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FOURIER TRANSFORM INFRARED SPECTROSCOPY ANALYSIS OF UREA INTERCALATED BIOCHAR

Olubisi Oluwasegun John[1*], Adesanwo Olusola Olajumoke[1], Fagbenro John Adeniyi[2], Azeez Musibau Oyeleke[1], and Olufemi Temitope Akinwale[1]

[1]Department of Soil Science and Land Resources Management, Obafemi Awolowo University, Ile Ife, Osun - State, Nigeria.

[2]Department of Crop Production, Soil and Environmental Management, Bowen University, Iwo, Osun State, Nigeria.

[*]Corresponding Author's Email: oluwasegun557@gmail.com

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ABSTRACT: *Numerous studies have demonstrated the efficacy of biochar in enhancing soil physical and chemical properties. However, biochar's inherent nitrogen content is exceedingly low. This research sought to investigate the potential for elevating the nitrogen content of biochar through intercalation with Urea, employing chemical methods. Urea intercalated municipal biochar (UMB) fertilizer was created by blending a urea solution with the original Municipal biochar at a 1:1 ratio (20 ml: 20 g). Subsequently, it underwent an oven drying process at 65°C to eliminate moisture content. One gram of adhesive polymer (starch) was introduced into the mixture and allowed to air dry. The final product was subjected to characterization through Fourier Transform Infrared Spectroscopy (FTIR) within the range of 400 to 4000 cm-1, utilizing Attenuated Total Reflectance techniques. Selected chemical properties of municipal biochar, urea, and urea intercalated biochar, such as pH and total nitrogen (N), were determined. Notably, the nitrogen content of municipal biochar was substantially lower than that of urea-intercalated biochar. FTIR analysis of the intercalated biochar was compared with the original biochar, revealing the emergence of numerous absorption sites in regions corresponding to amides (3600 - 3300 cm-1), carboxylic acid groups (1750 - 1700 cm-1), nitrile groups (2260 - 2210 cm-1), and hydroxyl groups (OH) at 2950 cm-1. These features were absent in the FTIR results of the original municipal biochar. The predominant peak in the municipal biochar spectra was attributed to the COOH group at a wavelength peak of 1630 cm-1. The outcomes of this study validate the feasibility of enhancing the nitrogen content of biochar through intercalation. Furthermore, the presence of numerous functional groups in urea-intercalated municipal biochar fertilizer suggests its potential as a slow-release nitrogen fertilizer. Nonetheless, additional research is warranted to confirm its efficacy.*

KEYWORDS: FTIR; Municipal biochar; Urea intercalated biochar; Functional group.

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INTRODUCTION

The utilization of biochar as a sustainable technology for enhancing the fertility of highly weathered or degraded tropical soils has garnered global attention ((Lehmann and Rondon, 2006). Biochar applications exhibit the potential to improve plant growth by enhancing various soil characteristics (Yamato *et al.*, 2006, Uchimiya *et al.*, 2011) , including physical attributes (such as bulk density, water-holding capacity, and permeability), chemical properties (including nutrient retention and availability), and biological factors (Glaser *et al.,* 2002; Lehmann and Rondon, 2006, and Chen *et al.*, 2011) and collectively contributing to increased crop productivity (Lehmann and Rondon, 2006, Biederman and Harpole, 2013). Additionally, biochar applications have shown promise in mitigating soil N2O and CH4 emissions through carbon sequestration (Van Zieten *et al.*, 2009, and Cayuela *et al.*, 2014).

Despite its recognized benefits, the use of biochar is still in an experimental stage in Nigeria. Some researchers have employed it in charcoal form, witnessing improvements in both the physical and chemical properties of the soil (Ndor *et al.*, 2010). Arable lands throughout Nigeria are experiencing declining soil productivity due to continuous agricultural practices without adequate restorative measures (Ndor *et al.*, 2010). Furthermore, the consistent application of mineral fertilizer has led to nutrient imbalances within the soil, resulting in the leaching of NO_3^- from ammonium fertilizers, posing a threat to groundwater contamination and the environment (Bhargava and Sheldarkar, 1993; Ozacar, 2003; Laird *et al.*, 2010).

Biochar application presents a viable alternative. However, its nitrogen content is inherently low, as it is susceptible to loss through the pyrolysis reaction, especially at higher temperatures. Therefore, the augmentation of nitrogen content in biochar is of urgent concern. While some attempts have been made to manually mix nitrogen fertilizer with biochar, these methods are labor-intensive, and the homogeneity of the mixture remains uncertain. In an effort to enhance the nitrogen use efficiency of certain mineral fertilizers, particularly Urea, coatings with substances like neem, tar, and starch have been explored (Smith and Harrison, 1991). Additionally, inorganic materials such as zeolite, montmorillonite, and halloysite minerals have been utilized to coat Urea, thereby regulating nitrogen release, but these techniques tend to be cost-prohibitive.

In developed countries, the intercalation of nitrogen fertilizer with biochar or other organic materials has proven effective in improving soil nitrogen content and the efficiency of applied fertilizers. However, this method has yet to be attempted in Nigeria. This paper presents a characterization study of urea intercalated biochar, with the objective of assessing its feasibility for use in Nigerian soils. The study's primary goal is to identify structural distinctions between Municipal Biochar (MB) and Urea Intercalated Municipal Biochar (UIMB) using chemical methods.

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MATERIAL AND METHODS

This study was conducted at the soil laboratory of the department of soil science and land resources management, faculty of agriculture, Obafemi Awolowo University, Ile-Ife, Osun State, Nigeria.

Preparation of Urea Intercalated Biochar.

Urea solution was formulated through the dissolution of 20 grams of fertilizer in 20 ml of deionized water, establishing a weight-to-volume ratio of 1:1 (20 g: 20 ml). The resulting mixture underwent thorough blending, after which it was heated on a hot plate at a constant 105°C with continuous stirring until all crystals had dissolved completely. Subsequently, 20 grams of Municipal biochar were meticulously combined with the urea solution and subjected to drying in a hot air oven at 65°C until all moisture had completely evaporated. To this mixture, one gram of adhesive polymer, starch, was added and allowed to air dry (Manikandan and Subramanian, 2013). The product derived from these procedures was pulverized and employed as a urea-intercalated biochar fertilizer, referred to as UMBF. The ensuing steps involved the determination of various physical and chemical properties for both the biochar and urea-intercalated biochar, including pH levels, organic matter content, total nitrogen (N), available phosphorus (P), and electrical conductivity.

FT-IR Analysis.

Both the biochar and the resulting intercalated fertilizer product underwent analysis utilizing Fourier Transform Infrared Spectroscopy (FTIR) within the spectral range of 400 to 4000 cm–1. This analysis was conducted employing the Attenuated Total Reflectance (ATR) technique at the Institute of Agriculture, Research and Training (IAR&T) in Ibadan.

RESULTS AND DISCUSSION

Selected properties of urea fertilizer, municipal biochar and urea intercalated biochar

Results of some chemical properties of urea fertilizer (UF), municipal biochar (MB) and urea intercalated biochar were presented in Table 1.

Table 1: Selected properties of Urea (UF) and Urea Intercalated fertilizer (UMBF).

Treatment	pH (0.01 M CaCl2)	Total Nitrogen (%)
MB	8.0	3.36
UF	8.5	46
UMBF	8.7	24.5

The pH values of 8.5, 8.0 and 8.7 were recorded for urea fertilizer, municipal biohar and intercalated material respectively. The values fall in the alkaline region thus confirming its potential as a liming material with the exception of urea fertilizer which usually leave acidic residue due to the biochemical transformation of amide to nitrate with release of proton into

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the soil system. The nitrogen content of municipal biochar was very low as previously was reported (Glaser et al., 2002; Lehmann and Rondon, 2006; Chan et al., 2008). Great improvement in nitrogen content was observed when the biochar was intercalated with Urea fertilizer.

The FTIR results of municipal biochar and urea intercalated municipal biochar were presented in Figures 1 and 2.

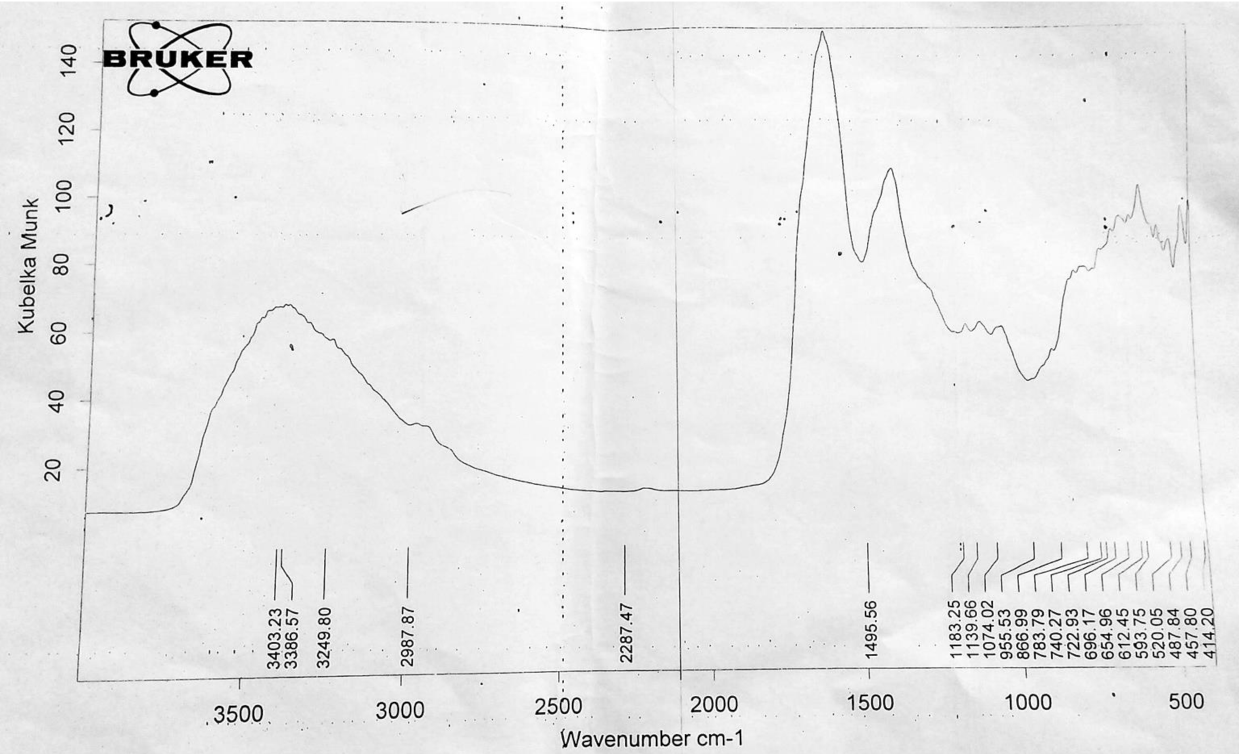

Fig.1. FTIR spectra (4000–400 cm-1) for Municipal biochar

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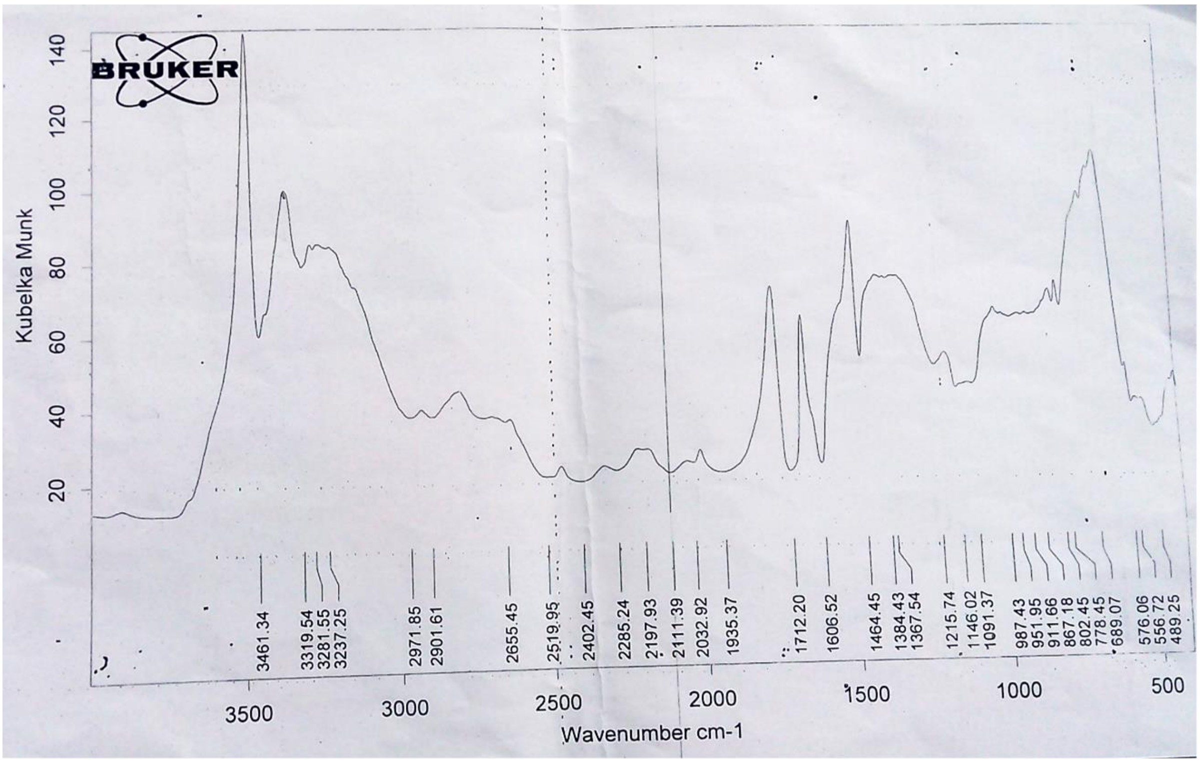

Fig. 2 FTIR spectra (4000–400 cm^{-1}) for urea-intercalated municipal biochar (1:1)

The FTIR spectra clearly indicated OH stretch at wavelength between 3000 – 3500 cm-1 (Fenglin *et al.*, 2015), the presence of Aliphatic group of C – H wavelength at 2287.47 cm-1 (Fenglin *et al.*, 2015), and carbony group between 500 – 1000 cm-1. Additionally, the FTIR spectrum of urea intercalated fertilizers revealed appearance of so many absorption sites in region corresponding to amides (3600-3300 cm-1), carboxylic acid group (1750-1700), nitrile group (2260 -2210), hydroxyl group (2950 cm-1) which were not visible in FTIR result of original municipal biochar. The dominant peak at 1630 cm-1 in municipal biochar spectra belongs to COOH group . It is therefore envisaged that adsorption of nitrogen on organic functional groups will slow down the release of N from the fertilizer during the plant cultivation.

The results of this study confirmed feasibility of improving nitrogen content in biochar through intercalation. In addition, the presence of many functional groups in urea intercalated municipal biochar fertilizer is an indication of its potential for a slow release of N, however more studies should be carried out to ascertain its agronomic effectiveness.

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CONCLUSION

The findings of this study validate the potential for enhancing nitrogen content in biochar through intercalation. Furthermore, the identification of various functional groups in urea-intercalated municipal biochar fertilizer suggests its suitability for a controlled and gradual release of nitrogen. However, additional studies are warranted to confirm its agronomic efficacy.

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