CHEMICAL OXYGEN DEMAND (COD) REDUCTION IN INDUSTRIAL WASTEWATER USING ACTIVATED CARBON PREPARED FROM FOXTAIL PALM FRUIT

by:

NAJIEHAH BINTI MOHD AKHIR

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DECLARATION

I declare that this thesis entitled 'Chemical Oxygen Demand (COD) Reduction in Industrial Wastewater Using Activated Carbon Prepared from Foxtail Palm Fruit' is the result of my own research except as cited in the references. The degree has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ___________________________

Name : NAJIEHAH BINTI MOHD AKHIR

Date : 8 JANUARY 2020

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Chemical Oxygen Demand (COD) Reduction in Industrial Wastewater Using Activated Carbon Prepared From Foxtail Palm Fruit

ABSTRACT

Industrial wastewater has become one of the major sources that contributes to pollution in water bodies. The water discharge from the industrial activities carries out pollutant which can cause several impacts towards the human and ecosystem. Textile industry specifically have great amount of chemicals input which eventually affect the value of acceptable conditions for discharge of industrial effluent especially the value of chemical oxygen demand (COD). Thus, this research was carried out to evaluate the reduction of chemical oxygen demand (COD) in industrial wastewater from the adsorption of activated carbon derived from *Wodyetia bifurcata* fruit or commonly known as foxtail palm fruit. The adsorption study was done on the treatment of wastewater collected from the textile industry located in Kota Bharu, Kelantan. Chemical activation with nitric acid (HNO_3) was used for the conversion of foxtail palm fruit powder to activated carbon. A series of batch adsorption were carried out to study the effect of adsorbent dosage, contact time and concentration of wastewater on the percentage of COD reduction. The optimum condition for the adsorption process in this study was obtained at 90 minutes of contact time with the concentration of 5 % using 2 g of activated carbon. The percentage reduction of COD value obtained at these conditions was 71.23 %.

Pengurangan Permintaan Oksigen Kimia (POK) Dalam Air Sisa Industri Menggunakan Karbon Aktif Daripada Buah Pokok Ekor Tupai

ABSTRAK

Air sisa industri telah menjadi salah satu sumber utama yang menyumbang kepada pencemaran air. Pelepasan air dari aktiviti perindustrian membawa kepada pencemaran yang boleh menyebabkan beberapa kesan terhadap manusia dan ekosistem. Industri tekstil khususnya mempunyai banyak input kimia yang akhirnya memberi kesan kepada nilai syarat yang boleh diterima untuk mengeluarkan efluen perindustrian terutamanya nilai permintaan oksigen kimia (POK). Oleh itu, kajian ini dijalankan untuk menilai pengurangan permintaan oksigen kimia (POK) dalam air sisa industri dari penjerapan karbon aktif yang dihasilkan daripada buah *Wodyetia bifurcata* atau dikenali sebagai buah pokok ekor tupai. Kajian penjerapan dilakukan terhadap rawatan air sisa yang dikumpulkan dari industri tekstil yang terletak di Kota Bharu, Kelantan. Pengaktifan kimia dengan asid nitrik (HNO_3) digunakan untuk penukaran serbuk buah pokok ekor tupai kepada karbon aktif. Satu siri penjerapan telah dijalankan untuk mengkaji kesan dos penjerap, masa sentuhan dan kepekatan air sisa pada peratusan pengurangan POK. Keadaan yang paling optimum untuk proses penjerapan dalam kajian ini diperoleh pada masa 90 minit dengan kepekatan 5% menggunakan 2 g karbon aktif. Peratusan pengurangan nilai POK yang diperoleh pada keadaan ini adalah 71.23%.

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LIST OF ABBREVIATIONS

AC	Activated Carbon
AN	Ammoniacal Nitrogen
BOD	Biological Oxygen Demand
COD	Chemical Oxygen Demand
DO	Dissolve Oxygen
DOE	Department of Environment
$FeCl_3$	Ferric Chloride
H_2O_2	Hydrogen Peroxide
HNO_3	Nitric Acid
TSS	Total Suspended Solids
WQI	Water Quality Index

LIST OF SYMBOLS

mg/L	milligram per Litre
%	Percentage
m^2/g	metre square per gram
mL/g	milli Litre per gram
mm	millimetre
g/mL	gram per milli Litre
g	gram
mL	milli Litre
g/L	gram per Litre
ºC	degree Celsius
µm	micro meter
rpm	revolution per minute

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The development of chemicals, pharmaceutical and industries had released variety and a great amount of contaminants into the water bodies. Industrialisation in specific had become one of the factors that contribute to the environmental degradation thus create the pollution of water, soil and air. For instance, industrialisation from the textile industry had contributed to water pollution as the contaminants from the production process of textile will be released to the water bodies in the form of wastewater. These contaminants will eventually affect the amount of organic and inorganic compounds in the water bodies (Fawaz *et al*, 2013). As a consequence from this condition, the level of biochemical oxygen demand (BOD), chemical oxygen demand (COD) and other parameters of water quality assessment were affected.

Several treatment methods of physical, chemical and biological treatment had been proposed to remove or treat organic compounds from the wastewater. However, the effectiveness of these methods were compromised due to several factors. The high cost of preparation and a long period of experiments were some of the factors that limit the success of the treatments thereby giving the optional to practice adsorption method using activated carbon. Activated carbon is a microporous, homogenous structure with a high surface area which is widely used in the industrial process. The preparation cost of the activated carbon is very low making it to be a desirable material for treatment of wastewater (Pradhan, 2013). Activated carbon prepared from biomaterials have been a great interest in order to find inexpensive and effective alternatives to the existing commercial activated carbon (Thomas & George, 2018). Commercial activated carbon are produced from coal, lignite, wood peat, petroleum coke, bone char and other biomass sources making it to be more costly compared to the activated carbon from agricultural waste (Saleem *et al*., 2019).

According to Zhenisbekovna *et al* (2016), active carbon can be used as an adsorbent for cleaning of environmental pollutants of various composition. Their study shows the adsorptive capacity of the active carbon depends on the porosity and surface chemistry of the active carbon. Chemical and physical activation methods along with the microwave radiation method are widely used methods in the preparation of active carbon. Due to the porous structure, large surface area and high adsorption capacity, activated carbon is often used for remediation of pollutants in soil and water *(*Sabir *et al*, 2014).

In this study, wastewater collected from textile industry was treated with the activated carbon to identify its efficiency in reducing the COD value. *Wodyetia bifurcata* fruit or commonly known as foxtail palm fruit was used as the activated carbon due to its characteristics that have the potential to be an adsorbent. Foxtail palm fruit can be found abundantly as its trees are fast-growing and can reproduce a great amount of fruits. Its characteristics which are economically cheap, easily available and highly adsorptive can be considered as a good material for activated carbon for treatment of wastewater (Daud *et al*, 2016).

1.2 Problem Statement

Industrial wastewater has become one of the major sources that contributes to pollution in water bodies. According to Lahot and Tiwari (2016), there has been a huge amount of industrial wastewater that has been discharged into the water bodies like rivers, coastal areas and lakes. This has caused negative impacts towards the environment especially the ecosystem and human's health. Malik *et al*. (2017) in their study had mentioned the major sources of water pollution were domestic sewage, industrialisation, population growth and many more. Waste discharged from the industries' sectors caused the water bodies like river to be polluted thereby affecting the quality of the water in terms of odour and colour. As a result from this pollution, the flora and fauna around the rivers have been affected which resulted in their habitats to decline.

In research done by Qing and Yang (2016), the discharge of pollutants from the by-products of rapid economic development and industrialization has caused widespread of toxic pollutant, organic pollutant and eutrophication. These have affected the value of water quality parameters like biological oxygen demand (BOD) and chemical oxygen demand (COD). As a result from this problem, it has also led to increase in health risk as water is the source of living for both human and animals.

The increase of COD value in water will decrease the amount of dissolved oxygen that is available for aquatic organisms. This will cause harmful effects towards the organism as they are living in the area. It is very important to reduce the amount of COD value in wastewater to avoid or reduce the impact from this problem. Hence, in order to solve this problem, many methods were introduced to treat the wastewater for the reduction of COD value. One of the methods was adsorption using activated carbon which has its special characteristics that enable it to be an excellent and versatile adsorbent. Activated carbon can be used as adsorptive removal of colour, odour and taste. It also can be used to treat undesirable organic and inorganic pollutants especially in industrial wastewater (Bansal & Goyal, 2005).

The abundance of agricultural waste in Malaysia has been a great opportunity for the production of activated carbon from the agricultural waste as approximately, Malaysia produces 168 million tonnes of waste from the sector per year (Ozturk *et al*. 2017). Foxtail palm which is a fast growth tree with abundance of fruits has contributed to the amount of waste generated, thus providing the intention to reduce the waste by preparing activated carbon from its fruits. Foxtail palm fruit is a suitable material to prepare activated carbon due to its properties which are low ash content and high fixed carbon content (Munasir, 2018). Thus, the potential of activated carbon prepared from foxtail palm fruit for reduction of COD value in industrial wastewater especially textile industry was studied.

1.3 Objectives of Study

The objectives of this study are:

i. To determine the COD value in textile wastewater.
ii. To optimize the reduction of COD value from textile wastewater using activated carbon prepared from foxtail palm fruit.

1.4 Scope of Study

This study was conducted to evaluate the reduction of COD treated by the activated carbon prepared from foxtail palm fruit. Batch adsorption experiment regarding three different parameters were performed to identify the optimum value of each parameter for COD reduction. The three parameters were the dosage of adsorbent, contact time and concentration of wastewater. UV-Visible spectrophotometer was used to analyse the parameters involved to compare results before and after the treatment process.

1.5 Significance of Study

This study was conducted to evaluate the potential of *Wodyetia bifurcata* fruit or foxtail palm fruit as activated carbon for the reduction of chemical oxygen demand (COD) in industrial wastewater. Foxtail palm fruit was used in this study as it can be found abundantly and are able to help in reducing the waste from agricultural sector as it can be used as activated carbon to treat wastewater. In this study, a batch adsorption experiment was done to determine the optimum conditions for the activated carbon to act as adsorbent to its optimum value. Three different parameters which were adsorbent dosage, concentration of wastewater and contact time were done to obtain the best condition combination to optimize the COD reduction. Hence, the intention of this study was to identify the optimum conditions for COD reduction using foxtail palm fruit so it will provide optimum resources for further research.

CHAPTER 2

LITERATURE REVIEW

2.1 Water Quality

Water quality standards are important to ensure there is a standard of quality water for a designated purpose. According to Roy (2019), water quality can be defined as the chemical, physical and biological properties of water. Physical properties of water quality are the turbidity and temperature of the water while chemical properties include parameters such as biological oxygen demand (BOD), chemical oxygen demand (COD), dissolve oxygen (DO) and pH of water. Biological indicators include the presence of phytoplankton and algae in the water that affect the quality of the water (Fawaz *et al*, 2013).

In Malaysia, the department that is responsible for standardising water quality is the Department of Environment (DOE). According to Huang *et al*. (2015), the Water Quality Index (WQI) has been introduced by the DOE to serve as the basis or guide for the assessment of the water quality. The quality of water is assessed as it is the factor that determined the quality of the water regarding to its sustainable survival of the riverine ecosystem (Vishnuradhan *et al*, 2017).

There are many parameters that can be used to assess the quality of water, but only six parameters are involved in the calculations of water quality index. The six parameters are BOD, COD, DO, pH, total suspended solids (TSS) and ammoniacal nitrogen (AN). The result of the calculation will indicate the classification of the pollution level. There are five classes of pollution levels. The first class shows the lowest pollutant level while the fifth class shows the highest pollutant level (Huang *et al*, 2015)

2.2 Chemical Oxygen Demand (COD)

According to J. Rank (2017), chemical oxygen demand (COD) is the alternative to the BOD test to determine the potential of the wastewater sample to consume oxygen. It is used to measure the water capacity to consume oxygen during the decomposition of organic matter and the oxidation of inorganic chemicals such as ammonia and nitrite. By referring to its name, it can be said that the carbonaceous oxygen demand or organic compound is chemically oxidized in the COD test (Ellis, 2004).

The COD test was run on natural water or wastewater samples that have been contaminated by the pollutant discharged from the human activities like domestic or industrial activities. COD is one of the parameters used in the water quality index calculation to indicate the level of pollution of the water body. It provides an index to assess the impact of the wastewater discharged from the domestic and industrial sectors to the receiving environment.

The discharged of wastewater must comply with the value as prescribed in Environmental Quality (Industrial Effluent) Regulations 2009. The effluent was categorized into Standard A and Standard B which Standard A is used for any effluents discharged into any inland waters within the catchment areas as listed in the Sixth Schedule in Environmental Quality Act while Standard B for any effluents discharged into any other inland water. For the discharge of effluent containing COD, for industrial effluent of specific trade or industry sector, the conditions follow the Seventh Schedule while for mixed effluent containing COD follow the conditions under the Eight Schedule in Environmental Quality Act (Environmental Quality (Industrial Effluent) Regulations 2009).

The unit to measure COD is in milligrams of oxygen per litre (mg L^{-1}). A high COD value means there is a great amount of oxidize organic material in the water sample (Ellis, 2004) and a high level of water pollution in the area (Fawaz *et al*, 2013). COD level is relatively related to the dissolve oxygen (DO) level where the higher the COD value, the lower the DO level. The reduction of DO level will cause anaerobic condition which will give negative impact to the higher aquatic life forms. It will cause deleterious to the higher aquatic life forms and caused disturbance to the aquatic ecosystem (Hajali, 2016).

Compared to the BOD test, the COD test will give a quick and accurate result, thus it can reduce the time consuming to run the experiment for testing the level of oxygen demand in the water sample. COD analysis method is a chemistry method that involves two hour of digestion procedure at a high temperature (Huang *et al*, 2015). The COD value will be read in mg/L.

There are two types of COD vials for COD digestion method which are low range and high range. COD vials of low range will be used for samples with a concentration of 0 mg/L to 150 mg/L while for higher concentrations of COD value which has range of reading between 0 mg/L to 1500 mg/L, the high range COD vials should be used. Usually, the high range COD vials will be used for sample from industrial sources. If the COD value exceeds 1500 mg/L which is over range, the sample should be diluted. The dilution is done to bring the reading in range so the spectrophotometer are able to read the value.

Chloride is the primary interference for reading of COD value. Vials that contain mercuric sulphate can be used to eliminate chloride interference. Samples with high chloride concentrations should be diluted in order to obtain accurate reading of COD. The sample is diluted based on the dilution factor to reduce the chloride concentration.

2.3 Wastewater Treatment Method for COD Reduction

In general, there are many methods that can be used to treat COD in wastewater. Example of methods that can be used are coagulation-flocculation process, Fenton process (Yadav et al., 2013) and adsorption method (Daud *et al*., 2016). According to research done by Rajasulochana and Preethy (2016), the methods that can be used to treat wastewater include the chemical precipitation, carbon adsorption, ion exchange, evaporations and membrane process while a study by Atkinson *et al*. (2015) stated that wastewater can be treated by the technique of swirl flow bioreactor with the aid of copper-alginate beads.

Coagulation-flocculation can be used during the process of water and wastewater treatment. Coagulation is defined as the method which very fine solid suspensions are destabilized so the suspension solids can agglomerate under the desirable condition while flocculation is the process which the particles that have been destabilized are consolidate into larger complex thus making it possible for the particles to be separated from the wastewater. It is a spontaneous and endothermic process with a positive change in entropy of the system. The biomaterials, organic and inorganic materials that promote accumulation and sedimentation of suspended particles in solution are called as coagulants and flocculants (Nharingo, 2015).

Polymer and compounds like ferric chloride will be added to wastewater to destabilise the colloidal materials. Thus, it will cause the small particles to agglomerate or clustered into complete larger flocs. For instance, ferric chloride ($FeCl_3$) can be used as coagulant to treat organic matter in leachate, colour removal, municipal wastewater treatment and surfactant removal from microelectronic plant wastewater (Aygun and Yilmaz, 2010).

The result from study done by Abdulhassan *et al*. (2006) showed 88% of COD reduction from microelectronic plant wastewater was obtained from the treatment of coagulation-flocculation using $FeCl_3$. However, the efficiency of COD reduction was low when the treatment used only $FeCl_3$.Thus, to improve the efficiency, addition of polyelectrolyte and clay minerals as the coagulant aid was needed in the treatment. Apart from that, numerous studies showed the chemicals used in the coagulation-flocculation process were remained in the treated water. This problem might affect the human health if the treated water is used as drinking water (Rebah and Siddeeg, 2017).

The other method to remove COD is Fenton process. The process is first described by H.J.H Fenton in 1984. It is also known as Fenton reaction and Fenton reagent. It is an alternative method for the conventional oxidation process. The process involved the oxidation of substrate by iron (II) and hydrogen peroxide (H_2O_2). It is a process used to treat wastewater contaminated with contaminants like pesticides, phenols, formaldehyde and organic compounds. It is one of the methods that can be used to treat organic pollutants by oxidation process. The reaction from the Fenton process will cause dissociation of oxidant and formed highly reactive hydroxyl radicals. These radicals will attack and destroy the organic pollutants thus reducing the level of pollution. As the amount of organic compound has been reduced from the process, thus it also reduces the level of COD value (Pawar and Gawande, 2015).

Fenton process has been widely used to treat wastewater as it is a simple treatment system. The study done by Ziati *et al*. (2018) showed a reduction of COD value up to 87% under pH of 3.5. The reaction of this process is only efficient at pH below than 6 which is quite difficult to maintain. Plus, in some cases, when the chemical oxidation is done in high amount, it will lead to increase in toxicity level due to formation of toxic oxidation by-products (Pawar and Gawande, 2015).

The system of swirl flow bioreactor coupled with copper-alginate beads is a system that requires low capital and running cost to treat wastewater. It is a simple system as its construction from commercially available plumbing pipes and fittings. However, due to some circumstances, there have been a limitation to the system demonstrated. The study stated that the system still need an improvement as the beads were affected by the high value of COD due to competing ions within the waste. Thus, it affects the interaction between the copper-alginate within the gel matrix (Atkinson *et al.*, 2015).

Adsorption method using activated carbon is most commonly implemented for the removal or low concentrations of non-degradable organic compounds from groundwater, drinking water preparation, process water or as tertiary cleansing after, for example, biological water purification. Adsorption takes place when molecules in a liquid bind themselves to the surface of a solid substance. Adsorbents have a very high internal surface area that permits adsorption. Active carbon is by far the most commonly used adsorbent and is particularly suited to the removal of a polar compounds. Active carbon adsorption is a proven and much used technique because of the low energy and maintenance costs, simple and reliable (Emis, 2015).

2.4 Activated Carbon

Activated carbon is a media with complex structure which is primarily composed of carbon atoms. The pores network of the activated carbon were formed due to the rigid skeleton of disordered layers of carbon atoms that linked together by the chemical bonds. These networks have created high porous structure making the activated carbon desirable for the adsorption process. The intrinsic pores network of the activated carbon allow the activated carbon to perform adsorption to remove impurities or contaminants from the media. It can be performed towards liquid and gaseous media. Carbonaceous materials like rice husk, coconut shell and palm fruit are suitable to produce activated carbon with the aid from the activation process (Bansal & Goyal, 2005).

2.4.1 Properties of Activated Carbon

Activated carbon has high internal surface area due to the raw characteristics and enhancement of pore distribution during the activation process. The total surface area of the activated carbon can be up to 1500 m^2/g or even more which is relatively equal to the surface area of a soccer field. The higher the internal surface area, the higher the effectiveness of the carbon for adsorption (Bansal & Goyal, 2005).

The other properties of activated carbon is the total pore volume and pore radius. Total pore volume is refers to the pores spaces of the activated carbon particles. It is measured in millilitres per gram (mL/g). The effectiveness of the carbon will be higher if the total pore volume is also high. Different type of activated carbon has different pore volume distribution. It can be classified into three which are micropores, mesopores and macropores. Different source of raw materials and type of activation method will give the activated carbon different structure. It is depends on the carbon and ash content of the raw materials (Bansal & Goyal, 2005).

2.4.2 Type of Activated Carbon

There are three types of activated carbon which are powdered activated carbon, granular activated carbon and extruded activated carbon. The size of powdered activated carbon is only 0.1 mm while granular and extruded activated carbon are 0.2 to 5 mm and 1 to 5 mm respectively. The commonly produced activated carbon are powdered activated carbon and granular activated carbon. Powdered activated carbon can be used in liquid phase adsorption and its processing cost is low (Bansal & Goyal, 2005). Figure 2.1 shows the image of powdered activated carbon.

Figure 2.1 Powdered activated carbon

(Source: https://www.promacwt.com/activated-carbon.html)

Granular activated carbon can be used in liquid and gas phases. It is irregular shaped particle formed by sieving and milling. It is longer lasting compared to the powdered activated carbon and can be used multiple times. It can also performed in fixed and moving systems. Extruded activated carbon is a cylindrical pellet with diameter of 1 to 5 mm. It can be used in gas phase adsorption and can be performed in the automotive sectors (Bansal & Goyal, 2005). Figure 2.2 shows the image of granular activated carbon and extruded activated carbon.

Figure 2.2 Granular activated carbon (left) and extruded activated carbon (right).

(Source: https://www.promacwt.com/activated-carbon.html)

2.5 Factors Affecting Efficiency of COD Reduction Using Activated Carbon

The efficiency of COD reduction using activated carbon is affected by some factors. The factors involved are dosage of adsorbent used, contact time of treatment, pH of solution and temperature (Nayl *et al.*, 2017). Usually, batch adsorption experiment will be performed to identify the optimum value of those parameters in wastewater treatment. Optimum value is the optimal or minimum value used corresponding to the highest reduction of the contaminant or pollutant (Shilpa *et al.*, 2012).

2.5.1 Effect of Adsorbent Dosage

According to research done by Sasikala and Muthuraman (2015), the percentage removal of COD will increase with the increase of adsorbent dosage. Based on their research, it was shown the optimum dosage for adsorbent is 0.4 g/mL where any further mass will give a decreased percentage of COD reduction for various activated carbon. On the other hand, based on the study done by Sanou and Pare (2017), the COD removal experiment was carried out in batch mode where various weight of adsorbent ranging from 0.1 to 0.5 g were stirred in 50 mL of wastewater. The result showed the optimum dosage of adsorbent for COD reduction was 10 g/L while study done by Nayl *et al*. (2017) showed an equilibrium was reached when the carbon dose was 0.1g in 100 mL of wastewater for the optimum percentage removal. After equilibrium, it shows that any increase of the adsorbent dose will give a constant removal of COD result.

2.5.2 Effect of Contact Time

The other parameter that involved in COD reduction experiment is contact time. The percentage of COD reduction will increase when the contact time increases. It was observed that the optimal reduction time lies between 60 to 90 minutes. However, different treatment shows different optimum removal within the range. The treatment using activated charcoal from rice husk shows the optimum removal was achieved during 60 minutes of contact time where the percentage of removal was 92% (Sanou and Pare, 2017). This study was supported by the same result found by El-Dars *et al*. (2013).

2.5.3 Effect of Wastewater Concentration

The initial concentration of wastewater will affect the percentage removal of the adsorption process. Study done by Attia *et al*. (2010) showed the percentage of sorption increased with decreasing of adsorbate concentration. It was due to the presence of sufficient active sites on the adsorbent for the adsorbate to occupy during adsorption process. Thus, at higher concentration, the removal percentage was low as the ions present in the wastewater not completely adsorbed due to the saturated binding sites. The same result was obtained in the study done by Ould Brahim *et al*. (2014) where at higher adsorbate concentration, the removal percentage was decreased.

2.5.4 Effect of Solution pH

According to Nayl *et al*. (2017), the pH value of the solution will affect the effectiveness of the adsorption. Based on their study, the removal percentage of COD increase at an acidic pH to pH 6. When the pH value is increased, the removal percentage of COD decreased. The same result was obtained by the research done by Said et al. (2015) and Daud *et al*. (2017) where the adsorption process decreased for solutions with pH value above 7 and 6 respectively. The rate of COD reduction will decreased if the pH of the solution is below 7 or higher than 9 (Abdulhassan *et al*., 2006). This can conclude that the adsorption efficiency will decrease at alkaline solution.

2.6 Foxtail Palm

Foxtail palm is a monocot plant that has a solitary growth habit. The tree is named as foxtail regarding its appearance, which its leaf looks like a foxtail. Its scientific name is *Wodyetia bifurcata* and it is grouped under member of Arecaceae family which is a palm family. Fast growth, adaptability and beauty makes the foxtail palm tree as desirable tree for nurseries and landscaping sectors (Perez *et al*, 2009). Foxtail palm tree has abundance of fruits which is clustered. At early stage, the fruits will appear in green colour and once it has ripened, it will appear into orange-red colour. Since the fruit cannot be eaten and be found abundantly, it is suitable to produce activated carbon. Plus, the characteristics of the fruits which are low ash content and high fixed carbon content makes the foxtail palm fruit suitable to produce activated carbon (Munasir, 2018).

Figure 2.3 Foxtail palm tree

(Source: https://www.123rf.com/photo_36862269_granular-activated-carbon-for-water-filter.html)

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials and Instruments

Wodyetia bifurcata fruit or commonly known as foxtail palm fruit was used as the material for preparing activated carbon while wastewater from textile industry was collected as the sample water for the treatment using the activated carbon. The chemicals and instrument used in this study were listed in Table 3.1 and Table 3.2 respectively.

Table 3.1: List of chemicals used in the study

Chemicals	Brand
Concentrated Nitric Acid (HNO_3)	Merck (M) Sdn. Bhd.
HACH's COD Digestion Vials (High Range)	Merck (M) Sdn. Bhd.

Table 3.2: Instrument used in the study

Instrument	Brand
UV-Visible spectrophotometer	HACH

3.2 Sample Collection

3.2.1 Foxtail Palm Fruit

Wodyetia bifurcata fruit or commonly known as foxtail palm fruit was collected from its tree that is located at residential area in Jeli district, Kelantan. It was used to prepare activated carbon for Chemical Oxygen Demand (COD) reduction from the industrial wastewater. After the collection, the fruits then were transported to the laboratory and it were washed thoroughly with distilled water to remove the impurities and dirt on the fruits. The fruits were dried overnight in the oven at 100ºC to cut off the water molecules present in the fruits. The dried fruits were left outside to let it cool in room temperature (Zakir, 2013). Then, the dried fruits were stored in a tight polyethylene bag in the desiccator for further use.

3.2.2 Wastewater

Wastewater from textile industry, 'Kedai Adnan Batik' in Kota Bharu, Kelantan (6°10'38.3"N 102°15'15.9"E) was collected at the point of discharge from the industry as shown in Appendix A. Figure 3.1 shows the location of the textile industry.

Figure 3.1 Map of 'Kedai Adnan Batik', Kota Bharu, Kelantan.

3.3 Preparation of Activated Carbon

The foxtail palm fruits prepared from the previous method were used to prepare the activated carbon. The dried fruits were carbonized in the furnace at 300ºC for two hours. This was to cut off the water molecules and to enhance the pore development for adsorption. Next, the char produced from the carbonized fruit was cooled in room temperature before being crushed into small pieces using mortar and pestle. Then, it was sieved through 250 µm (Zakir, 2013). The char was stored in a tight polyethylene bag and kept dried in the desiccator. This was to ensure the char will not interact with the outer surrounding (Munasir, 2018).

3.4 Process of Chemical Activation

For the process of chemical activation, the process was referred to the method proposed by Chin (2014). About 40 g of oven-dried foxtail palm fruit prepared from previous process was soaked and impregnated with 80 mL of concentrated nitric acid (HNO_3) in the beaker. The mixture was mixed vigorously for 30 minutes with a constant stirring until it becomes a paste. Then, the paste was left impregnated overnight in the fume hood to ensure the chemical had fully reacted with the sample. It was carbonized at 500ºC for 2 hours and 30 minutes in the furnace. Next, it was kept in the desiccator to let it cool in room temperature. After the carbonization process, the activated carbon was washed using distilled water to adjust its pH to neutral. Then, the activated carbon was dried in the oven for 3 hours at 150ºC before was stored in a tight polyethylene bag and kept in the desiccator for further use.

3.5 COD Value

The method used to test the unknown chemical oxygen demand (COD) value was based on the method prescribed by USEPA Reactor Digestion Method (1980). About 100 mL of wastewater were measured and blended for 30 seconds. Then, 2 mL of the wastewater was pipetted into the COD vial. 2 mL of deionized water was pipetted into the other vial which was used as the blank. All the vials were put into the reactor for two hours at 150ºC. Next, the vials were cooled at room temperature and COD value was analysed using the UV-Visible spectrophotometer.

3.6 Batch Adsorption Experiment

3.6.1 Effect of Adsorbent Dosage on COD Reduction Efficiency

First, 20 mL of wastewater with COD reading of 441 mg/L was poured into a 100 mL Erlenmeyer flask. The wastewater was treated with 5 various weights of activated carbon prepared from previous process ranging from 1 g to 5 g. The mixture was stirred at 200 rpm within 60 minutes in an isothermal shaker and was filtered using 0.45 µm Whatman filter afterward. The treated sample was then analysed using UV-Visible spectrophotometer (Sanou & Pare, 2016).

3.6.2 Effect of Contact Time on COD Reduction Efficiency

The experiment was repeated referring to the method proposed by Sanou and Pare (2016) using different parameter by keeping the adsorbent dosage with the highest percentage of removal constant. The contact time was vary from 60 minutes to 180 minutes. The volume and concentration of wastewater used was as the same as the previous method.

3.6.3 Effect of Wastewater Concentration on COD Reduction Efficiency

The third batch was done where the highest removal percentage of adsorbent dosage and contact time were kept constant while the concentration of wastewater was vary. The concentration of wastewater used in this experiment were 5%, 10%, 20%, 25% and 50% from the initial concentration of wastewater. The procedure was the same as to analyse the effect of adsorbent dosage and contact time referring to the method proposed by Sanou and Pare (2016). Table 3.3 shows the dilution factor for wastewater concentration.

Table 3.3 Dilution factor for wastewater concentration

Volume of wastewater (mL)	Volume of distilled water (mL)	Dilution factor
1	19	5
2	18	10
4	16	20
5	15	25
10	10	50

3.7 Reduction Efficiency

The efficiency of the COD reduction was calculated using the equation below (Equation 3.1).

$$\text{Reduction (\%)} = \frac{(C_o - C_i)}{C_o} \times 100 \quad (3.1)$$

where;

C_o is the initial reading of COD value (mg/L)

C_i is the final reading of COD value (mg/L)

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Foxtail Palm Fruit Activated Carbon

Wodyetia bifurcata fruit or commonly known as foxtail palm fruit was prepared to produce activated carbon. Chemical activation using concentrated nitric acid (HNO_3) was used in this study. Chemical activation was done to enhance the development of porous structure in carbon thus providing more surface area for the adsorption process (Atef, 2016).

The activated carbon was washed before the treatment to neutralize its pH. This was to ensure the efficiency of the adsorption process as pH could affect the reading of the adsorption. This was proven from study done by Sumrit Mapoung *et al*. (2015) where at low pH of activated carbon, the surface of the adsorbent was positively charged, thus the performance for adsorbing cation was low and vice versa.

The type of activated carbon produced in this study was powdered activated carbon. Powdered activated carbon were generally used in batch adsorption experiment for liquid phase adsorption as it was more easily handled in term of dosage (Bansal & Goyal, 2005). The physical appearance of the activated carbon prepared from foxtail palm fruit was as shown in Appendix A.

4.2 COD Value

The value of chemical oxygen demand (COD) of the wastewater at 10% concentration was 441 mg/L. The acceptable value of COD for textile industry was 250 mg/L for effluent under Standard B as prescribed in the Environmental Quality (Industrial Effluent) Regulation 2009. The high value of COD obtained in this study was applicable to be treated with the activated carbon since the value exceeds the value of acceptable conditions for the industrial effluent containing COD. The COD value was kept constant at 441 mg/L as the reference for subsequent readings of COD reduction in the batch adsorption experiment.

4.3 Batch Adsorption Experiment

4.3.1 Effect of Adsorbent Dosage on COD Reduction Efficiency

The effect of different activated carbon dosage was studied to observe the optimum dosage for the reduction of COD from textile wastewater. Five different dosage of activated carbon ranging from 1 g to 5 g was treated in 20 mL of wastewater. The orbital shaker was set up at constant setting which are 200 rpm with contact time of 60 minutes. The result obtained for the effect of adsorbent dosage on COD reduction efficiency was as shown in Table 4.1.

Table 4.1 Effect of adsorbent dosage on COD reduction efficiency

Adsorbent dosage (g)	Removal percentage (%)
1	52.15
2	58.96
3	46.26
4	36.73
5	24.72

High percentage removal was obtained using dosage of 2 g of activated carbon which was 58.96%. As can be observed from Figure 4.1, the percentage removal of COD was increased from 52.15% to 58.96% using 1 g and 2 g of activated carbon respectively. According to El-Dars *et al*. (2013), the percentage removal of COD arise with the addition of adsorbent dosage due to increase of adsorbent surface area and the number of available adsorption sites.

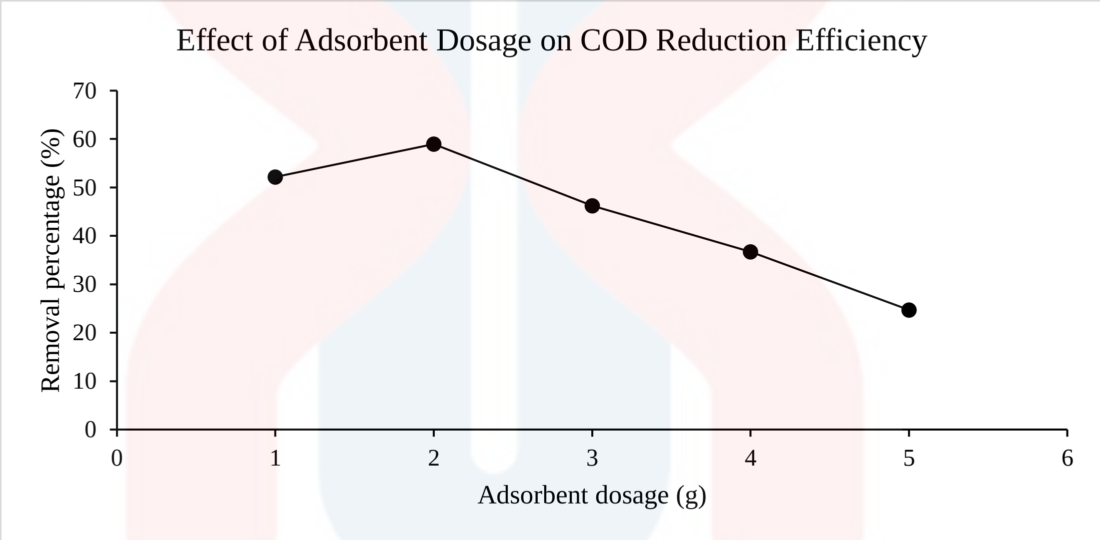

Figure 4.1 Effect of adsorbent dosage on COD reduction efficiency

The increase of adsorbent surface area was resulted from the increase of total pores available for the adsorption process for COD reduction to occur. The process of chemical activation have enhanced the development of pores for the adsorption of COD using the activated carbon. More binding sites were available for the adsorption process to take place (El-Dars *et al*., 2013). Thus, the percentage reduction of COD value increased with the increased dosage of activated carbon from 1 g to 2 g.

Nevertheless, the percentage removal started to decrease at 3 g to 5 g of adsorbent dosage. This might due to the over dosage of adsorbent that does not correspond to the amount of wastewater. Maryam Khodaie *et al*. (2013) in their study stated the adsorption capacity was influenced by the concentration and volume of the adsorbate. At constant concentration and volume of adsorbate, the sorption sites were not saturated thus giving a better performance of adsorption process.

Apart from that, Maryam Khodaie *et al*. (2013) in their study mentioned adsorbent dosage also contributed to the efficiency of adsorption process. They stated that high adsorbent dosage will affect the particulate interaction such as aggregation, thus lead to decrease in total surface area of the adsorbent. This explained the reason of the percentage declined for the COD reduction efficiency at 3 g, 4 g and 5 g of activated carbon dosage which were 46.26%, 36.73% and 24.72% respectively.

The volume of wastewater used in this study was 20 mL. This might be the reason for the declining of percentage reduction of COD value at a higher amount of activated carbon. The amount of water used was not correspond to the amount of activated carbon, thus resulting in aggregation.

4.3.2 Effect of Contact Time on COD Reduction Efficiency

The effect of contact time on COD reduction using foxtail palm fruit activated carbon was studied. The textile wastewater was treated with activated carbon dosage with the highest percentage of removal obtained from the previous method which was 2 g with the same amount of wastewater. The wastewater was treated for duration of 3 hours which for every 30 minutes of interval, starting from the 60th minute, the treated water was analysed using UV-Visible spectrophotometer. The result for the effect of contact time on COD reduction efficiency was as shown in Table 4.2.

Table 4.2 Effect of contact time on COD reduction efficiency

Contact time (min)	Removal percentage (%)
60	60.09
90	65.31
120	65.08
150	65.08
180	64.85

From Figure 4.2, it can be observed a high percentage of COD reduction was obtained for the first 60 minutes. The reason was, at the beginning of adsorption process, the pores or the surface area of the activated carbon has not been occupied yet, thus resulting a high percentage of COD reduction. An increase of percentage removal by 5.22% between contact times of 60 minutes to 90 minutes was obtained with the reading of 60.09% and 65.31% respectively. The highest percentage of COD reduction was achieved at the 90th minute of contact time.

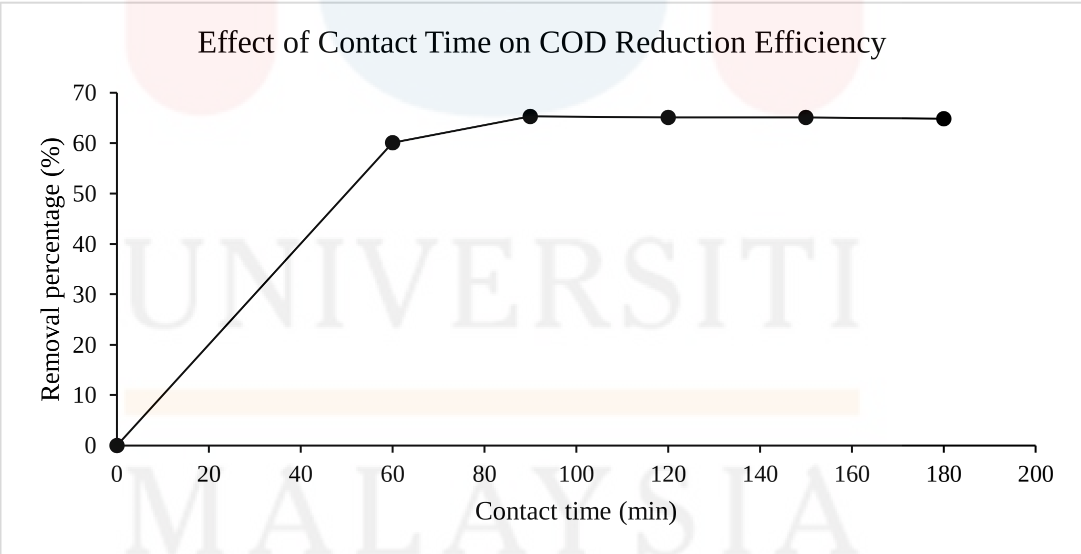

Figure 4.2 Effect of contact time on COD reduction efficiency

A slightly difference of percentage removal was obtained after the 90th minute with the reading of 65.08% of COD removal for both reading at 120th minute and 150th minute. As mentioned in the study done by Abdel-Gawad and Abdel-Aziz (2019) and

El-Dars *et al*. (2013), as the contact time for the adsorption process increase, the percentage removal will decrease. The inclination of the result was due to saturation of the adsorbent pores that enabled for continuous adsorption process to take place.

4.3.3 Effect of Wastewater Concentration on COD Reduction Efficiency

The optimized value of adsorbent dosage and contact time of the treatment obtained from the previous treatment was set for the third batch experiment. Different concentration of wastewater which were 5%, 10%, 20%, 25% and 50% were treated under the optimized condition which were 2 g of adsorbent dosage with 90 minutes of contact time. The result obtained was as shown in Table 4.3.

Table 4.3 Effect of wastewater concentration on COD reduction efficiency

Wastewater concentration (%)	Removal percentage (%)
5	71.23
10	65.08
20	24.10
25	19.78
50	7.65

The efficiency of COD reduction was depending on the concentration of the adsorbate and availability of binding sites on surface of adsorbent. The relation between both factors has been proven in the study done by Ould Brahim *et al*. (2014) at which, the efficiency of the removal was depending on both factors. At low concentration of adsorbate, the percentage of removal was high and the reading started to decline with the increase of the adsorbate concentration.

As can be observed from Figure 4.3, the percentage of COD reduction decreased as the concentration of the wastewater increased. High percentage of reduction was achieved as the concentration of the wastewater was 5%, which was the lowest concentration of wastewater among the other concentrations. The reading recorded for the concentration of 10% and 20% of wastewater were 65.08% and 24.10% respectively. A significant difference of reduction percentage was obtained between both concentrations of wastewater which was 40.98% of difference.

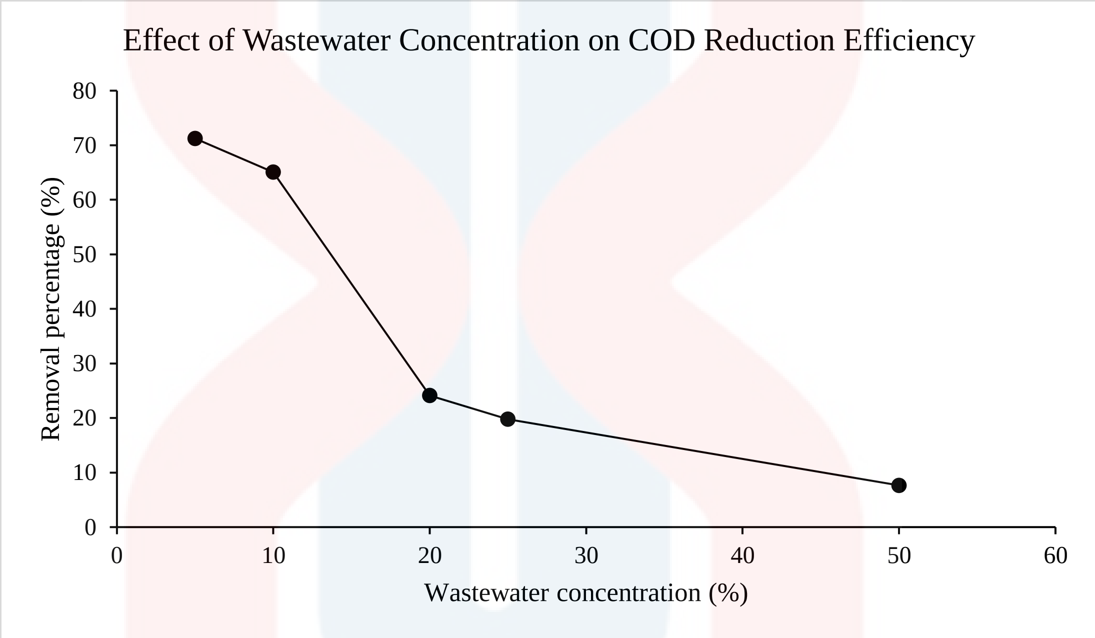

Figure 4.3 Effect of wastewater concentration on COD reduction efficiency

The lowest removal percentage was obtained at wastewater concentration of 50%. This might be due to the presence of high amount of molecules in the wastewater compared to the low concentration of wastewater. A great amount of molecules present in high concentration of adsorbate, therefore lacking of binding sites required for the adsorption process to take place (Ould Brahim *et al.*, 2014). Thus, the higher the concentration of wastewater, the lower the percentage of removal.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In this study, the optimum conditions for the reduction of chemical oxygen demand (COD) from the industrial wastewater especially textile industry were at 90 minutes of contact time with adsorbent dosage of 2 g in 5% of wastewater concentration. At this optimum conditions, the percentage of removal obtained was 71.23%. The efficiency of the activated carbon for COD reduction in this study was low as it was affected by the amount of adsorbent dosage which was not correspond to the volume of wastewater used.

Lower percentage of COD reduction was obtained with the increased of contact time and dosage of adsorbent. Higher amount of activated carbon used in the treatment resulted in decreased of COD reduction efficiency as the amount of the activated carbon were not corresponding to the volume of wastewater. This was due to the interference with the activated carbon particles which caused aggregation of particles. This condition has reduced the efficiency of the COD reduction using the activated carbon prepared from foxtail palm fruit.

Apart from that, the concentration of wastewater also affected the efficiency of COD reduction in industrial wastewater. As wastewater from industrial activities has great amount of particles, thus it affected the process of adsorption to take place. The presence of particles caused interference during the process as it saturated the adsorbent pores available for the adsorption sites. The higher the concentration of wastewater, the higher the amount of particles present in the wastewater thus, the lower the percentage of COD reduction. As a result from this, the efficiency of the COD reduction was decreased.

5.2 Recommendation

The volume of wastewater used in this study was not corresponding to the amount of activated carbon used in the treatment. Hence, it is recommended to use higher volume of wastewater in further research. It is so that the activated carbon can be fully reacted with the wastewater for a better result. Contrariwise, if the volume of the wastewater is to remain the same, less amount of activated carbon can be applied in the treatment. This is to make sure the volume of wastewater and the amount of activated carbon is corresponding to avoid aggregation.

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APPENDIX A

Figure A-1 : The site for sampling of textile wastewater

Figure A-2 : Foxtail palm fruit

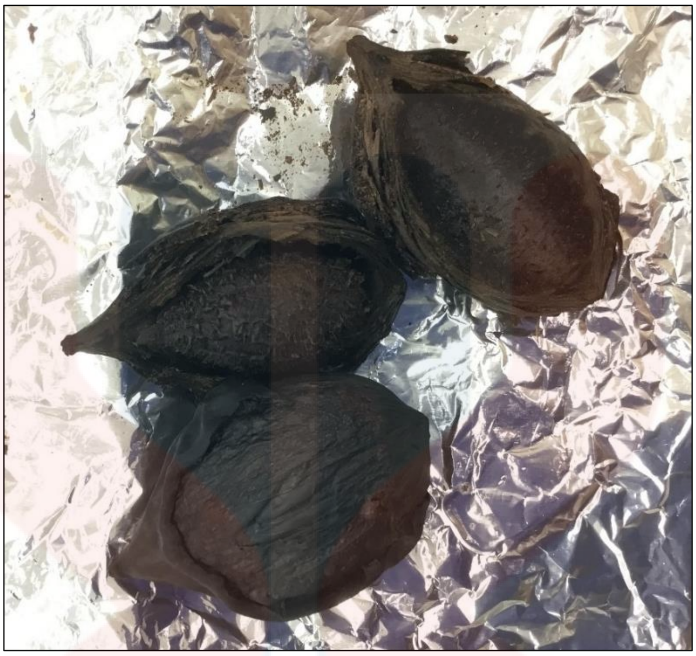

Figure A-3 : Carbonized foxtail palm fruit

Figure A-4 : Activated carbon prepared from foxtail palm fruit