

## PREPARATION AND CHARACTERISATION OF ACTIVATED CARBON FROM E-WASTE AND ITS APPLICATION ON DYE DECOLOURIZATION

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### ABSTRACT

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Electronic Wastes (E-Wastes) predominantly consists of Printed Circuit Boards which contains both metallic and non-metallic fractions (NMFs). While the metallic fractions are extracted and recycled, the NMFs are disposed of which are a major source of pollutant to the environment. Thus, it becomes necessary to find novel methods to effectively use this waste. In the current study, it focuses on the conversion of the NMFs into the activated carbon by physical and chemical methods. NMFs are subjected to pyrolysis from 300-700°C to find the optimum temperature for maximum adsorption. Potassium hydroxide was impregnated with the char powder and activated at optimum temperature (600°C). The prepared activated carbon was characterized by FTIR, SEM and XRD. The Methylene blue dye was used as an adsorbent in this study as it imparts intense coloration. The percentage removal of 10 mg/ml adsorbate is 98% at an optimum contact time of 300 minutes. BET analysis to study the porosity and the surface area was found to be 225.7 m<sup>2</sup>/g. Adsorption isotherms such as Langmuir & Freundlich were plotted and the R<sup>2</sup> value was found 0.996 and significant.

### INTRODUCTION

E-Waste is the fastest growing waste stream, increasing at a disquieting rate which causes an irreversible impact on the environment if proper methods are not adopted for the disposal of these wastes (Hadi et al., 2013). The production of E-wastes has increased due to innovations and up-gradation of technology, an increase in the population in the urban sector, intense marketing of electrical and electronic equipment and their replacements. The total E-waste generated worldwide was estimated to be 41.8 million metric tonnes in the year 2016 and is expected to reach 52 million metric tonnes by 2020 (Guo et al., 2008). The global growth reported for E-waste accounts for about 3.5% per year (Kumar et al., 2018). Due to the rapid increase in the production of these E-wastes, a novel and an economic method must be adopted in order to eliminate the major social hazards, health issues e.g. increased blood lead levels, stillbirths, premature births, reduction in birth weight and environmental threat like the accumulation of heavy metals in *Oryza sativa L.* (Fu et al., 2008 and Marques et al., 2013). The recycling of

Printed Circuit Boards (PCB), a composite material, is of major concern as they are the fundamental component of the electrical and electronic industry accounting to about 3% wt of the whole of E-waste which contains metals e.g. Cu, Al, Fe, Sn, Sb, Pb and non-metals e.g. the Thermo-setting resins, reinforcing materials and brominated flame retardants (Marques et al., 2013; Eswaraiahm et al., 2008; Li et al., 2008; Birloaga et al., 2013; Yi-hu et al., 2013). Problems in the recycling of PCBs are faced due to their complex physical and chemical characteristics (Guo et al., 2009; Li et al., 2014). Metallic fraction and non-metallic fractions are the two classifications of PCBs based on their components (Yi-Hu et al., 2013; Guo et al., 2009). By the mechanical separation method, the metallic fractions of the PCBs are recycled and metals recovered are used while a large amount of NMF is left behind (Hadi et al., 2013).

The NMF fractions are however recycled by the process of incineration which leads to the formation of the highly toxic compounds e.g. polybrominated dibenzodioxins and polybrominated dibenzofurans while leaching of the groundwater occurs through

landfilling by the brominated flame retardants (Kan et al., 2015; Zheng et al., 2009; Guo et al., 2009). The NMF that are recycled by physical methods finds its application as filler in construction materials, adhesive, insulating material. Other chemical methods such as gasification, depolymerization with a supercritical fluid, pyrolysis, and hydrogenolysis methods are adapted to recycle the NMF (Khodale et al., 2013). Pyrolysis of NMF lead to the formation of chars, oil, gas and hence can be used as a source of fuel and chemical feedstock (Hadi et al., 2013). In order to obtain an economically feasible and a cost-effective source the NMF are converted into activated carbon and chemical activation can be achieved by treating the activated NMF with  $H_3PO_3$ ,  $ZnCl_2$ ,  $H_3PO_4$  and KOH (Budinova et al., 2006).

Carbonaceous materials are the major source for preparation of activated carbon by subjecting it to different conditions. Physical and chemical activation are the main parameters to activate the compounds. The physical activation of NMFs was subjected to high temperatures ranging from 300°C to 700°C. Chemical activation is achieved by treating the activated carbon with KOH and the same is used to treat the industrial effluent containing the textile dye as these textile dye contains surfactants, dissolved solid and certain heavy metal e.g. Cr, Ni and Cu in high quantities (Kannan and Sundaram., 2001; Al-Qodah and Shawabkah., 2008; Min and Hui 1987; Coia-Ahlman and Groff., 1991). Methylene blue is a monovalent, cationic, basic dye with molecular weight 373.9. Methylene blue is predominantly used in silk, paper, leather, cotton industries and the discharge of the same into the environment results threat for toxicological and aesthetical reasons for the light penetration and disturbs the food chain significantly (Wang et al., 2005; Shendrik et al., 1989; Cooper 1985; Nowak 1989). Thus, it is more than customary to remove the Methylene blue from the textile effluent before the toxicity level increases to a greater extent. Physical, chemical, biological methods are used to remove the Methylene blue concentration among which adsorption is accounted as the most effective method (Ke et al., 2013; Bozdogan and Goknil., 1987; Brower and Reed., 1985; Grau 1991). An advantage of adsorption over the other methods is that it is cost-effective and exhibits a stable effect. Therefore, this study concerns the adsorption of the methylene blue dye from the industrial effluent by means of activated carbon obtained from the NMF of the PCBs (kumaar et al., 2005).

This study was carried out using charcoal as control to compare its adsorption capacity with the activated carbon produced from NMFs. The adsorption of methylene blue was studied by varying the contact time and the concentration of the dye. In addition,

elemental analysis such as XRD, EDS, CHNS, FESEM, FTIR and BET were done. Freundlich and Langmuir adsorption isotherm was plotted to study the adsorption kinetics

## MATERIALS AND METHODS

The non-metallic fractions (NMFs) of waste printed circuit boards (PCBs) were provided by Victory Recovery and Recycle Technologies Pvt Ltd., Thiruvallur, Tamilnadu, India after separating the metallic contents.

### Pyrolysis

Fine powder of NMFs and charcoal were activated at temperatures ranging from 300°C -700°C for 3 hours in a muffle furnace (Kan et al., 2015). Activated products were impregnated with solid KOH (1:1 w/w) for 1 hour at 100°C. Acid wash was done using 1 N HCl to dissolve the excess ash content present in the impregnated samples (Yi-Hu et al., 2013). The dissolved samples were further washed with distilled water until the pH dropped to 7. After washing, the samples were air dried overnight at room temperature and was stored to carry out dye and effluent adsorption studies.

### Characterization of activated Nmf's

Various analytical measurements were done to analyze the physical, chemical and topographical information of the activated NMF samples. Elemental analysis of carbon-hydrogen-nitrogen- sulphur (CHNS) measurements was estimated by classical dumas method (Ke et al., 2013). Energy dispersive spectroscopy (EDS) was done to further characterize the chemical composition of the NMF sample and the adsorption capacity of silica, clay, and epoxy was determined (Flandinet et al., 2012). Topographical and structural information was observed by Field emission scanning electron microscopy (FESEM) (Flandinet et al., 2012). The specific surface area of the NMF samples was evaluated by Brunauer-Emmett Teller (BET) analysis. Fourier- transform infrared spectroscopy (FTIR) analysis was done to identify the functional groups present in the activated NMFs (Yuan et al., 2011). The amorphous nature of the activated NMF was confirmed by X-ray diffraction analysis (Salleh et al., 2011).

## RESULTS AND DISCUSSION

### CHNS elemental composition

CHNS elemental analysis of the raw NMFs and the activated NMFs (Table 1) indicated that the precursor comprised around 56% carbon while the remaining amount of it is burnt off after the activation process. This result can be justified by the catalysing effect of KOH stated by some other authors for similar

reactions (Ke et al., 2013).

#### Fourier-Transform infrared spectroscopy (FTIR)

Infrared spectroscopy has been used to obtain information about the surface chemical structure of the activated material. Fig. 1, compares the FTIR spectra of the raw and the activated NMF, and illustrates the change of the surface chemistry of the precursor due to the activation process (Table 2).

#### BET analysis

Brunauer Emmett Teller (BET) surface area indicates the total adsorptive area available for adsorption of gas molecules. Raw NMF was found to have a surface area of 1.5 m<sup>2</sup>/g which upon activated increased to around 225 m<sup>2</sup>/g. The activated NMF has comparatively a large Surface area which could possibly account for the decolorization process. This

**Table 1.** Composition of carbon, nitrogen, oxygen and sulphur content of the NMF and charcoal.

Sample	C (%)	H (%)	N (%)	S (%)
Raw NMF	56.284 ± 0.343	4.658 ± 0.231	1.0015 ± 0.095	0.001
Activated NMF	52.130 ± 0.579	2.120 ± 0.926	1.657 ± 0.028	0.000
Charcoal (Control)	85.222 ± 0.138	4.834 ± 0.622	0.694 ± 0.053	0.011

**Table 2.** FTIR analysis of functional groups present in NMFs.

Frequency (per cm)	Functional group
3769	Amide N-H stretch
3644	Amide N-H stretch
2321	Nitrile C=N stretch
1757	Aromatic C=C bending
711	Aromatic C=H bending

result is in accordance with the works published by Hadi et al., 2013 (Table 3).

#### EDS-FESEM

**SEM micrographs:** The SEM images of Raw NMF and activated NMF at 600°C were obtained to draw a comparative study of surface morphology between the two. The Raw NMF contained needle like particles which upon activation has appeared to be well disintegrated (Fig. 2).

**Energy dispersive spectroscopy:** Different patterns depicting the elements present in the NMF. EDS represents the elemental composition of the NMF. The carbon content was found to be around 59.46% which can be regarded as an indicator for the process of adsorption (Fig. 3).

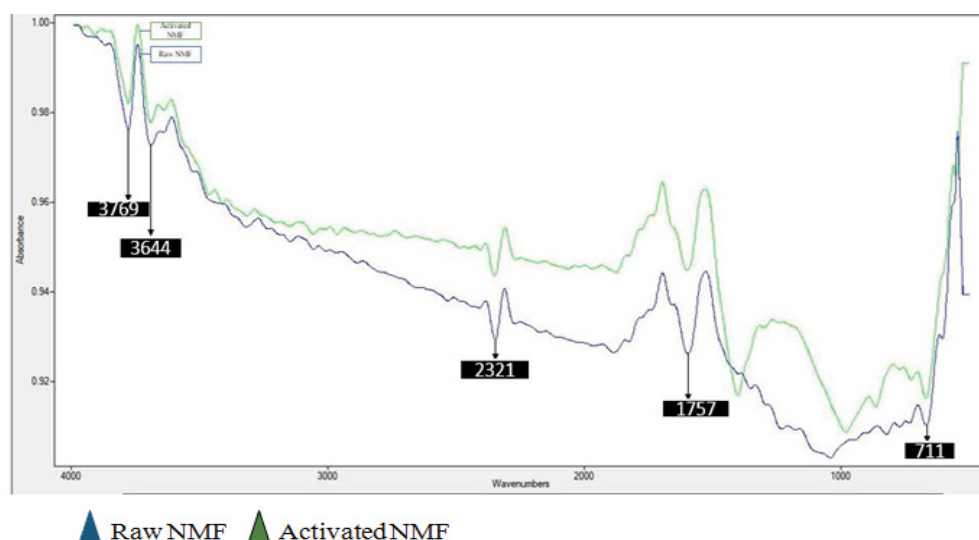
**X-ray diffraction (XRD):** Fig. 4 shows the XRD pattern of raw NMF, activated NMF and KOH impregnated NMF. In comparison with the XRD pattern of raw NMF, the peak of activated NMF is broad and the peak of impregnated NMF almost disappears. The results suggest that the graphite micro particles be etched at different extends after physical or chemical activation. Impregnated NMF has a lower content of metallic residues than activated NMF indicating that washing with HCl after chemical activation can effectively remove residues (Fig. 4).

**Adsorption studies:** Absorbance of methylene blue was studied using different concentration at wavelength of 664 nm (Table 4).

#### Decolorization studies

Decolorization of methylene blue at different stages (0.5 g/ml) by activated NMF (1 g/ml) with the time interval of 24 hrs (Fig. 5).

(a) Methylene Blue dye solution.



**Fig. 1** FTIR spectra of raw NMF and activated NMF.

**Table 3.** BET analysis of activated carbon.

Sample	m <sup>2</sup> /g
Raw NMF	1.5
Activated NMF (600°C)	225.731
Charcoal (600°C)	3792

**Table 4.** Absorbance of methylene blue at 664 nm.

Concentration (mg/ml)	Absorbance (at 664nm)
1	0.284
2	0.458
3	0.638
4	0.886
5	0.978

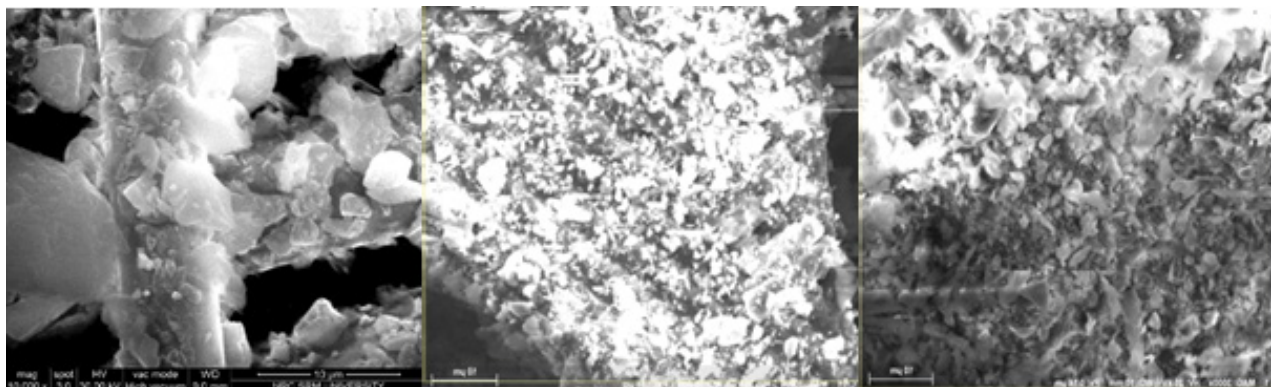
(b) Addition of activated NMF (600°C) to methylene blue solution.

(c) Partial decolorization of methylene blue solution after 24 hrs.

(d) Complete decolorization of methylene blue solution after 48 hrs.

(e) Complete decolorization of methylene blue due to the action of activated NMF (600°C).

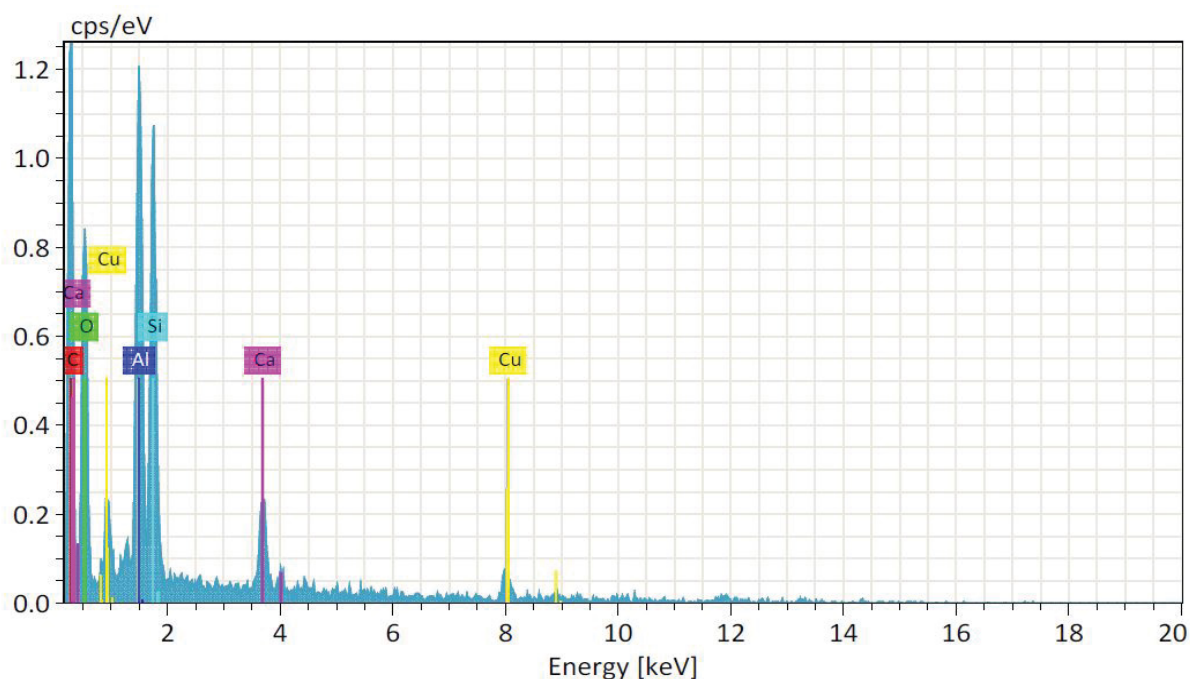
**Varying concentration of Methylene Blue:** The concentration of Methylene Blue dye was varied from 10-50 mg/L and a constant amount (5 g) of activated NMF and Charcoal (control) were used for adsorption studies. The maximum adsorption



(a)

(b)

(c)

**Fig. 2** SEM images of (a) Raw NMF, (b) Activated NMF and (c) Charcoal.**Fig. 3** Elemental composition of activated NMFs at 600°C.

occurred for the concentration of dye at 10 mg/L by the NMF (98.95%), while for the same concentration adsorption by charcoal was 97%. It is evident from the graph that on increasing the concentration of methylene blue, the percentage removal reduces gradually. It is also clear that the percentage removal of the dye by the activated NMF is higher than that of activated charcoal (Fig. 6).

**Varying contact time:** Methylene Blue adsorption studies were carried out with varying time intervals (60-420 min) until the adsorption rate almost became constant. It can be inferred from the graph (Fig. 7) that the percentage removal of the dye increased gradually until 300 minutes after which it became constant. The maximum percentage removal was 98% by the NMF and occurred at the 300<sup>th</sup> minute (Fig. 7).

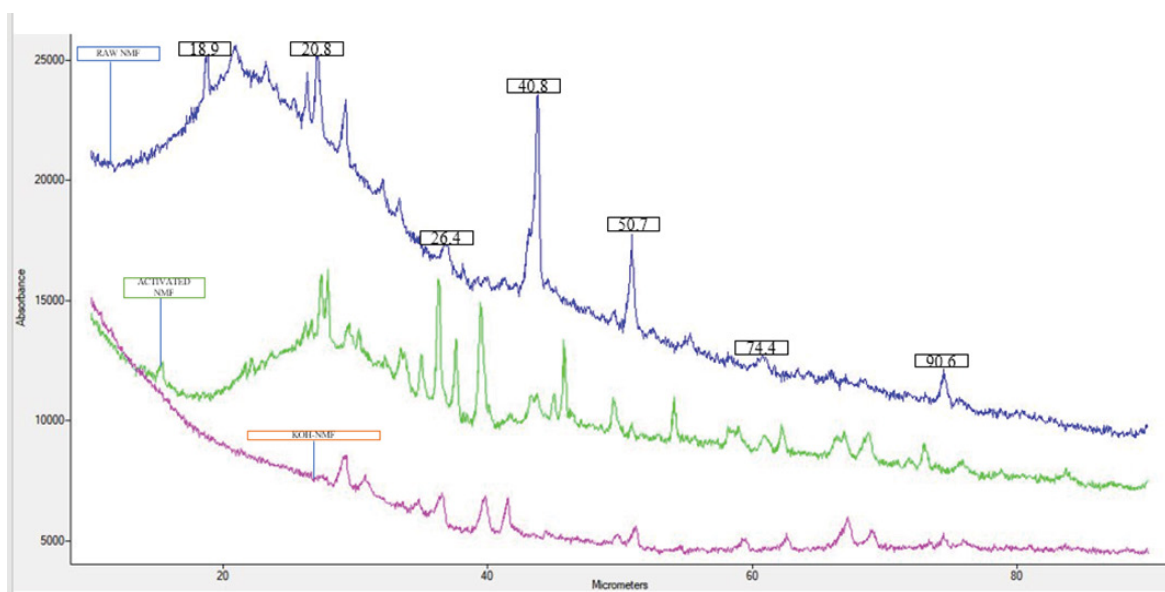


Fig. 4 XRD peaks for raw NMF, activated NMF and impregnated NMF (KOH).

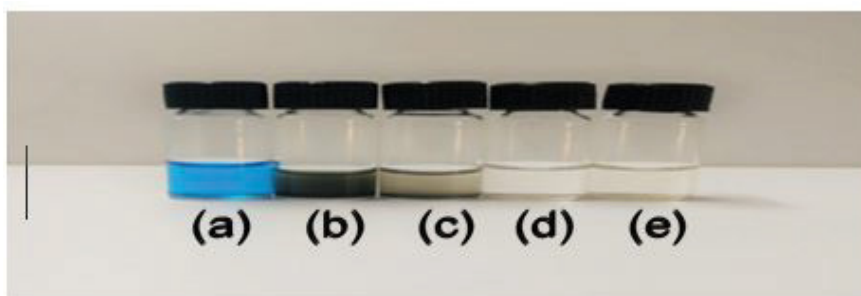


Fig. 5 Stages of methylene blue (0.5 g/ml) decolorization by activated NMF (1 g/ml) with the time interval of 24 hrs.

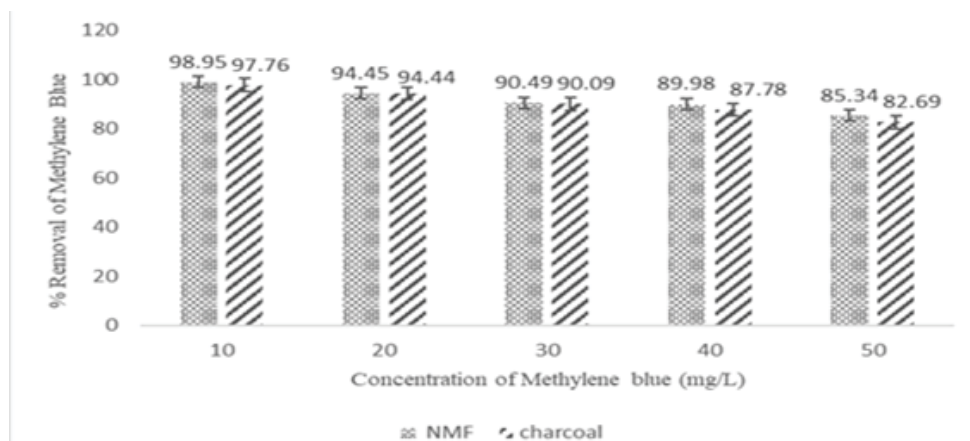


Fig. 6 Percentage removal of methylene blue (%) vs. concentration of methylene blue (mg/L).



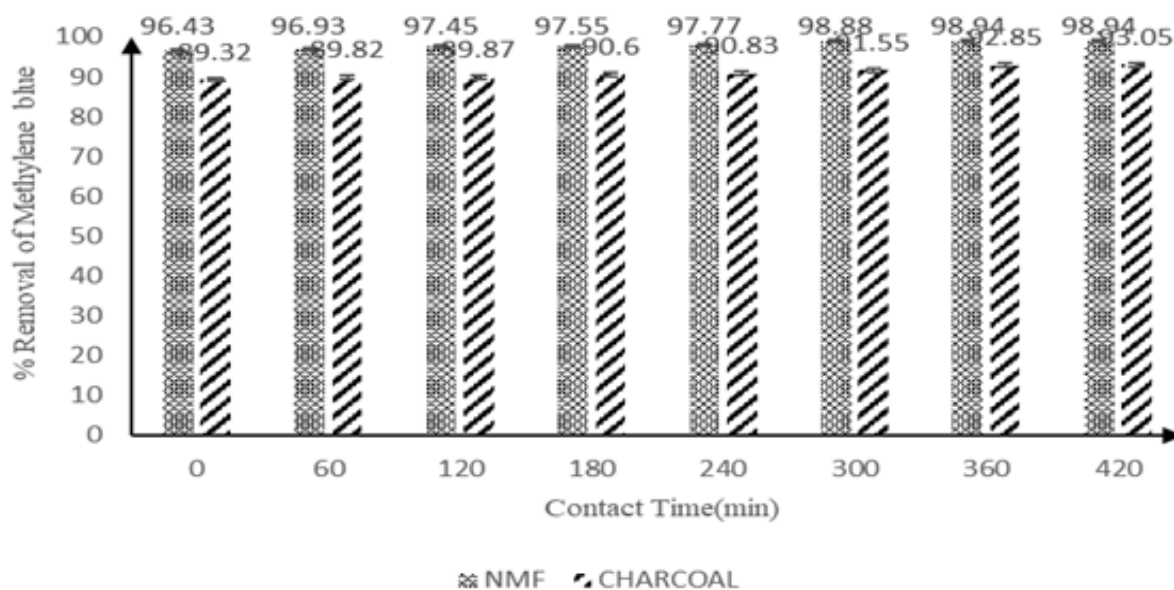


Fig. 7 Percentage removal of methylene blue vs. contact time.

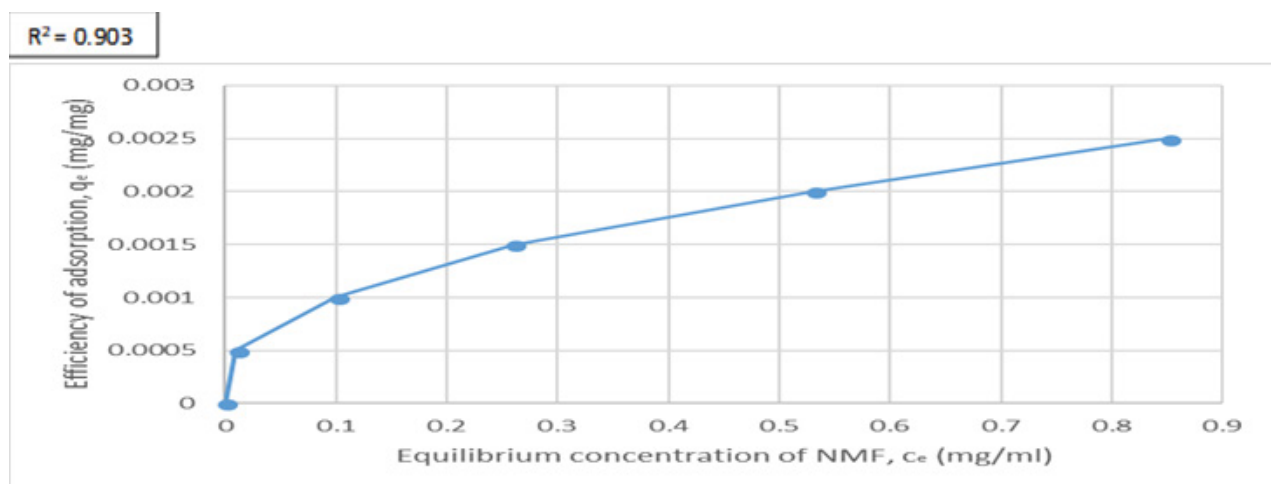


Fig. 8 Freundlich model for adsorption of methylene blue dye by the NMF with a time interval of 1- hour.

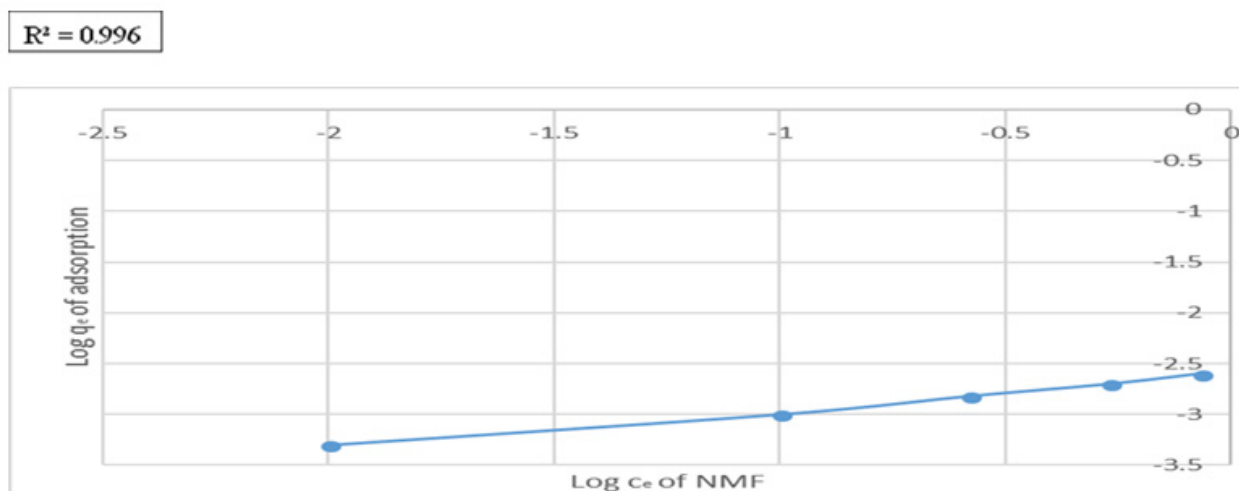


Fig. 9 Langmuir model for adsorption of methylene blue dye by the NMF with a time interval of 1-hour.

### Adsorption isotherms

Freundlich model and Langmuir model for adsorption of methylene blue dye by the NMF with a time interval of 1- hour as shown in Fig. 8 and Fig. 9.

### CONCLUSION

Based on the results obtained within the framework of this research work, it seems that NMFs of printed circuit boards may constitute a suitable precursor for the production of an activated material through activation with potassium hydroxide to be used as a potent adsorbent for the removal of methylene blue dye from industrial effluents. One of the commercially used adsorbent- charcoal has been used for comparison purposes in the same test conditions. The results showed that the adsorption capacity of the novel material increased with increasing concentrations of adsorbent used and considerably reduced with increasing concentrations of dye incorporated. The results also indicate that the NMFs have a better adsorbing efficiency than the commercially used charcoal. High surface area-225 m<sup>2</sup>/g was obtained for the KOH impregnated NMF sample which is indicative of the high adsorptive efficiency of the material. From the above results, it can be concluded that the NMFs of E-Waste PCBs can be chemically activated with KOH to increase the adsorption capacity of the same and this material can be considered as a potential adsorbent for removing dyes and toxic substances from industrial effluents.

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