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Removal of pollutants from wastewater using activated carbon from durian peel

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Abstract. The efforts in reducing food waste can lead to better water management with less land required for food production and indirectly having a positive impact on the livelihoods and the environment. In order to support the practice of reducing food waste in Brunei Darussalam, one must find ways to recycle or reuse them, particularly waste generated from fruit peels. This paper investigates the feasibility of utilising Durian (*Durio zibethinus)* rinds to remove pollutants in wastewater, namely Chemical Oxygen Demand (COD), and Nitrates by the process called adsorption. Durian is a local fruit and has high consumption in the country. Hence, it produces a large amount of waste, particularly in the form of peels or rinds. In the present research, the fruit peels are treated by dehydration and carbonisation method prior to utilisation. The variable parameters will be investigated such as the effect on particle size, adsorbent dosage, pH, contact time, and agitation. The maximum COD removal percentage was found to be 68.2 % with an adsorption capacity of 690 mg/g. Overall, carbonised samples gave a better outcome than dehydrated samples. Possibly due to the presence of a unique pore structure in carbonised samples that allows organic matter to be adsorbed on the surface.

1. Introduction

Having access to clean water is very important to humans for their basic needs. Therefore, it is critical to treat and monitor the water quality to ensure that the water supply is clean and safe. Brunei's growing population increases water utilization, which leads to an increase in wastewater. Wastewater is water that has been used for domestic uses such as toilets, kitchens and gardening etc. These waters usually contain pollutants such as Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Suspended Solids (TSS), Pathogens, Nutrients and Heavy Metals, which may include arsenic, cadmium, chromium, copper, lead, mercury, nickel, silver and zinc [1].

Many wastewater treatment technologies have been developed to reduce water pollution [2]. There are seven common wastewater treatments; coagulation/flocculation, sedimentation, filtration, disinfection, sludge drying, fluoridation, pH Correction, electro-chemical technique [3]. Despite that, there are many shortcomings in utilizing the processes mentioned, such as high maintenance and operational costs, generation of toxic sludge, the treatment has a complicated procedure and many more. Due to this, adsorption is considered a better alternative because of its convenience and simplicity of the operating process [4]. Therefore, this paper will be focusing on the adsorption of pollutants in wastewater using Activated Carbon (AC) derived from Durian peel.

AC is carbon that has been treated with oxygen to open up millions of tiny pores between the carbon atoms. This treatment resulted in a very porous substance that has a surface area of 200-3000 m^2/g. There are two main methods of contaminants removal utilizing AC. The first method is carbon filtering by chemical absorption, where a bed of activated carbon is used to remove pollutants. The carbon particles or granules provide a large surface area or pore structure where the contaminants will be exposed to the active sites within the filter media. The second method is by using the adsorption method, where the pollutants present in water are trapped inside the pore structure of the carbon substrate.

AC can be obtained from a high carbon content organic compound such as by processing fuel (coal, lignite, petroleum pitch, tar) or from biomass (sugarcane, rice husks, coconut husks, agriculture/food/fruit wastes) [5]. Due to increasing concerns about the extracting of non-renewable resources, this research will be concentrating on bioorganic-derived activated charcoal.

There are many potentials that can be utilized from the fruit wastes such as composting, the feedstock of bioenergy or biogas production, increasing soil fertility, production of enzymes and applications in domestic and wastewater treatment [1]. Brunei has an abundance of seasonal fruits, such as Durian, *Durio zibethinus* and Rambutan, *Nephelium lappaceum* etc. When it is in season, the waste from these two fruits are high as the fruit has a high rind to edible fruit ratio. Rambutan consists of 47.5 % skin [6] and Durian consists of up to 70 % skin [7]. As the rinds are high in cellulose content and very fibrous, it is very attractive to be used to make activated charcoal for adsorbent in the wastewater treatment plant.

This research aims to optimize the removal efficiency of chemical oxygen demand of wastewater using AC by adsorption using Durian Peels as a bio-adsorbent. The aim can be achieved by synthesising the carbonised Durian Peels using different temperatures and different carbonisation time. The efficiency can be further improved by manipulating the dosage of carbonised Durian Peels for the adsorption, using different particle sizes of the adsorbents, manipulating the pH of wastewater, changing the adsorption reaction time (contact time) and using different agitation speeds.

2. Materials and methods

Durian rinds were obtained locally from the Pasar Pelbagai Guna, Gadong, Brunei. The wastewater effluent is obtained from the Gadong Wastewater treatment plant, Jabatan Kerja Raya, Ministry of Development, Brunei. The chemicals used for this experiment are supplied by Hach Company, USA. These chemicals are Sodium hydroxide (NaOH) and Phosphoric acid (H_3PO_4). The pH of the wastewater was measured by a portable multiparameter (HACH HQ40D) analyser. All the experiments were run by batch adsorption process and the amount of COD were analysed using split beam spectrophotometer (Hach DR 3900).

2.1 Adsorbent preparation

The durian peels were cut into small pieces and washed using tap water a few times until there was no more dirt observed. After that, the rinds were rinsed with distilled water. The rinds were then sun dried until the weight of the rinds was constant. The dried rinds were stored in an airtight container.

2.2 Carbonisation process and optimise the parameters optimisation

The samples were then divided into three parts and charred in a muffle furnace at 200 °C, 250 °C for 3 hours and 4 hours and 350 °C for 3 hours. After that, the samples were put into a desiccator at least overnight to cool, ensuring there was no condensation and preservation of the sample.

Figure 1. Dehydrated Durian Peels

Figure 2. Charred Durian Peels

The samples were then crushed using a mechanical blender for 20 seconds and sieved by using a sieve with 425µm, 300µm, 212µm, and 150µm sizing. The process is done by using a mechanical sieve shaker. This process is done for 10 minutes. The samples are then collected and stored in different containers.

2.3 Effect of the adsorbent dosage on adsorption capacity

Adsorbents that were used for this experiment were synthesized at 250 °C for 3 hours with a particle size of 212 µm. The adsorbent dosage was varied from the amount of 0.01 g to 0.4 g in 100 ml of wastewater. The mixture was agitated at 210 rpm using a magnetic stirrer for 30 minutes.

2.4 Effect on the particle size on adsorption capacity

The particle size used for these experiments were varied by using adsorbents of size 150 µm, 212 µm, 300 µm and 425 µm. The batch adsorption experiments were done on adsorbent synthesized at 250 °C for 3 hours with an adsorption dosage of 0.02 g mixed into 100 ml of wastewater. The mixture was agitated at 210 rpm for 30 minutes for every experiment.

2.5 Effect of wastewater pH on adsorption capacity

For this experiment, the pH was manipulated by adding 5 N NaOH for increasing the pH and H_3PO_4 to make the wastewater acidic. The adsorbent used was the size of 212 µm synthesized at 250 °C for 3 hours, the dosage 0.02 g mixed into 100 ml of wastewater. For each experiment, the mixture was mixed at 210 rpm for 30 minutes.

2.6 Effect of contact time on adsorption capacity

The experiment on the effect of contact time was done by varying the contact time from 15 minutes to 60 minutes with 15 minutes increments. The adsorbent used was at the size of 212 µm synthesized at 250 °C for 3 hours, the dosage 0.02 g mixed into 100 ml of wastewater. For each experiment, the mixture was mixed at 210 rpm for 30 minutes.

2.7 Effect of Agitation on adsorption capacity

For this experiment, the magnetic stirrer was varied from 50 rpm, 210 rpm, 360 rpm and 420 rpm. The mass of adsorbent used is 0.02 g in 100 ml of wastewater and 212 µm synthesized at 250 °C for 3 hours. The experiment was run in batches for 30 minutes.

2.8 Measurement of COD from wastewater

The mixture is then filtered using filter paper after every treatment to remove the adsorbent from the wastewater solution. Then the mixture is stirred for 1 minute to ensure that the sample is homogenized. At the same time, the test vial of Hach TNT 821 is inverted several times to mix the settled solid in the vial. Then 2 ml of the sample is pipetted into the vial. The vial is inverted several times to mix the mixture and then put into DR200 for digestion for 2 hours at 150 °C. After that, the mixture is cooled until the temperature reaches 120 °C and the vial is inverted several times, then let cool until the temperature decrease to room temperature. The cooled vial is put into the split beam spectrophotometer (Hach DR 3900) cell holder and COD reading is taken in mg/L.

The percentage removal of COD can be calculated as follow:

$$\%\ COD\ removal = \frac{C_i - C_f}{C_i} \times 100\% \qquad \text{Eq. (1)}$$

Adsorption Capacity (q_t) of the AC can be calculated as follow (Mukhlish *et al.*, 2014):

$$q_t = \frac{(C_i - C_f)V}{W} \qquad \text{Eq. (2)}$$

Where C_i is the initial COD concentration of wastewater (mg L^{-1}), C_f is the final COD concentration after adsorption (mg L^{-1}), V is the Volume of wastewater (L) and W is the mass of the AC used (g).

3. Results and discussions

3.1 Carbonisation process and optimise the parameters

The results obtained from the research, when using a carbonized durian peel synthesized at 200 °C, 250 °C for 3 hours and 4 hours and 350 °C for 3 hours and compared the results as shown in Figure 3, as the temperature of the carbonisation was increased from 200 °C to 350 °C. The adsorption experiments were run for 30 minutes for each parameter. We can observe that the percentage of COD removal increased from 61.59% to 65.46%. However, comparing the adsorption capacities between the temperatures, it is evident that the carbonized durian peel synthesized at 200 °C shows a lower adsorption capacity of 618.93 mg/g than carbonized durian peel synthesized at 350 °C, 676.5 mg/g.

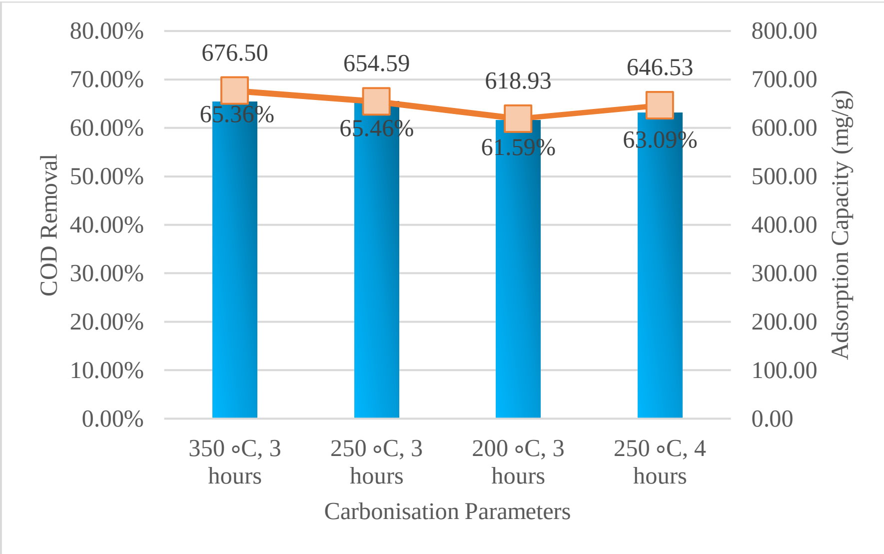

Figure 3. COD Removal efficiency with different carbonisation parameters

It is clear that the performance of the adsorption can be manipulated by synthesizing the durian peels by using different carbonisation parameters. The results show that the performance of the AC from the durian fruit peel increases as the carbonisation temperature increases. For all the experiments, we used the AC particles produced at 250 °C for 3 hours of carbonization.

3.2 Effect of adsorbent dosages

The solid to wastewater ratio or the dosage of adsorbent needed for the batch adsorption study is essential in determining the capacity of the adsorbent. For this research, the effect of increasing the adsorbent dosage can be shown in Figure 4. The experiments were run for 30 minutes using 212 μm adsorbent particle size. It can be observed that, in general, the percentage of COD removal with an increase in adsorbent dosage is relatively constant with the COD % at the range of 51% to 44% for adsorbent dosage of 0.1 g L^{-1} to 1.0 g L^{-1}. Whereas, for the adsorption capacity, there is a more drastic drop in adsorption capacity as the dosage increases from 0.1 g L^{-1} to 1.0 g L^{-1}.

The percentage of COD removal increases from 51 % to 52 % when the adsorbent dosage is increased from 0.1 g L^{-1} to 0.2 g L^{-1}. Then, as the adsorption dosage is increased from 0.2 g L^{-1} to 4 g L^{-1} the percentage of COD removal is then decreasing gradually from 51 % to 15 % at an adsorption dosage of 4 g L^{-1}. The increase in the percentage of COD removal for dosage 0.1 g L^{-1} to 0.2 g L^{-1} may be the result of increasing binding sites available on the adsorbent surface. However, a further increase in the dosage decreases the COD removal percentage. As for the adsorption capacity, it decreases significantly from 734 mg/g to 381 mg/g as the dosage increases from 0.1 g L^{-1} to 0.2 g L^{-1}. After that, the adsorption capacity of the carbonized durian peel decreases gradually as the dosage increases from

0.2 g L^{-1} to 4 g L^{-1}. These results are similar to the research done by Mahdavi et al. on COD removal from landfill leachate using the carbonized walnut shell. This phenomenon occurs as a result of a partial aggregation of the adsorbent particles. Therefore, the available adsorption sites cannot be saturated with the adsorbates [8].

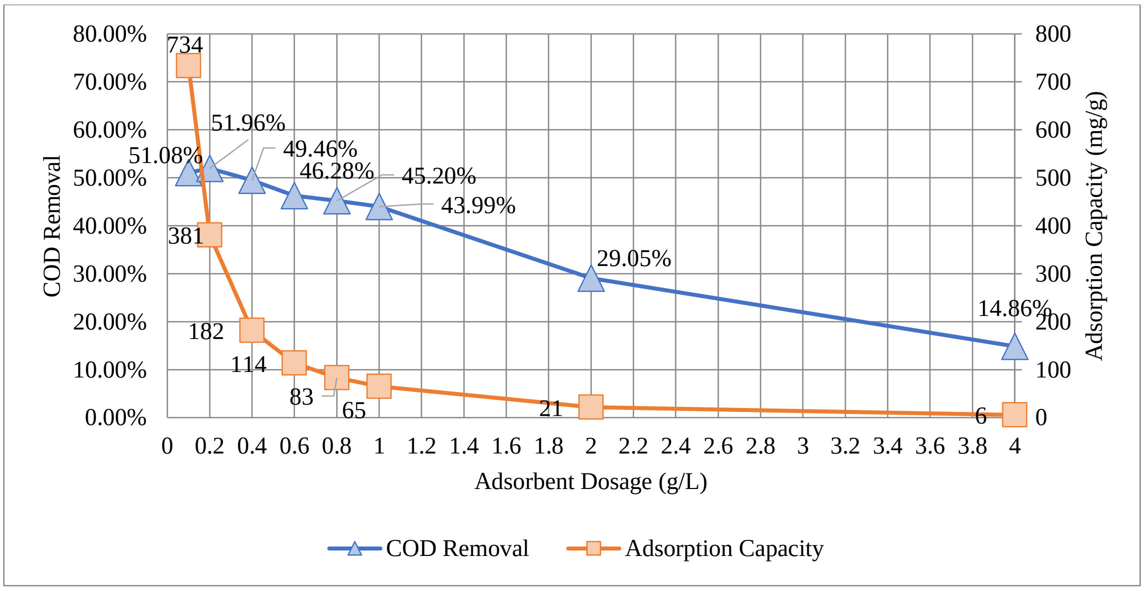

Figure 4. COD removal efficiency when varying the adsorbent dosage

From a study done by Lazim et al., on using durian peel as a potential adsorbent for Bisphenol A removal in an aqueous solution, their team had achieved a lower adsorption capacity of the durian peel at the adsorbent dosage was increased [9]. They have suggested that the adsorption efficiency is highly dependent on the amount of adsorbent and the volume. The decrease in the adsorption capacity with an increase in the adsorbent dosage suggests that with the increasing mass of adsorbent, the amount of pollutant per unit mass was also reduced. For the remaining experiments, 0.2 g of adsorbent in 1 L of wastewater was used as it shows the highest COD removal with good enough adsorption capacity.

3.3 Effect of adsorbent particle size

The effect of the adsorbent particle size can be shown in Figure 5. There is not much difference in the COD removal percentage for the particle sizes 150 μm to 425 μm but the percentage is increasing in the range of 64 % to 69 %. However, a slight increase in the COD removal percentage from 64 % to 65 % can be observed when the adsorbent particle size was increased from 150 μm to 212 μm. Then the COD removal percentage increased from 65 % to 69 % when the adsorbent particle size was increased from 212 μm to 300 μm and stayed around relatively constant when 425 μm adsorbent particle size was used.

Consequently, a similar trend was observed for the adsorption capacity as the COD removal percentage. There is not much difference in the adsorption capacity for the particle sizes 150 μm to 425 μm but the adsorption capacities are increasing in the range of 650 mg/g to 685 mg/g However, a slight increase in the adsorption capacity from 649 mg/g to 686 mg/g can be observed when the adsorbent particle size was increased from 150 μm to 212 μm. Then the adsorption capacity increases from 655 mg/g to 686 mg/g when the adsorbent particle size was increased from 212 μm to 300 μm and stays constant when 425 μm adsorbent particle size was used.

Therefore, for this research, it can be said that the particle size of durian peel carbonized at 250 °C does not have any significant influence on the COD removal from wastewater. From research done by Devi's team on the removal of COD and BOD from wastewater using avocado peel carbon and commercial activated carbon, they have found that there was a significant influence in the percentage reduction of COD and BOD concentration using both types of carbons. They used the particle size range from 0.75-0.25 mm. As per their results, the smaller particles have a higher surface area compared to the larger particles was the reason behind the decrease in COD and BOD reduction. Therefore, implying in direct relation to the adsorption capacity based on the net surface area as smaller particles at any mass of the carbon powder has a larger surface area than larger particles. Therefore, higher adsorbent – adsorbate interaction on the active site of the carbon occurred more in the smaller particles.

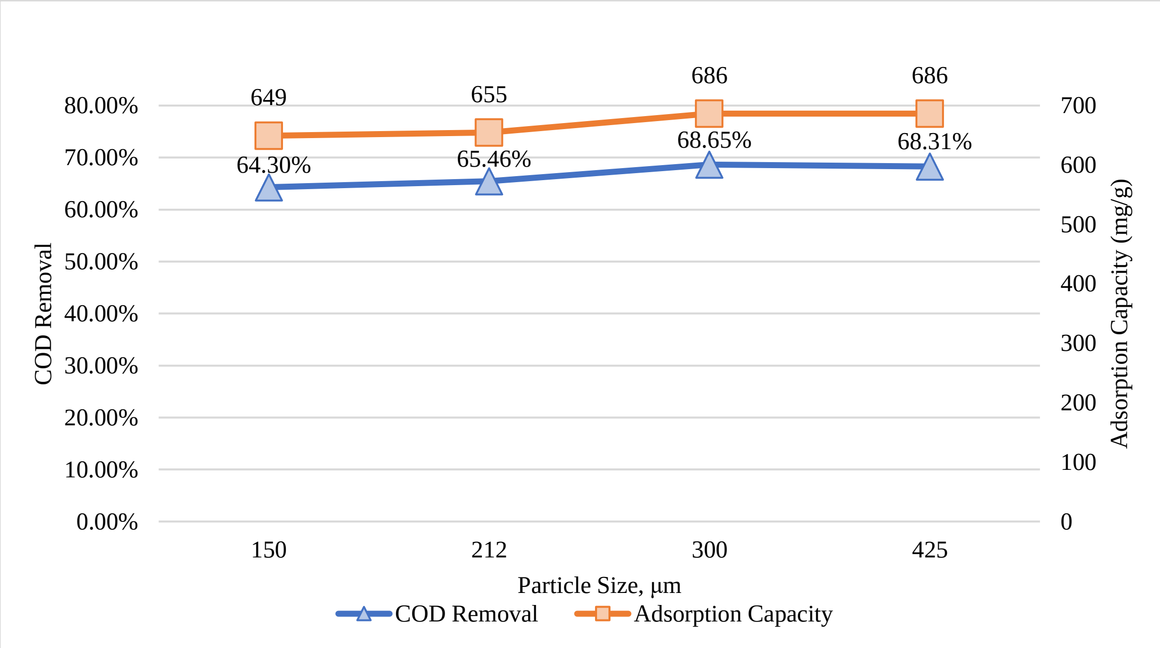

Figure 5. COD Removal efficiency with different particle sizes

3.4 Effect on pH on COD removal efficiency

The effect of COD removal with different pH can be shown in Figure6. The pH for these experiments were pH 1.5, 5.8, 7.8, 10 and 13. From the graph, the COD removal percentage is increasing as the pH increases. At pH 1.5, the carbonised durian peel was able to remove 48 % of the COD and the COD removal increased to 50 % and 52 % at pH 5.8 and 7.8, respectively. Then, the COD removal percentage decreased when the pH was increased to pH 10 to 42 %. When the pH is further increased to higher alkalinity at pH 13, the COD removal percentage reduces drastically and it was observed that the COD in the wastewater increases, thus having a negative value in COD removal at -16 %.

The pH of the solution affects the degree of ionisation and the surface charge of the adsorbent. According to Mahdavi et al., the COD is amongst the pollutants affected by the pH of the solution [8]. The wastewater influent has a fluctuation of pH as it receives both industrial and domestic wastewater. The pH is also influenced by rainwater and runoffs [10]. From the wastewater samples taken from Gadong wastewater treatment plant, the wastewater is usually in the range of pH 6 to pH 8. Therefore, it is important to find out the effect of pH in COD removal and the sensitivity of the results in different pH of the water to ensure that the highest efficiency of the pollutant removal is achieved.

As for the adsorption capacity of the carbonised durian peel, the resulting trend is similar to the COD removal percentage. At pH 1.5, the carbonised durian peel has an adsorption capacity of 312 mg/g. When the pH was increased to 5.8 and 7.8, the adsorption capacities also increased to 341 mg/g and 354 mg/g, respectively. A further increase in the initial wastewater pH to 7.8 reduces the adsorption capacity to 287 mg/g. In the same case as the COD removal, an increase in pH to higher initial wastewater pH to pH 13 causes a drastic decrease in adsorption capacity to - 122 mg/g which had suggested that the wastewater contains a higher COD compared to the initial reading.

From the results, it can be concluded that the initial wastewater pH has a significant effect on the performance of COD removal. It may suggest that pH of the solution may affect the physiochemistry of the active sites on the adsorbents and thus making the characteristic of the adsorbent differ at different initial wastewater pH. The COD removal in this paper shows that the carbonised durian peel favours slightly acidic conditions to neutral pH. This may be due to the electrostatic attraction between the positively charged adsorbent surface and the negatively charged COD.

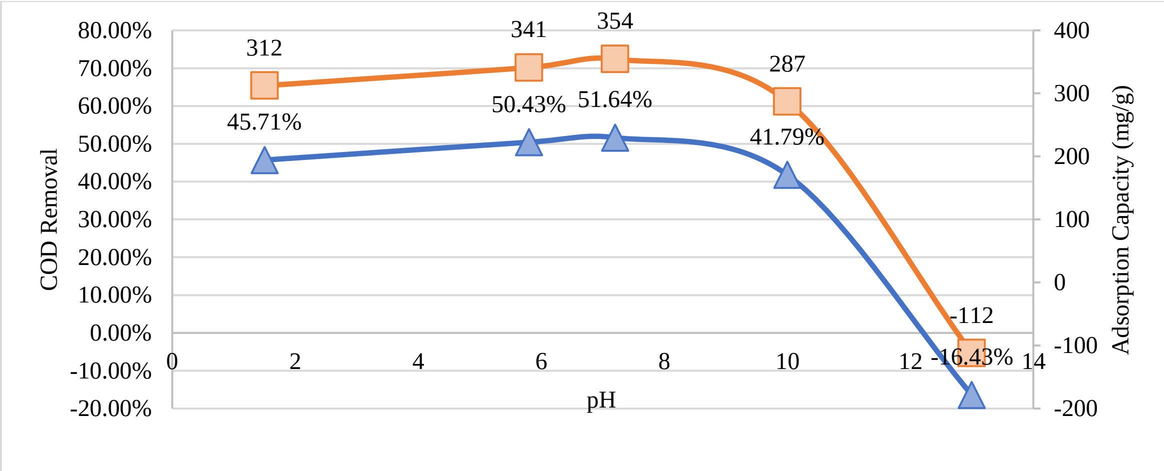

Figure 6. COD Removal with different pH of wastewater

When comparing the results of using Durian peel as an adsorbent by Lazim et al. on removing Bisphenol A, the removal favours Bisphenol an acidic solution. They suggested that the sorption capability of the sorbent (durian peel) did not increase by an increase in the negative charge of the initial Bisphenol A solution. Their study also suggested that as the initial pH solution increases, the electrostatic repulsion between Bisphenol A and the sorbent becomes stronger. Due to the greater electrostatic repulsion from OH^- ions, the binding affinity of the sorbent is lower as the overall charge of the sorbent surface gets more negative [8]. Therefore, this led to a low percentage of removal and adsorption capacity of Bisphenol A by the adsorbents under alkaline conditions [9]. From this, we can observe a similar result for this research which can be implied that the adsorption using Durian peel is least favourable in a higher initial pH of solution.

From Mukhlish et al research on adsorption and desorption study of the uptake of Indosol Dark-blue GL dye from aqueous solution by water hyacinth roots powder, the adsorption favours low acidic initial solution at pH4. At higher pH, greater than the point of zero charges, there is a weak physical force such as the van der Waals attraction which highly influences the adsorption process [11]. This suggests that for this research, it can be assumed that the point of zero charges for the carbonised durian peel carbonised at 250 °C for 3 hours has the point of zero charges of around pH 6 to 9 based on the peak observed in Figure6. However, a more accurate point of zero charges can be obtained by conducting a surface charge experiment on the adsorbent using titration and a plot graph of the surface charge against the pH of the solution.

3.5 Effect of contact time

Contact time is the time the wastewater is mixed or agitated with the adsorbent to reach its equilibrium. The adsorbent is effective if the rate of it reaching its equilibrium is fast and has a high removal of pollutants. Investigating this parameter is essential to know the optimal contact time of the reaction for the designing process of the adsorption reactor, which directly affects the cost of the building and its operation.

The percentage removal of the COD in wastewater increases with increasing contact time of the adsorption reaction and then stays relatively constant once it reaches its equilibrium. This can be observed in Figure7. When the adsorption reaction for a batch process was run for 15 minutes, the COD removal percentage is at 49.12 %. The COD removal percentage was then increased to 65.5 % as the experiment ran longer to 30 minutes. After that, the percentage of COD removal stays relatively constant of variation of 0.5 % as the experiments were run longer to 45 and 60 minutes. Therefore, the adsorption reaction of COD removal by using durian peel carbonised at 250 °C for 3 hours is reached at 30 minutes.

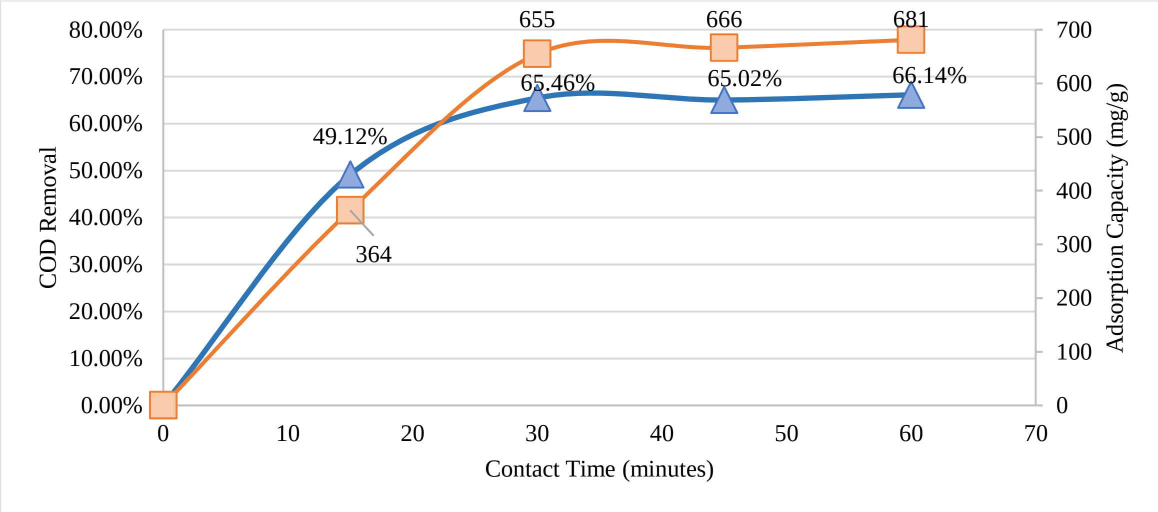

Figure 7. COD Removal with different contact time (minutes)

A similar trend is also observed for the adsorption capacity, where the adsorption capacity increases as the contact time increases. The adsorption capacity then stays relatively constant after it reaches its equilibrium. For the adsorption reaction that ran for 15 minutes, the adsorption capacity of the adsorbent was observed to be 364 mg/g and then increased to 655 mg/g when the adsorption reaction was run for 30 minutes. Then, as the adsorption reaction was run longer to 45 minutes and 60 minutes, the adsorption capacity was observed to be 666 mg/g and 681 mg/g, respectively.

The increase in COD removal and adsorption capacity increased for the first 30 minutes for the carbonised durian peel. This may be due to a higher affinity towards the pollutants as the morphology effect of the adsorbent surface and the readily available binding site for the adsorbates on the adsorbent. As the reaction was run longer than 30 minutes, the adsorption becomes exhausted and a stationary state of the carbonised durian peel has been achieved, which suggests that the adsorbent binding sites were saturated [9].

3.6 Effect of agitation speed

The results of the effect of increasing the agitation speed can be shown in Figure8 where all parameters were kept constant by using durian peels that were carbonised at 250°C for 3 hours with the particle size of μm, adsorption reaction of 30 minutes with an adsorbent dosage of 0.2 g L^{-1}.

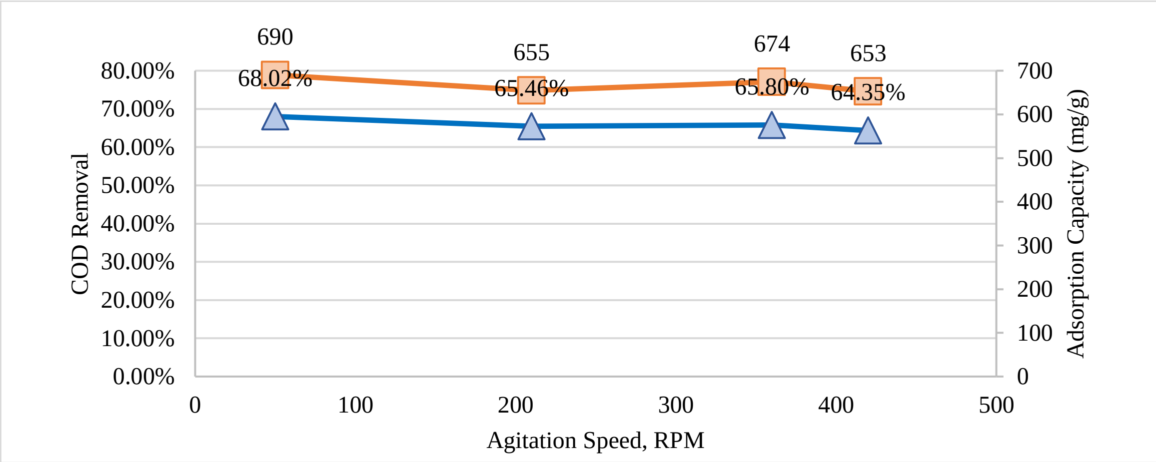

Figure 8. Effect of agitation speed on the COD removal

The COD removal and adsorption capacity reduce slightly as the agitation speed is higher. At 50 rpm, the COD removal percentage is at 68 % and dropped to 65.5 % when the speed was increased to 210 rpm. The COD percentage removal was observed to increase to 65.8 % and dropped again to 64 % as the speeds were increased to 360 rpm and 420 rpm, respectively. While for adsorption capacity, at 50 rpm, the adsorption capacity is at 690 mg/g and dropped to 655 mg/g when the speed was increased to 210 rpm. The adsorption capacity was observed to increase to 674 and dropped again to 653 as the agitation speeds were increased to 360 rpm and 420 rpm, respectively.

The decrease in the efficiency of the carbonised durian peel was observed as the agitation speed gets faster. From Figure 7, the equilibrium of the adsorption reaction was achieved at 30 minutes of contact time. So, at 30 minutes, all the adsorbent active binding sites were saturated. As the agitation speed gets faster, the adsorbates that were loosely bound onto the active site may be detached and re-enter the wastewater mixture [12-14]. Therefore, this may explain the increase in the final COD reading in the mixture.

In the overall research, the optimised results for each parameter were tabulated in Table 1 and were compared with the parameters with the highest COD removal percentage and the highest adsorption capacity.

Table 1. The optimum conditions for each parameter

Parameters	Best Parameters	Highest COD Removal Percentage	Highest Adsorption Capacity
Carbonisation	350°C for 3 hours	250°C for 3 hours	250°C for 3 hours
Adsorbent Dosage	0.2 g L^{-1}	0.2 g L^{-1}	0.2 g L^{-1}
Adsorbent Particle Size	300 µm	212 µm	212 µm
Wastewater pH	7.2	neutral	Neutral
Mixture Contact Time	60 minutes	30 minutes	30 minutes
Agitation Speed of the Mixture	50 RPM	50 RPM	50 RPM
COD Removal	67.80 %	68.65 %	68.02 %
Adsorption Capacity	671 mg/g	686 mg/g	690 mg/g

4. Conclusions

Activated carbons were prepared from a durian shell at different activation temperatures and timings. Different parameters' effects like adsorbent dosage, particle size, pH, contact time, and agitation on adsorption capacity have been optimized. The results for each parameter were discussed and were compared with the parameters with the highest COD removal percentage and the highest adsorption capacity. The maximum COD removal percentage was found to be at 68.2 %, with an adsorption capacity of 690 mg/g.

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