Rice Husk-derived Activated Carbon as an Eco-Friendly Adsorbent for Water Purification

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ABSTRACT

Industrial wastewater discharge is one of the most important causes of water pollution. Adsorption using activated carbon is commonly used to remove these pollutants. However, commercial activated carbon is mostly derived from non-renewable resources such as coal and production is expensive. This project aims to produce activated carbon from an eco-friendly precursor, rice husks, and compare the properties of activated carbon obtained from different methods of activation with commercial activated carbon. The activated carbons were successfully synthesized with physical and chemical activation with zinc chloride. A novel method of chemical activation was developed by replacing water (conventional solvent) with ethanol. The activated carbon obtained was characterised by SEM and EDS. Iodine number test was also conducted to evaluate the porosity of the carbon synthesised. Adsorption studies were carried out to investigate the percentage removal of Methylene blue, iron(III) ions and ibuprofen by the activated carbons synthesized. The effect of initial concentration of pollutant solution on adsorption was investigated by varying the concentration of methylene blue from 100-400ppm and iron(III) ion from 75-300ppm. These studies revealed that all the activated carbon synthesized from rice husk have higher percentage removal of iron(III) ions than commercially activated carbon. Among the activated carbons synthesised, chemically activated carbon using ethanol as the solvent has the greatest percentage removal of all three pollutants and maximum adsorption capacity for methylene blue and iron(III) ions, hence showing great promise to be used in wastewater to remove methylene blue, iron(III) ions and ibuprofen from industrial effluent.

1. Introduction

1.1 Literature review

Industrial wastewater discharge is one of the most important causes of water pollution which have many negative effects on the ecosystem and the human's life. There are many types of industrial wastewater based on different industries and contaminants. A wide variety of pollutants are found in these wastewaters, ranging from organic chemicals such as dyes and pharmaceutical waste to inorganic pollutants such as metal ions. Specifically, heavy metals are discharged by industries such as agrochemical, petrochemical, and fertilizer, whereas dyes are principally found

in effluents from dye manufacturing industries, electroplating factories, distilleries, and food companies (Mohammedia et al., 2014).

Dyes in water are highly visible and undesirable (Crini, 2006). Colour affects the nature of the water and inhibits the penetration of sunlight into the receiving water bodies, which has a deleterious effect on photosynthesis and thus aquatic life (Arami et al., 2005). Dyes such as azo dyes are also found to be carcinogenic and mutagenic (Patil & Shrivastava, 2010). On the other hand, heavy metal ions are toxic to living organisms and are non-biodegradable. Metal ions such as iron(III) ions cause water to have an unpleasant metallic taste and promotes undesirable bacterial growth within a water works and distribution system, resulting in the deposition of a slimy coating on the piping (WHO, 1996).

Drug residue is an emerging pollutant. These pharmaceuticals comprise one of the few groups of chemicals that are specifically designed to act on living cells, which presents a special risk when they enter, persist and are dispersed into the environment. Ibuprofen is a nonsteroidal anti-inflammatory drug which is widely used in the treatment of rheumatic disorders, pain and fever (Mestre, Pires, Nogueira, & Carvalho, 2007). Due to relatively low metabolism and absorption in the human body, most of the administered ibuprofen is excreted via feces and urine in the form of its parent compound or water-soluble metabolites and consequently released into the environment (Buser, Poiger, & Muller,1999).

Wastewater treatment methods aim to remove contaminants such as metal ions and dyes from water, with the adsorption technique gaining popularity due to a wide range of adsorbents available. Activated carbons (AC) are effective adsorbents to remove pollutants as they are very porous and have a high surface area. Carboxylic, lactonic, phenolic, aldehydic, and other organic functional groups found at the edges of the hexagonal carbon layer planes are responsible for surface reactivity of activated carbon (Momčilović et al., 2011). Thus this makes them effective adsorbents, but current preparations of activated carbon are limited by the high costs (Anirudhan & Sreekumari, 2011). Therefore, there is a need to lower the production costs of activated carbon through the usage of biowastes (Momčilović et al., 2011).

One such waste material is rice husk, an undesirable by-product of the rice milling industry. Rice production all around the world generates abundant rice husks as agricultural waste, which are usually burned or discarded, which is unfavourable to the environment. The estimated annual rice production is 500 million tonnes, and much of this amount is not utilized (Wahab, Nemr, Sikaily & Khaled, 2005). Rice husks can be processed and transformed into AC with good adsorption properties. This would solve the environmental problems of disposal of rice husks,

while producing AC for wastewater treatment. Furthermore, AC derived from rice husk can be produced at lower costs as rice husks are readily available and cheap.

Rice husk contains 32.24% cellulose, 21.34% hemicellulose, 21.44% lignin, 1.82% extractives, 8.11% water, and 15.05% mineral ash which comprises mostly silica (Wahab, Nemr, Sikaily & Khaled, 2005). The high cellulose content of rice husk renders it a good source for the production of activated carbon.

1.2 Objectives

- To synthesize activated carbon from rice husk through physical activation and chemical activation by zinc chloride.
- To evaluate the effectiveness of activated carbon derived from rice husk in adsorbing methylene blue, iron(III) ions and ibuprofen as compared to commercial activated carbon
- To study the effect of initial concentration on the adsorption capacity of the risk husk derived activated carbon on methylene blue, iron(III) ions and ibuprofen.

1.3 Hypothesis

- Activated carbon synthesised by chemical activation will have greater adsorption capacities on pollutants than activated carbon synthesised by physical activation.
- Activated carbon synthesised by chemical activation will be more porous than that synthesised by physical activation, and comparable with commercial activated carbon.
- Activated carbon synthesised will have a comparable adsorption capacity with commercial activated carbon.

2. Materials and Methods

2.1 Materials

Rice husks was obtained from World Farm Co Pte. Ltd. Methylene blue was procured from Unichem while ibuprofen was procured from Sigma-Aldrich. Iron (III) nitrate, zinc chloride, sodium alginate and calcium chloride were obtained from GCE Chemicals. Ethanol was obtained from Labscan.

2.2 Synthesis of activated carbon

2.2.1 Physical activation

Rice husks were first washed with hot deionized water to remove dirt and dried in an oven at 70 °C until constant mass. The dried rice husks were then carbonized in an ashing furnace at 400 °C for 40 minutes. The carbon was then ground to fine powder.

2.2.2 Chemical activation using water as solvent

The method for chemical activation was carried out according to Chen et al., (2011) where 15g of zinc chloride was added to 300 ml of deionised water, after which 15g of dried rice husks were added into the solution and boiled for an hour. The treated rice husks were then dried in an oven at 70 °C until constant mass, after which it was carbonized at 400 °C for 40 minutes. The obtained activated carbon was then washed with deionized water until the filtrate was free of chloride ions by testing with aqueous silver nitrate. The washed carbon was then dried in the oven at 70 °C until constant mass. The carbon was then ground to fine powder.

2.2.3 Chemical activation using ethanol as solvent

The problem with the reported method was that when dissolved in water, zinc chloride reacts with water to consistently produce a dense white precipitate. This is due to zinc hydroxychloride an insoluble solid being formed (Peacock, 1918), which precipitates out (equation 1).

$$ZnCl_2\ (s) + H_2O\ (l) \rightarrow Zn(OH)Cl\ (s) + HCl\ (aq) \quad \ldots\ldots(1)$$

Hence zinc ions available to increase the porosity of the carbon was reduced, affecting the porosity of the carbon. To circumvent the problem, a novel method of impregnation with zinc chloride was developed in this study where ethanol is used as the solvent instead of water. Zinc chloride does not react with ethanol and yet dissolves in it, rendering it a more suitable solvent for activation.

15 g of zinc chloride was dissolved in 300 ml of ethanol, after which 15g of rice husks were added into the solution. The mixture was then boiled under reflux for an hour. The carbonization, drying and washing procedures were carried out as described in 2.2.2.

2.3 Iodine number test

Iodine number is a relative indicator for porosity. 10ml of 5% (v/v) hydrochloric acid was added to 0.1g of carbon and the mixture was boiled for 30 seconds. 100ml of iodine solution (0.1N) was

added to the mixture and shaken for another 30 seconds. The mixture was then filtered and titrated with sodium thiosulfate using starch as an indicator.

The iodine number was then calculated with the following formula:

$$\frac{X}{M} = \frac{(A-(DF)BS}{M}$$

where
$\frac{X}{M}$ = mass of iodine adsorbed per gram of carbon (mg/g)
A: normality of iodine solution (N), DF: dilution factor
B: normality of $Na_2S_2O_3$ (N)
S: volume of $Na_2S_2O_3$ (ml)
M: mass of beads used = 0.1 g

2.4 Adsorption Studies

2.4.1 Adsorption tests (Batch study)

The adsorption tests were carried out by mixing 0.1g of activated carbon with 20 ml of pollutant solution and stirred using a magnetic stirrer for 24 hours. The control for the tests were prepared by only adding the pollutant solution without an adsorbent. For each pollutant, 5 replicates were conducted. The mixture was then centrifuged and the supernatant analysed for the concentration of the remaining pollutant. Methylene blue and ibuprofen solutions were analysed using a UV-VIS Spectrophotometer (Shimadzu UV 1800) at 664 nm and 222 nm respectively, while iron(III) ion was analysed using a colorimeter (HACH DR890).

The percentage of pollutant adsorbed was calculated using the following formula:

$$Percentage\ removed = \frac{Initial\ concentration - Final\ concentration}{Initial\ concentration} \times 100\%$$

2.4.2 Effect of initial concentration and isotherm studies

The initial concentration of methylene blue was varied from 100 to 400 mg/l while the initial concentration of iron(III) ions was varied from 75 to 300 mg/l. For each concentration, 5 replicates were conducted and the adsorption capacity was obtained by the following formula:

$$Q\ (mg/g) = \frac{(C_i - C_f)(V)}{M}$$

where C_i = initial concentration in mg/l;
C_f = final concentration in mg/l;
V = volume of solution in dm^3;
M = mass of activated carbon in g

The equilibrium concentration data were then fitted into Langmuir and Freundlich isotherms (Appendix, page 13-18) to investigate the mechanism of adsorption and to determine the maximum adsorption capacity of the activated carbon.

3. Results and discussion

3.1 Characterisation of activated carbon synthesised

3.1.1 By scanning electron microscope (SEM)

Figure 1: SEM image of Physically activated carbon

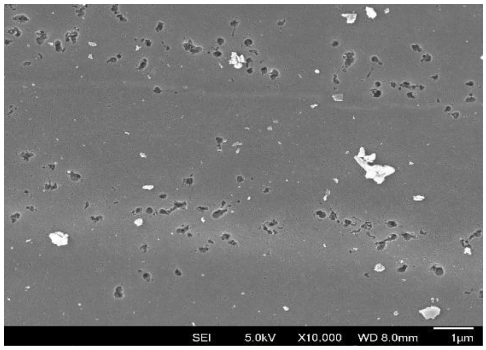

Figure 2: SEM image of commercial activated carbon

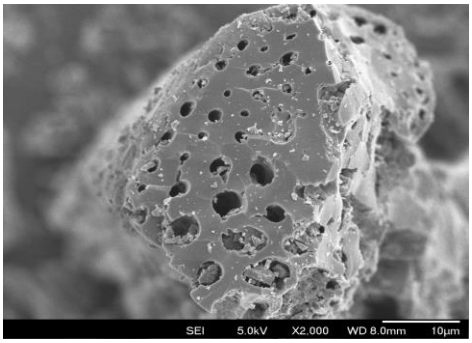

Figure 3: SEM image of chemically activated carbon (water)

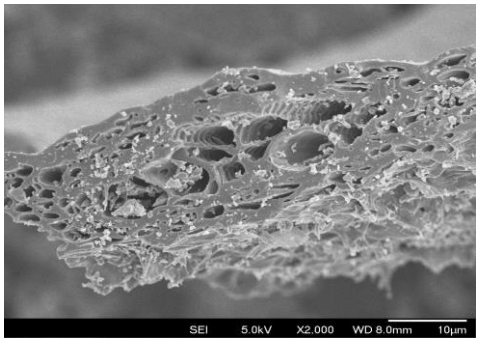

Figure 4: SEM image of chemically activated carbon (ethanol)

Figure 1 shows that physically activated carbon has a lack of pores on its surface, as compared to chemically activated carbons (Figure 3 and 4). The difference in the morphology of the different types of activated carbon suggests that zinc chloride had been effective in increasing the porosity of the carbon. In chemically activated carbon samples, there were presence of macropores and mesopores, while in commercially activated carbon, there were micropores (Figure 2).

3.1.2 By energy-dispersive spectroscopy (EDS)

The EDS spectra (Figures 6 and 7) reveal the presence of carbon, oxygen and silicon as the main elements present in both the physically activated carbon and chemically activated carbon derived from rice husk. In contrast, commercial activated (Figure 5) carbon contains mainly of carbon and oxygen.

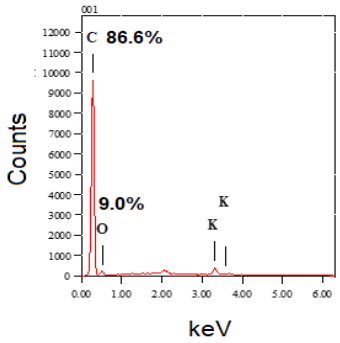

Figure 5: EDS spectrum of commercial activated carbon

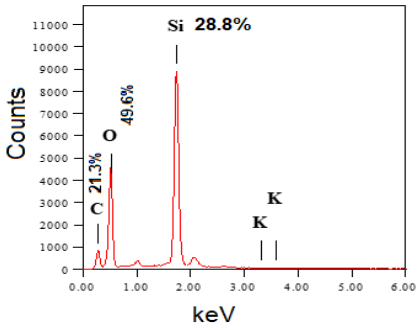

Figure 6: EDS spectrum of physically activated carbon derived from rice husk

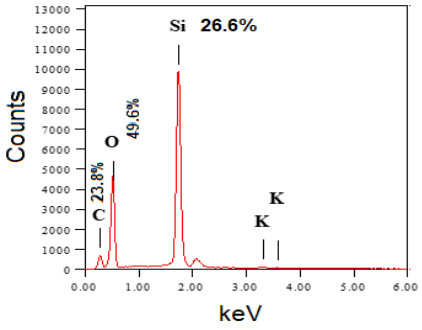

Figure 7: EDS spectrum of chemically activated carbon (water) derived from rice husk

3.2 Iodine number tests

Figure 8 shows the iodine number results, which is an indicator of porosity. Commercially activated carbon has the greatest porosity, possibly due to the abundance of micropores on the carbon surface, which increases the total porosity of carbon. All synthesised carbons have increased porosity than untreated rice husks and chemically activated carbon (ethanol) has the greatest porosity among all synthesised carbons, possibly due to the availability of more zinc ions to form pores on the carbon surface.

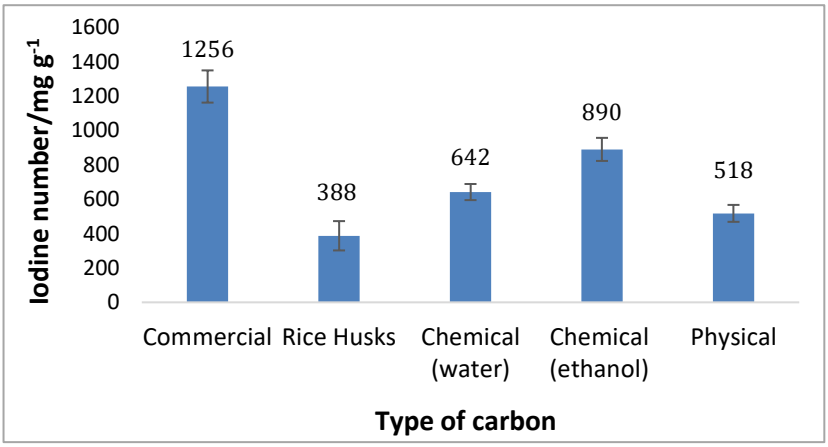

Figure 8: Iodine number of different activated carbons

3.3 Adsorption studies

3.3.1 At 50 mg/l concentration (Batch study)

For the adsorption tests for methylene blue and ibuprofen (Figure 9 and 10), physically activated carbon has the lowest percentage removal, followed by chemically activated (water) carbon, commercially activated carbon and chemically activated (ethanol) carbon. The similar trend observed for both pollutants is likely due to similar adsorption mechanisms, both pollutants being organic. Aromatic rings present in both ibuprofen and methylene blue interact with the aromatic

rings in activated carbon via pi-stacking (Li et al., 2013). Physically activated carbon is the least effective in adsorbing both pollutants, possibly due to the lack of pores on the carbon surface, reducing total surface area. Both types of chemically activated carbon from rice husks can remove methylene blue, iron(III) ions and ibuprofen to a great extent, with the percentage removed close to that of commercially activated carbon, which is both more expensive and energy intensive to produce.

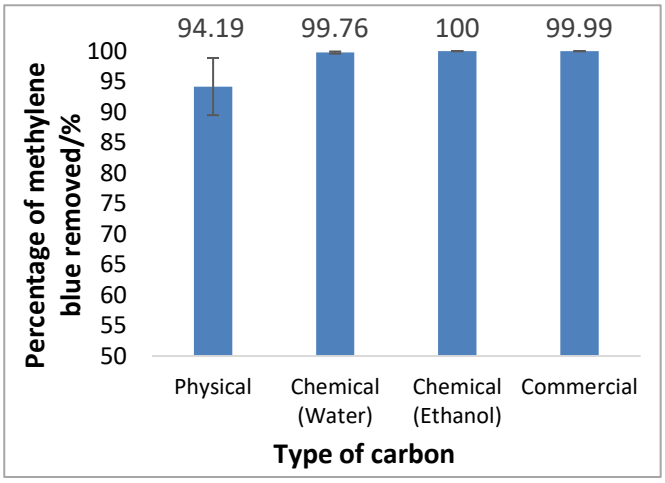

Figure 9: Adsorption of methylene blue by different activated carbons

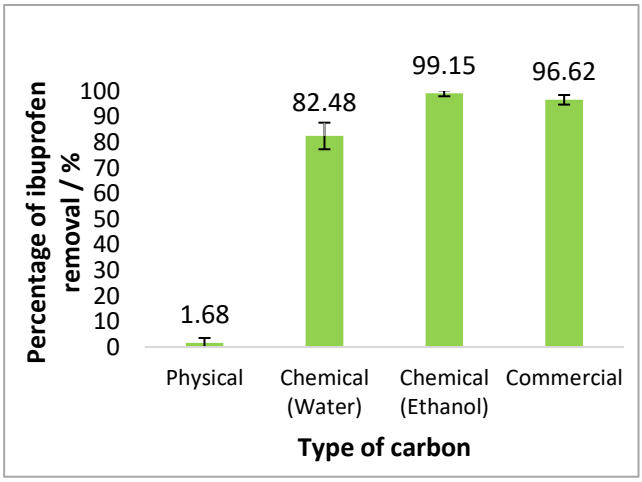

Figure 10: Adsorption of ibuprofen by different activated carbons

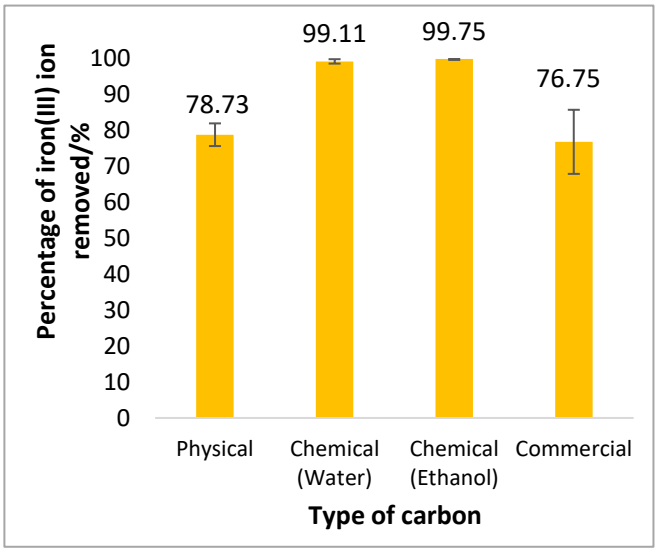

Figure 11: Adsorption of iron(III) ions by different activated carbons

For the adsorption tests on iron(III) ions (Figure 11), all the synthesised carbons had higher percentage removal than commercially activated carbon, possibly due to the presence of higher oxygen content in the chemically activated carbon due to the presence of silica in it, as supported by oxygen content revealed in EDS data (Figure 3). Oxygen provides lone pairs of electrons which allow the activated carbon to form dative bonds with iron(III) ions (Banerjee et al., 2016), hence removing it from solution.

The results were analysed using Mann Whitney U-Test (Table 1).

Table 1: Mann Whitney analysis (synthesised carbon against commercial carbon)

	Methylene blue		**Iron (III) ions**		**Ibuprofen**	
Comparison	P-value	Inference	P-value	Inference	P-value	Inference
Physically activated carbon vs commercial activated carbon	0.012	Significant	0.834	Insignificant	0.0121	Significant
Chemically activated (ethanol) carbon vs commercial activated carbon	0.144	Insignificant	0.0121	Significant	0.0366	Significant
Chemically activated (water) carbon vs commercial activated carbon	0.012	Significant	0.0121	Significant	0.0121	Significant

From the statistical analysis, it can be inferred that the chemically activated (ethanol) carbon is comparable to commercial activated carbon in adsorbing methylene blue as there is no significant difference between it and commercial activated carbon, unlike the other two carbons which differ significantly from commercial activated carbon. The adsorption of iron(III) ions by both the chemical activated carbons is significantly different from commercial activated carbon. All three carbons synthesized differ significantly from commercial activated carbon in the adsorption of ibuprofen.

3.3.2 Effect of initial concentration on adsorption of methylene blue and iron(III) ions

The data of the initial concentration tests were fitted into Langmuir and Freundlich isotherms (Appendix, page 13-18). All data sets had a better fit for the Langmuir isotherm except for the adsorption of methylene blue by commercially activated carbon. This suggests that the adsorption of methylene blue and iron(III) ions by the synthesised activated carbon are monolayer, except for the adsorption of methylene blue by commercially activated carbon which has a multilayer adsorption. The maximum adsorption capacities of the carbons synthesised were derived from the gradient of equations obtained from Langmuir isotherm (Table 2). Maximum adsorption capacity of commercial activated carbon on methylene blue could not be determined as the data is a better fit for Freundlich isotherm, which means maximum adsorption has not been reached yet.

Table 2: Maximum adsorption capacities of the different activated carbons

Type of carbon	Maximum adsorption capacity for methylene blue (mg/g)	Maximum adsorption capacity for iron(III) ions (mg/g)
Chemical (Water)	22.1	18.2
Chemical (Ethanol)	**27.3**	**23.9**
Commercial	-	12.6
Physical	9.9	12.5

Chemically activated (ethanol) carbon has the highest maximum adsorption capacity among all synthesised carbons for both pollutants, and even outperforms commercial activated carbon in adsorbing iron(III) ions, suggesting that it has great potential to be used as an adsorbent to remove iron(III) ions from wastewater.

4. Conclusions and Future Work

Activated carbon was successfully synthesised from rice husks via physical and chemical activation. Only activated carbons prepared by chemical activation were observe to possess macropores and mesopores. A novel chemical activation method using ethanol was implemented in this study and results show that the resulting activated carbon outperforms that synthesized using the conventional method which uses water as a solvent. Chemically activated (ethanol) carbon has the highest porosity and greatest maximum adsorption capacity on methylene blue and iron(III) ions. Physically activated carbon is least porous and least effective in adsorbing all 3 pollutants. Interestingly, both the chemically activated carbon were more effective than commercial activated carbon in adsorbing iron(III) ions, with the chemically activated (ethanol) carbon having almost twice the maximum adsorption capacity of commercial activated carbon. The best performing chemically activated (ethanol) carbon shows great potential to be an eco-friendly alternative to the expensive commercial activated carbon in water treatment.

In future, the best performing activated carbon can be embedded into calcium alginate beads for practical usage. The use of beads rather than powder allows the adsorbents to be separated easily, without the need for filtration or centrifugation. Other extensions include extending the isotherm studies to ibuprofen. Kinetics studies could also be carried out the compare the rate of adsorption of the activated carbon synthesized with that of commercial activated carbon. The range of pollutants studied can also be expanded to include pesticides and other metal ions.

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Appendix: Adsorption Isotherms of Methylene blue and Iron (III) ions

The equilibrium concentration data obtained from initial concentration tests on methylene blue and iron(III) ions were fitted into Langmuir isotherm and Freundlich isotherm.
The Langmuir isotherm assumes that the adsorbed material (such as methylene blue) is adsorbed over a uniform adsorbent surface at a constant temperature.

The linear form of Langmuir isotherm equation is given by:

$$\frac{C_e}{q_e} = \frac{1}{bq_m} + \frac{C_e}{q_m}$$

Where C_e is the equilibrium concentration of pollutant (mg/L), q_e is the equilibrium capacity of the sorbents (mg/g), b is the Langmuir constant that indicates the sorption intensity and q_m is the maximum sorption capacity (mg/g).

The Freundlich isotherm assumes that the adsorption occurs on a heterogeneous surface.
The linear form of Freundlich equation is given by:

$$log(Q_e) = log(K_F) + \frac{1}{n} log(C_e)$$

Where C_e is the equilibrium concentration of pollutant (mg/L), Q_e is the equilibrium capacity of the sorbents (mg/g), K_F, a constant, is related to sorption capacity and n corresponds to sorption intensity.

The Langmuir and Freundlich isotherm plots are shown below:

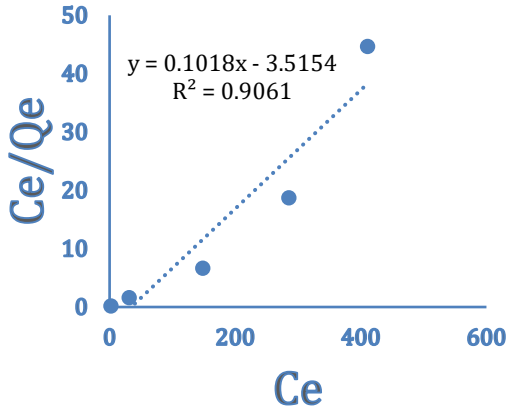

Figure 12: Langmuir isotherm of physically activated carbon on methylene blue

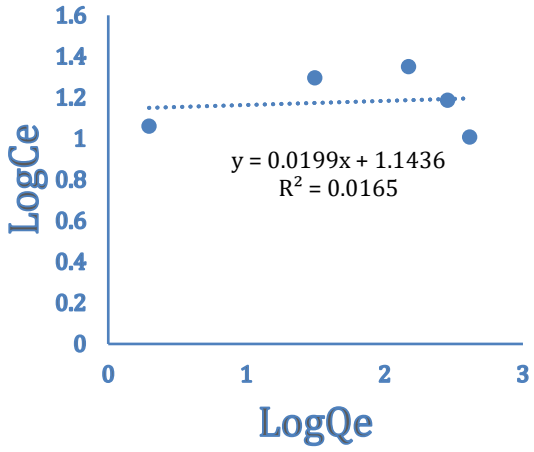

Figure 13: Freundlich isotherm of physically activated carbon on methylene blue

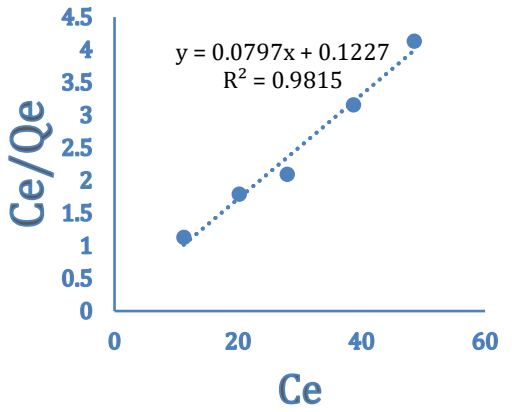

Figure 14: Langmuir isotherm of physically activated carbon on iron(III) ions

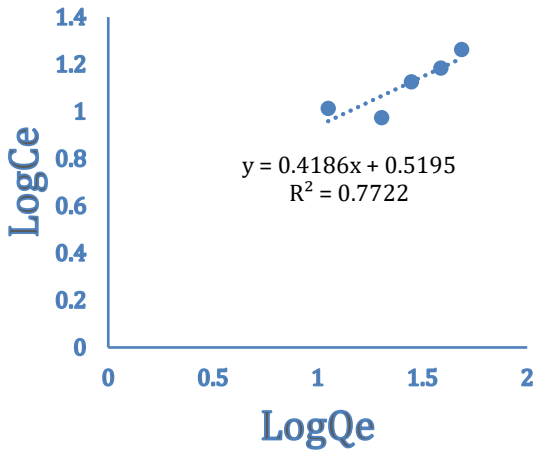

Figure 15: Freundlich isotherm of physically activated carbon on iron(III) ions

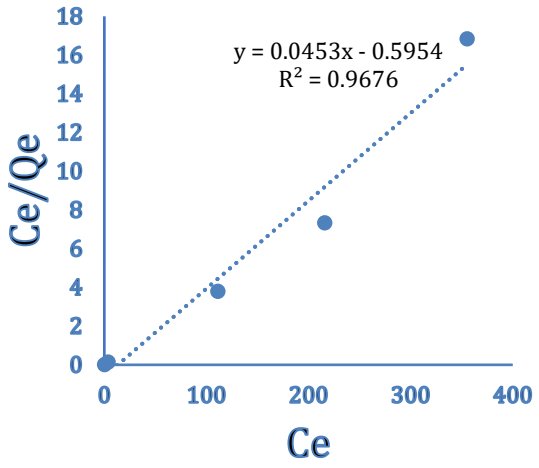

Figure 16: Langmuir isotherm of chemically activated (water) carbon on methylene blue

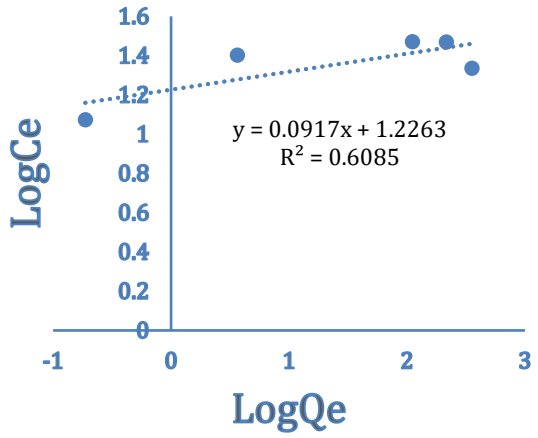

Figure 17: Freundlich isotherm of chemically activated (water) carbon on methylene blue

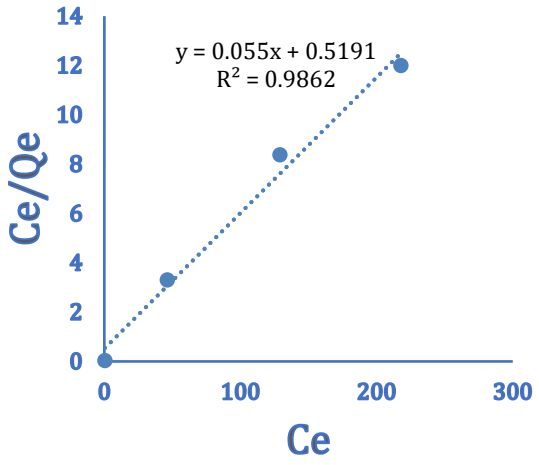

Figure 18: Langmuir isotherm of chemically activated (water) carbon on iron(III) ions

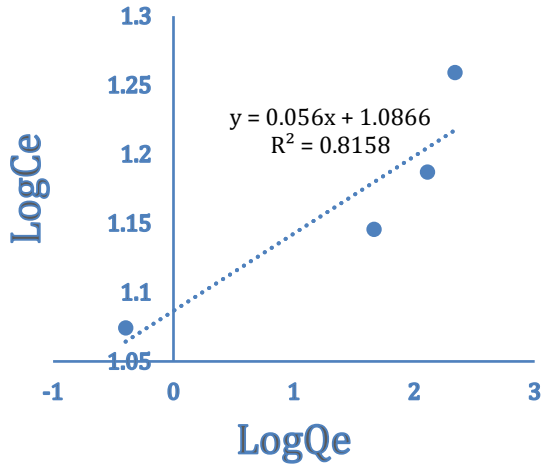

Figure 19: Freundlich isotherm of chemically activated (water) carbon on iron(III) ions

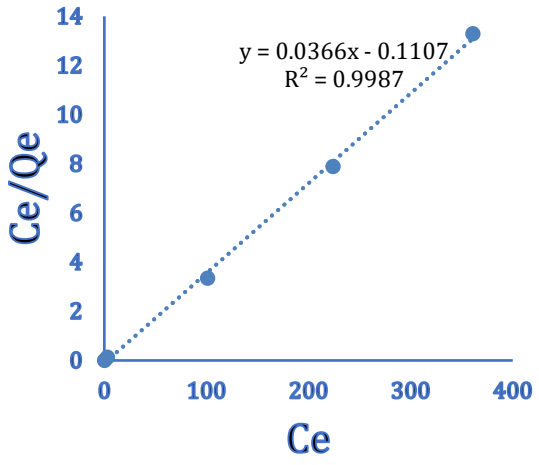

Figure 20: Langmuir isotherm of chemically activated (ethanol) carbon on methylene blue

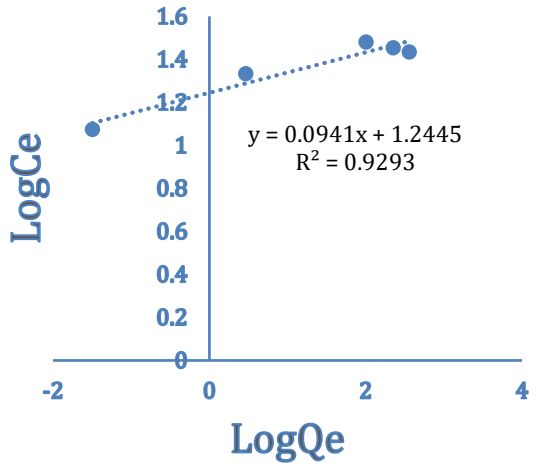

Figure 21: Freundlich isotherm of chemically activated (ethanol) carbon on methylene blue

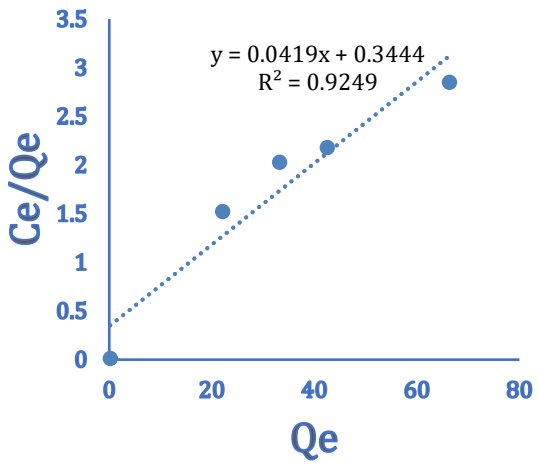

Figure 22: Langmuir isotherm of chemically activated (ethanol) carbon on iron(III) ions

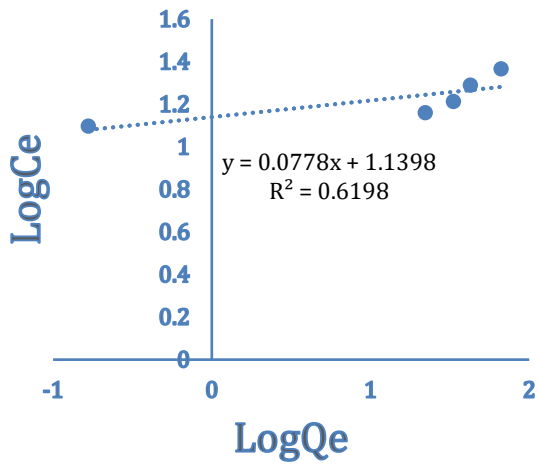

Figure 23: Freundlich isotherm of chemically activated (ethanol) carbon on iron(III) ions

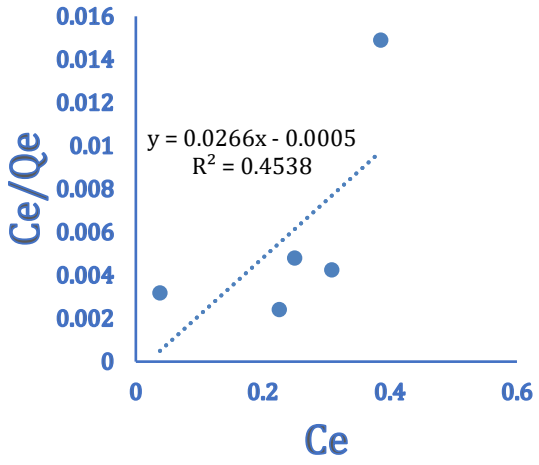

Figure 24: Langmuir isotherm of commercially activated on methylene blue

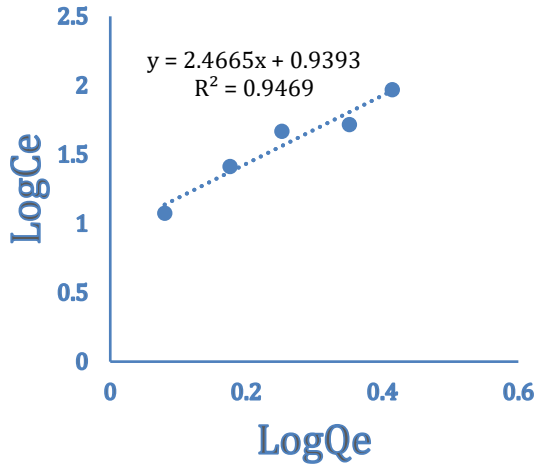

Figure 25: Freundlich isotherm of commercially activated on methylene blue

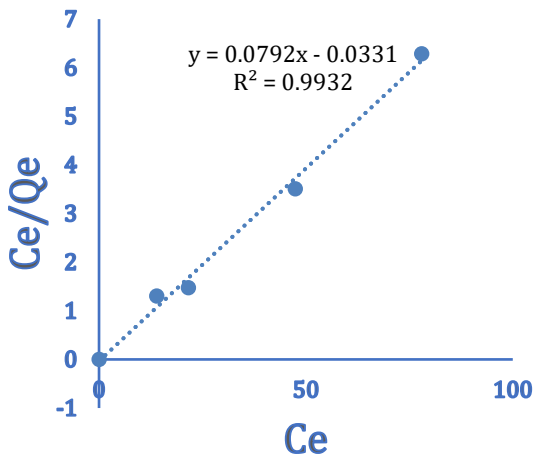

Figure 26: Langmuir isotherm of commercially activated on iron(III) ions

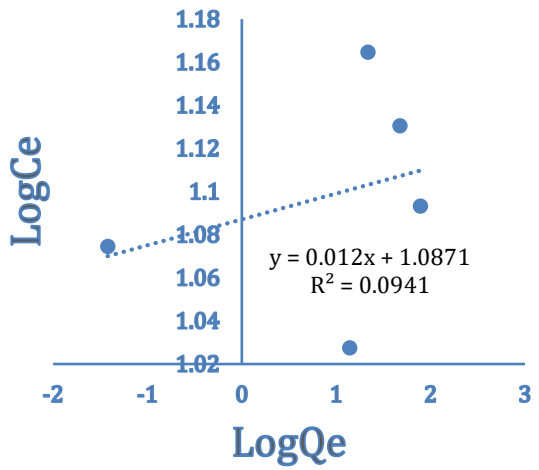

Figure 27: Freundlich isotherm of commercially activated on iron(III) ions

If the equilibrium concentration data fits the Langmuir isotherm, adsorption can be inferred to be monolayer. Important information such as the maximum adsorption capacity of the activated carbon can be derived from the inverse of the gradient of Langmuir linear equations.

In contrast, if the equilibrium concentration data fits the Freundlich isotherm, adsorption can be inferred to occur on a heterogeneous surface and adsorption is multilayer. A comparison between the R^2 values of the graph can determine which model the data set fits better (Table 3).

Table 3: R^2 value comparison of Langmuir and Freundlich isotherms

	Methylene Blue		**Iron(III) ions**	
Type of carbon	R^2 value for Langmuir isotherm	R^2 value for Freundlich isotherm	R^2 value for Langmuir isotherm	R^2 value for Freundlich isotherm
Commercial	0.454	**0.947**	**0.993**	0.094
Chemical (Water)	**0.968**	0.609	**0.986**	0.816
Chemical (Ethanol)	**0.999**	0.929	**0.925**	0.620
Physical	**0.906**	0.017	**0.982**	0.772

Most data sets had a better fit for the Langmuir isotherm, implying that the carbon has a homogenous surface with monolayer adsorption. Commercially activated carbon acting on methylene blue had a better fit for the Freundlich isotherm, implying that the carbon has a heterogeneous surface with multilayer adsorption.