
PREFACE

Earthly biomass can be a significant source for the synthesis of mesoporous carbon compounds with different specific surface areas and porosities. Up to 71% of the Earth's surface is made up of water for all purposes. Fresh water, which is found underground and in glaciers, lakes, and rivers, makes up just 1%. Metals, dyes, and other organic liquids are the main sources of pollution, and the industries' waste chemicals are contaminating this fresh water. Dye, which is a colouring chemical utilized for various purposes, is discarded mainly by textile industries into the surface water is a big concern of wastewater pollution. These pollutants cause various environmental harm, like carcinogenic, mutagenic, skin irritation, poisonous and DNA and RNA intercalation. The goal of the research is to thoroughly close the gap mentioned above while taking into account all of the study's parameters. The thesis uses the physico-chemical features of mesoporous carbon and its composite to eliminate dangerous dyes such as Orange G, Rhodamine B, and Crystal Violet. The objective of the study is as follows. Tectona Grandis sawdust is a readily accessible feedstock, and zinc chloride is used as a chemical activator to produce mesoporous carbon. Using urea and an aluminium nitrate precursor, gamma-alumina is created using the solution combustion process.

Gamma-alumina decorated mesoporous activated carbon (MAC@Al) adsorbent is synthesized using a hydrothermal method. Laboratory-scale investigation of the removal of crystal violet (CV) staining dye from aqueous solution. The MAC@Al adsorbent was regenerated utilizing a chemical-sonication technique. The MAC@Al adsorbent's characteristics and the removal research of CV dye in comparison to other co-adsorbents. The crystal violet removal study compares MAC and MAC@Al, bare activated carbon (BAC), and gamma-alumina decorated bare activated carbon (BAC@Al). Interference investigation with metal salts such as Ca^{2+} , Mg^{2+} , Cr^{3+} , and As^{5+} to verify the limits of adsorption removal. To further select corn husk biomass from agricultural sources, mesoporous carbon can be chemically synthesized in a nitrogen atmosphere using orthophosphoric acid as a chemical activating agent.

The investigation of impregnation with corn husk and chemical activating agent to improve the mesoporous carbon adsorbent. The mesoporous activated carbon (CHMAC) derived from maize husks was characterized through the use of XRD,

FTIR, BET, HR-TEM, SEM, and XPS analysis. The removal of two carcinogenic dyes, Orange G and Rhodamine B, was investigated using CHMAC adsorbent at varying temperatures. Kinetic, isothermal, and regeneration analysis of CHMAC's removal of rhodamine B and OG dye. Overall analysis comparing these dyes with other studies in the literature that use these adsorbents. Conclusion of these findings about our desired and obtained outcomes. Then, crystal violet, a cationic dye, comes into contact with human skin, it irritates it and has toxic properties. Rhodamine B, which is a xanthene dye commonly used in paper, textiles, and leather industries, cause reproductive toxicity in aquatic organisms and has mutagenic impact. Orange G, which is an azo dye often observed in the wastewater used in textile industries, causes mutagenic and carcinogenic effects in aquatic media. So, the removal of these hazardous dyes from wastewater utilizing biomass-derived mesoporous carbon materials from teak saw dust and corn husk (*Zea mays*) biomass can be excluded from the wastewater media. Chemical agents like zinc chloride salts and orthophosphoric acid were observed to be best for reforming biomass materials to mesoporous carbon materials after synthesis in nitrogen presence. The high specific surface area for the adsorbents MAC@Al (393 m²/g) and CHMAC 2 Imp 7 days (1668.18 m²/g) gave the suitability to the mesoporous range for the dye removal from wastewater media.

The mesoporous carbon is synthesized in the presence of nitrogen gas. First, the biomass which was chemically activated with zinc chloride and phosphoric acid up to a fixed time with a biomass/ chemical ratio. Then it was pyrolysed at 550 degrees Celsius and then washed several times to remove the chemical using acid and bases, then activated at 700 degrees Celsius to obtain mesoporous carbon of porosity between 2-50 nm range. Solution combustion methodology was utilized to synthesize gamma-alumina using urea as fuel with aluminium nitrate precursor. The support of this gamma alumina adsorbent on the surface of MAC improved and enhanced the removal percentage of crystal violet dye. A fixed (500 ppm) concentration of crystal violet dye solution was made and serially diluted to 20, 40, 60, and 80 ppm concentrations for analysis purposes in the work. The equilibrium analysis was checked for the CV dye removal within 0-30 minutes of duration. The removal efficiency was greater than 98 per cent using MAC@Al adsorbent as compared to other adsorbents synthesized without zinc chloride activation. These adsorbents were BAC (bare activated carbon) and BAC@Al (gamma alumina-supported bare activated carbon). Four different isotherms, Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich, were employed during CV dye removal using MAC@Al adsorbent. Four different kinetics to know the rate and nature of adsorption were

employed, namely as pseudo-first order, pseudo-second order, Elovich, and Intraparticle diffusion model. MAC@Al adsorbent was checked for surface charge analysis by using the pH_{ZPC} method. Metal salts interfere as discussed above; the objective showed that some metals have increased the removal efficiency within the solution of crystal violet dye, and some have decreased. When all metal salts were mixed in the solution of the CV dye, they caused interference within the solution of dye and has decreased the dye removal efficiency as compared to 40 ppm CV dye itself, utilizing MAC@Al adsorbent. The dose of the adsorbent was 1 g/L. Ganga water was utilized from the Varanasi area, and in that water, crystal violet dye removal was checked at pH 7, and it was observed to be higher (greater than 98 %) removal using MAC@Al adsorbent. So, the removal rate of CV dye in synthetic and Ganga-made wastewater was higher utilizing this adsorbent. Thermodynamic analysis was performed on CV dye at 298, 303, 308, and 318 K. The application of the Van't Hoff equation led to the conclusion that adsorption is exothermic and possible at all temperatures. Using a 0.1 Molar solution base NaOH, water bath shaking, and sonication at predetermined intervals, the adsorbent MAC@Al was regenerated. The adsorbent underwent five cycles of regeneration, with removal rates over 80% until the fourth cycle, after which they fell below 80% in the fifth cycle. The CV dye's adsorption loading on the MAC@Al adsorbent surface was 14.8 mg/g, according to Langmuir analysis. This study filled the gap of previously utilized activated carbon and other adsorbents used already in CV dye removal from wastewater media.

Corn Husk biomass was collected from the Banaras Hindu University Agriculture campus, Varanasi, to synthesize mesoporous carbon from this biomass. Phosphoric acid was utilized with a fixed molar concentration solution and fixed w/w ratio of biomass/acid at times 12 hours and 7 days. A total of six adsorbents were synthesized and compared for the cationic and anionic dye removal from wastewater, was Rhodamine B and Orange G. Out of all six adsorbents, CHMAC 2 Imp 7 days (corn husk-derived mesoporous carbon of w/w (2) impregnated 7 days duration adsorbent) was best in the removal of these dyes. Removal percentage was above 98 % in the case of both dyes. A fixed concentration (250 ppm) solution of the dyes was made and serially diluted to 20, 40, 60, 80, and 100 ppm. These concentrations of dyes with volume (60 mL) were used for removal analysis using 50 mg CHMAC 2 Imp 7 days adsorbent. The removal time was fixed from 0 to 2 hours. The dose of the adsorbent during analysis was 0.8 g/L. pH_{ZPC} analysis was done in the case of CHMAC 2 Imp 7 days adsorbent to know the surface charge. It was observed that

pH_{ZPC} was near to 7, showing an electrically neutral nature on the surface of the adsorbent at this pH value. The four different isotherm analyses were conducted, namely Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich isotherms. Kinetic analyses of the dyes using CHMAC 2 Imp 7 days were checked using the kinetics pseudo-first order, pseudo-second order, Elovich model, and Intraparticle diffusion model. From Langmuir analysis, the adsorption loading of the OG and RhB dye on the adsorbent surface was above 95 and 105 mg/g. Regeneration study of the adsorbent was done seven times using HCl and an ethanolic water mixture of fixed concentration using water bath sonication. The desorption efficiency was above 80 per cent up to seven cycles discussed in the study. This thesis is divided into five chapters: Introduction, materials and methodology, removal of CV dye using MAC@Al, removal of OG and RhB dye using CHMAC 2 Imp 7 days adsorbent, and conclusion. Earlier in the literature, teak and corn husk-derived mesoporous carbon materials were not utilized for the dye elimination from wastewater medium. CHMAC 2 Imp 7 days was used to check the removal of both dyes at different pH values. The higher removal after analysis was observed at neutral pH in the case of both dyes. Thermodynamic analysis was performed on OG and RhB dye at 303, 308, and 318 K. The application of the Van't Hoff equation led to the conclusion that adsorption is exothermic and possible at all temperatures 303, 313, and 323 K. After analysis of the thermodynamics study, it reveals that the adsorption of both dyes at all temperatures is spontaneous and exothermic in the case of OG dye and endothermic in the case of RhB dye.

The randomness was checked by entropy values, which were positive in the case of RhB dye and negative in the case of OG dye. Phosphoric acid treatment in the biomass made the carbon much mesoporous and of higher specific surface area. After adsorption, SEM, FTIR, XPS, and EDS analyses have shown the adsorption of both dyes successfully onto the surface of CHMAC 2 Imp 7 days adsorbent. Raman analysis was also conducted before and after scanning to know the change in defects of the adsorbent before and after adsorption of these dyes. Structural properties have changed the surface defects in the adsorbent materials after adsorption. In the case of OG dye, adsorption defects have increased as compared to pristine materials, and it is decrease after adsorption of RhB dye. Freundlich and Langmuir adsorption isotherms out among other isotherm models, were fitted best by defining the multilayer and monolayer adsorption of the dye by the adsorbents, respectively. Novelty was obtained when the removal capacity increased by the other literature studied adsorbents, including activated carbon. Crystal violet, rhodamine B, and

orange G showed exothermic, endothermic, and exothermic nature of thermodynamic property after adsorption analysis using the different mesoporous carbon materials. Pseudo-second order kinetics with a chemisorption nature of dye removal was shown by each dye using different adsorbents, respectively. The use of biomass and teak sawdust has shown the best modification to form mesoporous carbon materials. These biomasses are rich in lignin content after literature analysis. Mesoporosity in the case of the adsorbent has played a good role in the removal of cationic and anionic dyes from the wastewater medium. MAC@Al adsorbent has shown the multilayer chemical adsorption of crystal violet dye, and CHMAC 2 Imp has shown the monolayer chemical adsorption nature of OG and RhB dye, respectively. The utilization of biomass is economical for the synthesis of mesoporous carbon materials through the chemical method. It was observed that it can be industrially applied in the removal of textile dyes, which are cationic and anionic in nature.

The batch mode study to remove these dye pollutants from the wastewater medium was utilized. RPM value was 150-200 during shaking, and what man filter of the best grade was utilized for filtering purposes during analysis. The residual concentration of the dyes was evaluated using a calibration plot made of a fixed concentration, utilizing the UV-Vis instrument. Hydrochloric acid and sodium, and potassium hydroxide were used for pH maintenance of the dye solutions. Buffer capsules of pH 3, 5, 7, 9, and 12 were utilized for pH adjustment and calibration. The origin 2018/2021 pro software was utilized for graph plotting of different data. The utilization of gamma alumina on the surface of the MAC adsorbent was done because it is a good adsorbent, amphoteric, and does not react with water. XPS results have shown that nitrogen, sodium Sulphur, and chlorine were observed bound with carbon, confirming the adsorption of these triphenyl methane, xanthene and azo dye. SEM analysis after adsorption showed that the surface pores were filled with adsorbate, and the topography of the adsorbents changed.

Mechanism of the dye removal using MAC@Al and CHMAC 2 Imp 7 days adsorbent was proposed, showing that the crystal violet dye can do π - π interaction, hydrogen bonding, ionic bonding, and van der Waals force of attraction between adsorbate and adsorbent. In the case of CHMAC 2 Imp 7 days adsorbent, OG dye may cause ionic bonding, π - π interaction, and hydrogen bonding, and in the case of RhB dye, it may be due to dipole-dipole interaction, π - π interaction, and hydrogen bonding. Higher per cent removal and higher loading capacity of these dyes on the surface adsorbent made these biomasses full impact to be utilized for mesoporous

carbon synthesis. These biomass sources are rich in lignin, are available worldwide, and can be utilized for the various types of activated carbon synthesis. BET and BJH analysis showed that both adsorbents have average particle diameters in the meso range (2-50 nm) with pore volume below 1 cm³/g. So, these results have drawn the attention of biomass utilization to synthesize mesoporous carbon materials to remove industrial dyes from surface runoff wastewater. Based on future perspectives these adsorbents can also be employed in the removal of other pollutants from wastewater. The chapter-wise discussion of the work is compiled in each chapter, and the references are listed at the end of the last chapter. Chapters 3 and 4 are the removal and characterization chapters for the adsorbents synthesized.