# CHAPTER IV

# RESULTS AND DISCUSSIONS

## 4.1 Properties of coconut shells

Raw coconut shell was preliminarily analyzed the proximate component as shown in Table 4.1. It was observed that raw coconut shell was 6.8 % moisture, small ash 1.3 %, high volatile matter 80.1% and fixed carbon 18.9%. It was because of the low fixed carbon, the carbonization was needed to change the higher fixed carbon before activation. Figures 4.1-4.2 showed before and after activation.

Table 4.1 Proximate analysis of coconut shells.

| Coconut Shells | Dry basis | | |
|---|---|---|---|
| M (%) | Ash (%) | VM (%) | FC (%) |
| 6.80 | 1.30 | 80.10 | 18.90 |

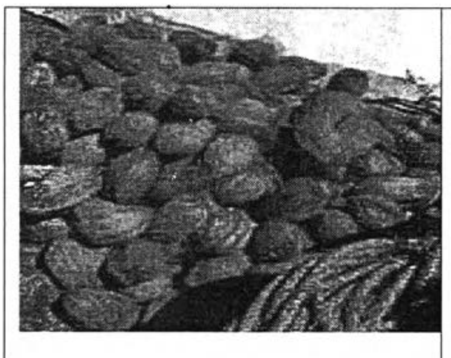

Figure 4.1 Coconut shells before carbonization.

Figure 4.2 Coconut shells after carbonization.

## 4.2 Results and discussion of the experiments

### 4.2.1 Carbonization

Carbonization was carried out at the temperature of 300, 350, 400 and 500$^{0}$C. Each temperature was varied at the time of 45, 60, 90 and 120 min for carbonization. The final products of this step are called "char". The proximate analysis of chars was shown in Table 4.2 and Figures 4.3-4.10.

Table 4.2 Characteristics of carbonized at different temperatures and times.

| Raw materials* | | | | On dry basis | | |
|---|---|---|---|---|---|---|
| Temperature (°C) | Time (min) | YC (%wt) | M (%wt) | A (%wt) | VM (%wt) | FC (%wt) |
| 300 | 45 | 39.08 | 1.68 | 1.19 | 31.93 | 55.19 |
| | 60 | 38.70 | 2.38 | 1.41 | 31.13 | 58.08 |
| | 90 | 38.56 | 1.692 | 1.71 | 29.72 | 65.39 |
| | 120 | 38.02 | 1.709 | 1.81 | 29.52 | 67.76 |
| 350 | 45 | 31.78 | 1.70 | 1.64 | 25.32 | 72.63 |
| | 60 | 31.52 | 1.88 | 1.88 | 26.09 | 72.03 |
| | 90 | 31.24 | 2.02 | 1.66 | 25.03 | 73.31 |
| | 120 | 31.18 | 2.12 | 1.73 | 26.10 | 72.16 |
| 400 | 45 | 29.92 | 1.99 | 1.72 | 24.39 | 73.89 |
| | [illegible] | 29.53 | 1.98 | 1.19 | 24.86 | 73.95 |
| | 90 | 28.07 | 1.54 | 1.75 | 24.85 | 73.40 |
| | 120 | 28.99 | 1.73 | 1.73 | 25.57 | 72.70 |
| 450 | 45 | 27.85 | 2.20 | 1.76 | 22.36 | 75.88 |
| | 60 | 27.17 | 1.85 | 2.04 | 21.58 | 76.38 |
| | 90 | 27.01 | 2.33 | 1.76 | 20.06 | 78.18 |
| | 120 | 24.81 | 2.66 | 1.77 | 21.87 | 76.36 |
| 500 | 45 | 26.29 | 2.96 | 2.55 | 20.08 | 77.37 |
| | 60 | 24.09 | 2.66 | 2.82 | 18.92 | 78.26 |
| | 90 | 23.77 | 2.24 | 2.29 | 18.83 | 78.88 |
| | 120 | 22.93 | 2.03 | 1.74 | 18.40 | 79.86 |

* These values in this table were average, which the characteristics of each sample was repeated twice.

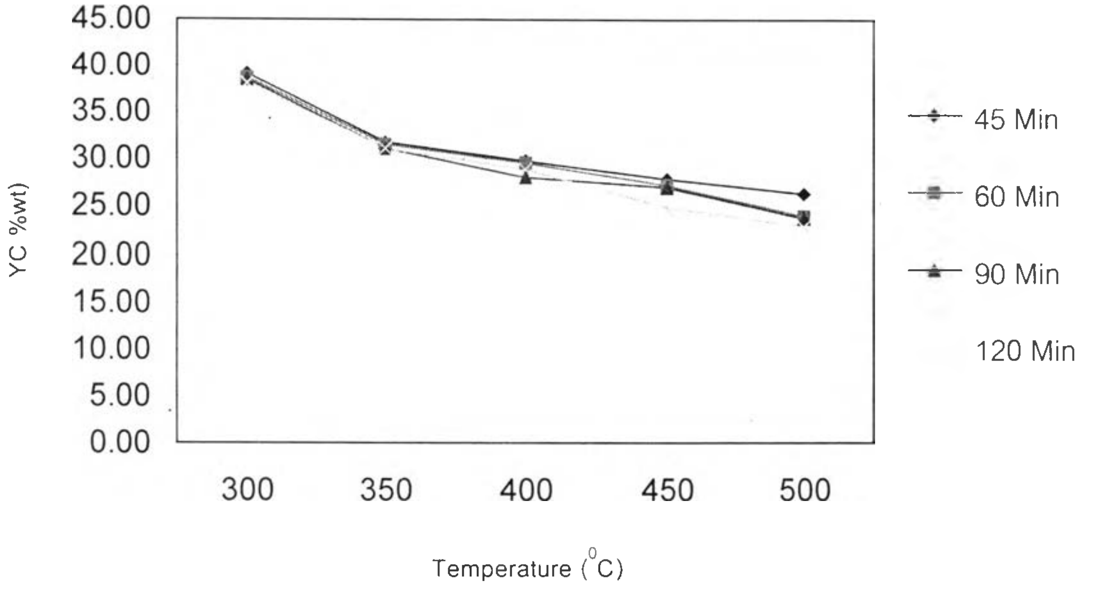


Figure 4.3 Effect of temperature on % yield at different times.

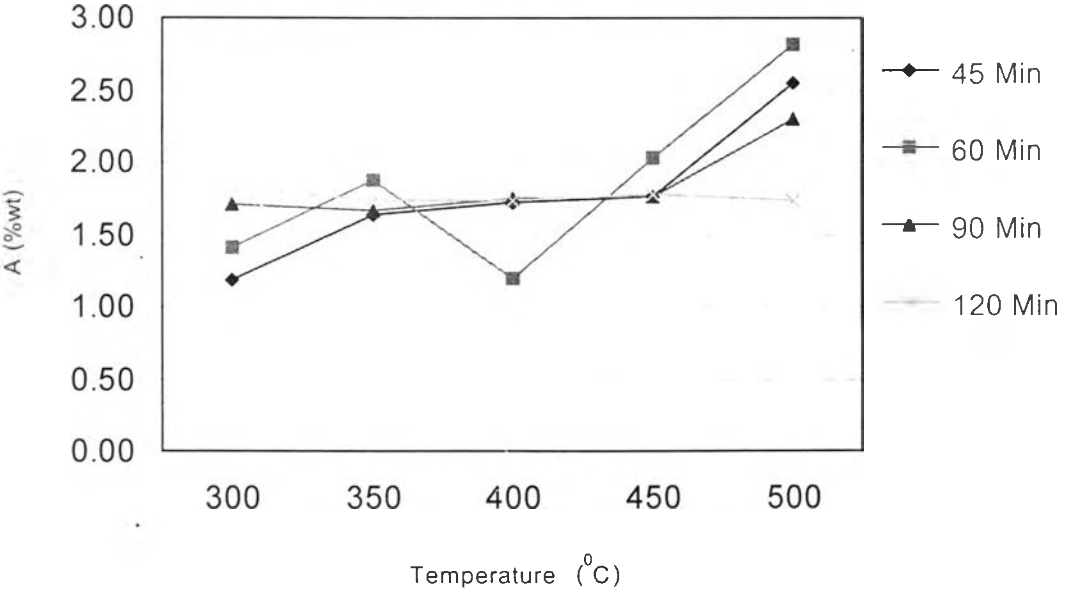


Figure 4.4 Effect of temperature on % ash at different times.

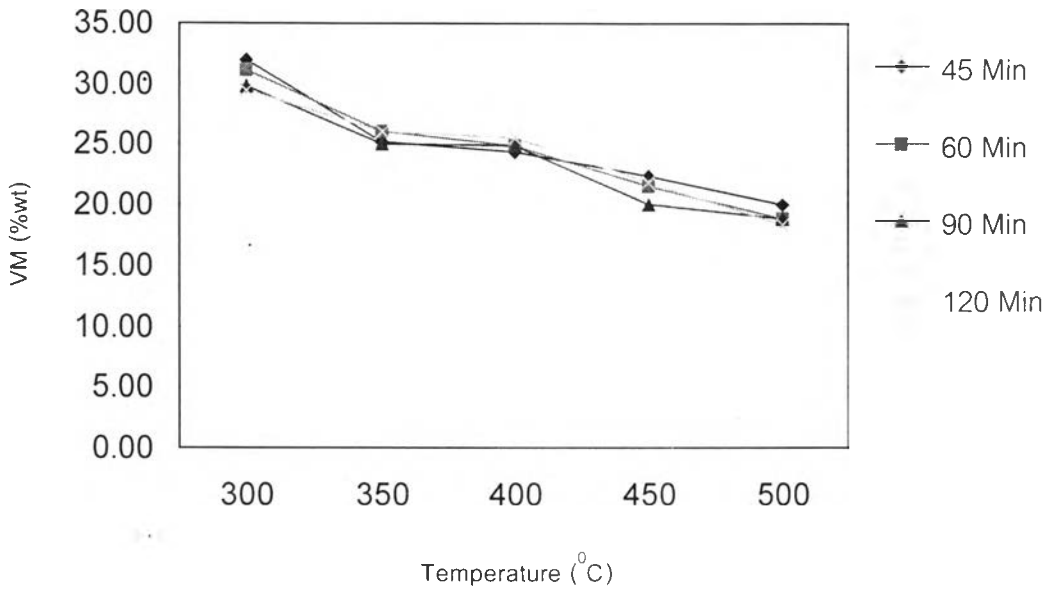


Figure 4.5 Effect of temperature on % volatile at different times.

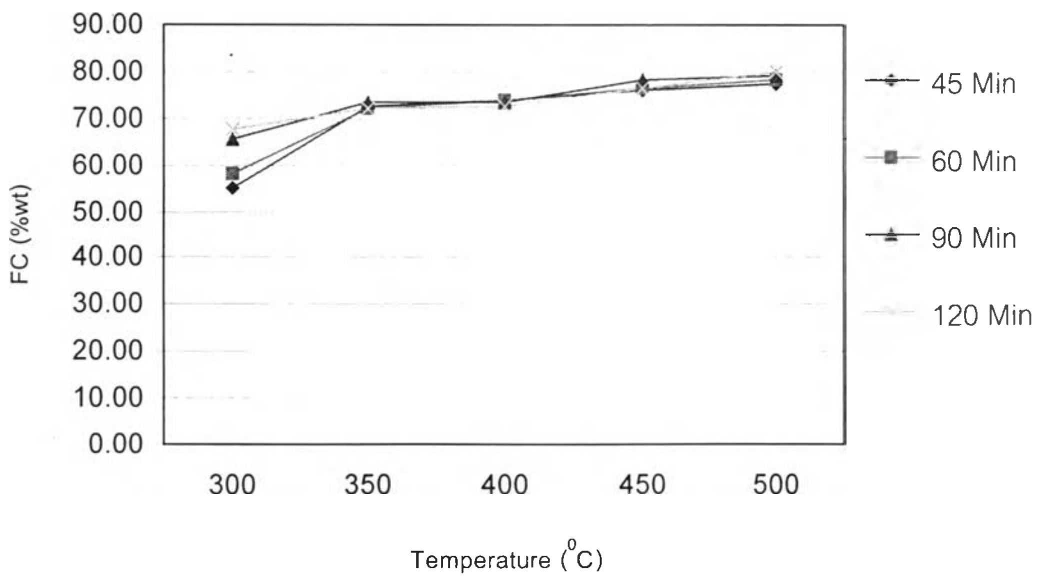


Figure 4.6 Effect of temperature on % fixed carbon at different times.

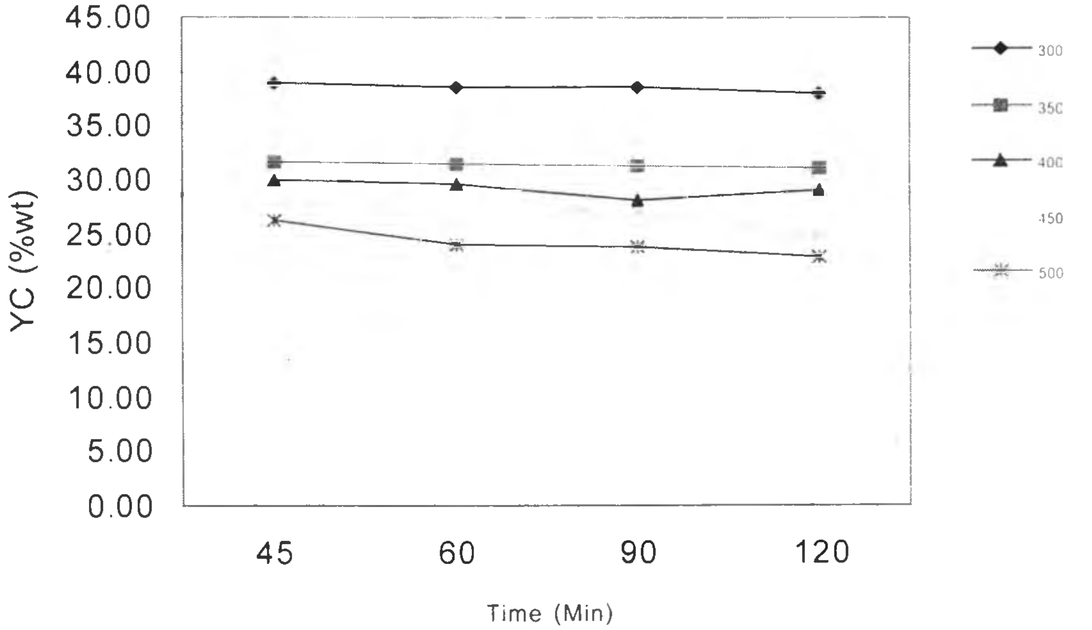


Figure 4.7 Effect of time on % yield at different times.

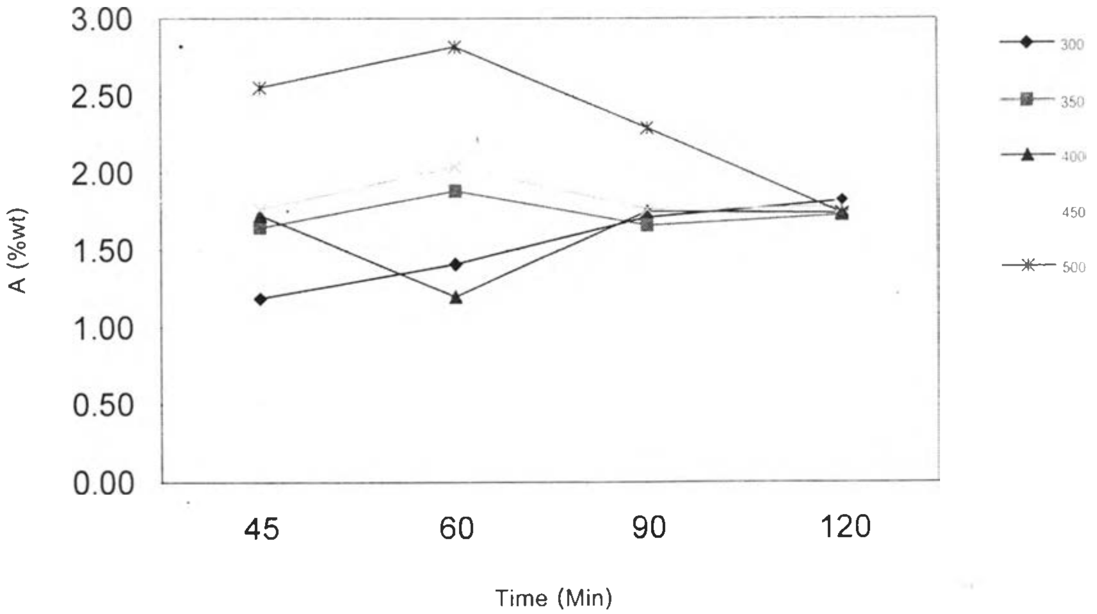


Figure 4.8 Effect of time on % ash at different times.

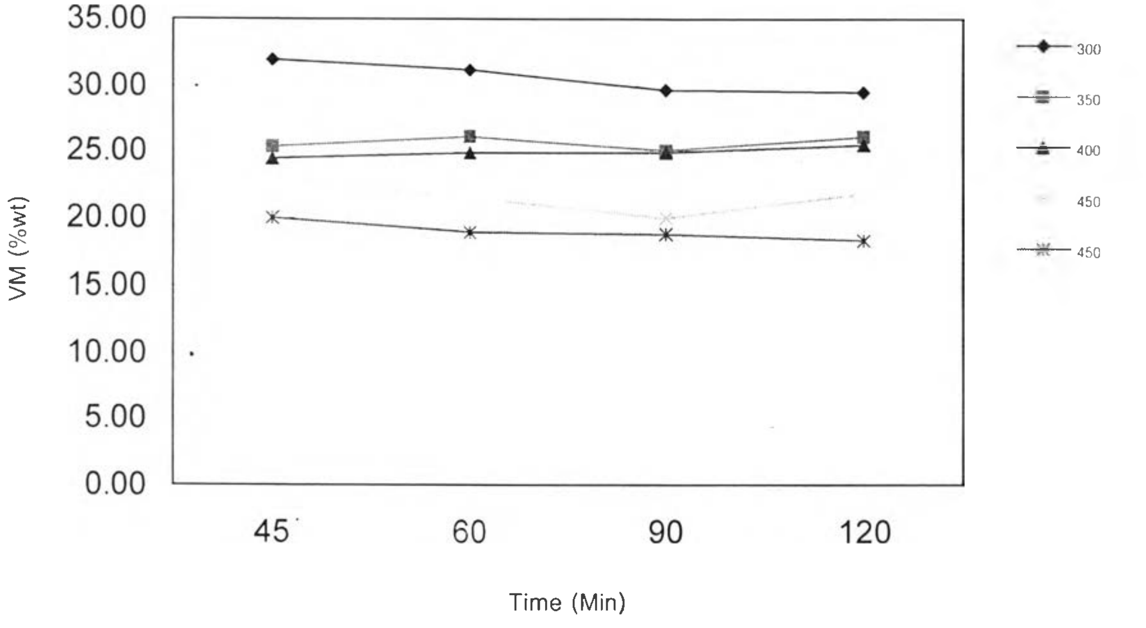


Figure 4.9 Effect of time on % volatile at different times.

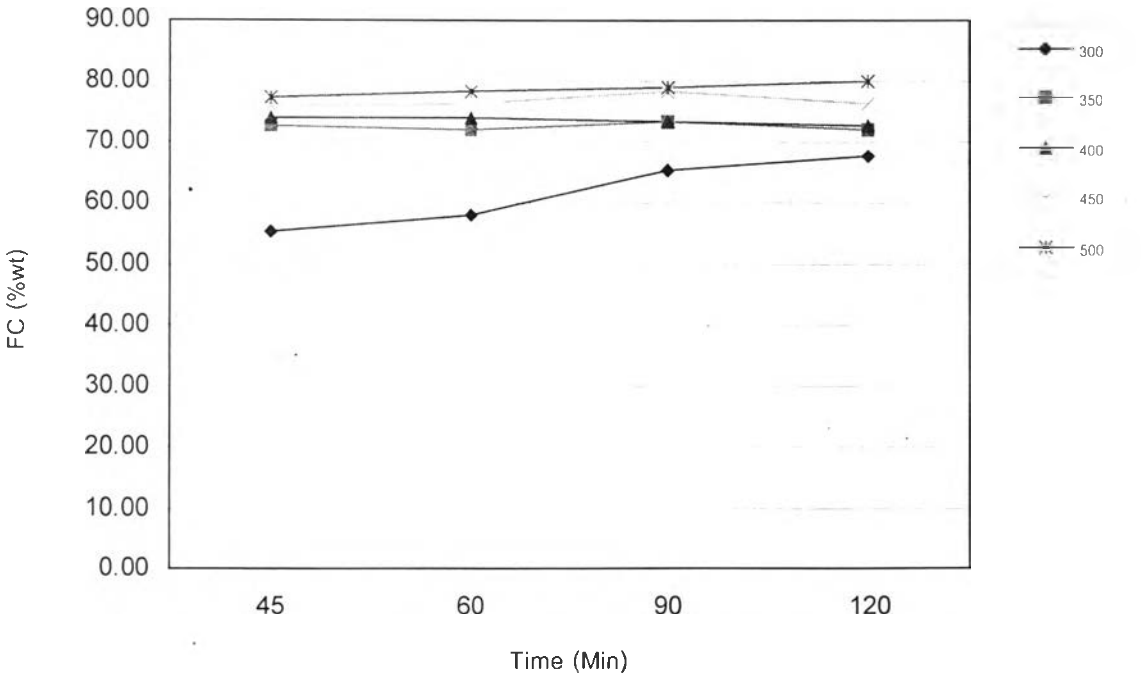


Figure 4.10 Effect of time on % fixed carbon at different times.

#### 4.2.1.1 The effect of temperature for carbonization

**Table 4.2** and **Figure 4.3** show that the % yields of various **carbonization** decrease while the temperature increase because at high temperature, the carbon in coconut shells is progressively gasified and diffused with volatile, so the volatile in the products decreases, resulting in the decrease of %yield. The %change of %yield showed in **Table 4.3**.

**Table 4.3** The % change of characteristics of char from coconut shells when carbonization temperature increases from 300 to 500 $^{0}$C for 45, 60, 90 and 120 min.

| Time (min) | Temperature ($^{0}$C) | % YC | %A | %VM |
|---|---|---|---|---|
| 45 | 300 | 39.08 | 1.19 | 31.93 |
| | ↓ | ↑ | ↓ | ↑ |
| | 500 | 26.29 | 2.55 | 20.08 |
| | % change | -32.73 | +114.29 | -37.11 |
| 60 | 300 | 38.70 | 1.41 | 31.13 |
| | ↓ | ↓ | ↑ | ↑ |
| | 500 | 24.09 | 2.82 | 18.92 |
| | % change | -37.75 | +100 | -39.22 |
| 90 | 300 | 38.56 | 1.71 | 29.72 |
| | ↓ | ↓ | ↑ | ↑ |
| | 500 | 23.77 | 2.29 | 18.83 |
| | % change | -38.36 | +33.92 | -36.64 |
| 120 | 300 | 38.02 | 1.81 | 29.52 |
| | ↓ | ↓ | ↑ | ↑ |
| | 500 | 22.93 | 1.94 | 18.40 |
| | % change | -39.69 | +7.18 | -37.67 |

↓: increase ↑: decrease

From Table 4.2 and Figure 4.4, the change of characteristics of %ash in temperature range of 300$^0$C to 400$^0$C is small because at low temperature, only the volatile is removed. But at high temperature, some reaction too extremely and change into ash. Thus, when the temperature increases from 400$^0$C to 500$^0$C, the %ash in range of these temperatures is shown in Table 4.3.

In Table 4.2 and Figure 4.5, the volatile matter in the temperature range of 300-400$^0$C decreases quickly because the low temperature has largely effected the rate of diffusion of the volatile out of the surface of particle. In contrast, this effect is less when the temperature is above 400$^0$C. With this effect, the rate of diffusion is more or less stable, and the % change of volatile matter for 300-500$^0$C is shown in Table 4.3.

In Table 4.2 and Figure 4.6, while the temperature increases from 300 to 350$^0$C, the % fixed carbon increases largely owing to immediately taking place of carbonization. The % fixed carbon has the highest increase at 400$^0$C. But above 400$^0$C, it would be a little bit increasing or decreasing. When the temperature increases above 400$^0$C, some C-C bonds in this structure was broken. Then carbon can react more with air and changes finally into ash thus the % fixed carbon has a trend to decrease. At 450-500$^0$C the % fixed carbon's trend is constant.

The optimum condition for carbonization of used tired in this experiment is 400$^0$C because the characteristic of the chars at this temperature has 20-25 % of volatile matter which is appropriate for activation. These carbonized coconut shells, or chars, have high fixed carbon, low ash and suitable yield.

4.2.1.2 The effect of time for carbonization.

Table 4.2 and Figure 4.7, the increase of carbonization time leads to the decrease of the % yield because carbon in coconut shells is progressively gasified and diffused out with volatile. The % change of % yield is shown in Table 4.4.

Table 4.4 The % change of characteristics of char from coconut shells when carbonization temperature increases from 45 to 120 min for 300, 350, 400, 450 and 500 $^{0}C$

| Temperature ($^{0}C$) | Time (min) | % YC | %A | %VM | %FC |
|---|---|---|---|---|---|
| 300 | 45 | 39.08 | 1.19 | 31.93 | 55.19 |
| | ↓ | ↓ | ↓ | ↓ | ↑ |
| | 120 | 38.02 | 1.18 | 29.52 | 67.76 |
| | % change | -2.71 | -0.84 | -7.55 | +22.78 |
| 350 | 45 | 31.78 | 1.64 | 25.32 | 72.63 |
| | ↓ | ↓ | ↑ | ↑ | ↓ |
| | 120 | 31.18 | 1.73 | 26.10 | 72.16 |
| | % change | -1.88 | +5.49 | +3.08 | -0.65 |
| 400 | 45 | 29.92 | 1.72 | 24.39 | 73.89 |
| | ↓ | ↓ | ↑ | ↑ | ↑ |
| | 120 | 18.99 | 1.73 | 25.57 | 72.70 |
| | % change | -36.53 | +0.58 | +4.84 | -36.64 |
| 450 | 45 | 27.85 | 1.76 | 22.36 | 75.88 |
| | ↓ | ↓ | ↑ | ↓ | ↑ |
| | 120 | 24.81 | 1.77 | 21.87 | 76.36 |
| | % change | -10.92 | +0.58 | -2.19 | -36.64 |
| 500 | 45 | 26.29 | 2.55 | 20.08 | 77.37 |
| | ↓ | ↓ | ↓ | ↓ | ↑ |
| | 120 | 22.93 | 1.94 | 18.40 | 79.86 |
| | % change | -12.78 | -23.92 | -8.37 | -37.67 |

↓ : increase ↑ : decrease

In Table 4.2 and Figure 4.8,the % ash increases with and increase in time because at long time, carbon in coconut shells is higher progressively gasified than at short time. The % change of % ash is shown in Table 4.4.

In Table 4.2 and Figure 4.9, the decrease of volatile matter in the time range of 45-60 min is faster than at 60-90 min because in the time range (45-60 min), only volatile at the surface is removed. When time increases above 60 min, volatile in the interior particle would be removed, but with more difficulty than volatile at the surface. The % change of volatile matter is shown in Table 4.4.

In Table 4.1 and Figure 4.10, the % fixed carbon increases with an increase in time because increase of time leads to the increase of volatile diffusion.

Consider from these results and characteristics, 45 and 60 min are better time for carbonization than 90 min. But 60 min is the best, because the chars with have been carbonized in 60 min will have the highest % fixed carbon. Thus the optimum time for carbonization of coconut shells is 60 min.

The optimum condition for carbonization of coconut shells in this experiment was 400 $^{0}$C for 60 min. The chars in this condition obtained yield of 29.53%, ash of 1.19%, volatile matter of 24.86% as and fixed carbon of 73.95%.

4.2.2 Activation

The chars obtained from the optimum in carbonization step was crushed and sieved to the particle sizes of 0.355-0.60,0.60-1.18,1.18-2.36 and 2.36-4.75 mm. The activation was carried out for determination the effect of parameters following these;

4.2.2.1 The effect of temperature and time for activation.

The 10 g of 1.18-2.36 mm of chars were charged into the fixed bed reactor followed with $CO_2$ and the air at the flow rate of 0.5 l/min and 0.005 l/min, at 800,850, 900, 950 and 1,000 $^0$C, respectively. Then the superheated steam at the flow rate of 0.01 l/min was passed through the reactor for 60,120, 180 and 240 min. The results are shown in **Table 4.5** and **Figures 4.11-4.23**.

**Table 4.5** Characteristics of activated carbon from coconut shells at temperatures and times (size 1.18-2.36 mm, 10 g, air 0.005 l/min, $CO_2$ 0.5 l/min and superheated steam at the flow rate of 10 g/min)

| Condition | | | On dry basis | | | | |
|---|---|---|---|---|---|---|---|
| Temperature ($^0$C) | Time (min) | Y (%) | BD (g/cm$^3$) | Ash (%) | IA (mg/g) | MB (mg/g) | $S_{BET}$ (m$^2$/g) |
| 800 | 60 | 52.22 | 0.6652 | 2.24 | 480 | 101 | 467 |
| | 120 | 51.16 | 0.6380 | 2.32 | 515 | 105 | 469 |
| | 180 | 49.25 | 0.5969 | 2.46 | 559 | 109 | 518 |
| | 240 | 47.13 | 0.5444 | 2.49 | 567 | 117 | 663 |
| 850 | 60 | 49.91 | 0.6146 | 2.39 | 508 | 110 | 492 |
| | 120 | 48.37 | 0.6057 | 2.47 | 538 | 118 | 537 |
| | 180 | 46.32 | 0.5465 | 2.57 | 588 | 120 | 592 |
| | 240 | 44.49 | 0.5337 | 2.86 | 673 | 135 | 670 |
| 900 | 60 | 41.41 | 0.6057 | 2.59 | 642 | 134 | 817 |
| | 120 | 37.24 | 0.5654 | 2.83 | 660 | 162 | 875 |
| | 180 | 34.93 | 0.5301 | 3.10 | 706 | 181 | 920 |
| | 240 | 31.22 | 0.5002 | 3.15 | 781 | 183 | 970 |
| 950 | 60 | 33.72 | 0.5822 | 3.80 | 954 | 187 | 982 |
| | 120 | 34.31 | 0.5543 | 3.82 | 948 | 195 | 935 |
| | 180 | 30.87 | 0.5121 | 4.36 | 905 | 197 | 972 |
| | 240 | 27.64 | 0.4807 | 4.34 | 894 | 198 | 988 |
| 1,000 | 60 | 30.71 | 0.5541 | 4.45 | 902 | 199 | 911 |
| | 120 | 26.66 | 0.5096 | 5.22 | 845 | 201 | 844 |
| | 180 | 19.18 | 0.4725 | 6.19 | 714 | 203 | 812 |
| | 240 | 14.95 | 0.4288 | 6.71 | 653 | 206 | 809 |

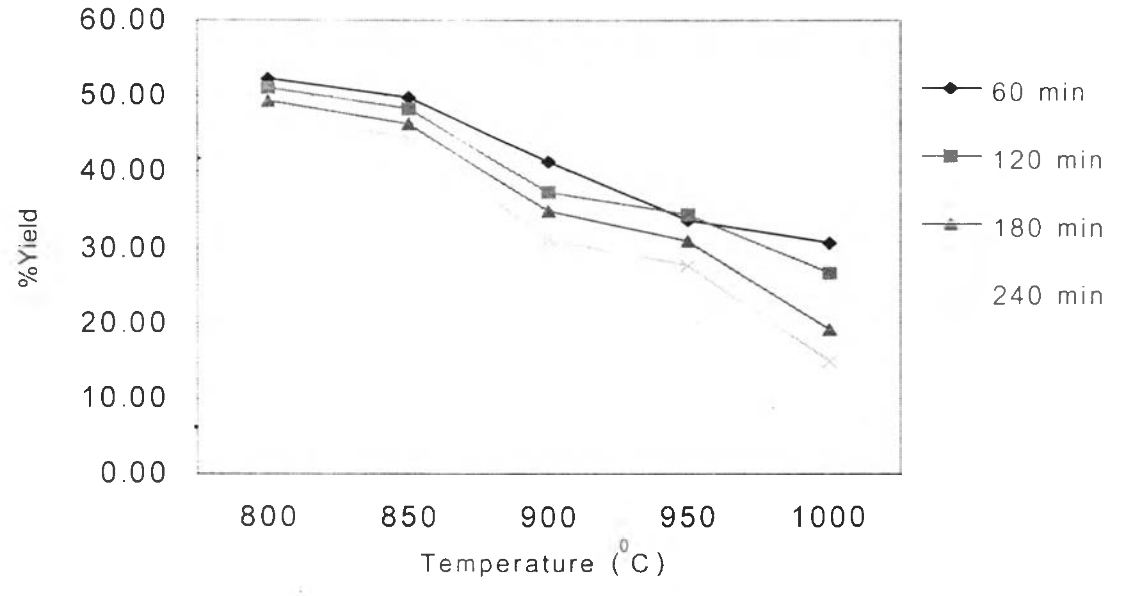


Figure 4.11 Effect of temperature on % yield at different time (size 1.18 – 2.36 mm, 10 g, air 0.005 l/min, $CO_2$ 0.5 l/min and $H_2O$ 0.01 l/min).

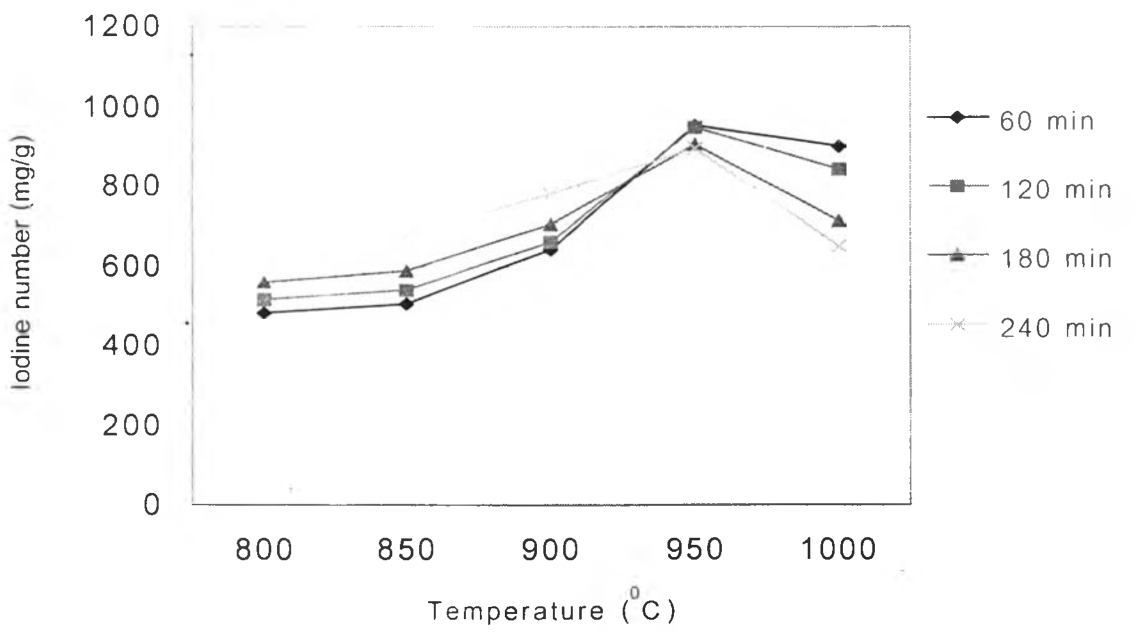


Figure 4.12 Effect of temperature on % iodine number at different time (size 1.18 – 2.36 mm, 10 g, air 0.005 l/min, $CO_2$ 0.5 l/min and $H_2O$ 0.01 l/min).

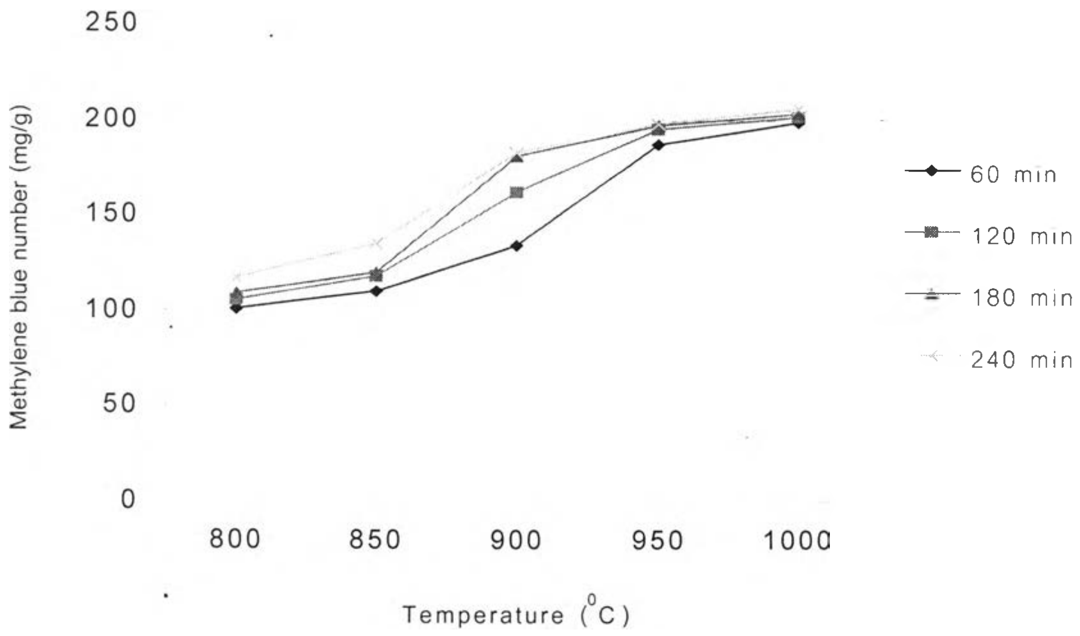


Figure 4.13 Effect of temperature on methylene blue number at different time (size 1.18 – 2.36 mm, 10 g, air 0.005 l/min, $CO_2$ 0.5 l/min and $H_2O$ 0.01 l/min).

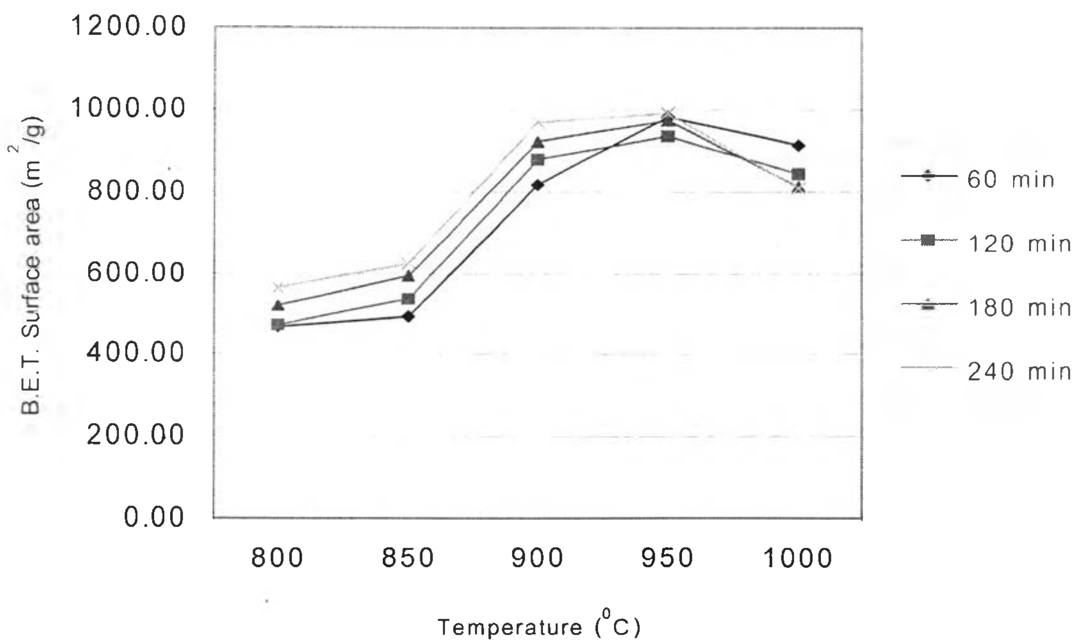


Figure 4.14 Effect of temperature on B.E.T. surface area at different time (size 1.18 – 2.36 mm, 10 g, air 0.005 l/min, $CO_2$ 0.5 l/min and $H_2O$ 0.01 l/min).

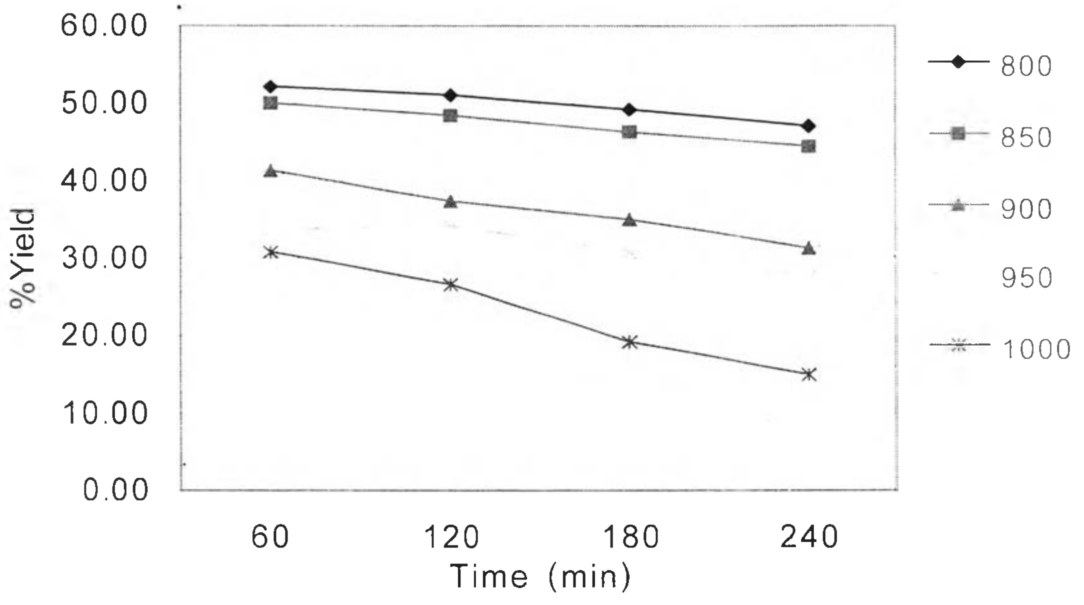


Figure 4.15 Effect of time on % yield at different time (size 1.18 – 2.36 mm, 10 g, air 0.005 l/min, $CO_2$ 0.5 l/min and $H_2O$ 0.01 l/min).

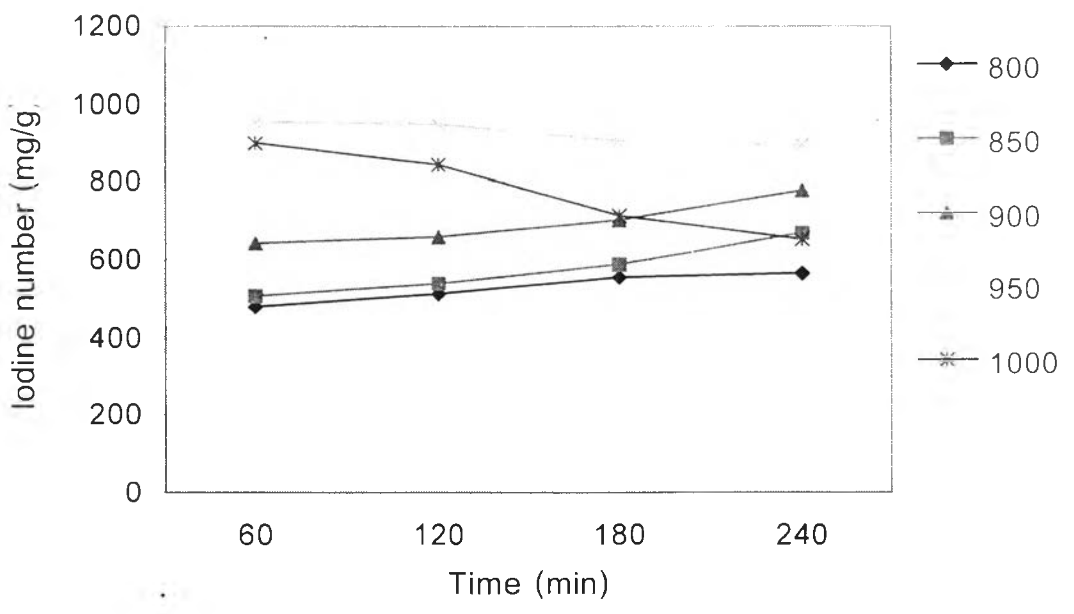


Figure 4.16 Effect of time on iodine number at different time (size 1.18 – 2.36 mm, 10 g, air 0.005 l/min, $CO_2$ 0.5 l/min and $H_2O$ 0.01 l/min).

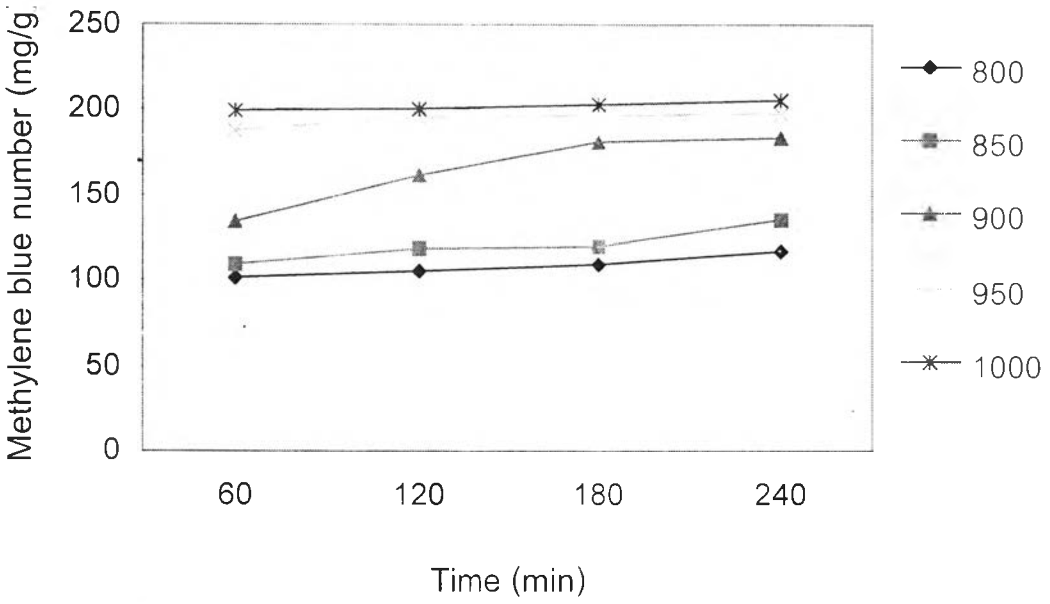


Figure 4.17 Effect of time on methylene blue number at different time (size 1.18 – 2.36 mm, 10 g, air 0.005 l/min, $CO_2$ 0.5 l/min and $H_2O$ 0.01 l/min).

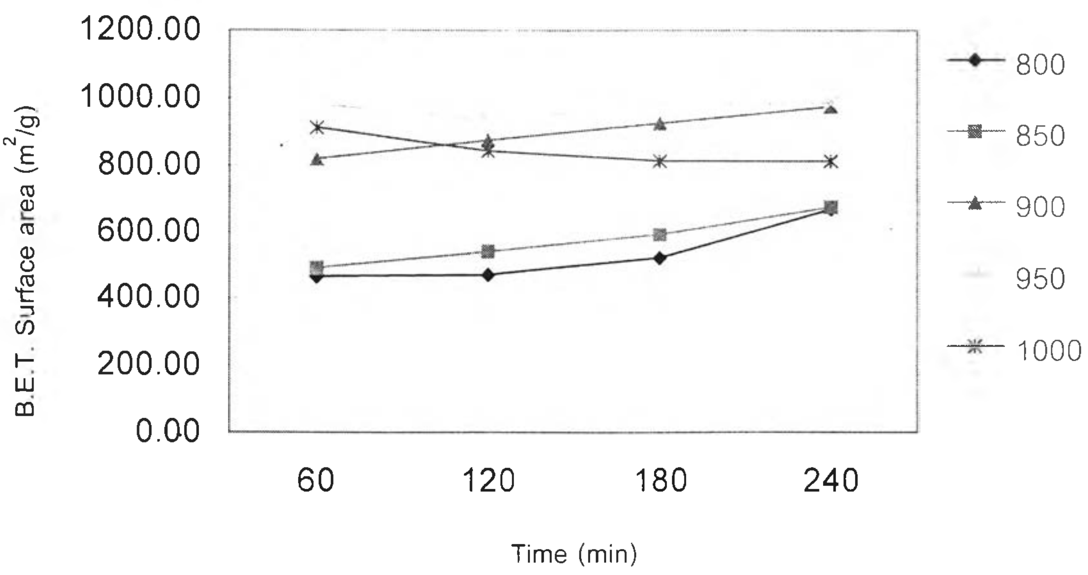


Figure 4.18 Effect of time on B.E.T. surface area at different time (size 1.18 – 2.36 mm, 10 g, air 0.005 l/min, $CO_2$ 0.5 l/min and $H_2O$ 0.01 l/min).

-The effect of activation temperature.

**Table 4.5** and **Figures 4.11-4.14** showed that when temperature increases from 800 to 1,000 $^0$C, the amount of product decreases. When the temperature is high, tar and volatile matter, which obstruct absorption of activating substance in pores, disappear. Thus, more activating substance spreads into the structure. Then char, which is partly burnt, changes into ashes. When temperature increases from 800 to 950 $^0$C, iodine absorption capacity increases slightly because the temperature is not high enough to chase volatile matter and tar from the structure. Tar and volatile matter obstructing in the pores cause activating substance to uncomfortably expanding into the structure, instead, the substance only passes the outer surface of coal. Thus only a little number of pores were produced at this temperature. However, when temperature changed from 800-950 $^0$C, iodine absorption capacity increases dramatically because VM and Tar obstructing in pores are immediately chased away. Thus, activating substance can better spread onto the char structure. New pores were produced and old pores were expanded.

From **Table 4.5**, when the activation time increases, % ash increases. Because of high temperature, the carbon in char stucture is higher gasified from particles than at low temperature. But the % change of ash in activation step increases lower than those of with carbonization step, it seems that the steam reduces the change of carbon into ash. Then in the final activation, it is necessary to feed steam into the activator for a moment before finishing the activation. The % change of ash is shown in **Table 4.6**.

From **Table 4.5 and Figure 4.11**, the % yield decreases when activation temperature increases from 800 to 1,000$^o$C. Because at high temperature, superheated steam and carbon dioxide attack chars better than at low temperature. Thus the increasing of temperature leads to the increasing of the porosity development; as a result, the weight of activated carbon decreases. Moreover, the %change of % yield showed in **Table 4.6**.

Table 4.6 The % change of characteristics of activated carbon from coconut shells when activation temperature increase from 800 to 1,000 °C for 60, 120, 180 and 240 min (size 1.18 – 2.36 mm, 10 g, $CO_2$ 0.5 l/min, $H_2O$ 10 g/min and air 0.005 l/min).

| Time (min) | Temperature ($^0C$) | %Y | BD ($g/cm^3$) | %Ash | IA (mg/g) | MB (mg/g) | $S_{B.E.T.}$ ($m^2/g$) |
|---|---|---|---|---|---|---|---|
| 60 | 800 | 52.22 | 0.6652 | 2.24 | 480 | 101 | 467 |
| | ↓ | ↑ | ↑ | ↓ | ↓ | ↓ | ↓ |
| | 1,000 | 30.71 | 0.5541 | 4.45 | 902 | 199 | 911 |
| | % change | -41.19 | -16.70 | +98.66 | +87.92 | +97.03 | +95.03 |
| 120 | 800 | 51.16 | 0.6380 | 2.32 | 515 | 105 | 469 |
| | ↓ | ↑ | ↑ | ↓ | ↓ | ↓ | ↓ |
| | 1,000 | 26.66 | 0.5096 | 5.22 | 845 | 201 | 844 |
| | % change | -47.89 | -20.13 | +125 | +64.08 | +91.43 | +79.67 |
| 180 | 800 | 49.25 | 0.5969 | 2.46 | 559 | 109 | 518 |
| | ↓ | ↑ | ↑ | ↓ | ↓ | ↓ | ↓ |
| | 1,000 | 19.18 | 0.4725 | 6.19 | 714 | 203 | 812 |
| | % change | -20.84 | -20.84 | +151.63 | +27.73 | +86.24 | +56.60 |
| 240 | 800 | 47.13 | 0.5444 | 2.49 | 567 | 117 | 663 |
| | ↓ | ↑ | ↑ | ↓ | ↓ | ↓ | ↓ |
| | 1,000 | 14.95 | 0.4288 | 6.71 | 653 | 206 | 809 |
| | % change | -68.28 | -21.23 | +169.48 | +15.17 | +76.07 | +21.91 |

↓ : increase , ↑ : decrease

These results show that the optimum temperature is 950 $^0C$ since it has maximum iodine and B.E.T. surface area.

-The effect of activation time.

In **Table 4.5** and **Figure 4.15** showed that when the % yield decrease while activation time increases because the partical chars are progressively gasified by air and the porosity development increases with an increase in time, so the % yield decreases. The % change of % yield is shown in **Table 4.7**.

In **Table 4.5** and **Figures 4.20-4.22** show that the iodine number, the methylene blue number and B.E.T. surface area increase when the activation time increase from 800-950 $^0$C. Because tarry matter and the products of deposition in pores are removed while the superheated steam and the carbon dioxide attack chars; as a result, a lot of new pores are developed. Above 950 $^0$C for 120 min of activation time, iodine number and the B.E.T. surface area are slowly decreasing while the methylene blue number still increases because the micropores are chaged into the mesopores which can adsorb the methylene blue well, so the methylne blue number increases. The % change of iodine number, methylene blue number and B.E.T. surface area are shown in **Table 4.7**.

**Table 4.7** The % change of characteristics of activated carbon from coconut shells when activation time increases from 60 to 240 min for 800, 850, 900, 950 and 1,000$^0$C. (size 1.18 – 2.36 mm, 10 g, $CO_2$ 0.5 l/min, $H_2O$ 10 g/min and air 0.005 l/min).

| Temperature ($^0$C) | Time (min) | % Y | BD (g/cm$^3$) | %Ash | IA (mg/g) | MB (mg/g) | $S_{B.E.T}$ (m$^2$/g) |
|---|---|---|---|---|---|---|---|
| 800 | 60 | 52.22 | 0.6652 | 2.24 | 480 | 101 | 467 |
| | ↓ | ↑ | ↑ | ↓ | ↓ | ↓ | ↓ |
| | 240 | 47.13 | 0.5444 | 2.49 | 567 | 117 | 663 |
| | % change | -9.75 | -18.16 | +11.16 | +18.13 | -15.84 | +42.04 |
| 850 | 60 | 49.91 | 0.6146 | 2.39 | 508 | 110 | 492 |
| | ↓ | ↑ | ↑ | ↓ | ↓ | ↓ | ↓ |
| | 240 | 44.49 | 0.5337 | 2.86 | 673 | 135 | 670 |
| | % change | -10.86 | -13.16 | +19.67 | +32.48 | +22.73 | +36.22 |
| 900 | 60 | 41.41 | 0.6057 | 2.59 | 642 | 134 | 817 |
| | ↓ | ↑ | ↑ | ↓ | ↓ | ↓ | ↓ |
| | 240 | 31.22 | 0.5002 | 3.15 | 781 | 183 | 970 |
| | % change | -24.61 | -17.42 | +21.62 | +21.65 | +36.57 | -18.80 |
| 950 | 60 | 33.72 | 0.5822 | 3.80 | 954 | 187 | 982 |
| | ↓ | ↑ | ↑ | ↓ | ↑ | ↓ | ↑ |
| | 240 | 27.64 | 0.4807 | 4.34 | 894 | 198 | 988 |
| | % change | -18.03 | -17.43 | +14.21 | -6.29 | +5.88 | +0.64 |
| 1,000 | 60 | 30.71 | 0.5541 | 4.45 | 902 | 199 | 911 |
| | ↓ | ↑ | ↑ | ↓ | ↑ | ↓ | ↑ |
| | 240 | 14.95 | 0.4288 | 6.71 | 653 | 206 | 809 |
| | % change | -51.32 | -22.61 | +50.79 | -27.61 | +3.52 | -11.22 |

↓ : increase ↑ : decrease

The above results show that the optimum time for the activation is 60 min because the the activated carbon obtained the optimum methylene blue number and yield. Thus, the optimum condition for the activation was 950$^0$C for 60 min. The properties of the activated carbon in this optimum condition obtained yield of 33.72 %, bulk density of 0.5822 $g/cm^3$, ash of 3.80 %, iodine number of 954 mg/g, methylene blue number of 187 mg/g and B.E.T. surface area of 982 $m^2/g$.

#### 4.2.2.2 The effect of particles size.

The influence of particle size was determined for different four sizes of chars. They were 0.355-0.6, 0.6-1.18, 1.18-2.36 and 2.36-4.75 mm. The mass of 10g of each sample of the coconut shell char was carried out the activator followed a flow rate of air 0.005 l/min, $CO_2$ 0.5 l/min and superheated steam at the flow rate of 0.01 l/min at 950 $^0C$ while superheated steam was passed through reactor for 60 min. The results are shown in Table 4.8 and Figures 4.19-4.22.

Table 4.8 Characteristics of activated carbon from coconut shells at different sizes (air 0.005 l/min, $CO_2$ 0.5 l/min and superheated steam at the flow rate of 10 g/min, 10g, 60 min, 950 $^0C$).

| | | | On dry basis | | | |
|---|---|---|---|---|---|---|
| Size of char | %Y | BD | Ash | IA | MB | $S_{BET}$ |
| | | ($g/cm^3$) | (%) | (mg/g) | (mg/g) | ($m^2/g$) |
| 0.355-0.6 | 30.05 | 0.5721 | 3.99 | 986 | 187 | 994 |
| 0.6-1.18 | 31.31 | 0.5648 | 3.87 | 999 | 188 | 996 |
| 1.18-2.36 | 33.72 | 0.5522 | 3.80 | 992 | 187 | 983 |
| 2.36-4.75 | 35.05 | 0.5221 | 2.20 | 614 | 181 | 659 |

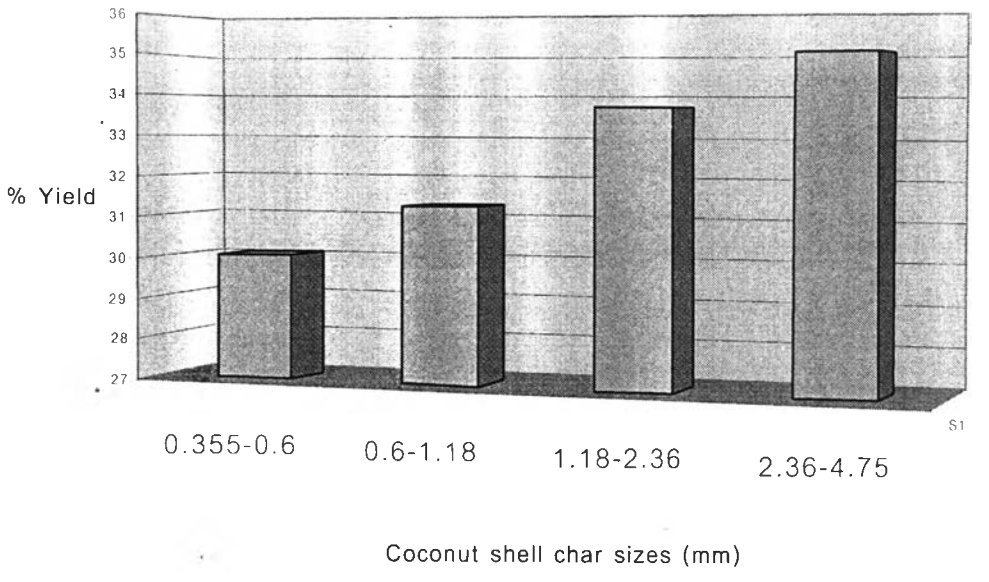


Figure 4.19 Effect of size on % yield (air 0.005 l/min, $CO_2$ 0.5 l/min and superheated steam at the flow rate of 10 g/min, 10g, 60 min, 950$^0$C).

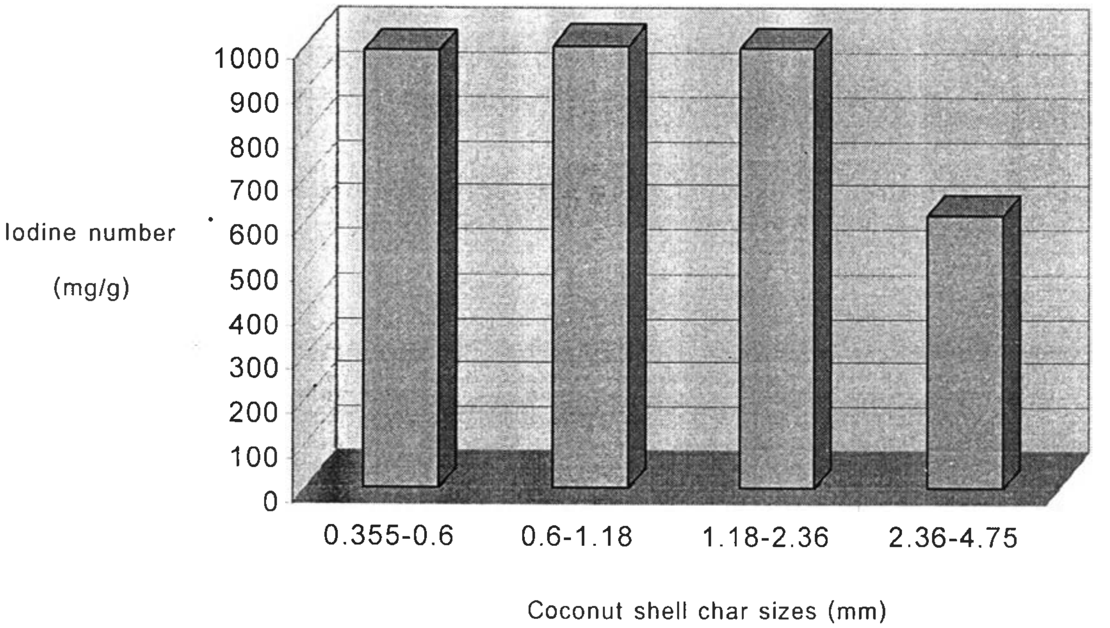


Figure 4.20 Effect of size on iodine number (air 0.005 l/min, $CO_2$ 0.5 l/min and superheated steam at the flow rate of 10 g/min, 10g, 60 min, 950$^0$C).

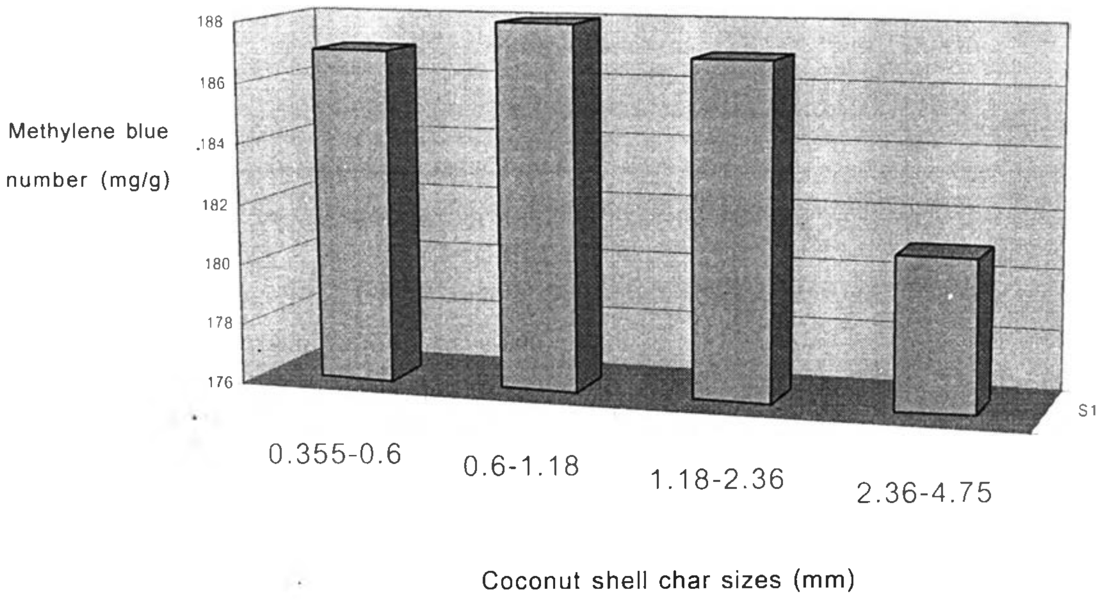


Figure 4.21 Effect of size on methylene blue number (air 0.005 l/min, $CO_2$ 0.5 l/min and superheated steam at the flow rate of 10 g/min, 10g, 60 min, 950$^0$C).

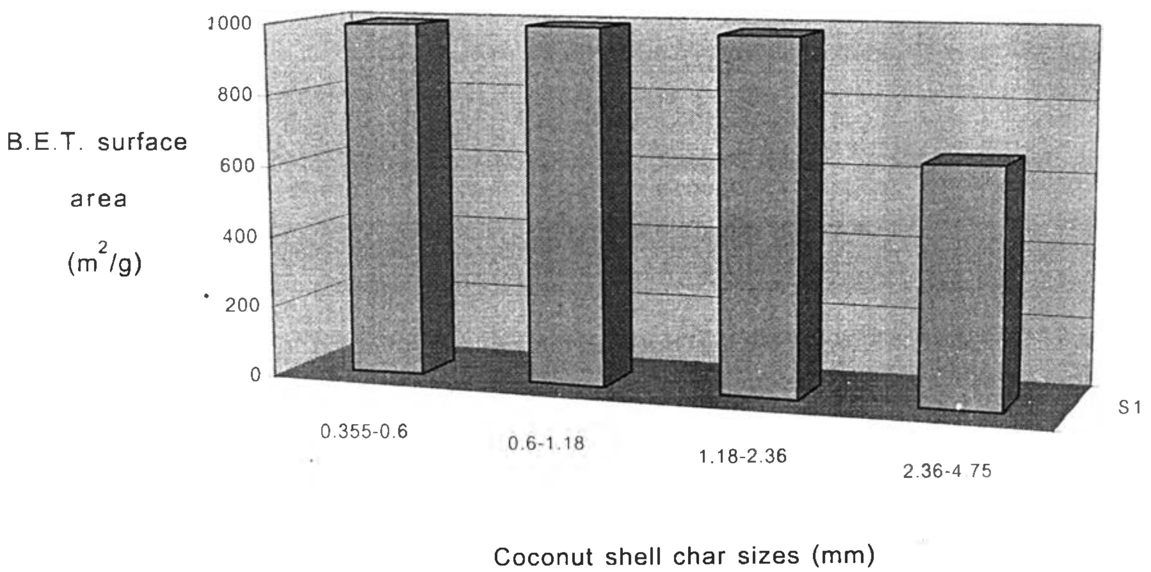


Figure 4.22 Effect of size on B.E.T. surface area (air 0.005 l/min, $CO_2$ 0.5 l/min and superheated steam at the flow rate of 10 g/min, 10g, 60 min, 950$^0$C).

**Table 4.8** and **Figures 4.19-4.22** show that the iodine number, the methylene blue number and the B.E.T. surface area of 2.36-4.75 mm size of chars are less because this size of chars is the biggest. So they pack tightly in the fixed bed; as a result, the gaps between particles are wide and the amount is high, thus the superheated steam and the carbon dioxide flow through easy to attack the surface of chars. Furthermore, the small particles burn out early due to faster reaction rates resulting in pore coalescence which increasing the iodine number. When the size of particle is 0.60-1.18 mm, the iodine number and the methylene blue number are maximum. But when the size of particle is 1.18-2.36 mm the iodine number decreases They decreases to the minimum When the size of particle is 2.36-4.75 mm. Because the bigger chars partcles pack loosely, so the gaps between big particles are larger than those small particles as a result, the superheated steam and the carbon dioxide attack chars so easily. The reaction time on the bigger particle is less because the superheated steam and the carbon dioxide flow quickly through the bed, thus they attack the inner particles so difficult. So the reacted surface of big particles is lower than that of the medium particles resulting in the decreasing of iodine number, methylene blue number and the B.E.T. surface area.

Thus the optimum size for activation is 0.6-1.18 mm because the activated carbon obtained the highest iodine number, methylene and the B.E.T. surface area. It's properties were yield of 31.31 %, bulk density of 0.5648 g/cm3, ash of 3.87% iodine number of 999 mg/g, methylene blue number 188 mg/g and B.E.T. surface area of 996 $m^2$/g.

4.2.2.3 The effect of different carbon dioxide (% weight) on composition

The composition of $CO_2$ (by weight) at 0, 4.2, 6.5, 8, 14 and pure $CO_2$ approximate 10 g of 0.60-1.18 mm of chars were treated at 950$^0$C for 60 min with fixed superheated steam flow rate at 10 g/min and air at 0.005 l/min. The results are shown in **Table 4.9** and **Figures 4.23-4.26**.

**Table 4.9** Characteristics of activated carbon from coconut shells at different flow rate of $CO_2$ (950$^0$C for 60 min, size 0.60-1.18 mm, 10 g, air 0.005 l/min and superheated steam 10 g/min)

| | | | On dry basis | | | |
|---|---|---|---|---|---|---|
| The composition of $CO_2$ | Y | BD | Ash | IA | MB | $S_{BET}$ |
| (by weight) | (%) | (g/cm$^3$) | (%) | (mg/g) | (mg/g) | (m$^2$/g) |
| 0 | 27.69 | 0.5922 | 4.06 | 1009 | 190 | 1027 |
| 0.5 | 29.52 | 0.5879 | 3.98 | 1006 | 189 | 1015 |
| 0.8 | 30.26 | 0.5743 | 3.92 | 1002 | 188 | 1008 |
| 1 | 31.31 | 0.5648 | 3.87 | 999 | 188 | 996 |
| 2 | 32.43 | 0.5105 | 2.98 | 624 | 184 | 725 |
| Pure $CO_2$ | 35.09 | 0.5006 | 2.91 | 590 | 181 | 658 |

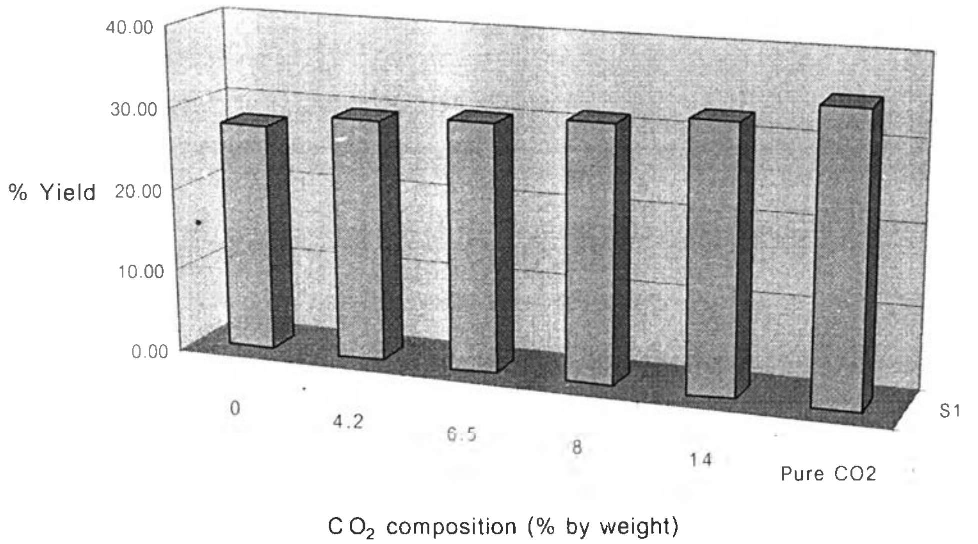


**Figure 4.23** Effect of $CO_2$ (% weight) composition on % yield ($950^0C$ for 60 min, 0.60-1.18, 10g, air 0.005 l/min and superheated steam 10 g/min).

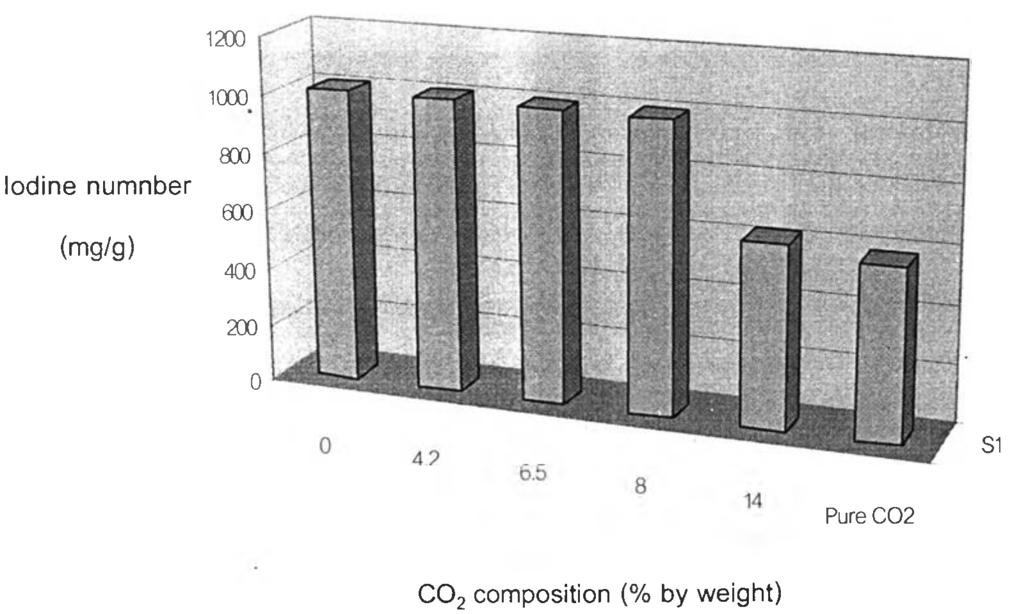


**Figure 4.24** Effect of $CO_2$ (% weight) composition on iodine number ($950^0C$ for 60 min, 0.60-1.18, 10g, air 0.005 l/min and superheated steam 10 g/min).

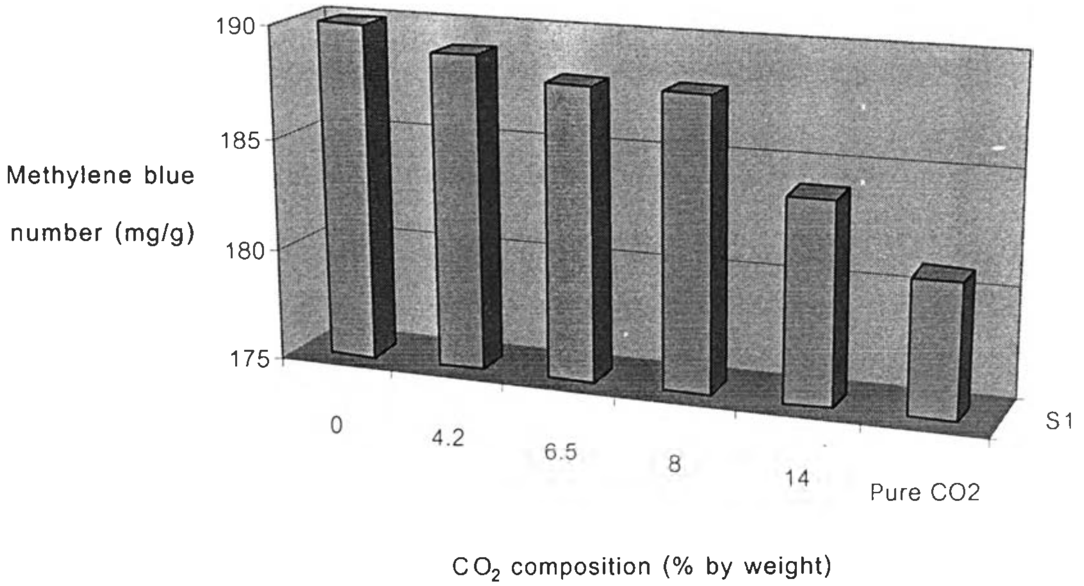


**Figure 4.25** Effect of $CO_2$ (% weight) composition on methylene blue number (950$^0$C for 60 min, 0.60-1.18, 10g, air 0.005 l/min and superheated steam 10 g/min).

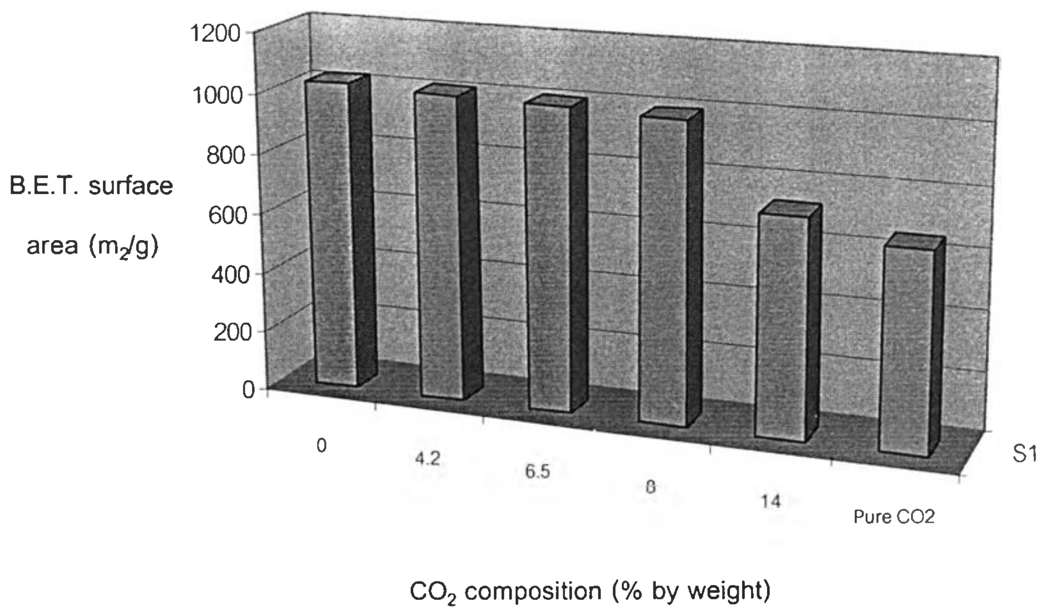


**Figure 4.26** Effect of $CO_2$ (% weight) composition on B.E.T. surface area (950$^0$C for 60 min, 0.60-1.18, 10g, air 0.005 l/min and superheated steam 10 g/min).

For the mechanism of reaction of carbon with steam is presented with reasonable confidence by the following set of equations:

$$C + H_2O \rightleftharpoons C(H_2O)$$

$$C(H_2O) \longrightarrow H_2 + C(O)$$

$$C(O) \longrightarrow CO$$

The reaction of carbon and water vapor is accompanied by the secondary reaction of carbon monoxide with water vapor (the so-called homogeneous water-gas reaction) catalyzed by the carbon surface :

$$CO + H_2O \longrightarrow CO_2 + H_2 \qquad \Delta H = -42 \text{ kJ/mol}$$

With activation with carbon dioxide, it is accompanied by mechanisms of interaction of carbon dioxide with the carbon surface are proposed :

$$C + CO_2 \longrightarrow C(O) + CO$$

$$C(O) \longrightarrow CO$$

Following this is shown about activation with carbon dioxide which in a less energetic reaction than that with steam and requires a higher temperature. The activation agent used in technical practice is flue gas to which a certain amount of steam is usually added, so that actually this is a case of combined activation.

was activated with superheated steam, the maximum iodine number, the methylene blue number and the B.E.T. surface area were obtained. Those values were decreased sharply at composition 14% to pure carbon dioxide namely 41% iodine number, 1.5% methylene blue number and 34% B.E.T. surface area. It could be explained that pure superheated steam reacted slowly with char and developed through out micropores. All the surface (SEM in Figure 4.28), while carbon dioxide reacted strongly with char and caused large pore size distribution. It was confirmed by looking at the iodine number decreased when the composition of $CO_2$ (by weight) more than 14% whereas methylene blue number stayed constant at 180 mg/g because carbon dioxide change from micropores to mesopores. This phenomenon was confirmed by this mechanism and also discovered by Sa Ahmed and Krishnaian[20].

Figures 4.27-4.30 showed the scanning electron microscope (SEM) for study morphologies of activated carbon from coconut shell at 1000 enlargement in case of coconut char activated carbon activated by pure superheated, carbon dioxide at composition 14% and pure carbon dioxide respectively. It was observed clearly that narrow pore size distribution in case, activation with pure superheated steam was shown in Figure 4.28 and decreased in case of mixed carbon dioxide and superheated steam and pure carbon dioxide which corresponded to the surface specific 1,027, 996 and 658 $m^2/g$ respectively whereas coconut char was about 112 $m^2/g$.

4.2.3 Study morphologies of activated carbon with the scanning electron microscope (SEM)

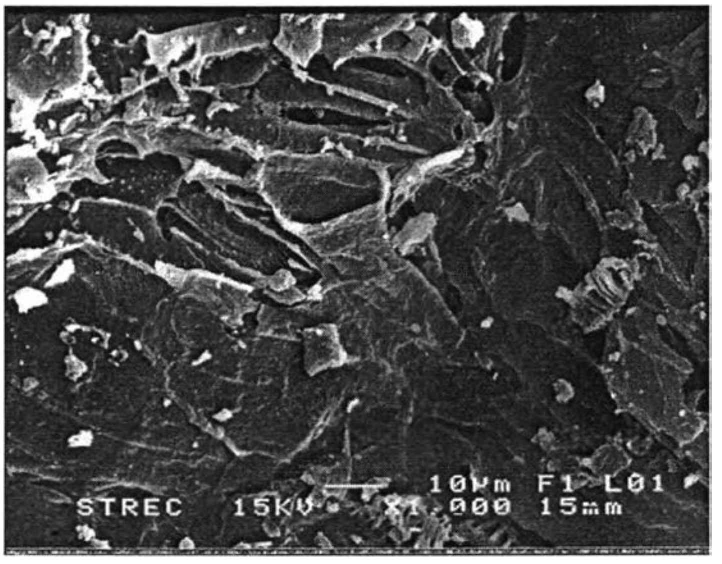


Figure 4.27 Coconut shell char sizes 1.18-2.36 before activation.

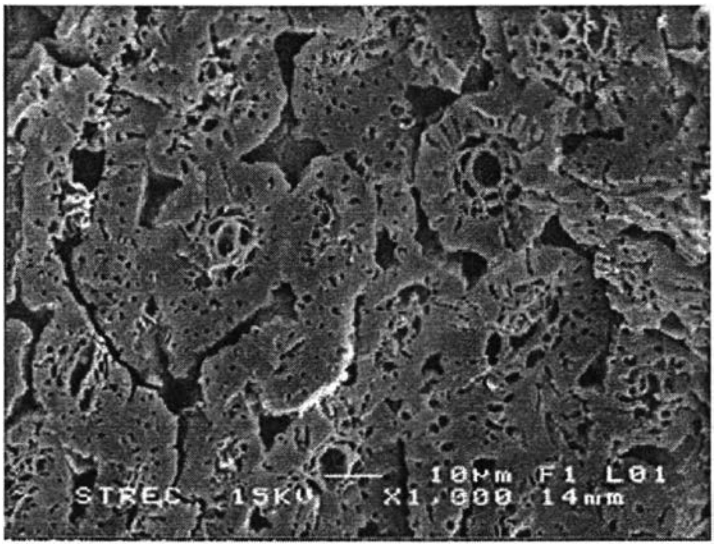


Figure 4.28 Activated carbon from coconut shell sizes 0.6-1.18, 950$^{0}$C 1 hr., flow rate of air 0.005 l/min and flow rate of pure superheated.

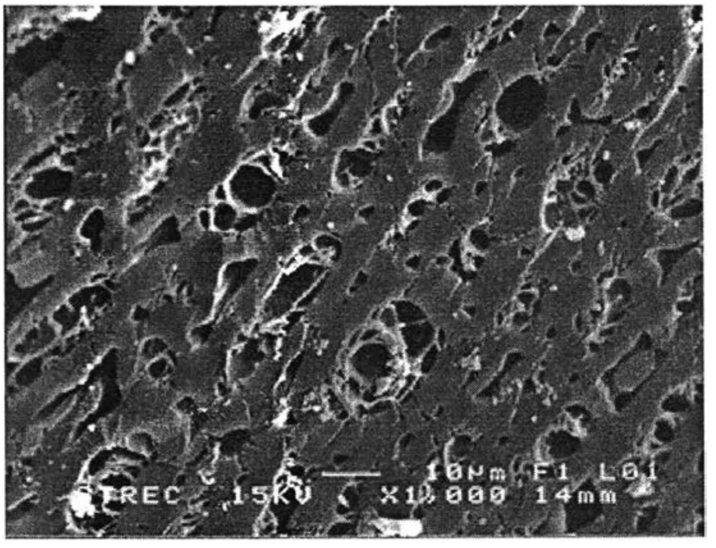

Figure 4.29 Activated carbon from coconut shell sizes 0.6-1.18, 950$^0$C 60 min, flow rate of air 0.005 l/min and carbon dioxide at composition 8%

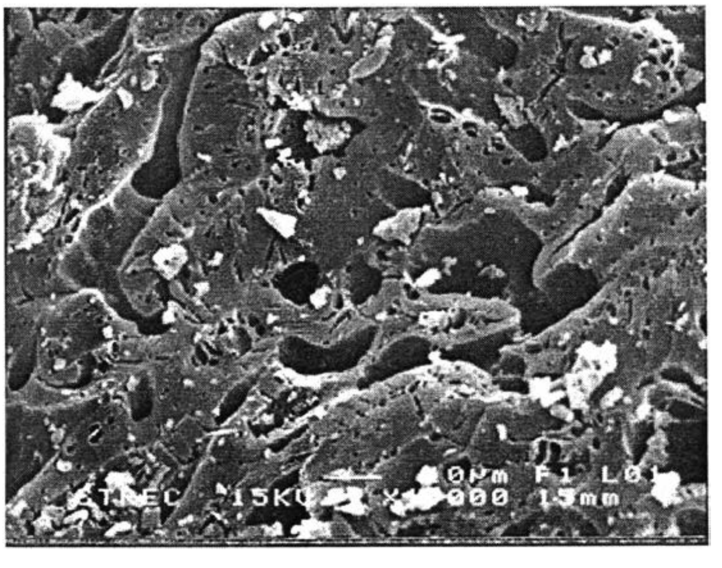

Figure 4.30 Activated carbon from coconut shells sizes 0.6-1.18, 950$^0$C 60 min, flow rate of air 0.005 l/min and pure carbon dioxide.

### 4.3 Comparison of this work with other works

The comparison of this work with that of Sai *et al.* (1997)[23] , Gergova *et al.* (1993)[17] and Torkai(1979) )[16] are presented in **Table 4.10.**

**Table 4.10** Comparison of this work with other works.

| | This work (2001) | Sai *et al* (1997) | Gergova *et al* (1993) | Torkai (1979) |
|---|---|---|---|---|
| Raw material | Coconut shells | Coconut shells | Coconut shells | Used tires |
| Carbonization | $400^0C$ for 1 hr. | $400^0C$ for 2 hr. | $700^0C$ for 2 hr. with air | $550^0C$ for 30 min with He |
| Activation | $950^0C$ for 1 hr. with air | $850^0C$ with air | | $900^0C$ with He |
| Activating agent | Steam and $CO_2$ | Steam and $CO_2$ | steam | $CO_2$ |
| %yield | 31.31 | - | - | - |
| IA (mg/g) | 999 | 950 | - | - |
| MB (mg/g) | 189 | - | - | - |
| $S_{BET}(m^2/g)$ | 996 | 1,260 | 700 | 400 |

The results of this work, comparing to other works show that the temperature of carbonization ($400^0C$ for 1 hr.) in this work is lower than others ($400^0C$-$700^0C$ for 2 hr.). Because they had different raw material, so air will help removing volatile matter and oxidizes some carbon from different raw material. The reaction of air oxidized carbon is exothermic, so the volatile matter is removed quickly.

The temperature of the activation in this work is similar to the others (700-950$^0$C). Comparing to Gergova *et al*(1993)[17], his temperature is high, so B.E.T. surface area is lower than ours due to longer activation time but in Sai' s(1997)[23], the temperature is high, so B.E.T. surface area is highest, due to their preparation of fluidized bed reactor in a 100 mm diameter and 1,250 mm length. Experimental data showed that an increase in reaction time, fluidizing velocity, particle size, and the temperature resulted in better activation. However, at higher reaction times, a decrease in iodine numbers was observed, which was due to coalescence or widening of already formed pores. Static bed heights greater than the diameter of the column gave lower iodine numbers due to poor gas-solid contact.

Torkai(1979)[16] observed the maximum surface area of 400 m$^2$/g at 900$^0$C for activated carbon.from used tires using carbon dioxide as the activating agent. From the comparisons, it can be seen that by using steam as an activating agent, the B.E.T. surface area of activated carbon is more than that by using carbon dioxide. Because carbon dioxide expanded the pores of the activated carbon, which causes less B.E.T. surface area.

In this work, the activation process is carried out with the mixture of superheated steam and carbon dioxide as activation agents similar to industrial condition. For economic reason, we could use less quantity of steam to cut cost of the activated carbon production by mixing steam with carbon dioxide which is one kind of flue gases; this kind of flue gases has high concentrations. The activated carbon produced from this method obtained B.E.T. surface area of 996 m$^2$/g and iodine number of 999 mg/g.

The above iodine number, more or less meets the standard of the commercial activated carbon of TS 900-1989[23], that is over 600 mg/g. The process condition used in this work is a good starting point to optimize the production of activated carbon from coconut shells.