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RESEARCH ARTICLE

CHEMICAL FUNCTIONALIZATION AND CROSS-LINKING OF BIOCHAR FROM COW DUNG FOR USE AS SUPER ABSORBENT MULCH SUBSTRATE FOR AGRICULTURAL PURPOSE

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Abstract

Water scarcity significantly affects agricultural productivity, particularly in arid and semi-arid areas. There is a pressing need for developing mulching substrates that are sustainable and effective in conserving water and nutrients. In this study, cow dung collected from a farm in Thika, Kiambu County, converted into biochar through pyrolysis (burning under limited oxygen supply). It was subsequently functionalized using Ethan1,2 diol, Glycerol, Phosphoric acid that introduced hydroxyls (hydrophilic group) and phosphates to improve the biochar's affinity for water. The funtionalized biochar was cross-linked with phosphoric acid, citric acid, and urea to form a polymer-like network (gel-like structure when wet), with improved the swelling capacity and water retention.The functionalization were confirmed with the FTIR spectrum which showed peaks for -OH at 3350.73 cm^{-1}, P-OH at 788.87 cm^{-1}, P=O at 1279.5 cm^{-1}, C-N at 1045.23 cm^{-1}, N-H at 1622.80 cm^{-1}, C-H at 2919.68 cm^{-1} and C=O at 1602.5 cm^{-1}. This confirmed that the diol, phosphate and amino used for functionalization and cross-linking had been successively anchored. This was recommended as a sustainable modification of biochar to super absorbent mulch.

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Introduction:-

Background Information

Mulching is the act of covering the topsoil with a layer of materials such as grass clippings, wood chips, straw, chopped leaves, twigs, sawdust, and even paper. Their utilization as mulching materials has been used in agriculture (Martín-Closas et al., 2016). In nurseries, grass clippings have been the most widely used mulch because they are readily available, prevent excessive evaporation, and also moderate soil temperatures (Wells et al., 2016). These mulching materials have been used to improve soil moisture, beautify the land, control weeds, and improve soil fertility, but they do not retain water for long, hence the need for a more reliable mulch.

Biochar is the lightweight black residue that remains after biomass has been burnt in the absence of oxygen. It is more porous (Chrisman, 2015) and can hold more water for longer than natural mulches. It has been made in the past from biomass materials like rice husk, olive shell, crop residues, pig manure, cattle manure, and wood (Ma et al., 2019). Biochar in the soil has many benefits, some of which include increasing water infiltration, promoting better root development, reducing fertilizer run-off (especially nitrogen and phosphorus), binding sandy soil together, and improving water holding capacity (Lonappan et al., 2019). It adsorbs and immobilizes heavy metals and reduces their uptake by plants and subsequent ingestion by humans and animals (Li et al., 2018). It has carbon bonds that readily accept functional groups to form a hydrogel (Huff et al., 2016). Most of the chemically improved biochar has been used in the adsorption of heavy metals, but there is limited information on the use of chemically improved biochar in agricultural practices.

In Kenya, the intensive livestock farming system is a major source of environmental pollution, especially in urban areas where people live in small plots of land. Even though cow dung has been used for biogas production (Mwangi et al., 2019), the biogas slurry poses a problem in its disposal.

There are many adverse impacts of climate change, especially in the agricultural sector, which is heavily affected by drastic weather and climate change, resulting in food insecurity. There is therefore a need to search for an amicable solution to mitigate the impact of climate change on agriculture, which is the backbone of our economy. This can be done through the development of super absorbents such as functionalized and cross-linked cow dung biochar with the ability to retain water, nutrients, and keep the crops and trees growing even when the weather conditions are not favorable.

Cow dung is the undigested residue of plant matter that passes through the digestive system of bovine animal species. It is rich in nutrients like nitrogen, phosphorus, potassium, and high levels of ammonia (Randhawa et al., 2011). It also consists of lignin, cellulose, and hemicelluloses (Singh et al., 2014). It has several benefits, some of which include burning as fuel, mosquito repellent, plaster for buildings, bio-pesticides, and as a cleansing agent (Mwangi et al., 2019). Cow dung manure increases humus, and this results in better water seepage and water binding capacity, together with an improved cation storage capacity (Bio-fertilizers, 2014). It is also used for making biogas, which is used for cooking, thus lowering dependency on charcoal, firewood, and fossil energy (Guo et al., 2013). Cow dung poses a problem in its disposal since it is produced in large volumes, especially in town set-ups where people live in small plots of land (Dupla et al., 2024).

Biochar is a form of charcoal produced through the thermochemical conversion of biomass under low oxygen conditions known as pyrolysis (Guo et al., 2013). When air is excluded, oxygen for burning is removed from the biomass, leading to its reduction to carbon single bonds of charcoal (Chen et al., 2017). In ordinary burning, biomass would be oxidized into ash, carbon (IV) oxide, vapors, and other gases (Ekebafe et al., 2013). It has many benefits in the soil, like increasing water penetration and water retention capacity, improving soil configuration, thus improving root development, which leads to better yield (Chrisman, 2015). Biochar-amended soils improve nitrogen use efficiency, adsorption of ammonium, phosphate, and calcium ions (Richardson et al., 2011). It enhances fertility, nutrient retention, soil biological activity and diversity, and reduces fertilizer run-off, especially nitrogenous and phosphate (Zahra et al., 2021).

Biochar from animal waste is generally high in nutrient content, particularly nitrogen, phosphorus, calcium, and magnesium, compared to plant residue. This has been stated by Utomo and Nugroho (2011), whose research looked at the production of a simple biochar from coconut and animal waste and its application in Lombok and the other eastern parts of Indonesia. They also found that biochar made from animal waste was less alkaline (pH 8.9) compared to that from coconut shell, which had a pH of 9.9 (Utomo et al., 2011).

Chemical functionalization of biocharmaterials

Use of organic acids and alkali solutions has been reported in the pretreatment of biochar materials to improve the internal surface area (Usman et al., 2015). Chemical modification alters the outside chemistry of biochar, thereby leading to improved water holding capacity in comparison with un-functionalized biochar. The activated and functionalized biochar has several applications in diverse fields, which include soil recreation, carbon dioxide sequestration, and removal of heavy metals and degradation of organic pollutants (Yakout et al., 2015).

The commonly used activating chemicals include H_2SO_4, H_3PO_2, KOH, $ZnCl_2$, and K_2CO_2. Chemical functionalization can be done by introducing specific chemical functional groups like $–SO_2H$ and –COOH into the biochar matrix (Ma et al., 2019). In the present study, functionalization of cow dung biochar by incorporating functional groups inside the polymer chain and then cross-linking it enhanced water holding capacity, nutrient adsorption, and increased swelling after water absorption.

Hydrogels are polymers that are loosely cross-linked and exhibit the capacity to swell and hold a large amount of water within their structure, but do not dissolve in it (Alkhasha et al., 2018). The capability of hydrogel to take up water arises from the hydrophilic functional groups attached to the polymeric backbone, while its resistance to dissolution arises from cross-links between its network chains (Wu et al., 2021). They can be classified depending on their physical properties, method of preparation, nature of swelling, ionic charges, rate of their biodegradation, and nature of cross-linking (Larrañeta, 2018).

Hydrogels are extensively implemented as input materials in various biomedical applications due to their spongy, stretchy, hydrophilic, and quasi-solid nature (Ahmed, 2015). They are also used in bio-separation, tissue engineering, proteomics, and chromatography, where good water-holding capacity is of prime importance (Ullah et al., 2015).

Three polymerization techniques can be applied to form a gel. They include:

- **Homo-polymeric hydrogel**, which is derived from the same species of monomer (Ahmed et al., 2019).
- **Co-polymeric hydrogel**, which comprises two or more different monomer species.
- **Multi-polymer interpenetrating polymeric hydrogel**, which results from two free cross-linking synthetic or natural polymer components (Chrisman, 2015).

Superabsorbent hydrogel acts as humus, which joins loose soil, hence forming loam soil that helps better root penetration (Ahmed et al., 2019). It increases the plant-accessible water in the soil, encourages faster growth of plants even under the best water conditions, and lengthens the survival of plants under water shortage (Ekebafe et al., 2013).

Materials and Methods:-

Sampling of Cow Dung and Pre-treatment

Cow dung was collected by random sampling from Thika in Kiambu County from Freshian breed cattle. It was sun-dried for 6–7 days until there was no change in weight. Stones and other foreign materials were removed, and then the cow dung was burnt at a temperature of 300 ºC in the absence of air to make biochar.

Preparation Procedure of Biochar

Two hundred grams of cow dung was placed in a tight-lid tin where it was burnt for 30 minutes at a temperature of 300 ºC. It was allowed to stay in that state for 10 minutes, after which it was cooled at room temperature for one hour (Pattanik et al., 2019).

Experimentation Procedure for Functionalization of Biochar with Phosphoric Acid

The reaction of biochar with phosphoric acid was performed by adding 100 cm^3 H_3PO_4 (85%) to 50 g of biochar sample at 40 ºC as described by Gyarmati et al. (2015). The system temperature was raised to 100 ºC and maintained for 40 minutes while stirring at 10-minute intervals. The reaction mixture was then cooled in an ice bath at a temperature of 15 ºC.

The mixture was decanted from the resulting suspension and washed four times with water. Ten grams of urea was added to phosphorated biochar and the temperature raised to 150 ºC for cross-linking to take place in the biochar chains. The cross-linked biochar hydrogel was then analyzed using Fourier Transform Infrared Spectroscopy (FTIR).

Functionalization of Biochar with Ethan-1,2-diol

The functionalization procedure of biochar with ethan-1,2-diol was performed as per Yakout et al. (2015) with slight modifications. Ten grams of biochar was mixed with 100 ml of ethan-1,2-diol in the presence of sulfuric acid at 120 ºC to increase surface area and porosity of biochar.

The resulting ester was then cross-linked with phosphoric acid by heating under reduced pressure in the presence of an inorganic base at a temperature of 150 ºC. The crude phosphate ester was purified by washing first with a chelating agent and then with water, after which it was dried.

Functionalization of Biochar with Glycerol

In the presence of sulfuric acid, 10 g of biochar was reacted with glycerol at 120 ºC for 2 hours to form an ester. The cross-linking method was adopted from Gyarmati (2015).

The ester was heated at 165 ºC for 10 minutes. Then 100 g of the ester was placed in a beaker with 0.1 M NaOH solution (pH 12.00), necessary for cross-linking and stability due to the alkali environment. The liquid was stirred at 250 rpm for 1.5 hours and discarded.

The residue was washed four times using 400 ml of water for 1 hour and then dried on a porcelain tray in the open for 4 hours. The treated sample was added into 600 ml of 0.6 M citric acid solution, stirred at 400 rpm for 40 minutes. The acid-treated hydrogel was washed with water to remove excess citric acid until the effluent turned non-turbid with lead (II) nitrate in buffer solution at pH 4.6. Drying was then done in an inert environment.

Procedure for Functionalization of Biochar with Phosphoric Acid and Cross-linking with Urea and Citric Acid

The hydrogel in procedure 3.2.3 was cross-linked again with citric acid. Two hundred grams of cross-linked biochar with urea was placed in 4 L of 0.1 M NaOH (pH 12.00), necessary to provide an alkali environment for cross-linking. The mixture was stirred at 250 rpm for 1 hour and the liquid discarded.

The residue was washed four times with 400 ml of water for 1 hour and then dried on a porcelain tray in the open for 4 hours. The treated biochar sample was added to 120 ml of 0.6 M citric acid solution, stirred at 400 rpm for 1 hour. The acid-treated hydrogel was washed with water to remove excess citric acid until the effluent turned non-turbid with lead (II) nitrate in buffer solution at pH 4.6. It was then dried in an inert environment.

Results and Discussion:-

Functionalization of Biochar and Cross-linking

Functionalization of Biochar with Phosphoric Acid followed by Cross-linking with Urea

Phosphoric acid was linked to the biochar chain in the form of a phosphoric acid group by ester linkage through reactions with biochar hydroxyl groups to form phosphorylated biochar. The phosphorylated biochar was then cross-linked by the use of urea. The temperature was raised to 150 ºC because the melting point of urea is 144 ºC (Martinez-Villadiego et al., 2024).

Figure 1. Reaction Scheme for the functionalization and cross-linking of biochar with phosphoric acid followed by urea

Functionalization of biochar with Ethan-1,2-diol followed by cross-linking with phosphoric acid:
It involved an esterification reaction of ethan-1,2-diol with biochar in the presence of sulphuric acid which was then cross-linked with phosphoric acid to give a phosphoric Ester hydrogel

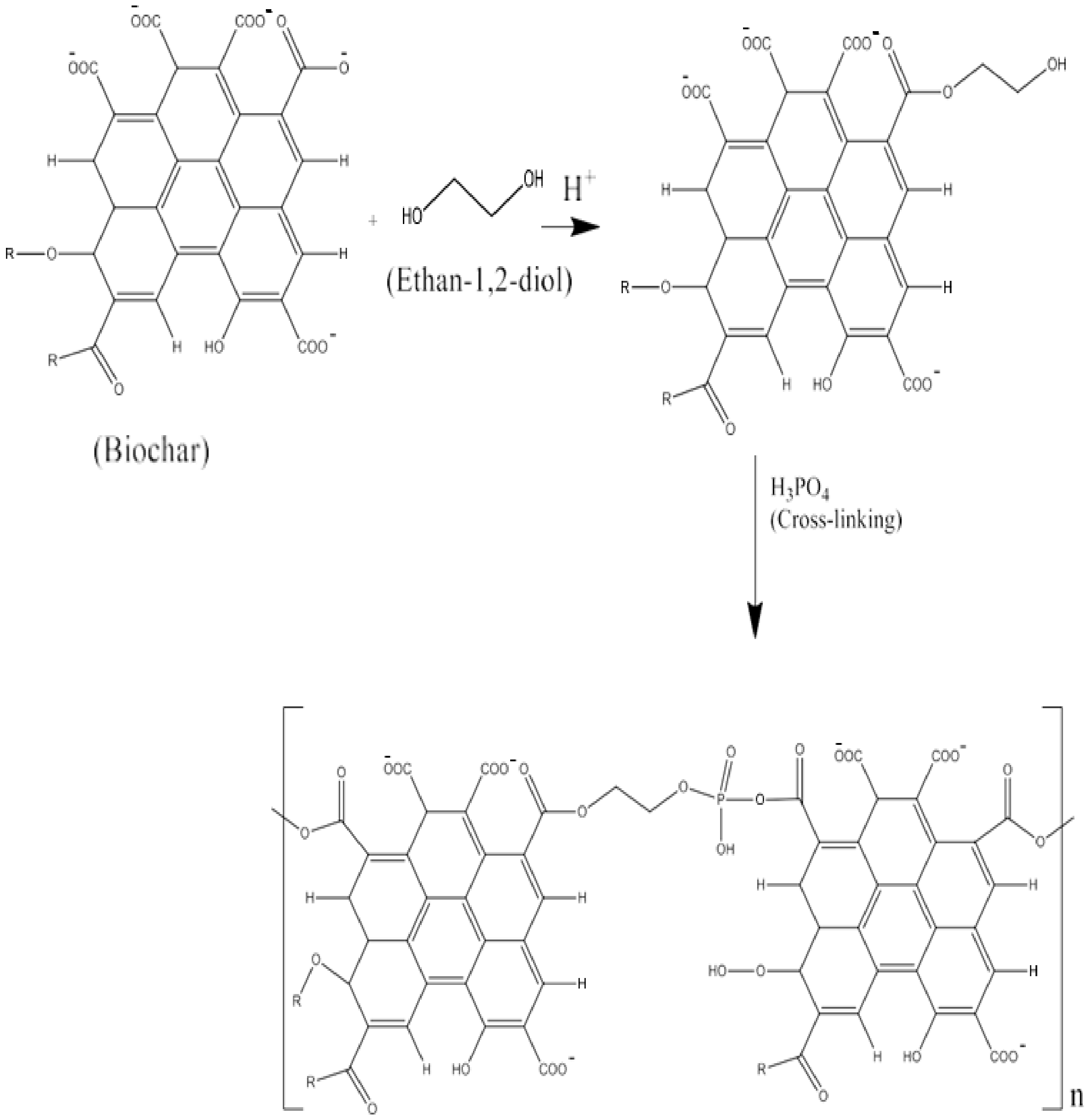

Figure 2. Reaction Scheme for the functionalization of biochar with Ethan-1,2-diol and cross-linked with phosphoric acid

Functionalization of biochar with glycerol and cross-linking with citric acid:-
It involved reacting 10g of biochar with glycerol at 120 ºC for two hours in the presence of sulphuric acid to form an ester. The cross-linking method was adopted from Gyamati, 2015 where the ester was heated at 90º C for 20 minutes after which 100g of the ester was placed in a beaker with 0.1M NaOH solution at a pH of 12.00 necessary for cross-linking to attain stability due to the alkali environment. The liquid was discarded and a carboxylation reaction followed as shown by the reaction scheme Figure 3.

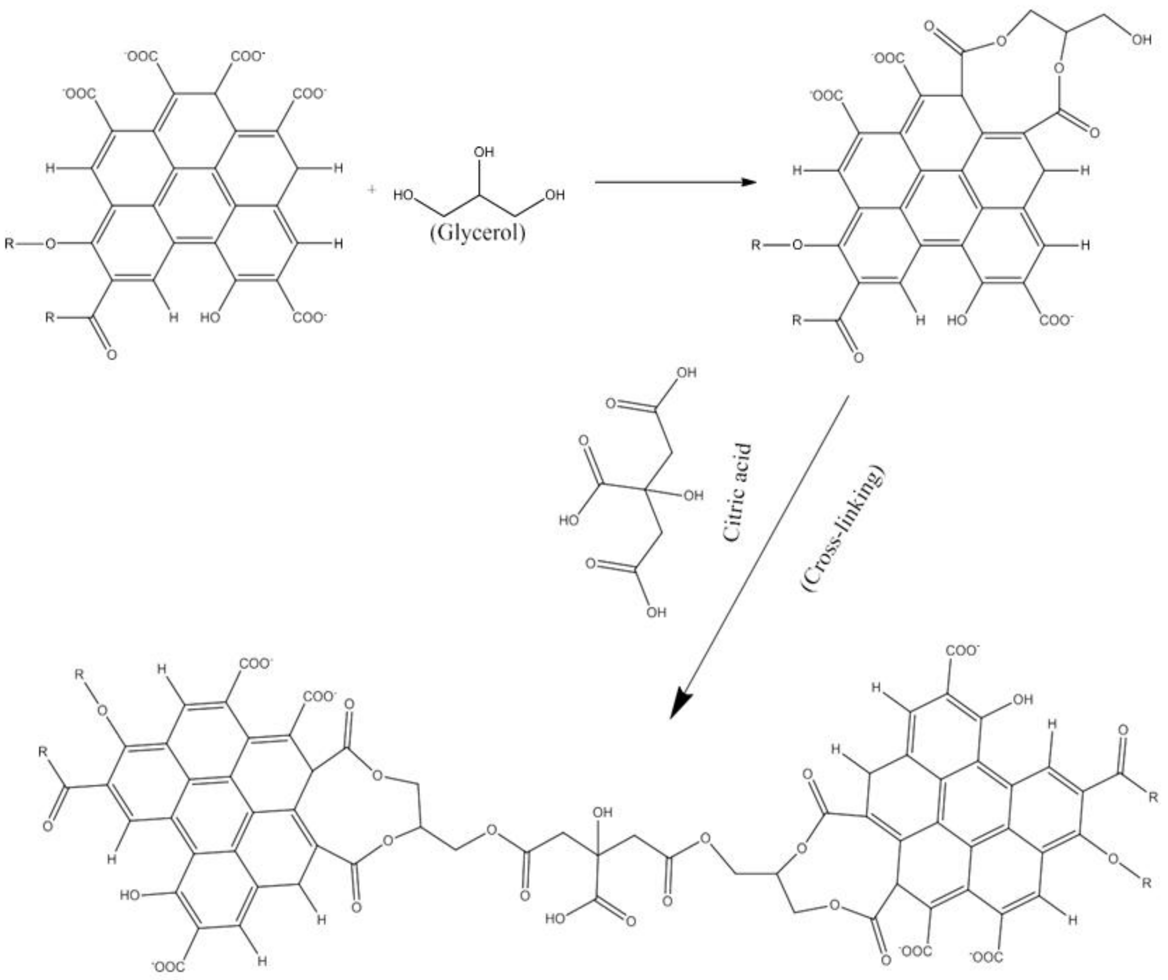

Figure 3. Reaction scheme of functionalization of biochar with glycerol then cross-linked with citric acid

Functionalization of biochar with ethan-1,2-diol:

Biochar was reacted with ethan-1,2-diol by ester linkage and then cross-linked twice by the use of phosphoric acid to get a phosphoric Esther followed by a second cross-link with urea to give urea phosphate. The mulch did not hold a lot of water as one would expect as compared to the mulches with single cross-link since its swelling was 7.76 times its weight as compared to phosphate functionalized with urea cross-link which had a swelling capacity of 12.09 times its weight. This result was in line with a study which was done by An et al., (2021) on the relationship between cross-linking and swelling capacity. He found out that if too much cross-linking agent is used, then the high cross-linking degree causes a low swelling degree of hydrogel hence little water is held. However, if the amount of cross-linking agent is too small, then the physical interaction between polymer bonds breaks easily causing the hydrogel to become water soluble.

FTIR Spectra characterization of modified and unmodified biochar

Functionalized and non-functionalized mulch were characterized using FTIR. The spectra of biochar, phosphate functionalized and urea cross-linked biochar are given in Figure 4.1

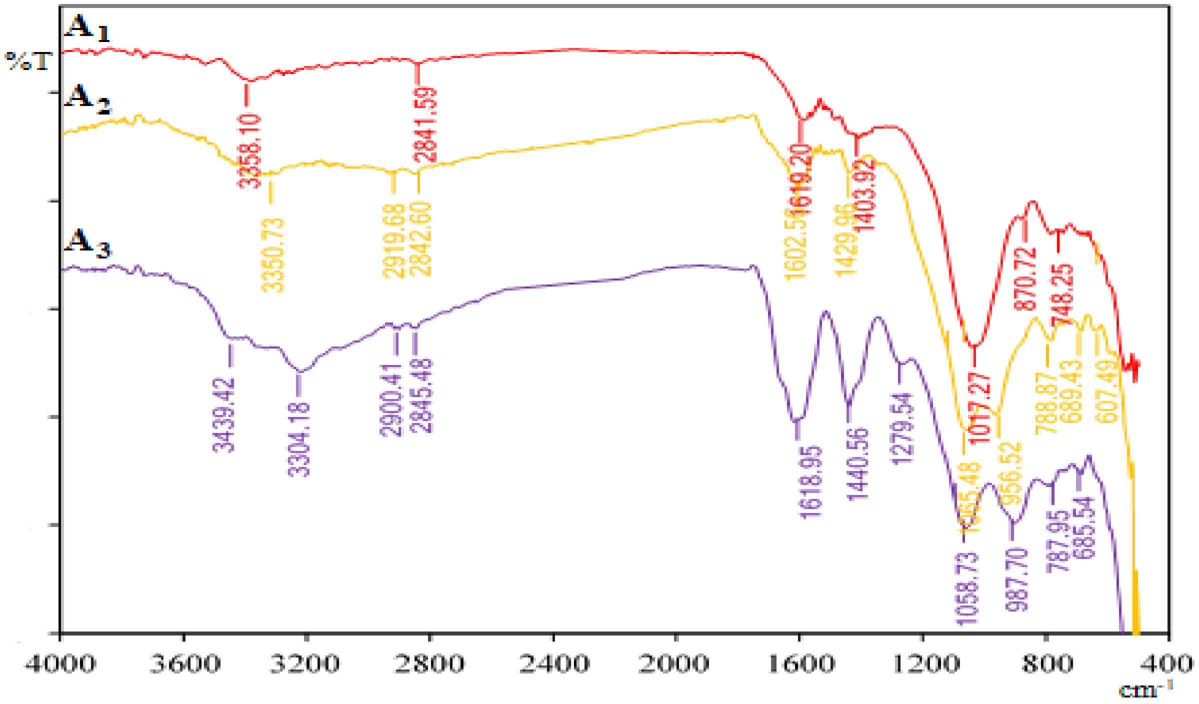

Figure 4.1: **The FTIR spectra of non-functionalized biochar (A_{1}), phosphate functionalized Biochar (A_{2}) and urea cross-linked mulch (A_{3})**

All the spectra bands about 3350.73-3439.42 cm^{-1} are attributed to either -NH or -OH groups (Petit et al., 2018). The bands at 2919.68cm^{-1} and 2900.41cm^{-1} were attributed to C-H stretches while 1618.95 cm^{-1}, 1610.20 cm^{-1} and 1602.5cm^{-1} were attributed to stretching C=O, N-H or -OH (Nandiyanto et al., 2019). The peaks at 788.87 and 956.52 cm^{-1} correspond to P-OH stretching vibration mode of phosphoric group (Zhang et al., 2021) which was used to functionalize biochar. A band at 1279.5 cm^{-1} corresponds to the P=O stretching mode (Kubicki et al., 2012) and a peak at 1058.73 cm^{-1} was attributed to C-N stretching which indicate a successful cross-linking with urea.

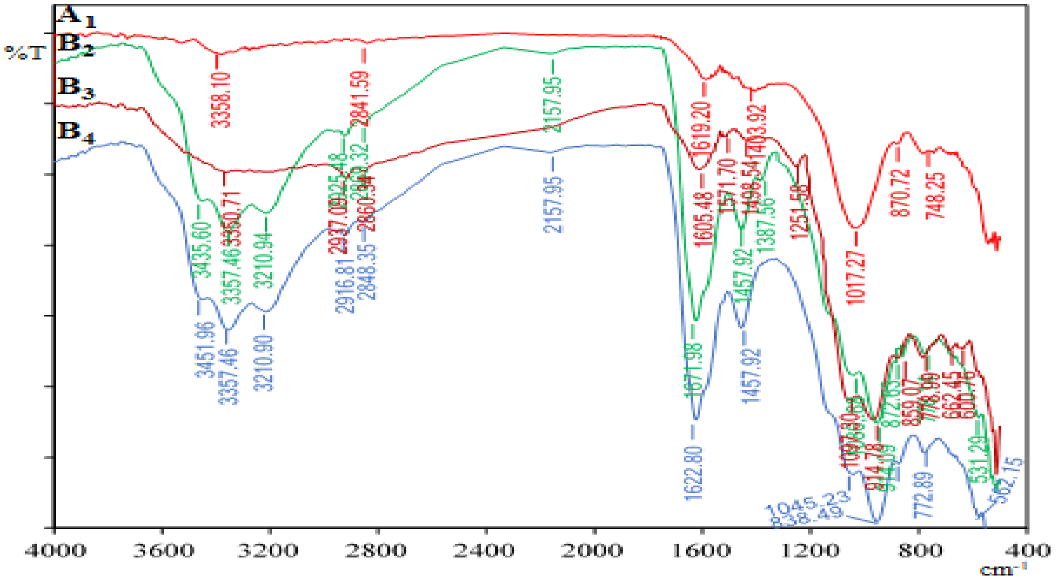

Figure 4.2 FTIR spectra of non-functionalized biochar (A_1), Ethan-1,2-diol functionalized (B_2), Phosphate cross-linked (B_3) and second cross-linking with Urea (B_4)

There are broad and strong bands at about 3451.96 cm^{-1}, 3435.60cm^{-1} and 3358.10 cm^{-1} which were attributed to -OH or -NH groups (Enders et al., 2021) from biochar. A peak at 1387.56 cm^{-1} corresponds to C-H stretching from Ethan-1,2-diol while a band at approximately 1251.58 cm^{-1} correspond to the P=O stretching mode (Kubicki et al., 2012) and a peak at 859.07 cm^{-1} is attributable to P-OH stretching vibration mode (Frost et al., 2013).A band at approximately 3357.46 cm^{-1} corresponding to N-H stretching vibration mode (Branca et al., 2016), a peak at 1622.80 cm^{-1} can be attributed to N-H bending vibration of amines according to Asyana, (2016) and a band at approximately 1045.23 cm^{-1} correspond to C-N stretching vibration mode (Krichen et al., 2019)

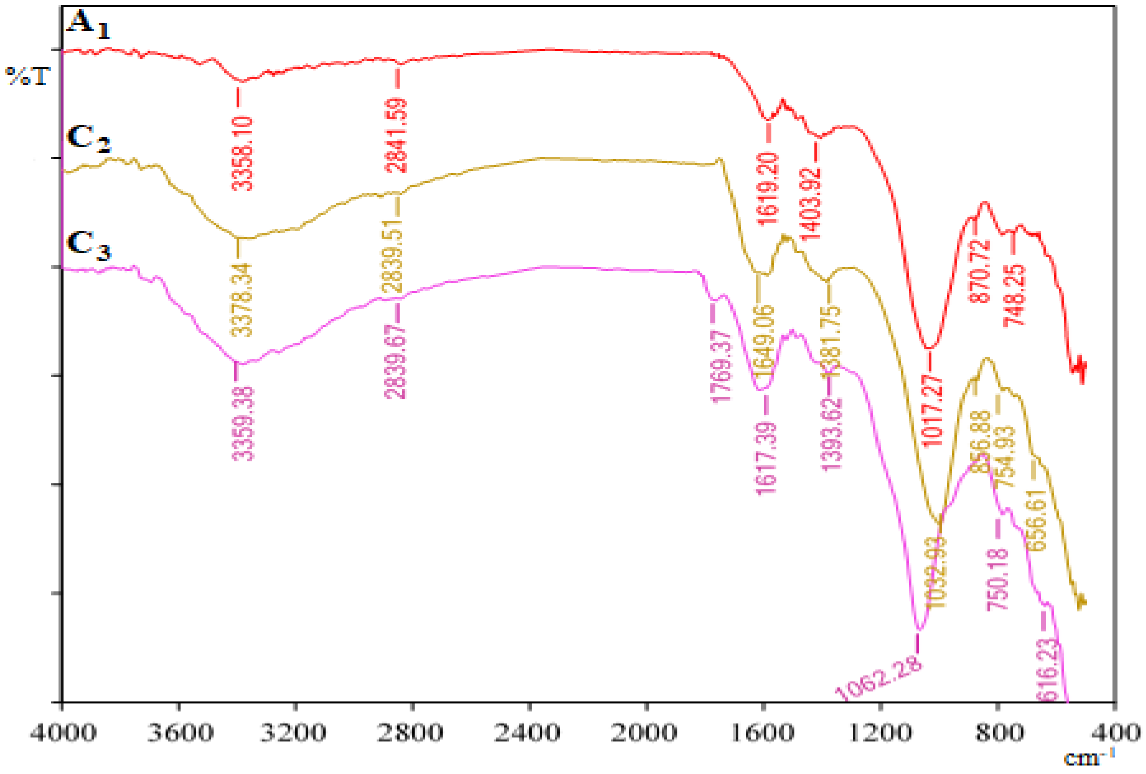

Figure 4.3: FTIR spectra of non-functionalized biochar (A_1), glycerol functionalized (C_2) and citric acid cross-linked (C_3).

The observed peaks at 3359.38, 3378.34 and 3558.10 cm^{-1} in all the three spectra's is due to -OH Stretching vibrations (Fontues et al., 2021), the peak around 1617.39 cm^{-1} stretching was attributed to a carbonyl (Cai et al., 2019) which indicates that the C=O from citric acid cross-link was successively anchored.

Conclusions:-

The functionalization with Ethan-1,2-diol, glycerol, phosphate and cross-linking with urea, phosphate and citric acid were confirmed with the FTIR spectrum which showed peaks for -OH at 3350.73 cm^{-1}, P-OH at 788.87 cm^{-1}, P=O at 1279.5 cm^{-1}, C-N at 1045.23 cm^{-1}, N-H at 1622.80 cm^{-1}, C-H at 2919.68 cm^{-1} and C=O at 1602.5 cm^{-1}. This confirmed that the diol, phosphate and amino used for functionalization and cross-linking had been successively anchored.

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