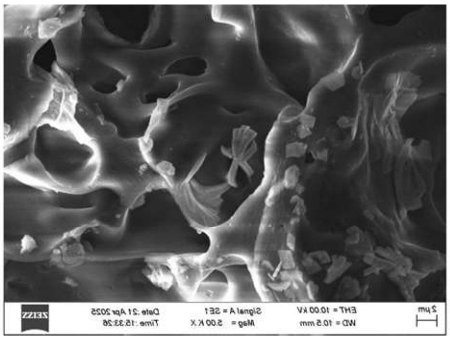
For the embedding process, 2 grams of prepared biochar adsorbent is mixed with deionized water and stir it using magnetic stirrer for 15 mins to ensure uniform mixture. Then slowly add 2 grams of iron oxide nanoparticles into the biochar adsorbent mixture while continuously stirring at a RPM of 420 for about 30 mins. After mixing, the mixture is filtered. Lastly the material is dried and then, 1.5 grams of iron oxide nanoparticles embedded biochar adsorbent is obtained and it is ready to use for the dye and heavy metals removal from wastewater.

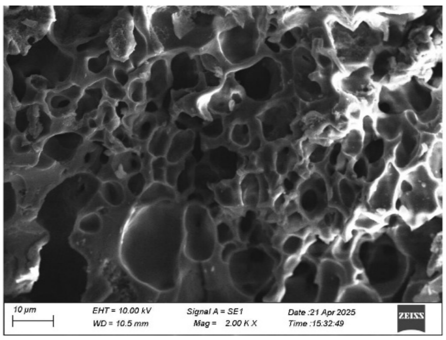
3. RESULTS AND CONCLUSIONS:

3.1. CHARACTERIZATION

3.1.1. SEM Analysis

The six SEM images collectively reveal significant variations in the surface morphology and porous structure of biochar samples for dye adsorption applications. The first image shows a relatively smooth surface with minimal porosity, indicating limited activation or insufficient pyrolysis conditions, which may hinder effective dye adsorption due to low surface area. In contrast, the second image displays a rougher texture with increased pore openings, suggesting a partially activated biochar structure better suited for adsorptive interactions. The third image reveals a highly porous and fragmented surface, possibly resulting from chemical activation (e.g., with KOH or H_3PO_4), which enhances dye uptake by increasing surface area and active sites. The fourth image also shows well-developed macro- and mesopores, which are essential for rapid dye diffusion and adsorption efficiency. Meanwhile, the fifth image appears to exhibit collapsed or blocked pores, potentially caused by incomplete washing or excessive pyrolysis temperature, reducing the functional surface available for adsorption. Finally, the sixth image demonstrates an interconnected porous network with relatively uniform pore distribution, indicating an optimally prepared biochar likely to perform well in dye removal applications. Overall, the morphological differences observed across the images highlight the critical influence of activation methods and preparation conditions on the adsorptive performance of biochar materials.





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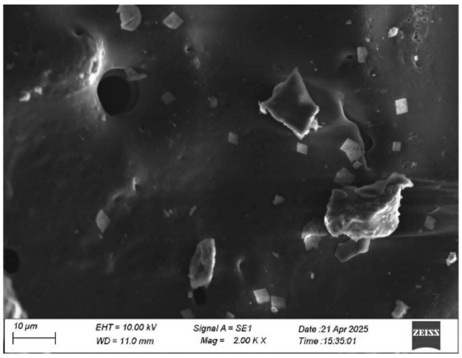

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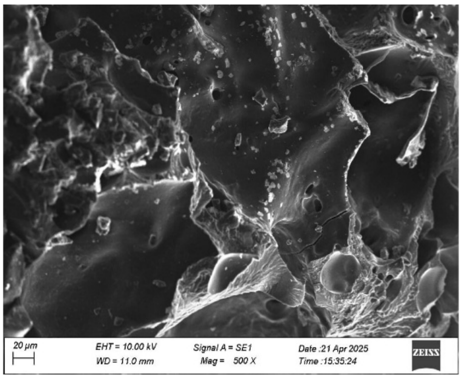

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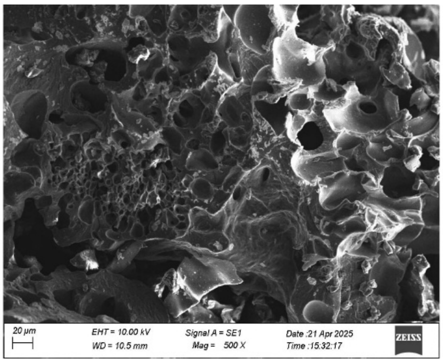

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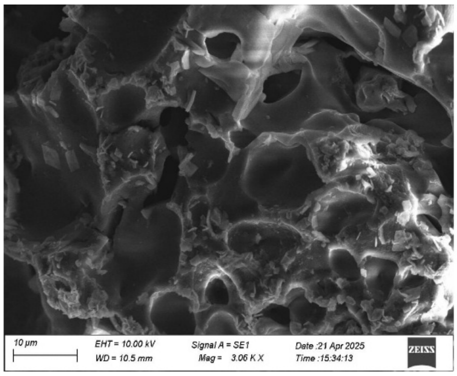

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3.1.2. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

This IR (Infrared) spectrum provides insight into the functional groups present in a compound by showing the absorption of infrared light at specific wavenumbers (cm^{-1}), corresponding to different bond vibrations. A broad peak around 3324.84 cm^{-1} indicates the presence of an O–H group, typical of alcohols or phenols. The peak at 2921.06 cm^{-1} corresponds to C–H stretching, commonly seen in alkanes. A strong, sharp absorption at 1736.99 cm^{-1} suggests a carbonyl (C=O) group, which is characteristic of esters, aldehydes, or carboxylic acids. The signal at 1636.95 cm^{-1} may represent C=C stretching, indicating the possible presence of alkenes or aromatic rings. Additional peaks at 1462.99, 1237.38, and 1054.14 cm^{-1} point to C–H bending and C–O stretching, the latter being typical in alcohols, ethers, or esters. The lower frequency absorptions at 881.01 and 741.94 cm^{-1} suggest out-of-plane C–H bending, often found in aromatic compounds. Overall, the spectrum implies the compound contains hydroxyl, alkyl, carbonyl, and possibly aromatic or alkene groups.

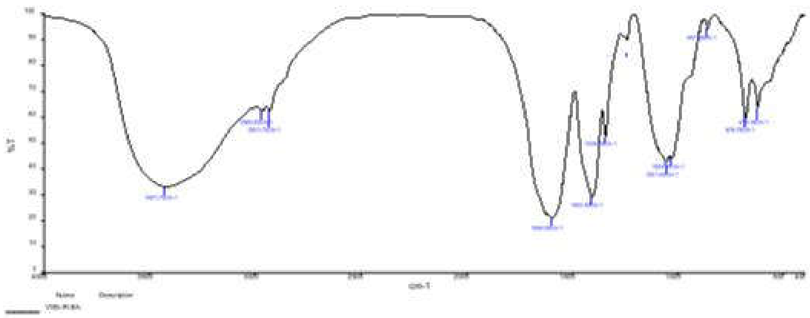


Fig-3.1.2-FTIR

3.1.3. UV-Visible Spectroscopy

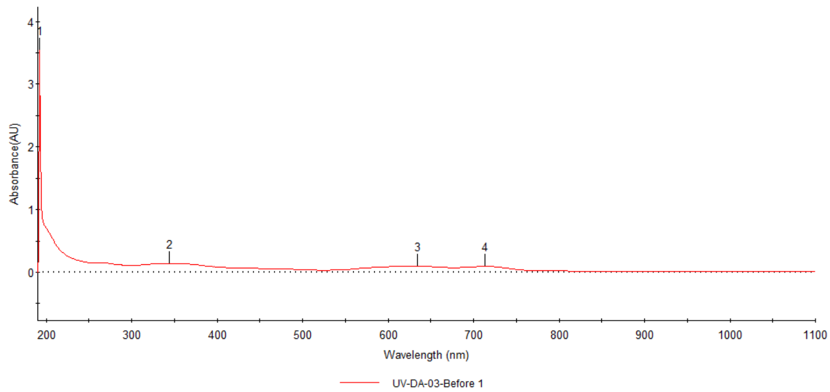


Fig-1 Before adsorption

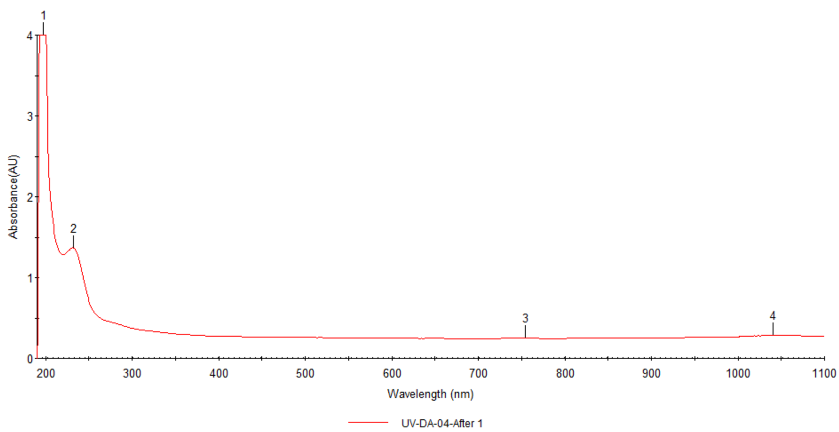


Fig-2 After adsorption

The two UV-Vis absorbance spectra shown represent the dye solution before and after treatment, indicating the effectiveness of dye removal. In the first image the absorbance is significantly higher, especially at lower wavelengths (around 200–300 nm), suggesting a high concentration of dye in the solution. Peaks are clearly observed and marked at specific wavelengths, corresponding to the characteristic absorbance of the dye components. In the second image, the absorbance intensity has noticeably decreased across the entire spectrum, particularly at the same peak wavelengths, indicating a substantial reduction in dye concentration. This reduction in peak intensity after treatment is a clear sign of successful dye degradation or removal, likely due to a chemical, physical, or biological treatment process. The disappearance or decrease in the sharpness of the peaks further supports the effectiveness of the treatment method in breaking down the dye molecules or removing them from the solution. The efficiency is 75%.

3.2. ADSORPTION STUDIES

3.2.1. EFFECT OF TEMPERATURE

Temperature plays a crucial role in determining the adsorption capacity of materials with distinct effects observed in physical and chemical adsorption. The effect of temperature on adsorption capacity is a critical factor in adsorption processes. Generally, adsorption is an exothermic process, meaning it releases heat. As a result, an increase in temperature typically leads to a decrease in adsorption capacity. the thermal behavior of a specific adsorption system is essential for optimizing its efficiency and performance in industrial applications such as gas separation, wastewater treatment, and air purification.

3.2.1.1. BIOCHAR ADSORBENT

3.2.1.1. Table

PARAMETER	SAMPLE 1	SAMPLE 2	SAMPLE 3
Mass of sample(peels)	34.8	10	10
Temperature(°C)	600 °C.	550°C.	650°C.
Time(min)	25 mins	20 mins	25 mins
Concentration(g)	11.7	7.07	4.07

3.2.1.2. IRON OXIDE NANOPARTICLES

3.2.1.2. Table

PARAMETER	SAMPLE 1	SAMPLE 2	SAMPLE 3
Ferrous sulphate(g)	9.5	11.5	2.5
Ferric chloride(g)	10.5	12.5	3.5
Temperature(°C)	50	55	65
Time(min)	25 mins	20 mins	25 mins
Concentration(g)	7.9	8.2	1.2

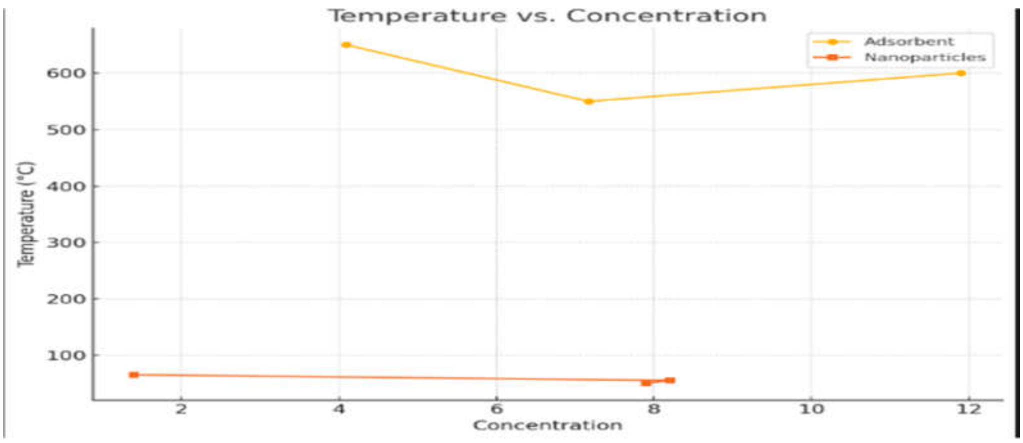


Fig-3.2.1 (EFFECT OF TEMPERATURE)

3.2.2. EFFECT OF pH

The effect of pH plays a crucial role of a material and the chemical state of the compound involved. The pH of a solution plays a crucial role in determining the efficiency and outcome of many chemical and biochemical processes. In contrast as the **pH increases**, these effects may diminish, creating a more favorable environment for the process, thereby enhancing product formation or adsorption.

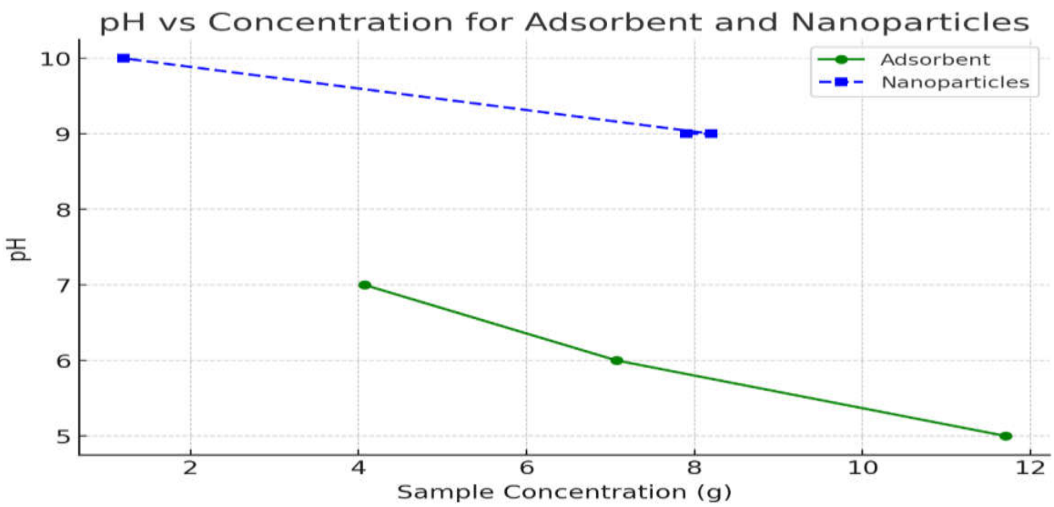


Fig-3.2.2 (EFFECT OF pH)

3.2.3. EFFECT OF CONTACT TIME

Contact time plays a crucial role in determining the efficiency of chemical reaction or an adsorption process. Initially, the chemical rate of reaction or adsorption is rapid due to the abundance of available active sites or reactive molecules. Eventually, equilibrium is reached where no significant increase in product formation or adsorption is observed, even with extended contact time. Therefore, optimizing contact time is essential to ensure maximum efficiency without unnecessary delays or energy consumption. This optimal time varies depending on factors such as type of dye, Characteristics of the adsorbent, solution, pH, temperature and initial dye concentration. Eventually, the system reaches equilibrium, where no significant increase.

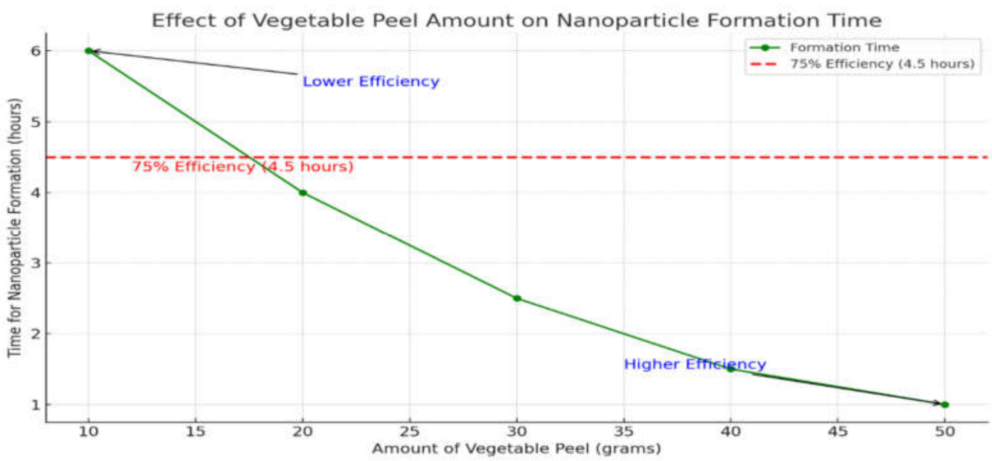
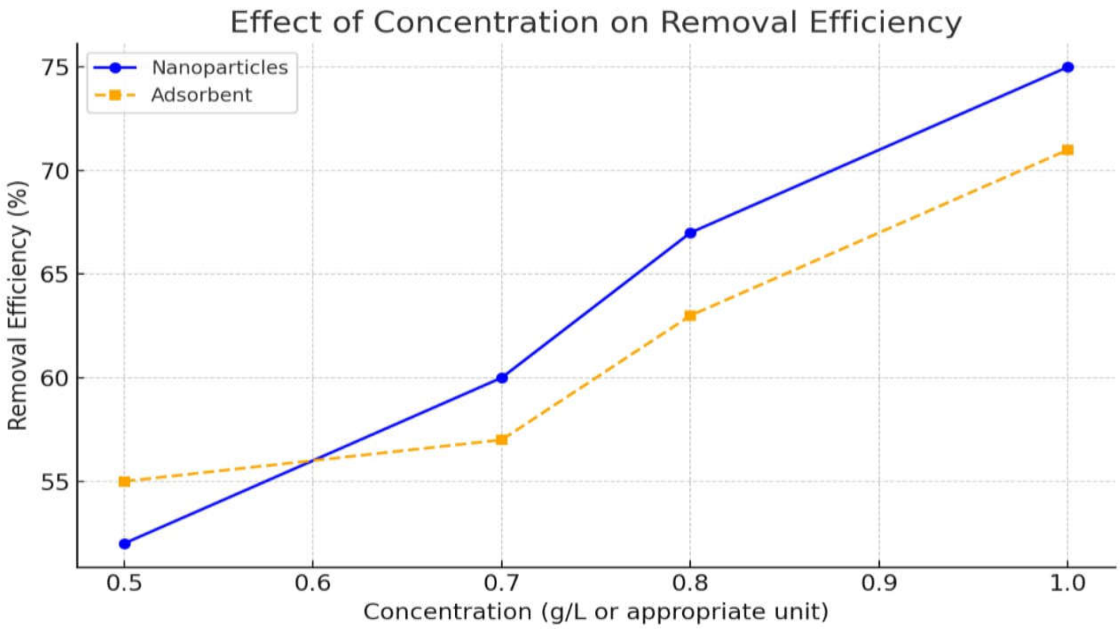


Fig- 3.2.3 (EFFECT OF CONTACT TIME)

A line graph showing the rise in adsorption efficiency of nanoparticles over time, reaching **75% efficiency at 4.5 hour**. This suggests rapid performance improvement within the first hour of use.By using 10 grams it takes 6 hours to form nanoparticles, while taking 50 grams it takes 1 hour to form nanoparticles.It shows that at low reaction time the efficiency is higher than the highest reaction time.

EFFICIENCY Vs CONCENTRATION

EFFICIENCY: 75%



(EFFICIENCY Vs CONCENTRATION)

The graph showing that the efficiency of nanoparticles is higher than adsorbent. The efficiency of nanoparticles is 75 % whereas for adsorbent it is less than 75 %.

4. CONCLUSION:

Biochar adsorbent from vegetable peels by iron oxide nanoparticles have been synthesized in this study by employing the exclusive physicochemical properties of adsorbents. The modified adsorbent embedded nanoparticle is very promising for separating dyes from wastewater. The present study shows the removal efficiency & adsorption of dyes in aqueous solution using biochar adsorbent with iron oxide nanoparticles (Fe_2O_3) prepared using wet impregnation method. Visual interpretation of the dyes is controlled by both external surface film diffusion phase & a pore filling adsorption phase. The highest efficiency reached by the nanoparticles is around 75% by 34.8 grams of biochar adsorbent which is prepared at around 600 °C temperature & 1 hour reaction time. The results also indicate that dye (from wastewater) adsorbed to the surfaces of the adsorbents embedded in nanoparticles suggesting that these forms of nanoparticles have great potential to be a good & reusable adsorbent for wastewater treatment.

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