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Research paper



Self-Purging Pyrolysis of Sugarcane Bagasse biomass to disordered microporous Biochar production

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Abstract

In this study, biochar was produced from sugarcane bagasse in a modified muffle furnace without using a purging gas at different temperatures; 400°C to 700°C with increments of 50°C at 25°C/min heating rate. Biochar yields were significantly influenced by the devolatization of biomass during the pyrolysis and were inversely proportional to the temperature. The holding time was also found to have a significant effect on biochar yields. The maximum biochar yields from sugarcane bagasse, at 10, 20 and 30 minutes holding times, were 27.11, 26.35, and 28.48%, respectively. The highest biochar yield at different holding times was obtained at a temperature of 400°C. The physicochemical properties of biomass and biochar were analysed. The results obtained from characterization showed that SCBC30 showed the highest BET surface area of 348.12 m²/g. From the FTIR results, SCBC10 showed fewer peaks, which indicated a smaller presence of functional groups than SCB-BC20 and SCB-BC30 due to an incomplete transformation of raw SCB wastes to biochar.

Keywords: Biochars pyrolysis; sugarcane bagasse; self-purging; agriculture residues

1. Introduction

Over the last few years, biochar and activated carbon derived from sugarcane bagasse residue have been extensively studied [1, 2]. In 2010, Inyang and co-workers produced biochar derived from sugarcane bagasse by pyrolysis at 600°C in a nitrogen environment [3]. However, the proposed method only produced 18% (by weight) biochar from the digested residue, 23% (by weight) from raw bagasse and 14.07 m²/g BET surface area in the presence of nitrogen gas. Furthermore, in 2017, Varma reported on the pyrolysis of sugarcane bagasse in a semi batch reactor with char yield decreases with N² flow rate [2]. The optimal proposed method produced 33.63% (by weight) biochar with 10.85m²/g BET surface area.

The quantity and quality of pyrolysis products such as biochar are significantly influence by operating parameters. None of the reports on the self-purging pyrolysis of sugarcane bagasse are available in the literature. Besides, to date, very few studies on pyrolysis without the use of a purging gas have been conducted [4, 5]. In order to sweep the volatiles and pyrolysis gas, the purging gas is needed as well as to decrease the vapour residence time which can avoid secondary char to form. There's a conflict has been reported that an increase in purging gas flow rate had no significant effect on biochar yield [6, 7] and contradictly few researchers claimed that the purging gas decreased the biochar yield [2, 8]. In particular, when the nitrogen flow rate reaches 100 cm³/min, the biochar yield nearly becomes constant [9]. Similar results of no significant change in biochar yield, where nitrogen flow rate reaches a certain value, were also reported for the fast pyrolysis of corncob [10]. In this work, the physiochemical characteristics of biomass from sugarcane bagasse and their biochars were studied, the effects of

different operating parameters (temperature and holding time) of a self-purging pyrolysis on biochar yield were also investigated. Temperature range and holding time were set to 400–700°C, and 10–30 min, respectively. Meanwhile, the heating rate of 25°C/min was kept constant.

2. Materials and methods

2.1 Sample preparation

The SCB was sun-dried before being thoroughly washed with distilled water. The bagasse was then oven-dried for two days at 60 °C. Next, it was further crushed in a home blender to obtain powdered form (Raw SCB). A modified programmable digital muffle furnace (WiseTherm, FP-03, 1000 0C, 3 Lit.) was used to synthesis biochars without a purging gas by manipulating the heating time (10, 20 and 30 minutes) and temperature (400°C to 700°C). The produced biochar was then washed with distilled water until the pH became neutral. Finally, the black residue was sieved to obtain SCB with an average particle size of less than 150 mm. This was then stored in an air tight plastic container. The samples were characterized by TGA, XRD, SEM, BET and FT-IR. Biochar yields were determined as the ratio of the produced biochar weight to the dry weight of SCB subjected to pyrolysis (as shown in Eq. 1):

Biochar yield (%) = $(W_2/W_1) \times 100;$

where W_1 is the dry weight of the SCB sample prior to pyrolysis and W_2 is the biochar weight.



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3. Results and discussion

3.1 Thermal analysis of raw SCB

The TGA curve of raw SCB showed 82% weight loss up to 800°C (Figure 1(a)). Then there was a sharp weight loss up to 400°C due to a loss of the organics and volatile materials. There was negligible weight loss from 400°C upwards; which is attributed to the oxidation of residual carbon. Thermal decomposition of the organic components began at a temperature of around 300°C in air.

3.2 SCB BC Yields

The biochar yields significantly decreases when the pyrolysis temperature increased; except for 10 minutes holding time (Figure 1(b)). The yield produced at 10 minutes holding time was inconsistent, as it decreased until 550°C and then increased at 600°C. However, it decreased again when the temperature reached 700°C. The most rapid decrease of yield occurred between 400 and 550°C. A relatively stable yield of BC was maintained above 600°C. However, at 700°C, a slightly lower yield was observed at 20 and 30 minutes holding times. This phenomenon can be explained by the gasification of chars at temperatures above 700°C.

The reduction of biochar yield is possibly due to further removal of volatile substances through opening of the closed pores. It is therefore expected that beyond 800°C, yield difference will either level off or become insignificant.



Figure 1: (a) TGA curve of raw SCB and (b) Effect of pyrolysis temperature and holding times on biochar yields



Figure 2: (a) and (b). (a) FT-IR spectras of SCBC10, SCBC20, and SCBC30, (b) XRD diffractometer of raw SCB and SCB BC produced at different holding times

3.3 Physical characterization of SCB BC

 Table 1: Mean values of biochars BET surface area at different carbonization temperatures.

As the SCB BC produced at 650°C showed little difference at 3 holding times, it was selected for further analyse. SCBC10, SCBC20, and SCBC30 were denoted as biochar produced at 650°C at 10, 20 and 30 minutes holding time, respectively. Table 1 presents the BET surface area for all biochars produced. The results presented in Table 1 indicate that for all samples examined, the increase of pyrolysis time resulted in an increase of biochar surface area. The devolatization of feedstock developed porosity in the biochars, resulting in particles with an essentially micromacropore structure.

tion temperatures.	
Sample	BET surface area [m ² /g]
SCBC10	107.23
SCBC20	212.18
SCBC30	348.12

The FTIR spectra of the biochars derived under

650 °C at different holding times were chosen to illustrate the effect of holding time on their spectral properties. As presented in Figure 2(a), the typical spectrum had several absorption peaks. The broad peak at between 3400 and 3550 cm⁻¹ was assigned to the O–H stretching of structural and free hydroxyls in organic silicon or phenolic compounds on the surface of biochar [11]. The

peak at around 1705 cm⁻¹ was assigned to the absorption of free carbonyl groups and is therefore a typical hemicellulose marker [12]. The increasing absorption intensities, at bands 1600 cm⁻¹ and 780 cm⁻¹, with increasing holding time, indicates the decrease of aliphatic hydrocarbon and development of the aromatic structure.

The crystalline structure and phase purity of the SCBC (as shown in Figure 2(b)) were determined by XRD. The XRD diffractogram of the SCBC suggests that it is amorphous. XRD patterns of SCBC10, SCBC20 and SCBC30 show two broad peaks at 2θ of about 22° and 42° ; corresponding to the (002) and (100) plane reflections, respectively, which were assigned to the disordered crystalline structure of carbon.

For SCBC30, these two peaks increased in intensity and became sharper; thus indicating that some partial crystalline structure of cellulose was formed [14].

The SEM images of raw SCB and SCBC, produced at different holding times, clearly shown in Figure 3, reveal that various heterogeneous pores and rough texture are present. A planar, constricted and blocked by deposited tarry substances of raw SCB, was observed in Figure 3(a). The extraction of some materials, such as the dissolution of lignin and other components from the bagasse during pyrolysis, and the escape of the developed volatile compounds from within the particle, gradually increased when reaching the maximum holding time during the heating process. Consequently, the porosity of the biochar and the size of the voids increased with rising holding time [15].

In the fingerprint region of SCBC (i.e., $1500-400 \text{ cm}^{-1}$), the spectra consist of several peaks that indicate the C–O stretching region between 1200 and 950 cm⁻¹, that are most probably due to cellulose-related absorptions [13]. Both SCBC held at 20 and 30 minutes, seemed to sharpen these bands, and might indicate changes in the interaction between the C–OH, C–O–C and other functional groups. With the holding time increase, the absorption intensities (at approximately 3500 cm⁻¹) became broad; thus suggesting that large amounts of hydroxyl groups were created during pyrolysis. The band at 3500 cm⁻¹, with increasing intensity, may also be assigned to O–H vibration with interchain H–bonds between the cellulose fibres [13].



Figure 3: Morphology of (a) raw SCB, (b) SCBC10, (c) SCBC20 and (d) SCBC30 at 50µm. (offset image refers to pores taken from different angle).

4. Conclusion

The optimal pyrolysis conditions of SCB were found at 400°C and 30 min holding time, which resulted in a biochar yield of 28.48%. The successful production of biochar using the self-purging pyrolysis reactor confirmed the possibility of using a low-cost pyrolysis unit to explore the potential of SCB residues as pyrolysis feed-stock and the optimization of biochar yield. Pyrolysis temperature and holding time significantly affect the properties of biochar

derived from SCB. Increasing temperature and extended holding time may reduce the volatile matter content, enhance carbon enrichment, and develop an aromatic structure in biochar; thus producing biochar with high specific surface area and an extensively porous structure.

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