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Abstract

Discharge of industrial effluents containing dyes, metal ions and pesticides poses a serious threat to the environment. Examples include dye pollution in Asian countries like Bangladesh as well as the Flint water crisis in Michigan, USA. Banana peel is a widely available food waste that has potential to be used as a biosorbent. Carbonising banana peel enhances its adsorption capacity, while magnetising it renders the separation after adsorption simple and convenient via a magnet. Magnetic carbonised banana peel (MCB) was prepared by dispersing 3g to 5g of carbonised banana peel into aqueous iron salts. Results show that the percentage removal of brilliant green dye, lead(II) ions and atrazine by MCB was more than 95% and the maximum adsorption capacity of MCB derived from the Langmuir isotherm was comparable to commercial activated carbon for lead(II) ions but slightly lower than that of commercial activated carbon for brilliant green dye. The effectiveness of MCB in removing the two pollutants did not decrease significantly and close to 100% of MCB could be retrieved using a magnet for 3 progressive cycles of adsorption and desorption, unlike commercial activated carbon. MCB shows great promise as a versatile, eco-friendly and reusable adsorbent for water purification.

Keywords

Banana peel, magnetic, eco-friendly, reusable, purification

1 Introduction

With a rapid growth of industries, water pollution becomes a pertinent issue in today's context. Pollution is associated with an estimated 9 million deaths a year, with water pollution contributing to 1.8 million deaths [1].

One common pollutant commonly discharged into water bodies is dye. In the textile industry, up to 200,000 tonnes of these dyes are lost to effluents every year due to the inefficiency of the dyeing process [2], presenting major environmental problems for developing countries like Bangladesh [3]. One example of dye is brilliant green (BG), a toxic cationic dye that is widely used in the textile industries [4]. Discharge of BG into the hydrosphere has an adverse effect on humans, causing irritation to the gastrointestinal and respiratory tract, as well as symptoms such as nausea, vomiting and diarrhoea [5].

Another type of common water pollutant is heavy metal ions, such as lead(II) ions. Lead contamination generally occurs as a result of corrosion of lead-containing plumbing systems and surface runoffs of lead-based materials like paints [6]. A recent example is the drinking water crisis that occurred in Flint, US, which resulted in hundreds of people being poisoned by lead contaminated water [7]. Lead(II) ions are known to cause neurological diseases that impair basic mobility functions, growth defects and even death [8].

Atrazine is one of the most commonly used herbicides in the United States and Australia and is frequently detected in ground, surface and drinking water, such as in the lower Mississippi river and its tributaries [9]. Atrazine is an endocrine disruptor for mammals and aquatic life [10]. Human exposure to atrazine has been linked to adverse health effects like breast and prostate cancer as well as infertility [11], thus treatment of herbicide-containing water is of paramount importance.

Current methods of removing toxic dyes and pesticides include chemical coagulation, adsorption [12] and advanced oxidation processes [13]. Metal ions are removed by chemical precipitation, ion exchange and adsorption. Among these methods, adsorption by activated carbon is one of the most effective methods because of its efficiency, capacity and scalability for commercial usage [14]. However, synthesis of activated carbon requires high temperatures of up to 800°C [15], leading to high energy and capital costs [16]. Retrievability is inefficient as it requires filtration or flocculation in order to remove the adsorbent-contaminant complex from water [6].

Banana peels have attracted attention as a widely available and inexpensive biological waste [17] to be used as ecofriendly adsorbents for water purification. The number of bananas consumed annually in the world is more than 100 billion, making it the fourth most important food crop after wheat, rice and corn [18]. Banana peels present a high adsorption capacity for metals and organic compounds [19], primarily due to the presence of the hydroxyl and carboxyl groups of the pectin [20]. Carbonisation of banana peel can potentially enhance its adsorption capacity [21] while magnetisation (via magnetite coating) serves as an easier and faster way of separating the adsorbent from the adsorbentcontaminant complex in place of filtration or flocculation.

Although there have been studies on the use of magnetized adsorbents such as waste tea to remove metal ions [6], to date, there have been limited studies on the use of magnetic banana peel to remove a range of pollutants including dyes, pesticides and metal ions.

This study aims to synthesise magnetic carbonised banana peel (MCB) via co-precipitation, evaluate its adsorption capacity on brilliant green dye, lead(II) ions and atrazine as compared to commercial activated carbon (AC), investigate its reusability after progressive cycles of adsorption and desorption and construct a prototype for the application of MCB for industrial use.

2 Materials and Methods

2.1 Materials

Iron(III) chloride hexahydrate, iron(II) sulfate heptahydrate, 25% (w/w) aqueous ammonia and lead(II) nitrate were procured from GCE Laboratory Chemicals; commercial

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activated carbon from Unichem; brilliant green and atrazine from Sigma Aldrich. Banana peels were obtained from a fruit stall in school.

2.2 Synthesis of Magnetic Carbonised Banana Peel (MCB)

Banana peels were washed with deionised water, dried and crushed. They were then carbonised in a furnace at 450°C for 40 min under atmospheric conditions, and ground into powder. 6.66g of iron(III) chloride hexahydrate and 13.39g of iron(II) sulfate heptahydrate were dissolved in 45 ml of deionised water. Subsequently, carbonised banana peel (3g, 3.5g, 4g, 4.5g and 5g) was mixed and stirred in 15ml of the iron salt solution. 25ml of 25% (w/w) aqueous ammonia was added into the solution to induce co-precipitation of magnetite onto the carbonised banana peel. The mixture was stirred and left to stand for 30 minutes before being filtered using vacuum filtration. The residue was washed until neutral pH and dried at 60°C until constant mass. The chemical reaction for the co-precipitation of magnetite is shown in equation (1).

Fe2+ (aq) + 2Fe3+ (aq) + 8OH- (aq)
$$\rightarrow$$

Fe3O4 (s) + 4H2O (l) (1)

The magnetic carbonised banana peels (MCB) were labelled according to the mass of carbonised banana peel used in the synthesis, as shown in Table 1.

Table 1: Sample ID of magnetic carbonised banana peel (MCB)

Sample ID	Mass of carbonised banana peel used in synthesis/g	Volume of iron salt solution used/ml
MCB (3g)	3.0	15
MCB (3.5g)	3.5	15
MCB (4g)	4.0	15
MCB (4.5g)	4.5	15
MCB (5g)	5.0	15

2.3 Batch Adsorption Studies on Magnetic Carbonised Banana Peel (MCB)

0.2g of the various MCBs mentioned in Table 1 was added to 20 ml of brilliant green solution (50 mg/L), lead(II) ion solution (50 mg/L) or atrazine solution (5 mg/L) and shaken on an orbital shaker at 150 rpm for 24 hours. The concentration of atrazine was lower than the other two pollutants as it has limited solubility in water. A magnet was used to separate the MCB and the supernatant was obtained. Final concentration of brilliant green was analysed using a UV-VIS spectrophotometer (Shimadzu UV 1800) at 627 nm while that of lead(II) ions was analysed using an Atomic Absorption Spectrophotometer (AA 6300 Shimadzu). Pollutant solutions which do not contain any adsorbent served as the controls for the experiments. The adsorption studies were also conducted on commercial activated carbon (AC), carbonised banana peel and unmodified banana peel for comparison with MCB. These non-magnetic adsorbents were separated from the mixture using a centrifuge, whereas MCB was separated from the solution using a magnet. The percentage of brilliant green, lead(II) ions and atrazine removed was calculated using the following formula:

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

For each pollutant, out of the 5 MCBs synthesised, the MCB that could remove the highest percentage of pollutant while retaining its ability to be magnetically separated from the solution was selected for the subsequent reusability and adsorption isotherm studies.

2.4 Adsorption Isotherms

Adsorption isotherms which provide insights on adsorption mechanisms and maximum adsorption capacity were determined by introducing 0.2g of MCB, carbonised banana peel or commercial AC to brilliant green and lead(II) ion solutions of concentrations from 50 to 1800 mg/L. Data was fitted into Langmuir and Freundlich isotherms (Annex A, Pages 7-8).

2.5 Reusability of Magnetic Carbonised Banana Peel (MCB)

After each adsorption study, 100 ml of 70% ethanol was added to the used adsorbent (MCB or commercial AC) to desorb the dye for 24 hours, after which the adsorbent was separated and dried. The regenerated adsorbent was tested on its ability to re-adsorb brilliant green. The same procedure was conducted for lead(II) ions, except that 70% ethanol was replaced by deionised water.

2.6 Iron Leaching Tests on Magnetic Carbonised Banana Peel at various pH

To simulate electroplating wastewater, a solution containing 25.0 mg/L of lead(II) ions and 13.9 mg/L of copper(II) ions with a pH of 6 was prepared according to literature [22]. Batch adsorption studies were conducted in a similar way to that described in Section 2.3 of this report and the concentration of iron ions leached out into the solution was measured using a colorimeter (HACH DR 890). The procedure was repeated for pH 2 and pH 4.

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3 Results and Discussion

3.1 Characterisation of Magnetic Carbonised Banana Peels (MCB)

3.1.1 By Scanning Electron Microscopy

Banana peel has a rough, uneven surface (Figure 1) and becomes porous after carbonisation (Figure 2).



Figure 1: SEM image of banana peel at 10,000x magnification



Figure 2: SEM image of carbonised banana peel at 10,000x magnification

Figure 3 shows magnetite particles. After magnetisation, a coating of magnetite particles can be seen blocking the pores on the surface of the carbonised banana peel (Figure 4). The Scanning Electron Microscopy (SEM) images of MCB prepared by other masses of carbonised banana peel are similar to that seen in Figure 4.



Figure 3: SEM image of magnetite particles at 100,000x magnification



Figure 4: SEM image of MCB (3g) at 100,000x magnification

3.1.2 By X-Ray Diffraction

The X-Ray Diffraction (XRD) pattern of MCB (3g) (Figure 5) exhibits 2 theta peaks at 30.45° , 35.74° , 43.40° , 53.81° , 57.32° , 62.93° corresponding to crystal planes of (220), (311), (400), (422), (511) and (440) respectively, which is similar to that reported by Loh et al. in 2008 [23]. This suggests that magnetite has been successfully coated onto the carbonised banana peel.



Figure 5: XRD Spectrum of MCB (3g)

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3.2 Batch Adsorption Studies of Magnetic Carbonised Banana Peel (MCB)

3.2.1 Brilliant Green

Figure 6 shows that commercial AC has the highest adsorption capacity on brilliant green dye, followed by carbonised banana peel, the various MCBs and banana peel. This is likely due to the high porosity and surface area of both adsorbents (Figure 2). Comparing the percentage removal of brilliant green by MCB (3g) to MCB (5g), it can be deduced that as the mass of carbonised banana peel used in the synthesis increases, the percentage removal of brilliant green increases.



Figure 6: Adsorption of brilliant green by different adsorbents. N=5

To explain the trend, acid digestion was carried out by dissolving various MCBs in concentrated nitric acid to determine the mass of magnetite coating per gram of MCB. Interestingly, as the mass of carbonised banana peel used during magnetisation increases, mass of magnetite coating on the carbonised banana peel decreases (Figure 7), and the percentage removal of brilliant green increases (Figure 8). This suggests that the presence of magnetite coating compromises adsorption capacity of MCB by blocking pores or binding sites in MCB, resulting in less area of contact for adsorption. MCB (3.5g) was selected for subsequent tests on brilliant green as MCB (4g) to MCB (5g) were not as magnetic and not easy to retrieve using a magnet after adsorption.



Figure 7: Mass of magnetite coating in MCB (3g) to (5g). N=3



Figure 8: Adsorption of brilliant green by MCB (3g to 5g). N=5

Brilliant green is likely to be adsorbed via pi-pi interactions between aromatic rings of brilliant green and carbon, as supported by FTIR where a shift in peak (corresponding to C=C stretch of aromatic ring) from 1602 to 1583 cm⁻¹ (Figure 7) was observed. The proposed adsorption mechanism agrees with the study by Calvete et al., 2010 [24].



Figure 9: FTIR spectrum of MCB before and after adsorption of brilliant green dye

3.2.2 Lead(II) Ions



Figure 10: Adsorption of lead(II) ions by different adsorbents. * denotes significant difference based on Mann-Whitney U test at significance level of 0.05. N=5

Figure 10 shows that MCB synthesised using various masses of carbonised banana peel all have 100% or close to 100% adsorption for lead(II) ions. Commercial activated carbon (AC) has the next highest adsorption capacity, followed by carbonised banana peel, and lastly, banana peel. There is a significant difference in the results of MCB (3g) and MCB (3.5g) when compared to commercial activated carbon (Figure 10). Energy Dispersive Spectroscopy (EDS) was then conducted to find out why.

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Figure 11: EDS of (a) commercial activated carbon and (b) MCB (3g)

Lead(II) ions are adsorbed via the formation of dative bonds between electron-deficient lead(II) ions and lone pairs of electrons [25]. Figure 11 reveals that MCB has a greater oxygen content than AC, suggesting that there is more extensive dative bonding and hence, more effective adsorption of lead(II) ions. MCB (3g) was selected for future tests as it is most effective, requires the least carbonised banana peel for synthesis and yet magnetic.

3.2.3 Atrazine

Figure 12 shows that commercial AC has the highest adsorption capacity, followed by carbonised banana peel and MCBs. There is no significant difference in the results of various MCBs, but each MCB exhibited at least 95% of atrazine removal.



Figure 12: Adsorption of atrazine by different adsorbents. N=5

3.3 Adsorption Isotherms

The equilibrium concentration data of MCB, carbonised banana peel and commercial activated carbon on brilliant green and lead(II) ions were fitted into Langmuir and Freundlich isotherms (Annex A, Pages 7-8). The Langmuir isotherm is a better fit for all 3 adsorbents, suggesting that the adsorption is monolayer. The maximum adsorption capacities of the 3 adsorbents were derived by taking the reciprocal of the gradient of Langmuir plots and the results were compared with adsorbents synthesised by other researchers (Tables 2 and 3).

rilliant green			ions		
Type of adsorbent	Qmax/mg g ⁻¹	Reference	Type of adsorbent	Q _{max/mg} g ⁻¹	Reference
Commercial AC	227	This study	MCB	41.3	This study
Carbonised banana	196	This study	Commercial AC	39.2	This study
MCB	189	This study	Carbonised banana	38.8	This study
Citrus fruit peel	143	[5]	Pine cone AC	27.5	[28]
ZnO-AC	143	[26]	Coconut shell AC	26.5	[29]
Chitosan beads	135	[27]	Eichhornia AC	16.6	[30]

For brilliant green, although MCB has a lower maximum adsorption capacity than commercial AC and carbonised banana peel, its maximum adsorption capacity is higher than that of several eco-friendly adsorbents reported in literature (Table 2). For lead(II) ions, Table 3 shows that MCB has higher maximum adsorption capacity than both commercial AC and carbonised banana peel and it also outperforms various ACs synthesised by other researchers. These promising results suggest that MCB has great potential to be used as an effective adsorbent to remove dye and metal ions in wastewater treatment.

3.4 Regeneration and Reusability of Magnetic Carbonised Banana Peel (MCB)



Figure 13: Regeneration of MCB and Commercial AC for re-adsorption of brilliant green. N=5



Figure 14: Regeneration of MCB and Commercial AC for re-adsorption of lead(II) ions. N=5

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Figure 13 shows that after 3 cycles of adsorption and desorption, MCB is still able to effectively adsorb brilliant green, with the decrease in the percentage of dye removed being less than that of commercial AC. It is worth noting that in Cycle 3, there is no longer a significant difference in the results of AC and MCB. One possible explanation for this result is that the interactions between MCB and brilliant green are relatively weaker, thus allowing the dye to be easily desorbed. For lead(II) ions, Figure 14 shows that MCB can adsorb lead(II) ions for 3 cycles of adsorption and desorption without a significant decrease in the percentage of lead(II) ions removed. In contrast, there is a significant drop in percentage of lead(II) ions removed by commercial AC from Cycle 2 to Cycle 3, suggesting that MCB has a greater potential than commercial AC in adsorbing lead(II) ions over multiple cycles of adsorption and regeneration and can be implemented as a sustainable adsorbent to lower the gross cost of production as it can be effectively reused.

Another advantage of MCB over commercial AC is that due to its magnetic property, as much as 97% of MCB could be retrieved after each cycle of adsorption via a magnet (Figure 15), while only about 60% of AC could be retrieved via filtration. Hence, MCB evidently outperforms commercial AC in terms of reusability and sustainability, and is much easier to adsorption retrieve after than commercial AC.



Figure 15: Separation of MCB using a magnet

3.5 Iron Leaching Tests at various pH





The results for the iron leaching from MCB in simulated electroplating wastewater are shown in Figure 16. On average, MCB (3g) leached 0.20 mg/L and 0.08 mg/L of iron(III) ions in simulated electroplating wastewater at pH 4 and 6 respectively. According to WHO guidelines [31], approximately 0.5–50 ppm of iron can be found naturally in fresh waters. Hence the magnetite coating on carbonised banana peel does not cause significant leaching of iron into

the waters in concentrations sufficiently high that it will pose an environmental or a health hazard to humans. In addition, the concentration of iron leached out is well below NEA allowable limits of iron (1 ppm) for trade effluent discharge [32]. However, at pH 2, the magnetite coating on carbonised banana peel, formed by basic oxides, reacts with acid at pH 2 via a neutralisation reaction, resulting in a higher concentration of iron(III) ions being leached out. However, this is not a significant issue in real life as the pH of electroplating wastewater from plating baths tends to be around 5.6 [22], and the problem can be easily solved by neutralising acidic wastewater before using MCB to adsorb the heavy metal ions.

3.6 Cost Considerations

One of the greatest advantages that MCB has over commercial AC is its cost of production. Table 4 details the costs of various materials and equipment needed for the synthesis of MCB and they were derived from materials of analytical grade of at least 99% purity. The cost of 1kg of commercial AC used for this study is \$96, which is 4 times more expensive than 1kg of MCB, which costs about \$24 to produce (Table 4).

Tables 5 and 6 consider the costs of using both MCB and AC over 3 cycles of adsorption and desorption. MCB is regenerated by soaking it in 70% ethanol (for brilliant green) and deionised water (for lead(II) ions), while AC is regenerated by heating it to 800°C for 40 min to ensure that its effectiveness does not decrease over multiple cycles. Clearly, MCB is a much more affordable option than commercial AC and would be an attractive product for textile and electroplating companies looking to simultaneously increase their profits, save the environment as well as protect human health.

Table 4:	Estimated	cost of MCB	
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Synthesis of 1 kg of	Cost per
MCB	kg/SGD
Iron(III) chloride and	1.27
iron(II) sulfate	
Water	0.61
Aqueous ammonia	2.10
Carbonisation in furnace	12.00
Shipping	8.00
Total	24.18

Table 5: Cost of MCB and commercial AC in 3 cycle	es
of adsorption and desorption for brilliant green	

Adsorbent	Cycle 1	Cycle 2	Cycle 3	Total
MCB	\$24.18	\$15.78	\$15.78	\$55.74
Commercial AC	\$96.00	\$22.80	\$22.80	\$141.60
[able 6: Cost of	MCB and	commerc	ial AC in	. 3 cy
Table 6: Cost of f adsorption an	MCB and d desorptio	commerc on for lead	tial AC in d(II) ions	3 cycle

	Adsorbent	Cycle 1	Cycle 2	Cycle 3	Total
	MCB	\$24.18	\$0.10	\$0.10	\$24.38
	Commercial AC	\$96.00	\$22.80	\$22.80	\$141.60

4 Conclusion

Magnetic carbonised banana peel (MCB) was successfully synthesised via co-precipitation of magnetite onto carbonised banana peel. MCBs were effective in removing brilliant green, lead(II) ions and atrazine, with the percentage removal being more than 95%. Langmuir isotherm was a better fit for the adsorption of both pollutants, suggesting that the adsorption by MCB is monolayer. Magnetising the carbonised banana peel lowers the maximum adsorption capacity on brilliant green slightly but enhances the

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maximum adsorption capacity of lead(II) ions. The magnetic property of MCB allows for a rapid, simple and convenient way of recovery by the use of a magnet, enabling it to be recycled effectively. MCB can be reused for at least 3 cycles of adsorption and desorption without a significant drop in effectiveness, potentially saving cost and making the use of it in water treatment even more eco-friendly and sustainable.



Figure 17: Proposed model for use of MCB in treatment plants

Using programming, robotics technology and LEGO Mindstorms, a fully automated prototype was constructed to showcase how it can be used in industries for water treatment and regeneration. After adsorption, a magnet will be used to attract the MCB from the adsorption tank and transport it to a regeneration tank where the MCB can be desorbed. Such a fully automatic system facilitates the rapid and efficient retrieval of MCB, making the purification process less timeconsuming and more efficient.

Although capital is required for the initial investment in the new technology, MCB's low cost and reusability will benefit the company in the long run and it has great potential to be scaled up and utilised in industries. In the future, isotherms studies and reusability tests can be extended to atrazine. MCB can also be used in the removal of other forms of pollutants like pharmaceuticals and other heavy metal ions.

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Annexes

Annex A: Adsorption Isotherms of MCB, Carbonised Banana Peel and Commercial AC

The equilibrium concentration data obtained from adsorption isotherm studies on brilliant green and lead(II) ions was fitted into the Langmuir isotherm and Freundlich isotherm.

The Langmuir isotherm assumes that adsorbed material is adsorbed over a uniform adsorbent surface at a constant temperature, while the Freundlich isotherm assumes that the adsorption occurs on a heterogeneous surface at a constant temperature.

The linear form of Langmuir isotherm equation is given by:

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

where C_e is the equilibrium concentration of adsorbate (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 adsorption capacity (mg/g).

The linear form of Freundlich isotherm equation is given by:

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

where C_e is the equilibrium concentration of adsorbate (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 for **brilliant** green are shown below:



commercial AC

igure 23: Freundlich isotherm fo commercial AC

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The values q_m (maximum adsorption capacity) were computed from the slope of Langmuir isotherm plots of C_e (mg/L) versus C_e/Q_e (g/L). Similarly, the values of n were computed from the slope of the Freundlich plot of $log(Q_e)$ versus $log(C_e)$. The isotherm parameters obtained are summarised in table 7.

Table 7: Isotherm parameters for different adsorbents on brilliant green

	Langmuir Isotherm Freundlich Parameters Param		Isotherm	
Adsorbent			Para	Parameters
	gm (mg/g)	\mathbb{R}^2	n	\mathbb{R}^2
MCB	189	0.979	1.51	0.903
Carbonised Banana	196	0.946	1.80	0.924
Commercial AC	227	0.963	1.41	0.941

Comparison of the coefficient of determination (R^2) of the linearized forms of both isotherms suggests that the Langmuir model yields a better fit for the equilibrium adsorption data of brilliant green onto MCB, carbonised banana peel and commercial AC. The maximum adsorption capacity, q_m , of AC is the highest, followed by carbonised banana peel and MCB.

The Langmuir and Freundlich isotherm plots for **lead(II)** ions are shown below:



The isotherm parameters obtained are summarised in table 8:

	Langmuir Isotherm		Freundlich Isotherm		
Adsorbent	Paran	Parameters		Parameters	
	g _m (mg/g)	R ²	n	R ²	
MCB	41.3	0.961	5.97	0.931	
Carbonised Banana	38.8	0.980	2.61	0.953	
Commercial AC	39.2	0.963	4.83	0.914	

Comparison of the coefficient of determination (R^2) of the linearized forms of both isotherms suggests that the Langmuir model yields a better fit for the equilibrium adsorption data of lead(II) ions onto MCB, carbonised banana peel and AC. The maximum adsorption capacity, q_m , of MCB is the highest, followed by commercial AC and carbonised banana peel.

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