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# ALKALI MODIFICATION: AN APPROACH TO IMPROVE SURFACE PROPERTIES OF BIOCHAR FROM EASILY AVAILABLE FEEDSTOCKS

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*Key words*: Rice Stubble Biochar, Saw Dust Biochar, KOH modification, SEM, Surface characteristics, Nutrient status

**Abstract**

Biochar is a stable carbon-rich material whose potentials can be greatly enhanced by modification with simple treatments. Easily available two different biochars such as rice stubble biochar (RSB) and saw dust biochar (SDB) were slowly pyrolyzed (450±50°C) and modified with KOH as RSB-M and SDB-M respectively. Represented biochars were analyzed to know their physicochemical characteristics and nutrient contents. Results revealed that nutrient contents such as total N, P and S of RSB and SDB were found maximum whereas RSB-M and SDB-M showed the lowest nutrient contents. All the biochars possessed pH value higher than 7. In case of organic C, the values of RSB-M (19.97%) and SDB-M (17.83%) were the highest and RSB (17.32%) and SDB (14.80%) were the lowest. All the biochars showed very low concentration of heavy metals (such as Cr, Cd, Pb and Ni). Water holding capacity, average particle size and surface area were higher in the RSB-M and SDB-M compared to unmodified RSB and SDB. These findings showed that alkali-modified biochars had better surface properties than unmodified biochars.

## Introduction

Biochar is a fine grained high carbon form of charcoal produced by pyrolysis of biomass in an oxygen-limited environment and can be applied to agricultural land as part of agronomic management. It is found in soils around the world as a consequence of forest fires and historical soil managements practices. The idea of biochar for soil amendment originated from soils particular to Amazonian Basin and can be produced from a variety of feedstocks ranging from agricultural byproducts to waste materials. Biochar has been used as a soil amendment for at least 2000 years[(1)]. It converts agricultural waste into a soil enhancer that can hold carbon, boost food security and discourage deforestation, remediate contaminated soil and water.

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According to Lehmann and Joseph[2], the char produced by pyrolysis process is called biochar when its application towards environmental management benefits to soil. Biochar is an alkaline and stable organic source of nutrients and can be regulated in the soil through improved cation exchange capacity[3]. Additionally, as a soil ameliorant, it has different positive effects on soil and can contribute to the recovery of nutrients from waste and increase crop yields. Biochar has high porosity and surface area, and alters soil's physical properties such as bulk density, soil porosity, water-holding capacity, hydraulic conductivity, surface area, and penetration resistance[4]. It also has significant impact on soil carbon sequestration, microbial community and greenhouse gas emissions and has many additional long lasting benefits for plant nutrient cycling, such as increasing nutrient retention and use efficiency, and reducing leaching, thereby improving soil fertility[5].

Biochar has gained attention in the past few years because of its potential applications in waste management, renewable energy, carbon sequestration, greenhouse gas (GHG) emission reduction, soil and water remediation, enhancing soil quality and crop production[6]. Biochar has broad specific surface area and porous structure with abundant surface groups and they can be used as a sorbent to remove pollutants (inorganic and organic) from both water and soil[7]. The complex and heterogeneous chemical and physical composition of biochar provides an excellent platform for adsorption removal of contaminants.

Unmodified biochars may have limited ability to selectively adsorb contaminants of high concentrations[8]. Biochars potential can be greatly enhanced by modifying them with simple treatments. Modification can be done with several treatments like physical, chemical or magnetic treatments. Impregnation with mineral oxides/ hydroxides is getting huge attention recently. Resulting modified biochars should have increased surface area and surface functional groups than that of unmodified biochars[9] and become significantly better treatment for pollutants (both organic and inorganic) remediation.

We assume that, KOH-modified biochar will have better surface functionality. Therefore, the main objective of this research is to identify the physicochemical properties of biochar and modified biochar produced from easily available feedstocks.

## Materials and Methods

*Feedstock selection:* Two different types of easily available feedstocks were collected to produce biochars. Saw dusts (Eucalyptus) were collected from sawmill (Malek Timber and Saw Mill) from Mohadebpur, Naogaon. Rice stubbles (BRRI Dhan-51) (Rice husk+ Rice straw) were collected from a farmer of Kalushoher village, Mohadebpur, Naogaon. After collection, the materials were packed in polythene bags, and the bags were tied with strings to avoid any air exchange between the atmosphere and the sample itself.

*Processing and production of biochar:* Before biochar production, all feedstocks were air-dried under the sunlight for few days. After drying properly feedstocks were processed and pyrolyzed in a specially designed kiln that was made with a wasted pressure cooker, stainless steel pipe, and heat resistant rubber (Fig. 1). The pipe was attached to the upper part of the cooker and the whole pressure cooker was made air tightened by the heat resistant rubber in the head of the cooker. The pipe was used to the syngas produced in the cooker.


Fig. 1. Biochar production using specially designed kiln.

Individual feedstocks were placed in the cooker and then the head of the cooker is locked in such a way that no oxygen can enter inside. The cooker was then placed on the gas Stover for burning. After one hour of burning, an approximate temperature of 450-500°C was maintained for 3.5 hours. After completion of the process, the cooker was removed from the stover and kept on the floor to cool down to room temperature. Then, the lid of the pot was opened and screened through a 0.50 mm and 0.25 mm stainless sieve and preserved in plastic jars with paper tags indicating the source.

*Production and processing of modified biochar:* After production, biochars were further treated with 1M KOH in a ratio of 1: 10 at temperature 60-75°C for an hour with continuous stirring as described by Liu *et al.*[10]. After treatment, modified biochars were allowed to cool down and their pH was adjusted around 7 with deionized water. Then, the modified biochars were dried at temperature 80°C for 12 hours in an oven. Dried modified biochars were then cooled down to 25°C and preserved.

**Table 1. Symbols used for feedstocks, biochars and modified biochars.**

| Feedstocks | Biochars | Modified biochars |
|---|---|---|
| Saw Dust | Saw Dust (SDB) | Modified Saw Dust (SDB-M) |
| Rice Stubble (rice straw + rice husk) | Rice Stubble (RSB) | Modified Rice Stubble (RSB-M) |

*Laboratory analysis and analytical procedure:* To determine the water holding capacity by mass, ASTM(11) method was followed. The morphological properties of all biochars were analyzed by Scanning Electron Microscopic (SEM) imaging. A range of SEM images (Magnification: 2000× to 10,000×) was captured with a JEOL JSM-6490 operating at 20KV at the Center for Advanced Research in Sciences (CARS), University of Dhaka. Image analysis was done with ImageJ version 2.0 with appropriate threshold and size range values. The pH, electrical conductivity (1:10 ratio), and cation exchange capacity (CEC) of the unmodified and modified biochar samples were measured as described in Rayment and Higginson(12). Organic carbon of the unmodified and modified biochar samples was determined by the wet oxidation method of Walkley and Black(13). The total N of the samples was determined by the Kjeldahl steam distillation method(14). The concentration of P, K, and S in unmodified and modified biochar samples were analyzed after digestion with nitric-perchloric acid(14). Total P was measured colorimetrically using a spectrophotometer by developing yellow color with vanadomolybdate, total K by flame photometer, and total S by the turbidimetric method using a spectrophotometer(14). Total Calcium (Ca), Magnesium (Mg), Iron (Fe), Manganese (Mn), Zinc (Zn), and heavy metals like – lead (Pb), chromium (Cr), nickel (Ni), and cadmium (Cd) content of the digested samples were determined by a Varian atomic absorption spectrophotometer (VARIAN AA240).

Statistical analyses were done by using Microsoft excel 2010 and SPSS version 16.

## Results and Discussion

*Physical characteristics of unmodified and modified biochars:* The structural composition of the surface morphology of all the biochars was highly diverse. Generally, biochar consists of abundant minerals and organic structures. The SEM images showed the heterogeneous and amorphous structures of the biochars. Here are some Figure of SEM image analyses with 5000 times zoom (Fig. 2).

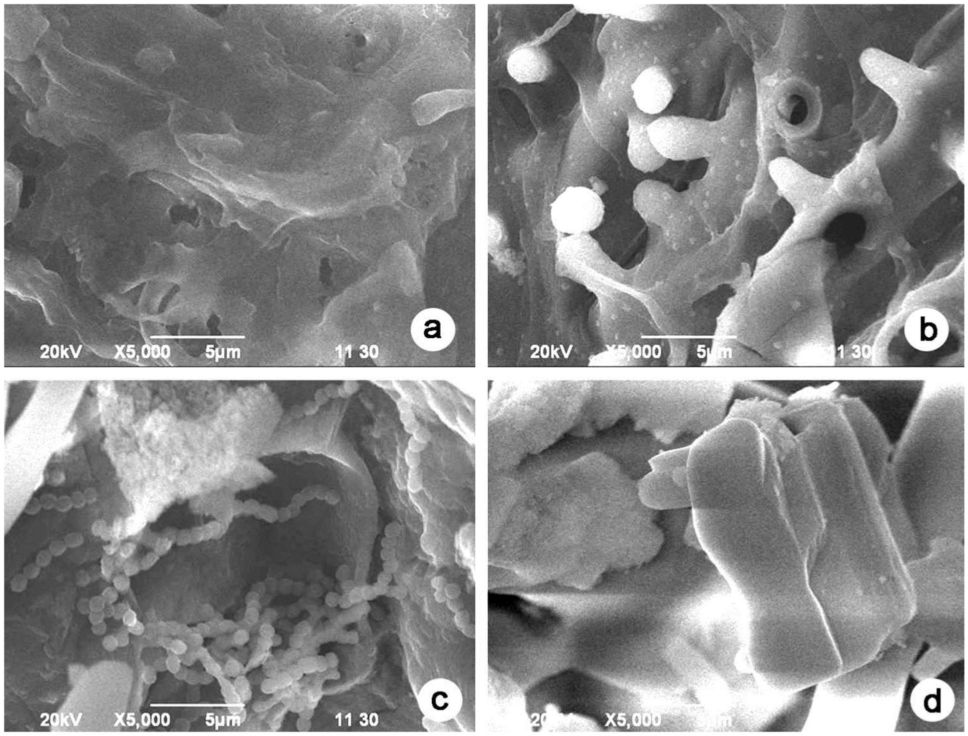


Fig. 2. Images obtained by Scanning Electron Microscopy (SEM). (a) Saw Dust Biochar (SDB), (b) Modified Saw Dust Biochar (SDB-M), (c) Rice Stubble Biochar (RSB), (d) Modified Rice Stubble Biochar (RSB-M).

Biochar surface is very sensitive to modification reagent. The selection of modification reagents for improvement of biochar performance requires a comprehensive understanding of structure and particle distribution. After analyzing the images with ImageJ software (Table 2 and Fig. 2) it was found that RSB-M possessed the largest average particle size (0.421µm$^2$), highest surface area (27.15%) occupied by particles, and highest water holding capacity (394.74%).

**Table 2. Physical characteristics of unmodified and modified biochars.**

| Properties | Biochar types | | | |
|---|---|---|---|---|
| | SDB | SDB-M | RSB | RSB-M |
| Water holding capacity (%) | 164.47 ± 2.11 | 263.16 ± 1.47 | 197.36 ± 1.14 | 394.74 ± 2.50 |
| Particle size (µm$^2$) | 0.112 ± 0.01 | 0.245 ± 0.04 | 0.234 ± 0.03 | 0.421 ± 0.01 |
| Area covered by particles (%) | 8.91 ± 0.41 | 14.19 ± 0.81 | 13.65 ± 0.50 | 27.15 ± 1.44 |

± = standard deviation.

Contrarily SDB contained the lowest average particle size (0.112 $\mu m^2$), the area occupied by particles (8.91%), and water holding capacity (164.47%). Potassium species ($K_2O$, $K_2CO_3$) may be formed during the modification with KOH due to the intercalation of $K^+$ in the layer of the crystallites that form the condensed C structure. These potassium species may diffuse into the internal structure of unmodified biochars matrix, widen existing pores and create new pores in the modified biochars[15]. The highly porous characteristics of these biochars may grant high surface area which is like to increase soil aeration, soil structure, water-holding capacity and nutrient retention, sorption of contaminants when applied to the soil. Hence, all biochars could be a suitable amendment to light soils especially for newly developed lands with high sand deposited, as provides high water holding capacity[16].

*Chemical characterization of unmodified and modified biochars:* All biochars found to be alkaline in nature, pH 7.48 to 8.60 (Table 3). The production methods and high temperature increase the biochars pH value probably in consequences of the relative concentration of non-pyrolyzed inorganic elements that are already present in the original feedstocks[17]. Biochars alkalinity can be attributed to four broad categories: surface organic functional groups, carbonates, soluble organic compounds, and other inorganic alkalis including oxides, hydroxides, sulfates, sulfides, and orthophosphates[18]. Alkaline biochar application to acid soils may help to increase soil pH as well as reduce Al toxicity and increase P availability[16].

**Table 3. Chemical properties of unmodified and modified biochars.**

| Biochar types | Parameters | | | |
|---|---|---|---|---|
| | pH | EC (mS/cm) | CEC ($Cmol_c$/Kg) | OC (%) |
| SDB | 7.62 ± 0.05 | 0.53 ± 0.01 | 23.33 ± 1.60 | 14.80 ± 0.41 |
| SDB-M | 7.48 ± 0.03 | 17.29 ± 0.19 | 33.33 ± 1.44 | 17.83 ± 0.00 |
| RSB | 8.60 ± 0.04 | 0.87 ± 0.02 | 32.50 ± 2.50 | 17.32 ± 0.41 |
| RSB-M | 7.72 ± 0.01 | 17.94 ± 0.14 | 39.17 ± 1.52 | 19.97 ± 0.31 |

Electrical conductivity was very high for RSB-M and then for SDB-M (Table 3) and other biochar had relatively lower EC. High EC resulting from modified biochars may be due to high soluble salt concentrations and their modification with KOH.

Cation exchange capacity (CEC) of biochars indicates their ability to hold cationic substances. Modified rice stubble biochar possessed the highest CEC 39.17 $Cmol_c$/Kg and SDB possessed the lowest CEC 23.33 $Cmol_c$/Kg. Potassium, Ca, Mg, Na, and P in the biomass promote the formation of O-containing groups on biochar surface during pyrolysis, resulting in higher CEC. Modification using KOH further increases the surface basicity[19] of unmodified biochars. Biochars with higher CEC, when applied to soil, can increase soil CEC, and soils with high CEC values are able to retain cationic fertilizers ($K^+$ and $NH_4^+$) in the root zone and prevent nutrient leaching. Biochars with higher CEC can also be an environmental management option for remediation of soil and water contaminated with pollutants[7].

Research results indicated that RSB-M (19.97%) and SDB-M (17.83%) possessed the highest organic carbon (OC) content whereas SDB has the lowest (14.80%; Table 3). Organic C and nutrient status improvement of biochars are due to thermal humiliation which indicates the loss of volatile compounds from the original material[20] and relatively small volatilization losses of alkaline nutrients[21]. Alkali modification enabled more removal of inorganic matters[10] from biochars and resulted in higher organic C in modified biochars. This stable and inert form of organic carbon would extensively affect the physicochemical properties of soil and tends to be present in the soils for hundreds to thousands of years[22].

*Nutrient status of unmodified and modified biochars:* Pyrolysis alters the nutrient content in the resulting biochars and affects nutrient availability to plants. Nutrient contents of biochars like N, P, K, and S can vary according to the variation in the feedstock and modification. Rice stubble biochar (3.04%) and RSB-M (1.84%) had the highest content of total N (Table 4). Modified saw dust biochar (0.39%) had the lowest content of total N.

**Table 4. Total nutrient contents of unmodified and modified biochars.**

| Biochar types | Total Nutrient Concentrations (%) | | | |
|---|---|---|---|---|
| | N | P | K | S |
| SDB | 0.47 ± 0.02 | 0.21 ± 0.03 | 0.36 ± 0.04 | 0.71 ± 0.08 |
| SDB-M | 0.39 ± 0.02 | 0.07 ± 0.01 | 5.90 ± 0.90 | 0.02 ± 0.01 |
| RSB | 3.04 ± 0.18 | 0.59 ± 0.05 | 0.58 ± 0.07 | 0.39 ± 0.02 |
| RSB-M | 1.84 ± 0.35 | 0.50 ± 0.02 | 6.07 ± 0.80 | 0.24 ± 0.01 |

The influence of feedstocks on biochar properties is prominent in the case of total P but a high concentration of P resulted in RSB (0.59%) (Table 4). Other biochars showed lower content of P. Modified rice stubble biochar and SDB-M possessed higher total K content than other biochars. In the case of total S, all biochars showed sulfur content below 1% and SDB showed the highest amount of S among them. The sulfur content of biochars varies depending on their production processes like pyrolysis or gasification (>700°C)[18].

Available nutrient content of biochars is an important factor for plant growth. Available N content was found in biochars but in very low amount. Biochars are generally very low in mineral forms of N that is, Nitrate-N, and Ammonium-N[23]. Available N content of SDB (0.0134 µg/g) (Table 5) and RSB (0.0328 µg/g) were relatively high, contrarily SDB-M (0.0078 µg/g) and RSB-M (0.0039 µg/g) possessed low available N.

**Table 5. Available nutrient contents of unmodified and modified biochars.**

| Biochar types | Available Nutrient Concentrations (µg/g) | | | |
|---|---|---|---|---|
| | N | P | K | S |
| SDB | 0.0134 ± 0.0005 | 0.14 ± 0.01 | 0.18 ± 0.03 | 0.39 ± 0.01 |
| SDB-M | 0.0078 ± 0.0020 | 0.97 ± 0.03 | 4.89 ± 0.09 | 0.01 ± 0.00 |
| RSB | 0.0328 ± 0.0006 | 0.24 ± 0.02 | 0.39 ± 0.05 | 0.02 ± 0.01 |
| RSB-M | 0.0039 ± 0.0033 | 0.18 ± 0.02 | 5.93 ± 0.10 | 0.10 ± 0.00 |

The pyrolysis process plays an important role on availability of P and K. Because of the same reason, a smaller amount of P is generally lost than C or N as it transforms into less soluble minerals resulting in a reduction of the available P in biochars[24]. Available K was higher in modified biochars than unmodified biochars. Available S content of all biochars was below 1 µg/g.

*Macro and micro nutrient content of unmodified and modified biochars:* The total Ca content of RSB (1.85%) was highest and RSB-M had the second-highest Ca content (1.53%) (Table 6). On the contrary, SDB-M showed the lowest Ca content. Magnesium contents were found low in all the biochars. High production temperatures resulted in reducing decomposable substances, volatile compounds, and elements such as O, H, N, S, and, as a result, enhanced concentrations of other essential nutrients including Ca, Mg, etc.[25] in biochars. The total Fe content of RSB was the highest 730.83 µg/g while SDB-M had the lowest amount of iron.

**Table 6. Total macro and micro nutrient contents of unmodified and modified biochars.**

| Biochar types | Nutrients | | | | |
|---|---|---|---|---|---|
| | Macro nutrients (%) | | Micro nutrients (µg/g) | | |
| | Ca | Mg | Fe | Zn | Mn |
| SDB | 0.49 ± 0.0001 | 0.11 ± 0.0001 | 4.57 ± 0.47 | BDL | 3.83 ± 0.15 |
| SDB-M | 0.29 ± 0.0001 | 0.07 ± 0.0001 | 3.87 ± 0.20 | BDL | 3.73 ± 0.34 |
| RSB | 1.85 ± 0.0001 | 0.28 ± 0.0002 | 730.83 ± 4.30 | 33.56 ± 1.08 | 267.80± 0.62 |
| RSB-M | 1.53 ± 0.0004 | 0.19 ± 0.0004 | 528.17 ± 3.86 | 21.55 ± 0.71 | 206.30 ± 0.61 |

BDL= Below Detection Limit.

In the case of SDB and SDB-M, total Zn content was below the detection limit. RSB contained 33.56 µg/g of Zn and RSB-M contained 21.55 µg/g Zn. Manganese was found highest in RSB (267.80 µg/g) and lowest in SDB-M (3.73 µg/g).

*Heavy metal contents of unmodified and modified biochars:* Biochars have heavy metals inherent within their structure, derived from the source material, which may be accumulated and concentrated in the ash fractions during pyrolysis[26]. All biochars demonstrated a very low concentration of heavy metals like Cr, Pb, Cd, and Ni. The highest amount of total Cr resulted in RSB 0.32 µg/g (Table 7). Both SDB and SDB-M had a nearly similar concentration of total chromium. Total Pb content in SDB and SDB-M was below the detection limit.

**Table 7. Heavy metal contents of unmodified and modified biochars.**

| Biochar types | Elements (µg/g) | | | |
|---|---|---|---|---|
| | Cr | Pb | Cd | Ni |
| SDB | 0.06 ± 0.02 | BDL | 0.47 ± 0.06 | 7.53 ± 0.21 |
| SDB-M | 0.04 ± 0.01 | BDL | 0.60 ± 0.00 | 6.43 ± 0.42 |
| RSB | 0.32 ± 0.06 | 1.74 ± 0.04 | 0.76 ± 0.06 | 10.97 ± 0.21 |
| RSB-M | 0.30 ± 0.03 | 1.23 ± 0.03 | 0.73 ± 0.12 | 6.63 ± 0.15 |

In contrast, RSB contained a high amount of Pb and Ni content among all. The reason for rice stubble containing more heavy metals may be the uptake of heavy metals from the agricultural fields where fertilizers with high metal content were used. The reduction of heavy metals and other nutrients in modified biochars could be due to the removal of inorganic substances during the alkali modification process.

The characteristics of different biochars varied depending on the types of feedstock and modification. Modified biochars possessed higher water holding capacity, surface area, particle size, OC, EC, K, and CEC while unmodified biochars were enriched with essential nutrient content. However, the biochars and modified biochars used in this study possessed higher potentiality to be adopted in the agricultural systems for amendment purpose, pollutants remediation, and environmental management. Feedstocks for biochar production, modification process, use efficiency, and cost-benefit ratio must be selected carefully to meet the needs of a particular soil crop combination and get the desired outcome. Biochar production and modification from agricultural byproducts and wood shavings can be a new field of research interest in Bangladesh. This research has shed some light on the importance and effectiveness of biochar modification and will create a further research opportunity to work on it.

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