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To cite this article: Mohd Sukhairi Mat Rasat et al 2020 IOP Conf. Ser.: Earth Environ. Sci. 549 012067

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Torrefaction's Optimization of Multiple Responses Analysis on Torrefied Biochar from Oil Palm Empty Fruit Bunch

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Abstract. In this study, biomass resource is chosen as a renewable energy source based on the abundance of oil palm waste generated monthly. The aim of this study is to find an optimal value of torrefection process on the torrefied biochar from oil palm empty fruit bunches (OPEFB) in order to produce biomass energy source by using the Box-Behnken design of response surface methodology (RSM). The OPEFB has been torrefied based on the three independent variables which are particle size (250, 500 and 750μ m), holding temperature (200, 250 and 300°C) and residence time (30, 60 and 90 minutes). Torrefied biochar are being optimized in regards of six dependent variables which are mass yield, moisture content, volatile matter, ash content, fixed carbon and calorific value. The optimization process from the RSM shows that the most optimal value for OPEFB torrefied biochar is at 750μ m (particle size), 274°C (holding temperature) and 90 minutes (residence time) of torrefaction process in order to produce a high energy content of biomass.

1. Introduction

Energy demand and consumption are growing due to increasing population numbers. Renewable energy, which is the alternative energy, becomes really necessary to be used to offset the use of nonrenewable fossil fuel energy, and the issues are getting worse due to climate change [1,2]. More carbon dioxide is emitted to the atmosphere because of the industrial revolution and the combustion of fossil fuel. Climate change increase the global temperature, hence causing global warming and the world getting warmer. Human consumptions of energy and electricity keep increasing and thus more fossil fuel is needed as the source of energy. In Malaysia, the analysis shows that the energy production will be increasing almost seven times more than 2005 till 2030 and it relies on the fossil

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fuel which is the non-renewable energy as a sources of energy generation and only small portion of energy production that is contributed by renewable energy source [3].

Biomass is an alternative energy generated from the biodegradable organic materials that undergo decomposition of plants and animals [4,5]. Energy of biomass is derived from the interaction between plants and the sunlight, also known as photosynthesis. Biomass is an energy that is sustainably produced to reduce the consumption of fossil fuel [6,7]. This energy is able to derive about 44% from biomass that mostly based on wood and wood-derived materials [8,9]. Biomass production in Malaysia is about 168 million tonnes such as oil palm waste, municipal waste and rice husks every single year [10-12]. The abundance of biomass that is derived from the oil palm waste is able to cover the issue of generating a sustainable energy sources [13]. They act as a fuel by generating steam in the process of generating power by the movement of turbines [14]. Hence, the alternative energy will contribute in reducing the issue of global warming which is one of the solutions towards green energy production.

Malaysia is the second largest producer of palm oil after Indonesia. Thus, during the harvesting and extraction of oil palm fresh fruit bunch (FFB), oil palm mills produced numerous amount of oil palm waste. To find an alternative way to replace fossil fuel, oil palm waste biomass is seen as an alternative renewable energy that can generate energy from biomass. The oil palm waste such as oil palm empty fruit bunch (OPEFB) is able to replicate the ability of fossil fuel in energy generating by undergo the process of torrefaction, at the same time able to reduce the issues of climate change and global warming.

Currently, Malaysian government is focused on replacing 5.5% of the electricity source with renewable energy as the country moves towards becoming a developed country [14]. Based on the 2010 Malaysian Renewable Energy Act that was submitted to Parliament for approval in accordance with the Sustainable Energy Development Authority (SEDA) Act, the study focused on to promote the role of biomass as part of the Malaysia energy mix strategy.

Thus, in this study, torrefaction was proposed as an alternative method that suitable in improving the biomass properties for the biomass energy generation. Hence, the optimization of torrefied OPEFB as biochar energy is needed through optimizing the appropriate parameters which are particle sizes, holding temperature and residence time. To be precise, this study focus on to generate the experimental design through analyse torrefied OPEFB biochar using response surface methodology (RSM), Box-Behnken design according to those three parameters, and to identify the optimized torrefied OPEFB biochar regards to six responses of mass yield, moisture content, volatile matter, ash content, fixed carbon and calorific value.

2. Material and Methods

2.1 Sample Preparation

Raw OPEFB has been washed using tap water for oils and impurities removal that present on the samples. The clean samples have been dried under direct sunlight to dry the samples for a day. Then, the samples have been dried in the oven about 1 hour at $80\pm5^{\circ}$ C to reduce the moisture content in the samples. Then, the dried OPEFB has been grinded to get smaller particle size.

The samples have been screened into three different sizes which are 250, 500 and 750μ m in order to obtain desired particle size and to prevent the contamination.

2.2 Torrefaction Process

The OPEFB fibre has been undergone torrefaction process which is the thermal pre-treatment method and being analyzed. The torrefaction process has been set at three holding temperature which are 200, 250 and 300°C without the presence of oxygen under slow heating rate. The samples have been torrefied at three different residence time which are 30, 60 and 90 minutes of torrefaction process. The OPEFB are called torrefied OPEFB biochar after the torrefaction process is completed.

2.3 Response Data

In order to get optimize data of torrefied samples, a statistical technique which is RSM by using the Box-Behnken design has been used in this study. This statistical technique has been applied to design the experiment run, and the response data can be used to optimize the process as well. The six responses of dependent variables (response data) are mass yield, moisture content, volatile matter, ash content, fixed carbon and calorific value, that have been obtained in the experiment has been keyed in into the Minitab software. The measurement of each response has been obtain via different analysis.

2.4 Multi Response Optimization

Using the RSM, the Box-Behnken design is generated based on the three parameters; particle size, holding temperature and residence time for those six responses of dependent variables, sample optimization have been performed. The response of data recorded has been analyzed via regression. Data optimization has been run using Minitab 15.

This method describes the multiple response surface aspect on RSM technique based on several responses that have been analyzed. All the six responses have been set based on desirable goal. Torrefied OPEFB biochar must have a high mass yield, calorific value, fixed carbon and possess low moisture content, volatile matter and ash content in order to produce high-quality biomass energy sources. Thus, maximum and minimum goals has been chosen based on desirable target.

3. Results and Discussion

The design of experiment run that has been suggested by Box-Behnken as shown in Table 1. The three parameters are denoted as PS (particle size), HT (holding temperature) and RT (residence time). The six experimental responses are denoted as MY (mass yield), MC (moisture content), VM (volatile matter), AC (ash content), FC (fixed carbon) and CV (calorific value).

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Run	PS	HT	RT	MY	MC	VM	AC	FC	CV
	(µm)	(°C)	(min)	(%)	(%)	(%)	(%)	(%)	(MJ/kg)
1	500	250	60	80.29	9.59	77.12	1.79	11.5	17.70
2	750	300	60	36.72	8.46	51.53	3.98	36.03	18.78
3	750	250	30	90.40	10.76	79.81	1.48	7.95	17.58
4	500	250	60	88.52	9.65	76.89	1.82	11.64	17.71
5	250	200	60	97.64	10.86	81.91	1.36	5.87	17.77
6	750	200	60	97.26	9.29	81.75	1.44	7.52	17.18
7	250	250	30	88.42	7.82	81.80	2.02	8.36	17.79
8	500	300	90	31.95	7.83	47.77	4.75	39.65	19.12
9	250	250	90	72.26	9.23	73.70	2.52	14.55	18.17
10	500	250	60	80.09	9.71	77.04	1.85	11.40	17.74
11	500	250	60	81.08	9.62	76.98	1.80	11.60	17.70
12	250	300	60	35.05	11.05	47.34	5.25	36.36	18.30
13	750	250	90	73.31	7.83	73.70	1.68	16.79	18.04
14	500	200	30	97.86	11.43	83.24	1.14	4.19	17.22
15	500	200	90	96.84	9.98	80.40	1.65	7.97	17.47
16	500	300	30	50.44	9.10	60.62	3.02	27.26	18.38
17	500	250	60	80.86	9.69	76.73	1.79	11.79	17.33

Table 1. The experiment run

3.1 Mass Yield

The mass yield of the OPEFB shows decreasing value from the initial weight compared to the final weight after being torrefied. The initial weights are approximately 5g for all run. The final weight of OPEFB after torrefaction process shows that the mass has loss up to 68%. Mass yield represent the

percent ratio of the actual ratio of OPEFB mass [15]. The higher the mass yield, the higher the energy sources in the OPEFB samples. Lowest mass yield is 31.95%, while the highest mass yield is 97.86%.

Due to the high concentration of volatile composition in OPEFB, high mass reduction occurs on the basis of mass yield obtained. High mass loss also occurs during drying periods, where high levels of moisture are decreased before being put in the torrefaction reactor. In order to obtain most desirable energy source, the mass yield of the torrefied

OPEFB biochar has been targeted to be maximum [15]. However, it is a desire to obtain reaction conditions within the ranges that could produce biochar with maximum mass yield [16].

3.2 Proximate Analysis

This study is focused on minimizing the moisture content and produce torrefied OPEFB biochar with higher calorific value. The elimination of water is an integral part of the process [17]. Coal water content also has a significant effect on the coal's utilization. Reducing the moisture content of the torrefied OPEFB biochar add to the rise in the energy content calorific value.

Volatile matter is referring to the components of the biochar after being torrefied without the presence of oxygen excluding the moisture content. It measure the gases that has been formed during torrefection process. The higher temperature correspond to a more intense reaction, resulting in more volatile generation that escape mainly in the form of gas. Thus, the volatile matter has been increased as the holding temperature and residence time of torrefaction increased. The analysis is aimed at minimizing volatile matter and previous research showed that volatile matter increased but could be minimized by optimizing torrefaction factors [18].

Ash content is a part of the torrefied biomass that has been made of the incombustible mineral material. A large amount of ash content is not favour due to the potential released of ashes during the process of combustion [19]. The study shows that ash content in the measured biomass decreased as the holding temperature and torrefaction period rise. The increase in the composition of ash is more proportional to the shift of the original components of biomass. As the biomass loses some of the moisture and volatiles during the process, the ash content is more of a relative increase with respect to the original components.

Fixed carbon is a residue portion of the char after combustion processes, excluding the volatile, humidity and ash content [20]. In the meantime, there is no change in the residence time in the fixed carbon as the time increased. It can therefore be inferred that holding temperature has led to changes in fixed carbon while residence time has not been relevant to changes in fixed carbon value.

3.3 Calorific Value

The calorific value is the amount of heat generated during the combustion process. It can also measure the torrefied biochar's heating capacity by estimating the amount of biochar needed to produce a certain amount of heat. A higher calorific value in char indicates the higher energy content in the char. Recent studies showed that the impact of torrefaction on the OPEFB gradually enhanced the calorific value of the biomass [21].

3.4 Multi Responses Analysis

The optimization plot of mass yield, moisture content, volatile matter, ash content, fixed carbon and calorific value are based on Figure 1. Within the experimental range, three-dimensional surfaces are plotted to show the effect of each factor along with interaction effects between different factors to each responses. The vertical red line can be moved forward or backward to increase or decrease the factors values. By moving the red lines, there are changes happened in each six responses. Thus, the clear relationship can be observed straight away from this plot.

IOP Conf. Series: Earth and Environmental Science 549 (2020) 012067 doi:10.1088/1755-1315/549/1/012067

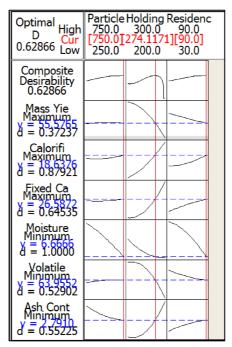


Figure 1. Optimization plot of all six responses

The current factor settings are shown by the red writing and red numbers on the upper part noted with 'cur'. There is also the plot of composite desirability shown in the first graph row. The composite desirability computes an optimal condition of factors regards to the response goal input and presents them in the optimization plot. The higher composite desirability is necessary in case to virtualize the data into experimental data. The composite desirability is changed by moving the red line regards to the parameters value and the desired responds. In this study, the optimization plot shows the composite desirability is 0.62866 which is acceptable and valid in this study because it is nearer to 1 rather than 0.

For each response, there is a y- value and d value. The y-value showed that the optimum value of the response at the respected current parameter applied, while the d value is the composite desirability of each response. The d value must be near to 1 as for the response to be valid and acceptable, while near to 0 is unacceptable.

In this case, the mass yield is 55.58% when applying the parameters of particle size of 750 μ m, holding temperature of 274.12°C and residence time of 90 minutes. However, the composite desirability to gain the mass yield of 55.58% is almost invalid because the d value is 0.37237.

The relation between each parameter to each response is also shown in this plot. The steep slope of the particular graph shows the significant effect of that parameter on the particular response. For the mass yield, the particle size shows very small changes as the size is increased or decreased, while holding temperature gave a significant change of mass yield as the temperature increased. The graph of holding temperature for mass yield shows negative slope, which indicates that the relationship between holding temperature to the mass yield is inversely proportional. As the temperature is increased, the value of mass yield is decreased. Residence time also shows slightly negative slope.

The y-value of calorific value shows that the optimum value is 18.64 MJ/kg at particle size of 750 μ m, holding temperature of 274.12°C and residence time of 90 minutes. The calorific value shows a slightly positive slope as the size increased. Holding temperature gave a very significant change on the calorific value as the temperature increase due to the steep positive slope. Residence time also shows a slightly positive slope and as the time increased, the calorific value is slightly increased. The composite desirability is near to 1, which is 0.87921.

The y-value of fixed carbon shows that the optimum value is 26.59% at particle size of 750 μ m, holding temperature of 274.12°C and residence time of 90 minutes. There is no visible change in fixed

carbon as the particle size increased. While holding temperature shows a significant change on the fixed carbon as the temperature increased. The increment of residence time also contribute to the calorific value increment.

The moisture content is at the optimum value of 6.67% when particle size is 750 μ m, holding temperature is 274.12°C and residence time is 90 minutes. All the three factors show significant changes since they show steep negative slopes which indicated as the size, temperature, and time is increased, the value of moisture content is decreased significantly. The acceptance of moisture content to reach the minimum value is acceptable as the composite desirability is 1.

The volatile matter is at the optimum value of 63.96% when particle size is 750 μ m, holding temperature is 274.12°C and residence time is 90 minutes. Particle size shows a slightly increasing slope of volatile matter as the size is increased. Holding temperature shows a very steep negative slope as time temperature increased. Residence time also shows a negative slope. As the time is increased, the volatile matter is decreased.

The optimum value of ash content is 2.79% at particle size of 750 μ m, holding temperature 274.12°C and residence time of 90 minutes. Particle size shows a negative slope regards to the ash content value. Holding temperature shows a steep positive slope as the temperature increased indicated that higher holding temperature caused higher ash content. Residence time shows a positive slope which contributes to the increment of ash content as the torrefaction time is increased.

The fixed carbon, ash content and calorific value are increase with the torrefaction temperature and time while the volatile matter content, mass yield and moisture content are decrease. This finding had been agreed by Nobre *et al.* [22]. Ash content of the biochar is targeted to be minimum since it will reduce the calorific value and the carbon content of the torrefied biochar. As result of the torrefaction process, volatile matter is decreased and fixed carbon are increased. The increment in fixed carbon and ash content leads to a densification of torrefied OPEFB biochar as more volatile components are removed.

This study shows that the changes in particle size do not give a significant impact on the results of those six responses. Meanwhile, holding temperature gives a significant impacts on each responses. While, the residence time gives a slightly impact on each of the responses. To obtain an optimal result, the suggested composite desirability was 0.62886 to generate an optimal torrefied biochar.

4. Conclusion

The torrefied OPEFB biochar has been successfully generated and analysed by using an experimental design created via RSM, Box-Behnken design according to the parameters; particle size, holding temperature and residence time of torrefaction process. The RSM analysis has been selected based on the method's ability to separately analyse each of the variables. The optimized torrefied OPEFB biochar has been successfully identified based on the six responses of mass yield, moisture content, volatile matter, ash content, fixed carbon and calorific value. The study proved that the holding temperature was found to exhibit the most significant effect on the responses. Based on the multiple optimization plot, the optimal value of the responses in regards of factors applied at 750μ m of particle size, 274.12°C of holding temperature and 90 minutes of residence time, the maximum mass yield can be obtained was 55.58%. In the other hand, the maximum value of calorific value and fixed carbon are 18.64MJ/kg and 26.59% respectively. Meanwhile, the minimum value of moisture content volatile matter and ash content are 6.67%, 63.96% and 2.79% respectively. RSM is able to give an analysis involving multiple factors and multiple responses. RSM is able to analyse the relationship between the factor levels to the responses. For future, considering on other factors which gives great impact on the responses and remove the particle size because it is insignificant factor. It will thus increase the validity of the experiment.

Acknowledgements

The authors gratefully acknowledge the Universiti Malaysia Kelantan on the SGJP-Matching grants awarded (R/SGJP/A0700/00093A/001/2018/00571) and Ministry of Education, Malaysia in support of

this study through the Research Acculturation Grant Scheme (R/RAGS/A08.00/01080A/001/2015/000211) and Fundamental Research Grant Scheme phase 1/2013 (R/FRGS/A08.00/A00800A/001/2013/00114). Millions thanks also goes to Rose Nadiah Binti Abu Hasan for her assistance and contributions in carrying out the research on this study.

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