TECHNICAL NOTE: EVALUATION OF A CRUCIBLE FURNACE RETORT FOR LABORATORY TORREFACTIONS OF WOOD CHIPS[1]

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Abstract. Torrefaction is a thermal process that improves biomass performance as a fuel by property enhancements such as decreased moisture uptake and increased carbon density. Most studies to date have used very small amounts of finely ground biomass. This study reports the testing of a crucible furnace retort that was fabricated to produce intermediate quantities of torrefied material and to allow processing of wood chips. Yields ranging from 51 to 96% were impacted to a greater extent by differences in temperature than time of treatment. Although temperature-control issues (gradients, slow heating) were experienced with the crucible furnace retort, this equipment proved to be useful for its intended purpose.

Keywords: Biofuels, biomass, moisture, particle size, pyrolysis, thermal degradation.

INTRODUCTION

Torrefaction is a thermal process by which biomass can be made more amenable to fuel applications. Conditions are typically anaerobic with atmospheric pressure and temperatures ranging from 200 to 300°C. Yields of solid material from torrefaction of woody biomass feedstocks are reported to range between 70 and 90% (Ciolkosz and Wallace 2011). Property enhancements include decreased moisture uptake, greater resistance to microbial degradation, higher carbon density, and increased grindability (Arias et al 2008; Chen and Kuo 2011; Medic et al 2012). Most torrefaction studies to date have used small amounts of finely ground biomass in quartz tube furnaces. Little is known about the processing of large particles (Ciolkosz and Wallace 2011). Muffle furnaces equipped with box-type chambers (retorts) have been used in only a few studies to produce more substantial amounts of torrefied material (Pentananunt et al 1990; Pimchuai et al 2010; Stelte et al 2011). Commercially practical particle sizes (wood chips) have been processed in box-type chambers (Phanphanich and Mani 2011; Singh et al 2013) and in a pilot-scale torrefaction plant (Meng et al 2012). This study reports our efforts to develop equipment to produce intermediate quantities (50-500 g) of torrefied material needed for application assessments (adsorbents, soil amendments) and the ability to accommodate wood chips.

MATERIALS AND METHODS

The retort used in this study was fabricated by Thermal Product Solutions (Williamsport, PA) for a programmable Lindberg Model 5661 crucible furnace with a Watlow EZ-Zone PM (Winona, MN) programmable integral derivative (PID) controller. A basic schematic of the retort is shown in Fig 1. Fitted to the flanged

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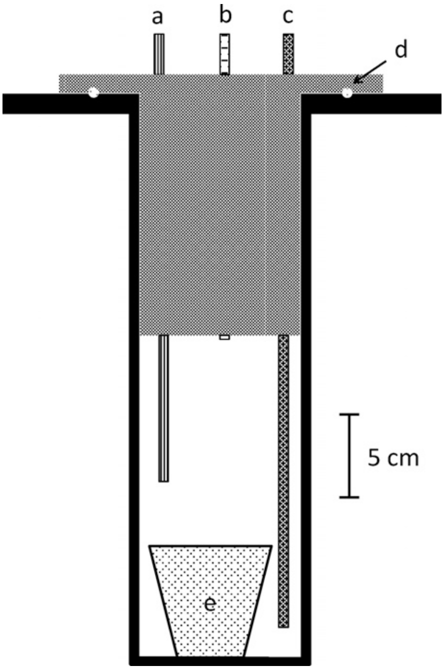

Figure 1. Schematic of crucible furnace retort with positioning of crucible used for sample containment showing (a) fixed internal thermocouple, (b) purge gas inlet, (c) purge gas outlet, (d) O-ring seal, and (e) crucible.

retort chamber was an insulated O-ring-sealed plug-type cover that, when installed, provided an approximate internal volume of 1.6 L. The cover was equipped with inlet and outlet tubes for purging. The flow rate of the purge gas was monitored using a Key Instruments (Trevose, PA) P/N: 60510 glass tube flowmeter. The cover was also equipped with a monitoring thermocouple sheathed in an Inconel 600® protection tube. Temperatures at different positions within the retort were determined by inserting a flexible Inconel 600® overbraided ceramic fiber-insulated thermocouple through the purge gas inlet port. Thermocouple temperatures were measured with an Omega HH501AJK digital thermometer. To assess the time needed for retort cooling, tests were conducted in which the temperature at the bottom of the heated retort (260°C) was monitored with the retort remaining in the furnace or placed in a cooling stand with or without air circulation using a box fan.

Pulp-grade pine wood chips (mostly *Pinus taeda* L.) were obtained from a local chip mill (Winnfield, LA). The moisture content (103 ± 2°C) of the air-dry wood chips was 9.6%. Wood chips (50 g) were transferred to a high-form porcelain crucible (250 mL) that was then placed in the bottom of the retort. The retort cover was installed with specific attention given to the placement of the outlet purge tube to ensure that it was aligned past the side of the crucible (Fig 1). Under a constant purge of laboratory-grade N_2 ($1\ L\cdot min^{-1}$), the retort was heated from room temperature to one of three targeted operating temperatures (230, 260, and 290°C). Total run times ranged from 2 to 4 h including the time (less than 1 h) needed to reach the targeted operating temperature. When the torrefaction run was complete, the retort was removed from the furnace and placed on a stand to cool before stopping the flow of N_2 and removing the sample-containing crucible. Yields were based on the dry weights of both the torrefied product and the starting wood chips. A two-way analysis of variance was conducted using PROC GLM in SAS/STAT 9.3 (SAS Institute, Inc., Cary, NC).

RESULTS AND DISCUSSION

A concern with using a box-type chamber (retort) was very slow heat dissipation occurring after turning off the electrical power to the muffle furnace (Stelte et al 2011); this would allow the torrefaction process to continue, albeit at a decreasing rate as a function of the time needed to cool to a reasonable temperature before sample removal. In other studies using muffle furnaces, hot samples were immediately removed and allowed to openly cool (Pimchuai et al 2010; Phanphanich and Mani 2011), the concern here being the potential for sample oxidation. Thus, the retort used in this study was designed to be lifted from the furnace and placed in a stand for faster cooling while maintaining a continuous purge of N_2. Testing and calibration of the retort are subsequently described; however, given the concerns related to retort cooling, trials with the retort remaining in the furnace

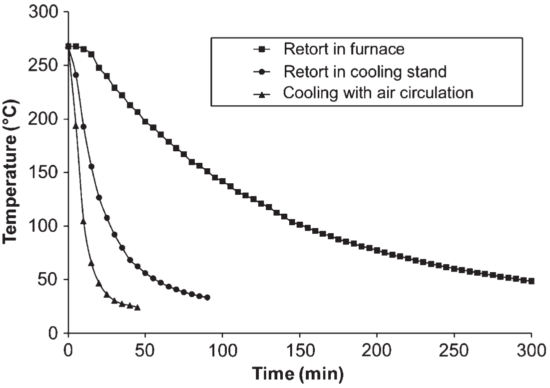

Figure 2. Temperature monitoring of torrefaction zone during cooling of hot retort (260°C target torrefaction temperature) under different conditions.

required 2.5 h to cool the torrefaction zone (lower 100 mm of retort) from 260°C down to 100°C (Fig 2). When the retort was removed and placed in the cooling stand, the time to reach 100°C was decreased to 30 min. With a fan for air circulation, this time was further decreased to 10 min.

Monitoring the measured temperature reading on the PID controller showed it to overshoot the programmed temperature setting by about 20°C before stabilizing at the programmed temperature within 60 min of operation (Fig 3). Initial operations also revealed a temperature gradient within the retort that was greater than anticipated. After temperature stabilization (steady-state operation), the temperature at the fixed internal thermocouple (Fig 1) was approximately 75°C lower than at the furnace thermocouple. With a flexible thermocouple inserted through the retort air inlet port, the temperature at the bottom of the retort was approximately 35°C lower (Fig 3). Thus, a torrefaction experiment with wood chips (265 g) placed directly in the retort afforded a gradient of wood chip colors, from light to dark brown, indicative of different degrees of torrefaction from top to bottom. Placement of an empty high-form porcelain crucible (250 mL) in the retort gave a relatively uniform temperature zone, ultimately limiting the sample size to 50 g. Temperature calibration was performed and gave a linear ($R^2 = 0.99$) plot (not shown). The higher temperature setting programmed into the PID controller to achieve the target torrefaction temperature could then be calculated using the corresponding equation ($t = 0.964c - 28.965$, where t = temperature experienced by sample and c = temperature setting on the PID controller). For example, heating the feedstock to a target temperature of 260°C required the PID controller to be programmed to a temperature of 300°C.

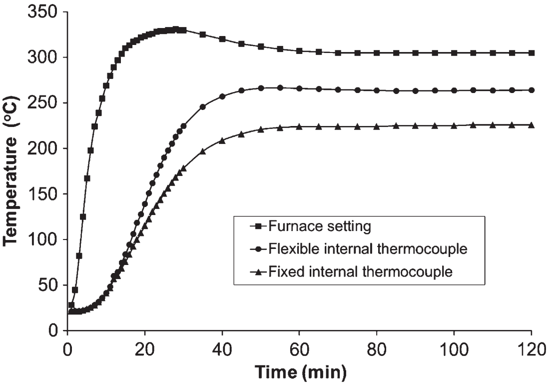

Figure 3. Temperature monitoring of torrefaction zone and at fixed internal thermocouple during initial 2 h of heating cycle (260°C target torrefaction temperature).

Torrefaction of the air-dry (9.6% MC) wood chips, contained in the aforementioned crucible, gave relatively homogeneous product colorations. Torrefied wood chip colors were increasingly darker with increasing time and temperature. The most dramatic difference in color change was with increasing temperature for a given time. This observation was readily apparent in the data with yields being impacted to a greater extent by temperature as opposed to time (Table 1). Using the intermediate temperature of 260°C, all yields were well within the range (70-90%) commonly cited in the literature (Ciolkosz and Wallace 2011). At the lowest temperature (230°C), all yields were greater than 90%, whereas at the highest temperature (290°C), all yields were less than 70%. At 290°C, the yield at 5 h (50.14 ± 0.81%) was the same as that at 4 h of treatment (51.24 ± 1.27%). Torrefaction times longer than 4 h were therefore not pursued. Given the high yield (low thermal degradation) when torrefying

Table 1. Percentage yields of torrefied wood chips for different temperature and time conditions.

	Temperature (°C)		
Time (h)[a]	230	260	290
2	95.64 (0.25)[b]	85.30 (1.88)	67.36 (1.13)
3	92.95 (0.76)	82.01 (1.47)	61.61 (0.45)
4	91.53 (0.39)	78.41 (0.01)	51.24 (1.27)

[a] Torrefaction time includes the time (less than 1 h) needed to reach the target torrefaction temperature.

[b] Standard deviations shown in parentheses; two-way analysis of variance showed all values to be statistically different ($P < 0.0001$) across time and temperature.

for 2 h at 230°C, shorter torrefaction times were not pursued. It is acknowledged that commercial torrefaction systems for the mass production of biochars would undoubtedly be designed for shorter residence times. Equipment temperature-control limitations demonstrated here precluded the pursuit of kinetic studies. Also, condensation that occurred within the plug-type cover precluded the quantitative collection of condensable volatiles for analysis.

Monitoring the fixed internal thermocouple during the torrefactions showed that the temperature lagged behind that obtained during testing with an empty crucible (Fig 4). The temperature of wood chips themselves could not be determined given the current retort design; however, it was unlikely that any temperature deviations were greater than those at the fixed internal thermocouple. Subsequent processing of oven-dry wood chips gave plots that were essentially identical to those with the empty crucible (plots not shown). The previously mentioned temperature deviations at the fixed internal probe were therefore attributed to moisture within the wood chips. Admittedly, it was not anticipated that as little as 5 g of water in 50 g of wood chips could impact the internal temperature of a crucible furnace retort with a volume of 1.6 L, with a mass of 15 kg, and being operated under a continuous purge of dry N_2.

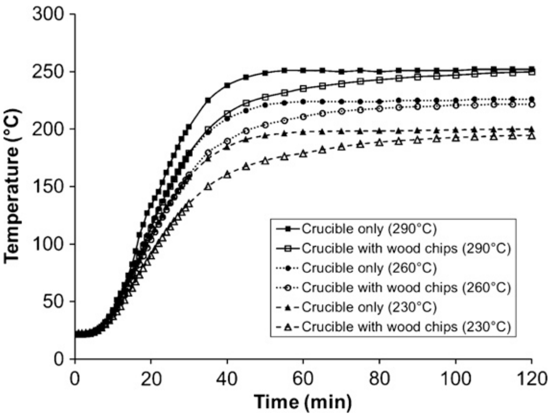

Figure 4. Temperature at fixed internal thermocouple during the first 2 h of heating cycle (to target torrefaction temperature) with empty crucible or crucible containing wood chips.

CONCLUSION

Temperature-control limitations (gradients, slow heating) preclude the use of a crucible furnace retort for kinetic studies; however, this equipment proved to be useful for producing intermediate quantities of experimental torrefied wood samples covering a wide range of yields.

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