

## Effect of Microwave-Assisted CO<sub>2</sub> Pyrolysis on the Production of Activated Carbon from Confiscated Cigarettes

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### ABSTRACT

*Confiscated cigarettes are a significant problem worldwide, and in Malaysia, the Royal Malaysian Customs Department (RMCD) Perlis had confiscated 1.5 billion sticks of cigarettes from 2016 to 2019 and the cost of disposal reached RM1.2 billion. Confiscated cigarettes are commonly disposed by incineration which can cause environmental pollution. In this study, the cigarette tobacco was converted into activated carbon (AC) via microwave-assisted CO<sub>2</sub> pyrolysis. The tobacco was first carbonized at 300 °C, followed by carbon dioxide (CO<sub>2</sub>) activation under microwave heating. The highest yield, 31% was obtained at 616W for 6 min. The BET surface area for tobacco, char and the best AC produced were 1.99, 1.21 and 1.69 m<sup>2</sup>/g, respectively. Response surface methodology (RSM) of dye removal from AC showed that the optimum condition with the best adsorption properties was achieved at 364 Watt for 2 minutes, which resulted in 87.4% removal of methylene blue (MB). Due to the low surface area, the capability of the AC to adsorb MB dye might be assisted by a diversified factors that contributes to the adsorption mechanism. Modelling of the adsorption data also showed that the adsorption process occurs in multilayers of the adsorbent, best fit to the Freundlich isotherm. Microwave-assisted CO<sub>2</sub> pyrolysis shows potential in the production of AC from tobacco and could be further improved to increase the surface area.*

**Keywords:** Activated carbon, Microwave heating, Tobacco, Adsorption, Methylene blue.

### 1. INTRODUCTION

Tobacco is the main constituent in a cigarette, containing nicotine which is highly addictive, resulting in repeated use of cigarettes (Gaalema *et al.*, 2022). Cigarettes are often illegally traded around the world, including Malaysia. From Oxford Economics (2019), the highest market of illicit cigarettes was in Sarawak, which was 83.5%, followed by Sabah and Pahang. The combination of few factors such as availability and affordability had caused the market of illicit cigarettes to increase, and more cigarettes is expected to be confiscated.

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Based on the records from the Royal Malaysian Customs Department (RMCD), a total of 1,894,318,528 cigarettes were confiscated in Malaysia from 2018 to September 2020 (JKDM, 2020). The total disposed cigarettes and the cost of disposal in 2016 and 2019 were 1,535,854,514 cigarettes and RM1,227,995, respectively. All the confiscated cigarettes were disposed by RMCD through incineration or landfills. This is considered a huge waste of resources because tobacco have the potential to be converted into value-added products, for example activated carbon (AC). A variety of raw materials had been tested to produce AC. The biggest requirement of the raw material is that is must contain high carbon. Moreover, ash and trace impurities are also part of a crucial role as they will exist in the final product and can influence the properties of the AC produced. The most generally utilized raw materials to produce AC are charcoal (including anthracite coal, bituminous, subbituminous, and wood coal), coconut shells, soft wood such as sawdust, hard wood and peat.

The choice of methods to produce AC will determine its adsorptive characteristics, such as in dye removal application (Ahmad *et al.*, 2021). Yusop *et al.*, (2021) stated that the pyrolysis process with the aid of microwave oven were used to produce AC via two main steps. First, the raw material must undergo carbonization which is a thermal degradation process without the presence of oxygen. In the carbonization process, the precursor will be converted into char by exposing it to a temperature generally between 300°C to 400°C. The main function of carbonization is to increase the carbon content within the precursor and to form a series of preliminary pores. The second step is an activation steps where the char will be activated to form AC. The activation process of a normal furnace had disadvantages such as high energy consumption and long activation period (Yusop *et al.*, 2021). Hence, researchers are exploring alternative techniques for the activation process such as microwave-assisted pyrolysis. Thus, this study employs microwave irradiation technique to produce AC by using tobacco as the raw material to investigate the AC adsorption properties on the removal of methylene blue (MB) dyes.

## 2. MATERIALS AND METHODS

### 2.1 Sample Preparation

The cigarettes were obtained from the RMCD of Perlis. The tobacco were separated from the cigarette and stored until further use. First, 260 g of tobacco was loaded into a furnace for carbonization at 300°C, for 10 minutes under N<sub>2</sub> flow to create pyrolysis atmosphere. The carbonization process was controlled to prevent the temperature from fluctuating as this will affect the quality of final product AC. After carbonization, the samples were then transferred into a container and cooled down to room temperature under N<sub>2</sub> environment. Activation process was carried out by using an improvised microwave oven (EMM2001W, Sweden) with different radiation power and duration with the purging of CO<sub>2</sub> gas. The radiation power was set at 364 W, 490 W and 616 W, for 2, 4 and 6 minutes. The CO<sub>2</sub> was purged at a rate of 150cm<sup>3</sup>/min to give a physical activation effect to the biochar. The sample was then cooled down in the microwave and packed in a zip bag to prevent it from absorbing moisture (Lin, Yan, & Sheng, 2016). The yield of AC from the precursor was calculated according to the following equation 1:

$$\text{Yield \%} = \frac{W_f}{W_i} \times 100 \quad (1)$$

where  $W_f$  is the mass of dried AC, and  $W_i$  is the dried precursor.

## 2.2 Adsorption properties of AC in dye removal

This experiment was conducted by adding 0.1 g of AC into 100 ml of 50 mg/L MB (HmbG Chemicals) dye solution in a conical flask. The agitation speed and temperature was set at 150 rpm and 30°C, respectively. The experiment was stopped when the AC reached the state of equilibrium. Then, the absorbance of the remaining dye solution was measured at 665nm using UV-visible spectrophotometer. The quantity of dye adsorbed, together with the percentage removal of MB were analyzed using Equations 2 and 3, respectively (Pathania *et al.*, 2017):

$$q_e = (C_i - C_e) \frac{V}{M} \quad (2)$$

$$\text{Removal \%} = \frac{C_i - C_e}{C_i} \times 100\% \quad (3)$$

where  $q_e$  is the amount of dye in mg per gram of adsorbent (mg/g),  $C_i$  is the initial concentration,  $C_e$  is the concentration of MB at equilibrium (mg/l),  $V$  is the volume of solution (L), and  $M$  is the mass of adsorbent (g).

## 2.3 Effects of Contact Time for Equilibrium Studies

The experiment was conducted by adding 0.1 g of AC into 100 ml of 50 mg/L MB dye solution in a conical flask. The agitation speed was set at 150 rpm and temperature at 30°C. The absorbance of the dye was taken at different intervals (0, 15, 30, 60, 90 min, followed by 6, 22, 24 and 25 h).

## 2.4 Isotherm models of MB dye Adsorption

To describe the behaviour of isothermal adsorption, Langmuir and Freundlich model were used (Kuang *et al.*, 2020). Data of  $q_e$  and  $C_e$  from the dye removal were extracted and used in both isotherm model. The experiment was carried out with different initial concentrations, 20, 40, 60, 80, 100 mg/L of MB with 0.1 g of AC in 24 hours until equilibrium was reached.

The Langmuir model (Equation 4) is mainly focused only on one monolayer. Furthermore, it assumes that all adsorption sites on the surface of adsorbent are homogeneous and contains the precise equal adsorption capacity (Kuang *et al.*, 2020):

$$\frac{C_e}{q_e} = \frac{1}{q_{max}} C_e + \frac{1}{q_{max} K_L} \quad (4)$$

where  $C_e$  is the equilibrium concentration (mg/L),  $q_e$  is the amount of adsorbed dye at equilibrium (mg/g),  $q_{max}$  is the Langmuir constants that are correlated to the adsorption capacity (mg/g), and  $K_L$  is the adsorption rate (L/mg).

On the other hand, the Freundlich isotherm model assumes that multilayer adsorption processes take place on heterogeneous surfaces. Equation 5 stated the Freundlich isotherm model (Kuang *et al.*, 2020):

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e \quad (5)$$

where  $C_e$  is the equilibrium concentration (mg/L),  $q_e$  is the amount of adsorbed dye at equilibrium (mg/g),  $K_f$  is a constant related to the adsorption energy (mg/g), and  $n$  is the number of active sites.

## 2.5 Functional groups analysis of AC

The samples were prepared by grounding the AC with KBr salt at a ratio of 1:50 and compressed it into pellet using a hydraulic press. Then, the pellet was inserted into the FTIR machine (Hussein *et al.*, 2015). The FTIR (Perkin Elmer, USA) spectrum was chosen within the range of 550cm<sup>-1</sup> to 4000cm<sup>-1</sup> with a resolution of 1 cm<sup>-1</sup> for each scan (Şencan & Kiliç, 2015).

## 2.6 Surface area analysis of AC

Isothermal adsorption of nitrogen method was used to analyze the pore characteristics and surface area of the AC. The AC samples were degassed at 105°C for 4 hours before carrying out the measurement (Lin *et al.*, 2016). Relative pressures ( $P/P_0$ ) ranging from 0.05 to 0.35 was carried out to acquire the information of its porosity characteristics and the specific surface area of AC.

## 2.7 Scanning electron microscopy (SEM) imaging

Tobacco, char, and AC produced from different conditions were prepared for SEM analysis (Hitachi TM-3000, Japan). About 0.5g sample was loaded onto the metal plate. The magnification of SEM was carried out at 1000x with 20kV.

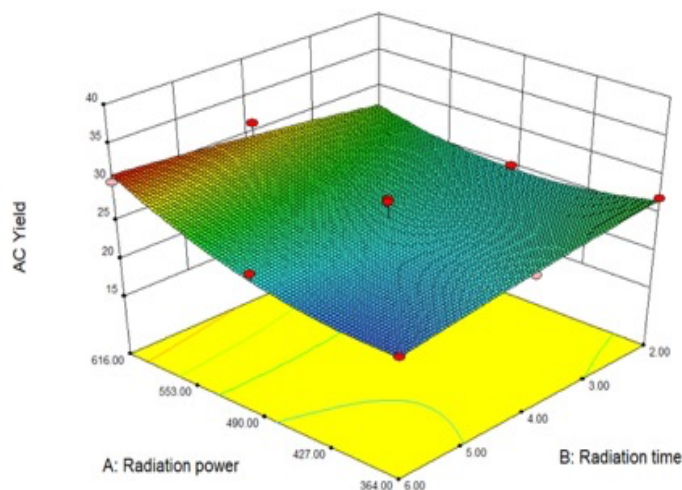
# 3. RESULTS AND DISCUSSION

## 3.1 Yield of AC Produced from Cigarette Tobacco

AC was successfully produced from the cigarette tobacco via CO<sub>2</sub> pyrolysis with radiation power of 364 W, 490 W and 616 W for 2, 4 and 6 minutes. During pyrolysis, the precursor, which was the tobacco, experienced a thermal decomposition where the formation of micropores and mesopores occurred. For the response of AC yield (Figure 1), a quadratic model was used. The final equations of the empirical models in terms of coded factors that relates the responses and variable are given as follows:

$$Y = 24.94 + 2.25X_1 - 0.075X_2 + 1.95X_1 X_2 + 2.43X_1^2 - 0.30X_2^2 \quad (6)$$

$Y$  is the yield of AC in terms of coded factors,  $X_1$  is the radiation power (Watt) and  $X_2$  represents the radiation time (minutes). The yield of the AC was around 22% to 31% which didn't differ much at different conditions tested. The highest yield, 31% was obtained at 616W for 6 min.



**Figure 1.** Response surface plot of AC yield vs radiation power and time.

### 3.2 Outcomes of CCD response

Central Composite Design (CCD) was used to design the experiment for the production of AC from tobacco with different parameters which includes radiation power (Watt) and radiation time (minutes). The percentage of dye removal is shown in Table 1.

**Table 1.** Experimental Design for AC Preparation

Run	AC Preparation Variables		Responses		
	Radiation Power (Watt)	Radiation Time (Minutes)	Absorbance	Final Concentration (mg/L)	Percent Dye Removal (%)
1	364	2	0.109	6.3	87.4
2	364	4	0.126	6.5	87.0
3	364	6	0.159	9.7	80.6
4	490	2	0.147	9.0	82.0
5	490	4	0.11	6.4	87.2
6	490	4	0.137	7.9	84.2
7	490	4	0.119	6.7	86.6
8	490	4	0.118	6.6	86.8
9	490	4	0.119	6.7	86.6
10	490	6	0.125	7.7	84.6
11	616	2	0.144	8.5	83.0
12	616	4	0.171	10.0	80.0
13	616	6	0.181	12.5	75.0

#### 3.2.1 Analysis of Variance (ANOVA)

The ANOVA results for the MB dye removal are shown in Table 2. It can be concluded that the model's p-value of 0.0243 indicated that the model is statistically significant. In this case A and B are significant model terms. Based on Table 3, a negative Predicted R-Squared implies that the overall mean is a better predictor of the response than the current model. Adequate Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Thus, this model can be used to navigate the design space.

**Table 2.** ANOVA for Response Surface Quadratic Model of MB Removal

Source	Sum of Squares	df	Mean Square	F-Value	p-value Prob > F	
Model	221.60	5	44.32	5.35	0.0243	significant
A- Radiation Power	80.67	1	80.67	9.73	0.0168	significant
B-Radiation Time	49.31	1	49.31	5.95	0.0448	significant
AB	9.61	1	9.61	1.16	0.3173	
A <sup>2</sup>	23.73	1	23.73	2.86	0.1345	
B <sup>2</sup>	27.08	1	27.08	3.27	0.1136	
Residual	58.01	7	8.29			
Lack of Fit	52.20	3	17.40	11.98	0.0182	significant
Pure Error	5.81	4	1.45			
Cor Total	279.61	12				

**Table 3.** Statistical Values for MB Removal

Parameters	Values
Standard Deviation	2.88
Mean	83.55
CV %	3.45
PRESS	539.87
R-Squared	0.7925
Adjusted R-Squared	0.6443
Predicted R-Squared	-0.9308
Adequate Precision	7.609

From the data above, a final equation in terms of coded factors is shown in Equation 7. The equation shows how a mathematical model of coded parameters is connected to the factors that determine the MB percentage removal.

$$\text{Percentage Removal} = 86.35 - 3.67A - 2.87 B - 1.55 AB - 2.93A^2 - 3.13B^2 \quad (7)$$

where A indicates radiation power (Watt), and B stands for radiation time (minutes).

### 3.2.2 Response Surface of MB Dye Removal

The effect of the parameter on the results and responses can be known better by studying the three-dimensional response surface. A contour plot can also be used to understand the relationship between two parameters. The plot shows the maximum and the minimum point of effect of two parameter towards certain responses.

Since ANOVA results had shown that both the parameters A and B are significant to the MB removal response, therefore the 3D plot only shows the effect of the variable. Based on Figures 2 and 3, the lowest MB dye removal by AC took place when the radiation time is at 6 minutes with a radiation power of 616 Watt. On the other hand, the AC with radiation time of 2 minutes and radiated at 364 Watt appeared to be the highest in MB removal. Based on Foo & Hameed (2012), excessive energy subjected to the sample could cause some of the carbon content inside to scorch and demolish the existing pores and evaporating more volatile matter. This is a major reason that cause the adsorption properties to decrease and the ability to remove MB dye is greatly reduced when using higher radiation power. This could be explained by the physical characterization via BET and SEM analysis.

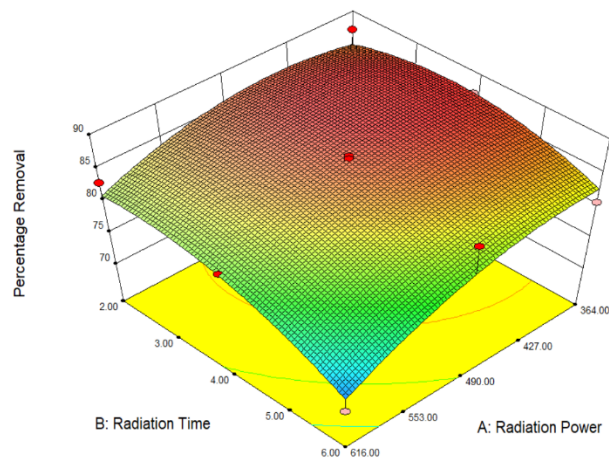


Figure 2. Response surface plots for MB dye removal

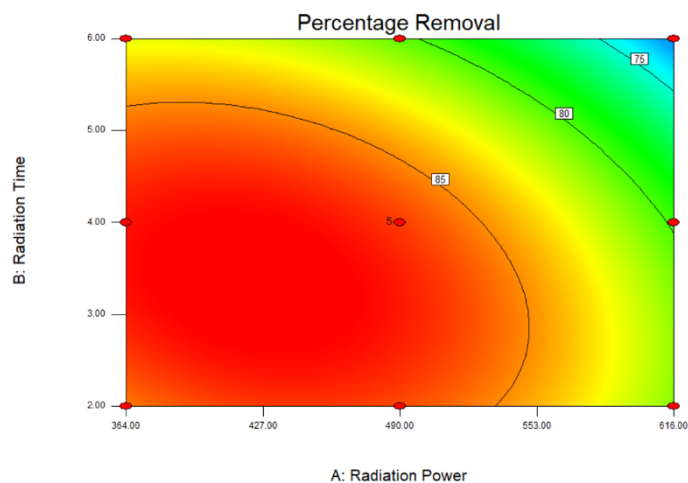


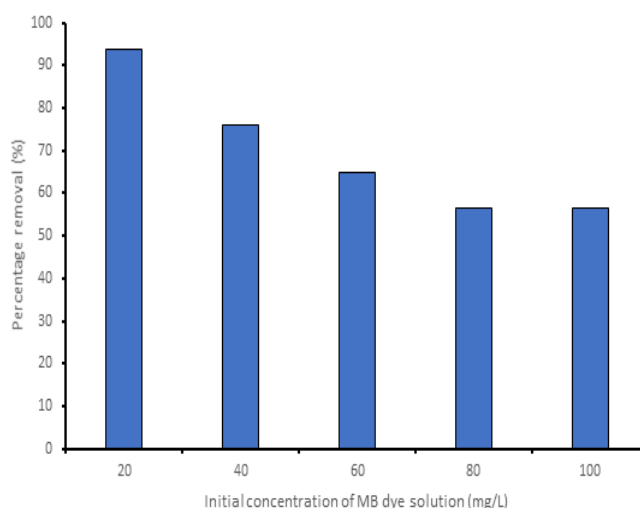
Figure 3. Contour plot for MB dye removal

### 3.3 Adsorption of MB by AC

#### 3.3.1 Effect of Different Initial MB Concentration towards AC

The initial concentration of MB dye solution used in the experiment were 20, 40, 60, 80 and 100 mg/L. The experiments were carried out for 24 hours until the equilibrium had been reached. Based on Figure 4, the percent removal of MB by AC decreased gradually from 20 mg/L to 80 mg/L and finally come to a constant at 100 mg/L.

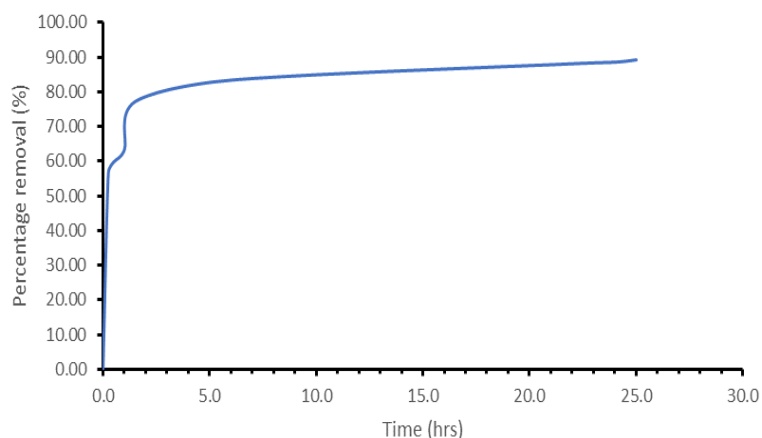
The decreasing trend shown in Figure 4 might be caused by the maximum capacity of the AC had been reached and the AC have no more vacant spaces for further removal of MB dye. The removal of the MB dye declined with the increase of initial MB dye concentration might be due to the deficiency of active sites under high concentration (Kuang *et al.*, 2020). The MB removal at low concentration was high because there are more vacant sites compared to the MB dye molecules.



**Figure 4.** Effects of initial concentration of MB on percent removal by AC

### 3.3.2 Effect of contact time of AC on MB removal

The MB dye removal by AC were carried out for 25 hours. This is to determine the equilibrium time needed for the AC to remove MB. From Figure 5, the initial removal of MB was fast within the first hour. The MB removal reached 56% and 63 % dye removal after 15 min and 1 h, respectively, before reaching equilibrium. This is due to the limited reaction sites which were filled with MB molecules, and the remaining dye can no longer be adsorbed to the surface of the AC.



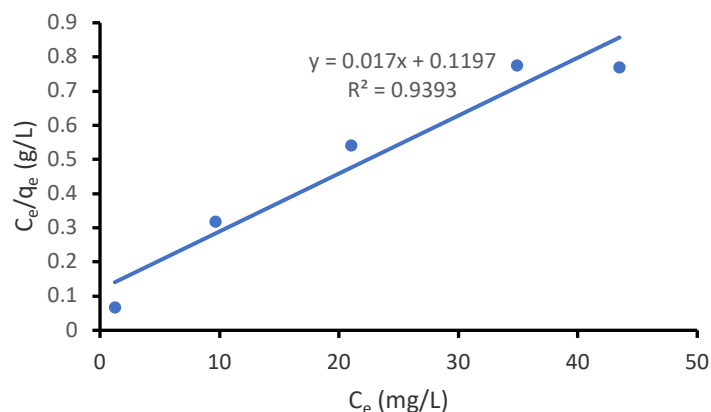
**Figure 5.** The MB dye adsorption uptakes at different contact time

### 3.4 Adsorption Isotherms

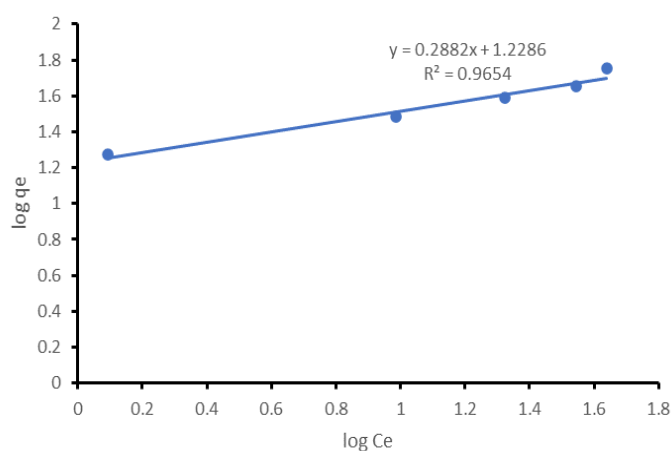
To seek for the best isotherm to fit the adsorption data, the graph's correlation coefficient, R<sup>2</sup> values were evaluated. Based on Figure 6, the results presented using the Langmuir model had produced a satisfactory adjustment where the R<sup>2</sup> value was 0.9393.

However, it is apparent that Freundlich model achieved higher R<sup>2</sup> value, 0.9654 as shown in Figure 7. The Freundlich model seemed to be more suitable for the MB adsorption on AC, signifying that the MB dye were adsorbed on multilayer of the adsorbent. The MB dye molecules adsorbed on the surface of the adsorbent heterogeneously. Based on Table 4, the K<sub>F</sub> and n<sub>F</sub> generated by Freundlich isotherm was 16.928 (mg/g) (L/mg)<sup>1/n</sup> and 3.470, respectively, while the Q<sub>m</sub> and K<sub>L</sub> were 58.824 mg/L and 0.142 L/mg, respectively. The n<sub>F</sub> in the Freundlich equation is 3.470 which means it is a favorable adsorption condition when the value of n is greater than 1 (Hameed *et al.*, 2008).





**Figure 6.** Langmuir adsorption isotherms.



**Figure 7.** Freundlich adsorption isotherms.

**Table 4.** Adsorption isotherm parameter.

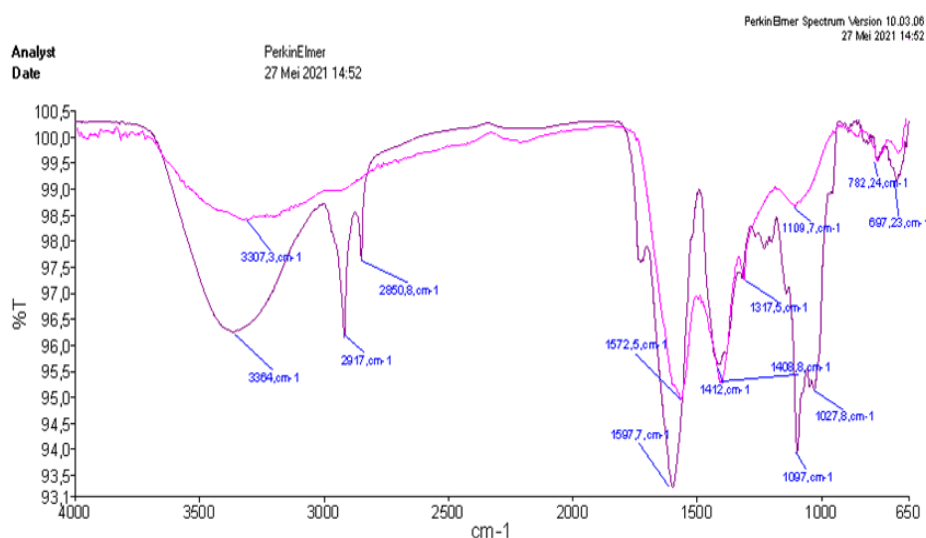
Isotherms	Parameters	
Langmuir	$Q_m$ (mg/g)	58.824
	$K_L$ (L/mg)	0.142
	$R^2$	0.9393
Freundlich	$n_F$	3.470
	$K_F$	16.928
	$R^2$	0.9654

### 3.5 Characterization of the Activated Carbon

The capability of AC to adsorb MB dye solution greatly depends on the structural development and also the surface region of the AC. Different radiation power and radiation time affected the adsorption properties of AC. Hence physical characterization was carried out to evaluate the AC produced from tobacco using FTIR, SEM and BET. The samples that have the best adsorption properties, which is AC produced at 364 W and 2 minutes was used for this characterization.

### 3.5.1 Chemical Bonds

The FTIR spectrum for precursor tobacco and AC is shown in Figure 8. The spectrum for both samples were estimated to be around 650 cm<sup>-1</sup> and 3700 cm<sup>-1</sup>.



**Figure 8.** FTIR spectra for the precursor tobacco (purple line) and AC (pink line).

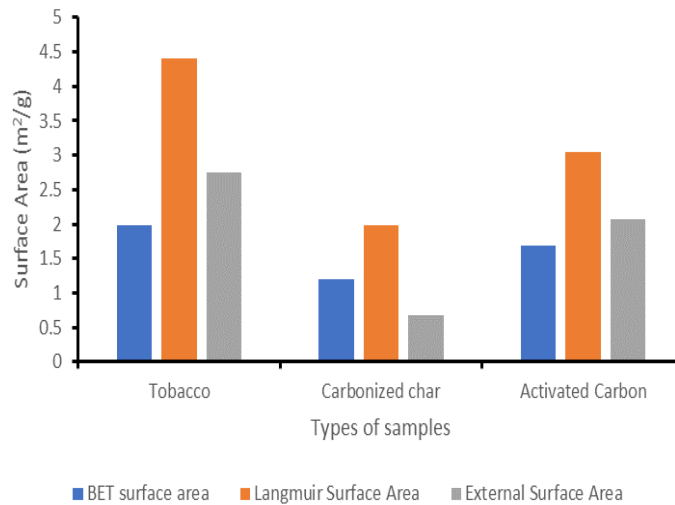
The peaks for tobacco shows a strong and broad band at 3364 cm<sup>-1</sup> represents O-H stretching vibrations due to the existence of hydroxyl functional groups and adsorbed water (Yakout & Sharaf El-Deen, 2016). The band of 2917 cm<sup>-1</sup> was addressed to the C-H stretching which represents the existence of alkane functional group. The strong peak at 1597 cm<sup>-1</sup> stands for N-O stretching for nitro compound, while peak at 1412 cm<sup>-1</sup> bending represents O-H functional group of alcohols (LibreTexts, 2021). The strong peak of 1097cm<sup>-1</sup> and 1027 cm<sup>-1</sup> are alcohol and C-N stretching amine group, respectively. Lastly, the peak which transmitted a weak spectrum of 697 cm<sup>-1</sup> represents monosubstituted benzene.

The FTIR spectrum for the AC shows a lower intensity peak at 3307 cm<sup>-1</sup> which means that the hydroxyl functional groups and adsorbed water was greatly reduced and removed during the processes. The peak of alkane group (C-H) was eliminated. Moreover, the peak for secondary alcohol and C-N stretching amine group at were also critically decreased.

In short, the peaks of hydroxyl functional groups (O-H), alkane group (C-H), secondary alcohol and C-N stretching amine group are strongly visible in precursor tobacco but are significantly diminished in the spectrum of AC. Heat treatment during carbonization of tobacco and activation of char had greatly reduced the moisture and volatile material. According to Zhang & Li, (2017), fibre treated at high temperature, under inert conditions eliminated the non-carbon element and volatile gasses such as water, ammonia and methane. The MB dye will have an affinity towards these functional group in AC and this enable the AC to remove the dye from aqueous water as well.

### 3.5.2 Surface area of AC

Figure 9 shows the result of the BET test. It was found that the surface area for tobacco, carbonized char and AC were 1.98, 1.21 and 1.69 m<sup>2</sup>/g, respectively.

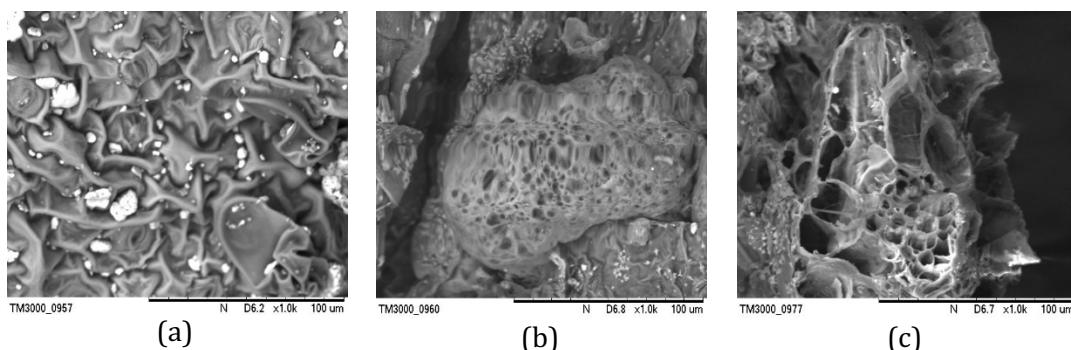


**Figure 9:** Surface area of tobacco, carbonized char and AC

The BET surface area was not much different from each other. When comparing with Yusop *et al.* (2021) studies, the BET surface area for acacia wood was 1.12 m<sup>2</sup>/g. However, after carbonization, the value increased to 425.41 m<sup>2</sup>/g in char. This shows that the surface area of AC produced from tobacco using microwave treatment resulted in very low surface area.

### 3.5.3 Surface Morphology

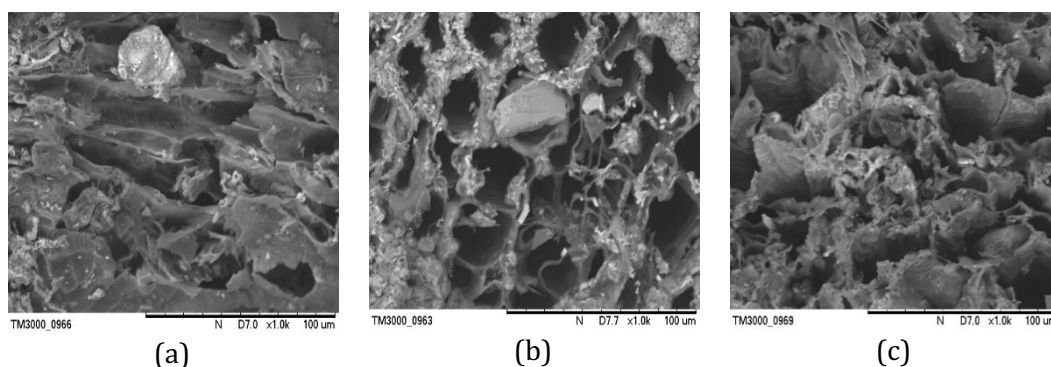
Figure 10 shows the surface of tobacco, char and the AC produced. The SEM images of tobacco surface shows uneven wrinkle-like structure with no pores. According to Suresh Kumar (2019), adsorbents need to have a high surface area with high adsorption capacity. A high surface area comes from pores which have a variety of pore sizes and different pore size distribution. Based on the SEM imaging, pores were more apparent after microwave-assisted irradiation under CO<sub>2</sub> on the AC produced. The treatment by using CO<sub>2</sub> pyrolysis had caused further treatment towards the surface of the char and also the internal side of the pores.



**Figure 10.** SEM images of (a) precursor tobacco, (b) carbonized tobacco (char) and (c) AC, at 1000x magnification

Figure 11 compares the AC magnification images between different irradiation times (2, 4 and 6 minutes) for 364 W. It shows that the pores were largest for the ones irradiated for 4 minutes,

followed by 2 minutes. However, when irradiated for 6 minutes, the pores seemed to deteriorate which might be the reason for the lower MB removal. This might be due to excessive energy causing large amounts of carbon to be burnt and rupturing the pores. Instead of producing AC, the char is burnt into ash, and it greatly reduces the adsorption properties.



**Figure 11.** SEM images of AC produced at (a) 364W for 2 minutes, (b) 364W for 4 minutes, and (c) 364W for 6 minutes, at 1000x magnification

Due to the low surface area, the capability of the AC to adsorb MB dye might be assisted by diversified factors that contribute to the adsorption mechanism (Manfrin *et al.*, 2021). One of the factors could be adsorption towards the functional groups remaining in the AC as described by Tran *et al.* (2017).

The findings from this study suggested that cigarette tobacco has potential to be used as AC when irradiated with microwave under CO<sub>2</sub>.

#### 4. CONCLUSION

Cigarettes confiscated by RMCD Perlis have been successfully utilized as precursors to produce AC by microwave-assisted CO<sub>2</sub> pyrolysis. The treatment by using CO<sub>2</sub> pyrolysis had caused further treatment towards the surface of the char and also the internal side of the pores. This caused the development of pores to be further enhanced. The highest yield of AC, 31% was obtained at 616-Watt irradiation for 6 minutes. However, the AC produced at radiation power of 364 Watt for 2 minutes exhibited the highest MB dye removal, 87.4%. Based on surface area and functional groups analysis, the removal of MB dye was not heavily dependent on the porosity on the AC, but also rely on the functional groups in the AC. The functional groups can further attract MB for adsorption and thus resulting in better adsorption properties. Freundlich isotherm model generated satisfactory adjustment with R<sup>2</sup> values of 0.9654, higher than Langmuir model, 0.9393, showing that adsorption of MB dye occurred on multilayers of the adsorbent.

#### ACKNOWLEDGMENT

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