



ABILITY OF UREA IMPREGNATED BIOCHAR FERTILIZERS FOR SECURING THE SLOW RELEASE OF NITROGEN IN SOILS – PRELIMINARY STUDY

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Abstract- Nitrogen use efficiency of conventional fertilizers hardly exceeds 30-35 % on crop production. An experiment was conducted to develop biochar based slow release nitrogen fertilizer in order to improve the use efficiency. Slow pyrolysed (350°C) biomass material was low in surface area, cation exchange capacity and ion exchange properties. *Prosopis juliflora* hard wood based biochar recorded maximum recovery (30%) and carbon (>70%) than other crops. Sorption of four different forms of nitrogen showed higher adsorption characteristics. The amount of desorbed $\text{NH}_4^+\text{-N}$ increased as pH and initial NH_4^+ concentration increased. Biochar fabricated nitrogen reduced the N-lose of ammonia volatilization. The nitrogen loading efficiency (16.3 - 18.5 %) was confirmed, using Powder X-ray diffraction (XRD), Fourier Transform Infra-Red Spectroscopy (FT-IR), Raman Spectroscopy and Scanning Electron Microscopy (SEM). Nitrogen release pattern of 18 days study confirmed that the slow release characteristics of biochar based N fertilizer

Key words- Biochar, Nitrogen, Production, Slow Release Fertilizer, Release Pattern.

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Introduction

Nitrogen considered as “*element of the sun*”. It is important constituent of chlorophyll and which captures the energy of the sun for aiding plant growth. It is a key to increase agriculture production and plays significant role in photosynthetic activity as well as crop yield. The productivity of current year decreased than last year on accordance with “*Law of diminishing marginal returns*” stated that with reference to fertilizers usage in agriculture, “Fertilizer use improves crop production and productivity of farms and gardens; but at some point, adding more and more fertilizer improves the yield less per unit of fertilizer, and excessive quantities can even reduce the yield” and fetches illness to our mother soil/earth. However, nitrogen is found in plants to the extent of from 1 to 4%. Desorbed nitrogen had been leached as nitrate in soil, ground water and volatilized as ammonia in atmosphere. Increasing nitrogen losses in soil and atmosphere and particularly with increased use of nitrogenous fertilizers for crop production and intermittently causes pollution in water system [1]. Hence, we must take more steps on improving the efficiency of fertilizers, to mitigate the imprudent conventional fertilizer application and its impacts on crop production. Biomass based fertilizers development will reduce the ill effects and increases nitrogen use efficiency.

Prosopis juliflora- (Swartz) DC (PJ) is a small tree and native of Mexico and it was first introduced to India in 1877 where on it had become an invasive weed. It is selected for biochar production and also availability is high on wastelands, pond or lake border area. Commonwealth Scientific and Industrial Research Organization (CSIRO), Australia referred biochar as a carbonized material. It is by-product from slow pyrolysis of biomass material and also it has been utilized as amendment for soil and plant nutrition [2]. Recently, a few studies provided the importance on nitrogen cycle [3], its role as an alternative N-fertilizer for ammonia capture [4], bioavailability studies on adsorbed ammonia [5] and effect on nitrogen and carbon dynamics in soil [6] and arid soil management. Black

coloured solid carbon, which performs as soil carbon sequestration agent [7]. Studies explained that the biochar production could be possible with different cellulosic biomass and wastes. Agricultural and forestry biomass residues (500 - 550 MT), papers, sewage sludge, plant or animal manure and city or urban wastes, are heated under limited oxygen present and or nil in a specially designed pyrolysis-stove at different temperature from 300°C to 750°C and produced stable carbon [6]. Utilization has been increased as a soil amendment/conditioner to improve physical properties of the soil and fertilizer use efficiency. Its application also significantly reduces greenhouse gas emission. It is non-selective adsorption potential. In preparation of fertilizer, biochar act as adsorbent and synthesized using inorganic components. From this idea, following objectives were studied on preparation, sorption and development of biochar based N fertilizer using four forms of N sources and characterized with nutrient release pattern.

Material and Method

Biochar Production

Prosopis juliflora (PJ) hardwoods were collected from Krishnampathi Lake, Coimbatore, Tamil Nadu. Biochar was produced by slow pyrolysis method using hardwoods at 350°C for an hour under the absence of oxygen with resident time of 8h. Fine ground through milling machine of 4 times and spreads under polyhouse of a 72 h for moisture evaporation. Biochar was sieved through <0.5 mm sieve and dried for a few hours at 60°C.

Sorption study

Nitrogenous fertilizers Ammonium Sulphate (AS), Potassium Nitrate (PN), Urea (U) and Ammonium Nitrate (AN) utilized for sorption study. One gram of biochar with 30 ml varying concentrations from 20 to 200 mM of N were shaken for 48h. The initial ammonium concentration in the extracting solution varied from 100 to

600 mg L⁻¹. The polypropylene tubes were laid horizontally on a mechanical shaker and agitated continuously for 48 h followed by centrifugation at 2,000 rpm for 10 min. The supernatant was decanted and exchangeable ammonium determined by modified kjeldahl using an UV- Spectrophotometer [8]. Adsorbed ammonium quantified by reduction of ammonium in the solution.

Fertilizer fabrication

Novel slow release fertilizer prepared with urea solution and emulsion heated on a hot plate at 105°C with constant stirring for destroying crystals. Equal amount (w/w) basis of biochar and urea emulsion was mixed and dried in a hot air oven at 65°C till the moisture is completely exhausted. 10% of adhesive polymer (starch) was added to the mixture and air dried, powdered and used as a slow release fertilizer.

Characterization

The characterization was done for the biochar before and after loading of nitrogen [Table-1]. The pH, Electrical Conductivity (EC) was determined using a saturated paste approach 1g biochar: 6.25 ml of water. Moisture content was estimated by loss of ignition method. Bulk density, particle density, pore space volume and porosity were determined by cylindrical method. Total carbon, total nitrogen and total phosphorus were analysed using standard procedure [9]. Cation exchange capacity (CEC) of sample was estimated by silver-thiourea method using ICP-AES [10]. Particle size distribution analysis, zeta potential distribution was measured by (Laser light scattering technique) using Horiba nanoparticle analyser. A known quantity of (0.5 mg) sample had taken in 50 ml beaker and added 20 ml of distilled water. It is sonicated for 10 minutes in ultrasonic processor with 10 seconds On-Off cycle. Add a drop of 0.1% Tween 80 after sonication to avoid agglomeration of particles. Particle size distribution was performed at 25°C at scattering angle 173° with glass cuvettes and cumulative analysis of dispersion determined the mean hydrodynamic diameter. The surface charge and zeta-potential distribution determined based on principle of electrophoretic mobility of particles in the colloidal suspension, which were performed using folded capillary cells in automatic mode. X-ray diffraction (XRD) patterns were recorded using a Bruker D8 Focus X-ray powder diffractometer at the Karunya University using CuK α radiation (λ = 0.154 nm) over a 2 θ range of

3–90°, with a step size of 0.02° and a step time of 1 s to identify any crystallographic structure in the fine grained sample and crystalline compounds were identified by comparing diffraction data against a database compiled by the joint committee on powder diffraction and standards. Raman Spectra are collected with an instrument Raman Systems Model- R- 3000- QE and RSIQ software version: 1-0-4-9. Agiltron Inc. Technique based on inelastic scattering of monochromatic light, usually from a laser source 785 nm. Dried samples and dark room condition is required for raman analysis. Samples were spread to an extent of 1 cm² in glass vial and raman probe was placed for data calibration. Fourier Transform Infra-Red Spectroscopy using attenuated total reflectance (ATR) technique used for determination of surface organic functional groups, chemical nature and molecular bonding of sample. Generally, it measures a range from 600 to 4000 cm⁻¹. To obtain the observable FT-IR spectra, sample was ground and mixed with KBr to 0.1% and then pressed into pellets and quantified. SEM FEI QUANTA 250 was used to characterize the size and morphology of the biochar and developed fertilizer (biourea) with gold sputter-coated. Various magnifications were used and compared the biochar structure and surface phenomena of the sample beam energy 20 kV. Energy dispersive X-ray spectroscopy (EDAX) images can provide semi-quantitative elemental composition of particular selected portion of sample. About 40 - 50 mg of sample was dusted on the carbon conducting tape for image. The surface sampling depth of 1-2 microns recorded at FEI QUANTA 200 EDAX.

Nitrogen release pattern

Nitrogen release pattern was studied with designed the percolation reactor system (PRS) [11]. PRS made up of glass and cylindrical in shape (internal diameter= 2.5 cm, height= 15 cm) with 4 pore holes and experiment was conducted under laboratory. Circulation of water is necessary. Processed ten grams of black soil with one gram of biochar or fertilizers (biochar, biochar + urea, biourea and urea alone) homogenised and which comprised of 4 treatments along with control (soil alone). The 50 ml of deionized water was added to above from 1-20 days. Solutions were collected to determine ammonium and nitrate ions as per water quality standard protocol [8]. By this technique, comparative study of slow release tendency for adsorbents and fertilizers would be estimated, to develop a novel fertilizer with statistical analysis using standard method [12].

Table - 1 Physio-chemical properties of biochar and biourea (1:1)

Property	Biochar	Biourea
pH(1: 6.25ml ratio)	9.16	8.1
EC(dSm-1) (saturated paste)	0.15	0.23
Moisture (%)	6	4
Bulk Density(Mg m-3)	0.5	0.61
Particle Density(Mg m-3)	0.71	0.67
Particle size distribution (nm)	6000-8000	8000-10000
Zeta potential (mV)	-0.0159	-0.01
Pore space volume(ml)	12	1.2
Pore space (%)	30	15
Total Organic carbon (%)	14.31	ND
Total nitrogen (%)	1.56	16.5
Total phosphorus (%)	0.07	0.04
Total potassium (%)	3.12	0.09
Calcium *(%)	12	0.2
Magnesium* (%)	0.42	0
Sodium *(%)	0.48	0
CEC(cmol(p+) kg-1)	20	11.5
Inorganic carbon (%)	67.24	

Note: * Quantified through SEM- EDAX

Results and Discussion

Biochar is produced through slow pyrolysis (350°C) with *Prosopis juliflora* hard woods biomass material under limited or non-oxygenated environment. Higher recovery recorded (28-30 ±2 per cent) than other hard wood biomass. It may be biomass type, initial moisture (less than 10%) content and pyrolysis temperature. Similar results were observed [6] and oxidation process, heating influenced the surface nutrient loss.

Biochar characterisation

Structural variations of biochar particles were determined by powder X-ray diffraction (XRD). XRD patterns of biochar indicated the broad peaks, which exhibit disordered characteristics of biochar particles [Fig-1]. The observed biochar micro sized particle XRD patterns profile matched exactly to standard semi crystalline to quasi crystalline carbon patterns. The peaks at $2\theta = 23^\circ$, 26.8° , 39.5° , 47.6° , 64° , 75.8° and 81.5° was observed for biochar and showed the maxima at $2\theta = 26.8^\circ$ which corresponds to d spacing = 3.32 Å. The XRD data confirms that the particles are mostly micro sized carbon material.

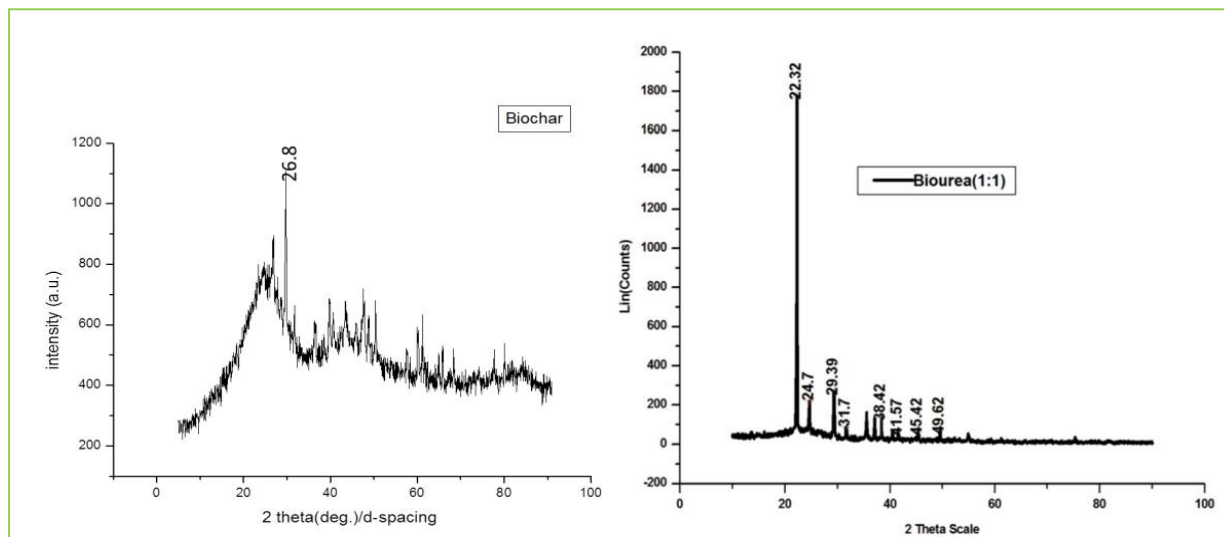


Fig-1 Powder X-ray diffraction (XRD) patterns of biochar and biourea (1:1)

The peaks represent the hexagonal structure. There is overlap of broad reflections from the biochar and which informed us it disordered material. It also suggest that the especially semi crystalline phase samples have intermediate structures between graphite and a few amorphous state called random layer lattice structure or turbostratic structure. Biochar particles were

produced raman shifts [Fig-2] between 190 to 1965 cm^{-1} . Seven shifts were observed at 190, 799, 1690, 1742, 1779, 1885 and 1965 cm^{-1} . The above raman shifts were lattice internal vibrations, Si-O, water molecules, C=C, aromatic C-C and C=C functional groups of biochar respectively. It reflects that the all the nutrients excited under high temperature (350°C).

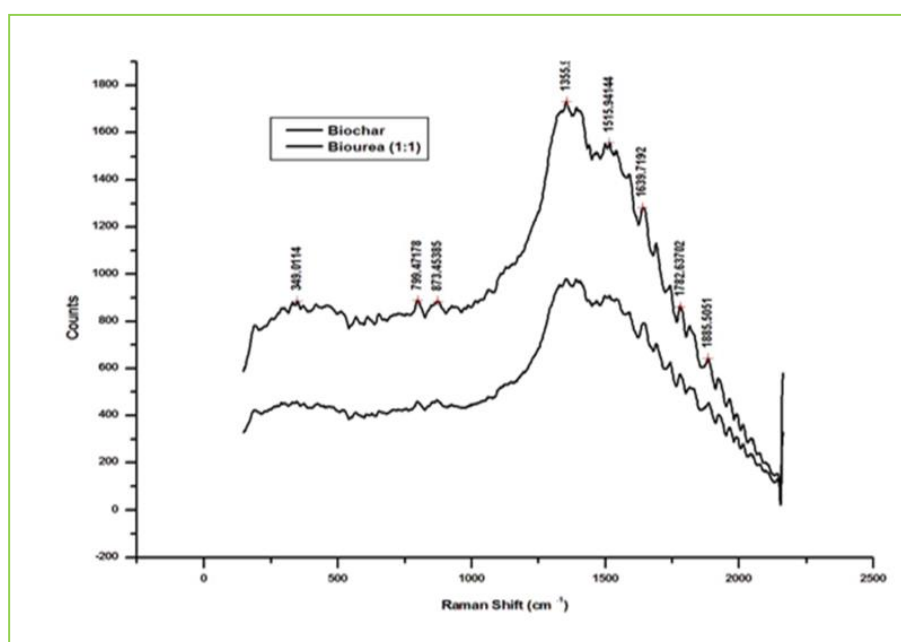


Fig-2 Raman Shifts for biochar and biourea (1:1)

Fourier Transform Infra-red Spectroscopy (FT-IR) spectral reflections were taken in the vibrations of lattice biochar at a 4000-400 cm^{-1} . The results obtained that

the spectra of biochar [Fig-3] agreed well with the spectra of reference of carbon. Maximum peaks observed in four regions 4000- 3400, 2900-1900, 1610-1030

and 880-470 cm^{-1} . The peaks were observed at 3977, 3907, 3734, 3695, 3479 and 3444 cm^{-1} were caused by O-H functional groups of biochar. The reflections at 2924, 2854, 2380 and 2314 cm^{-1} are caused by C-H vibrations of biochar. The peaks at 1990 and 2017 cm^{-1} were produced by S-H functional groups of biochar. The peaks at 1608, 1585 and 1450 cm^{-1} produced by aromatic chain vibrations of biochar. The peak at 1512 cm^{-1} was produced by $-\text{NO}_2$

asymmetric vibrations of biochar. The reflections observed at 1273, 1195, 1168, 1099 and 1033 cm^{-1} were produced by C=S functional groups of biochar. The peaks were observed at 1323, 875, 601 and 578 produced by alicyclic and aliphatic chain vibrations of biochar. The structural peaks observed at 547 and 470 cm^{-1} caused by Si-O-Si functional groups of biochar.

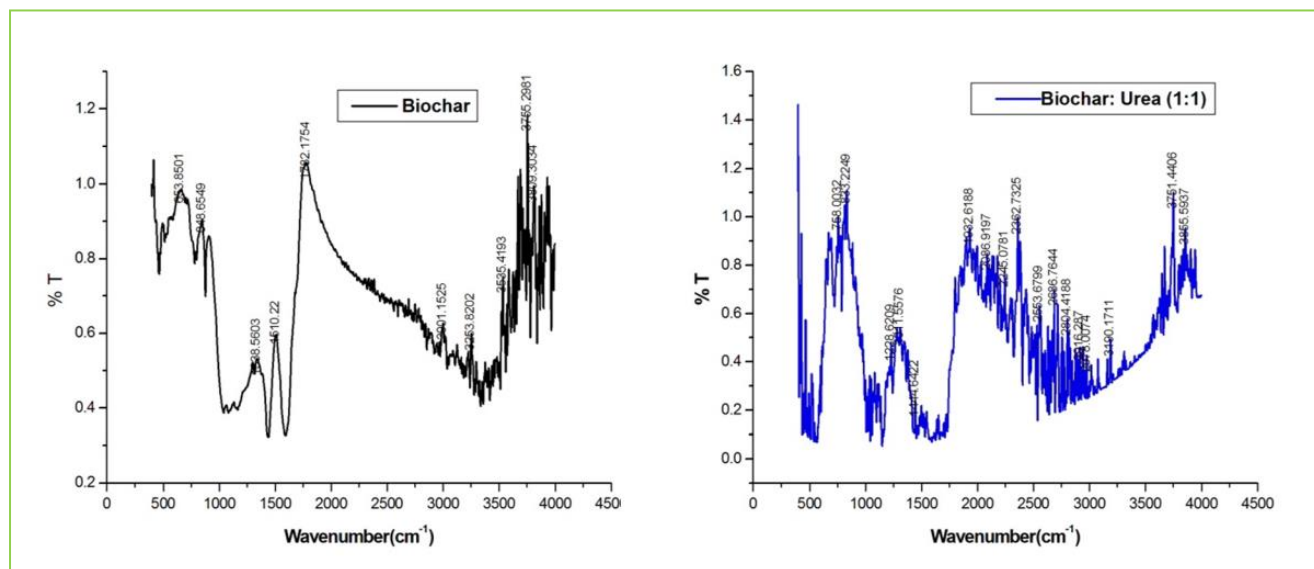


Fig-3 FT-IR Spectra for biochar and biochar:urea (1:1)

A SEM image [Fig-4] of crudely pulverized prosopis based biochar clearly reflects that the hard wood biomass pattern of higher resolution (data not shown). Different shapes of broken edges with grittiness at micro-scale level and the particles were aggregated in random manner. Macro and micro porous structure were observed on surface scatter manner. SEM-EDAX analysis indicated the

presence of elements of small surface area composed of carbon (86 %), oxygen (12 %) and calcium (0.2 %) presumably in mineral form. The results were similar to various [6,13,14] studies. They reported lower content of calcium (1%) and magnesium (0.036 %) on prosopis. Carbon content (68.6 %) and XRD patterns with lower number of ash were produced on gasification of sorghum biomass.

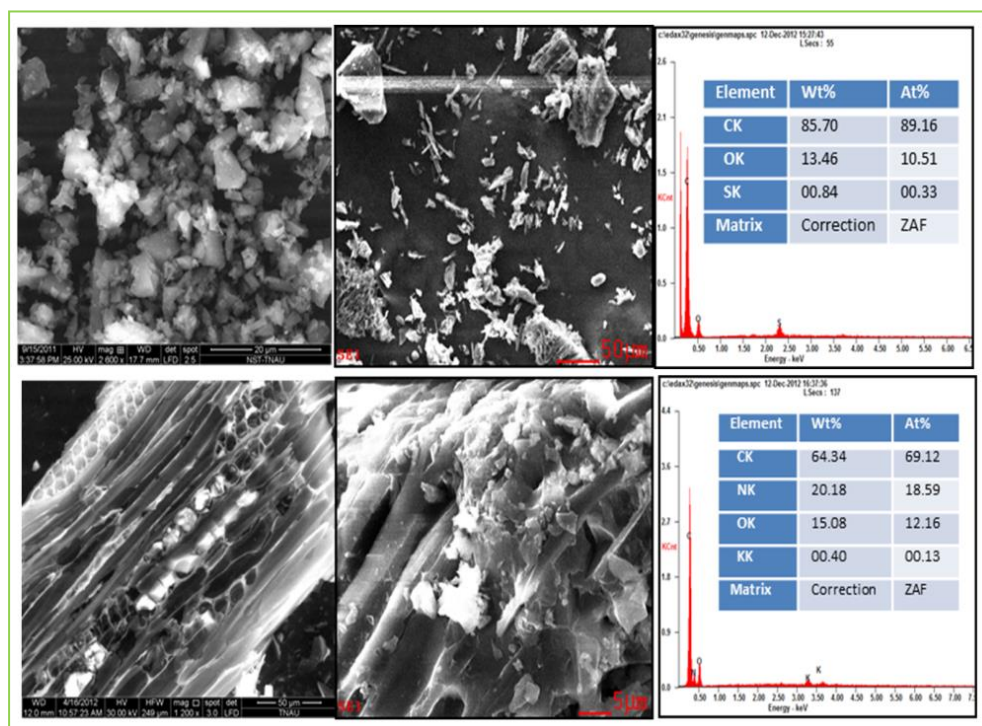


Fig-4 SEM image and EDAX micro analysis report of biochar and biochar:urea (1:1)

Sorption on nitrogen forms

The purpose of this sorption experiment with different forms of nitrogen fertilizers was to identify the suitable conventional fertilizer for fabrication of novel

impregnated fertilizer with biochar. Desorption was considered coexistence process of adsorption. Desorption curves of Urea, AS, AN and PN on biochar were explained that the on both forms of ammonium and nitrate ions at 20- 200

mM L⁻¹. Desorption was highest on higher concentration. Cumulative desorption of NH₄⁺-N among the nitrogenous fertilizers are 19100, 1600, 36900 and 8600 mg l⁻¹ for AN, PN, AS and Urea respectively. Desorption of NH₄⁺-N showed a linear pattern correspondingly with the concentrations [Fig-5]. Cumulative desorption of NO₃⁻-N among the nitrogenous fertilizers are 13500, 8300, 7200 and 1100 mg l⁻¹ for AN, PN, AS and Urea respectively. Desorption NO₃⁻-N increased with increasing concentrations [Fig-5]. Biochar order of desorption

sequence of ammonium ion trend was decreased from Urea < AS < PN < AN and for nitrate ion desorption trend was also decreased from Urea < PN < AS < AN. The results of mean data showed that the conventional urea for further fertilizer preparation experiment. The findings were corroborate with [5,15,16] demonstrations and ammonium ion adsorption higher than nitrate ion. High negative surface charge may be reason for adsorption of cation.

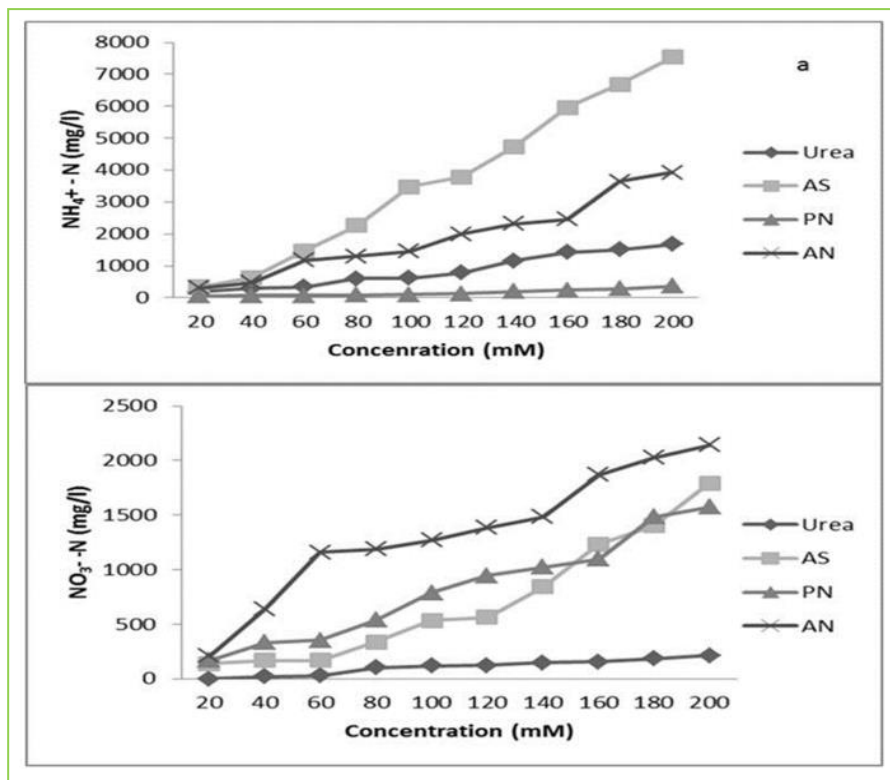


Fig-5 Desorption of ammonium and nitrate nitrogen on biochar

Biourea fertilizer fabrication

Impregnation of urea was done with different w/w ratio of biochar. The results on total N revealed that total N varied significantly due to the influence of treatments. The highest total N (16.3 to 18.5 %) was found in 1:1 w/w ratio of impregnated biochar fertilizer. The lowest total N (0.2%) was observed in 1:10 w/w ratio of impregnated biochar. Biochar loaded with urea emulsion adsorbs N on micropores and starch kept N intact. The fabricated fertilizers of 1:1 w/w ratio of urea impregnated adsorbents were further characterized. The results were in confirmation with [17,18] researchers. The loading efficiency of urea on montmorillonite varied from 1.8 to 21.8 on 1:1 ratio of intercalation.

Biourea characterisation

The observed biourea Powder X-Ray Diffraction (XRD) patterns profile were not similar to standard patterns of fertilizer [Fig-1]. The peaks at $2\theta = 22.3^\circ, 24.7^\circ, 29.5^\circ, 31.7^\circ, 38.4^\circ, 41.5^\circ, 45.4^\circ$ and 49.6° was observed for biourea and showed the maxima at $2\theta = 22.3^\circ$ which corresponds to d spacing = 3.98 Å. After impregnation of urea we were observed the reduction of d spacing and which indirectly confirms the adsorbed ammonium or other ions on its interlayer lattice. The XRD data confirms that the fabricated biourea particles are mostly micro sized and maxima reduction was observed. The peaks represent those structural changes on biochar. Biourea particles were produced raman shifts [Fig-2] between 180 to 1965 cm⁻¹. There are seven shifts were observed at 187, 797, 1356, 1646, 1690, 1742, 1781, 1888, 1922 and 1965 cm⁻¹. The Raman shifts at 187 cm⁻¹ caused by lattice internal vibrations. The raman shifts 799 cm⁻¹ produced by Si-O functional groups of fabricated biourea. The raman shifts 1356 cm⁻¹ caused by C-NO₂ asym. The raman shifts 1646 cm⁻¹ caused by the C-N vibrations. The raman shifts 1690 cm⁻¹ caused by the C-O and 1742 cm⁻¹ pertains

to water molecules the C=C functional groups of biourea. The raman shifts 1781 cm⁻¹ produced by aromatic C-C functional groups. The raman shifts 1888, 1922 and 1965 cm⁻¹ produced by C=C stretching vibrations of biourea. The Fourier Transform Infra-red Spectroscopy spectra were taken in the lattice vibration of biourea, 400-4000 cm⁻¹. The results obtained that the spectra of biourea [Fig-3] slightly agreed well with the spectra of reference of biochar with peak changes. Maximum peaks observed in 3 regions 750-1450, 1900-3200 and 3700-3900 cm⁻¹. The peaks were observed at 758, 833, 1228, 1311, 1444, 1932, 2086, 2286, 2245, 2362, 2553, 2686, 2804, 2916, 2978, 3190, 3751 and 3855 cm⁻¹. The peaks represented corresponding functional groups of 758 (SiO₂), 833, 1228, 1311 and 1444 (Nitrate) 1932 and 2086 (C-C), 2245 and 2362 (CO₂), 2553 and 2686 (CH₂), 2804, 2916 (C-H stretch), 2978 and 3190 (Ammonium) 3751 and 3855 cm⁻¹ (N-H) of biourea. A Scanning Electron Microscopy (SEM) image of biourea hardly reflects any shape and structure [Fig-4]. Different shapes of broken edges with grittiness at micro-scale level and the particles were aggregated in random manner. Some parts of showed patch appearance and other showed white glooms were presence of urea on biochar. The observed Energy dispersive X-ray spectroscopy (EDAX) of small surface area composed of carbon (64 %), nitrogen (20 %) oxygen (15 %) and potassium (0.4 %). The results were in confirmation with [16-20] researchers. Biochar with well-organized macro pores on outer surface with varying shapes and size on different temperature pyrolysis. Composition of nutrients highly varied on different biochar. XRD patterns of urea-montmorillonite 1:1 intercalation recorded maximum lameller spaces and which confirmed the adsorbed ammonium.

Percolation Reactor study

The release of NH₄⁺-N and NO₃⁻-N from adsorbents and fabricated fertilizers are

presented [Fig-6]. The data illustrated that the $\text{NH}_4^+\text{-N}$ release from urea, biochar + urea (1:1) and biourea (1:1) were 65, 50 and 28%, respectively, in the fertilizer formulations detected on the first day of the experiment. The first day of leachate

solutions had 2716, 3842, and 2910 mg L^{-1} of $\text{NH}_4^+\text{-N}$ and 490, 420 and 280 mg L^{-1} of $\text{NO}_3^-\text{-N}$ in urea, biochar + urea (1:1) and biourea respectively. The release of N from urea (irrespective of $\text{NH}_4^+\text{-N}$ and $\text{NO}_3^-\text{-N}$) ceased to exist within 4 days

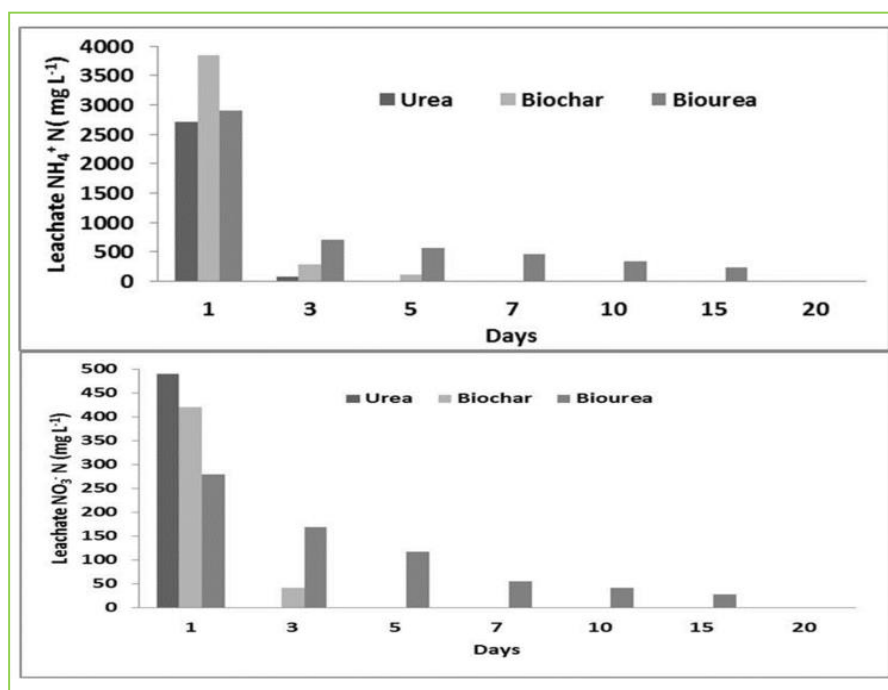


Fig-6 Desorption of ammonium and nitrate nitrogen on biochar

of experiment. The same set of treatments showed N release for 4, 5 and 18 days, respectively. Continuous presence of tubular or through (T) pores increased the adsorption capacity and retained for 4-18 days. The results mean data exhibited the slow release characteristics of urea loaded biochar fertilizers. The results were confirmed with [17-18] researchers. They reported that the acidic biochar captures ammonia on pores and confirmed that acid functional groups. Nutrient impregnated charcoal released up to 360 h under continuous flow state and urea-montmorillonite nano composite on its simple process of exfoliates clay lamella into urea matrix and its dissolution behavior explained slow release pattern.

Conclusion

Pyrolysed bio-materials used in adsorption are highly responsive due to the presence of inherent, persistent micro-pores and which related to their continuous tubular or tunnel internal structure. Acidified biochar have the capability to absorb the ammonium ions. To adsorb required quantity of $\text{NH}_4^+\text{-N}$, it is necessary to perform some useful changes in the internal and external surface. However, continuous drying process at high temperature/steam and chemical treatment like diluted acid leaching under controlled manner to modify their conditions their structure. Characterisation confirmed the ammonium ion presence on fabricated biourea (1:1) with maximum on surface pores. Dissolution and relatively slow release pattern was observed than conventional fertilizers under laboratory conditions. It could be additional supplement for carbon sequestration and improves nitrogen use efficiency (NUE) under less fertile or sterile soils. The developed biochar based slow release fertilizers would directly improve nutrient use efficiency and soil fertility status. Hence, trails must be conducted on various crops with protected conditions, on and off farm experiments in order to confirm the nutrient absorption.

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List of abbreviations used

XRD - X-ray diffraction

FT-IR- Fourier Transform Infra-Red

SEM- Scanning Electron Microscopy

PJ- *Prosopis juliflora*

AS- Ammonium Sulphate

PN- Potassium Nitrate

U- Urea

AN-Ammonium Nitrate

(w/w) - weight/weight

EC-Electrical Conductivity

ICP-AES –Inductively Coupled Plasma Atomic Emission Spectroscopy

ATR-Attenuated Total Reflectance

EDAX-Energy dispersive X-ray spectroscopy

PRS- Percolation reactor system