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CHARACTERIZATION AND METAL REMOVAL STUDIES OF NANO BIOCHAR ENERGY PARTICLES DERIVED FROM CAPSICUM ANNUAM

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Abstract

Intensified environmental concerns in recent decades have prompted a global effort to reduce pollution, particularly in the disposal of solid wastes and metals from industry. Nano biochar (N-BC) is gaining popularity as a result of its specific ecological properties. But, knowledge on Nano BioChar's synthesis, physicochemical properties, and stability is limited. The major product of the pyrolysis process is Biochar, which is an eco-friendly product. In the present work, Biochar was prepared from chilli stalk and nano biochar particles are prepared from the biochar particles. Finally the prepared biochar particles are characterized by Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), Particle Analysis, Thermo gravimetric Analysis (TGA) and Brunauer-Emmett- Teller (BET) surface area to interpret their physio-chemical, surface morphology and molecular structural changes so as to check whether they could be suitable to remove metals from waste waters.. The results of the characterization will help researchers better understand the creation of N-BCs, as well as their environmental destiny and conduct in soil and water.

Keywords: Biochar, Nanoparticles, Capsicum Annuam, Characterization, Thermogravimetric analysis, FTIR Analysis.

1. Introduction:

Biochar is mostly composed of aromatic molecules that are not arranged in a uniform layer[1]. Increases in pyrolysis temperature between 400 and 700°C result in higher aromaticity and hydrophobicity of biochar, as well as larger specific surface area and pore volume[2]. Biochar can be made from a variety of biomass sources, including waste from wood processing, municipal trash, sewage sludge, waste from animal breeding, and crop residues. Many researchers have worked on the removal of contaminants i.e., herbicides and pesticides onto several adsorbents (biosorbents, activated carbon, raw biomass and biochar, hydro char and modified biochars) using several techniques. Few researches also worked on synthesis of carbon nano particles. Fe_3O_4 magnetic nanoparticles were made by coprecipitating Fe_{2+} and Fe_{3+} ions in an ammonia solution and heating them hydrothermally [3]. Researchers are becoming interested in biochar (BC) made from biomass pyrolysis [4-6]. Biochars are commonly used for agronomic and environmental purposes. Several investigations have recently revealed that biochars can be physically degraded into nanoscale particles. Nano biochars were found to have superior mobility in natural soils and even travel into groundwater as compared to biochars. Nanobiochar, as a transporter, may enhance the transfer of natural solutes and pollutants, in contrast to biochar's beneficial properties, such as retaining nutrients and inactivating harmful compounds. [7]

In the present work, biochar which was prepared from chilli stalk was characterized and carbon nano particles were synthesized from the prepared biochar. Characterization of Carbon Nano particles were also done to investigate the differences among raw chilli stack biomass, biochar and nanoparticles.

2. Materials and methods:

The biomass (chilli stack) was collected from agriculture fields of Vadlamudi, Guntur district, Andhra Pradesh. All other chemicals required for experimentation were purchased from Sigma Aldrich.

2.1 Instruments:

Weighing balance (Shimadzu), pH meter (startorious PB-11), Muffle furnace (Tempo Instruments), Water bath (Labsolv Scientific Pvt.Ltd.), Stirrer (D -Lab), Micro-Oven (Tempo Instruments), Vacuum Filter (TarsonsRockyvac) and Sieves (Test Sieves) was purchased from Orlab technologies, Hyderabad, Telangana.

2.2 Preparation of Biochar:

The biomass (chilli stack) was washed with distilled water for several times and oven-dried at 900°C for 4hours. The dried biomass was then ground into small pieces and later, grinded to smaller particles. Then, the grinded biomass was transformed into pots and sealed with m-seal and

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kept in muffle furnace at 300°C for 2 hours. After that the biochar was removed from furnace and stored for further use. (Figure 1)

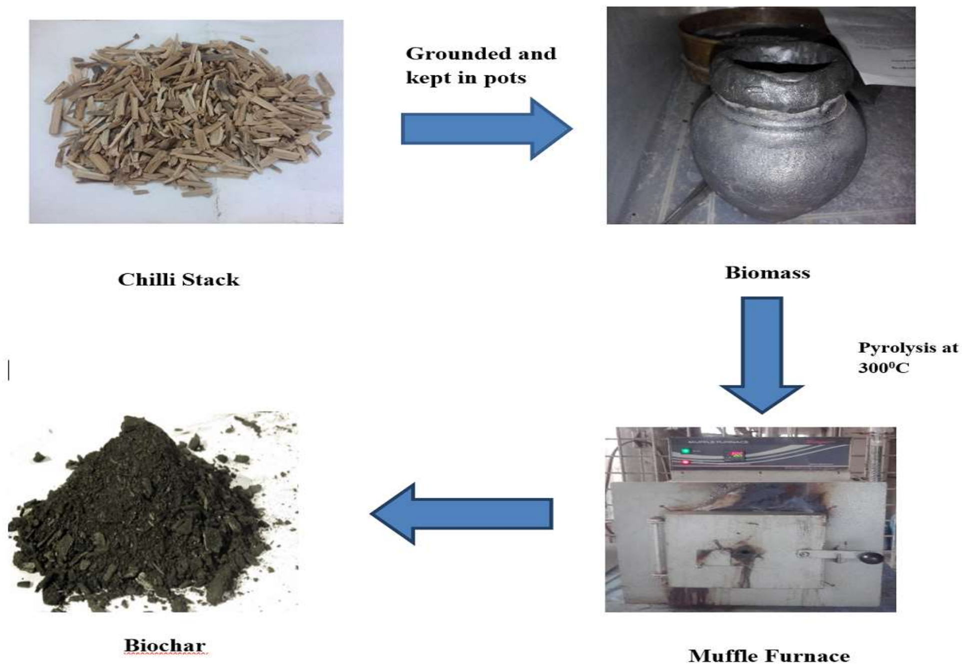

Figure 1: Preparation of Biochar Particles

2.3 Preparation of nanoparticles from biochar:

The biochar was rinsed in water and dried for 72 hours in a convection oven at 40 degrees Celsius. These were then broken down into smaller particles measuring between 0.1 and 0.2 mm in diameter. The co-precipitation process was used to make chilli stack biochar. In a 100mL water bath, 6.1 g of $FeCl_3.6H_2O$ and 4.2 g of $FeSO_4.7H_2O$ were dissolved and heated to 90°C. Rapidly and successively, a solution of 10 mL ammonium hydroxide (25 percent) and a solution of 1 g biochar dissolved in 200 mL water were added. The reaction medium's pH was set to 10. The mixture was agitated for 30 minutes at 80°C before being cooled to room temperature. The black Fe_3O_4–biochar precipitate was filtered, cleaned to neutral with water, dried at 50°C for 24 hours, and then kept for future use. (Figure 2)

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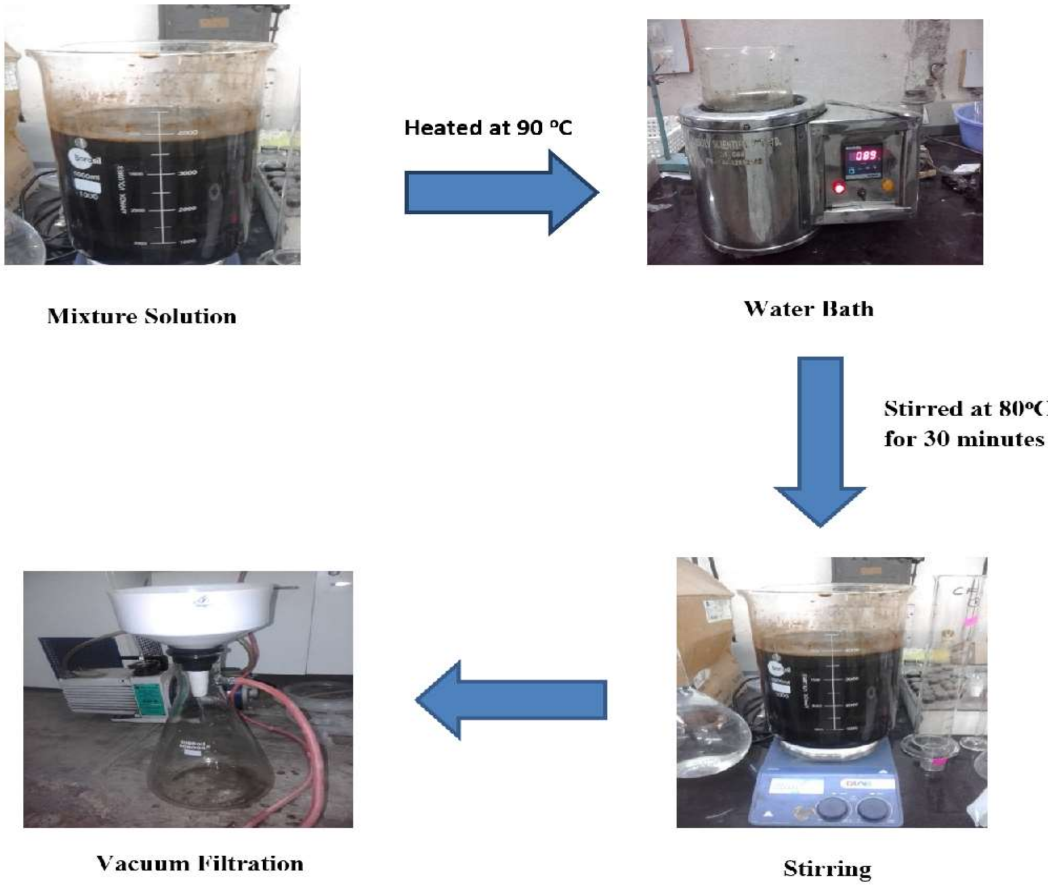

Figure 2: Preparation of Nano-Biochar Particles

2.4 Characterisation Techniques:

2.4.1 SEM Analysis:

SEM microscopy, often known as SEM analysis or SEM microscopy, is a powerful tool for microanalysis and breakdown properties of solid inorganic materials. High magnification electron microscopy yields higher scans and accurately quantifies very minute characteristics and substances.

2.4.2 FTIR Analysis:

FTIR is a technique for acquiring an infrared spectrum of the absorption or emission of a solid, liquid, or gas. An FTIR spectrometer simultaneously obtains high-spectral-resolution data over a wide frequency range.

2.4.3 X-Ray Diffraction:

XRD is a rapid method that can provide knowledge on micro - structure and is commonly used for crystalline material structural characterization. The sample is ground and mixed and the quantity content is standardized.

2.4.4 BET Analysis:

Physical adsorption of gas molecules on a solid surface is known as Brunauer–Emmett–Teller (BET), and it is the framework for an essential evaluation method for determining the specific surface area of a substance, as well as the porous structure. Because the rate of dissolution is proportional to the specific surface area, this data is used to predict it. As a result, solubility can be predicted using surface area.

2.4.5 TGA Analysis:

TGA Analysis, or Thermogravimetric Analysis, examines the amount of weight loss of a material as a result of rising temperature or constant temperature as a function of the time in an inert atmosphere. TGA is a sort of thermal treatment that determines the amount of a substance as temperature changes with time.

RESULTS AND DISCUSSIONS

3.1 Characterisation of Nanoparticles:

3.1.1SEM Analysis- The surface morphologies of raw biomass, Biochar, and Nano Biochar particles were examined using a scanning electron microscope (SEM) and are shown in Figure 3.1.1. When comparing raw biomass with biochar particles, it was discovered that Nano biochar particles have a greater number of pore volumes.

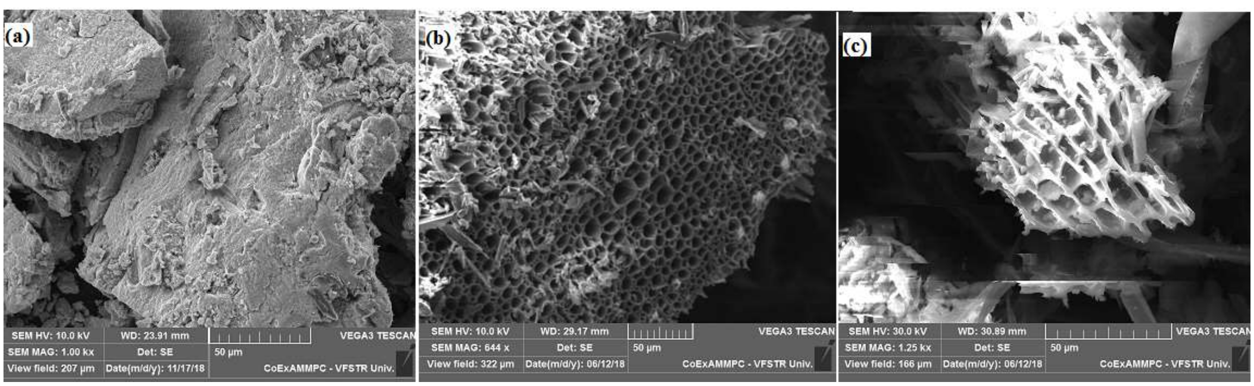

Figure 3.1.1: SEM images of of raw biomass, Biochar, and Nano Biochar particles

3.1.2 FTIR Analysis:

FTIR spectra of biochar nanoparticles (100°C for 24 hours) were obtained as thin coatings between KBr windows. To improve the signal-to-noise ratio, a total of 40 scans were produced. On a Thermo Nicolet Nexus 670 Spectrometer, the spectra were obtained at 4 cm^{-1} resolution in the range of 500–3600 cm^{-1}. The largest peaks in the spectrum for Nanoscale biochar belong to O-H stretching vibrations in the range of 3200-3600cm^{-1}, confirms the formation of alcohol, and remaining peaks are C-H,N- H and =C-H stretching vibrations at 2850-3000, 1550- 1640 and 675-1000 cm^{-1} indicating the presence of alkane, amines and amides and alkene functional groups, respectively as shown in the fig 3.1.2[8]. The detailed FTIR spectral analysis is shown in Table 1.

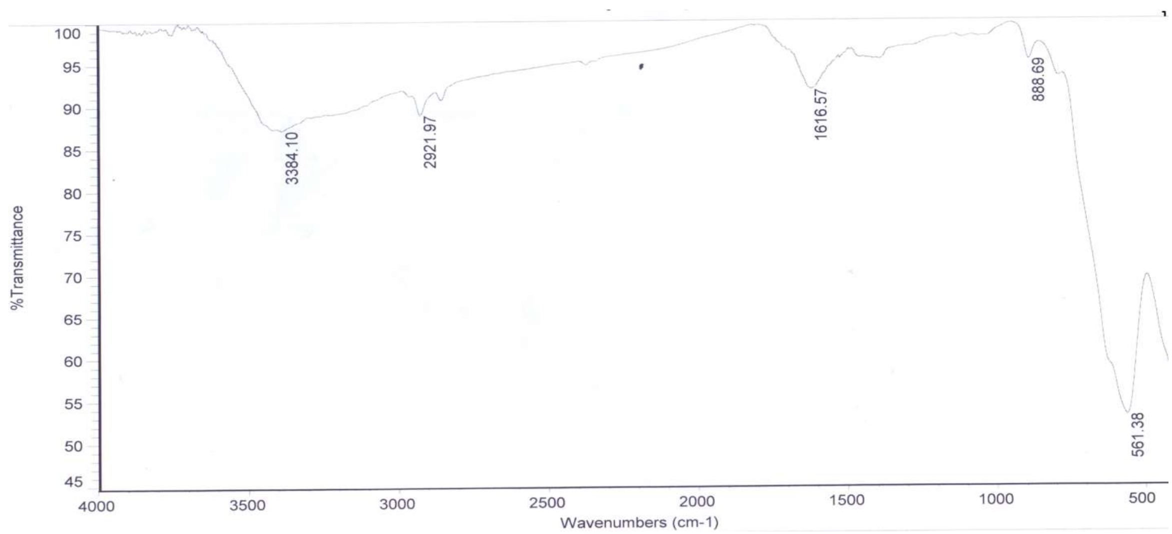

Figure 3.1.2 FTIR of Nano Biochar particles

Table 1: FTIR analysis of Nano Biochar particles

S.No.	**Wavelength (cm^{-1})**	**Functional Group**	**Type of Vibration**
1.	3200-3600	O-H(Alcohol)	(stretch, H-bonded)
2.	2850-3000	C-H(Alkane)	stretch
3.	2100-2260	-C=C-(Alkyl)	stretch
4.	1550-1640	-N-H(Amines and Amides)	Bending
5.	675-1000	=C-H(Alkene)	Bending
6.	500	C-I(Alkyl Halide)	stretch

3.1.3 XRD Analysis:

X-ray diffraction is a frequently used technique for determining the crystallinity of biomass and the structure of biochar. Cu K radiation at 40 kV and 130 mA was used in the X-ray diffraction examination (Bruker D8Advanced). The samples were scanned at a pace of 1°/min in the range of 2= 2–80°. Biochar's XRD spectra revealed a broad peak at 2h values of roughly 15-25. The appearance of a graphitic structure in Nano biochar was evidenced by the peak for biochar, which showed the growth of progressively carbonised material. The synthesis and sequential arrangement of aromatic carbon in Nano biochar results in this peak [9]. The XRD pattern corroborated the presence of a larger amount of aromatic component in the biochar, as evidenced by FTIR spectra. (Fig 3.1.3.)

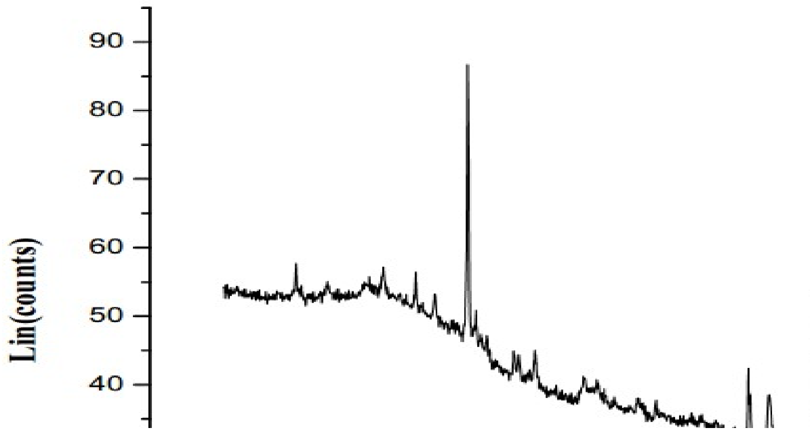

Figure: 3.1.3 X-ray diffraction of Nanobiochar particles.

3.1.4 TGA Analysis:

As a temperature dependent, the thermo gravimetric weight loss curve (TG, wt. percent) was observed. The amount of cellulose, hemicelluloses, and lignin in the sample determines the rate of heat decomposition. Thermo gravimetric analysis was performed at a 20 °C/min heating rate, as shown in Fig. 3.1.4 Raw chilli stalks were discovered to be in two stages of degradation. The first stage was achieved between 230°C and 350°C, with a weight reduction of 63.06 percent, while the second stage was achieved between 450°C and 630°C, with a weight loss of 43.02 percent. (Fig 3.1.4)

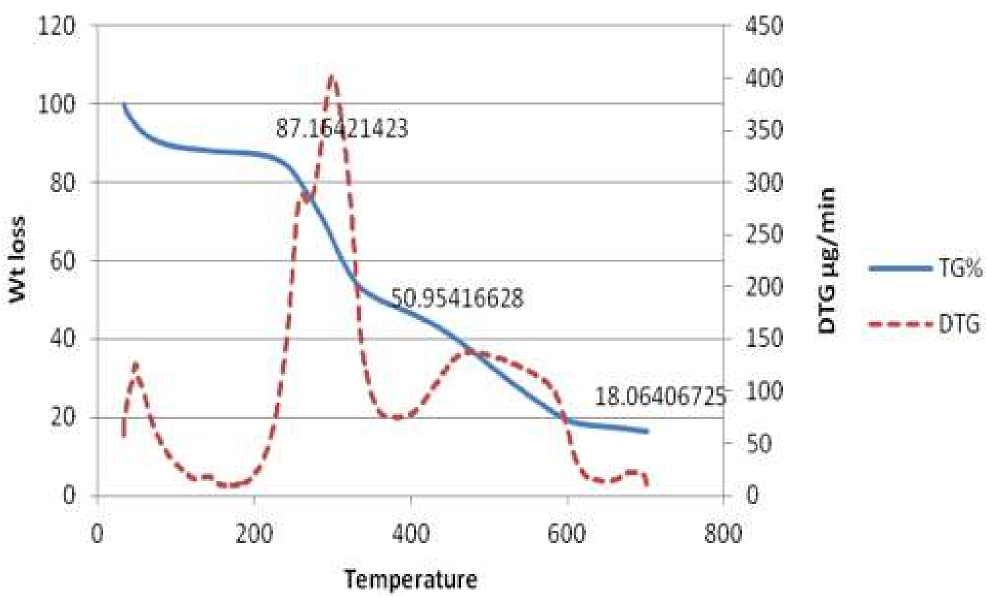

Figure: 3.1.4 TG-DTG Graph at 20°C heating rate

Conclusion:

The nanoparticles under investigation in this study were derived from Capsicum Annuum biochar, a fascinating organic material with potential applications in environmental remediation and adsorption processes. In order to comprehensively characterize these biochar-derived nanoparticles, a series of analytical techniques were employed, shedding light on their unique properties and suitability for specific applications. The characterization techniques encompassed in this analysis included Scanning Electron Microscopy (SEM) Analysis, Fourier Transform Infrared (FTIR) Analysis, X-ray Diffraction (XRD) Analysis, Brunauer–Emmett–Teller (BET) Analysis, Thermo gravimetric/ Derivative Thermo gravimetric Analysis (TG/DTG), and Particle Size Analysis. SEM Analysis revealed distinct features and morphological aspects of the nanoparticles, allowing for a visual examination of their structure and surface characteristics. This exploration provided valuable insights into the particle size and shape, which are essential parameters for understanding their performance in various applications.

The FTIR Analysis of the nanoparticles exhibited specific absorption bands, with the highest peaks appearing in the spectrum within the range of 3200-3600 cm^-1, corresponding to O-H stretching vibrations. These peaks suggest the presence of alcohol functional groups on the surface of the nanoparticles, which can play a vital role in their adsorption and chemical reactivity. XRD Analysis confirmed the presence of a graphitic structure in the Nano biochar, further supporting its potential as an effective adsorbent for various contaminants, especially heavy metals.

One of the notable findings of this investigation was the significant increase in pore volumes in the Nano biochar particles when compared to raw biomass and conventional biochar particles. This expanded pore structure enhances the surface area available for adsorption and underscores the promising potential of these nanoparticles for pollutant removal and remediation.

In conclusion, the results of the comprehensive characterization suggest that the prepared nano biochar particles possess a unique combination of structural features and surface chemistry, making them well-suited for applications involving the removal of metals and other contaminants. The insights gained from this study open new avenues for utilizing biochar-derived nanoparticles in environmental and material science applications, underscoring their significance in addressing contemporary environmental challenges.

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