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Biochar from anaerobically digested sugarcane bagasse

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ABSTRACT

This study was designed to investigate the effect of anaerobic digestion on biochar produced from sugarcane bagasse. Sugarcane bagasse was anaerobically digested to produce methane. The digested residue and fresh bagasse was pyrolyzed separately into biochar at 600 °C in nitrogen environment. The digested bagasse biochar (DBC) and undigested bagasse biochar (BC) were characterized to determine their physicochemical properties. Although biochar was produced from the digested residue (18% by weight) and the raw bagasse (23%) at a similar rate, there were many physiochemical differences between them. Compared to BC, DBC had higher pH, surface area, cation exchange capacity (CEC), anion exchange capacity (AEC), hydrophobicity and more negative surface charge, all properties that are generally desirable for soil amelioration, contaminant remediation or wastewater treatment. Thus, these results suggest that the pyrolysis of anaerobic digestion residues to produce biochar may be an economically and environmentally beneficial use of agricultural wastes.

1. Introduction

The conversion of biomass into value-added products such as biofuel and biochar has attracted tremendous research interest. This can be attributed to the rising energy demands and concerns over greenhouse gas emissions, as well as worldwide soil degradation (Laird et al., 2009; Lehmann, 2007). As one of the most popular bioenergy conversion technologies, thermal pyrolysis of carbon-rich biomass is unique because it produces biochar (charcoal) in addition to biofuel. Recent studies have highlighted the benefits of biochar technologies, particularly with respect to carbon sequestration via land application of biochar (Laird et al., 2009; McHenry, 2010). As a result, the conversion of biomass into biochar and biofuel has been receiving greater attention from government regulation agencies and the general public. For example, the 2008 Farm Bill established the first federal-level policy in support of biochar production and utilization programs nationally, and biochar has been mentioned in the United Nations Framework Convention on Climate Change in December 2009.

Sugarcane bagasse is the residual material derived from sugarcane after extracting cane juice. Like most agricultural residues, bagasse is a carbon-rich biomass, highly abundant and suitable for biofuel or biochar production. Several studies have been conducted to explore the potential of biofuel production from bagasse through pyrolysis (Mothe and de Miranda, 2009), but limited attention has been paid to biofuel production from anaerobic digestion of bagasse or possible uses of its residues. Over 850,000 tons of bagasse generated in Florida in the United States are either burnt directly as fuel in sugar mills or disposed of in landfills (Burnham, 2010). Anaerobic digestion of bagasse could be an additional source of biofuel (Osman et al., 2006).

Bagasse is a complex lignocellulosic material which consists primarily of 50% cellulose, 25% hemicellulose, and 25% lignin, in addition to other components such as pentosans, α-cellulose, and inorganic compounds referred to as ash (Pandey et al., 2000). Anaerobic digestion of most lignocellulosic materials like bagasse proceeds at low loading rates, long solid retention times and low conversion efficiencies (Kivaisi and Eliapenda, 1995). A few studies showing the feasibility of biogasifying sugarcane bagasse for biofuel (mainly methane) production, have indicated the hydrolysis of cellulose as the rate limiting step and the crystallinity of cellulose as a major obstacle in the digestion process (Kivaisi and Eliapenda, 1995; Rodriguezvazquez and Diazcervantes, 1994). In overcoming these challenges, researchers have suggested the use of steam explosion, and acid and alkaline pre-treatment methods to enhance the digestion of bagasse to methane (Amjed et al., 1992).

Using a variety of these anaerobic digestion pre-treatment methods, a maximum bagasse digestibility of 75% by weight has been reached (Rodriguezvazquez and Diazcervantes, 1994). Consequently, at least 25% of the bagasse will remain as residue after the digestion process. Large-scale anaerobic digestions of sugarcane bagasses, therefore, would require recycling of the digested residues (sludge) in an economic and environmentally friendly way.

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Traditionally, residues obtained from anaerobic digestion are applied as compost to soils directly. But increasing concerns on the potential contamination of the food chain by toxic trace elements have necessitated alternative methods of sludge recycling (Tyagi et al., 1988). Pyrolysis of anaerobically digested bagasse residue to produce biochar has been proposed as an beneficial product that could be obtained from digestion residuals (Sialve et al., 2009).

This study examined the conversion of sugarcane bagasse into biochar and biofuel using anaerobic digestion and thermal pyrolysis. Anaerobic digestion of bagasse was carried out to generate methane and possibly improve the stock material properties for biochar production. Two feedstock materials were employed in the pyrolysis study: raw bagasse and the residue obtained from anaerobically digested bagasse. These materials were converted into biochar and biofuel at 600 °C. The conversion rates of biochar and biofuel were determined. In addition, physicochemical properties of the biochar produced were characterized. Our objectives were to: 1) determine the methane potential of sugarcane bagasse via anaerobic digestion, 2) examine the feasibility of using the digested sugarcane bagasse residue as a feed stock for biochar production, and 3) compare the physicochemical properties of biochar obtained from digested bagasse residue to those of biochar obtained from pyrolysis of sugarcane bagasse directly.

2. Methods

2.1. Raw materials

The feed stock, sugarcane bagasse (sized 0.5–1 mm), was obtained from Florida Crystals, Okeelanta, Florida and stored in airtight ziploc bags and refrigerated until ready for use. Prior to the digestion of the samples, 150 g aliquots of the refrigerated bagasse were dried in an oven at 105 °C for 24 h. Volatile solid (VS) content of bagasse was determined by ashing 100 g of the dried samples in a muffle furnace at 550 °C for 2 h and determining the ash-free dry weight (Koppar and Pullammanappallil, 2008). The total solids (TS) and volatile solids (VS) content of the feedstock were determined gravimetrically before and after the digestion process.

2.2. Anaerobic digestion of bagasse

A thermophilic anaerobic digester was used to biogasify the raw bagasse (Fig. 1). The design and procedures of the anaerobic digestion experiment were similar to those of Koppar and Pullammanappallil (2008). In brief, 400 g of fresh bagasse (wet weight) was added to the digester and mixed with porous volcanic rocks (average grain size 25 mm, from a landscaping supplier) to prevent compaction of the solids. To initiate the anaerobic digestion process, 2 L of mixed liquor (inoculum), obtained from a currently operational, active and stable thermophilic digester was added to the vessel containing the feedstock. The digester was then sealed and incubated at a constant temperature of 55 °C until the end of the experiment. The pH of the mixture was monitored daily. Biogas produced from the anaerobic digester under batch conditions was monitored with a positive displacement gas meter consisting of a clear PVC U-tube filled with anti-freeze solution, solid state time delay relay, a float switch, a counter, and a solenoid valve. The U-tube gas meter was calibrated in-line to determine volume of biogas. A gas syringe was used to draw samples from the digester port daily and concentrations of methane and carbon dioxide produced were determined with a gas chromatograph (Fisher Gas Partitioner 1200). Anaerobic digestion was considered complete when no further gas production was recorded by the gas meters. The sealed digester was opened and emptied and the solid residue was separated from the inoculum and dried at 105 °C in the oven. A fraction of the dried residue was analyzed for TS and VS content and the remaining mass was used for biochar production. The methane yield from the anaerobic digestion of bagasse was reported in terms of the values of VS obtained.

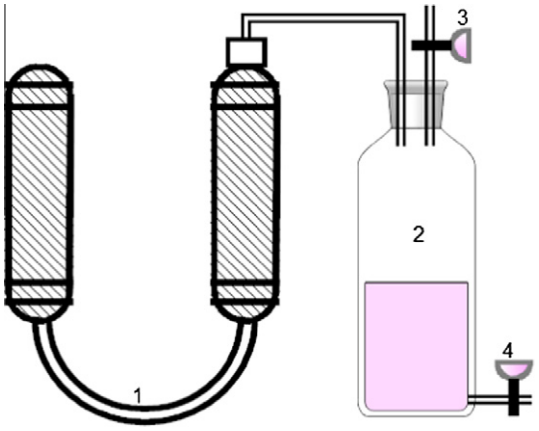

Fig. 1. Schematic of the experimental set-up for anaerobic digestion. (1) U-tube Gas meter, (2) Anaerobic digester, (3) Gas sampler port, and (4) Drain valve.

2.3. Biochar and biofuel production

Both raw bagasse and digested bagasse residue were converted into biochar using a bench-scale pyrolyzer. For each experiment, 15 g of dried samples were fed into a mini tubular reactor (6 cm diameter cylinder 28 cm long) designed to fit inside a bench-top furnace (Barnstead 1500 M). The tubular reactor was first purged with nitrogen gas (10 psi) and an oxygen sensor attached to the reactor ensured that the oxygen content in the reactor was less than 0.5% before it was inserted into the furnace. The reactor was purged again with N_2 along with the furnace and sealed for pyrolysis. The controller of the bench-top furnace was programmed to drive the furnace temperature to 600 °C at a rate of 10 °C/min and held at the peak temperature for 1.5 h before cooling to room temperature. Biochar produced from the pyrolysis was crushed and sieved into two size fractions: <0.5 mm and 0.5–1 mm. Only the latter was used in the characterizations to minimize the influences of residual ash particles.

2.4. Physicochemical properties of biochar

A range of physicochemical properties (e.g., pH, surface properties, elemental compositions, etc.) of the digested bagasse biochar (DBC) and the undigested bagasse biochar (BC) were determined. The pH of the biochar was measured by adding biochar to de-ionized water in a mass ratio of 1:20. The solution was then hand shaken and allowed to stand for 5 min before measuring the pH with a pH meter (Fisher Scientific Accumet Basic AB15). The surface area of the biochar was determined through a surface area analyzer (NOVA 1200) using the Brunauer–Emmett–Teller (BET) nitrogen adsorption method at 77 K.

The surface charge of the samples was determined by measuring the zeta potential (ζ) of colloidal biochar according to the procedure of Johnson et al. (1996). About 1 g of each sample was added to 100 ml of de-ionized water and the solution was shaken at 250 rpm for 30 min using a mechanical shaker. The shaken solution was then placed in a sonic bath to break the particles into colloids and the solution filtered using a filter paper. The ζ of each supernatant solution obtained was determined using a Brookhaven Zeta Plus (Brookhaven Instruments, Holtsville, NY). Smoluchowski's formula was used to convert the electric mobility into zeta potential.

Elemental carbon, hydrogen, and nitrogen of the raw bagasse, DBC, and BC was determined using a CHN Elemental Analyzer (Carlo-Erba NA-1500) via high-temperature catalyzed combustion followed by infrared detection of resulting CO_2, H_2 and NO_2 gases, respectively. The oxygen content was determined by weight difference assuming that the total weight of the samples was made up of C, H, N and O only.

Cation exchange capacity (CEC) and anion exchange capacity (AEC) of the samples were determined simultaneously using the point of zero net charge method (Zelazny et al., 1996). The samples were mixed with KCl solutions to saturate the biochar's exchangeable cation and anion sites. $NaNO_3$ solutions were used to displace the bound K^+ and Cl^-. Concentrations of the displaced K^+ and Cl^- were determined using flame atomic absorption spectrometry (FAAS; Varian 220 FS with SIPS, Walnut Creek, CA) and an ion chromatograph (Dionex ICS90), respectively. CEC and AEC of the samples were calculated based on the measured cation and anion concentrations and the sample weight.

Scanning electron microscope (SEM) imaging of the raw materials and biochar samples were carried out using a Hitachi S-4000 FE–SEM with maximum resolution of 1.5 nm. Varying magnifications were used to compare the structure of the bagasse and biochar samples before and after the anaerobic digestion. The accelerating voltage of the instrument was maintained at 10 kv.

Fourier Transform Infrared (FTIR) analysis of BC and DBC was carried out to characterize the surface functional groups present on these samples. To obtain the observable adsorption spectra, BC and DBC were ground and mixed with KBr to 0.1 wt.% and then pressed into pellets. The spectra of the samples were measured using a Bruker Vector 22 FTIR spectrometer (OPUS 2.0 software).

3. Results and discussion

3.1. Methane yield from anaerobic digestion of sugarcane bagasse

The total methane yield from the anaerobic digestion of sugarcane bagasse was about 84.75 L/kgVS at the end of 40 days (Fig. 2). Similar low yields of methane from the digestion of untreated bagasse have been reported by Osman et al. (2006) with a total biogas (includes methane and carbon dioxide) production of 0.02 L/kgVS.

The methane yield was much lower from anaerobic digestion of bagasse than from other feedstock materials such as beet pulps (336 L/kgVS) and sugar beet tailings (295 L/kgVS) (Koppar and Pullammanappallil, 2008; Liu et al., 2008). This can be attributed to the crystalline cellulosic structure of sugarcane bagasse, which usually has a very low biodegradability. Nevertheless, the cellulose in bagasse was sufficiently degraded by the inoculum to alter the appearance of the digested residue and create a more porous structure in comparison to the raw bagasse (Fig. S1a and b, supplementary data). The low yield of methane from bagasse in this study compared to other feedstock materials could also be attributed to pH inhibition of the digestion process. During anaerobic digestion of the bagasse, pH in the digester increased from 7.6 to 9.4 (Fig. 3), which was above the optimum value of 7.0–7.5. The average pH during the first 30 days of digestion was 8.5 which increased to 9.0 thereafter. Increase in pH has also been observed during batch digestion of biomass feedstocks like sugarbeet pulp, citrus pulp and sorghum stalks. This could be due to mobilization of cations like K and Ca from the biomass during digestion. High pH conditions have been found to suppress methanogen growth requiring methanogenic Archae to expend more energy for homeostasis than anabolism, resulting in slow degradation of the substrate (Gutierrez et al., 2009). Because there was no detectable nitrogen in the DBC (Table 1), the growth of bacteria, particularly methanogens, in the substrate could be limited by nitrogen deficiency, which may have also caused the low methane yield.

Advancement in research efforts for improving the digestion of bagasse including hemicellulose hydrolysis and conversion of crystalline cellulose to more fermentable sugars could make bagasse digestion a more economically attractive process for biofuel production.

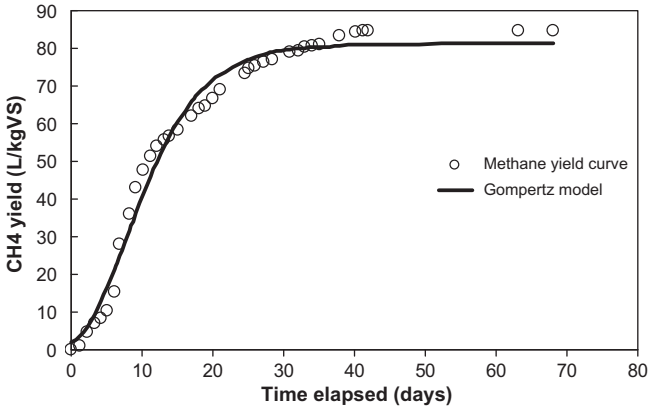

Fig. 2. Time course of methane yield during anaerobic digestion of sugarcane bagasse.

3.2. Modeling methane yield from sugarcane bagasse

Methane production in an anaerobic digester is a microbially associated growth product and is often described using sigmoidal curve bacterial growth models such as the Gompertz equation (Koppar and Pullammanappallil, 2008). In this study, the modified Gompertz equation derived by Zwietering et al. (1990) was used to simulate methane evolution from sugarcane bagasse, such that:

$$y = A\ \exp\left\{-\exp\left[\frac{\mu_m \cdot e}{A}(\lambda - t) + 1\right]\right\} \quad (1)$$

where y is the cumulative methane production (L/kgVS), A is the maximum methane yield potential (L/kgVS), μ_m is the maximum methane production rate (L/kgVS/day), e is the Euler's number

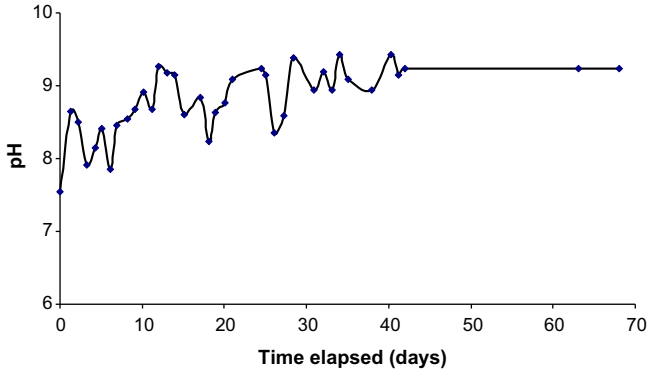

Fig. 3. Time course of pH during sugarcane bagasse digestion.

Table 1
Elemental analysis of raw bagasse and biochar samples.

Sample	% C	% H	% N	% O
Raw bagasse	46.08	6.88	0.74	46.30
DBC	73.55	2.41	–	24.04
BC	76.45	2.93	0.79	19.83

(2.72), the λ is the duration of the lag phase (day), and t is time (day). The model successfully reproduced the experimental data with a goodness of fit statistic, R^2, exceeding 0.98 (Fig. 2). The model-estimated A, μ_m, and λ were 81.29 L/kgVS, 5.08 L/kgVS/day, and 1.96 days, respectively. These values suggest that the anaerobic digestion efficiency of sugarcane bagasse is relatively low in comparison to other feedstock materials (Koppar and Pullammanappallil, 2008; Liu et al., 2008). The digested sugarcane bagasse residue, therefore, has the potential to be used as a feedstock material for biochar and biofuel production through pyrolysis.

3.3. Biochar and biofuel production from digested and undigested bagasse

The amount of biochar produced from the pyrolysis of digested bagasse residue and undigested bagasse was similar with efficiencies of 18% and 23% of initial dry weight, respectively (Fig. 4). The slightly lower rate of biochar production from pyrolyzed digested bagasse is probably because of the slight reduction in the carbon content of the bagasse after degradation as indicated by elemental analysis (Table 1). Generally, decreased formation of char during volatilization of biomass is accompanied by increased yield in bio-oil products (Demirbas et al., 2006). The biofuel (i.e., bio-oil and non-condensable gas) production rates from the pyrolysis of digested bagasse residue and undigested bagasse were 82% and 77%, respectively; suggesting that substantial amount of biofuel can still be extracted from the digested bagasse residue through pyrolysis. These results also suggest that it is feasible to use digested bagasse residue as a feedstock for both biochar and further biofuel production.

3.4. Effect of anaerobic digestion on biochar properties

Biochar can be used as a soil amendment to improve soil quality which, due to its refractory nature, will also sequester atmospheric carbon for long time periods, and as a low-cost adsorbent to remove contaminants from wastewater (Cao et al., 2009; Liu and Zhang, 2009; Novak et al., 2009). The effectiveness of biochars in these potential applications will be determined by its physicochemical properties, such as pH, surface charge, surface area, CEC, and AEC. Laboratory characterizations of the DBC and BC revealed that anaerobic digestion had a substantial effect on these physicochemical properties (Table 2).

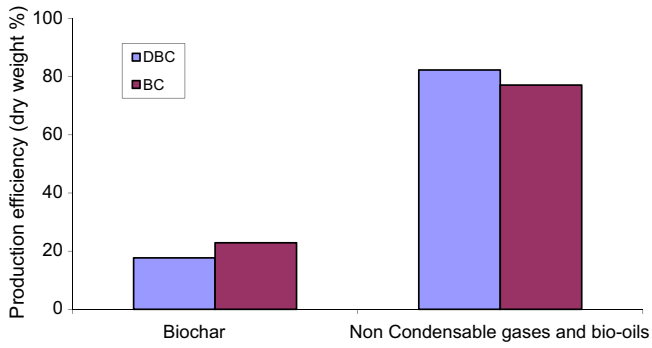

Fig. 4. Biochar and biofuel production from digested and undigested bagasse via pyrolysis.

Table 2
Summary of the physicochemical properties of biochar samples.

Sample	pH	Zeta potential (mv)	BET surface area (m^2/g)	CEC (cmol/kg)	AEC (cmol/kg)
DBC	10.9	−61.7	17.66	14.30	11.19
BC	7.7	−28.1	14.07	4.19	6.64

Measurements of biochar pH showed DBC had a higher pH (10.9) than BC (7.7) (Table 2). The high pH of DBC can be attributed to the fact that anaerobic digestion may concentrate recalcitrant cationic species (Pb, Cd, Zn, Cr, Cu, Ni) as well as exchangeable cations (Ca, Mg, Na) into the residue (Gu and Wong, 2004; Hanay et al., 2008). The DBC also had a much lower zeta potential of −61.7 mV in comparison to BC (−28.1 mV), indicating that the surface charge of the DBC was more negative than that of BC. Corresponding to the SEM images (Fig. S1c and d, supplementary data), the BET surface area of DBC (18 m^2/g) was higher than that of BC (14 m^2/g) and may reflect microbial utilization of more labile pore in-filling organic matter during digestion, leaving the refractory pore framework intact (Zimmerman, 2010). Because pH, surface charge, and surface area are among the most important factors that govern a material's interaction with chemical compounds, particularly with respect to cationic metal species. The digested bagasse biochar, therefore, may have a better ability to sequester metal species than non-digested bagasse biochar.

The measured CEC and AEC of DBC were 14.30 cmol/kg and 11.19 cmol/kg, respectively, which were higher than those of BC (6.64 cmol/kg and AEC 4.194 cmol/kg, respectively). When used as a soil amendment, DBC would likely be better able than BC to improve the nutrient holding capacities of the soils. However both biochars would significantly improve the exchange properties of soils and act similarly to enrichments in natural organic matter. It is further notable that the AEC found for both of these chars has not previously been measured in any biochar (Cheng et al., 2008; Liang et al., 2006).

The effect of anaerobic digestion on the properties of biochar produced can be further discriminated through its surface functional groups as determined by FTIR spectroscopy (Fig. S2, supplementary data). It has been reported that surface functional groups present in biochar are mainly a function of the pyrolysis temperature and pyrolysis conditions under which it was produced (Chun et al., 2004). Here, however, it was found that biomass pre-treatment may also play a role in the resulting functional group distribution. The infrared spectra of DBC were characterized by four significant bands at wave number 3452 (O–H functional group), 2349 (carbonyl, O=C=O bond group), 1626 (alkene, C=C bond group), and 646 (C–H aromatic group) cm^{-1}(Fig. S2, supplementary data). The spectrum of BC was characterized by four significant bands at wave number 3130 (O–H functional group), 1600 (alkene, C=C bond group), 1090 (phenolic, C–O stretch absorption band), and 826 (C–H aromatic group) cm^{-1}. So the major differences include the appearance of the dominant phenolic component in the undigested biochar only and the presence of carbonyls (O=C=O bond group) in the digested biochar only.

All of the observed functional groups have been reported as chemical groups characterizing many carbon sorbents (Cao et al., 2009; Nguyen et al., 2009; Ozcimen and Karaosmanoglu, 2004; Purevsuren et al., 2003; Suhas Carrott and Carrott, 2007) as well as in other biochars (Rutherford and Wershaw, 2008; Rutherford et al., 2004). The presence of an additional phenolic, C–O stretch band with high absorption intensity in BC at wave number 1090 cm^{-1} suggests that the alkalinity of BC was lower than that of DBC because the phenolic functional group promotes acidity in the biochar (Lopez-Ramon et al., 1999). This result is corresponding to the pH measurements. Furthermore, the presence of oxygen-containing functional groups in BC would produce a relatively more hydrophilic character than DBC which has a greater degree of aromaticity as indicated by FTIR. The digested bagasse biochar, therefore, may have a better ability to adsorb organic compounds than the raw bagasse biochar.

Based on the characterization of its physicochemical properties, it is evident that anaerobic digestion of bagasse enhances the adsorption and ion exchange abilities of biochar produced from digested relative to undigested bagasse residues. Therefore, the method of combining anaerobic digestion and pyrolysis can be used to produce additional biofuel or heat while generating high quality biochars to be used as low-cost adsorbents or as soil amendments.

4. Conclusion

Anaerobic digestion of bagasse was carried out to investigate the effects of the digestion process on biochar production via pyrolysis of the digestion residues. Biochar produced from anaerobically digested bagasse residue had a higher pH, surface area, CEC and AEC, and hydrophobicity, as well as a more negative surface charge in comparison to the undigested bagasse biochar. These characteristics suggest that the digested bagasse biochar may be efficiently used as a soil amendment to improve soil quality, to serve as a contaminant remediation barrier, or a low-cost adsorbent to remove contaminants from wastewater.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.biortech.2010.06.088.

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