



Effects of Operating Parameters on the Higher Heating Value of Kitchen Waste during Hydrothermal Carbonization

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Abstract

Hydrothermal carbonization (HTC) is a promising waste to energy technology for conversion of organic wastes with high water contents. There have been relatively few studies focused on evaluating the HTC of solid waste (SW). The studies have provided valuable information regarding HTC feasibility and potential environmental benefits. However, they lack the data necessary to understand how carbonization product composition and reaction extent change with the variation of operating parameter. This study sought to characterize kitchen waste from a university restaurant, to assess the impact of operating parameters on hydro-char and evaluate its potential as a solid fuel. The methodology for characterization was based on proximate analysis, ultimate analysis and calorific value. A ratio of dry sample to water of 1:9 was used to make a wet sample and stirred at 180 rpm in a thermal reactor under controlled HTC conditions. The results from proximate analysis revealed that moisture content, volatile matter, ash content and fixed carbon content were 9.6%, 65.1%, 13.6%, and 11.9% respectively. The ultimate analysis showed that carbon, hydrogen, nitrogen, Sulphur and oxygen were 48.8, 0.5, 0.4, 0.5 and 45.3% respectively. Mass yield of hydro-char decreased for all samples with increasing HTC reaction temperature. ANOVA confirmed the adequacy of the model, yielding R² and adjusted R² values of 0.8630 and 0.7398, respectively with a P value of 0.003 and F-value of 7.00. The high heating value, ultimate analysis and proximate analysis suggested that the hydro char from kitchen waste can potentially be utilized as a solid fuel.

Keywords: Hydrothermal carbonization; Higher heating value; Kitchen waste; Waste-to-energy; Solid fuel

Introduction

HTC is a thermochemical conversion method that uses hot, pressurized liquid water to react with wet organic substrates (i.e., those with moisture contents even higher than 80 wt. %). Temperatures in the process range from 180 °C to 250 °C, and the pressures are those needed to keep the liquid state of water (10–40 bar). The average length of stay is a few to several hours. Hot pressured water behaves as an acid/base catalyst precursor, operating as both a solvent and a reactant, and displaying

a greater ion product than at ambient circumstance (Kruse and Dinjus 2007). Such unusual characteristics produce a highly reactive behavior that can encourage the transformation of organic matter and its carbon enrichment. There are several possible applications for hydro-char including as a fuel, soil improver, sorbent, highly functionalized carbon material and activated carbon precursor (Demir-Cakan et al. 2009, Lu et al. 2011, Unur 2013, Stemann et al. 2013).

Several studies have been reported on the hydrothermal process for the upgrading of lignocellulose biomass; however, studies on a hydrothermal process for municipal solid waste are limited, Lu et al. (2011) studied the fuel quality and combustion performance of municipal solid waste treated using Hydrothermal treatment. Their results showed that hydrothermal treatment improved the fuel properties of municipal solid waste, in terms of reducing the water content, producing a uniform shape, and increasing the energy density of the fuel.

HTC is interesting for a variety of industrial applications due to the typical process parameters, including the temperatures and pressures involved, and the type of products obtained. Regarding other thermochemical procedures like supercritical water gasification (Fiori et al. 2012), the HTC plant would require much reduced investment and operating costs, as well as easier solutions to face safety issues. Moreover, if compared to common organic waste treatments (e.g., composting or anaerobic digestion), HTC involves shorter residence times and reducing equipment size.

HTC is also capable of managing possible variations of the chemical and physical characteristics of the input feedstock, which would be detrimental for biochemical processes. This is a crucial aspect when dealing with waste materials, which usually present a high grade of heterogeneity (Berge et al. 2011).

The general objective of the Study is to determine the effects of operating parameters on the higher heating value of kitchen waste during hydrothermal carbonization. The objectives are to characterize waste and to assess the effect of operating parameters (time, temperature and pressure) on hydro char yield. The study could have positive effects on society by converting kitchen waste into hydro char, which would reduce pollution, promote renewable energy, create job opportunities, provide a clean energy source, and eliminate health issues associated with waste dumping.

Materials and Methods

Materials

Food waste, Paper waste, Pressure reactor, Temperature sensors, Nitrogen, Energy meter, External temperature sensor, Timer, Sulphuric acid, Electrical heater and Freezer.

Characterization of SW

The University of Dar es Salaam's restaurants periodically gathered their food trash. All waste was weighed and then divided into four groups: (1) food items (apart from those with bones), (2) food with bones (such as chicken wings), (3) packaging materials (such as paper, plastic, condiment containers, and paper/plastic cups), and (4) others (e.g., plastic utensils, glass bottles). To determine the waste composition, each segregated fraction of the waste was weighed.

Proximate analysis test

The content of samples' volatile matter, fixed carbon, and ash was assessed by thermogravimetric analysis (TGA) on a TGA Q500. All gas flowrates were set to 50 mL per minute, and the sample heat rate will be 20 °C per minute. The proximate analysis was conducted in a nitrogen environment. Samples were heated to a temperature of 900 °C for 5 minutes after being heated to a temperature of 25 °C to 105 °C. The remaining sample was then heated to 900 °C for 10 minutes with the introduction of air. Moisture was defined as the mass that was lost in the nitrogen atmosphere at 105 °C. Volatile substances were defined as mass loss between 105 °C and 900 °C. Ash is the mass that is left over after combustion has finished. By deducting the percentages of moisture, volatiles, and ash from 100%, fixed carbon was calculated.

Moisture content test

According to ASTM Standard D 3175-89a for coal and coke Equation 1 was used to determine the sample moisture.

$$W_m = \left(\frac{W_i - W_d}{W_i} \right) \times 100 \quad 1$$

Where: W_m = moisture content (%), W_i = weight of sample before drying in oven at 105 °C for duration of 1 hour (g) and W_d =

weight of sample after drying in oven at 105 °C for duration of 1 hour (g).

Ash test

According to ASTM International (2002), the residual ash weight fraction was determined using Equation 2.

$$W_a = \left(\frac{W_b}{W_i} \right) \times 100 \quad 2$$

Where: W_a = ash content (%), W_b = weight of ash after burning the sample at 720 °C for 3 hours after heating (g) and W_i = initial weight of the sample utilized (g)

Volatile matter test

According to ASTM International (1997), the weight loss from burning the samples in an oxygen-starved environment while covering them with a cover to prevent any contact with oxygen below the atmosphere was used to calculate the fraction of volatile matter as shown in Equation 3.

$$W_v = \left(\frac{W_i - W_h}{W_i} \right) \times 100 \quad 3$$

Where: W_v = weight loss percentage in the analysis sample (%), W_i = initial weight of the sample utilized (g), and W_h = weight of the sample after heating at 925 °C for 7 minutes (g).

Fixed carbon value

The fixed carbon value will be determined using the proximate analysis test results mentioned above and ASTM D 7582-10, (2012) using Equation 4.

$$F_c = 100 - (W_m + W_a + W_v) \quad 4$$

Where: F_c is the analysis sample fixed carbon percent (%)

Ultimate analysis

A Flash 2000 Organic Elemental Analyzer was used to estimate the amount of carbon, hydrogen, nitrogen, and sulfur. The method was also applied by Sarakikya and Kiplagat (2015). By deducting the percentages of ash, carbon, hydrogen, nitrogen, and sulfur from 100%, the oxygen content was estimated. For the oxidation column of the analyzer, electrolytic copper and copper oxide were

utilized, and 2, 5-Bis (5-tert-butylbenzoxazol-2-yl) thiophene (BBOT) was used for calibration.

Gross calorific value

Calorimetry, as opposed to temperature measurement, is the science of measuring amounts of heat. Calorimeters are the devices used to make these measurements. The industry-standard tools for determining the calorific values of flammable liquid and solid samples are oxygen bomb calorimeters. The amount of heat units released by a unit mass of a sample when burned with oxygen in a container of constant volume is referred to as the calorific value (heat of combustion) of a sample.

1180P Oxygen Combustion Bomb, Bomb head support stand, Parr 6200 Oxygen Bomb Calorimeter, distilled water. Sample was dried for 24 hours at 105 °C in a crucible. The dried was gridded to a powder form. A gram of sample was weighed and put into the sample vial (0.5- 1.2 g).

Oxygen gas cylinder was opened and flow rate was set to (400 -3000 kPa). Distilled water chamber was filled to the mark. Calorimeter was turned on with the pump and heater activated for 20 minutes and the jacket temperature stabilized at 29 °C. Calorimeter bucket was filled with 2 L of distilled water and a prepared sample of 1.16 g 24 hours at 105 °C dried blended sample into capsule was set into the bucket. The vessel was then placed in the calorimeter and the testing sequence was initiated. After all, three tests were completed, the results were recorded.

Effect of operating parameters

The ratio of dry sample to de-ionized (DI) water in the reactor was 1:9. 100 grams of dry sample, were mixed with 900 grams of DI water. The reactor stirrer was set at 180 °C/minute once the reactor was sealed. From that point until the HTC process was complete, the reactor mixes were stirred. The hydro-chars were made at reaction temperatures between 180 °C and 260 °C, pressures between 1300 kPa and 4130 kPa, and residence times between 20 and 40 minutes. The following steps were used to heat the reactor:

- a) The reactor's contents were heated to the desired reaction temperature at an average rate of 80 °C per minute;
- b) After reaching the target reaction temperature, the reactor temperature was maintained for 30 minutes;
- c) Following the 30 minute residence period, the reactor was immersed in an ice-water bath until it reached 300 °C, which typically takes 10 minutes;
- d) Reactor stirring was halted once 300 °C was reached.

Produced gases were vented in a fume hood, and vacuum was used to separate the process liquid from the solid hydro-char by using filtration and a Whatman 41 filter (20 µm). With DI water, hydro-char was rinsed until the pH of the water reached that of the DI water. The wet hydro-chars were dried in a 105 °C oven for 24 hours before being placed in a Ziploc bag for later analysis.

RSM modelling and optimization

Table 1: Experimental factors that were in the CCD for the hydro char process.

Factors	Coded factors	Levels with coded and actual values				
		-α	-1	0	1	α
Temperature (°C)	T	153	180	220	260	287
Pressure (kPa)	P	64	200	400	600	736
Residence Time (min)	R	13	20	30	40	47

Quality of the fitted quadratic model was evaluated by using the significances test and analysis of variances (ANOVA). The ANOVA based on 5% significance level (alpha-value) was conducted to test the significance effects of each factor and their interactions on HHV. The effect of the parameter under consideration was significant and stayed in the model if the significance is less than 0.05 (P-value 0.05), which is in the region of the hypothesis rejection. On the other hand, the effect of the

In this study, a Response Surface Methodology (RSM) was used to analyze process parameters (temperature, pressure, and residence time) that affect the yield of hydro char as shown in Table 1. To avoid laying-off (unnecessary repetition of experiments), a Central Composite Design (CCD), which renders an equitable distribution of the experimental points was adopted by means of Minitab 17.0 statistical software package. The total experimental runs conducted are computed using Equation 5 (Bayuo et al. 2020, Sadhukhan et al. 2016).

$$N = 2^n + 2n + n_c \quad 5$$

Where *N* = total number of experimental runs, *n_c* = number of center points, and *n* = number of independent variables (factors).

parameter under consideration is not significant when the significance is more than 0.05 (P-value > 0.05). To find the best combinations of the three process input variables to maximize HHV, a response optimizer was used.

Mass Yield of Hydro-char

Mass yields was calculated using Equation 6 below and they show how much of the initial feedstock was converted into solid (hydro-char), liquid and gas.

$$\text{Solid Mass yield (\%)} = \frac{\text{Mass of dry hydro - char}}{\text{Mass of untreated dry feedstock}} \times 100\% \quad 6$$

Results and Discussion

Characterization of the waste

Kitchen wastes were collected in large bins, then continued with its preparation (pre-treatment), which consisted in the blending through a professional blender to obtain a

homogenous mixture. Each of the four huge dumpsters was used to gather about 40 kg of solid garbage every day. The composition of the food waste is shown in Table 2.

Table 2: Collected food waste composition

Categories	Composition %
Food waste (without bones)	52
Food waste (with bones)	1.7
Paper	3.5
Cardboard	6.5
Plastic	3.3
Others	33

The organic fraction was divided into small portions (approximately 2-3 kg), which were then placed into buckets. The food waste combination was then poured into small buckets and placed in the freezer so that its basic composition would not alter throughout the course of the trials.

Characteristics of hydro char yield

Proximate analysis

The proximate analysis, which compares the energy content of biomass and fuels by comparing the ratio of combustible to non-combustible materials, is a key feature of both. It calculates the samples' fixed carbon, moisture, volatile matter, and ash contents. Table 3 lists the findings of the proximate analysis of the optimized hydro-char made from kitchen solid waste (KSW), and the values are contrasted with those of several coals as presented by Sarkar and Bhattacharyya (2012).

Generally speaking, fuel with low moisture, low volatiles, and high fixed carbon is regarded as a good fuel since more fixed carbon increases HHV while higher volatile matter lowers fuel combustion performance. According to Table 3, the moisture content and volatile matter of kitchen waste were respectively 9.64% and 65.12%, however in the optimized hydro-char, these values dropped to 2.5% and 45.6%. Additionally, rice straw had a fixed carbon content of 11.86%, which climbed to 35.4% for the improved hydro-char. The reaction temperature affects both a rise in fixed carbon and a decrease in volatile matter since biomass degrades rapidly at higher temperatures, which causes a drop in volatile matter and ultimately an increase in fixed carbon content (Liu et al. 2013). A rise in the value of fixed carbon is indicated by a release of volatile matters taking place during the HTC.

The ash content increased from 13.56% of kitchen waste to 16.5% in kitchen waste hydro-char, which is in agreement to previous literature by Gao et al. (2019) and Reza et al. (2013). It was found that the rice hull's 21% ash concentration rose to 29.8% in the rice hull biochar, which is explained by the reaction temperature. The results of the optimized hydro-char's proximate analysis resemble those of different kinds of coal, as shown in Table 3.

Table 3: Properties of raw and treated surrogate KSWs by hydrothermal treatment.

Parameters	Raw surrogate MSW	Hydrothermally treated MSW
Moisture (a.r.)	9.64%	2.5%
Proximate analysis (wt. %, d.b.)		
Volatile matter	65.12%	45.6%
Fixed carbon	11.86%	35.4%
Ash content	13.56%	16.5%
Ultimate analysis (wt. %, d.b.)		
C	37.19%	48.8%
H	4.8%	5%
N	0.2%	0.4%
O	57.6%	45.3
Calorific value (MJ/kg, d.b.)	17.15%	19.61%
a.r., as received.		
d.b., dry basis.		

Ultimate analysis

The CHNS/O analysis describes the elemental composition (carbon, hydrogen, oxygen, nitrogen and Sulphur) of a material (Nwankwo and Amah 2016). Table 3 displays the final analysis of kitchen solid waste and the optimized hydro-char. The kitchen waste had a carbon value of 37.19%, however an optimized hydro-char increased that value to 48.8%. On the other hand, after going through the HTC process, the optimized hydro-char's oxygen content dropped from 57.6% of kitchen waste to 45.3%. Higher carbon and lower oxygen concentrations are thought to improve hydro-char's ability to burn as described by Sevilla et al. (2011). The deoxygenation reactions (decarboxylation and dehydration) that occur during the HTC, which are affected by the process parameters (reaction temperature and reaction time), are responsible for a rise in the carbon content and a decrease in the oxygen content, these effects were also shown by Kang et al. (2012).

After the HTC procedure, a little rise in the nitrogen content of hydro-char products was observed. Kitchen waste had a nitrogen concentration of 0.2%, which rose to 0.4% in the hydro-char, showing that the nitrogen is not eliminated by dissolution in hot water but is instead held in the hydro-char. The less reactive protein fraction during the process could be the cause of an increase in the nitrogen concentration of hydro-chars (Liu et al. 2013).

**Experimental results of HHV
RSM modelling of HHV**

Using RSM-CCD approach, the quadratic regression model as illustrated in Equation 7

Table 4: ANOVA for HHV

Source	DF	Adj SS	Adj MS	F-value	P-value	Remark
Model	9	63.8299	7.0922	7.00	0.003	Significant
Linear	3	42.7906	14.2635	14.08	0.001	Significant
T	1	31.7794	31.7794	31.37	0.000	Significant
P	1	1.1708	1.1708	1.16	0.308	Non-Significant
R	1	9.8404	9.8404	9.71	0.011	Significant
Square	3	19.7865	6.5955	6.51	0.010	Significant
T2	1	13.1351	13.1351	12.97	0.005	Significant
P2	1	5.7030	5.7030	5.63	0.039	Significant
R2	1	4.4476	4.4476	4.39	0.043	Significant

was generated to demonstrate the relationship between HHV and factors of temperature (T), pressure (P) and residence time (R). In the model T, P and R are linear terms, TR, TP and PR interaction terms and T², P² and R² are quadratic terms.

$$\begin{aligned}
 \text{HHV (MJ/Kg)} = & -14.1 + 0.300T + \\
 & 0.0032P + 0.488R - 0.000597T^2 - \\
 & 0.000016P^2 - 0.00556R^2 + 0.000037TP - \\
 & 0.000479TR + 0.000089PR
 \end{aligned}$$

The accuracy and confidence level of the model were determined by R-square and adjusted R-square; their values were 0.8630 and 0.7398 respectively. This suggests that the factors analyzed explain 86.30% of the HHV variability and that the model could not explain only 13.7% of the variation of the response. Analysis of variance (ANOVA) was used to determine the adequacy of the model. Table 4 presents the results of the quadratic response surface model fitting in the form of ANOVA. If the determined/calculated F-value is found to be greater than that of the tabulated F-value, then the model is a strong experimental data predictor as stated by Ofori-Boateng et al. (2014). The F-value obtained in the present study was 7.00 (Table 4) while critical F-value was 3.2 which suggested the fitness of the response surface model. More so, the significance of each of the model terms was evaluated using the probability of error value (P-values). In Table 4, the P-values less than 0.050 showed that the terms are significant.

2-way interaction	3	11.2528	3.4176	3.41	0.038	Significant
TP	1	0.7045	0.7045	0.70	0.424	Non-Significant
TR	1	5.2941	5.2941	5.29	0.041	Significant
PR		0.2542	0.2542	0.25	0.627	Non-Significant
Error 10	1	10.1306	1.0131			Significant
Lack-of-fit	5	10.1300	2.0260	0.39	0.412	Non-Significant
Pure Error	5	0.0005	0.0001			
Total	19	73.960				
R2	0.86					
	30					
Adj-R2	0.7398					

It was found that with the exception of the linear effect of pressure (P) and interaction effects of TP and PR, all model terms were statistically significant. The temperature (T) had the highest F-value of 31.37 implying that it had the most significant influence on the HHV in comparison with residence time (R) and pressure (P) which had 9.71 and 1.16 respectively. In Table 4, the lack of fit p-value of 0.415 showed the lack of fit is not significant relative to the pure error. Non-significant lack of fit, however, is good because the model is geared towards perfect fitness. It implied that a good correlation

between the factors and responses could be drawn by the developed model.

Effect of process variables on the HHV

The studied input hydro-char process parameters are seen to have an influence on HHV as illustrated in Figure 1. However, processing temperature and residence time are significant as evidenced by respective p-values <0.05 in Table 4 while pressure is not significant. Besides, temperature is more significant than residence time. This is as well depicted from Figure 1 with temperature covering a wide range of mean response.

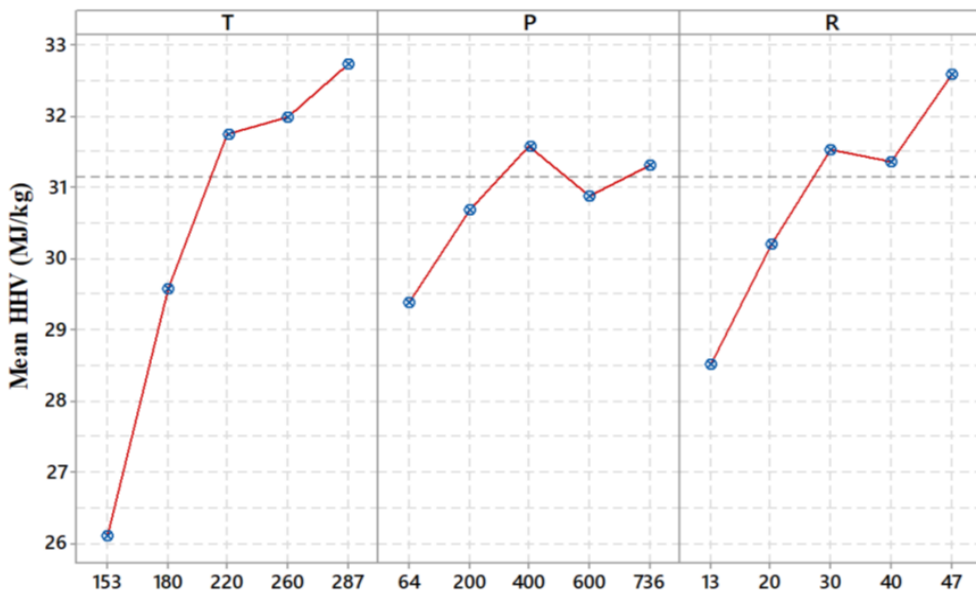


Figure 1: Main effect plot

The purpose of these plots is to simultaneously create understanding of the main effects and interactions among the factors for the selected response. The combined effects of the three process variables investigated on the HHV of the hydro-char are visualized in Figures 2-10.

Effect of Temperature and residence time

Figure 2,3 and 4 show the response surface and contour plots of the interactions between processing temperature and residence time

with regard to the hydro-char's high-temperature volatility (HHV) at a fixed pressure of 400 kPa. The maximum HHV, 33.61 MJ/kg, is shown by the combined plots of temperature and residence time at 250 °C and 40 min. The two main factors affecting the HHV are temperature and residence time. This could be explained by the fact that higher temperatures and longer residence times result in a greater degree of intermediate dissolution.

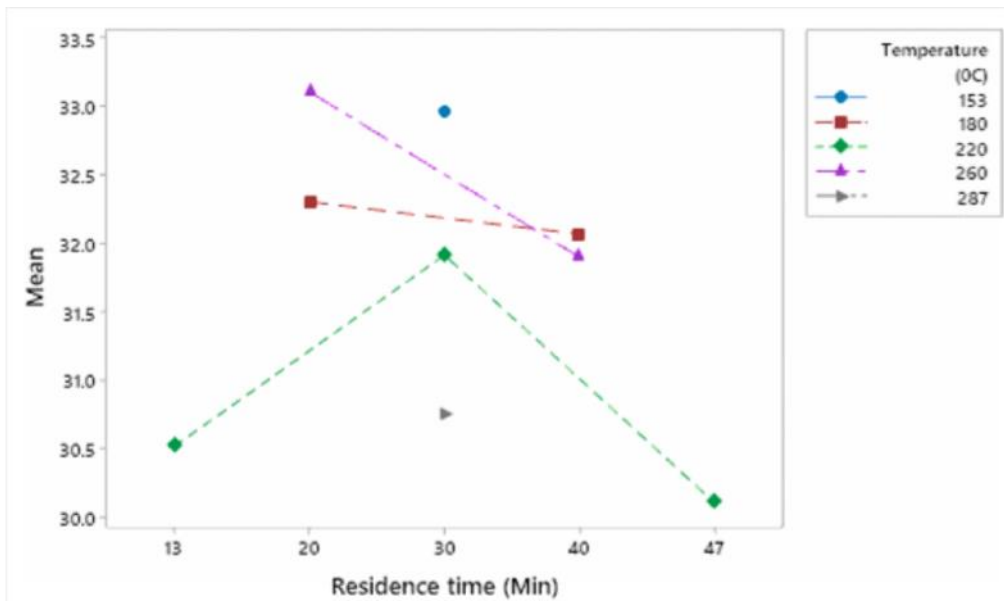


Figure 2: Interaction plot of temperature and residence time

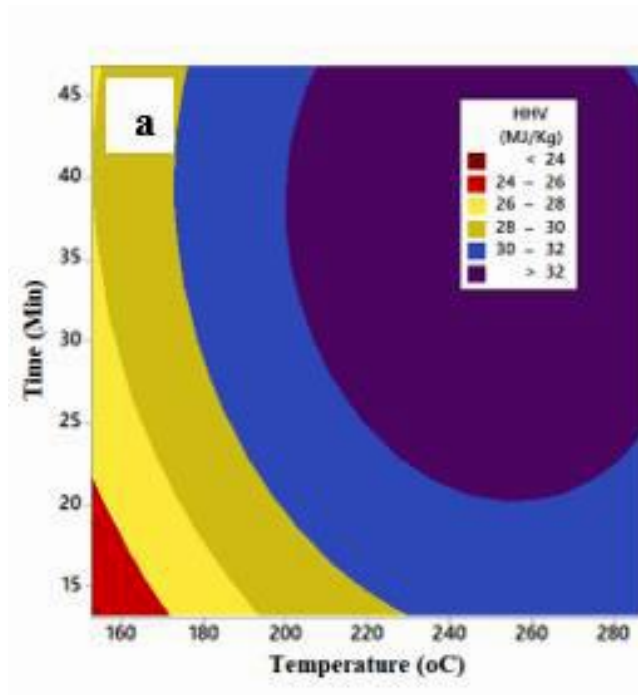


Figure 3: Contour plot of temperature and residence time

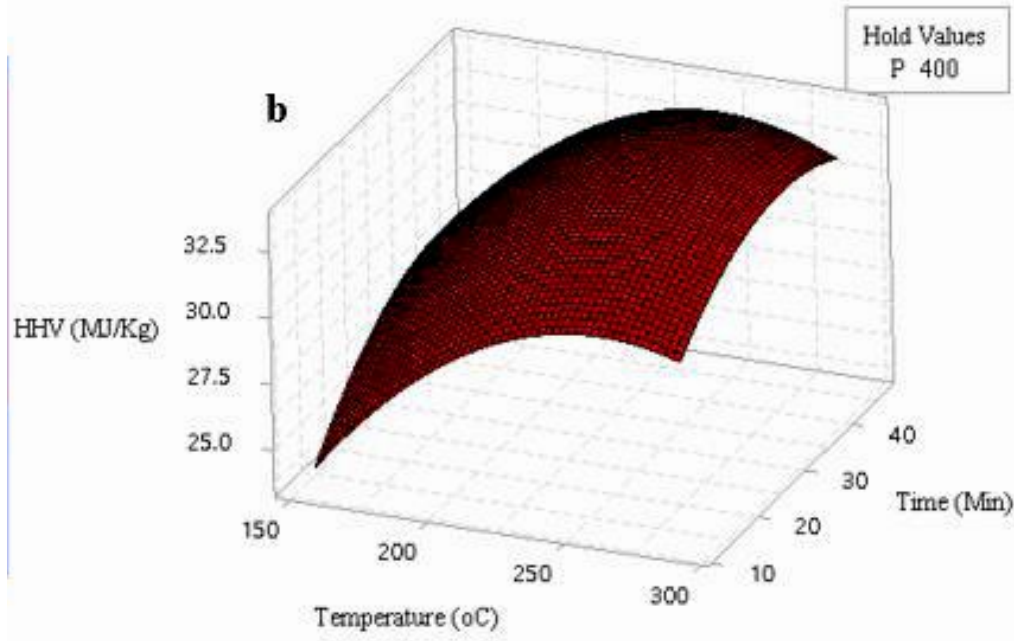


Figure 4: Response surface plot of temperature and residence time

However, the higher temperature (>250 °C) decreases intramolecular hydrogen bonding in cellulose He et al. (2008) and increases the polarity of the CH₂OH group. It also

encourages the solid-solid process that breaks down polysaccharides to generate hydro-char Chen et al. (2021).

Effect of temperature and pressure

Figure 5, 6 and 7 show the interactions between processing temperature and pressure on the hydro-char's HHV at a constant 30-minute residence time. The HHV increases from 26.88 to 32.64 MJ/kg when the temperature rises from 153 to 240 °C, according to the response surface and contour

plot, whereas at a constant temperature of 240 °C and a 30-minute residence period, the HHV slightly rises with pressure from 31.23 to 32.17 MJ/kg. The hydro-char HHV is little impacted by the temperature and pressure interactions during processing. The results of the ANOVA Table 4 also support this.

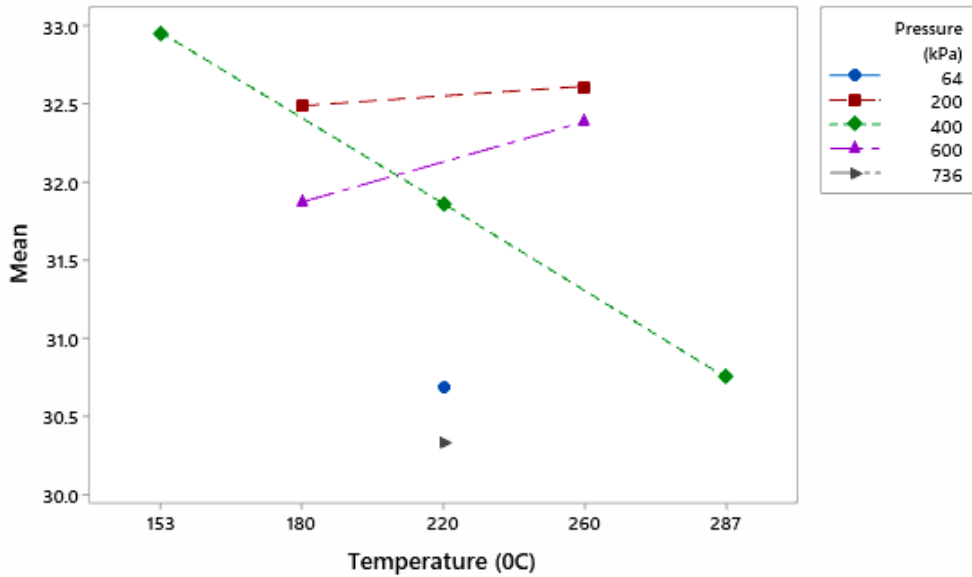


Figure 5: Interaction plot of temperature and pressure

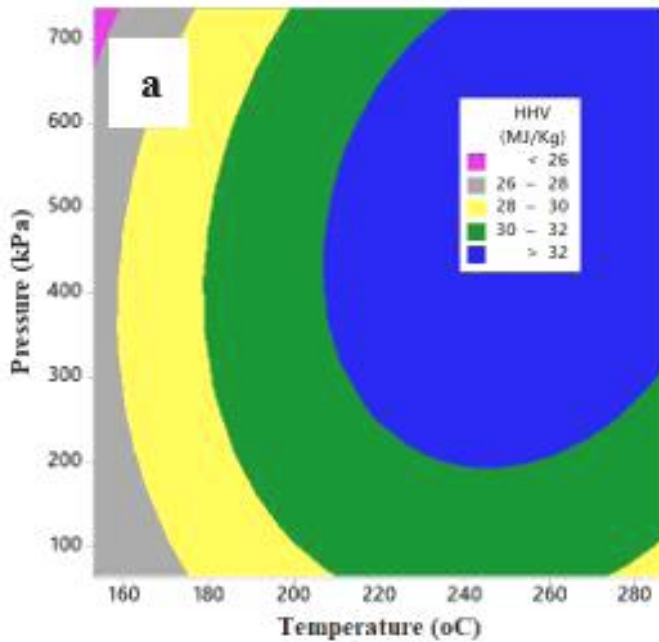


Figure 6: Contour plot of Temperature and Pressure

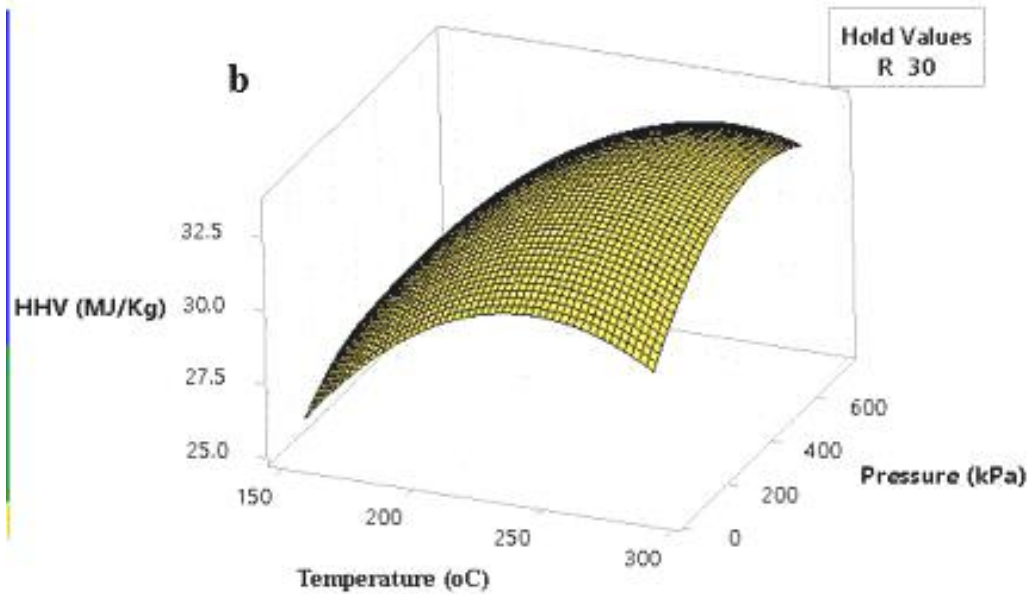


Figure 7: Response surface plot of temperature and pressure plot

Effect of residence time and pressure

Figure 8, 9 and 10 depict the response surface and contour plots of the residence time and pressure with respect to HHV at a constant temperature of 220 °C. HHV is a vital

parameter that indicates a correlation of how much of the waste is transformed into solid fuel during HTC. This interaction exhibited the lowest influence on the HHV.

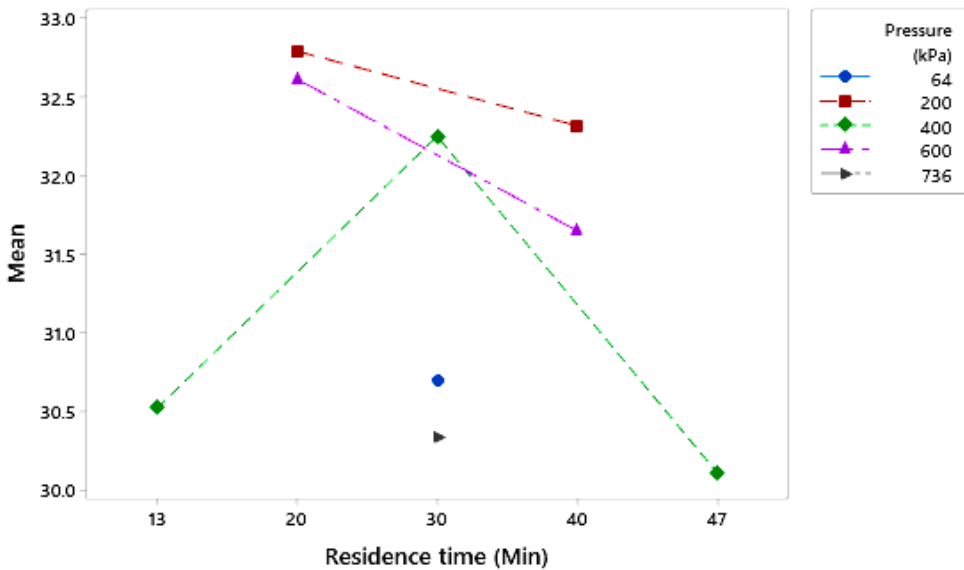


Figure 8: Interaction plot for residence time and pressure plot

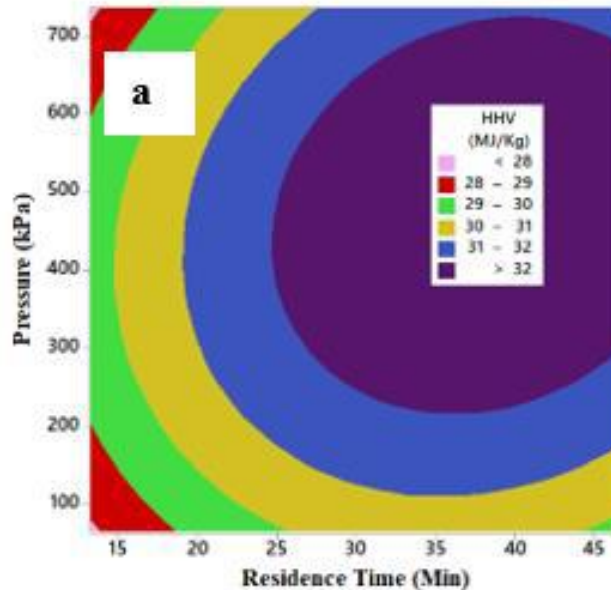


Figure 9: Contour plot for residence time and pressure

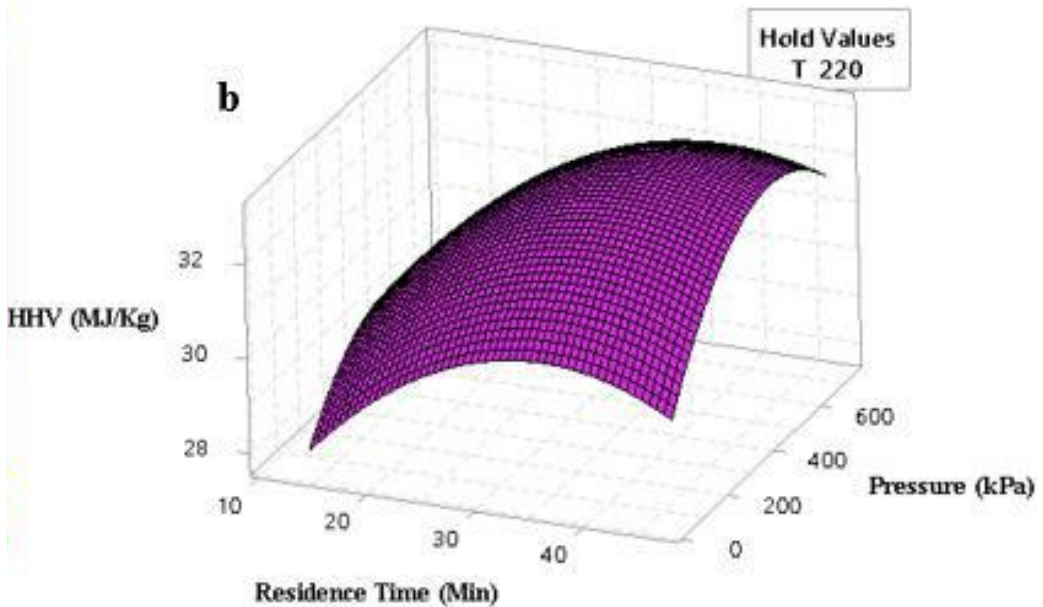


Figure 10: Response surface plot for residence time and pressure

Diagnostic plots for residuals

Diagnostic plots for scatter of the residuals such as normal probability plot of residual (a) plot and plot of residuals versus (b) fits and order were applied. The assumptions of normality, randomness and independence of the residuals were suitable, satisfied and

matched as illustrated in Figure 11. Thus, lack of fit of the model was insignificant. The normality assumption was also satisfied as the normal probability plot (Figure 11) approximated along a straight line.

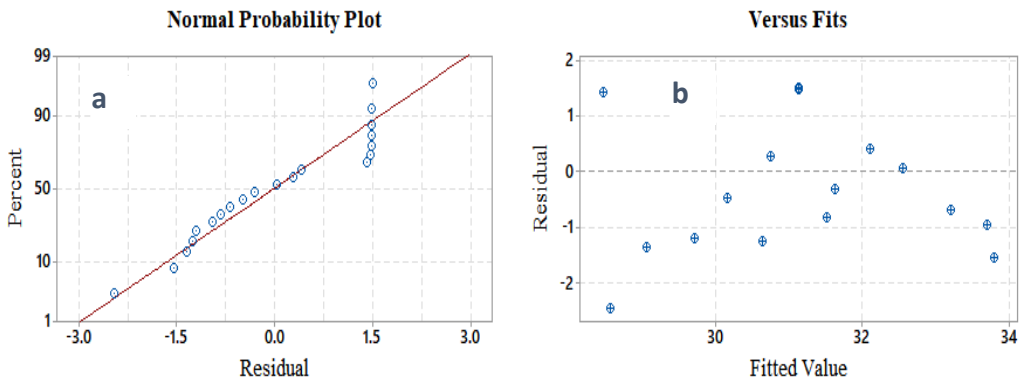


Figure 11: Residual plots of Normal Probability and plot of Residual versus fits

Optimization

Optimal points for the temperature, pressure and residence time were established to attain the maximum of HHV. The second order polynomial models developed in this analysis were utilized for the responses in demand to obtain specific optimum input factors.

$$HHV(MJ/KG) = -14.1 + 0.300T + 0.488R - 0.000597T^2 - 0.000016P^2 - 0.00556R^2 - 0.000479TR$$

Because the simulation model includes some probabilistic input variables, like the processing time, etc., the optimization problem taken into account in this study is stochastic in nature. Due to the interplay of

several components in the objective function, it is a nonlinear optimization problem (TR). The discrete values of the model control factors make it a discrete optimization issue. Furthermore, since just one answer (HHV) is maximized, the problem is a single objective optimization problem.

According to Equation 8, the ideal HHV of 33.59 MJ/kg was attained at 251.94 °C, 505.32 kPa of pressure, and 36.97 minutes of residence time. The ideal input settings for the hydro-char's HHV are shown in Figure 12. Therefore, the crucial conditions are 251.94 °C (a), 505.32 kPa (b), and 36.97 mts (c) in order to achieve the highest HHV.

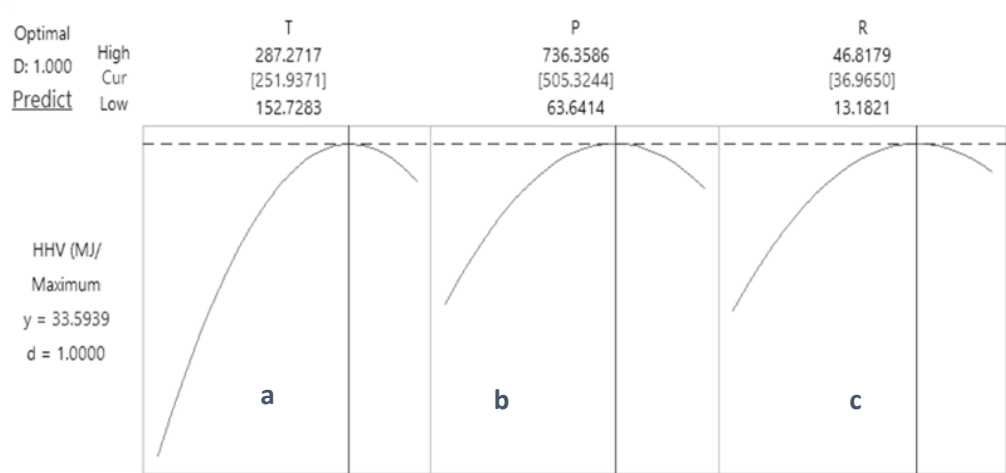


Figure 12: Optimization plot

Conclusions

This study demonstrated that organic/food solid waste from University of Dar Es Salaam restaurants can be converted to solid fuel (hydro-char) through HTC as well improving on organic waste management and treatment. Characterization of kitchen waste as well as hydro-char based on proximate analysis, ultimate analysis, calorific value and mass yield was elucidated. Further, the effect and response surface optimization (RSM-CCD) of the HTC process parameters (residence time, temperature and pressure) was also conducted to maximize the hydro-char yield. The effect of the temperature, residence time and pressure on the properties of the hydro-char were also examined.

For a better understanding of the environmental effects of HTC, more research analyzing the conversion of wastes by HTC is still needed. Additionally, greater knowledge is required regarding the energy properties of the gas and liquid phases. A life cycle evaluation of each activity is important when the appropriate data have been collected and will provide you a better understanding of the system's overall environmental impact.

Declarations

Ethics approval and consent to participate

This study was approved by the Department of Mechanical Engineering, Moi University, Kenya (Approval No. ENG/MES/3983/20).

Consent for publication

Not applicable

Availability of data and material

The raw data supporting the conclusions of this study are available from the corresponding author upon request.

Competing interests

The author declares that there is no conflict of interest regarding the publication of this paper.

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Authors' contributions

VGK and SM designed the study. VGK collected samples and performed the analytical work. SM supervised the study. VGK analyzed the collected data and wrote the first draft of the manuscript. All authors revised and approved the final manuscript.

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