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Destruction of Effluent Water Using Activated Carbon Synthesized by Morinda Citrifolia Leaves from Temple Solid Waste

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Abstract: ***This study explores the potential of utilizing Morinda citrifolia leaves, readily available in temple solid waste, for the synthesis of activated carbon (AC). Temple solid waste often contains significant amounts of these leaves, creating a disposal challenge. This waste stream can be converted into a valuable resource by transforming the leaves into AC, a highly porous material with diverse applications in adsorption, catalysis, and filtration. The research will focus on developing a sustainable and eco-friendly method for AC synthesis from Morinda citrifolia leaves. The study will investigate activation techniques, including chemical methods, to optimize the surface area, porosity, and adsorption capacity of the resulting AC. The influence of different activation parameters, such as temperature, time, and activating agent concentration, will be systematically evaluated. Characterization techniques such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscope (SEM),Energy-dispersive X-ray spectroscopy (EDX), Hydro analysis and UV VIS spectrophotometer will be employed to assess the structural and textural properties of the synthesized AC. Furthermore, the adsorption performance of the AC will be evaluated for various pollutants relevant to wastewater treatment.***

Keywords: Morinda citrifolia, Activated carbon, Hydro analysis, Methyl orange, Photocatalytic, UV spectrophotometer

1. Introduction

The chemical industry is one of the oldest industries in India. Tirupur is India's foremost textile hub. Based on the news report, the historically beautiful Noyyal River has been converted into a polluted sewer by the town's colorants and the bleaching appliances the fact that extend color and flare to its clothing, and the huge tracts of land for farming that that the aquatic body formerly supported have also been devastated. As a result, valuable farmland terrain of approximately two kilometers on both ends of the river has been turned unusable, causing countless agricultural communities to lose their means of subsistence [1]. The bleaching facilities as well as textile businesses throughout the neighbouring districts of Tiruppur along with Erode are currently attempting to take advantage of this opportunity by depositing waste water onto the Noyyal whenever rainwater reaches the river. The Noyyal water is not utilized for drinking purposes in Karur, despite being true that water exhibiting a Salinity of at least 500 is deemed suitable for overall consumption by individuals.[2]. Depending on the geographical situation, Due to the sewage, air pollution, water pollution, and chemical contamination around the Farming lands [3]. The contaminants in wastewater treatment encompass detergents, oils, grease, ammonium, sulfates, acidic solutions of hydrochloric acid, sulphuric acid, fluoride, and other compounds[4]. Colored dye wastewater, resulting from dye production and textile industry use, discharges over 7x10 tonnes annually from manufacturing operations and 10% from textiles and related industries [5].

A carbon type commonly referred to as activated charcoal, is a dark black powder or granule produced from diverse plant sources that is utilized in numerous applications, including commonly referred to as charcoal, including the carbon family, the air, water, and natural element removal [6]. Activated carbon (AC) is a highly effective adsorbent for removing various organic and inorganic pollutants from aqueous or gaseous media due to its high surface area, well-developed internal microporosity, and wide spectrum of surface functional groups. Carbon is relatively inexpensive. The high-temperature leftover carbon is activated carbon [7]. It is used in different fields shown in Table 1.

Table 1: Activated carbon approaches in different fields

Applications	End Uses
Industrial Applications	Purification Technique
Medical Applications	Treat positioning and overdoses, oral indigestion
Environment Applications	Groundwater remediation Air purification Water filtration Flexible applications
Gas purification	Hydrocarbons from the air Non-radio-active solid phases
Fuel storage	Store natural gas & hydrogen gas
Chemical purification	pharmaceutical processes
Food additive Applications	Birth control pills &Anti depressants
Agricultural	Biochar
Textiles Applications	Woven Coating Polymer coated carbon Impregnated carbon

The present research determines the environmental issues and spiritual ecology. The spiritual solid waste in India is a place in which there are many distinct religions, worshiping is an integral component of life, and individuals make many diverse compromises to the deities, among which flowering

memorials are widespread. Precisely an outcome, the proportion of specific leaf (*Morinda citrifolia*) recyclables in temple debris is change activated carbon[8].In the present research, activated carbon was prepared from morinda citirifolia leaves by using chemical activation followed by thermal treatments. The activated carbon was characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), and Fourier transform infrared spectroscopy (FTIR).

2. Materials and methods

2.1 Raw materials

The spiritual solid waste (*Morinda citrifolia*) was gathered from many temples in Salem, which come under the Periyar University jurisdictions. MS Scientific supplied all chemicals and reagents, including sodium hydroxide pellets and hydrochloric acid.

2.2 Synthesis of activated carbon

The *Morinda citirifolia* were washed with distilled water and exposed to shade drying for 18 days. The dried leaves were crushed with a mortar pestle, and powdered and sieved 1% NaOH pellets were used for activation. The powder and 1% solution were taken in different concentrations 1:10, 1:20, and 1:30 (w/w) weight to volume ratio, the powder was soaked in 1% NaOH pellets solution for 24 hours and the solution was filtered with filter paper. The wet solid mass is washed several times till the sample PH remains neutral[9]. The samples were dried in an oven at 100°c for 7 hrs to remove moisture content. For the activation, the samples were under a Tube furnace at 600°c for 3 hrs under nitrogen gas flow with a heating rate of 5°c min^{-1}, and settled down the room temperature[10]. The reacted sample was washed with 1.0 M hydrochloric acid to remove inorganic settled impurities and next with distilled water until the filtrate showed neutral pH. Finally, the material was dried in an oven at 80°c to get activated carbon.

2.3 Proximate analysis of Morinda citrifolia leaves

2.3.1 Moisture content

The precise measurement of moisture content under standard procedure (ASTM D2867). The activated carbon heat under the oven at 105°c for constant weight is achieved, indicating that the moisture content has been removed. The percentage of moisture was calculated on a percentage basis (Eq. 1).

$$\text{Moisture content}(\%) = \frac{\text{Initial weight} - \text{Dry weight} \times 100}{\text{Initial weight}} \quad (1)$$

2.3.2 Volatile matter

The dried morinda citrifolia leaves were placed in a crucible covered with a lid and the crucible was transferred to a Tubular furnace, and the temperature was maintained and increased to 600°c with a soak time of 8 mins under standard procedure ASTM D3175. The sample was allowed and cooled then weighed. The loss in weight was reported as a volatile matter on a percentage basis (Eq. 2).

$$\text{Volatile matter}(\%) = \frac{\text{Initial weight} - \text{residue weight} \times 100}{\text{Initial weight}} \quad (2)$$

2.3.3 Ash content

The ASTM D2886 method was used for this process. The dried powder was taken in the crucible (without lid) and heated to 600 ± 5°c for 60 mins in a Tubular furnace. The crucible was taken out and allowed to cool and weighed. The results of ash content on a percentage basis (Eq.3)

$$\text{Ash content}(\%) = \frac{\text{Residue weight} \times 100}{\text{Initial weight}} \quad (3)$$

2.3.4 Fixed carbon content

The fixed carbon content in activated carbon refers to the percentage of carbonaceous material that remains after the removal of moisture, volatile matter, and ash. The fixed carbon (FC) percentage was calculated using the following (Eq.4).

$$\text{Fixed carbon}(\%) = 100 - (\text{Moisture content} + \text{volatile matter} + \text{Ash content}) \quad (4)$$

3. Characterization of activated carbon

Scanning electron microscopy (SEM) Carl Zeiss Microscopy GmbH Germany, reveals particle size and morphology, providing insights into pore structure and surface features. Morinda citrifolia-derived activated carbon typically exhibits irregular shapes with rough surfaces, indicating the presence of well-developed pores. Smaller than 2 nm in diameter, appear as dark, smooth areas due to their limited resolution under SEM analysis. X-ray diffraction analysis reveals the presence and arrangement of atoms in the activated carbon structure[11]. For activated carbon, the main peaks (26° in 2θ) correspond to the graphitic planes of the carbon framework. Fourier-transform infrared (FTIR) spectroscopy is a powerful technique for characterizing functional groups. Common functional groups found on activated carbon include hydroxyl (-OH), carbonyl (-C=O), carboxyl (-COOH), and lactone (-O-C=O-)[12].

4. Results and Discussion

4.1 Proximate analysis

The results of the proximate analysis of the dry sample Morinda citrifolia are shown in Table 2. All the further experiments were conducted only with morinda citrifolia activated carbon; due to the presence of high ash and low carbon content. From the proximate analysis, the fixed carbon percentage was found to be 70% respectively[13]. One main reason for the high percentage of ash content is due to the air presence in the oven during the proximate analysis. In the presence of calcium (Ca), magnesium (Mg), Aluminium (Al), and Silica (Si) might be present in the ash content. Because it depends on the plant source and processing conditions[14]. Further, In the case of specific location and environmental factors, trace amounts of other elements like iron (Fe), manganese (Mn), and zinc (Zn) might also be present in the morinda citrifolia activated carbon[15].

Table 2: Proximate analysis of samples

Sample	Moisture (%)	Ash (%)	Volatile (%)	Fixed carbon
Morinda citrifolia leaves	2%	10 %	9 %	79

4.2 XRD analysis

The XRD analysis of *Morinda citrifolia,* activated carbon is shown in Figure 1. The activated carbon from morinda citrifolia leaves was more amorphous, and the same was observed from the XRD analysis. This AC showed some peaks at around 26°, 29°, 36.2°, 39.8°, 43.22°, 47.5°, and 48.9° respectively of 2θ. [16] The XRD investigation of morinda citrifolia activated carbon reveals indicates the material is mostly amorphous, with a large peak of about 26°. This peak indicates the existence of turbostratic graphite, a kind of graphite with tense stacking of layer structure of graphene. The presence of turbostratic graphene indicates that the activated carbon has undergone partial graphitization. The XRD examination also reveals that activated carbon includes a trace quantity of calcite (CaCO3). Calcite is a prevalent contaminant in activated carbon derived from natural sources like morinda citrifolia. Calcite can impact aspects of activated carbon, such as pH and ash concentration[17]. Overall, In the case of activated carbon from morinda citrifolia, good bonds were observed. The formation of activated carbon can also be confirmed by XRD analysis.

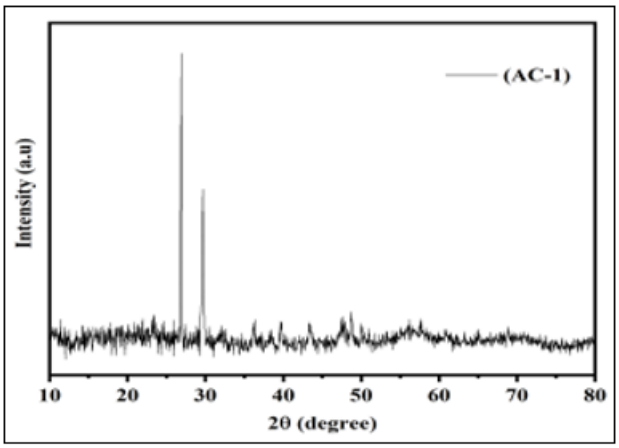


Figure 1: XRD spectra of activated carbon from morinda citrifolia leaves

4.3 Scanning electron microscope (SEM)

The investigation of the surface topography of morinda citrifolia activated carbon was carried out using SEM analysis, and the results are depicted in Figure 2. The SEM image indicates NaOH activation has covered the activated carbon outer surface with voids of various shapes and sizes. The carbon structure voids of various sizes result from NaOH evaporation during the carbonization[18]. SEM analysis reveals the presence of s highly heterogeneous surface with randomly oriented pits of several flaky particles, macro pores, and high porosity with a series of scattered holes. Although there were some micropores (less than 2 nm) and macro pores (greater than 50 nm) present, the majority of the pore size distribution fell into the mesopore range (2–50 nm). It was discovered that the activated carbon has 520 m2/g of surface area. According to the SEM study, activated carbon derived from the leaves of Morinda citrifolia offers a wide range of possible uses, including energy storage, catalysis, and adsorption.

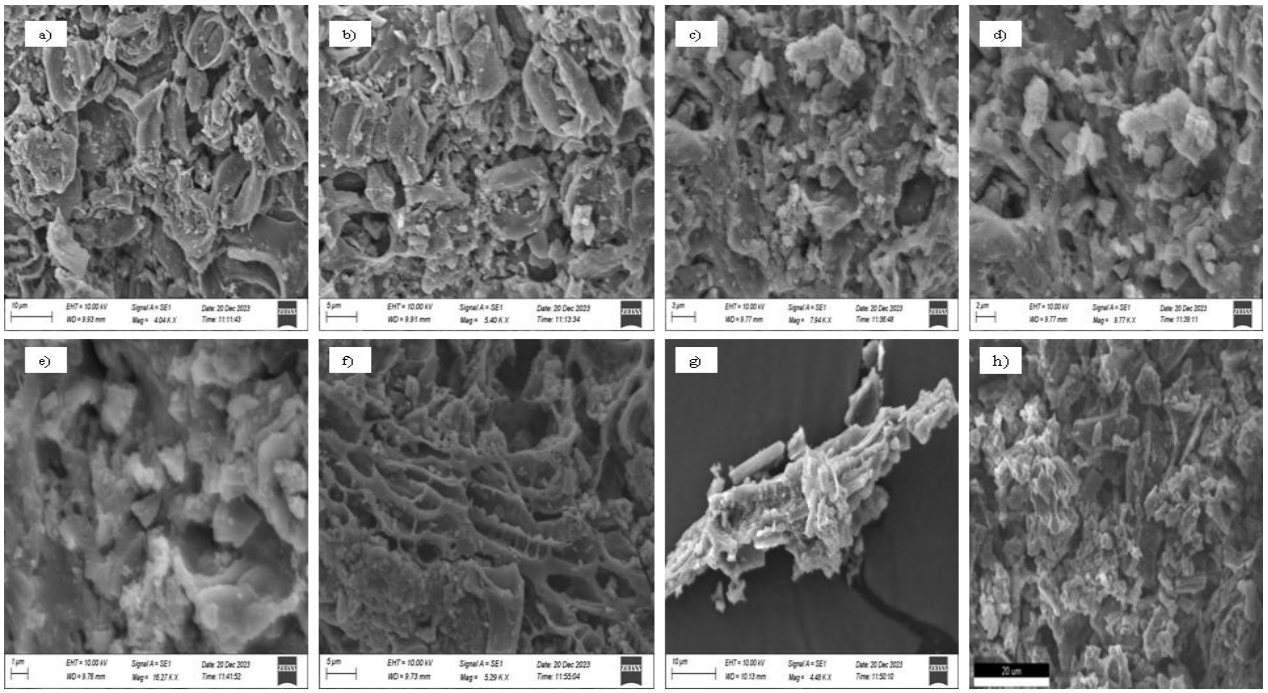
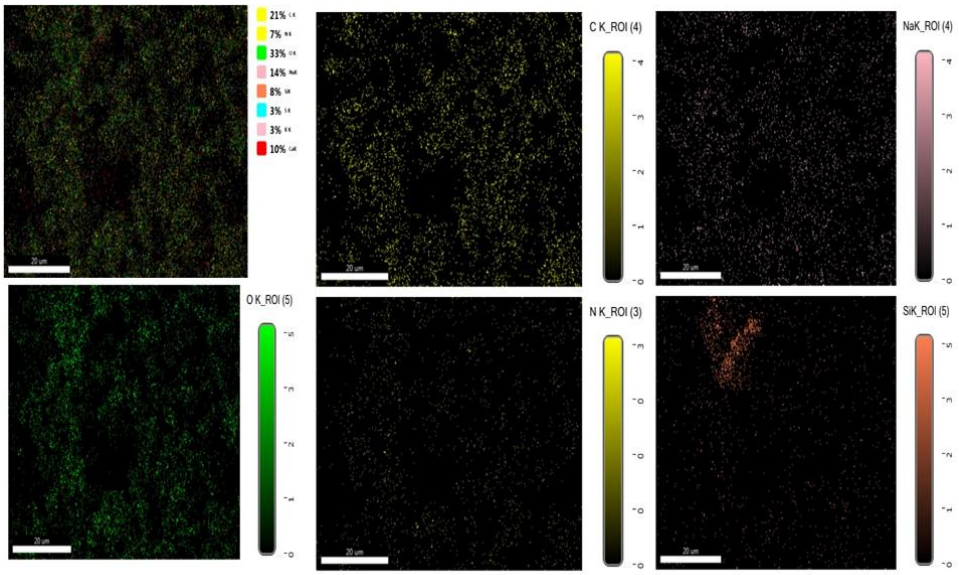
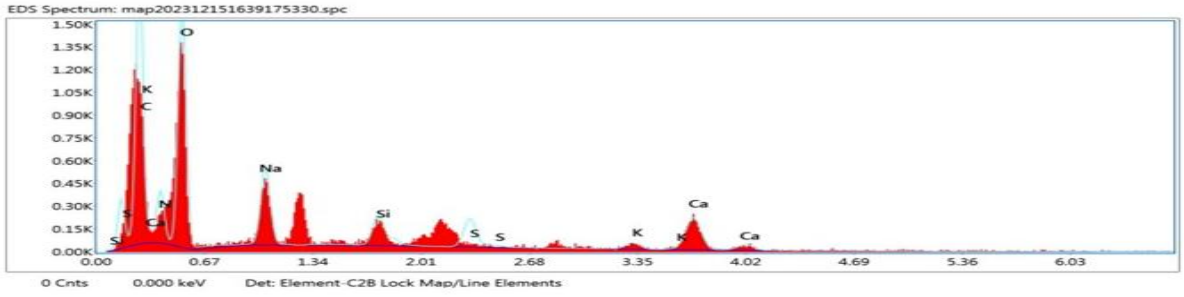


Figure 2: SEM images of activated carbon from morinda citrifolia leaves

4.4 EDX analysis

Energy-dispersive X-ray spectroscopy (EDX) is a technique that can be used to identify the elemental composition of a material. In the case of activated carbon, EDX can be used to determine the presence of elements such as carbon, oxygen, and various metals. The elemental arrangement and integration of carbon elements contained in the material are demonstrated by EDX analysis of the particles in Figure 3. The EDX spectrum showed that the activated carbon was mainly composed of carbon (32.15%) and oxygen (34.51%). There were also small amounts of other elements present, such as potassium (1.03%), calcium (7.21%), and Sodium (6.63%).





Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
C K	21.99	32.15	100.61	8.45	0.1173	1.0958	0.4866	1.0000
N K	10.95	13.73	23.59	13.39	0.0295	1.0637	0.2533	1.0000
O K	31.44	34.51	120.58	9.53	0.1189	1.0359	0.3650	1.0000
NaK	8.68	6.63	43.69	7.16	0.0577	0.9284	0.7144	1.0012
SiK	3.28	2.05	20.66	7.07	0.0280	0.9196	0.9239	1.0061
S K	4.90	2.68	21.09	9.90	0.0431	0.8951	0.9713	1.0118
K K	2.30	1.03	5.66	21.04	0.0200	0.8392	0.9949	1.0397
CaK	16.46	7.21	30.14	6.86	0.1407	0.8513	0.9969	1.0074

Figure 3: EDX spectra and mapping

4.5 Fourier-transform infrared (FTIR) spectroscopy

Fourier transform infrared spectroscopy was employed to investigate the functional group and gather information about the chemical composition of the generated Activated carbon. The FTIR spectra of activated carbon from Morinda citrifolia are shown in Figure 4. In the spectrum of the broad absorption band at 3697.59cm^{-1}. It is due to the stretching frequency of hydroxyl groups (-OH) and the absorption band at about 2925.01cm^{-1} corresponds to C-H stretching in methyl and methylene groups. In the case of a band about 1794.14 cm^{-1} is due to C=O stretching vibration. The absorption band is between 1417.02cm^{-1} to 874.24cm^{-1}. Due to carboxyl groups, esters, and Phenol groups. All of these chemical modifications verified the carbonization process[18].

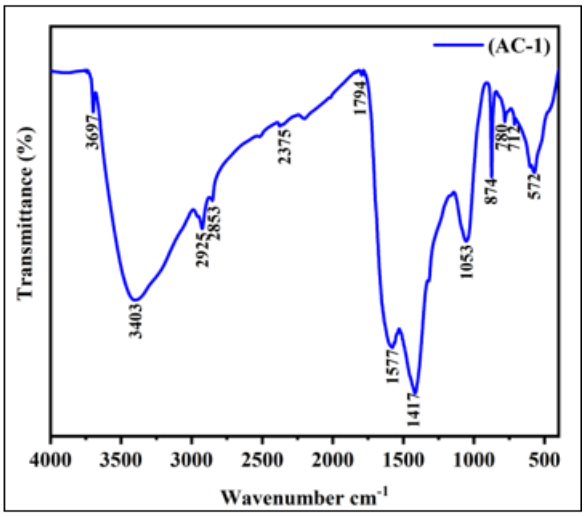


Figure 4: FTIR spectra of activated carbon

4.6 Waste water treatments

In this experiment, three different ratios (1:10, 1:20, 1:30)were used. The effluent water presents scouring, bleaching oils, chemicals, and acids[19]. The change in turbidity of effluent water was studied with respective to time and the results are depicted in figure 5. The turbidity content was found to decrease with time[20]. In this case, different time intervals (0, 10, 20, 30, 40, and 50 minutes) were treated with effluent water. The turbidity suddenly dropped from 1g to 5 ml in the first 5 mins and the turbidity value got stabilized at 5ml. In the case of morinda citrifolia-activated carbon, a gradual decrease in turbidity with treatment time was noticed[21]. Change in pH was also studied in both cases, and the morinda citrifolia activated carbon showed considerable change in pH from 6.73 to 7, whereas the solution decreased and resulted in the increased removal of efficiency due to the completing nature of hydrogen ions at low pH[22].

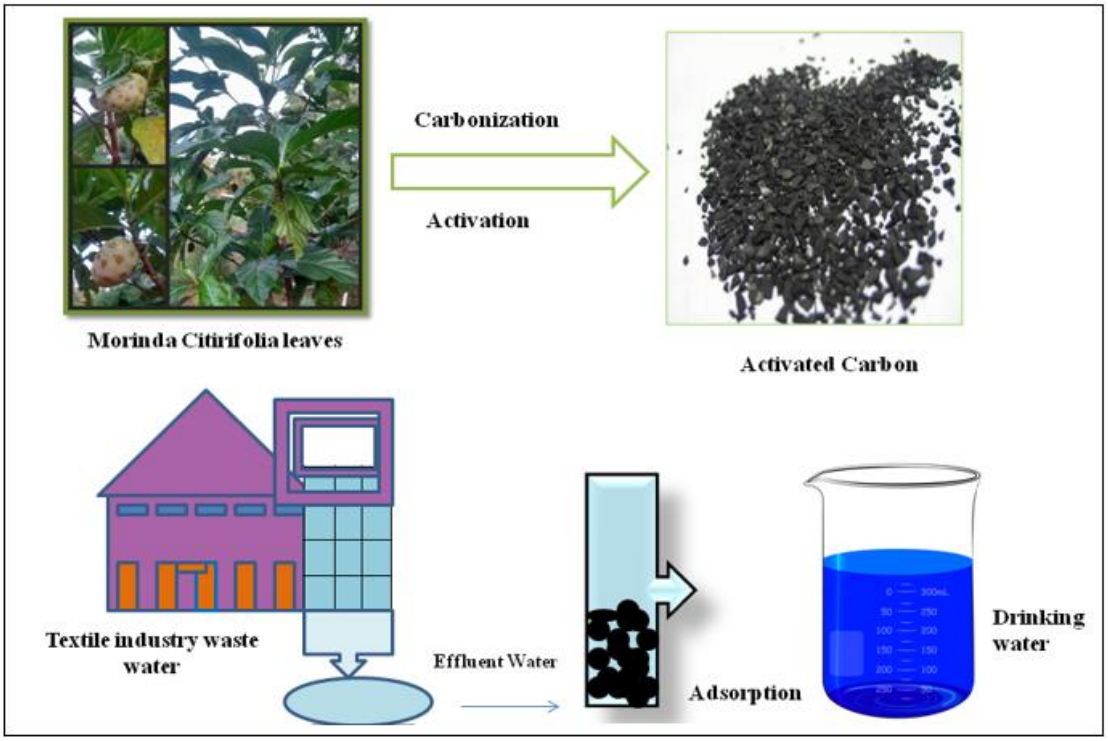


Figure 5: Overall process for effluent wastewater treatment

4.7 Hydro Analysis

4.7.1 Testing Parameters:

Table 3, shown hydro analysis (Efflent water and AC degraded water) insightful results regarding various parameters crucial for evaluating water quality. The pH level, registering at 8.47 effulent water higher than degraded water 7.46, falls comfortably within the optimal range of 6.5 to 8.5, indicating a neutral to slightly alkaline nature conducive to aquatic ecosystems and human consumption. Total Dissolved Solids (TDS) are measured effulent water at 7450 mg/L, degraded water at 9070 mg/L and, slightly below the acceptable limit of 500 mg/L, suggesting relatively high concentrations of dissolved substances in the water. Total Hardness, quantified effulent water at 84.07 mg/L and degraded water at 328.3 (Below detectable limit) as CaCO3, reflects a level within acceptable bounds, ensuring water's suitability for various applications. Calcium content is measured degraded water at 41.7 mg/L (high detectable limit) indicating a presence conducive to maintaining water hardness higher within acceptable limits.

Table 3: Hydro Analysis

S:No	Parameters	Test Method	Units	Acceptable Limit	Results	
					Effulent Water	Activated Carbon Treated water
1.	pH Value at 25°c	IS 3025 P.11.1983 RA 2017	-	6.5-8.5 Agreeable	8.47	7.46
2	Color	IS 3025 P.16:1984 RA 2021	HU	-	10	18
3	Total dissolved solids	IS 3025 P.16:1984 RA 2017	mg/L	500 mg/L	7450	9070
4	Total Hardness (as $CaCO_3$)	IS 3025 P.21:2009RA 2019	Mg/L	200 mg/L	84.07	328.3

4.8 Photocatalytic dye degradation

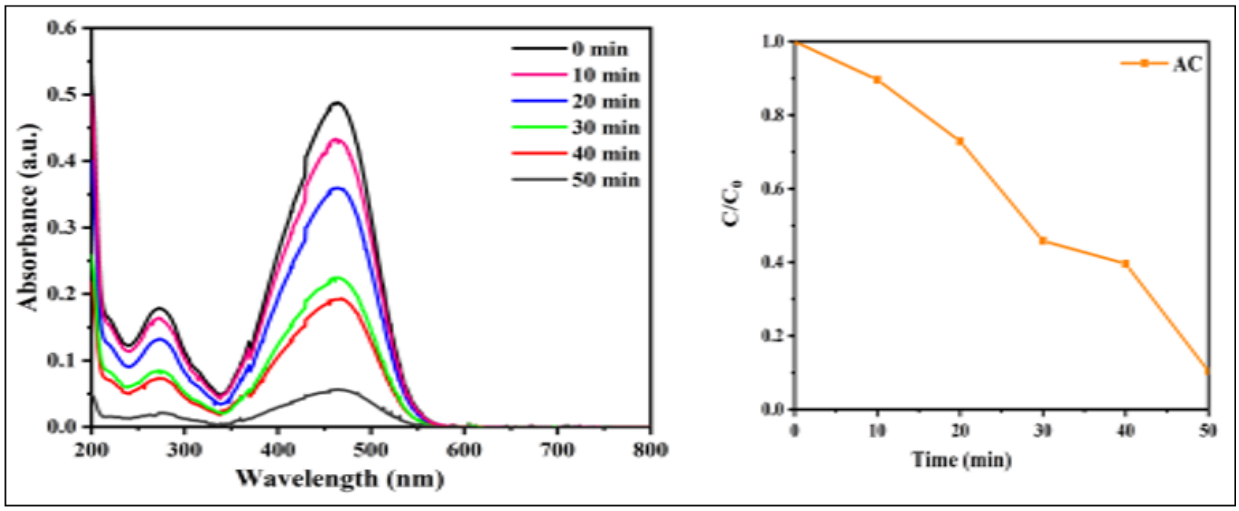
The UV VIS Spectrophotometer was used to analyze the photocatalyst performance during methyl orange dye degradation testing. The initial concentration of 0.01g under visible light irradiation. Before irradation, the dye solution(5ml) with the photocatalyst was agitated and held in the dark until absorption equilibrium was reached. The dye solution sample was taken at regular intervals of 10 minutes. Finally, the liquid was tested with a UV-VIS spectrophotometer to determine the absorbance, which was used to measure the quantity of methyl orange. The maximum absorption wavelength of the dye was 600nm. The absorption spectra of methyl orange dye solutions were examined at 10 minutes intervals. The rate of degradation was reported by computing the proportion of the concentration of the dye that remained over the original dye concentration. It obtained from the absorption standard curve. The degradation performance can be evaluated by considering the following equation(5) and (6).

$$\text{Degradation (\%)} = [(C_0 - C_t)/C_0)] \times 100 \quad (5)$$

where C_0 and C_t are the initial and final concentrations of methyl orange (mg/L) at reaction time (t), respectively.

$$\ln (C_0/C_t) = kt \quad (6)$$

where C_0/C_t is the normalized MO concentration, k is the first-order rate constant and t is the reaction time.



5. Conclusion

Utilizing Morinda citrifolia leaves from temple solid waste as a raw material for activated carbon synthesis promotes sustainable resource utilization and waste management. This eco-friendly approach contributes to the reduction of organic waste while providing a valuable resource for water treatment. The experiment considered different concentrations (1:10, 1:20, 1:30)and time intervals (0, 10, 20, 30, 40, and 50 minutes) to understand the optimal conditions for adsorption and characterized by Hydro analysis testing for effulent water treatements. The UV VIS Spectrometer test demonstrated that during the process of methyl orange dye degradation light, the catalyst breaks down the dye molecule in around 30 minutes, reaching a high of 89.59%. When exposed to light, the catalyst produces reactive oxygen, which attacks and breaks down the dye molecules. Conduct further studies to determine the optimal contact time, dosage, and regeneration protocols for specific types of effluent water and targeted pollutants. Further research and development are needed to optimize the process and ensure its scalability and practical implementation. This approach could potentially contribute to cleaner water resources and reduced environmental pollution, particularly in regions with temple waste management challenges.

Author Contributions

SM: Investigation; Methodology; performed the experiments; analyzed and interpreted the data; Original draft preparation; Material preparation.
LM: Supervision; Validation; Review and editing.

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Data Availability

The datasets generated during the current study can be requested from the corresponding authors upon reasonable request.

Ethics declarations

Ethics approval
Not applicable.

Consent to participate
All the authors agreed to participate in the work of the paper.

Consent for publication
All the authors agreed to publish the paper.

Competing interests
The authors declare no competing interests

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