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<https://doi.org/10.15760/honors.483>

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TORREFACTION KINETICS OF HEMP HURDS, AN EMERGING AGRICULTURAL
BYPRODUCT, IN A SMALL RETORT

BY

TEL JENSEN

A thesis submitted in partial fulfillment
of the requirement for the degree of

BACHELOR OF SCIENCE
IN
CIVIL AND ENVIRONMENTAL ENGINEERING

Thesis Advisor:
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ACKNOWLEDGMENTS

Dr. Johnson, obviously.

Emily and Julie, for graciously enduring the smoke.

Corina, for Matlab help and fizz and snacks and commiseration.

Henry, for the hurds.

Äkräs, for everything, but especially the turnips.

ABSTRACT

As knowledge concerning the consequences of unsustainable energy sources and agricultural practices grows, development of low-impact alternatives to both becomes more important. Sustainable sources of biomass offer solutions to both problems. This study characterized the reaction kinetics of torrefying an agricultural byproduct, the woody interior of hemp stems, or hurds. Torrefaction was carried out at 250 °C in small and simple retorts made of galvanized steel pipe. A first-order model most closely approximated the reaction. A sensitivity analysis was performed and the data was adjusted to correct for three potential sources of error, and regressed to find the best fit in each case. When corrected for torrefaction time, initial moisture content, and final mass loss, the best fit regressions for mass loss with time yielded governing equations $L = 0.5388 \times (1 - e^{-0.003069(t+113)})$, $L = 0.4523 \times (1 - e^{-0.003015t})$, and $L = 0.4088 \times (1 - e^{-0.008718t})$, respectively. Regression of uncorrected data gave the governing equation $L = 0.5388 \times (1 - e^{-0.004682t})$. Results of this study indicate that the conversion of hemp hurds via torrefaction follows first-order reaction kinetics and that reaction is most sensitive to the torrefaction time under fixed-bed, isothermal conditions.

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1.0 INTRODUCTION

1.1 Biomass

As the negative impacts of fossil fuel use, from extraction (Butt et al. 2013; Bouman et al. 2015; Griffith et al. 2012) through processing (Mangmeechai et al. 2014), transportation (Obida et al. 2017), and finally consumption (PecI et al. 2017), are better understood, the need for tools and alternatives to mitigate those impacts become clearer. A variety of methods using biomass as a raw material may accomplish both goals as they may sequester carbon (Abdel-Fattah et al. 2015) and/or provide alternative fuels (Yue et al. 2014), among other benefits.

Some forms and uses of biomass, however, have significant drawbacks. For example, agricultural practices used to grow rapeseed and corn (maize) for biodiesel and bioethanol production, respectively, increase N₂O emissions. The additions of N₂O may negate any climate benefits of a reduction in carbon emissions that result from replacing fossil fuel (Crutzen et al. 2016). Additionally, augmenting forest soils with biochar can lead to faster decomposition of native organic matter and higher rates of soil CO₂ respiration (Mitchell et al. 2015), again reducing climate benefits.

1.2 Hemp hurds

Many useful products are derived from hemp. The fibers are used for paper, textiles, insulation, and biocomposites. Hemp seeds and seed oil are used for human food and animal feed. Hemp seed oil is also used in oil-based paint, cosmetics, and as a raw material for plastics (Carus et al. 2013). Epidermal appendages, called trichomes, contain several terpenophenolic compounds, including cannabidiol, which has therapeutic potential for a number of disorders in addition to acting as an antioxidant (Cherney et al. 2016). Finally, the inner woody core of the hemp plant, called hurds or shivs, is used as animal bedding and, combined with lime, as a crack-proof construction material (Carus et al. 2013). This last portion of the hemp plant, the hurds, is the lowest value material but produced in the largest quantities, with 1.7 times the mass of hurds produced as hemp fiber (Ibid.).

As the market for hemp products continues to grow, research has shown hemp production can provide significant environmental benefits as compared to typical large-scale agricultural crops. For example, in Europe, hemp (*Cannabis sativa*) is cultivated without the use of synthetic chemical fertilizers or pesticides, even on farms that are not organic (Carus et al. 2013). A life cycle assessment found that hemp cultivation sequesters a net of 26.01 tons CO₂ equivalent/ha (Zampori et al. 2013). In 2016, the Oregon legislature passed a law allowing a limited amount of industrial hemp cultivation in the state. Depending on the outcome of these initial trials and the future political environment in Oregon and the USA, hemp hurds could become an abundant local agricultural byproduct and source of biomass.

1.3 Torrefaction

Torrefaction is pyrolysis of biomass that takes place between 225 and 300 °C. Woody and herbaceous biomass is largely made up of cellulose, hemicellulose, and lignin (Bergman 2005). Hemicelluloses are the most reactive compounds in woody biomass, and decompose at temperatures between 225 and 325 °C. Lignin decomposes gradually at temperatures between 250 and 500 °C. The temperature range at which cellulose decomposes, 305-375 °C, falls outside that considered for torrefaction (Prins 2006).

Biomass is often torrefied to make it more suitable for intended use (e.g., energy production). Torrefaction increases biomass's energy density by removing less-energy dense volatile constituents. It is also made easier to grind, pelletize, and transport (Bergman 2005).

This honors thesis project aims to characterize and optimize the torrefaction of various raw biomass materials using fixed-bed torrefaction and low oxygen content sample retorts. Initial experiments were conducted using hazelnut shells and hemp hurds. Further experiments and characterization of the torrefaction of hemp hurds was done to determine the efficiency of fixed-bed torrefaction for the production of hemp biochars. Data analysis included the determination of the best fit model for the order of reaction and kinetic coefficients of that reaction. A sensitivity analysis to determine the impact of various experimental conditions on the optimization of the torrefaction process was also conducted.

2.0 MATERIALS AND METHODS

2.1 Instrumentation, Apparatus, Experimental Procedure

Eight (8) trials using hemp hurds (Item number F-H3, Hemp Traders, Paramount, CA) were packed into four (4) small retorts made of threaded sections of galvanized pipe (120 mm long, 27 mm internal diameter) and capped tightly using a pipe wrench (Figure 1).

Figure 1. Galvanized steel pipe retort.

The mass of hurds in each retort was determined by weighing retorts before and after packing (Mettler PJ3600 DeltaRange). Retorts were placed side-by-side on the same rack in a small laboratory oven (Blue M Electric Company Stabil-Therm B-1206Q-A) (Figure 2). Temperature was recorded at one-minute intervals using a digital thermometer (6802 II) with a thermocouple probe inserted into the top of the oven. The oven temperature was set to reference number 25 and power set to high for all trials. At the end of each trial's time, the

oven was turned off and the door opened to allow consistent cooling while the retorts remained in the oven. After the retorts had cooled to room temperature, each was weighed. The torrefied hurds were emptied into aluminum weigh boats (Figure 3) and weighed, and the empty retorts were weighed again.

Figure 2. Retorts in lab oven with digital thermometer.

Figure 3. Hemp hurds torrefied at 250 °C for five hours.

It should be noted that no attempt was made to exclude oxygen from the retorts. They were packed under ambient atmospheric conditions, so some oxygen was present. Torrefaction is defined as thermal degradation in the absence of oxygen. However, the mass of oxygen in the retorts was small initially, and it would have decreased as the gas in the retort heated and was forced out through the threads. It is unlikely that more than a negligible amount of air was able to flow back into the heated retorts.

2.2 Data Analysis

Calculation of kinetic coefficients and constants roughly followed Chen and Kuo (2011). Specifically, the rate of reaction is given by the following equation:

$$\frac{dX}{dt} = k(1 - X)^n \quad (1)$$

where n is the order of the reaction, k is the reaction rate constant, t is torrefaction time, and X is the fractional conversion of the sample, defined as

$$X = \frac{L}{L_{48}} \quad (2)$$

L is the fractional mass loss of the sample, defined as

$$L = \frac{M_i - M}{M_i} \quad (3)$$

where M_i is the initial mass of the sample and M is the mass of the sample after torrefaction. L_{48} is the fractional mass loss after isothermal torrefaction for 48 hours, defined as

$$L_{48} = \frac{M_i - M_{48}}{M_i} \quad (4)$$

where M_{48} is the mass of the sample after 48 hours of isothermal torrefaction.

If n , the order of the reaction, is 1, integration of Equation (1) yields

$$\ln \frac{1}{1-X} = kt \quad (5)$$

and a plot of $\ln \frac{1}{1-X}$ against torrefaction time will give a straight line with slope equal to the rate constant, k . Solving this equation for conversion, X , gives

$$X = 1 - e^{-kt} \quad (6)$$

and solving for mass loss, L

$$L = L_{48} \times (1 - e^{-kt}) \quad (7)$$

If n is not equal to 1, integrating Equation (1) yields

$$(1 - X)^{1-n} - 1 = k(n - 1)t \quad (8)$$

and a plot of $(1-X)^{1-n} - 1$ against torrefaction time will give a straight line with slope equal to $k(n - 1)$.

2.3 Sensitivity Analysis

A sensitivity analysis was conducted to determine how sensitive the experimental results are to three experimental variables: the isothermal torrefaction time, moisture content of the hurds, and the optimal mass conversion. These experimental variables were chosen based on observations made during data collection. For example, it was not feasible to measure temperature inside the retorts, so the air temperature in the oven directly above the retorts was measured during torrefaction. As such, the true isothermal torrefaction time may vary across replicate experiments. Furthermore, complete mass conversion of the hemp hurds was assumed after 48 hours of torrefaction time, thereby allowing for only an estimation of the long-term maximum mass conversion. Measured data was adjusted to account for these experimental variables, data was then regressed, and the highest R^2 value was assumed to correspond with the most likely true value. Each adjustment (for each of the three experimental variables tested) was performed on the raw data separately. It may be of interest to note that an attempt was made to perform all three adjustments concurrently, but the analysis failed to converge on a best-fit solution.

3.0 RESULTS AND DISCUSSION

The experimental data was regressed assuming reaction kinetics following Chen and Kuo (2011) and presented in Equation (1). The order of reaction integer value n was varied from 1 through 10 and the intercept forced to zero (Figure 4). R^2 values for each value of n were compared, and the n with the highest R^2 representing the most likely (best fit) reaction order.

Figure 4. Plot of R^2 versus modeled order of reaction.

When the data was regressed using integer values of n from 0 through 10, the regression with $n=1$ gave highest R^2 , 0.7449 (as shown in Figure 4). All subsequent results assume a first-order reaction to describe the thermal conversion of raw hemp hurds to hemp biochar under torrefaction.

To further characterize reaction kinetics of the hemp hurds' thermal conversion, integration of the best-fit governing equation (see Equation 5) and regression of the transformed raw

data gave a reaction rate constant of $4.682 \times 10^{-3} \text{ day}^{-1}$ (Figure 5) with the following governing first-order equation:

$$L = 0.5388 \times (1 - e^{-0.004682t}) \quad (9)$$

Figure 5. Plot of mass conversion versus torrefaction time at 250 °C with linear regression. Error bars are one standard deviation of mean.

In an attempt to determine the effect/sensitivity of torrefaction time on the best-fit first order equation determined in this analysis, the torrefaction time, t , for each trial was shifted by between -180 and 180 minutes in 1-minute increments (Figure 6).

Figure 6. Plot of R^2 vs. torrefaction time correction.

The highest R^2 for adjusted torrefaction time was 0.8200 with a time shift of 113 minutes (Figure 6). Given that the temperature of the hurds lagged behind the measured air temperature in the oven, and that the air temperature of the oven did not reach torrefaction temperature of 200 °C for approximately 20 minutes after the beginning of heating and for approximately 60 minutes for 250 °C (see Figure 7), this suggests that the temperature of the hurds remained above torrefaction temperature well after the end of heating. Adjusting Equation 9 for torrefaction time (Figure 8) gives the governing equation:

$$L = 0.5388 \times (1 - e^{-0.003069(t+113)}) \quad (10)$$

Figure 7. Representative measured temperature profiles for torrefaction of hemp hurds.

Figure 8. Model corrected for torrefaction time plotted with measured data. Error bars are one standard deviation in each direction.

To account for initial moisture content, the initial mass, M_i (see Eq. 3), was reduced by between 0 and 20% in 0.1%-increments (Figure 9). It is assumed that all initial moisture was driven off during torrefaction.

Figure 9. Plot of R^2 vs. correction for initial moisture content of hurds.

The highest R^2 after adjusting for moisture content was 0.8166 at 15.9% moisture (Figure 10). This magnitude of moisture content falls in the range of equilibrium moisture contents (EMC) that Nilsson et al. (2005) found for hemp stalks, and agrees with the values of EMC that Kymäläinen and Pasila (2000) found for coarse green hemp hurds. Adjusted for initial moisture content, the governing equation becomes:

$$L = 0.4523 \times (1 - e^{-0.003015t}) \quad (11)$$

Figure 10. Model corrected for initial moisture content with measured data. Error bars extend one standard deviation in each direction.

To determine the sensitivity of the best-fit model to the assumed long-term final mass conversion time of 48 hours, the 48-hour torrefaction mass loss term, L_{48} , was adjusted by adding between -14 and 46% in increments of 0.25% (Figure 11).

Figure 11. Plot of R^2 vs. final mass loss correction.

The highest R^2 for adjusted final mass loss was 0.7944 at an adjustment to the final mass loss of -13% (Figure 12). This result suggests that the long-term final mass loss is less than the mass loss after 48 hours, which is physically invalid. This result is likely an artifact of the sensitivity analysis being conducted in discrete steps. Specifically, as mentioned previously, the sensitivity analysis conducted herein was performed for the three experimental variables separately. This means that the best-fit solutions obtained during this sensitivity analysis (as summarized in Table 1) provide lumped-term corrections for the various experimental parameters tested herein (i.e., torrefaction time, initial moisture content, and optimal mass conversion torrefaction time).

Further sensitivity analyses for these three variables with data adjustments done in sequence could benefit final analyses of these experimental results. For example, the adjusted data resulting from sensitivity analysis performed on the torrefaction time could

be tested for sensitivity to initial moisture content and so forth. Additionally, it could be that over 48 hours of torrefaction in the fixed-bed torrefaction system, air was able to circulate into the retorts and redox reactions took place. Further analysis of the hemp biochar for chemical composition could also benefit final analyses of these results. Modifying the governing equation for corrected final mass loss gives:

$$L = 0.4088 \times (1 - e^{-0.008718t}) \quad (12)$$

Figure 12. Model corrected for final mass loss and measured data. Error bars extend one standard deviation each direction.

Table 1. First-order reaction kinetics model coefficients.

Model	$L_{48}(\%)$	$k(\text{min}^{-1})$	R^2
Raw data	53.88	0.004682	0.7449
Time corrected	53.88	0.003069	0.8200
Moisture content	45.23	0.003015	0.8166
Final mass loss	40.88	0.008718	0.7944

4.0 CONCLUSIONS

4.1 Models

Four governing equations were developed to model the reaction kinetics of the torrefaction of hemp under isothermal conditions in a fixed-bed torrefaction system at 250 °C in small retorts. Results of the sensitivity analysis on three experimental variables (i.e., torrefaction time, initial moisture content, and optimal mass conversion) showed that the model including an adjustment for the isothermal torrefaction time had the highest R^2 value.

4.2 Future Studies

More advanced analytical instrumentation could be used to reduce error, but the expense of such instrumentation is difficult to justify. Instead, careful experimental design could accomplish similar reduction of error while adding much less to the cost of the study. To eliminate some of the sources of error identified, the following changes should be made:

Exclude Oxygen

Remove oxygen from the retorts by purging with nitrogen before heating begins. Maintain a flow of nitrogen into the retorts sufficient to exclude air from entering for the duration of torrefaction and until the retorts have cooled below temperatures that support thermal degradation.

Measure temperature inside retorts

The correlation between air temperature in the oven and the temperature inside the retorts is not known. Rather than trying to determine this correlation, it would likely be more reliable to measure the retort temperature directly.

Account for moisture content

As a hygroscopic material, the moisture content of hemp hurds fluctuates with relative humidity. Keeping the hurds sealed in a temperature-controlled and air-tight container would reduce the variation in moisture content between trials to negligible levels. A simple oven drying test for moisture content would then be sufficient to account for this variable. Alternately, oven drying each sample prior to weighing and torrefaction would also remove moisture content from the calculation.

4.3 Additional Experimental Data and Analysis

Temperature

Repeating these experiments under a range of temperatures would allow development of relations between kinetic coefficients and temperature, as in Chen and Kuo (2011).

Composition of hurds

Determining the composition of raw hurds, prior to torrefaction, would allow comparisons and correlations of kinetic models with other biomasses of known composition as well as isolated constituents, such as in Chen and Kuo (2011). Analyzing the composition of the hurds after torrefaction would allow a better understanding of the chemical processes involved and development of models with a theoretical basis rather than purely empirical data. Additionally, characterization of the biochars produced after torrefaction as either an alternative fuel or possibly as a soil additive would be helpful.

Composition of gaseous products

Gases evolved during torrefaction represent a substantial fraction of the chemical energy contained in biomass. Collecting these gases for use would be important for maximizing the economic and environmental utility of processing biomass. Collecting and analyzing torrefaction gases would complicate the study, but would yield useful information.

Energy and economic evaluations

For torrefaction to be a useful treatment for hemp hurds, it would need to be both economically and environmentally productive. If the energy required to accomplish the treatment is greater than the increase in energy density it affects, for instance, it would be difficult to justify for biomass energy uses (while the potential as a soil additive may be justified). Likewise, if the economic value of the products does not justify the expense of required energy and apparatus, the process is unlikely to be of practical use.

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