

## FT-IR Spectra, XRD and EDX Studies on the Adsorption of Methylene Blue Dye Present In Aqueous Solution onto Acid Activated Carbon Prepared From Mimosops Elengi Leaves

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**Abstract:** Adsorption studies were carried out by observing the effects of various experimental parameters removal of Methylene blue from aqueous solution. Morphology of adsorbent due to adsorption have been analyzed  $P^H$ , FT-IR spectra, EDX and XRD analysis. The data were fitted into the Langmuir and Freundlich adsorption isotherm equations. Thermodynamics parameters like change in free energy, enthalpy and entropy were calculated.

**Keywords:**  $P^H$ , FT-IR spectra, EDX and XRD analysis

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### I. INTRODUCTION

#### DYES POLLUTION

Dyes are complex and sensitive chemicals. A dye is a coloured substance that has an affinity to the substrate to which it is being applied. The dye is generally applied in an aqueous solution and may require a mordant to improve the fastness of the dye on the fiber. The Dyes are obtained from animals, vegetables, mineral origin, plants, roots, berries, bark, leaves and wood. Both dyes and pigments appear to be coloured because they absorb some wavelengths of light more than others. In contrast with a dye, a pigment generally is insoluble, and has no affinity for the substrate. But Dyes are soluble, some dyes can be precipitated with an inert salt to produce a lake pigment, and based on the salt used they could be aluminum lake, calcium lake or barium lake pigments [1].

Coloured wastes may contain chemicals which exhibit toxic effects towards microbial populations and can be toxic and/or carcinogenic to mammals. In general, dyes are poorly biodegradable. Conventional biological treatment processes are not very effective in dye removal [2]. Basic malachite green (MG) dye has been widely used for the dyeing of leather, wool, jute and silk, as in distilleries, as a fungicide and antiseptic in aquaculture industry to control fish parasites and disease [3]. Malachite green has properties that make it difficult to remove from aqueous solutions and also toxic to major microorganisms. Though, the use of this dye has been banned in several countries and not approved by US Food and Drug Administration, it is still being used in many parts of the world due to its low cost, easy availability and efficacy and to lack of a proper alternative [4]. Malachite green is environmentally persistent and acutely toxic to a wide range of aquatic and terrestrial animals. It is highly lethal to freshwater fish, in both acute and chronic exposures. It causes serious public health hazards and also poses potential environmental problem. Both clinical and experimental observations reported so far reveal that MG is a multi-organ toxin. It decreases food intake, growth and fertility rates; causes damage to the liver, spleen, kidney and heart; inflicts lesions on the skin, eyes, lungs and bones; and produces teratogenic effects. Malachite green is highly cytotoxic to mammalian cells.

#### Morphological study

Changes in the morphology of adsorbent due to adsorption have been analyzed with FT-IR spectra, EDX analysis and XRD patterns.

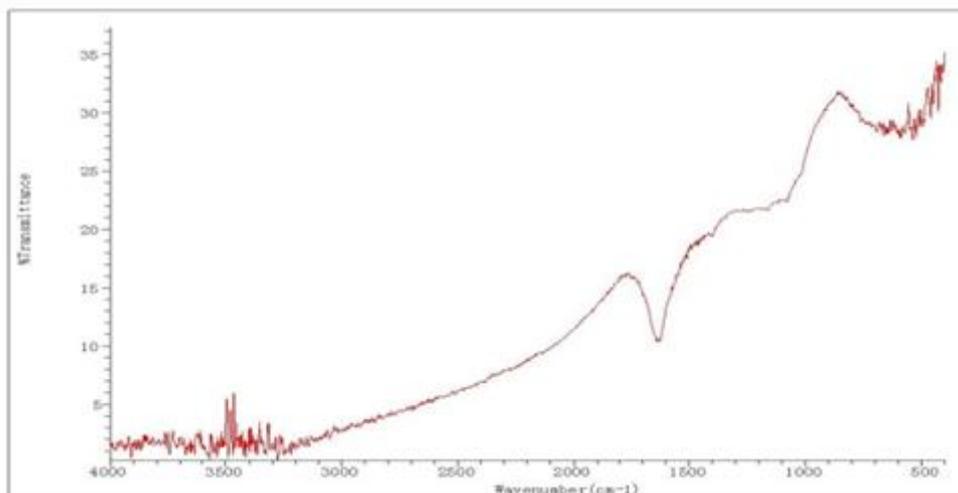
#### Fourier Transform Infrared Spectroscopy (FT-IR)

The instrumental evolution of the day makes non-destructive and quantitative and qualitative analysis possible, with significant accuracy and precision. Historically IR has been mostly used for qualitative analysis, to

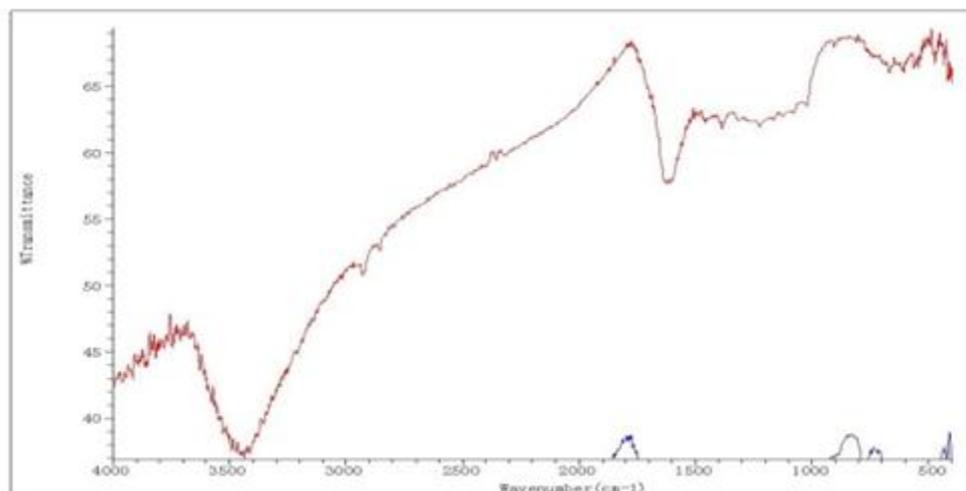
obtain structural information. The shift of the bands and the changes in signal intensity allow the identification of the functional groups involved in adsorption [5 – 7].

#### **FT - IR for PTMAC and MB dye loaded onto PTMAC**

The FT – IR spectrums of PTMAC and PTMAC loaded with MB dye were shown in figure 6.80, found except new peak at  $3450\text{ cm}^{-1}$  and  $1600\text{ cm}^{-1}$  in the spectrum of PTMAC loaded with MB dye. This may be due to chemical bond formation. The above examination infers that the major portion of adsorption took place with physical adsorption and a small portion with chemisorption.



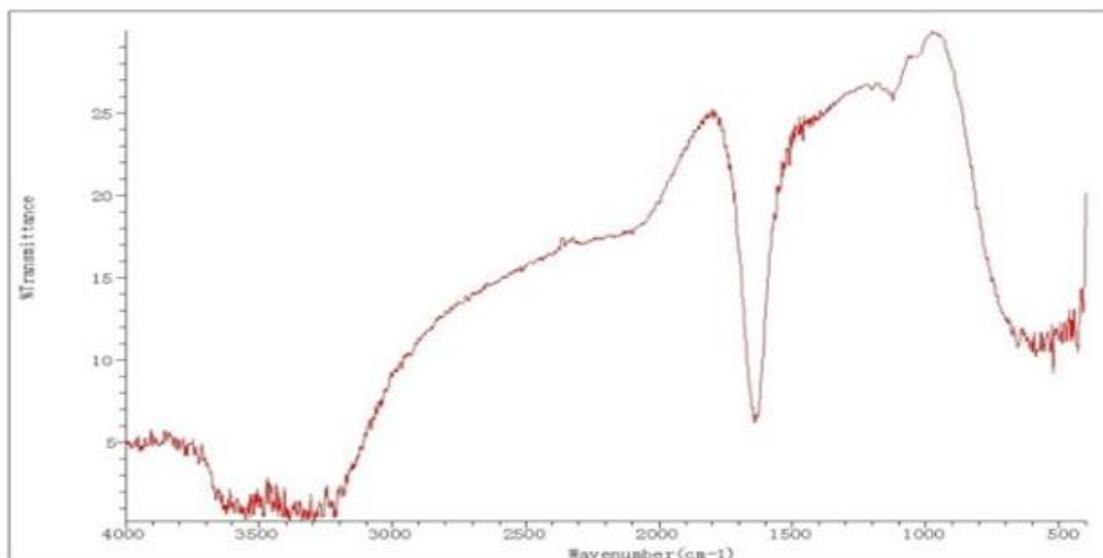
**Figure.1.(a) Before adsorption of MB dye onto PTMAC**



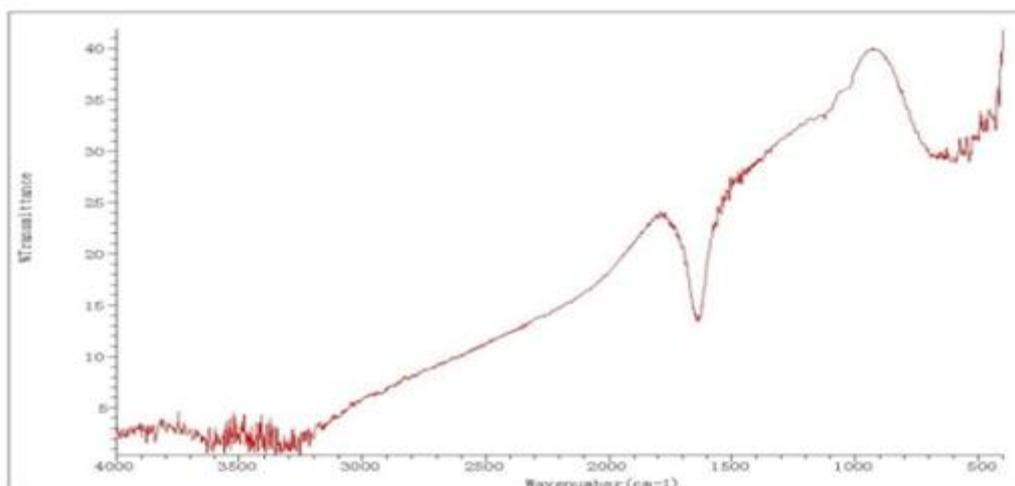
**Figure 1. (b) After adsorption of MB dye onto PTMAC**

#### **FT - IR for PTMAC and MB dye loaded onto STMAC**

The FT – IR spectrum of STMAC and the STMAC loaded with MB dye were shown in Figure 6.83. It could be seen from the spectra that almost there is no change in the spectral pattern before and after adsorption. This infers that the possibility of adsorption of MB dye onto STMAC adsorbent is by physical forces.



**Figure 2.2. (a) Before adsorption of MB dye onto STMAC**



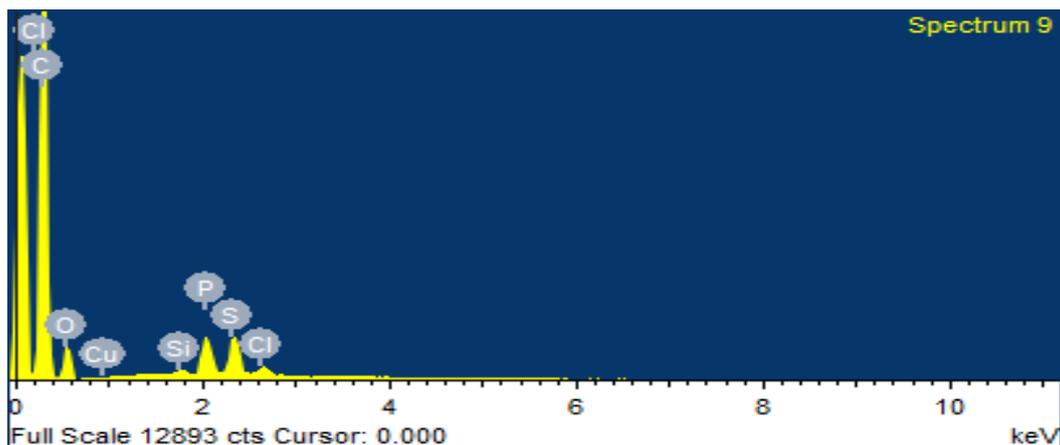
**Figure 3.(b) After adsorption of MB dye onto STMAC**

### **Energy Dispersive X-ray Spectroscopy (EDX)**

Energy Dispersive X-ray Spectroscopy (EDX) is an analytical capability that can be coupled with several applications including Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Scanning Transmission Electron Microscopy (STEM). EDX, when combined with these imaging tools, can provide elemental analysis on areas as small as nanometers in diameter. The impact of the electron beam on the sample produces x-rays that are characteristic of the elements present on the sample. EDX Analysis can be used to determine the elemental composition of individual points or to map out the lateral distribution of elements from the imaged area [8 – 9].

### **EDX studies for MB dye ion loaded Ptmac**

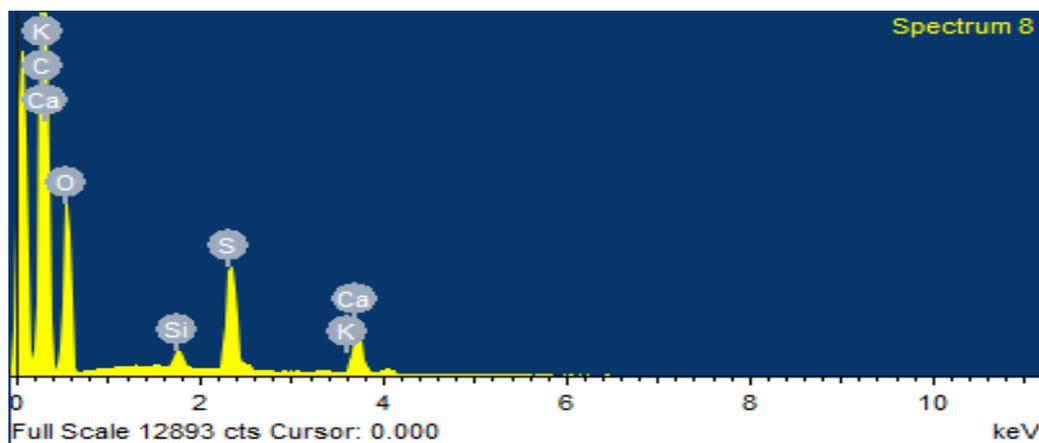
EDX analysis of MB dye loaded PTMAC were shown in Figure 6.86. The well- defined peak of adsorbent loaded with MB dye shows that MB dye were bound onto the PTMAC surface. Peaks of certain MB dye are very small which infers the surface adsorption through Vander Walls force.



**Figure: 3. (a) EDX studies for MB dye ion loaded PTMAC**

#### **EDX studies for MB dye ion loaded STMAC**

EDX analysis of MB dye loaded STMAC were shown in Figure 6.89. The well -defined peak of adsorbent loaded with MB dye shows that MB dye were bound onto the STMAC surface. Peaks of certain MB dye are very small which infers the surface adsorption through Vander Walls force.



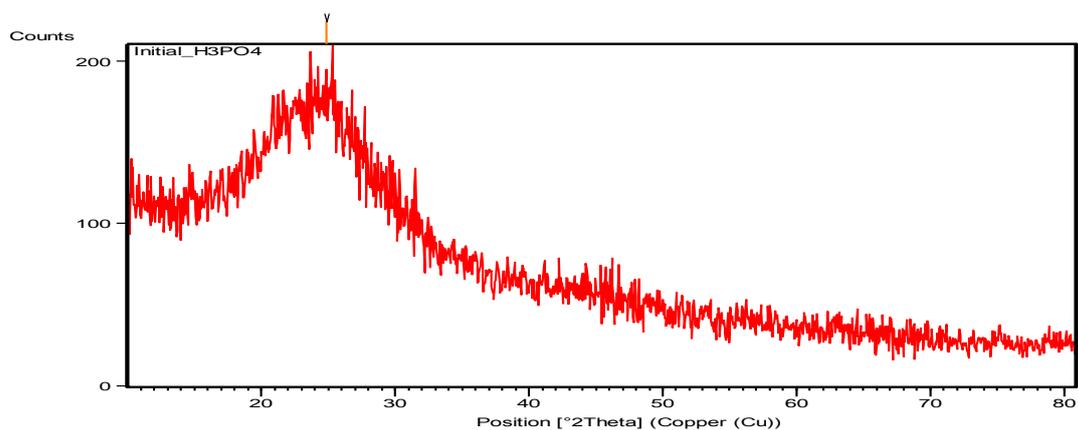
**Figure: 3. (b) EDX analysis of MB dye loaded STMAC**

#### **X ray diffraction studies**

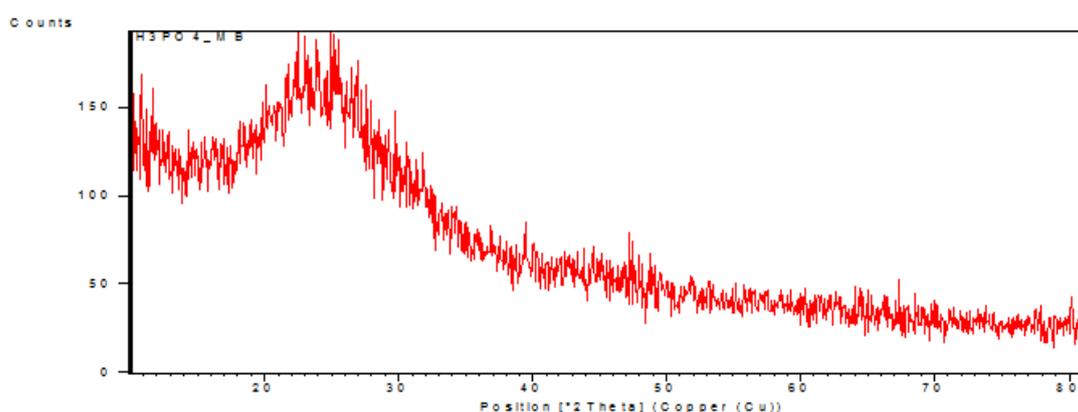
XRD studies show changes in the crystallinity of the adsorbent well defined peaks show the crystalline nature and the hallow peak shows the non- crystalline amorphous nature of the carbon [10 – 12].

#### **XRD pattern of MB dye loaded PTMAC**

The XRD pattern of PTMAC and after adsorption of MB dye is shown in Figure 6.94 & 6.95. The intense peak shows the presence of highly organized crystalline structure of raw activated carbon after the adsorption of metal ions the intensity of some peaks are diminished appreciably in the PTMAC loaded with MB dye. The peak between 20 and 30,  $2\theta$  values became shallow. This has been attributed to the adsorption of MB dye on the upper layer of the crystalline structure of the activated carbon surface.



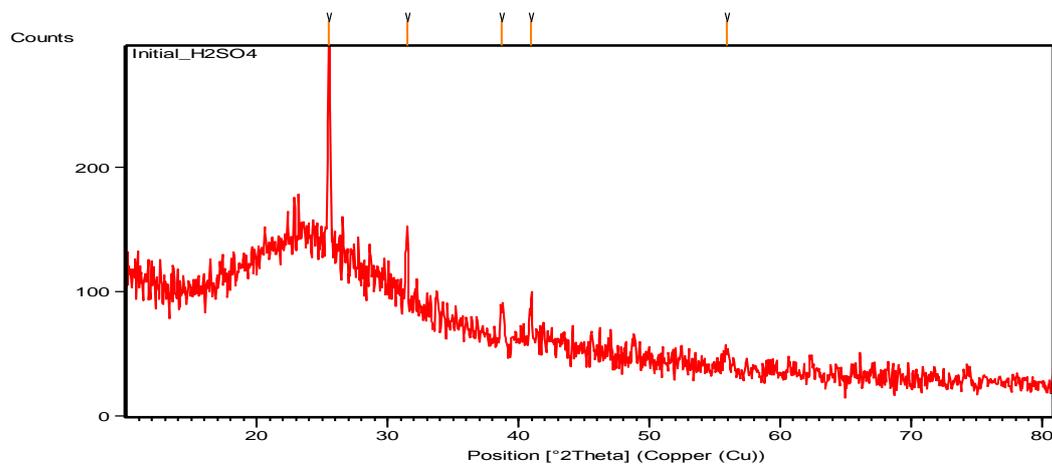
**Figure: 4 (a) PTMAC Before Adsorption**



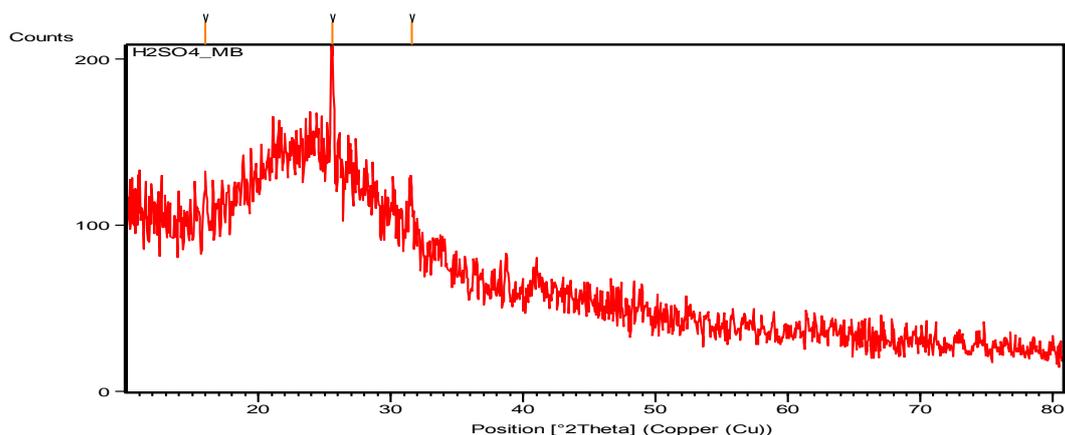
**Figure: 4. (b) After Adsorption of MB dyes onto PTMAC**

#### **XRD pattern of MB dye loaded STMAC**

The XRD pattern of STMAC and after adsorption of MB dye is shown in Figure 6.00 & 6.01. The intense peak shows the presence of highly organized crystalline structure of raw activated carbon after the adsorption of metal ions the intensity of some peaks are diminished appreciably in the STMAC loaded with MB dye. The peak between 20 and 30, 2θ values became shallow. This has been attributed to the adsorption of MB dye on the upper layer of the crystalline structure of the activated carbon surface.

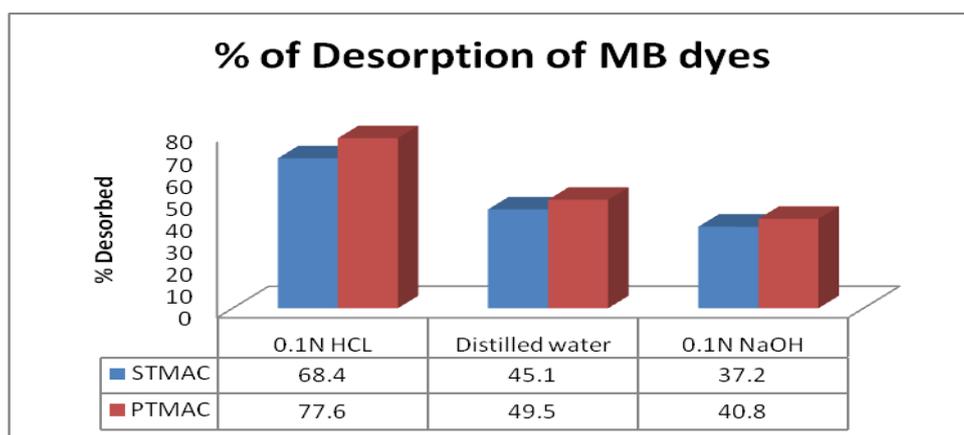


**Figure: 5. (a) STMAC Before Adsorption**



**Figure: 5. (a) After Adsorption of MB dyes onto STMAC**

Desorption study results pertaining to MB dye are presented as bar diagram in Figure 6.04. The effects of various reagents used for desorption studies indicate that hydrochloric acid is a better reagent for desorption, because more than 77% of adsorbed dye were removed from PTMAC and 68 % dye from STMAC. This indicates that the dyes were adsorbed on both the carbons, PTMAC and STMAC through physisorption mechanisms. <sup>6</sup>



**Figure: 6. Desorption of MB dye from PTMAC and STMAC**

## II. Conclusion

Studies inferred that pH 12.0 was the effective desorbing reagent for hydrochloric acid solution (0.1 N) MB dye. These result also favoured the physisorption mechanism. The FT-IR spectrums of PTMAC and STMAC unloaded and loaded adsorption of MB dyes indicated that the adsorption were physisorption.

The X-ray diffraction pictures of PTMAC and STMAC recorded unloaded and loaded adsorption of MB dyes indicate that the adsorption was physisorption.

The foregoing study demonstrated the evaluation of the low cost adsorbents prepared from the *Memsopselengi* leaves for the removal of MB dye from aqueous solution. Regeneration studies revealed the reuse of the adsorbates and the spent adsorbent. The experimental results obtained by batch experiments can be useful for the water engineers and environmental technologists in designing experimental set up while employing the adsorbate/adsorbent system studied. Yet additional research is needed to determine the useful capabilities and specific applications of these adsorbents with respect to other heavy metals, dyes and various real industrial waste water.

## Reference

- [1]. Gleeson, Tom, Wada, Yoshihito, Bier kens, Marc F. P, van Beek, and Ludovicus P. H , Nature, 488 (7410), (9 august 2012), 197–200.
- [2]. [http://www.wikiwand.com/en/Water\\_pollution](http://www.wikiwand.com/en/Water_pollution).
- [3]. Contaminants may include organic and inorganic, [www.coursehero.com/file/p2jjv35/Contaminants-may-include-organic-and-inorganic-substances-Organic-water/](http://www.coursehero.com/file/p2jjv35/Contaminants-may-include-organic-and-inorganic-substances-Organic-water/).
- [4]. Vijayaraghavan K and Yun Y S, Bacterial biosorbents and biosorption, *j.biotechadv*, (26) 3, (2008may-jun).

- [5]. Tarun Kumar Naiya<sup>1</sup>, BiswajitSingha<sup>2</sup> and Sudip Kumar Das<sup>2</sup>, FTIR Study for the Cr(VI) Removal from Aqueous Solution Using Rice Waste, International Conference on Chemistry and Chemical Process IPCBEE vol.10 (2011).
- [6]. Woranan Nakbanpotea<sup>1</sup>, Bernard A, Goodman<sup>b</sup> and PaitipThiravetyan ,Copper adsorption on rice husk derived materials studied by EPR and FTIR, Colloids and Surfaces A,Physicochemical and Engineering Aspects, Volume 304, Issues 1–3, 1 (September 2007), Pages 7-13.
- [7]. Ioannou Z<sup>1</sup>andSimitzis J, Adsorption of methylene blue dye onto activatedcarbons based on agricultural by-products, equilibrium and kinetic studies,Water Sci Technol. (2013);67(8):1688-94.
- [8]. S. Ghorbani-Khosrowshahi<sup>1</sup> and M. A. Behnajady<sup>1</sup>, Chromium(VI) adsorption from aqueous solution by prepared biochar from Onopordom Heteracanthom, Int. J. Environ Sci. Technol. (2016) 13, 1803–1814.
- [9]. HimaniMedhi and Krishna G. Bhattacharyya , Kinetic and mechanistic studies on adsorption of Cu(II) in aqueous medium onto montmorillonite K10 and its modified derivative,NewJournal of Chemistry, Issue 22, (2017).
- [10]. ReyadAwwadShawabkeh, Adsorption of chromium ions from aqueous solution by using activated carbo-aluminosilicate material from oil shale, Journal ofColloid and Interface Science 299 (2006) 530–536.
- [11]. EleonoraSoco and Jan Kalembkiewicz, Removal of Copper (II) and Zinc (II) Ions from Aqueous Solution by Chemical Treatment of Coal Fly Ash, Croat. Chem. Acta(2015), 88(3), 267–279.
- [12]. Arivoli, S, Hema, M, Parthasarathy, S. andManju, J. ChemPharm. Res., (2010), 2(5), 626-641.

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