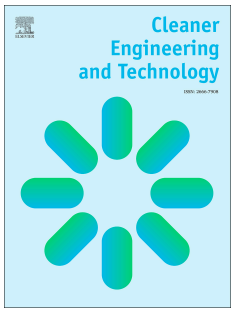

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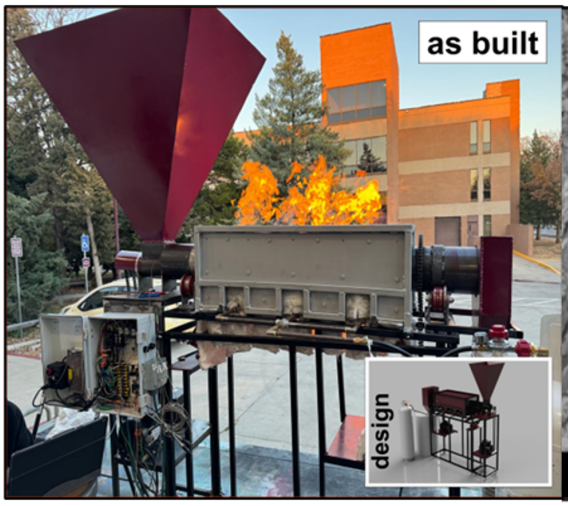

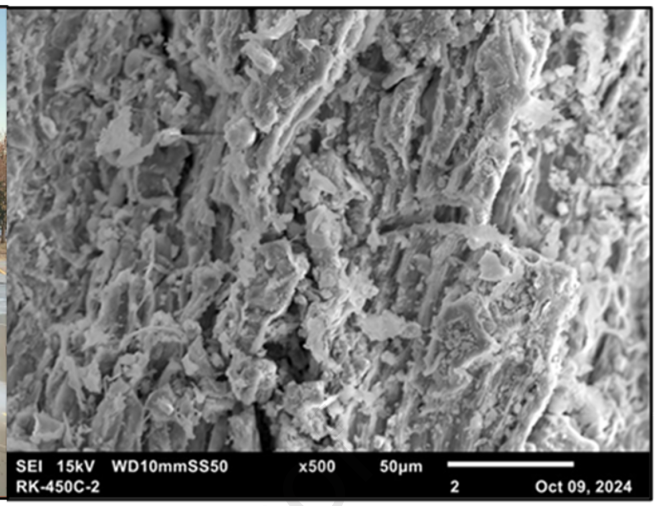

Ground cotton gin waste

Continuous process biochar, 450°C

Lab- and pilot-scale biochar production from cotton gin waste

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ABSTRACT

As much as 2.32×10^6 metric tons of cotton gin waste (CGW) is generated in the U.S. annually. Technologies for utilization of CGW, including improved nutrient cycling via conversion to biochar, are of high interest to the agroindustry seeking answers regarding technoeconomic feasibility. Yet there is generally a gap between what is understood about CGW and similar crop residue biochar made in the lab as opposed to that which might be made at larger scales. The purpose of this work was to examine the quality and quantity of biochar made from CGW, at lab- and pilot-scale, taken directly from a working gin. We considered scaling up of CGW biochar production at 350-600 °C. Specifically, we compared the reactor operation and the quality of biochar produced with a research-grade lab-scale muffle furnace to a less controlled pilot-scale rotary kiln. The average biochar yields were 37.1% ± 6.39% (mean ± sd) for the muffle furnace and 25.8% ± 8.99% for the rotary kiln, and these differences were significant ($p < 0.05$). We attribute many of the differences in yield and quality to unintended air entry into the rotary kiln. Porosity of the biochar was higher for the muffle furnace (0.929) compared to the rotary kiln (0.747). The estimated stability of all biochar was generally high (O/C < 0.2; >1,000-year half-life). The biochar nutrients most extractable by water were S, Mg, B, and K, with extraction fractions ranging from 6-59%. The technoeconomic potential of CGW biochar produced in the pilot-scale reactor was approximately $3,000/t; costs are expected to decrease substantially for a full-scale industrial process if a conservative economy of scale can be harnessed. More studies on increasing biochar production from lab to pilot scale are needed if biochar is to reach its potential for large-scale production and applications. Specifically, studies should focus on the unit cost of production and the consistency of biochar quality, since much of biochar's ultimate value depends on the demonstration of specific properties for each application.

KEYWORDS: Cotton production, crop residue, thermal treatment, biochar properties, technoeconomic analysis, sustainable agriculture, scalability, nutrient availability

1 INTRODUCTION

The U.S. Department of Agriculture Natural Resource Conservation Service (USDA-NRCS, 2006) reported that the eight leading U.S. crops produce more than 500 Mt of residue each year.[1] Cotton, eighth among those crops, generates Mt of residue after harvest. Cotton gin waste (CGW), also referred to as cotton gin trash or cotton stalk, is the residue from ginning, the separation of the fibers from the seeds.[2] The National Agricultural Statistics Service (NASS, 2023) reported that 0.46 M and 14 M bales of pima and upland cotton, respectively, were produced by the 17 southern U.S. states, respectively, generating as much as 2.32 Mt of CGW. The Texas High Plains, a major cotton-producing area, generates approximately 1-1.5 Mt of CGW annually.[3] For cotton gins, CGW disposal and management are inconvenient and expensive. Gin owners continue to seek technologies for value-added uses of CGW. Research has continued to show CGW potential in various applications, such as livestock feed, solid fuel for combustion and generation of gin dryer heat, building material, liquid and gaseous fuel generation (via gasification, anaerobic digestion, and biofuel production from fermentation), biochar for soil amendment, and activated carbon.[2]

Biochar properties have been explored for improving water retention and nutrient availability in soil, and for environmental management. Biochar is a highly porous, aromatic black carbon solid, often with a large surface area. There are several examples of CGW biochar studies for specialized (i.e., relatively small-scale) applications. Ndoun et al. (2023) used CGW pyrolyzed at 700 ºC and walnut shells processed through flash pyrolysis at 800 ºC for removal of pharmaceuticals from aqueous solutions. CGW biochar removed 99% of docusate and 50% of docusate and ibuprofen, respectively.[4] Hernandez-Maglinao et al. (2019) pyrolyzed CGW in a batch reactor at 600, 700 and 800 ºC, then activated with superheated steam to increase the biochar's surface area and pore volume to generate an activated carbon.[5] After pyrolyzing CGW using a fixed bed reactor, Zhang et al. (2016) reported a biochar containing 55.4% C, 2.75% H, 2.29% N, 1.84% S and 24.7% O.[6] These applications represent only a small amount of the CGW available. Larger scales of biochar production are needed to evaluate the potential of CGW biochar for larger (field-level) applications, enabling meaningful extents of CGW biomass management and increasing soil water holding capacity, soil organic carbon (SOC), and microbial communities. To date, there is no published evidence of studies considering the scale up of CGW biochar from academic (well-controlled lab-scale) reactors to pilot-scale or full-scale production.

Only a few studies have explored the possibility of biochar production from agricultural residues at the pilot scale. Veiga et al. (2021) converted from a batch to a continuous flow process for pyrolysis of sugarcane; batch reactors were easier to use but had the disadvantage of longer residence times.[7] Gomez et al. (2016) studied the effects of temperature on process performance for pilot-scale slow pyrolysis of biomass in a semi-continuous electrically-heated reactor at temperatures of 350, 450, and 550 °C; increases in temperature reduced the char production yield.[7, 8]

Selecting a type of reactor is an important aspect of technoeconomic analysis (TEA), especially for less-researched feedstocks like CGW. Temperature, feedstock compatibility, safety, biochar yield, heat source, residence time, energy, labor, and transport costs, and market considerations are among the crucial factors. Struhs et al. (2020) performed TEA for nutrient-rich biochar from cattle manure, measuring sustainability in terms of environmental (chemicals), economic (capital and operating costs), and safety (physical and chemical) measures. They concluded that such a biochar production strategy was sustainable and would stimulate the biochar production industry.[9] The Texas High Plains is home to substantial significant beef and dairy cattle production, suggesting synergies between manure and CGW utilization for biochar.

To date, biochar production at lab-scale with various feedstock types is generally well researched with established knowledge about the influence of pyrolysis conditions on biochar properties. However, there remains an important gap in scaling from lab to pilot production systems, especially for batch to continuous reactor designs at pilot-scale, and for CGW as a feedstock. The objectives for this research, therefore, were to:

(1) Develop a pilot-scale (10-50 kg/h) continuous pyrolysis reactor to produce CGW biochar for soil amendment, setting up a framework for adaptability to full-scale designs (500 kg/h or more).
(2) Characterize and compare the physical and chemical properties of biochar produced in lab- and pilot-scale systems under similar process conditions for residence time, heater set temperature, and loading rate.
(3) Estimate the cost per unit mass of CGW biochar produced at different scales.

2 METHODS

2.1 Cotton gin waste

CGW was used as received from a local gin (Briscoe County, TX) and consisted mostly of lint, seeds, hulls, leaves, sticks and burrs with small amounts of soil and fine dust particles **(Figure 1**). For the lab-scale experiments, the CGW was oven-dried for at least 8 h. For the pilot-scale experiments, the CGW was dried outdoors in the sun due to the large quantity of biomass. The bulk density of CGW varied due to differences in particle size, type, composition, and amount of compaction during biomass handling. Here, the uncompacted bulk density was 126 kg/m^3. The maximum dry bulk density achievable with compaction was 214 kg/m^3. In all pyrolysis experiments, we endeavored to homogenize the CGW as much as possible without resorting to complex processing. The aim was to evaluate the potential for pyrolysis scale up using the feedstock in its current form. Still, we homogenized the CGW by putting it in a large bag and physically shaking it. Being that CGW is a mixture of dry gripping material (fibers, errant seeds, bowl fragments, leaves, and stalks), true homogenization is difficult. The homogenization we achieved certainly did not visual homogenization. Individual components were still easily discerned. The process we used did limit the amount of variation due to the way that we grabbed material from a stockpile that was provided to us by the gin.

It is of interest to know the biomass component breakdown of CGW compared to other feedstock. We did not conduct this testing for this batch of CGW, but we did this on CGW from the same cotton gin (though a different year) as has already been published. The fractional breakdown of CGW from that study is (dry basis) 37.9% hemicellulose, 31.9% cellulose, 26.5% lignin, and 3.63% residual ash. The use of TGA is more approximate compared to extraction-based methods for which we provided results for CGW alongside our TGA-based results in **Table 1**. Our previous work did not separate extractives from other losses found on the TGA. It likely that some of these extractives, being lighter molecular weight compounds, were incorporated into the TGA mass loss we associated with hemicellulose. Thus, our value of hemicellulose (37.9%) likely includes actual hemicellulose + extractives which others found to be 19.0-28.3%. The cellulose we previously obtained is within the range provided by others (16.4-36.5%) and our lignin (26.5%) is slightly outside of the range found by other researchers (17.9-22.8%). Put together, these data show that the general range for hemicellulose, cellulose, and lignin in CGW thus far studied is approximately 14-31%, 16-37%, and 18-23%, respectively. Thus, there is a moderate amount of variation in any given CGW sample which would certainly influence the biochar product ultimately generated.

While we do not know what precisely the breakdown of this CGW is, we can say that it was consistently the same material used in all experiments.

Table 1. Biochemical composition for CGW provided in other studies. All values provided on a dry basis.

study	**McIntosh et al.[10]**		**Jordan et al. [11]**	**Howell et al.[3]**
sample name	CGW1	CGW2	CGW	CGW
extractives	28.1%	26.3%	19.0%	-
hemicellulose	18.5%	13.7%	30.9%	37.9%
cellulose	23.9%	36.5%	16.4%	31.9%
lignin	22.8%	17.9%	20.1%	26.5%
ash	13.1%	9.00%	0.530%	3.63%
other	-	-	13.1%	-
remainder	-6.40%	-3.40%	0.00%	0.00%

2.2 Lab-scale CGW biochar production

A Thermolyne large muffle furnace (model F6020C-33-80, Thermo Fisher Scientific) was used for the lab-scale reactions. The furnace was purged with high purity compressed nitrogen (99.998%) before, during, and after pyrolysis. The furnace had an exhaust opening on top to allow for the release of effluent gases. A 3-in. (7.6 cm) flexible aluminum ducting was connected to the exhaust and passed through a room temperature water bath to cool effluent gases and to prevent condensation of tar inside the fume hood. **Table 1** summarizes the experimental conditions for lab-scale biochar production. Clean ceramic crucibles were loaded with CGW at densities of 126 kg/m^3 and 214 kg/m^3 (**Figure 1)**. After completion of the pyrolysis hold time, the muffle furnace was turned off and allowed to cool slowly under nitrogen flow. The scales are indicated by arrows in the diagram with individual portions of CGW feedstock on the order of 0.1-2 cm, lab-scale production conducted in a heating space on the order of 10-50 cm, and the pilot-scale production heating over a space which is nearly 2 m long.

Table 2. Experimental conditions for lab- and pilot-scale CGW biochar production.

Lab-scale muffle furnace			**Pilot-scale rotary kiln**		
Retention time (min)	Feedstock density (kg/m^3)	Reaction temperature (°C)	Mean residence time (min)	Kiln rotation speed (RPM)	Reactor temperature (°C)
30	126	350	37	20	300
30	214	350	37	20	450
60	126	350	37	20	600
60	214	350	24	20	300
30	126	550	24	20	450
30	214	550	24	20	600
60	126	550	-	-	-
60	214	550	-	-	-

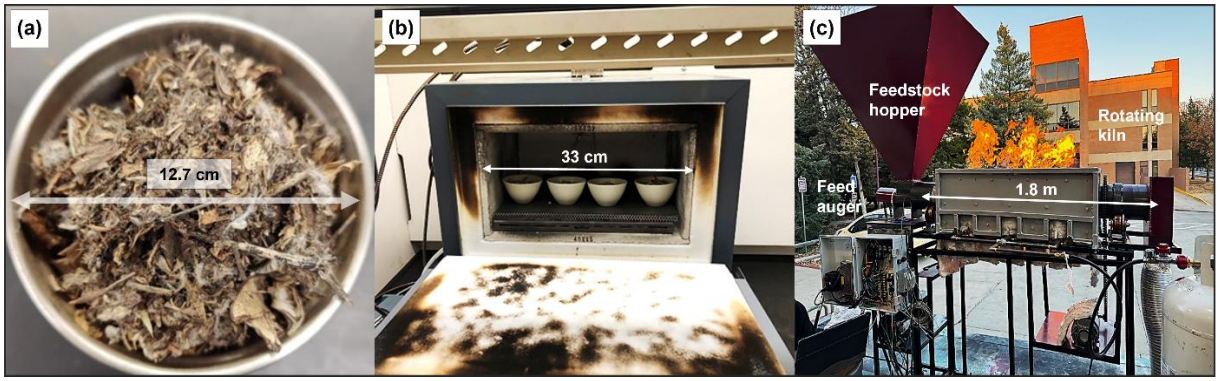


Figure 1. Two reactors were used to make CGW biochar via different processes and scales. (a) Unprocessed CGW under natural dry bulk density in a crucible. (b) Lab-scale production in a muffle furnace (100 g biomass/batch). Multiple crucibles were loaded and left uncovered during pyrolysis under N_2 flow. Samples were composited to represent the run. (c) Continuous, pilot-scale rotating kiln auger reactor heated externally by propane gas on the outside of a heat box (50 kg biomass/h). Arrows in the diagram are meant to show the effect of process scale.

2.3 Pilot-scale continuous feed biochar production process

The rotary kiln reactor (**Figure 1c**) was designed and built by undergraduate engineering students at West Texas A&M University as part of their capstone course. The setup utilized an externally-heated rotating pipe "kiln" as the reaction chamber with a feed auger. Both the auger and the kiln were rotated using a chain and sprocket system. The kiln was made of a 5-ft. (1.5-m) length of 6-in. (0.152-m) diameter carbon steel pipe that can be tilted between 0 and 2°, allowing alteration of the reaction time. The kiln was heated inside a 3-ft. (0.914-m) long chamber using cooking burners to maintain temperature in the reaction zone. The chamber was insulated with 1-in. (0.025-m) of ceramic wool. CGW feedstock was prepared by grinding in a Wiley Mill to size passing 2 mm screen to enable good particle flow through the feed auger. Each of the six experimental conditions was repeated in independent triplicates to ensure reproducibility of results for a total of 18 experiment runs. Samples from each triplicate were combined in equal masses to yield six representative composite samples. Critical differences between the lab-scale and pilot-scale biochar setups were (a) the lab-scale is batch process, and the pilot-scale is continuous process and (b) the lab-scale biochar was in a low O_2 environment which was controlled by high purity N_2 while the pilot-scale reactor did not use any N_2 displacement for outside air. Certainly, a higher quality biochar would likely result from the use of N_2 in the pilot-scale rotary kiln. However, our aim in much of this work was to strive towards economic feasibility which would make adoption by area cotton gin operators or entrepreneurs more likely.

The mean residence time (MRT) varied with the angle of tilt and auger feed motor speed. **Eq 1** was used to relate MRT to operational conditions.[12] Input variables were length of reactor (L), bulk

volumetric feed rate ($\dot{V}$), angle of inclination (θ), angle of repose (β), kiln rotation rate (n), and diameter of kiln (D).

$$MRT = \frac{0.1026L^3}{\dot{V}}\left(\frac{\theta}{\beta}\right)^{1.054}\left(\frac{\dot{V}}{L^3 n}\right)^{0.981}\left(\frac{L}{D}\right)^{1.1} \quad \text{Eq 1}$$

Tilt angles of 1° and 2° were used at a constant rotational speed across all experiments of 20 rpm, resulting in MRT values of 37 and 24 min, respectively (see **Table 2)**.

2.4 Biochar characterization

Yield (Y, %) was determined by dividing the dry mass of the produced biochar (m_b) by the dry mass of the CGW feedstock (m_{cgw}) using **Eq 2**:

$$\boldsymbol{Y} = \left(\frac{\boldsymbol{m_b}}{\boldsymbol{m_{cgw}}}\right) \times \mathbf{100}\% \quad \text{Eq 2}$$

The dry bulk density (ρ_b) was determined by dividing the dry mass of biochar (m_b) by the volume of the porcelain crucible using **Eq 3**:

$$\rho_b = \left(\frac{m_b}{V}\right) \quad \text{Eq 3}$$

Particle density was determined using a Ultrapyc 1200e automated helium pycnometer (Quantachrome, Boyton Beach, FL). Porosity (n) was calculated based on solid volume (V_s), particle density (ρ_p, **Eq 4**), and bulk density (**Eq 5**):

$$\rho_p = \frac{m_b}{V_s} \quad \text{Eq 4}$$

$$n = 1 - \frac{\rho_b}{\rho_p} \quad \text{Eq 5}$$

Proximate and ultimate analysis were conducted by a commercial laboratory (Hazen Research, Golden, CO, USA). Briefly, for moisture, samples were air dried overnight, then oven-dried at 130°C for 8-12 h. For volatile matter, oven-dried biochar was heated in a muffle furnace at 600°C for 3 h. Ash content was determined as the residue after volatile matter determination at 600°C for 3 h. Fixed carbon was determined by difference. Ultimate analysis was conducted as CHNS elemental composition with O determined by difference. Biochar pH was measured in 1:5 biochar/deionized water ratio after 1 h shaking on a reciprocating shaker at room temperature. Samples were allowed to stand for 30 min and then pH measured using a VWR Symphony benchtop meter B40PCID. Electrical conductivity (EC) was measured with a Hanna Combo pH/EC/TDS/temperature ppm tester. Scanning electron microscopy (SEM) was conducted in a

coated and uncoated fashion on a JEOL JSM-6010PLUS/LA instrument. SEM samples were prepared by applying copper tape to a sample stage and pouring the biochar onto the stage. Excess biochar was removed. Coated samples were sputter-coated with 5 nm of gold applied at 1-nm intervals with a 1-min cool-down time between coats.

2.5 Biochar nutrient determination

Total biochar nutrients were analyzed at ServiTech Labs (Amarillo, TX, USA) by acid digestion of biochar and quantification of elements by inductively coupled plasma mass spectroscopy (ICP-MS); nutrient contents (C_{bulk}) were reported on a dry biochar mass basis (m_{bc}). Water-extractable nutrients were measured using a mass ratio of 5:1 deionized water to biochar, shaken for 15 min on a reciprocating shaker, allowed to settle for 1 h, shaken again for 5 min, then centrifuged and filtered with a 1-µm filter paper. The filtrates were analyzed by ICP-MS. **Eq 6** provides nutrient extractability fraction, f^{i}_{extr}, in terms of C^{i}_{extr} (extractable concentration of nutrient *i*) and C^{i}_{bulk} (total concentration of nutrient *i*). The extractable fraction was used as a quantitative estimate of nutrients expected to be readily available when a biochar is used in cropping systems.

$$f^{i}_{extr} = \frac{C^{i}_{extr}}{C^{i}_{bulk}} \qquad \text{Eq 6}$$

2.6 Statistical comparisons

Where the number of conditions and replicates were justified, we did use one-way or two-way ANOVA to find differences in treatment effects for properties measured. The treatments are typically pyrolysis time (two levels), pyrolysis temperature (two or three levels), and biochar production scale (two levels, lab and pilot). For those wanting to see the details of the ANOVA sum of squares (SS) and F statistics, we have provided these in the Supporting Information. Throughout the writing here, we refer only to conditions of statistical significance and p-value thresholds.

3 RESULTS

3.1 Lab-scale biochar production

3.1.1 Yield

Table 3a summarizes the biochar yield, production conditions, ultimate, and proximate analysis for lab- and pilot-scale experiments. For the muffle furnace, biochar yields ranged from 29.9-54.9% (dry mass basis). Higher yields had been expected for lower temperature, shorter residence time, and higher bulk density based on heat transfer. The highest yield (unexpectedly) occurred for 550 °C, 60 min, and 126 kg/m^3. The next highest yield of 48% (as expected) was for 350 °C, 30 min, and 126 kg/m^3. The lower bulk density frequently resulted in higher biochar yields, and this was found to be statistically significant with three-way (time, temperature, ρ_b; $p < 0.05$) but not in one-way (ρ_b; $p = 0.079$) ANOVA. We can say that there is at least a nominal difference in yield based on feedstock bulk density where low density (126 kg/m^3) had a mean yield of 39.1% while high bulk density (214 kg/m^3) had a yield of 35.2%. Lower yield at higher bulk density is surprising, and this may have to do with higher temperature sustained inside the biomass due to its compaction. We also found a statistically significant difference for temperature when high yield ($p < 0.05$, Tukey HSD 95% confidence, $n = 16$ for each temp) where the yield was 42.3 ± 4.15% and 31.9 ± 2.00% at 350°C and 550°C, respectively. (Note these values not shown in **Table 3a** since it shows summary statistics for all experimental runs.)

We previously reported yield results in a muffle furnace for 60 min ranging 32-34% (450 °C) and 33% (600 °C) at an uncompacted bulk density. In the previous study, we did not quantify the exact bulk density, instead, we loaded the mass as naturally as possible, and we did not have a nitrogen source to our muffle furnace as we did in this time.[3] In the current study, we weighed out the material to achieve either 126 or 214 kg/m^3 exactly. The yields from our previous work generally agree with what we obtained this study. We see that small-scale yields can be "tuned" to a degree using temperature, use of nitrogen versus just covering the sample, and packing density of the feedstock. For comparison, Rehrah et al. (2014) produced biochar from pecan shells, peanut shells, switchgrass, and CGW at 300, 500, and 700 °C. They attributed higher biochar yields at lower pyrolysis temperatures to minimal condensation of aliphatic compounds and lower mass losses as CH_4, H_2, and CO. As pyrolysis temperatures increase, more mass is lost due to dehydration of hydroxyl groups and thermal degradation of lignocellulosic structures.[13]

3.1.2 Proximate and ultimate analysis

Biochar C content ranged from 58-63% (% as mass) and generally increased with increasing pyrolysis temperature. Higher temperatures drive off more volatiles and concentrate the carbon.[14, 15] At 550 °C, C content decreased slightly, 63% to 58%, as residence time increased. Bulk density did not have a strong impact on elemental composition. S and N contents were low for all biochar with no apparent correlations to temperature, time, or bulk density. The C contents here were higher than those reported by Zhang et al. (55.0 ± 3.0%) for CGW biochar made at 450 °C for 60 min; H and N proportions were similar.[6] Statistical comparisons (ANOVA, see SI for more details) showed differences in H, O, ash, volatile matter, Fixed C, H:C, and O:C. All differences were due to temperature and not either CGW feedstock bulk density or pyrolysis time.

3.2 Pilot-scale continuous biochar production

3.2.1 Yields and properties

Table 3b shows that yields for the rotary kiln reactor ranged from 12.0-37.3% (mean 25.8%). Decreases in yield were significant in temperature and mean residence time ($p < 0.05$, two-way ANOVA) but not in their interaction ($p = 0.780$). All individual levels of time (24 min, 37 min) and temperature (300, 450, 600 °C) were different based on Tukey HSD ($p < 0.05$). Biochar produced at different temperatures exhibited noticeable differences in physical structure and surface morphology as well; biochar color changed from black to dark gray with increasing pyrolysis temperature (**Figure 2**). All statistical summaries can be found in SI.

3.2.2 Biochar composition

Biochar carbon content decreased, and ash content increased with increasing temperature from 300 to 600 °C. Compared to the muffle furnace reactions, which occurred under an inert gas purge, the rotary kiln operated in the presence of oxygen, and a statistically significant lower yield was detected at the pilot-scale (39.2% lab vs. 25.8% pilot, $p < 0.05$, one-way ANOVA). The oxygen is expected to have caused partial combustion (supported by the grey color at the highest temperature), resulting in biochar yields and properties similar to those of biochar that had experienced higher temperatures under inert conditions.[16] Reaction temperature had the dominant influence on C and O contents compared to MRT, suggesting that the heat transfer in the continuous system was adequate for achieving the desired level of pyrolysis while MRT has a subtler influence. The only statistical element for which temperature made a difference was for H. A closer look at the proximate analysis (provided in **Figure 3**) reveals one way to explain the differences between the lab-scale batch and pilot-scale continuous biochar. The pyrolysis in the lab-scale biochar (3a) is increasing in ash (light gray bar) and fixed C (dark gray bar) with increasing time and temperature at the expense of volatile matter. Yet even the fixed C is not changing much. It is relatively constant at 48.2% ± 3.46% (mean ± sd, CV = 7%) over all the production conditions. We visualized the mean ash ± sd on the plots to help show the distinction between lab (24.7% ± 5.25%) and pilot-scale (42.9% ± 11.1%). The pilot-scale reactor (3b) is reducing in fixed C with increasing time and temperature with the balance being made up with ash. The pilot reactor's fixed C is 30.1% ± 7.29% (CV = 24%, much higher variation). The pyrolysis itself produces a biochar with a similar amount of volatile matter, but the pilot-scale reactor is either combusting the fixed C or combusting feedstock before it can be pyrolyzed due to the presence of oxygen which is likely entrained with the ground CGW.

Table 3. Ultimate and proximate analysis of CGW biochar produced (a) via lab-scale muffle furnace and (b) using a rotary kiln reactor at different temperatures and mean residence times. All yield statistics are reported as mean ± standard deviation based on independent replicates (n = 4 lab scale, n=3 pilot scale). Statistics on all rotary kiln experiments use all replicates together for yield (n =18) and all conditions for all other values (n = 6).

(a) Lab-scale muffle furnace

Conditions			Yield	Proximate (mass %)			Molar ratios	
T (°C)	t (min)	ρ_{bulk} (kg/m^3)	(Y, mass %)	ash	volatile matter	fixed C	O/C	H/C
CGW, raw				9.67%	71.6%	18.7%	0.585	1.32
350	30	126	48.3% ± 1.93%	15.6%	39.2%	45.2%	0.197	0.715
350	30	214	38.0% ± 1.57%	23.5%	33.4%	43.1%	0.137	0.620
350	60	126	42.2% ± 1.94%	21.8%	32.3%	45.9%	0.159	0.692
350	60	214	40.9% ± 0.70%	21.4%	30.7%	47.9%	0.132	0.626
550	30	126	31.0% ± 1.09%	27.4%	19.3%	53.4%	0.056	0.407
550	30	214	32.1% ± 0.40%	25.8%	22.3%	51.9%	0.066	0.399
550	60	126	34.8% ± 0.96%	30.9%	16.7%	52.4%	0.067	0.318
550	60	214	29.9% ± 1.00%	31.5%	18.8%	49.7%	0.079	0.385
All lab-scale			37.1% ± 6.39%	24.6% ± 5.14%	26.8% ± 8.09%	48.6% ± 3.74%	0.112 ± 0.0517	0.520 ± 0.158

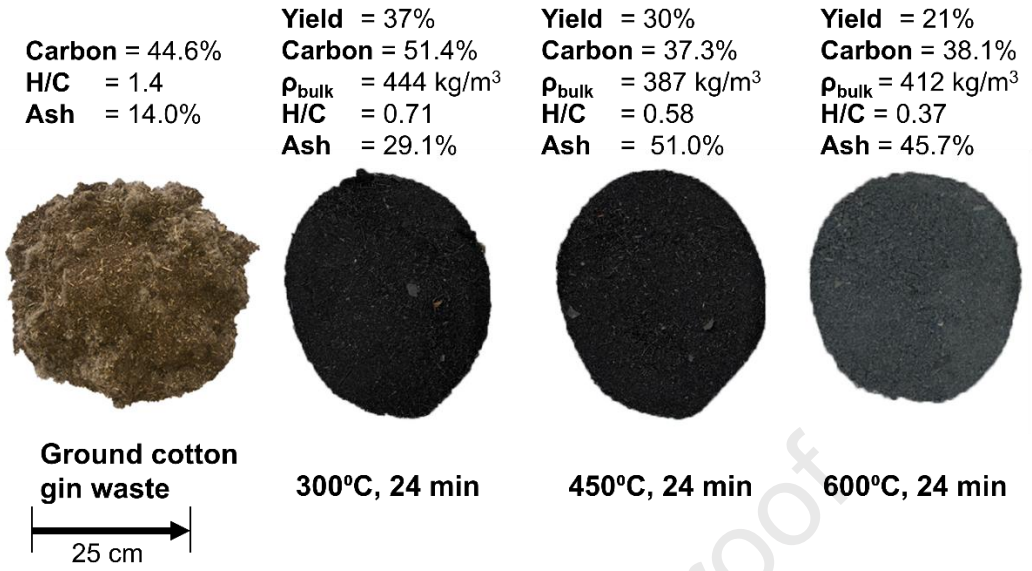
Conditions			Ultimate analysis (mass %)				
T (°C)	t (min)	ρ_{bulk} (kg/m^3)	C	H	N	O	S
CGW, raw			46.7%	5.15%	1.84%	36.4%	0.242%
350	30	126	61.8%	3.68%	2.54%	16.2%	0.221%
350	30	214	59.1%	3.05%	3.18%	10.8%	0.281%
350	60	126	59.4%	3.42%	2.59%	12.6%	0.197%
350	60	214	61.4%	3.20%	2.91%	10.8%	0.264%
550	30	126	63.2%	2.14%	2.30%	4.75%	0.238%
550	30	214	63.8%	2.12%	2.48%	5.61%	0.259%
550	60	126	59.7%	1.58%	2.20%	5.36%	0.221%
550	60	214	58.0%	1.86%	2.24%	6.12%	0.268%
All lab-scale			60.8% ± 2.07%	2.63% ± 0.795%	2.56% ± 0.341%	9.03% ± 4.18%	0.244% ± 0.0290%

(b) Pilot-scale rotary kiln

Conditions		Yield	Proximate (mass %)						Molar ratios			
temp (°C)	MRT (min)	(Y, mass %)	ash		volatile matter		fixed C		O/C		H/C	
CGW, ground			14.0%		67.8%		18.2%		0.573		1.40	
300	24	37.3% ± 5.13%	29.1%		33.3%		37.6%		0.185		0.713	
300	37	31.0% ± 2.65%	31.1%		31.2%		37.6%		0.181		0.610	
450	24	30.0% ± 2.00%	51.0%		22.1%		26.8%		0.141		0.580	
450	37	23.3% ± 4.73%	42.9%		29.0%		28.0%		0.0934		0.543	
600	24	21.3% ± 3.06%	45.6%		22.6%		31.8%		0.248		0.372	
600	37	12.0% ± 5.20%	57.4%		24.1%		18.5%		0.0646		0.428	
All pilot-scale		25.8% ± 8.99%	42.9% ± 11.1%	±	27.1% ± 4.77%	±	30.1% ± 7.29%	±	0.152 ± 0.0668	±	0.541 ± 0.124	±

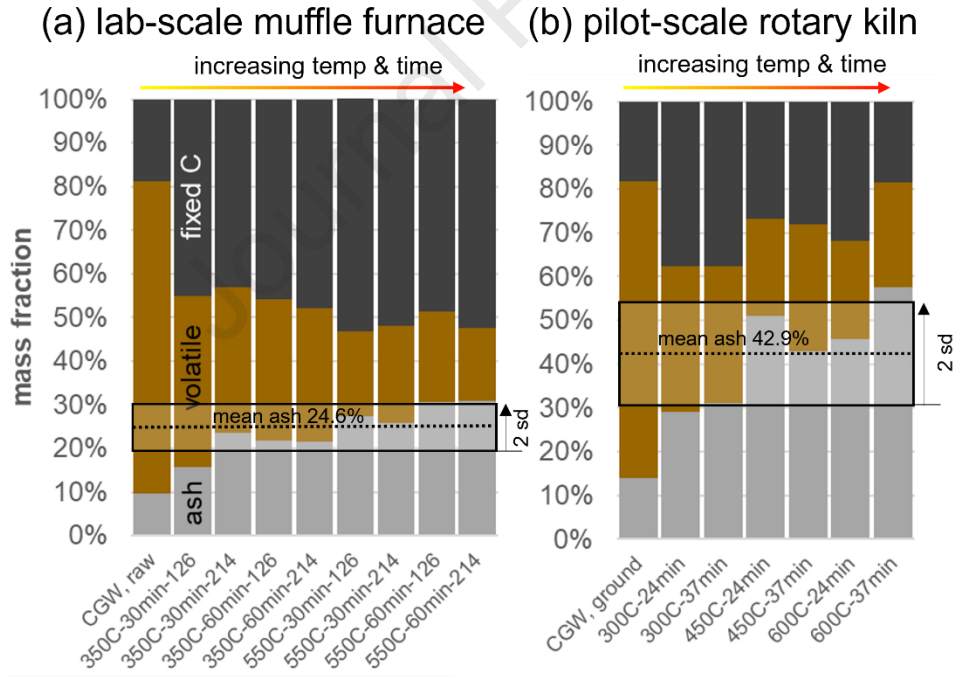
Conditions		Ultimate (mass %)							
temp (°C)	MRT (min)	C	H		N		O	S	
CGW, ground		44.6%	5.18%		2.07%		33.98%	0.221%	
300	24	51.4%	3.05%		3.51%		12.65%	0.262%	
300	37	50.4%	2.56%		3.37%		12.14%	0.356%	
450	24	37.3%	1.80%		2.53%		7.02%	0.333%	
450	37	44.3%	2.00%		3.02%		5.51%	0.433%	
600	24	38.1%	1.18%		2.06%		12.60%	0.369%	
600	37	35.7%	1.27%		2.01%		3.07%	0.558%	
All pilot-scale		42.9% ± 6.89%	1.98% ± 0.729%	±	2.75% ± 0.649%	±	8.83% ± 4.18%	±	0.385% ± 0.101%	±

[a]This particular yield does not follow the trends of all of the others. We have, in some cases, excluded it from statistical comparisons.



299

300 **Figure 2. Appearance and properties of feedstock and biochar made from CGW using pilot-scale**
301 **rotary kiln reactor. Photographs were taken on an indoor laboratory bench and placed inside of a**
302 **drawn circle which was approximately 35 cm in diameter. The scale bar shown is estimated but is**
303 **correct within 10% error. We did not change the lighting in the room during all photographs.**



304

305 **Figure 3. Proximate analysis of (a) lab-scale and (b) pilot-scale biochar. The final number in the series**
306 **of values in (a) on the x-axis refers to the starting bulk density of materials in kg/m^3 (126 or 216).**

3.3 Biochar comparison between scales and methods

3.3.1 pH and EC

Figure 4 shows the mean pH and EC values (n = 3 measurements) of biochar produced in the two reactor systems according to production temperature. Values ranged from 8.6 to 11.2, with the general trend that lower temperature pyrolysis resulted in lower biochar pH. The pH-EC clustering shows (dashed oval on plot) that, at 300-350 °C for reactor set temperatures, the lab-scale and pilot-scale biochar have similar properties. Weber et al. showed that treatment temperature is the most influencing factor on biochar pH and that pH increases with pyrolysis residence time.[17] Biochar EC values ranged from 3.46 mS/cm for the pilot-scale rotary kiln reactor to 2.94 mS/cm for the lab-scale muffle furnace. The highest values for EC (3.9, 4.7 mS/cm) were in the rotary kiln reactor at the highest temperature (600 °C), suggesting the highest concentrating effect of biomass mineral content into the biochar, as supported by the grey color, lower C content, and higher ash content.

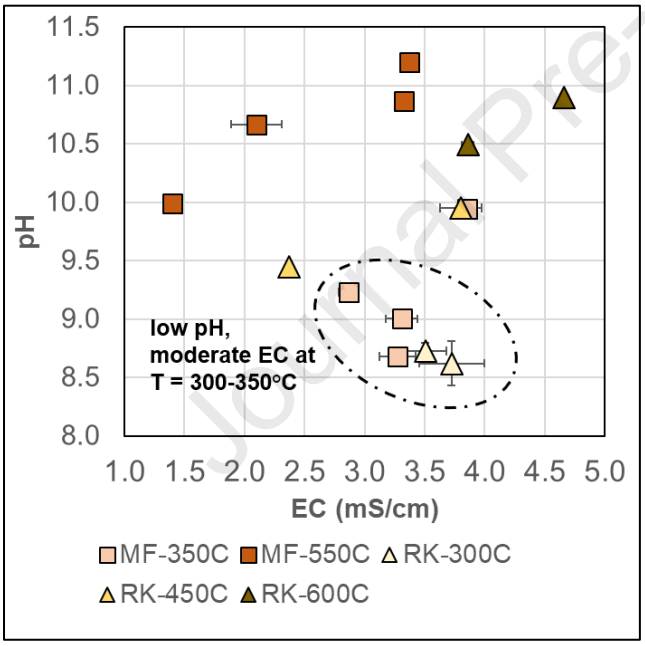


Figure 4. CGW biochar pH and electrical conductivity (EC) comparison for different reactors (MF, lab-scale muffle furnace; RK, pilot-scale rotary kiln) and reactor set temperatures. Values shown are means with error bars as standard deviations (n = 3).

3.3.2 Scanning Electron Microscope (SEM)

Figure 5a shows the fibrous nature of biochar produced using the muffle furnace at 350 °C. Decomposition can be seen, however, a large number of particles still have surface structures similar to the CGW feedstock. Even at this small scale of production, the more-densely packed biomass appears to need > 60 min pyrolysis time when the temperature is lower. **Figure 5b** shows

the biochar produced at 550 °C using the muffle furnace. The biochar particle sizes were mostly smaller than those of the feedstock (provided in the SI). The biochar surface structures only slightly resembled the feedstock: severe degradation appears to have occurred with pyrolysis. **Figure 5c-e** shows the biochar produced at 300, 450, and 600 °C using the rotary kiln reactor at 24 min MRT. At 300 °C, the surface remains like the feedstock, with pyrolysis decomposition largely occurring on the edges of the particles. More deterioration of the surface structures and smaller particles are observable in the biochar made at 450 °C. At 600 °C, pyrolysis is apparent with many of the particles having prominent pores and unrecognizable surfaces from the feedstock.

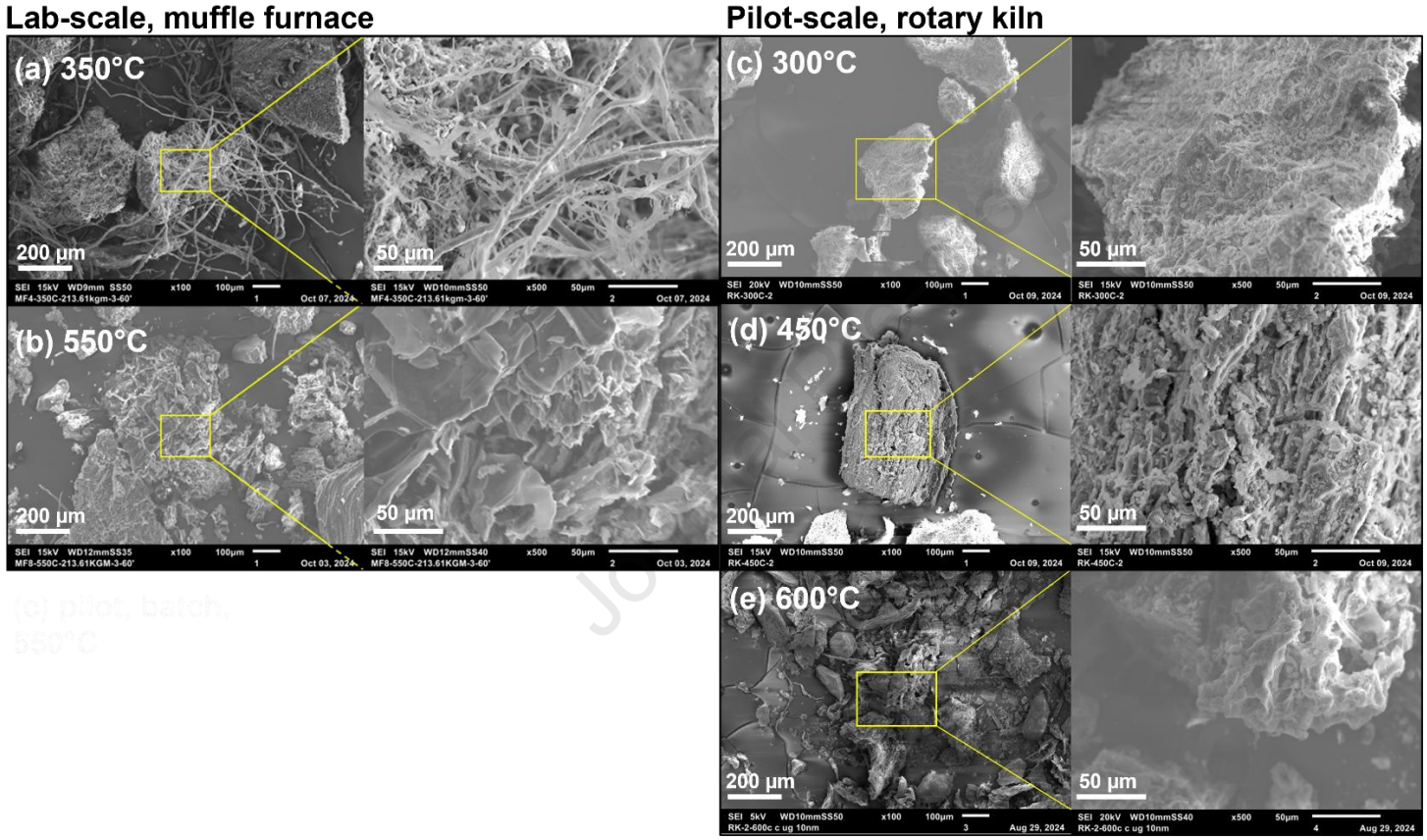


Figure 5. Scanning electron microscope images, far (100x) and close (500x), of biochar: (a) muffle furnace (lab-scale) at 350 °C, 60 min, 214 kg/m^3 feedstock bulk density, (b) muffle furnace (lab-scale) at 550 °C, 60 min, 214 kg/m^3 density, (c) rotary kiln 300 °C, 24 min, (d) rotary kiln 450 °C, 24 min, and (e) rotary kiln 600 °C, 24 min.

3.3.3 Porosity

The lab-scale and pilot-scale porosity values are provided in **Table 4.** Detailed statistical analysis data can be found at tables in SI. A pair-wise Pearson correlation analysis revealed no statistically significant relationship between any of the experimental variables and biochar porosity ($p > 0.05$). We also examined the experimental conditions on porosity according to two-way (pilot-scale; temp, MRT) and three-way (lab-scale: feedstock ρ_b, time, temp) ANOVA. Via that analysis, we also found no statistical significance ($p > 0.05$). A glance at the porosity values does show that the porosity of the lab-scale biochar (0.927 ± 0.0225, mean ± sd) and pilot-scale biochar (0.742 ± 0.0332) were quite different ($p < 0.05$, one-way ANOVA). Looking into the value which make up the porosity calculation, it is two independent measurements, biochar bulk density (ρ_b) and particle density (ρ_p). We performed one-way ANOVA on each of these with the production scale as the factor. We found that particle density was not significant ($p = 0.199$) while bulk density was significant ($p < 0.05$). Therefore, these results indicate that biochar bulk density was much higher in the pilot-scale biochar (0.448 ± 0.0650 kg/m^3) than the lab-scale biochar (0.115 ± 0.0316 kg/m^3) and it is this factor which yielded lower porosity overall in the material. Further analysis using specific surface measurements and microporosity would likely provide more insight and corroboration on these behaviors. Given what we saw with the difference in ash content, we speculate that the ash has relatively low porosity and higher bulk density while the fixed C has high porosity and lower bulk density. Thus, we do not think that the fixed C in the material has a difference in porosity in lab-scale versus pilot-scale. The difference arises, we speculate, to the increased amount of ash. The difference is in the proximate analysis and not in the fundamentals of the pyrolyzed carbon.

Table 4. Bulk density (ρ_b), particle density (ρ_p), and porosity for CGW biochar made using a lab-scale muffle furnace and a pilot-scale rotary kiln reactor.

Production conditions			**Biochar properties**		
T (°C)	Feedstock ρ_b (kg/m^3) [a]	Time (min)	ρ_b (kg/ m^3)	ρ_p (kg/ m^3)	Porosity (n)
Lab-scale muffle furnace					
350	126	30	0.0770	1.42	0.946
350	214	30	0.163	1.38	0.882
350	126	60	0.0840	1.40	0.940
350	214	60	0.117	1.50	0.922
550	126	30	0.0800	1.87	0.957
550	214	30	0.136	1.84	0.926
550	126	60	0.136	1.70	0.920
550	214	60	0.128	1.72	0.925
350	126	30	0.0770	1.42	0.946
		Summary			
		mean	0.115	1.61	0.927
		sd	0.0316	0.203	0.0225
		cv	27.4%	12.7%	2.42%
Pilot-scale rotary kiln					
300	-	24	0.444	1.59	0.721
300	-	37	0.393	1.59	0.753
450	-	24	0.387	1.64	0.764
450	-	37	0.546	1.76	0.690
600	-	24	0.412	1.91	0.784
600	-	37	0.507	1.97	0.742
		Summary			
		mean	0.448	1.74	0.742
		sd	0.0650	0.164	0.0332
		cv	14.5%	9.40%	4.47%

[a] The production bulk density for the rotary kiln is unknown. The feedstock was ground to particles < 2 mm for ease of introduction to feed auger. The feedstock material is rapidly spread into the reaction zone and thinned out along the wall, suggesting that bulk density may not be meaningful for the rotary kiln reactor as an experimental variable.

3.3.4 Biochar total and available nutrient content

Table 5 shows the total and extractable nutrients in the biochar, organized by major, secondary, and micro-nutrients. Two considerations for the use of these biochar in Texas Panhandle soils are that these soils are already alkaline and contain high amounts of calcium carbonate, raising potential concerns for the Ca and alkalinity added with the biochar (**Figure 3**). The biochar all exhibited Na concentrations ranging from 160-380 mg/kg which, combined with the biochar EC, may indicate a risk for soil salinization.

376 **Table 5. Total and water-extractable nutrient contents for CGW biochar made in the lab-scale**
377 **muffle furnace and pilot-scale rotary kiln: (a) major, (b) secondary, and (c) micro-nutrients.**
378 **Contents are expressed on a dry biochar mass basis. MRT, mean residence time; ρb, feedstock bulk**
379 **density.**

380 (a) Major nutrients, mass fraction (total) and mg/kg dry (extractable)

Production conditions				**N**		**P**		**K**	
scale	temp (°C)	ρ_b (kg/m^3)	MRT (min)	total (%)	extractable (mg/kg)	total (%)	extractable (mg/kg)	total (%)	extractable (mg/kg)
lab	350	126	30	1.30	1.95	0.26	1.9	0.94	4,075
lab	350	214	30	1.79	1.95	0.30	1.45	0.93	3,120
lab	350	126	60	1.67	2.15	0.26	0.005	0.90	3,965
lab	350	214	60	1.64	4.35	0.27	0.7	0.93	16,300
lab	550	126	30	1.21	1.20	0.38	6.3	1.10	5,350
lab	550	214	30	1.34	3.55	0.38	3.35	1.11	11,650
lab	550	126	60	1.27	1.20	0.39	2.15	1.08	3,225
lab	550	214	60	1.21	1.60	0.40	4.85	1.23	3,155
pilot	300	126	24	1.53	8.65	0.29	7.2	0.91	3,295
pilot	300	126	37	2.00	11.85	0.32	1.9	1.00	3,200
pilot	450	126	24	1.98	8.45	0.34	2.75	0.93	4,765
pilot	450	126	37	1.97	17.8	0.42	9.3	1.19	5,250
pilot	600	126	24	1.71	13.45	0.51	1.55	1.50	6,600
pilot	600	126	37	1.44	27.45	0.61	0.4	1.65	8,550

381 (b) Secondary nutrients, g/kg dry

Production conditions				**Ca**		**Mg**		**S**		**Na**	
scale	T (°C)	ρ_b (kg/m^3)	MRT (min)	total (g/kg)	extractable (g/kg)	total (g/kg)	extractable (g/kg)	total (g/kg)	extractable (g/kg)	total (g/kg)	extractable (g/kg)
lab	350	126	30	37.0	5.45	4.75	0.935	1.88	0.75	0.16	0.075
lab	350	214	30	43.1	0.56	6.07	0.510	2.19	0.99	0.16	0.0493
lab	350	126	60	38.5	5.25	5.06	0.790	1.80	0.71	0.17	0.056
lab	350	214	60	40.6	1.33	5.72	0.145	2.16	2.18	0.16	0.18
lab	550	126	30	54.5	0.106	6.72	0.150	1.90	0.78	0.22	0.077
lab	550	214	30	54.2	0.055	6.71	0.000	1.90	1.69	0.22	0.084
lab	550	126	60	56.1	0.129	6.49	0.096	1.80	0.93	0.22	0.0349
lab	550	214	60	55.8	0.099	6.86	0.110	1.92	0.75	0.24	0.0297
pilot	300	126	24	45.2	3.03	5.85	0.750	2.51	0.89	0.16	0.055
pilot	300	126	37	48.4	2.92	6.34	1.35	2.83	1.33	0.18	0.0495
pilot	450	126	24	49.8	0.449	6.30	0.41	2.55	1.56	0.17	0.077
pilot	450	126	37	67.2	1.03	7.73	0.81	3.28	2.05	0.24	0.0715
pilot	600	126	24	83.6	0.128	9.75	0.121	3.14	2.16	0.30	0.065
pilot	600	126	37	104	0.515	11.5	0.096	4.39	3.40	0.38	0.086

382
383

(c) Micronutrients, mg/kg dry

Production conditions				**B**		**Cu**		**Fe**		**Mn**		**Zn**	
	T (°C)	ρ_b (kg/m^3)	MRT (min)	total (mg/kg)	extr. (mg/kg)	total (mg/kg)	extr. (mg/kg)	total (mg/kg)	extr. (mg/kg)	total (mg/kg)	extr. (mg/kg)	total (mg/kg)	extr. (mg/kg)
lab	350	126	30	77	1.45	10	0.005	882	0.35	64	0.3	29	0.1
lab	350	214	30	98	8.15	13	BDL	1,390	0.35	77	0.05	34	0.05
lab	350	126	60	80	2.4	11	0.05	1,520	0.1	69	0.1	31	0.1
lab	350	214	60	90	3.75	11	0.05	806	0.8	68	0.1	39	0.05
lab	550	126	30	120	8	12	0.005	1,550	0.005	95	0.005	40	0.005
lab	550	214	30	120	13.3	11	0.2	1,650	0.005	95	0.005	39	0.005
lab	550	126	60	120	5.8	11	0.005	1,790	0.005	100	0.005	40	BDL
lab	550	214	60	110	4.45	12	0.005	2,080	0.005	99	0.005	41	0.005
pilot	300	126	24	96	7.3	16	0.05	2,930	3.2	89	0.35	56	0.2
pilot	300	126	37	110	12.7	23	0.2	5,910	4.25	109	0.45	51	0.15
pilot	450	126	24	110	20.6	21	0.15	5,100	0.6	110	0.05	63	0.05
pilot	450	126	37	150	20.9	33	0.3	10,000	1.1	154	0.1	100	0.05
pilot	600	126	24	180	11.45	55	0.1	23,200	0.005	227	0.005	95	0
pilot	600	126	37	210	10.15	42	0.2	9,880	0.005	198	0.005	308	0.05

BDL, below detection limit

Table 6 shows the fraction of each nutrient that was easily water-extractable, that is, expected to be available in soil pore water on a short time scale. S, B, and the alkaline (K, Na) and alkaline earth (Mg, Ca) elements were the most extractable. From a fertilizer perspective, CGW biochar is not a good source of readily available N, P, or micronutrients (Cu, Mn, Zn, Fe). If a typical field biochar application rate of 10 Mg/ha were used, the available S could meet or exceed the S fertilizer needs; a typical agronomic application rate for S is 0.01 - 6 kg/ha.[18]

Table 6. Fraction of nutrients (mean ± sd) in CGW biochar that is water-extractable, in order of most to least water-extractable.

Element	Pyrolysis Reactor Scale	
	Lab	Pilot
S	56% ± 25%	59% ± 15%
K	63% ± 51%	43% ± 8%
Na	41% ± 31%	30% ± 9%
Mg	6.4% ± 7.4%	9% ± 8%
B	5.5% ± 3.0%	11% ± 5%
Ca	4.2% ± 6.3%	2.6% ± 2.9%
Cu	0.36% ± 0.62%	0.6% ± 0.3%
Mn	0.11% ± 0.16%	0.15% ± 0.19%
P	0.07% ± 0.05%	0.11% ± 0.10%
Zn	0.12% ± 0.14%	0.13% ± 0.15%
N	0.02% ± 0.01%	0.09% ± 0.05%
Fe	0.02% ± 0.03%	0.03% ± 0.05%

3.4 Comparison to other studies

3.4.1 CGW biochar characteristics

Table 7 compares the biochar yields, composition, and molar ratios of CGW produced in two reactor systems in this study to those from other studies on cotton wastes.[6, 19-23] The method of CGW procurement was not always clear. The biomass is likely to vary with cotton cultivar, cultural practices, ginning methods, and storage conditions. The other studies were conducted at the lab scale under tighter control of conditions than could be achieved with the pilot-scale reactor in this study.

Table 7. Reaction conditions, yields, and properties of biochar produced from CGWs using lab-scale reactors.

Study	Feedstock pre-processing	Process: type, time, temperature [c]		Mass fractions (%)[a]						Molar ratio [b]	
			Yield	C	H	N	S	O	Ash	H:C	O:C
Sadaka[21]	Ground	torrefaction, 1 h, 280-400 °C	-	48.1	4.54	1.56	0.25	32.3	13.3	1.1	0.50
Qurat-ul-Ain, et al.[23]	Ground to ~2 mm	fluidized bed gasifier, 0.5 h, 760°C	15%	18.7	0.700	0.660	0.21	0.990	78.7	0.45	0.04
Zhang, et al.[6]	Air-dried, stored in sealed buckets	fixed bed pyrolysis, 1 h, 450 °C	42%	55.4	2.75	2.29	1.84	24.7	13	0.60	0.33
Gao, et al.[20]	Rinsed with tap water, cut to 3-5 cm pieces, dried at 105 °C for 24 h, pulverized by high-speed crusher	fixed bed pyrolysis, 2 h, 250 °C	71%	50.9	5.29	1.29	0.18	33.0	9.3	1.20	0.49
		fixed bed pyrolysis, 2 h, 350 °C	43%	60.3	4.17	1.56	0.24	19.2	14.6	0.83	0.24
		fixed bed pyrolysis, 2 h, 450 °C	45%	64.5	3.27	1.51	0.29	12.7	17.8	0.61	0.15
		fixed bed pyrolysis, 2 h, 550 °C	33%	66.7	2.54	1.33	0.26	10.8	18.4	0.46	0.12
		fixed bed pyrolysis, 2 h, 650 °C	31%	67.8	1.89	1.24	0.30	9.07	19.7	0.33	0.10
Cheng, et al.[19]	Dried in oven at 55 °C for 24 h, cut into 1-3 cm pieces	fixed bed pyrolysis, 2 h, 300 °C	47%	67.8	3.96	1.34	0.29	21.7	5.0	0.70	0.24
		fixed bed pyrolysis, 2 h, 350 °C	44%	67.9	2.87	1.28	0.29	21.2	6.4	0.51	0.23
		fixed bed pyrolysis, 2 h, 400 °C	37%	68.2	2.66	1.37	0.23	20.5	7.0	0.47	0.22
		fixed bed pyrolysis, 2 h, 450 °C	37%	69.8	2.57	1.37	0.32	18.3	7.7	0.44	0.20
		fixed bed pyrolysis, 2 h, 500 °C	33%	69.9	1.84	1.38	0.28	18.3	8.3	0.32	0.20
		fixed bed pyrolysis, 2 h, 550 °C	30%	71.4	1.80	1.26	0.26	16.8	8.6	0.30	0.18
This study	Oven-dried at 105 °C, natural feedstock density	muffle furnace, 1 h, 350 °C	42%	59.4	3.42	2.59	0.197	12.6	21.8	0.69	0.16
		muffle furnace, 1 h, 550 °C	35%	58.0	1.86	2.24	0.268	6.12	30.9	0.39	0.079
	Air-dried, ground to 2 mm	rotary kiln pilot, 0.4 h, 300 °C	37%	51.4	3.05	3.51	0.262	12.7	29.1	0.71	0.19
		rotary kiln pilot, 0.4 h, 450 °C	30%	37.3	1.8	2.53	0.333	7.02	51.0	0.58	0.14
		rotary kiln pilot, 0.4 h, 600 °C	21%	38.1	1.18	2.06	0.369	12.6	45.7	0.37	0.25

[a] All mass fractions comprise fixed carbon (FC) and volatile matter (VM) with the remainder of the balance as ash. All values provided on a dry basis. [b] All mass fractions converted to molar equivalent fractions determination of ratios. - indicates value was not provided in original publication. [c] Temperatures represent reactor set temperatures, not temperatures reached by the biomass.

Biochar yields from the lab-scale muffle furnace compared well to other studies at the lower temperatures (300-450 °C); most ranged from 40-45%. At the higher temperature here, the yield was anomalously high (55%) compared to the other studies (33-37%) at similar temperatures. The rotary kiln reactor had yields that were much lower compared to similar temperatures and residence times of others, which is attributed to the air present in the reactor.

In terms of elemental content, biochar C content in this study was lower than other studies with wide variation, 37-59%. Other studies reported narrower ranges and, at higher temperatures (450-600 °C), were able to achieve C contents nearing and sometimes exceeding 70%. The biochar ash content had wide variation and was higher than in other studies at >20%. Ash content may provide some of the nutrients needed in biochar soil amendment. The only other study reporting ash content exceeding 20% was that by Qurat-ul-Ain et al., which used gasification (intentional partial combustion) in a fluidized bed at a much higher reactor set temperature (760 °C).[23]

Figure 4 shows O/C and H/C ratios of biochar produced with the muffle furnace and continuous reactors. Molar O/C and H/C ratios have been used to describe biochar stability in soil: O/C ratios < 0.2 are considered the most stable with a half-life exceeding 1000 years, O/C ratios of 0.2 – 0.6 are considered intermediate with a half-life of 100-1,000 years, and O/C ratios > 0.6 are considered relatively unstable with a half-life of about 100 years.[24] Biochar with H/C ratios < 0.7 are considered thermochemically altered relative to the feedstock.[25] All biochar produced with the muffle furnace had O/C ratios lower than 0.2 and therefore can be classified as stable (orange circles in Figure 2). Most of the rotary kiln reactor biochar with O/C < 0.2, except for one at 600 °C and 24 min MRT, which may have had slightly insufficient time in the reactor to be "fully converted". The linear regression relating H/C and O/C is quite strong ($p < 0.01$, $R^2 = 0.88$) and indicates a pathway of pyrolysis from CGW feedstock (H/C-O/C, 1.3-0.58) to the most-converted muffle furnace biochar (H/C = 0.4, O/C = 0.08). The fact that many other studies do not fall on this line may be indicative of different pre-processing techniques or differences in cotton ginning practices than what we had available to us. Linear regressions of van Krevelen diagrams have been used by others to relate the pathway of a feedstock towards a graphite-like region and to illustrate the strong effects of temperature on the degree of pyrolysis.[26-28] We examined three studies in detail, two which involved what authors called "cotton stalk" and one of *Sida hermaphrodita*, a forb used for alternative energy solid fuel. The slope which we obtained (1.76) is very close to that of Gao et al. (2.07, cotton stalk) and Szwaja et al. (1.57, *Sida hermaphrodita*) but very different from Cheng et al. (5.93, cotton stalk).[19, 20, 27] It is difficult to know the carbonization pathway that CGW should follow based on the comparison studies we found because frequently scant information is provided

on the exact form of the CGW used. The processing information is provided, but it is not entirely clear if the harvesting and ginning process is common enough across multiple studies to make an effective comparison. More details on these comparison H:C vs. O:C regressions are found in SI.

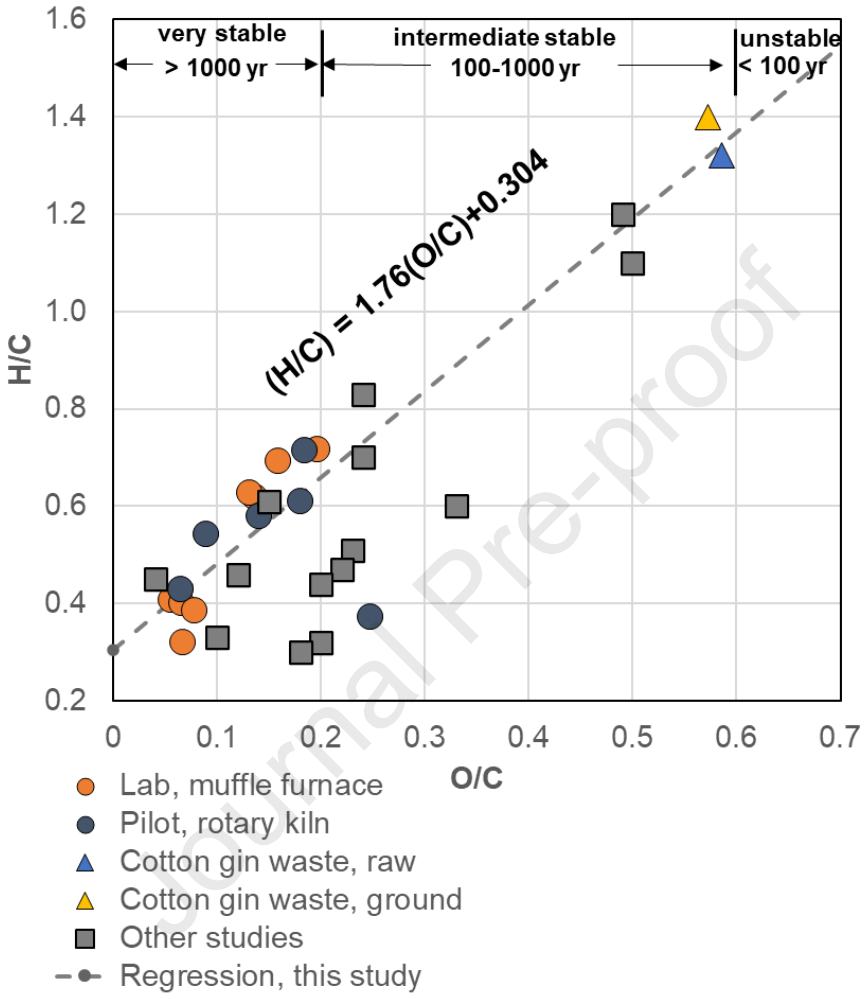


Figure 6. van Krevelen plot for CGW biochar produced using a lab-scale muffle furnace and pilot-scale rotary kiln reactor. Data from other studies are summarized in **Table 7**. The regression shown (H/C = 1.76*O/C + 0.304) is comprised of data from this study and is significant at $p < 0.01$. The regression shows a pyrolysis transition towards increasing carbonization which, at times, is different in lab-scale and pilot-scale reactors in this study as compared to others who have made biochar from CGW.

3.4.2 Examinations into pilot-scale production of biochar

A Web of Science search was conducted on 16 December 2024 related to the transition from lab to pilot to full-scale biochar production. Two search strings (ALL = “biochar” AND ALL = “reactor” AND ALL = “pilot”) and (ALL = “biochar” AND ALL = “reactor design”) returned 124 and 42

453 records, respectively. Of the returned records, 11 were selected for their relevance to scalability, as
454 summarized in **Table 8**. Most of these studies used feedstocks other than cotton gin trash.

455 **Table 8.** Summary of studies on biochar production at the pilot scale

Reference; Reactors; Feedstock	Pilot-scale feed rate	Typical yield	Intended application	Findings
Comparison studies				
Babler et al.[29]; rotary kiln, indirect heat; wood chips (spruce)	70-90 kg/h	31%	component in gasification biorefinery	Heat inefficiency is 30% as limited by heat transfer between reactor walls and feedstock.
Crespo-Barreiro et al.[30]; semi-continuous kiln; olive tree pruning	15 kg/h	-	soil amendment	Challenges in temperature control created biochar which was higher in pH and had phytotoxic effects compared to the lab-scale.
del Pozo et al.[31]; auger; coffee silverskin, grape pomace, olive mill waste	15 kg/h	32-41%	fuel, compost, soil, nutraceutical	Differences in scaling produced more differences in pyrolysis condensed liquids compared to biochar. Greatest biochar differences seen in olive mill waste due to its heterogeneity.
Gomez et al.[8]; semi-continuous electrically-heated tube; Olive stone, almond shell, pine wood, olive-tree pruning	5-10 kg/h	19-41%	soil amendment, sustainability of agriculture-bioenergy production	15-min residence time in the pilot plant allowed for self-sustaining heating. Using the feedstock "as received" lowered energy and logistics for pre-processing, but increased variation in bulk density and particle size, making biochar quality and yield less consistent.
Haryati et al.[32]; fixed bed; palm kernel shell	20 kg/batch	33-52%	soil amendment	N and S content was low in the biochar due to low protein content in non-reproductive plant components.
Kakku et al.[33]; auger; cotton stalk (CS), mustard husk (MH)	1-10 kg/h	CS: 17-51% MH: 20-47%	bio-oils for chemicals, biochar for soil nutrients	Both types of feedstocks contained high amounts of micronutrients which could replace some need for synthetic fertilizers if used in soils.
Nath et al.[34]; fixed bed; wheat straw and wheat straw mixture pellets	2 kg/batch	24-34%	biochar and syngas for on-farm applications	Fixed bed pyrolysis reactor was chosen for simplicity and expectation of higher biomass conversion. Solid product yield was highest for 300 °C; gas yield was highest for 600 °C.
Polin et al.[35]; fluidized bed fast pyrolysis; corn stover	8-22 kg/h	20-22%	biofuels and bio-based chemicals	Autothermal processes allowed for an increase in feedstock throughput. Fast pyrolysis was optimized for bio-oil yield. Oxidation of some biochar carbon reduced slag buildup.
Rosas et al.[36]; auger; vineyard woody residue	55 kg/h	26.7%	carbon sequestration, soil amendment, industry circularity	Higher temperature (728 °C) in mobile autothermal auger reactor increased fixed C content and biochar stability compared to the fixed bed reactor (550 °C). Autothermal operation was considered more feasible due to the energy surplus.
Semaan et al.[37]; auger; demolition wood, sawdust, pelletized refuse-derived fuel, waste tires, grape pomace	4-6 kg/h	-	agronomic, water treatment, construction catalysis, fuel, gas treatment	Syngas production is easier than biochar production because syngas has ready use for fuel. Biochar at pilot scale must have a clear connection between properties and local potential uses.

Reference; Reactors; Feedstock	Pilot-scale feed rate	Typical yield	Intended application	Findings
Veiga et al.[7]; lab-scale batch kiln, pilot-scale continuous rotary kiln; sugarcane bagasse	500 g/h	-	valorization of agricultural waste	Rotary kiln continuous biochar is suitable for scale-up when used under N_2 gas flow. Rotary kiln biochar is structurally different than batch-process biochar.
This study				
pilot-scale rotary kiln; cotton gin waste	50 kg/h	21-37%	soil amendment, cotton production circularity	Even a well-sealed rotary kiln reactor admits enough air to cause oxidation reactions during pyrolysis. Pilot-scale reactors need valve control or active flow of inert gas to prevent oxidation.

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"Pilot scale" was defined by the biomass feedstock processing rate rather than the size of the reactor. Studies that self-identified as "pilot" reported feed rates ranging from 0.5-90 kg/h for continuous reactors and 2-20 kg/batch for batch reactors. In general, authors did not attempt to justify or explain their classification of their reactor as a pilot. In this study, "pilot-scale" was considered to be an amount that is clearly too large to be a lab scale (typically 5-100 g/batch) but not large enough to be able to process the feedstock with sufficient control or throughput for commercial application. Here, the rotary kiln feed rate was 40 kg/h.

The most obvious application for biochar in the Texas Panhandle region is for soil amendment given the amount of land used for cropping and ranching. In comparison studies, use of biochar as a soil amendment for cropping was often considered, but also of interest were fuels, biorefining, nutraceuticals, carbon sequestration, and general valorization. The use of pilot scale reactors often seemed to be for the purposes of answering the general question, "*Can we make biochar at a meaningful scale, and what kinds of resources will be involved in doing so?*"

The studies reveal some valuable lessons about type and configuration of pyrolysis reactor. Most notable were the need to evaluate the energy balance of the reactor, the challenges of knowing and controlling the reactor temperature, and the effects of feedstock heterogeneity on the viability of the process.[8, 29-31, 36] Another class of lessons concerned the biochar product itself. These studies examined the possibility of nutrients in biochar that are available for cropping systems but also the possibility of toxicity.[30, 32, 33, 38] Other studies considered co-products, such as syngas or bio-oil, that have substantial impacts on process economics.[31, 34, 35, 37] Only one study specifically examined lab-scale and pilot-scale studies together to look at broader questions of scalability.[7] The difficulties in optimizing a pilot-scale reactor and producing biochar that are similar in yield and quality to biochar from lab-scale processes, suggest that more of these scalability examination studies are needed in order to advance biochar production and applications generally, especially for less typical feedstocks like CGWs.

4 DISCUSSION

4.1 Practical considerations for continuous and batch reactors.

While the batch reactor was able to handle unground CGW in a static mode, the rotary kiln required that the CGW feedstock be ground to enable consistent material movement and heat transfer. This comparison is important to understand how reactor design and operational techniques influence key biochar properties such as yield, porosity, and nutrient content. Such analysis provides insight into the trade-offs between operational efficiency and biochar quality and, therefore, guideposts for selecting reactor type. Biochar yield from the continuous rotary kiln reactor was generally low (25.8% ± 0.089) with a high ash content from partial feedstock combustion. Kakku et al. (2023) achieved a CGW biochar yield at similar conditions in an auger reactor (400 °C, 90 min) of 51.1% with 23.4% ash, whereas the yield here (450°C, 37 min) was 23% with 42.9% ash.[33] This suggests that the addition of an inert gas stream should be considered to increase biochar yield and quality, or that additional work might be done to prevent outside air from entering the reactor while still allowing biomass to enter.

Compared with continuous reactors, batch reactors are relatively simple to design and easy to construct for small-scale operations. Batch systems are, however, limited by the need for downtime during cooling, loading, and reloading. The steady-state operation of the rotary kiln reactor minimizes downtime—if the operational complexities and feedstock properties can be properly controlled to enable a steady state. The continuous system has greater potential for automation and process optimization and, therefore, is a more promising design for large-scale biochar production.

4.2 Techno-economic considerations

The estimated cost of biochar at the pilot-scale in the rotary kiln reactor was $2.93/kg. These costs include actual costs from study experiments, including reactor construction, land cost, heating, feedstock, repairs, and labor for a 25-year lifespan of the reactors. Scaling up from 12 to 60 kg/h increases production throughput (lab to pilot scale). Energy requirements and costs change significantly with pilot scale when propane gas can be used instead of electricity. A summary of production costs at different scales is shown in **Table 9**.

Table 9. CGW biochar production economic analysis at pilot and industrial scales. An economy of scale factor of m = 0.6 was used to determine the total costs at the industrial scale. This factor was not used for fixed and variable costs.

Factor	Unit	Pilot	Industrial
production specifics			
time	years	1	1
feedstock[1]	t	76.8	384
biochar	t	23	115
yield[2]	kg biochar/ kg feed	30%	30%
production rate, biochar	kg/h	12	60
reactor volume	m^3	0.0167	0.0463
scale factor	-	1	5
energy source	-	propane	process syngas
annual costs			
fixed costs	$/y	$485	-
variable costs	$/y	$66,995	-
total costs	$/y	$67,480	$177,239
unit costs, biochar			
unit cost	$/kg	$2.93	$1.54
unit cost	$/t	$2,929	$1,539

[1]Feedstock is assumed to be CGW at a typical natural packing density (uncompacted). [2]Yields were chosen to be at ~middle of the range determined experimentally.

Unit production costs are expected to decrease with increasing scale due to economies of scale and improved process optimization. Heating and labor were calculated as 29% and 57% of the total cost, respectively. While the production yields may remain similar at pilot and large scale, process automation and less expensive fuels will be critical in lowering costs. Unit costs at industrial scale were estimated to be 47% less using scale factor of m = 0.6 as suggested by Berthouex (**Eq 7)**.[39] C_i and C_p are the costs of production ($/y), while Q_i and Q_p are the capacity in kg/year, at the industrial and pilot scales, respectively.

$$\frac{C_i}{C_p} = \left(\frac{Q_i}{Q_p} \right)^m \qquad \text{Eq 7}$$

Further engineering for process design could reach a value of m = 0.5, providing a greater cost reduction of 55%. A recent review of costs for biochar produced from various (predominantly) wood and crop residues in the U.S. were in the range of $200-$5,000/t,[40] suggesting that the $1,539-$2,929/t estimates for CGW are reasonable. Future biochar research and value estimates are expected to consider both carbon quality and nutrient availability in the context of soil health, agronomy, and carbon sequestration—factors that are more nebulous yet important for assessing

527 long-term process feasibility. For CGW specifically, biochar yield and quality vary with the level
528 of compaction. Further technoeconomic analysis needs to account for the increased preprocessing
529 costs and reduced transportation costs that are expected with different options for feedstock
530 densification.

531

5 CONCLUSION

In the Texas High Plains and similar cotton growing regions, harvesting of cotton produces large quantities of CGW. There are no current examples of large-scale production of biochar from CGW. This study characterized the scalability of CGW biochar production based on biochar properties and concluded the following:

(1) The heterogeneous nature of CGW feedstock creates challenges for making the same biochar at the large scale as was made at the small scale. Some preprocessing of feedstock, such as particle size reduction and/or densification, is likely required.
(2) A well-designed and operated large-scale biochar production process should produce CGW biochar at yields on the order of 30-45% for pyrolysis temperatures in the range of 350-600 °C.
(3) A continuous feed pyrolysis process for CGW is possible but will need greater optimization than was achievable in this study. While heat transfer between a hot rotating kiln and the biomass was good, use of some inert gas or recirculation of flue gas may be required to reduce oxygen in the reactor atmosphere to avoid excessive ash and diminished biochar quality.
(4) The cost of cotton gin trash biochar estimated from this study is high ($2,930/t) and is in line with cost estimates reported elsewhere. Even with the expected reduction in costs with increases in scale, the use of pyrolysis gases in recycling or as a heat source is probably necessary. Other revenue streams, such as tipping fees or carbon credits, should be considered.
(5) Cotton gin trash biochar varies widely in terms of electrical conductivity, pH, H/C ratio, O/C ratio, and bulk density depending on pyrolysis method, time, and temperature. The O/C ratio of biochar in this study indicates stability of the biochar carbon for soil amendment applications. The other biochar properties should be aligned with the local soil quality needs for crop production or ranching.
(6) The density and water extractable availability of nutrients in the CGW biochar did not vary substantially with production scale. The highest nutrient extractability was found for S, K, Mg, and B. When these nutrients are deficient in each soil, the application of CGW biochar may provide a consequential enough quantity of these nutrients to avoid some input costs for farmers and ranchers.

There are still few examples of studies on the direct comparison of scale between biochar production methods. Such studies are needed to bridge the gap between promising results for lab-scale batch production and the challenges with control and consistency at the larger scale, especially for agricultural residues like CGW.

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Lab- and pilot-scale biochar production from cotton gin waste

Highlights

- Lab scale yields averaged 37.1%, while pilot continuous rotary kiln was lower at 25.8%
- Stability of biochar high regardless of scale with projected half-lives > 1,000 years
- Even heating was achieved in pilot rotary kiln, but an inert gas needed to avoid excessive ash
- Cotton gin waste biochar showed good water extraction fraction for nutrients S, Mg, B, K
- Feedstock will, in practice, likely require pre-processing for process upscaling
- Pilot biochar cost ~$3,000/t with lower costs achievable at industrial scale

Declaration of interests

☐ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☒ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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