Allium cepa L. peel-derived Activated Carbon as an absorbent for water purification, and evaluating its potential in increased carbon capture

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ABSTRACT

This project aims to produce activated carbon from *Allium Cepa L.* and compare the properties of activated carbon obtained from physically and chemically activated carbon with commercial activated carbon. The activated carbon were synthesised with physical and chemical activation with phosphoric acid, and the activated carbon obtained were characterised by SEM. Iodine number tests were conducted to evaluate the porosity of the activated carbon. Adsorption studies were carried out to determine the percentage removal of copper(II) ions, lead(II) ions and methylene blue by the activated carbon. Carbon capture tests were performed to determine the mass of carbon dioxide absorbed by the activated carbon. The studies showed that the physically activated carbon synthesised from *Allium Cepa L.* has the higher percentage removal of copper(II) ions compared to commercially activated carbon, whereas the chemically activated carbon synthesised from *Allium Cepa L.* had the lowest percentage removal of all three pollutants. Both physically and chemically activated carbon absorbed a larger mass of carbon dioxide than commercial activated carbon. This suggests that physically activated carbon synthesised from *Allium Cepa L.* is comparable to commercial activated carbon in pollutant adsorption while also being a cheaper alternative, and shows promise to be used in carbon absorption and wastewater treatment.

1. Introduction

1.1 Literature Review

Pollution has become a pertinent issue following rapid industrialisation, estimated to be associated with 9 million deaths yearly (Mayor, 2017). Dyes and heavy metals are the two main groups of contaminants commonly found in the wastewater from several industries.

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 (Mohammadi, Karimi, Yazdy, Shamspur, & Hamidian, 2014). Heavy metal ions such as lead(II) ions can decrease cognitive

performance in adults, and contribute to behavioural problems, learning deficits and lowered IQ in children (Wani, Ara, & Usmani, 2015) as they are mutagenic and carcinogenic (Hartwig, 1994).

Dyes in water are extremely visible and undesirable (Crini, 2006). They prevent penetration of light into water bodies, leading to decreased rates of photosynthesis, affecting aquatic life (Imran et al., 2015). Being highly toxic and potentially carcinogenic, consumption of textile dyes may bring about potentially fatal human diseases (Sharma, Dangi, & Shukla, 2018). Discharge of effluents constitute 80% of total emissions generated by the textile industry (Wang, 2016). Dyes exhibit high solubility in water, rendering them difficult to remove using conventional water treatment methods (Hassan & Carr, 2018).

Current wastewater treatments include electrochemical treatment techniques, ion exchange, irradiation, chemical precipitation, electrolytic method, and reverse osmosis (Wang et al., 2012). However, these treatment processes may be costly (Truong & Hart, 2001) and inefficient (Matamoros, Rodríguez, & Albaigés, 2015), rendering them unsuitable for use in small communities or developing countries. Activated Carbon, however, are also very effective adsorbents due to their extensive surface area and micro-porous structure (Anirudhan & Sreekumari, 2011). Their surfaces have unsaturated carbon atoms with many unpaired electrons that play a significant role in adsorption (Shewchuk et al., 2016), and are valued for their efficiency and scalability for commercial usage (Mattson & Mark, 1971). However, widespread use of commercial activated carbon is restricted due to high cost (Crini, 2006), thus demand for activated carbon production using cheaper and versatile materials in developing countries for wastewater treatment (Anirudhan & Sreekumari, 2011).

Recently, there has been growing interest and investments in the research of *Allium Cepa*. This is because it is one of the most popular horticulture vegetable crops and is widely cultivated around the world, which contain carbonaceous materials composed of large amounts of cellulose as well as vitamin and protein which reveals potential application in the field of absorption of heavy metal ions from water (Mehare, Deshmukh, & Dhoble, 2019). The enormous consumption of onions globally has led to production of tons of waste of onion peels, which are left to rot without proper utilization, leading to environmental issues due to the large amount of biowaste produced (Mehare et al., 2019). This has led to more than 500,000 tonnes of onion waste being produced annually in the European Union (Gray, 2011). Hence, readily available onion peels that are inexpensive may potentially be a useful source of biowaste to

produce activated carbon. This would solve the global problem of onion waste and also provide an effective solution to wastewater treatment.

Onion peels are composed of cellulose (41–50%), hemicelluloses (16–26%), and lignin (26–39%), which indicates that the predominant polysaccharide in onion peels is cellulose. This shows that onion peels are versatile and have a high potential for utilization as a new source of biopolymer resources (Reddy & Rhim, 2018).

1.2 Objectives

- To synthesise activated carbon from onion peels through physical activation and chemical activation by phosphoric acid.
- To evaluate the activated carbon for its potential in carbon capture.
- To evaluate the effectiveness of activated carbon derived from onion peel in absorbing dyes, lead ions and copper ions as compared to commercial activated carbon.

1.3 Hypothesis

- Chemically activated onion peel-derived activated carbon has the highest pollutant adsorption rate compared to commercial and physically activated carbon.
- Chemically activated carbon is more porous than commercially available activated carbon and physically activated carbon
- Synthesised activated carbon synthesised in this study has greater ability to adsorb carbon dioxide in atmosphere than commercial activated carbon

2. Materials and Methods

2.1 Materials

Onion peels were procured from Banu's Holdings Pte Ltd. Phosphoric acid, copper(II) sulfate and lead(II) nitrate were obtained from GCE Chemicals, while methylene blue and commercial activated carbon were obtained from Unichem.

2.2 Synthesis of Activated Carbon

2.2.1 Physical Activation

50g of onion peels were first washed with tap water to remove dirt before being washed with deionised water. The onion peels were left to dry overnight before drying in an oven at 70°C until constant mass. They were then carbonised in an ashing furnace at 400°C for 40 minutes.

The high temperature enables the onion peels to undergo pyrolysis. Heat and water vapor travel in the onion peels via diffusion and thermal transfer to activate more carbon molecules and generate more pores (Huang, Cheng, & Lin, 2015). The carbon was then grounded into fine powder using a mortar and pestle.

2.2.2 Chemical Activation

50g of onion peels were first washed with tap water to remove dirt before being left to dry overnight. 20g of dried onion peels were then added to 400 ml of 0.6M phosphoric acid and boiled for an hour. The treated onion peels were then dried in an oven at 70°C until constant mass, before being carbonised in an ashing furnace at 400°C for 40 minutes. The obtained activated carbon was then washed with deionised water until the mixture was free of hydrogen ions and had a pH of 7 by testing with universal indicator paper. The activated carbon was then dried in an oven at 70°C until constant mass again before being grounded into fine powder using mortar and pestle. The same steps were repeated for the remaining 30g of onion peels that were boiled in 600 ml of 0.6M phosphoric acid for an hour.

2.3 Iodine Number Test

Iodine number is an indication of porosity (Sutter & Bentz, 2017). 10 ml of 5% (v/v) hydrochloric acid was added to 0.1g of activated carbon and the mixture boiled for 30 seconds. 100 ml of 0.1 N iodine solution was then added to the mixture, and the mixture shaken vigorously for 30 seconds. The mixture was then filtered before a 25 ml aliquot of the filtrate was titrated with sodium thiosulfate to test for the amount of triiodide ions remaining in solution using starch as an indicator. The titration was repeated 2 more times for each type of activated carbon.

The iodine number was then calculated using the ASTM D4607-94 method according to Nunes & Guerreiro (2011):

$$X/M = \{(N_1 \text{ x } 126.93 \text{ x } V_1) - [(V_1 + V_{Hcl})/V_F] \text{ x } (N_{Na_2S_2O_3} \text{ x } 126.93) \text{ x } V_{Na_2S_2O_3}\}/M_c \quad (2)$$

$$C = (N_{Na_2S_2O_3} \text{ x } V_{Na_2S_2O_3}) \quad (3)$$

where N_I is the iodine solution normality, V_I is the added volume of iodine solution, V_{HCl} is the added volume of 5% HCl, V_F is the filtrate volume used in titration, $N_{Na2S2O3}$ is the sodium thiosulfate solution normality, $V_{Na2S2O3}$ is the consumed volume of sodium thiosulfate solution and M_C is the mass of activated carbon.

2.4 Adsorption Studies

2.4.1 Pollutant Solutions

Pollutant solutions were prepared by adding 0.197g, 0.050g and 0.100g of hydrated copper(II) sulfate, methylene blue powder and lead(II) nitrate respectively to 1L of deionised water in 1L volumetric flasks to obtain 3 bottles of 50 ppm solutions.

2.4.2 Batch tests

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. Control tests were prepared by only adding 20 ml of pollutant solution without an adsorbent. For each pollutant, triplicates were conducted for each type of activated carbon. After 24 hours, 12 ml of the mixture was decanted and then centrifuged, before transferring 10 ml of the supernatant into another centrifuge tube. The supernatant was then analysed for the concentration of the remaining pollutant. Methylene blue solutions were analysed using a UV-vis Spectrophotometer (Shimadzu UV 1800) at 664 nm, copper(II) ions was analysed using a colorimeter (HACH DR890), and lead(II) ions were analysed using the Atomic Absorption Spectrophotometer (Shimadzu AA-6300).

15 standard solutions of methylene blue of 5 varying concentrations from 1 to 5 ppm were prepared and analysed for the absorbance using the UV-vis Spectrophotometer at 664 nm. The respective absorbance values were then plotted against concentration on a graph to obtain the calibration curve for methylene blue (Appendix, page 14).
The percentage of pollutant adsorbed was calculated using the following formula:

$$\text{Percentage removed} = \frac{\text{Initial conc. - Final conc.}}{\text{Initial conc.}} \times 100\%$$

2.5 Carbon Capture Tests

2.5.1 Batch Tests

Carbon capture tests were performed by adding 0.100g of calcium carbonate into a conical flask and 25 ml of 1M hydrochloric acid into a burette. The conical flask was then placed below the burette and the opening of the conical flask was covered by the rubber stopper of the burette.

0.300g of cotton wool was added into a 20 ml syringe, followed by 0.500g of silica gel, before another 0.200g of cotton was added on top of it. 0.500g of activated carbon was then added before a final 0.500g of cotton wool was added to the syringe. The hub of the syringe was then

covered with a rubber tubing attached to a glass tubing extending from the rubber stopper covering the conical flask. This enables the passage of gases and water vapour produced from the reaction of copper carbonate and hydrochloric acid into the syringe without escaping into the burette as the burette valve was closed after the addition of hydrochloric acid. The chemical equation representing the reaction is as follows:

$$CaCO_3\,(s) + 2\,HCl\,(aq) \rightarrow CaCl_2\,(aq) + H_2O\,(l) + CO_2\,(g)$$

The mass of the syringe was measured before and 15 minutes after the addition of hydrochloric acid to calcium carbonate, and the change in mass was recorded. For each type of activated carbon, triplicates were performed and the average change in mass recorded.

2.5.2 Control Tests

Control setups for carbon capture were prepared by adding 0.300g of cotton wool, followed by silica gel and another 0.700g of cotton wool into the syringes. The mass of silica gel added to the syringes varied from 0.300g, 0.500g to 0.700g for the 3 syringes. The remaining procedures of the reaction and measurement of change in mass of syringes were carried out as described in 2.5.1.

3. Results and discussion

3.1 Characterization of activated carbon by scanning electron microscope (SEM)

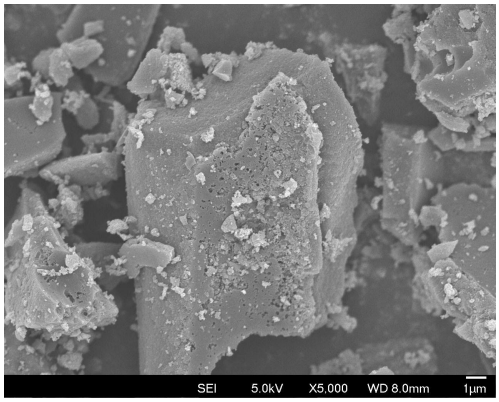


Figure 1: SEM image of commercial activated carbon

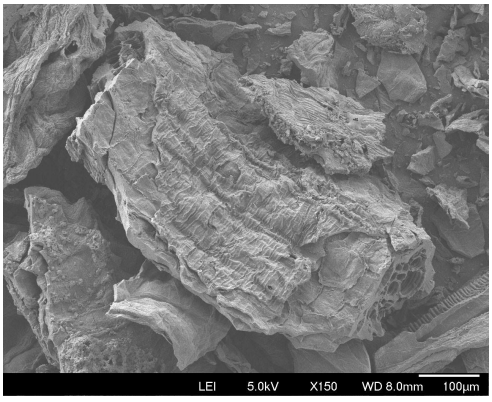


Figure 2: SEM image of chemically activated carbon

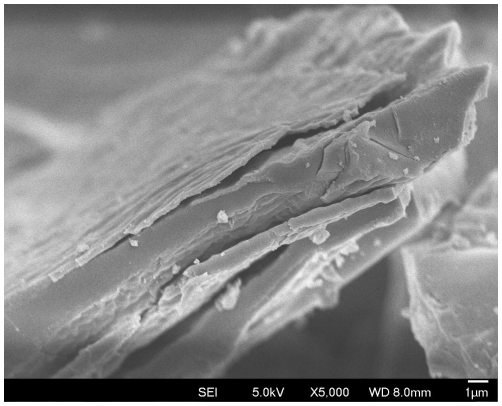


Figure 3: SEM image of physically activated carbon

Figure 2 shows that chemically activated carbon has a lack of pores on its surface, comprising mainly of macropores and mesopores. On the other hand, figures 1 and 3 show that commercial and physically activated carbon respectively have many micropores on its surface as compared to chemically activated carbon. The difference in morphology between the 3 types of carbon shows that phosphoric acid may not have been effective in ensuring widespread micropores covering a greater surface area on the chemically activated carbon.

3.2 Iodine Number Tests

We tabulated the average volume of sodium thiosulfate required for titration for all 3 types of activated carbon in Table 1 before calculating the iodine numbers (Appendix, page 14)

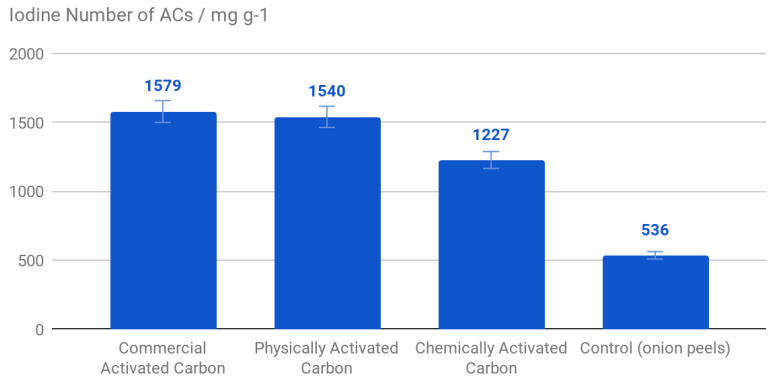


Fig 4: Comparison of iodine numbers of activated carbon

Based on the results shown in Figure 4, commercial activated carbon has the greatest porosity amongst all carbon, which is highly likely due to the abundance of micropores on its surface increasing its porosity. Physically activated carbon has a higher iodine number than chemically activated carbon, likely due to the greater number of micropores on its surface.

3.3 Adsorption Studies

For all 3 pollutants, the concentration of pollutants remaining in each sample was tabulated in tables 2, 3 and 4, and average concentration of pollutants remaining, followed by the percentage of pollutants removed calculated (Appendix, pages 15 and 16).

For the adsorption tests for methylene blue and copper(II) ions (Figures 5 and 6), physically activated carbon has the highest percentage removal, followed closely by commercial activated carbon, and chemically activated carbon. The high adsorption rate of methylene blue by all 3 types of activated carbon is likely due to the fact that both activated carbon and methylene blue are organic compounds, with aromatic rings present in methylene blue interacting with that in activated carbon through pi-pi stacking (Li et al., 2013). For copper(II) ions, the high adsorption rates of commercial and physically activated carbon is likely attributed to their highly porous morphology, with many micropores on their surfaces to adsorb ions (Figures 1 and 3). The low adsorption rate of chemically activated carbon is likely due to the lack of pores on the carbon surface (Figure 2), hence reducing surface area available for adsorption of copper(II) ions.

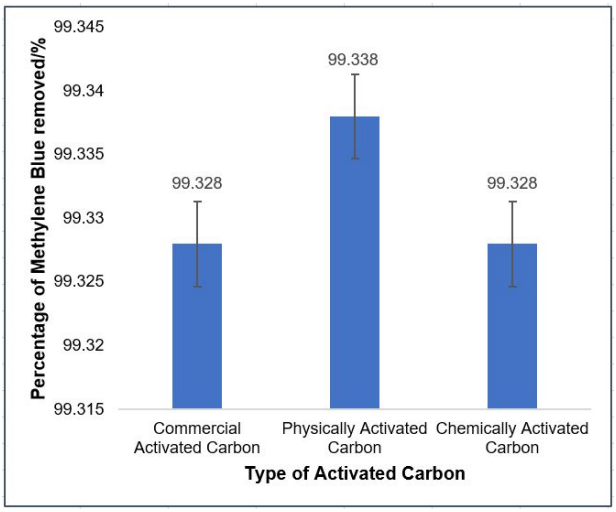


Figure 5: Adsorption of methylene blue by different activated carbon

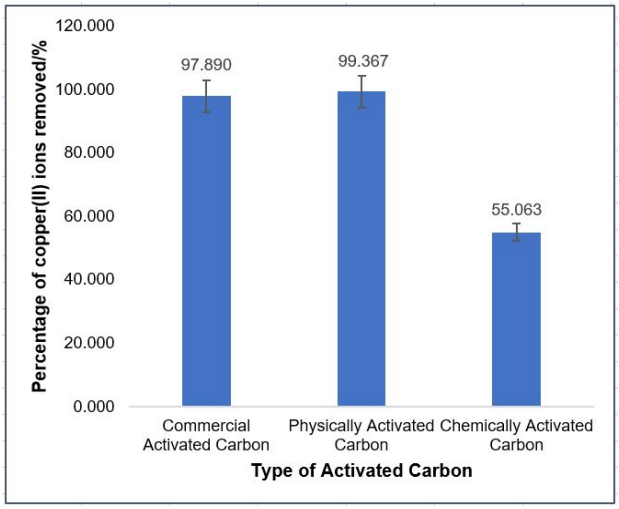
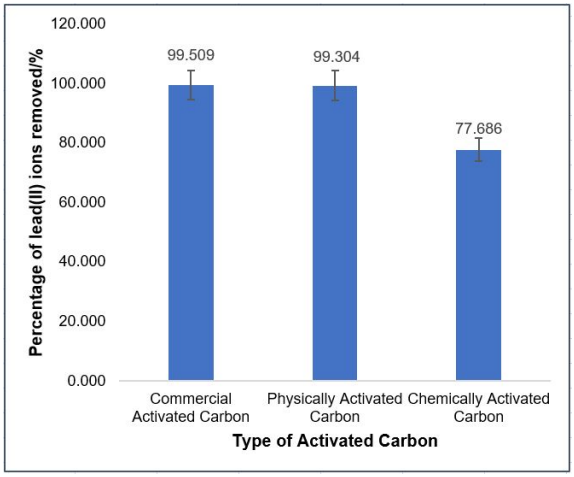


Figure 6: Adsorption of copper(II) ions by different activated carbon

For the adsorption test for lead(II) ions (Figure 7), commercial activated carbon had the highest percentage removal, followed closely by physically activated carbon. The high adsorption rates for these 2 types of carbon is again likely due to their high porosity, with many available micropores to adsorb the lead(II) ions. The low adsorption rates of chemically activated carbon



is largely due to the presence of only mesopores and macropores on the carbon surface, hence decreasing its total surface area available to adsorb the ions.

Figure 7: Adsorption of lead(II) ions by different activated carbon

3.4 Carbon Capture Tests

For all samples, the masses of the syringe before and after 15 minutes and the change in mass was tabulated in tables 6, 7 and 8 (Appendix, page 17).

3.4.1 Control Tests

For control tests, the change in mass of the syringes was found to be 0.005g across all 3 samples of varying silica gel masses (Table 5). This shows that the adsorption of water vapour by silica gel contributes to an increase in mass of 0.005g.

3.4.2 Batch Tests

For batch tests, the change in mass of the syringes for synthesised activated carbon was found to be 0.002g greater than that of commercial activated carbon on average without accounting for the adsorption of water vapour by silica gel (Figure 9). This shows that synthesised activated carbon have a greater carbon dioxide adsorption rate than commercial activated carbon, and thus have greater potential in the field of carbon capture, which is more expensive to produce.

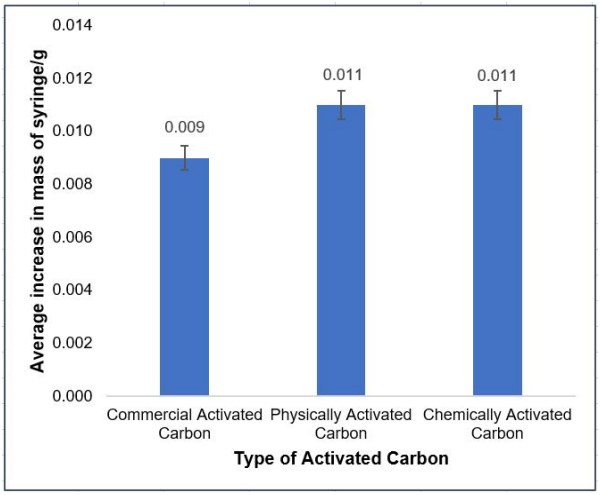


Figure 8: Comparison of change in mass of syringes for different types of carbon

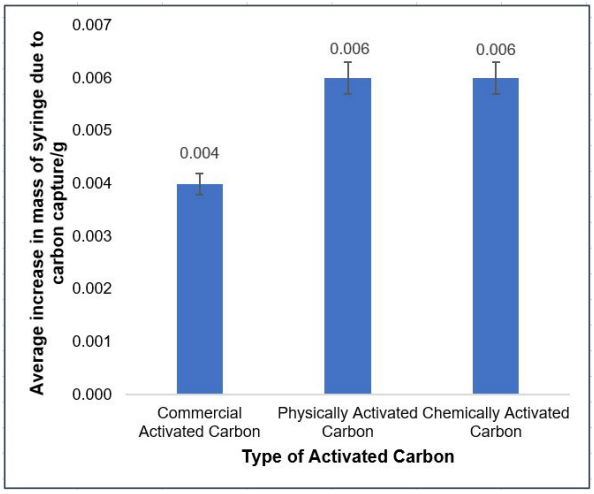


Figure 9: Comparison of change in mass of syringes for different types of carbon excluding adsorption of water vapour

4. Conclusions and Future Work

Activated carbon was successfully synthesised from onion peels using physical and chemical activation. Only activated carbon prepared by physical activation was observed to possess mesopores and macropores. The activated carbon was then compared to the commercial activated carbon derived from charcoal. Chemically activated carbon is the least porous and least effective in adsorbing copper(II) and lead(II) ions. The best performing activated carbon is physically activated carbon. It has the highest porosity and greatest adsorption of copper(II) ions, lead(II) ions and also carbon dioxide. This form of activated carbon shows great potential to be an eco friendly and cheaper alternative to the commercially activated carbon, which are non-renewable and costly as it displays similar, if not better, adsorbent properties.

In future, the synthesised activated carbon can be tested for its effectiveness in adsorbing other pollutants such as ammonia and organic solvents used in the medical industry, as well as other harmful gaseous pollutants. Adsorption kinetics studies could be carried out to compare the rate of adsorption between the commercial and synthesised activated carbon. The activated carbon can also be tested for its effectiveness when it contains inorganic impregnate such as silver, and different types of activated carbon such as granular activated carbon can be experimented with.

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APPENDIX

Methylene Blue Calibration Curve

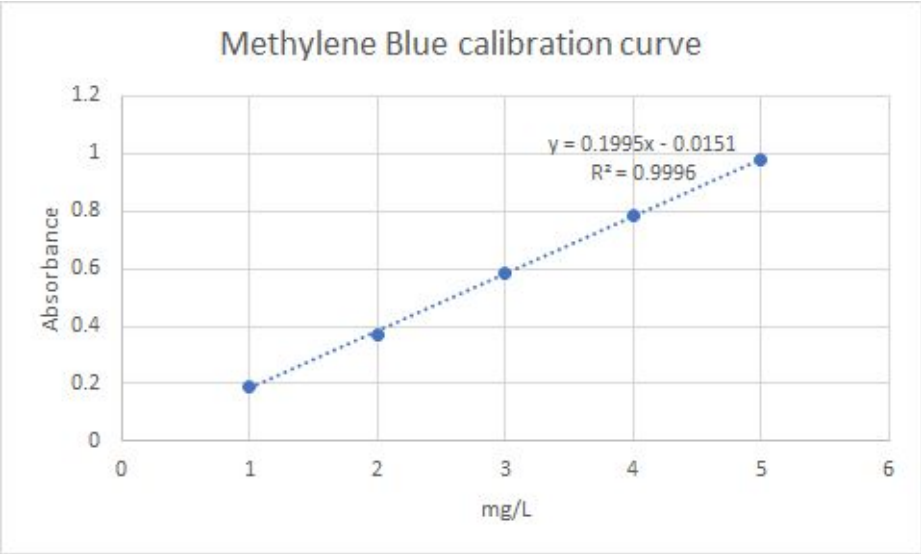

Figure 10: Methylene Blue Calibration Curve

Iodine Number Tests

Type of Activated Carbon	Vol. of $Na_2S_2O_3$ reacted/ml			Avg. Vol. of $Na_2S_2O_3$ reacted/ml	Iodine Number / mg g^{-1}
	1st titration	2nd titration	3rd titration		
Commercial Activated Carbon	19.90	20.10	19.70	19.90	1579
Physically Activated Carbon	20.00	20.00	19.90	19.97	1540
Chemically Activated Carbon	20.70	20.50	20.40	20.53	1227
Control (onion peels)	21.70	21.80	21.80	21.77	536

Table 1: Comparison of volume of sodium thiosulfate reacted for iodometric titration

Adsorption Studies

Sample	Absorbance / Au			Avg. absorbance /Au	Conc. / ppm	Percentage removed / %
	Sample 1	Sample 2	Sample 3			
Physical AC	0.063	0.046	0.044	0.051	0.331	99.338
Chemical AC	0.051	0.044	0.060	0.052	0.336	99.328
Commercial AC	0.045	0.067	0.044	0.052	0.336	99.328
Control	3.432	3.428	3.430	3.430	17.269	65.462
Original Solution					50	

Table 2: Adsorption rate for methylene blue by different types of carbon

Sample	Concentration / ppm			Avg. Conc. / ppm	Percentage removed / %
	Sample 1	Sample 2	Sample 3		
Physical AC	0.5	0.1	0.0	0.30	99.367
Chemical AC	21.5	21.0	21.5	21.3	55.063
Commercial AC	0.9	1.0	1.0	1.0	97.890
Control	46.8	46.7	46.0	46.5	1.899
Original Solution				47.4	

Table 3: Adsorption rate for copper(II) ions by different types of carbon

Sample	Concentration / ppm			Ave. Conc. / ppm	Percentage removed / %
	Sample 1	Sample 2	Sample 3		
Physical AC	0.1546	0.1312	0.1429	0.1429	99.304
Chemical AC	6.5775	1.3961	5.7624	4.5787	77.686
Commercial AC	0.1265	0.0750	0.1008	0.1008	99.509
Control	17.4744	19.2078	17.5213	18.0678	11.949
Original Solution	19.8637	22.0187	19.6763	20.5196	

Table 4: Adsorption rate for lead(II) ions by different types of carbon

Carbon Capture Tests

Sample	Mass of AC/g	Mass of silica gel/g	Mass of syringe before/g	Mass of syringe after/g	Change in mass/g
1	0	0.300	7.064	7.069	0.005
2		0.500	7.286	7.291	0.005
3		0.700	7.483	7.488	0.005

Table 5: Carbon Capture tests (control)

Sample	Mass of AC/g	Mass of silica gel/g	Mass of syringe before/g	Mass of syringe after/g	Change in mass/g
1	0.500	0.500	7.725	7.743	0.018
2			7.768	7.776	0.008
3			7.763	7.771	0.008

Table 6: Carbon Capture tests (physically activated carbon)

Sample	Mass of AC/g	Mass of silica gel/g	Mass of syringe before/g	Mass of syringe after/g	Change in mass/g
1	0.500	0.500	7.706	7.715	0.009
2			7.763	7.769	0.006
3			7.804	7.823	0.019

Table 7: Carbon Capture tests (chemically activated carbon)

Sample	Mass of AC/g	Mass of silica gel/g	Mass of syringe before/g	Mass of syringe after/g	Change in mass/g
1	0.500	0.500	7.688	7.700	0.012
2			7.723	7.732	0.009
3			7.765	7.772	0.007

Table 8: Carbon Capture tests (commercial activated carbon)