# CHAPTER-5

## Conclusion and Future Scope

## Chapter 5: Conclusion and Future Scope

The conclusion of the work is reported in four sections based on the chapter-wise data utilized in writing, with future scope. These are as follows:

### Chapter 1: Conclusion

Since day-to-day water scarcity is increasing due to it leads to diseases in humans and other biota. Various wastewater pollutants are being dumped by the industries, domestically and via other means. These are pesticides, surfactants, heavy metals, organic liquids and textile dye materials. There are several water pollutions, like groundwater pollution, surface water pollution, and agricultural water pollution. This pollution is caused by these sources, which are coming from human activities for their future needs. The mining sector, steel and iron manufacturing facilities, industrial laundry facilities, power plants, oil and gas fracking facilities, metal finishers, and the food and beverage sector are a few instances of industries that generate wastewater. Some other pollutant, including shampoo, body wash, dish soap, and laundry detergent, produces 1,4-dioxane as a contaminant for wastewater. Taste, odour, pH, BOD, COD level, and pathogenic problems are happening due to this pollution in the water system. Literature review shows that researchers are focusing on methodology to improve and want to remove it from wastewater without losing any side chemicals or hazardous byproducts, through which water becomes more scaring to be used by human use for drinking and washing purposes. India, which is the third largest producer of textile dyes in the world, makes various types of dyes for fabric, leather, cosmetics, and other coloured materials used in products. These are vat dyes, anthraquinone dyes, phenolphthalein dyes, indigo dyes, and azo dyes. The total suspended solid concentration increases, BOD and COD values change, pH and odour of water vary, and total dissolved solid concentration increases, when these dye goes into the water media.

For instance, dispersion yellow 3 has a carcinogenic effect with liver damage and the development of hepatocellular tumours, while methylene blue dye prevents the inhibition of microalgae growth. In certain Salmonella strains, rhodamine B possesses a reverse mutation that renders it mutagenic. Crystal violet dye is toxic to the lungs and can irritate the skin upon inhalation. In general, aquatic plants and animals are at risk from reactive red 120. Crystal violet is mostly used in biofilm, staining of biological molecules and in fingerprint film utilization. Toxic gases with a disagreeable odour, including Sulphur, formaldehyde, nitrogen oxides, volatile chemicals, particulate matter, and dusts, are released by the dye business. When chemical dye effluent and its byproducts are discovered in wastewater streams or textile industry dust, there are detrimental health hazards. These dyes belong to various families and are hazardous to biota.

These dye removals are done by various methodologies like photodegradation, ozonation, advanced oxidation process, biological method, and adsorption. In all of these methodologies, besides adsorption, all methods have some disadvantages, like sludge formation, hazardous side products formation within the media, higher cost of the gas or chemicals, and lower regeneration ability. So, the adsorption method is chosen for the dye removal from wastewater because of its good process, less time, no sludge formation, no hazardous side product formation, economical, multiple times regeneration ability, and stable at various temperatures based on the adsorbent. It is a phenomenon of dye removal by surface contact between the adsorbate and adsorbent. Various adsorbents have been utilized for dye removal, like silica oxide, chitosan, zeolites, biochar, activated carbon, and some other metal framework-related adsorbents. Out of all these, activated carbon has shown a remarkable impact in the removal of these dyes. It is because of their good porous nature, high specific surface area, long-term stability, economical (easily can be synthesized from chemical and biomass waste), meso, micro, and macro range availability, and remarkable loading of these dyes within a shorter time from wastewater medium. So, Mesoporous (2-50 nm) range carbon from various biomass sources like corn crop, sawdust was synthesized and used for the crystal violet, rhodamine B and orange G removal. This chapter concluded the objective to synthesize mesoporous carbon materials from tectona grandis sawdust and corn crop husk waste residue.

Using urea and an aluminium nitrate precursor, gamma-alumina is created by 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. To compare crystal violet removal studies, MAC and MAC@Al, bare activated carbon (BAC), and gamma-alumina decorated bare activated carbon (BAC@Al) were utilized. To further select corn husk biomass from agricultural sources, mesoporous carbon can be chemically synthesised in a nitrogen atmosphere using orthophosphoric acid as a chemical activating agent. The examination of impregnation with maize husk and chemical activation 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, 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 research in the literature that uses these adsorbents. These outcomes are in line with our desires and what we obtained.

**Chapter 2: Conclusion**

In this chapter, the synthesis of the adsorbents for dye removal was illustrated based on the previous methodology reported in the literature for the synthesis of mesoporous carbon. Biomass collection was done from the BHU campus and the nearby area. These biomass is widely available, mostly in Asian countries. The lignin content was enough in this biomass, as from the literature survey, and that's why these are utilized for dye removal after synthesizing in the form of mesoporous carbon. The chemical method of activation of biomass to mesoporous carbon was reported as the best and economical method. So, we have utilized the method for the production of mesoporous carbon, choosing zinc chloride and orthophosphoric acid as chemical activating agents. The support in one of the mesoporous carbons from teak sawdust biomass was done by synthesizing gamma alumina from the solution combustion method reported in literature, with some changes to the temperature and time duration of the reaction. The support was made using the hydrothermal method, since the removal of dyes alone by the mesoporous carbon was less. In case of the corn husk biomass-based mesoporous carbon synthesis, the orthophosphoric acid was utilized for the synthesis of CHMACs and rhodamine B and orange G were selected for dye removal. Various acids and bases are utilized for the pH and other work during synthesis. From this method, almost a good enough mesoporous carbon was synthesised as with a yield above 45 %. Various characterization method was employed to prove the synthesis of adsorbents and dye removal from wastewater media. These are XRD, FTIR, SEM, XPS, BET, and EDAX.

To know the characteristics of adsorption of dyes, various isotherms were studied and discussed in the chapter, are Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich isotherms models. Kinetics analysis was proposed using various kinetics, like Pseudo-First-Order, Pseudo-Second-Order, Elovich Model, and Intraparticle diffusion models. These methods of checking and elaborating from linear to non-linear models for studying the nature of adsorption were done using these adsorbents. To check the adsorption of dyes by the adsorbent was measured using a UV-Vis spectrophotometer. Calibration of the standard was also done using the spectrophotometer at different concentrations. Regeneration of the adsorbent was done using HCl, $C_2H_5OH$, NaOH, etc., for different dyes. Water bath shaker, ultra bath sonicator and heating methods are utilized for the regeneration study of the adsorbents.

**Chapter 3: Conclusion**

In this chapter, synthesized adsorbents MAC, BAC, MAC@Al, and BAC@Al are used for the removal of crystal violet staining dyes from synthetic wastewater and from Ganga water. During the removal of CV dye using the MAC, the BAC adsorbent was less, and then the elimination of CV dye was checked and contrasted with gamma-alumina supported mesoporous and bare activated carbon adsorbent, i.e, MAC@Al and BAC@Al. in both of these adsorbent MAC@Al chosen and proved to be the best for CV dye removal from both types of water media. XRD study revealed the successful synthesis of the MAC, BAC, MAC@Al and BAC@Al adsorbents with the planes in the diffractogram. Gamma-alumina was in the metastable phase with a cubic close-packed structure. The functionality of the surface of adsorbents is also characterized using the FTIR method, which was analysed with literature proving the formation of adsorbents and CV dye removal from wastewater. The BET/BJH analysis has shown that the adsorbent is mesoporous with a specific surface area of around 393 $m^2/g$. Raman analysis for MAC@Al was utilized to determine the defects and graphitic form on the adsorbent surface, proving that defects were less due to the higher peak intensity of the G-band in the Raman spectrum. It was compared with MAC also. The adsorbent was utilized to remove CV dye within 30 minutes, and it showed that removal was higher at 30 minutes, above 98 per cent. Teak sawdust was a wood material residue, which is why porosity was evenly distributed in the MAC adsorbent. In the SEM image of the BAC adsorbent, it has very wide pores which was not activated using zinc chloride. X-ray photoelectron analysis has shown that in MAC, only C 1s and O 1s elements with atomic weight per cent values are present in the major form. When gamma alumina was decorated on its surface Al 2p elemental peak was observed with the bonds O-Al and C-Al, etc. This analysis has proven the successful synthesis of support for gamma alumina on the MAC adsorbent. 20, 40, 60, 80 ppm concentration of CV dye was taken for removal purposes using batch mode analysis. A UV-Vis spectrophotometer was used to determine the residual concentration of CV dye. pH analysis for CV dye removal was done in acidic and basic media has shown that the removal percentage is higher in the basic range. The optimum dose was 1g/L during the removal process.

The isotherm study, which is discussed above, out of all of them Freundlich adsorption isotherm with multilayer adsorption characteristics and adsorbate-adsorbent interaction was accomplished after analysis. Pearson's R-squared value was close to unity, proving the best fit for the elimination of CV dye from wastewater. To check the rate of the adsorption of CV dye, elimination was followed by pseudo-second order kinetics, defining the chemical adsorption properties between the adsorbate and adsorbent. A thermodynamic study has revealed that adsorption is spontaneous at all temperatures, with an exothermic nature of adsorption. MAC@Al adsorbent regeneration study has revealed that it can remove CV dye above 80 per cent up to the fourth cycle, and it decreases in the fifth mode of regeneration below 80 per cent. Various types of binding and interaction may be the cause of the adsorption of CV dye using the MAC@Al adsorbent. These are pi-pi bonding, ionic bonding, covalent bonding, hydrogen bonding, and dipole-dipole types of interaction. The mechanism of the adsorption was assessed using FTIR, SEM, XPS, EDS, and elemental mapping analysis. These results have shown that the azo group and chloride bond with nitrogen and carbon peak in XPS, and the pore filling nature of dye in the adsorbent, post-adsorption analysed from SEM, confirms the CV dye removal using MAC@Al adsorbent. Positive shifts were also assessed in the O-Al peak, confirming the chemisorption adsorption property of CV dye. Pseudo-second order kinetics was followed during analysis, out of all other kinetic models, with a loading capacity of 14.8 mg/g. This chapter concluded the use of teak sawdust biomass to produce mesoporous carbon after zinc chloride activation, utilizing a tubular furnace and a nitrogen environment. Gamma-alumina was made as a support to improve the dye removal rate. Metal salts interference study has revealed that when we are utilizing salts separately at different concentrations, their removal efficiency increases in case of calcium and magnesium, but it decreases in other metal salt cases with

40 ppm CV dye concentration. This may be due to the nature of salts and their interaction with the CV dye. When we utilize all metal salts together of two different concentrations with 40 ppm CV dye, it shows a decrease in the removal percentage. It may be due to the gathering of all the metal salts together, adhering to the surface of the adsorbent, making the adsorbent less available for CV dye from the wastewater medium. Intraparticle diffusion study has revealed that pore filling properties of the adsorbent are not the major cause of CV dye removal, since it was not the best after analysis, as compared to pseudo-second order kinetics.

### Chapter 4: Conclusion

Corn husk biomass was selected to be used for the synthesis of mesoporous carbon. In this study, this biomass was chemically treated with phosphoric acid with six different w/w ratios of the biomass/chemical by fixing the weight of the biomass to 5 grams. Six different adsorbents, which were obtained after synthesis, were chosen for the two different dye removal studies, Orange G and Rhodamine B. These are cationic and anionic in their structural form. From stock concentration 20-100 ppm, the concentration of these dyes was utilized for the removal investigation. CHMAC 0.8 Imp 12 hours/7 days, CHMAC 1 Imp 12 hours/7days, and CHMAC 2 Imp 12 hours/7days adsorbent were obtained. Out of all these adsorbents, the highest removal efficiency was in the case of CHMAC 2 Imp 7 days adsorbent. This adsorbent was utilized for all the thermodynamic, kinetic and isotherms analysis of both dyes in the study.

Removal capacity was higher in the case of RhB dye removal as compared to OG dye from this adsorbent. pH analysis revealed that the removal of both dyes was higher at neutral conditions as compared to acidic and basic medium conditions of the OG and RhB dyes. It was 95 mg/g for OG dye and 105.6 mg/g in the case of RhB dye. Corn husk was selected as a biomass precursor for carbon materials because it was not previously utilized for the removal investigation of the cationic and anionic dye. The three different temperatures were used for thermodynamic analysis (303, 313, and 323 K) of both dyes. At all these temperatures, the adsorption was spontaneous and feasible with an exothermic adsorption nature for OG dye and an endothermic adsorption nature for RhB dye. Dose analysis for

CHMAC 2 Imp 7 days was checked using 10-50 mg adsorbent dose in 60 mL dye solution. The optimum dose, which was best for both dye removal, was 50 mg/60 mL dye solution within 120 minutes of adsorption reaction time. So, 0.8 g/L was the standard calculated and observed dose for the removal investigation of dye molecules from the wastewater. Four different kinetics were pseudo-first, second-order kinetics, with the Elovich and intraparticle diffusion model were analysed and concluded that pseudo-second order kinetics was best fitted for the elimination study of both dyes from the wastewater with chemisorption kinds of characteristics between adsorbent and adsorbate. Regeneration of the adsorbent CHMAC 2 Imp 7 days was done using HCl and an ethanolic-water mixture in the case of OG and RhB dye, respectively, revealing that it can be utilized up to seven times for the removal of OG and RhB dye above 80 per cent without losing the adsorbent adsorption loading ability. Langmuir adsorption was best fitted for both the dye removal with higher loading capacity and the monolayer nature of adsorption of the adsorbate on the adsorbent. $pH_{ZPC}$ analysis revealed that at neutral conditions, the removal rate of the dyes is higher. This adsorbent was observed to be best in the elimination of cationic and anionic dyes from wastewater. Overall, the use of the biomass corn husk was observed to be best in mesopore carbon formation using phosphoric acid as an impregnated material. This set of studies revealed that these can also be employed in the industrial field and can overcome the issue present in the wastewater due to the presence of colouring materials.

**Future Scope**

As the world population is increasing, the need for various colouring products for human and other purposes has also increased. Industry, basically in the textile field, making a large number of fabrics, fur, plastics, and other materials in colored form. These industries release day-to-day textile pollution into the surface runoff water. Due to the presence of these colouring materials, it is causing less growth of aquatic biota as well as decreasing the pleasant quality of this water medium. Mesoporous carbon with tunable pores and high specific surface area may become easily available, economical materials to sort out these dye materials from wastewater media. It was already discussed in the chapter with characteristic features of adsorbents for dye removal, which were anionic and cationic in form. Increasing the cyclisation use of adsorbent many times for these dye removal can enhance the motivation of researchers to focus on the use of meso-range carbon materials for dye removal studies from wastewater. One question arises: What will be next after the adsorption of dyes by these adsorbents? To solve this problem, various researchers are focusing on the use of spent adsorbent in construction materials, and desorbing these dyes through various approaches, dye material can be stored after water vaporization.

Spent adsorbent is a big query these days to be used in different fields for different purposes. It will be the future of solid waste management for these adsorbent materials. The goal of research should be that when we are working on something for environmental purposes, it should be free from a hazardous approach, making the environment polluted. There are various synthesised neutral dyes in the utilization, which should also be removed by functionalising the mesoporous carbon adsorbent via different non-hazardous chemicals. So overall conclusion of the thesis and future scope is that we have to make an adsorbent of well-defined porosity and high specific surface area from biomass utilization to observe a higher removal rate of dye pollution from wastewater with a higher number of regeneration cycles and higher loading capacity of the adsorbate on the surface adsorbent. These materials' future scope should be focused on the reuse and repurposing of the materials into an environmentally green approach, besides being discarded into the landfill openly.

Construction field use is one approach to using these spent adsorbents; we should focus it to be using them elsewhere for advantageous purposes. Biomass may be a valuable feedstock to synthesize these materials easily at a low cost without harming the environment. In future, these adsorbent materials can be applied for cationic as well as for anionic dye removal, and further modification of these adsorbents with a supportive adsorbent may enhance the adsorption loading capacity much higher. These adsorbents can also be applied in future for heavy metal, carbon dioxide capturing, and in supercapacitors after functionalization and can provide higher removal efficiency for other pollutants, so modification or functionalization of the adsorbent with metal organic framework, zeolites, and other porous materials can enhance its adsorptive and catalytic properties in future.