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Article

Influence of Pyrolysis Temperature on Rice Husk Char Characteristics and Its Tar Adsorption Capability

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Abstract: A biomass waste, rice husk, was inspected by thermoanalytical investigation to evaluate its capability as an adsorbent medium for tar removal. The pyrolysis process has been applied to the rice husk material at different temperatures 600, 800 and 1000 ℃ with 20 ℃/min heating rate, to investigate two topics: (1) influence of temperature on characterization of rice husk char and; (2) adsorption capability of rice husk char for tar removal. The results showed that subsequent to high temperature pyrolysis, rice husk char became a highly porous material, which was suitable as tar removal adsorbent with the ability to remove tar effectively. In addition, char characteristics and tar removal ability were significantly influenced by the pyrolysis temperature.

Keywords: rice husk char; tar removal; pyrolysis; adsorption

1. Introduction

In agricultural countries, lots of agriculture residues or biomass wastes, such as rice husk and woods, are produced every year. The world annual production of rice is more than 540 million metric tons [1]. These biomass wastes are one of the main assets for renewable energy. Consequently, there are numerous prominent technologies to transform biomass into energy. The worldwide well renowned technologies are the use of thermal processes such as combustion, gasification or pyrolysis. Pyrolysis is a decomposition process of biomass at high temperature in the absence of oxygen. In the end, after

transient high thermalization, producer gas, a carbon-rich residue called biomass char and tar will be produced. The proportion of these products depends on the operating conditions [2]. The producer gas has the ability to be used for various functions such as chemical production, heat resource, power generation, *etc.* Biomass char can also be used as a potential resource in diverse industries, depending on their characteristics, while the excess tar must be eliminated from the producer gas for downstream applications so the problem of tar blockage in pipes and engines can be prevented.

The biomass char is a solid carbonaceous residue with a high content of fixed carbon, which can be used directly as a fuel, fertilizer or precursor for activated carbon production [3]. One of the options for char utilization, which has been widely reported, is for adsorption purpose. Thermal processes will develop pores on the biomass char surface, which make char capable of acting as an adsorption medium material. Throughout the process, volatile matters are generated, while the physical nature of char is extensively altered. The raw biomass properties really influence the chemistry of char formation, with the pyrolysis temperature and the heating rate being the main operation parameters that have a strong influence on char's structure.

There are number of studies dealing with the relationship between the pyrolytic conditions and the char structure [1,3–6]. The operational parameters such as the heating rate, the reactor temperature and the residence time play the most significant roles in the operational control for the pyrolysis process. These factors influence both the product distribution and the product characteristics.

Various biomasses were pyrolyzed in a packed bed reactor at 500 ℃ with a solid residence time of 1 h [4]. The pore evolution and development were studied by BET and SEM characterization. The chars were seen to have surface areas as high as 600 m^2/g and were recommended for cheaper carbon adsorbent production. Rice husk char was studied in a fixed bed pyrolyzer at 200–650 ℃ at an interval of 50 ℃ with 10 ℃/min heating rate, aiming at determining the characteristics of the charcoal formation and its applicability as a solid fuel [1]. The relationship between gas composition/char properties and the pyrolysis temperature of rice husk was also analyzed [5]. The results showed that the char yield decreased in the temperature range from 600 to 1000 ℃. The maximum porosity appeared at 900 ℃. Another study also showed that the porosity increased gradually during a rapid pyrolysis reaction process [6]. Likewise, when different biomass samples (almond shell, walnut shell, almond tree prunings and olive pits) were subjected to the thermoanalytical investigation conditions to evaluate their thermal behaviour at the pyrolysis temperature of 600 ℃ with a residence time of 1 h [3] it was reported that at 600 ℃, most of the volatile matters were removed. The pyrolysis chars were further subjected to a steam gasification for activated carbon production. The rate of the thermal decomposition of the parent material played a determinant role on the porosity of the activated carbon produced and the pore sizes tended to be broader (greater volume of meso and macropores) with slow pyrolysis in the first stage. Activated carbons produced from almond tree prunings were recommended as the best solution for adsorption purposes. Other trees were also applicable for the gas adsorption application. The retention time was found to affect the phenol adsorption capacity and the modification-free carbonaceous materials (bio-chars) and bio-chars were suggested to be used as adsorbent rather than fuel for their greater economic prospect [7].

Seeing that the main operating parameters, which affect the conversion of biomass into char phase, are the pyrolysis temperature and the heating rate, this research investigates the influence of the

pyrolysis temperature on the characteristics of rice husk char at 600, 800 and 1000 ℃ with a 20 ℃/min heating rate and 1 h holding time at the target temperature.

Furthermore, the utilization of biomass char for tar removal adsorption purposes has a big advantage, considering the universal need for a cheap source of tar adsorbents for various biomass industries. In our previous paper [8], gasified rice husk char was proved to be capable of tar removal. Moreover, many researchers have reported their works on the tar removal performance of biomass char in gasification processes, which was also reviewed in the previous papers [8–12], confirming that rice husk char is capable of tar removal. In this research, the tar removal ability of each char produced from different pyrolysis temperature was investigated. Therefore, the aim of this work can be summarized in two topics: (1) influence of the pyrolysis temperature on characterization of rice husk char at 600, 800 and 1000 ℃ and; (2) adsorption capability for tar removal of rice husk char produced under different pyrolysis temperatures of 600, 800 and 1000 ℃.

2. Methodology

2.1. Rice Husk Material

Rice husk feedstock obtained from Thailand was prepared by drying in an oven at 105 ℃ for 8 h to obtain perfect moisture elimination before packing in the pyrolyzer. The characterization of rice husk feedstock is shown in Table 1.

Table 1. Characterization of rice husk feed stock.

Ultimate analysis (wt% dry basis)			
C	H	N	Cl
37.9	3.9	0.4	0.1
Proximate analysis (wt% dry basis)			
Volatile matter content	Fixed carbon		Ash
69.7	9.8		20.6

2.2. Experimental Setup

2.2.1. Rice Husk Char Preparation

The pyrolysis process was performed in a batch-type lab-scale equipment facility with feeding of nitrogen at 1.5 L/min as shown in Figure 1. The reactor was a made-to-order quartz glass unit (heat resistant to 1200 ℃) and was covered with a heater furnace. The sample was installed in the reactor before heating up at the heating rate of 20 ℃/min to reach the target temperatures of 600, 800 and 1000 ℃, then kept at the target temperature for 1 h holding time. Figure 2 shows the pyrolysis time intervals performed in this experiment and Table 2 lists the experimental conditions. The rice husk char was screened at 5–10 mm and dried in an oven at 105 ℃ for 8 h before being used as an adsorbent in the tar removal experiments.

Figure 1. Char preparation by pyrolysis at 600, 800 and 1000 ℃.

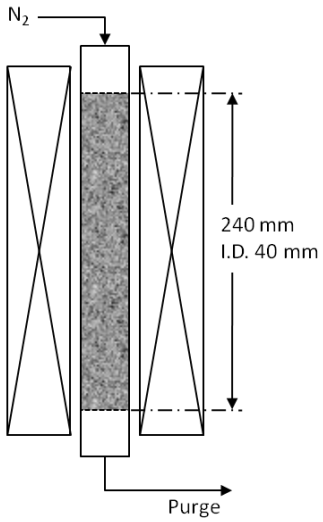


Figure 2. Char pyrolysis time interval at 600, 800 and 1000 ℃.

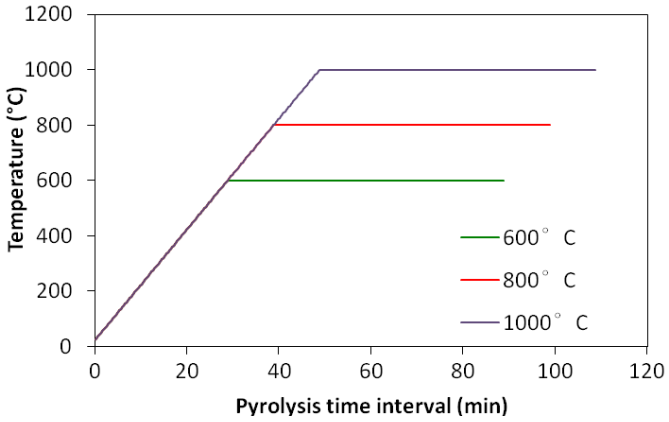


Table 2. Experimental conditions for char preparation.

Parameter	Experimental conditions
Material	Rice husk
Material size	Original size (8–11 mm)
Pyrolysis temperature	600, 800 and 1000 ℃
Heating rate	20 ℃/min
Holding time	1 h (after reaching the target temperature)
Carrier gas	Nitrogen
Carrier gas flow rate	1.5 L/min

2.2.2. Tar Removal Investigation

Rice husk chars pyrolyzed at three different temperatures were studied for their tar removal ability by an adsorption process. Adsorption studies were performed using a fixed-bed type adsorber, which was installed downstream of the reformer. The experimental setup is shown in Figure 3 and the setup conditions are shown in Table 3.

Rice husk feedstock (screened at 0.125–0.5 mm) was fed into the screw feeder at the feed rate of 0.6 g/min with a nitrogen carrier gas flow of 1.5 L/min. The feedstock was introduced into the pyrolyzer (SUS310 stainless steel; inner diameter 30 mm, height 280 mm), which combined with a reformer (SUS310 stainless steel; inner diameter 25 mm, height 1,300 mm). The pyrolyzer and the reformer were thoroughly controlled by an electric heater at 800 ℃.

The pyrolysis gas released at the bottom of the reformer was introduced to the adsorption bed through a high temperature resistant tube connector without any additional heating. The adsorption bed was kept at ambient temperature (25–28 ℃) and was filled with biomass char, which was prepared under each different temperature (600, 800 and 1000 ℃), with 100 mm bed height. At the exit of the adsorption bed, a tar measurement line was installed, consisting of ten impingers, each was filled with 100 mL of isopropanol and kept in cold baths as shown in Figure 3. After passing through the adsorption bed, the residual tar in the pyrolysis gas was collected by both condensation and absorption in the isopropanol solvent.

The pyrolysis gas was sampled at the flow rate of approximately 0.8 L/min for 48 min. After sampling, all of isopropanol sampling solvent in each impinger bottle was mixed together, filtrated and dried by a standard rotary evaporator in a water bath kept at 40 ℃. Then, the flask was weighed accurately and the amount of residue was determined, which was heavy tar. This measured heavy tar was defined as gravimetric tar. This tar measurement method has been well described in the previous work [8]. The ash product remained at the bottom of the pyrolyzer throughout the sampling time of 48 min.

Figure 3. Experimental setup for tar removal investigation.

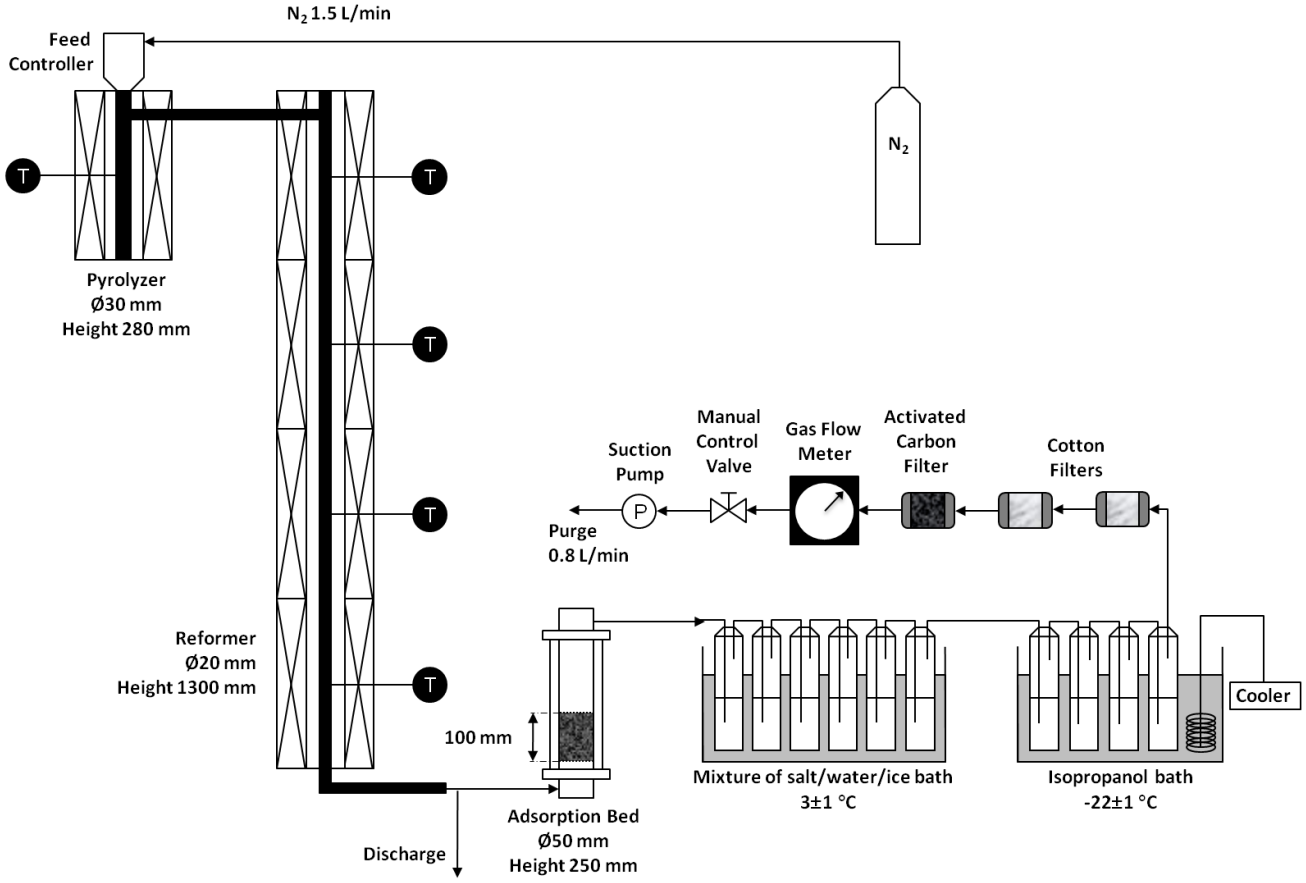


Table 3. Tar removal experimental conditions.

Parameter	Experimental conditions
Feed material	Rice husk
Feedstock size	0.125–0.50 mm
Feed rate	0.6 g/min
Pyrolyzer temperature	800 ℃
Reformer temperature	800 ℃
Carrier gas	N_2
Carrier gas flow rate	1.5 L/min
Tar adsorbent	Rice husk char pyrolyzed at 600, 800 and 1000 ℃
Char bed height	100 mm

2.3. Characterization of Char

2.3.1. Thermal Gravimetric Analysis (TGA)

Char samples were subjected to thermal gravimetric analysis using a Shimadzu DTG-50 (Shimadzu Corp., Nakagyo-ku, Kyoto, Japan), simultaneous DTA-TG instrument. The analysis was divided into two stages whose conditions are shown in Table 4.

Table 4. Thermal gravimetric analysis conditions.

Parameter	Stage 1	Stage 2
Heating rate	20 ℃/min	50 ℃/min
Target temperature	105 ℃	900 ℃
Keeping time at target temperature	5 min	30 min
Carrier gas	Nitrogen	Nitrogen and Air
Carrier gas flow rate	150 mL/min	150 mL/min

Remark: Nitrogen were used until reaching the target temperature of 900 ℃ and nitrogen was changed to air after passing 7 min at this target temperature.

2.3.2. Surface Characterization

The analysis of the specific surface area was carried out using a BelsorpII high precision surface area and pore size analyzer instrument (BEL Japan, Inc., Osaka, Japan), using the Advanced Free Space Measurement (AFSM) measurement principle. Prior to the measurements, the samples were pretreated in order to remove the moisture by heating up to 150 ℃ and holding for 2 h under vacuum. During the measurements, the pressure was raised under constant temperature and the physical adsorption of nitrogen gas in the sample was performed in order to measure the adsorption isotherm of nitrogen in the sample. The specific surface area values of the samples were calculated based on the theory of BET from the data of nitrogen adsorption.

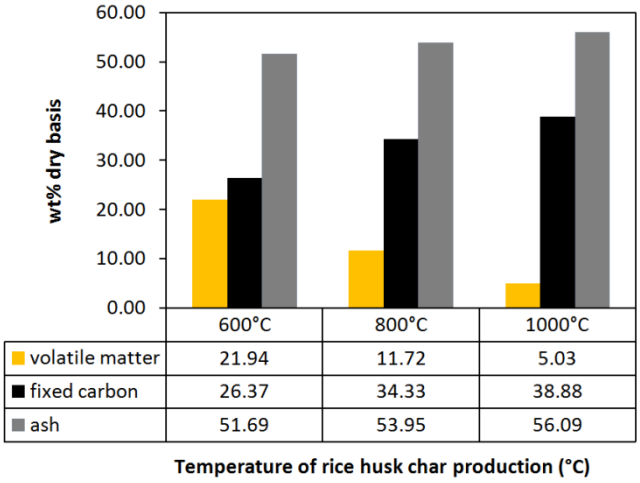
3. Results and Discussion

3.1. Thermal Effects on Characterizations of Rice Husk Char

In biomass pyrolysis processes, the target product is normally biomass char or biochar. The major factors that affect the characteristics of produced char are the pyrolysis temperature, the residence time at the target temperature and the heating rate [13]. Char is created mostly from the thermal decomposition of lignin and some extractive part of biomass, while the volatile matter is transformed into the gas phase and minerals in the biomass are left as ashes [14]. Hence, at the same heating rate and the residence time, the pyrolysis temperature is the most influencial factor for the product distribution.

In Figure 4, characterization of rice husk char produced at each pyrolysis temperature is presented. It can be seen clearly that, with higher pyrolysis temperature, more volatile matters have been forcibly expelled out of the char particles and less volatile matters are left in the particle form with 21.94, 11.72 and 5.03 wt% at 600, 800 and 1000 ℃ pyrolysis temperature, respectively. At the same time, higher pyrolysis temperature resulted in higher fixed carbon content. The fixed carbon content of the char is the carbon found after volatile matters are evaporated from the biomass char. Fixed carbon is determined by removing the mass of volatiles. Therefore, at higher pyrolysis temperature, more volatiles have been removed, which resulted in less volatile matters and more fixed carbon in the char particle with 26.37, 34.33 and 38.88 wt% at 600, 800 and 1000 ℃ pyrolysis temperature, respectively.

Figure 4. Characterization of rice husk char produced at each temperature (wt% dry basis).



3.2. Thermal Effect on Specific Surface Area of Rice Husk Char

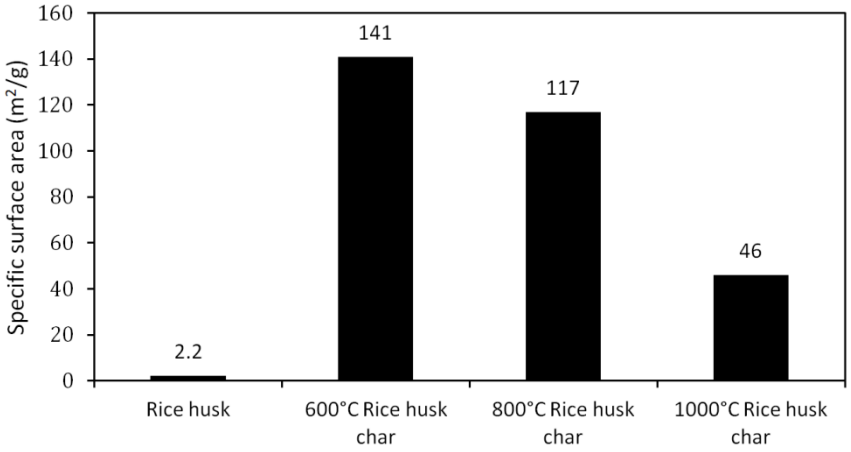
As stated above, the pyrolysis process was carried out during 1 h holding time with 20 ℃/min heating rate. These constant conditions were maintained in order to compare the results obtained from pyrolysis temperature variation at 600, 800 and 1000 ℃. Figure 5 presents BET specific surface area values of rice husk and rice husk chars. It is apparent that, after high thermal decomposition by the pyrolysis process, rice husk char has developed pores with dramatically increased the specific surface area. The specific surface area values varied significantly at different pyrolysis temperatures. Rice husk material has very low specific surface area of 2.2 m^2/g while rice husk char pyrolyzed at 600 ℃

showed the highest specific surface area of 141 m^2/g. It decreased to 117 and 46 m^2/g with the increase in the pyrolysis temperature to 800 ℃ and 1000 ℃, respectively.

In general, surface area of biomass char increases with the pyrolysis process, due to the generation of porosity. Hence, since this experiment involved slow pyrolysis, more micropores were likely to be created compared to macropores [5]. Nevertheless, the consequence of the carbonization step at a high temperature is as follows: too high a temperature will damage the development of porous structures in the char and the walls of the pores become so thin that they collapse and this causes a reduction in the available surface area [15]. Therefore, the chars were found to have decresed specific surface area when the temperature was increased, as reported by other researchers [16–21]. Fu *et al.* [16] and Pastor-Villages [22] also stated that above 500 ℃, high temperature may cause the occurrence of the structural shrinkage. Structural ordering and micropore coalescene during pyrolysis are responsible for the decrease in the surface area observed at too high temperatures, indicating the thermal annealing and thermal deactivation of the chars. Too high temperature led to deformation of particles resulting in smooth surfaces and large cavities [16,17,23,24].

The ash content is also examined. When the temperature increased, the ash content increased, which may sinter and block pores. In addition, ash does not contribute significantly to surface area and its presence can reduce the surface area [25]. Moreover, from Figure 2, it can be seen that at 1000 ℃, the operating time is longer than that at 800 ℃ and 600 ℃, which caused the char to decrease in the surface area which could be explained by the fact that continuing development of pyrolysis is detrimental to the pore structure of the char [6].

Figure 5. BET specific surface area values of rice husk and rice husk chars.



3.3. Tar Removal Ability Investigation

The main property of rice husk char that is focused on in this research is its adsorption capacity for tar removal. Figure 6 shows the concentration of heavy tar in the pyrolysis gas before (described as—no cleaning) and after the adsorption bed. The adsorption bed was packed with three types of adsorbent, which were rice husk chars produced under the pyrolysis temperatures of 600, 800 and 1000 ℃. It is clearly seen that the heavy tar concentration in the pyrolysis gas significantly decreased after adsorbtion by rice husk char. The transfer lines were high temperature resistant tubes without any additional heaters. The tar condensation inside the transfer line was checked by the weight change of

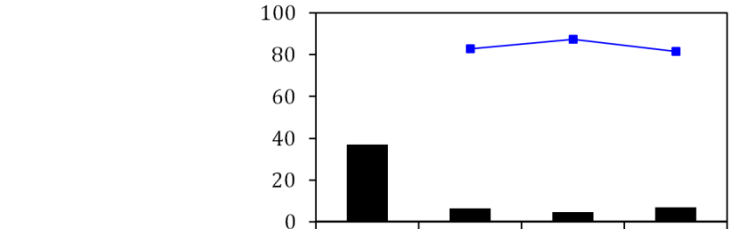
the tubes before and after the experimental operation. It was found that the weight difference was too small and was thus neglected.

In the present results, the 800 ℃ char showed the optimum tar removal performance whereby tar concentration could be reduced from 36.9 to 4.6 g/m^3, which corresponds to 87.5% tar removal, whereas 600 and 1000 ℃ char presented the performances of 82.9% and 81.6% tar removal, respectively. The adsorption capacity of rice husk char is related to the specific surface area and the fixed carbon content of the char.

More specific surface area means more channels for the tar molecules to be adsorbed physically by molecular attraction. At the same time, the fixed carbon content also plays an important role. Carbon surface will hold tar molecules by the weak force known as van der Waals force. Therefore, more fixed carbon content means large carbon surface for tar to be adsorbed. The 600 ℃ char had less fixed carbon content, while 1000 ℃ char had very low specific surface area, resulting in the 800 ℃ char being the most favorable char for tar adsorption in this research.

In Figure 6, the percentage of the tar removal in the char bed is noticeable. The main mechanism for this tar removal was adsorption. However it was found that some part of the heavy tar condensed at the bottom of the char bed. Therefore, tar removal by rice husk char was not only by adsorption of tar molecule in the pores of the char material, but also in the form of condensation of heavy tar molecules in the hot gas when passing through the adsorption bed at ambient temperature. The char at the bottom of the adsorption bed, at the gas inlet, was found to be a bit soaked by the condensed heavy tar. Nevertheless, most of the char was still dry after tar adsorption, which indicates that the main tar removal mechanism should be adsorption. This observation suggests that char adsorbent is more appropriate for light tar in the dry gas, to prolong the lifetime of the char adsorbent. Morover, this phenomenon assured that a heavy tar removal unit such as an oil scrubber must be installed before the char adsorption bed to remove heavy tars and moisture from the pyrolysis gas.

Figure 6. Concentration of heavy tar in the pyrolysis gas before (described as—no cleaning) and after the adsorption bed of each type of char.



	No cleaning	600°C Char	800°C Char	1000°C Char
Tar concentration (g/m^3)	36.9	6.3	4.6	6.8
%Tar removal		82.9	87.5	81.6

3.4. Analysis of Rice Husk Char after Tar Adsorption

Table 5 illustrates the proximate analysis and the specific surface area of each rice husk char adsorbent before and after tar adsorption. It can be noticeably seen that after tar adsorption, rice husk chars have more volatile matters and less fixed carbon, due to tar, which is volatile matter, has been adsorbed in the pores and on the surface of the chars. The 800 ℃ char showed the most significant

volatile matter increase after tar adsorption, from 11.7% to 30.0%, indicating the greatest capacity for tar adsorption among the samples tested. Moreover, the specific surface area of 800 ℃ char has significately decreased from 117 to 15 m^2/g after tar adsorption due to the adsorption of the tar molecules on the pore surfaces and the decrease of the surface area of the char particles.

Table 5. Proximate analysis of each rice husk char adsorbent before and after tar adsorption.

Proximate analysis (wt% dry basis)	600 ℃ Char		800 ℃ Char		1000 ℃ Char	
	Before	After	Before	After	Before	After
Volatile matter	21.9	36.1	11.7	30.0	5.0	18.7
Fixed carbon	26.4	12.3	34.3	17.0	38.9	27.7
Ash	51.7	51.6	53.9	53	56.1	53.6

4. Conclusions

Rice husks were inspected by thermoanalytical investigation to evaluate their capability as adsorbent media for tar removal. The pyrolysis process has been applied to the rice husk material under different temperatures; 600, 800 and 1000 ℃ at 20 ℃/min heating rate to investigate the influence of temperature. Experimental results have shown that rice husk char could be prepared for the purpose of tar removal. The pyrolysis temperature predominantly affected the properties of the char produced. Two properties of char that influence tar adsorption capacity were fixed carbon content and the specific surface area. The 600 ℃ char had less fixed carbon content, while 1000 ℃ char had very low specific surface area, resulting in 800 ℃ char being the most favorable char for tar adsorption in this research.

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