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Synthesis and characterization of biochar based phosphorus and biochar based nano-phosphorus

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Abstract
In this work, biochar based phosphorus and nano-phosphorus was synthesized by biological method (stevia leaf). Their size, morphology and microstructure have been investigated by particle size analyzer, Dynamic Light Scattering (DLS) and X-ray powder diffraction (XRD). The synthesized BBP (biochar based phosphorus) and BBNP (biochar based nano-phosphorus) was 87.6 and 110.8 nm in size, respectively and this was confirmed by X-ray powder diffraction (XRD). Further, zeta potential of the synthesized BBP and BBNP were determined by DLS (Dynamic Light Scattering) analysis. The zeta potential of BBP was -30.1 mV and BBNP was 9.2 mV.

Keywords: biochar based phosphorus, biochar based nano-phosphorus, zeta potential, XRD, DLS

Introduction
Biochar being a carbonaceous inert material has proven potential usage in agricultural systems owing to multiple benefits in improving soil fertility, crop productivity (Chan and Xu 2009; Glaser *et al*. 2002) [2, 4] and the porous physical structure of biochar induces a greater sorption capacity to conserve soil moisture and nutrients (Liu *et al*. 2012; Novak *et al*. 2012) [6, 10]. Recent reports highlighted the beneficial agricultural applications of fortified biochar including as organic manure to host a variety of plant nutrients and also as soil amendment (Ngulube *et al*., 2018) [9] for enhancing the soil physical properties and thereby improving the soil quality (Cornelissen *et al.*, 2013, Martinsen *et al.*, 2014 and Xu *et al*., 2015) [3, 7, 11]. However, biochar itself does not contain enough nutrients for crop growth (Asai *et al*., 2009) [1]. Grain yield of rice decreased significantly when only biochar was applied (Asai *et al*., 2009) [1]. This might be due to the poor nutrient supply capacity of biochar. Thus, supplementing biochar with certain nutrients renders biochar materials are more suitable for enhancing plant growth. Compared with chemical fertilization treatment, combined application of organic and inorganic fertilizer with biochar effectively increased the content of total nitrogen and potassium, available nitrogen, potassium and phosphorus (Mengyu *et al*., 2017 and Zang *et al*., 2016) [8]. Due to the agricultural and environmental advantages, biochar-based fertilizers are receiving increasing attention. Slow-release effect of biochar-based fertilizers has been reported by many researchers (Khan *et al*., 2008) [5].
Among the major nutrients, phosphorus is an essential nutrient for crop growth and yield. Further, in soil maintaining phosphorus under available condition is difficult and the applied P many a times is not utilized by crops. However, reducing the particle size of phosphorus to nanosize i.e. smaller than 100 nm, can further improve its properties, especially the absorption and utilization by plants which may lead to the reduction in the applied doses. Therefore, it is the dire need to synthesis and characterization of biochar based phosphorus and biochar based nano-phosphorus.

Materials and Methods
Synthesis and characterization of biochar based phosphorus (BBP) and biochar based nano phosphorus (BBNP)
Nanoscale phosphorus particles will be prepared using *Stevia* leaf extract as reducing and stabilizing agent. Take 10 g of dried *Stevia* leaf powder add 100 ml of distilled water into it. Then heat the content at 60 ^{0}C for one hour. Then filtered the content with wattman filter paper 40. After that Prepare 10 molar rock phosphate solution (422 g of rock phosphate dissolved in 1000 ml distilled water). Take 100 ml of stevia leaf extract and mixed with 100 ml rock

phosphate solution. Leave it for 2 days. Then centrifuge the content at 6000 rpm for 15 min. After centrifugation discard supernant solution and dry the solid portion (shade dry). After drying make it into powder with the help of pistle and mortar.
Biochar surface modification was done through acid wash (nitric acid) and appropriate binding reagent (gumacacia) powder was used for binding / loading of nano-phosphorous and phosphorus particles. The techniques such as size, DLS, and XRD used to characterize the biochar based phosphorus and nano phosphorus.

Dynamic Light Scattering (DLS)
Dynamic Light Scattering (DLS) technique is used to measure the hydrodynamic diameters (Size of the hydrosol) of the biochar based phosphorus and nano phosphorus (Nanopartica SZ-100, Horiba Scientific). This apparatus works on the principles of laser diffraction. The sample holder temperature was maintained at 25°C. Using this apparatus, the measurable particle size range is 0.3nm – 8µm. Depending on the configuration and application, the system can also be used to measure zeta potential. Zeta potential is widely used to denote the magnitude of the electrical charge surrounding the particles. The sample was prepared by centrifuging the particles suspension at 10,000 rpm for about 10 minutes and then the size (hydrodynamic diameter) and zeta potential of biochar based phosphorus and nano phosphorus

X-ray diffraction analysis (XRD)
The crystal-phase structure and the crystallite size of the nano particles were determined using X-ray diffractometer (Philips X‘Pert MPD, Japan) using monochromatic CuKa1 radiation of wavelength k = 1.5418Å from a fixed source operated at 40 kV and 30 mA in the 2θ scan range of 20–80°. The crystallite size was calculated using the Scherrer equation.

$$D = K\lambda / \beta \cos \theta$$

Where, k is the Scherrer constant (k=0.89), λ is the X-ray wavelength, β is full width of the peak at half maximum (FWHM) intensity (in radians) and θ is the Bragg‘s diffraction angle.

Results and Discussion:
Characterization of BBNP
The particle size of BBNP i.e. 87.6 nm and the zeta potential spectra of biochar based nano-phosphorus plotted zeta potential (mV) on x-axis and intensity (a. u) on y-axis. The zeta potential and electrophoretic mobility of BBNP was -31.0 mV and -0.000241cm^2 Vs^{-1} respectively (Fig 1 & 2).
In XRD, scanning of BBNP through a range of 2θ angles, all possible diffractions and d-spacings were recorded to characterize BBNP. Each nutrient have different diffraction pattern in XRD, based on diffraction pattern nutrient composition of the material is determined. BBNP diffraction peaks were observed at 14.82, 26.51, 29.31, 31.94, 33.05, 39.33 and 47.41, whereas d-spacing were observed at 5.96, 3.35, 3.04, 2.79, 2.70, 2.28 and 1.91. All these data were analyzed using ‘Search-Match’ algorithms of ICDD (International Centre for Diffraction Data, USA) (Fig.3.).

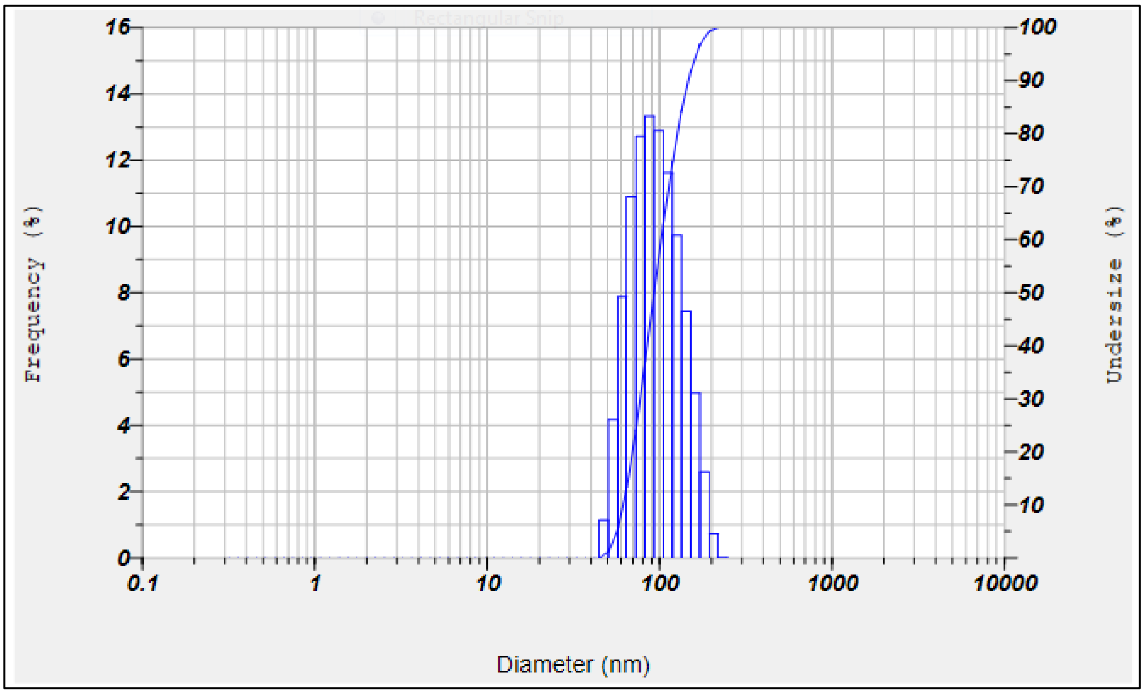

Fig 1: Histogram of BBNP particles of (mean size 87.6 nm) analyzed using Particle size analyzer

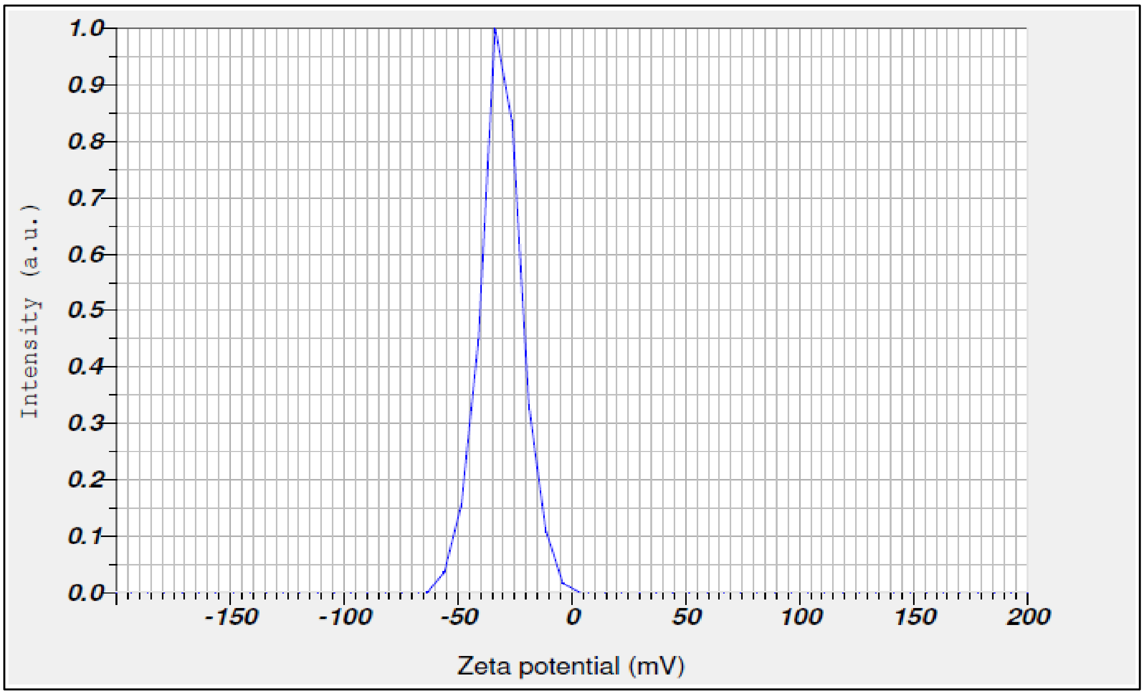

Fig 2: Zeta potential of BBNP (-30.1 mV) analyzed by Particle size analyzer

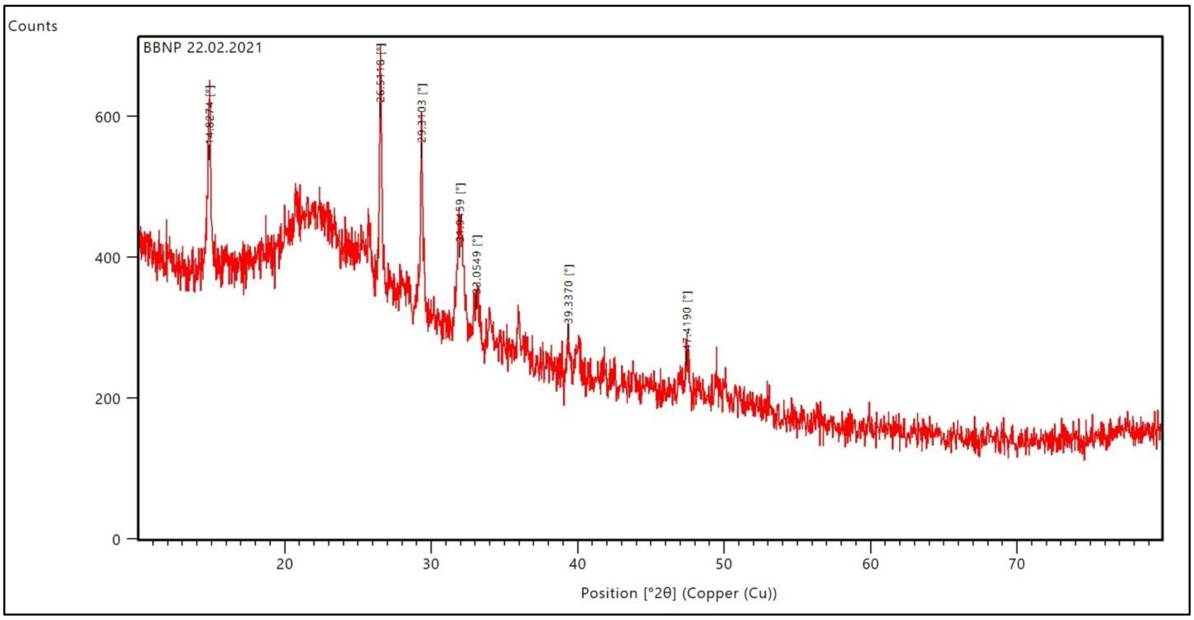

Fig 3: XRD pattern of prepared biochar based nano-phosphorus

Characterization of BBP

The particle size of the synthesized BBP sample i.e. 110.8 nm and zeta potential of biochar based phosphorus was 9.2 mV and its electrophoretic mobility mean recorded was 0.000072 $cm^2 Vs^{-1}$(Fig. 3 & 4).

X-Ray Diffraction technique is a non destructive technique which was used to study the characterization of prepared BBP. By scanning of prepared material through a range of 2θ angles, all possible diffractions and d-spacings were recorded, which helped in identification of given nutrient. The diffraction pattern of a known phase of a nutrient act as a ‘finger print, which can be used to identify that nutrient. With respect to prepared BBP, 2θ diffraction peaks measured at 14.84, 26.53, 29.33, 31.97, 33.09, 38.52 and 49.85 angle was 5.96, 3.35, 3.04, 2.79, 2.70, 2.33 and 1.82, by computer ‘Search-Match’ algorithms of ICDD (International Centre for Diffraction Data, USA) (Fig.6.).

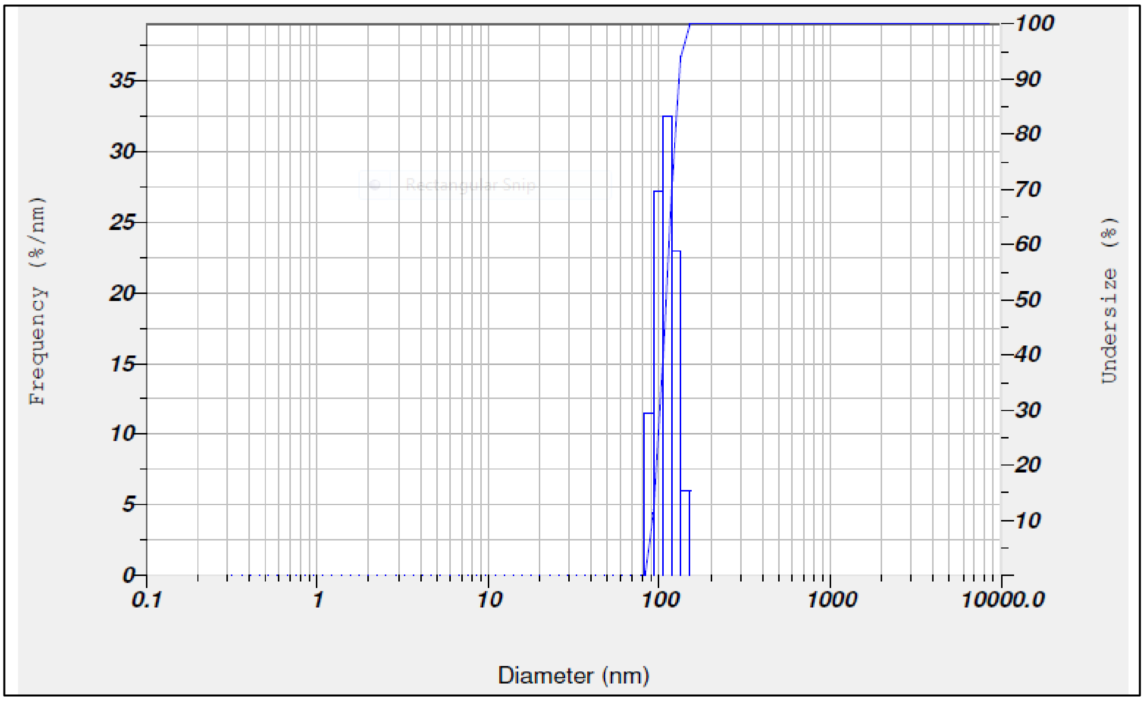

Fig 4: Histogram of BBNP particles of (mean size 110.8 nm) analyzed using Particle size analyzer

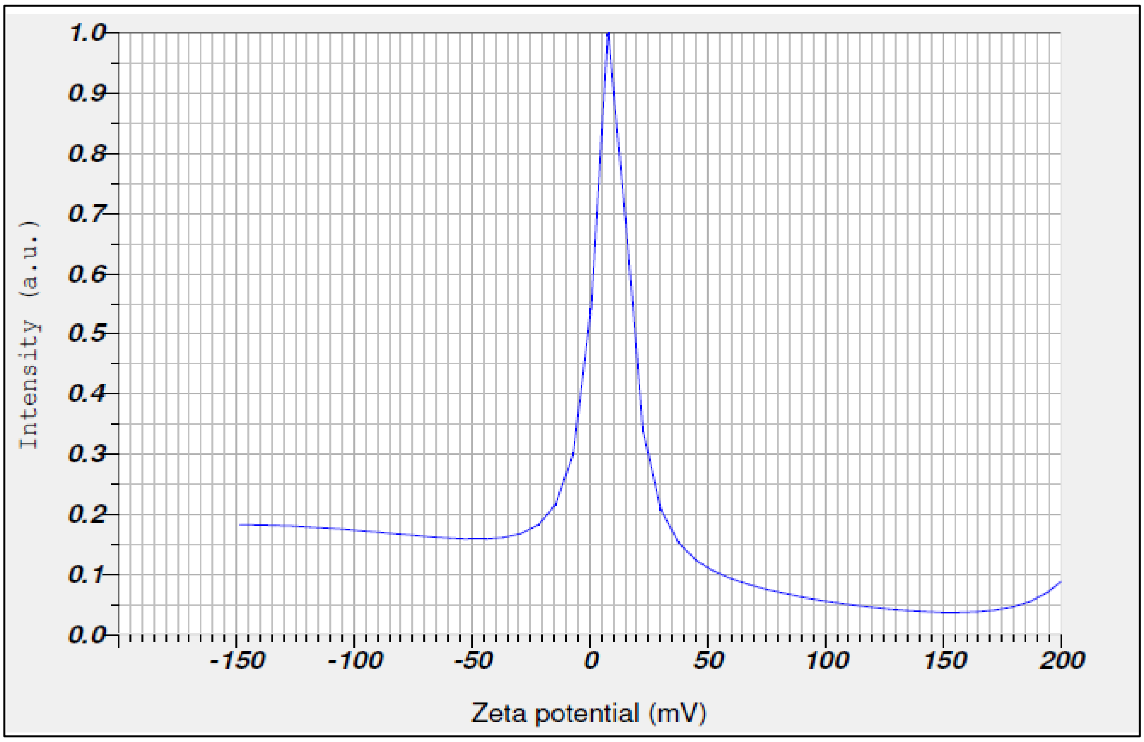

Fig 5: Zeta potential of BBP (9.2 mV) analyzed by Particle size analyzer

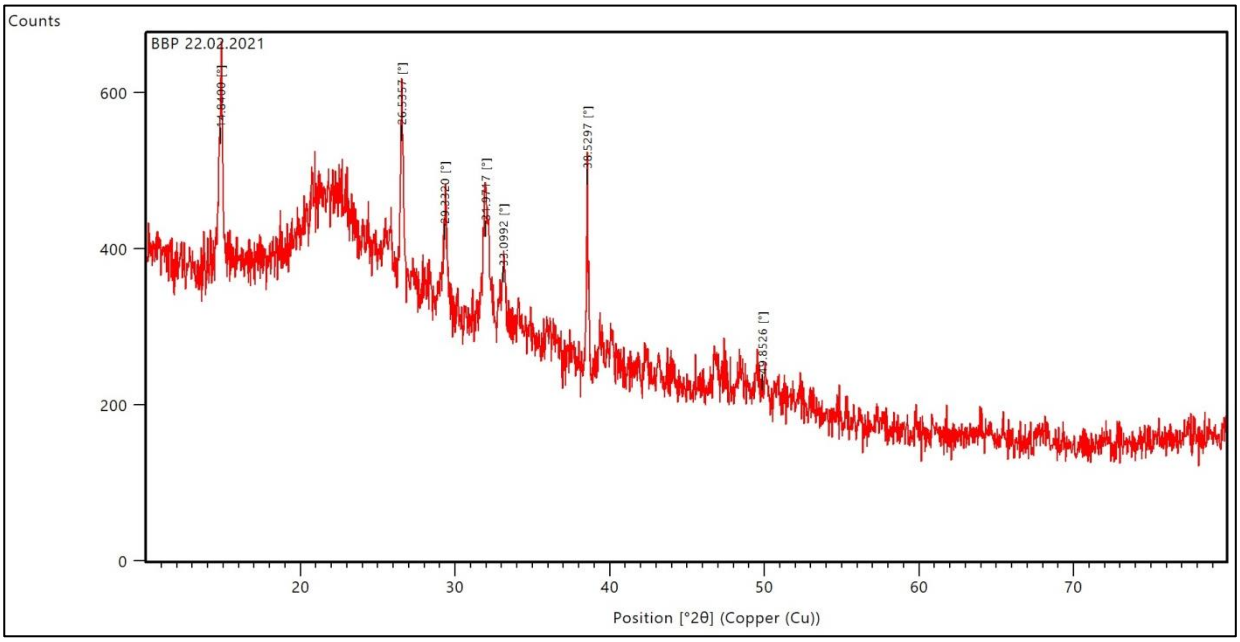

Fig 6: XRD pattern of prepared biochar based phosphorus

Characterization of NP

The particle size of the synthesized NP was 70.7 nm and the zeta potential of nanophosphorus was -22.0 mV and its electrophoretic mobility mean recorded was -0.000170 $cm^2 Vs^{-1}$(Fig.7&8).

In X-Ray Diffraction by scanning of prepared material through a range of 2θ angles, all possible diffractions and d-spacings were recorded, which helped in identification of given nutrient. With respect to prepared NP, 2θ diffraction peaks measured at 5.24, 14.80, 20.76, 21.89, 22.92, 25.74, 26.53, 28.04, 29.24, 30.11, 31.92, 32.17, 33.12, 34.06, 35.67, 36.46, 39.36, 40.09, 42.30, 43.77, 45.27, 46.90, 48.29, 49.50, 49.83, 50.84, 51.68, 52.33, 53.01, 56.19 and 57.27 d-spacing angle was 16.85, 5.97, 4.27, 4.05, 3.87, 3.45, 3.35, 3.17, 3.05, 2.96, 2.80, 2.77, 2.70, 2.62, 2.51, 2.46, 2.28, 2.24, 2.13, 2.06, 2.00, 1.93, 1.88, 1.83, 1.82, 1.79, 1.76, 1.74, 1.72, 1.63 and 1.60 by computer 'Search-Match' algorithms of ICDD (International Centre for Diffraction Data, USA) (Fig.9.).

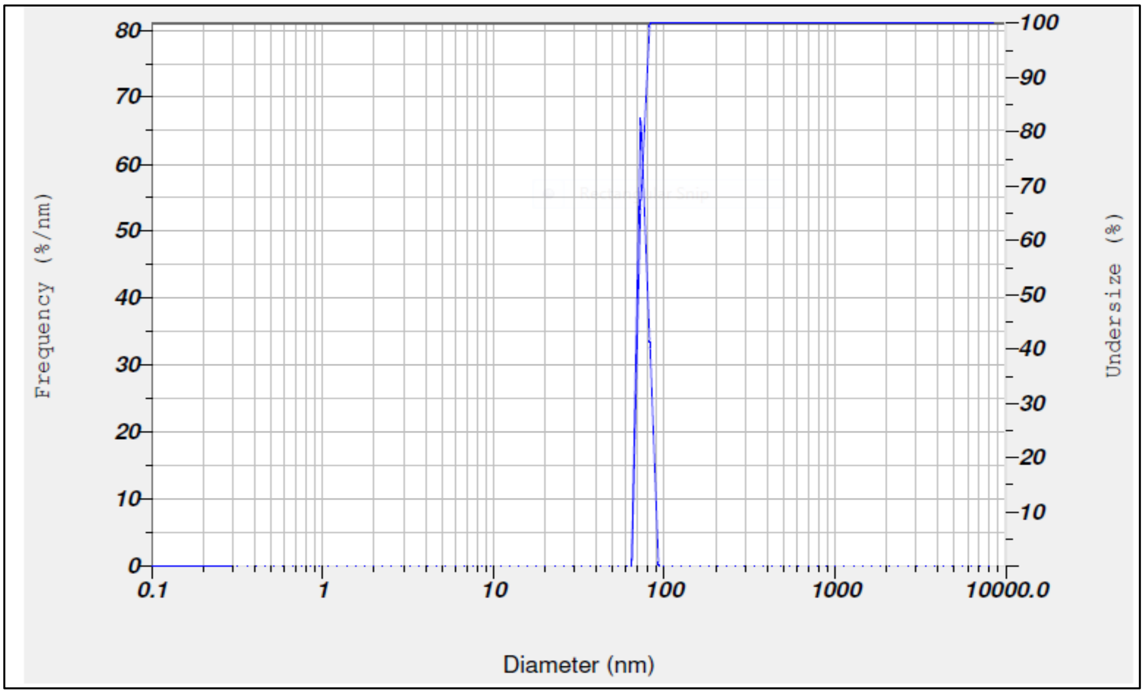

Fig 7: Histogram of NP particles of (mean size 70.7 nm) analyzed using Particle size analyzer

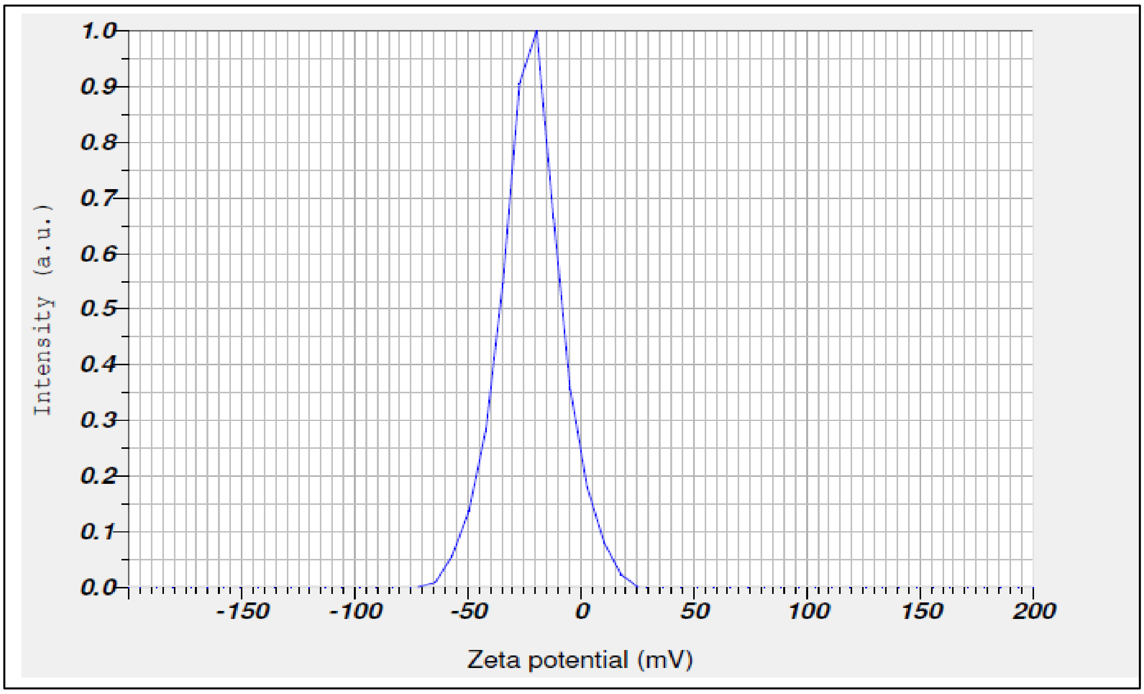

Fig 8: Zeta potential of NP (-22.0 mV) analyzed by Particle size analyzer

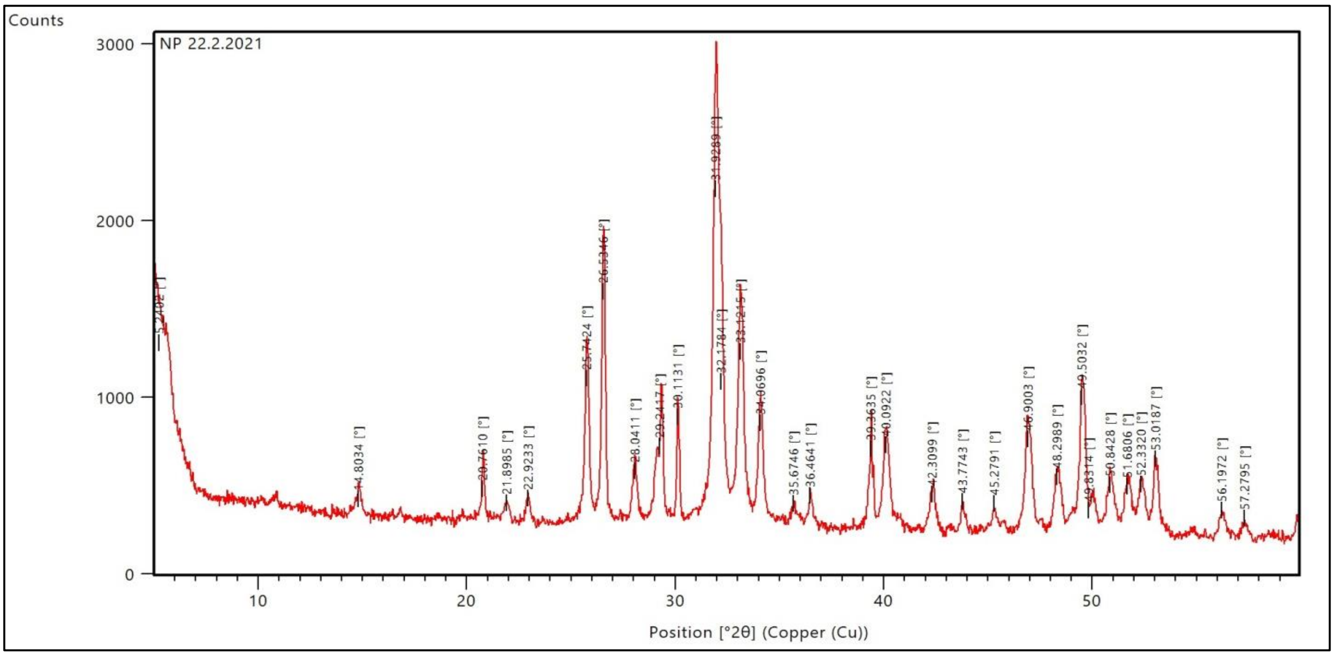

Fig 9: XRD pattern of prepared Nano-phosphorus

Summary and Conclusion

Nano-phosphorus was synthesized by biological method by using *stevia* leaf extract. Biochar surface modification was done through acid wash (nitric acid) and gumacacia powder was used for binding / loading of nano-phosphorous and phosphorus particles. The synthesized BBP (biochar based phosphorus) and BBNP (biochar based nano-phosphorus) was 87.6 and 110.8 nm in size, respectively and this was confirmed by X-ray powder diffraction (XRD). Further, zeta potential of synthesized BBP was -30.1 mV and BBNP was 9.2 mV. Synthesis of nano particles by biological method becomes critically important for promoting the development of environment friendly and sustainable agriculture. This new biological method by using *stevia* leaf can be good candidate as a dosimeter for various applications.

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