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Biogenic Carbon Nanofiber Enhances Soil Water Retention and Minimizes Nutrient Leaching

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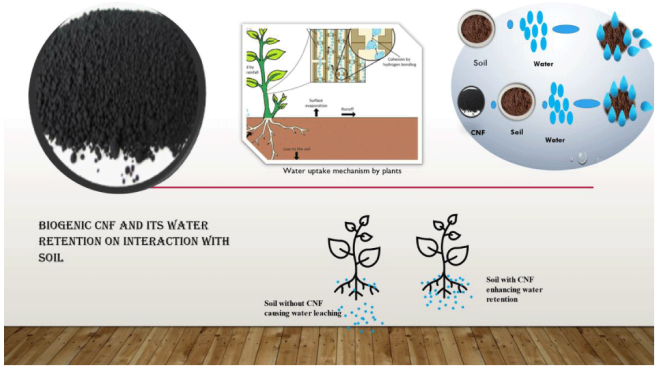
Abstract

This study focuses on the utilization of biogenic Carbon Nanofibers (CNF) derived from sugarcane bagasse waste. Due to their high surface area and porosity, biogenic CNFs exhibit excellent adsorption capabilities. We evaluated the water retention and leaching capacity of CNFs in Soilrite, incorporating varying amounts of CNF over 24 hours. The water was analyzed for pH, hardness, chloride ion concentration, alkalinity, and conductance before and after leaching. The results indicated that water retention capacity is significantly influenced by the concentration of CNF in Soilrite. The optimal water retention was found to be 38%, compared to just 14% in Soilrite without CNF. These findings highlight the potential of CNFs in enhancing soil water retention and ensuring the gradual release of water, making them a promising addition to soil management practices. Furthermore, the use of CNF as a carrier for nanofertilizers facilitates the slow release of water, promoting efficient crop growth without causing harm.

Introduction

Water occupies 71% of earth's surface out of which 96.5% of earths marine water is found in oceans, the rest is fresh water present in rivers, lakes, as ice, underground, and soil water. The generations in the near future are bound to face the problems of water crisis owing to the increasing population and their demands. This compels one to contemplate on every aspect of how one can achieve the needs of growing population. Multiple focus at every level employing use of upcoming technologies effectively in a scientific manner at domestic, industrial, agricultural is the Key to solve water CRISIS and enhance crop productivity. This research is focuses on agricultural



Graphical abstract representing interaction of CNF with soil for enhancement of water retention

sector using nanotechnology in such a way that it not only enhances the productivity issues to meet the needs of today but also saves the water required for growing crops. CNFs are conductive materials similar to graphite with cylindrical conical 1D nanostructures that range from few to 100 nm in diameter and lengths varying less than microns to mm (Sharon & Sharon 2021) [1]. CNF may be derived from biological sources (plants, animals, microbes, enzymes etc) or non-biological hydrocarbon sources. In the present work biogenic CNF synthesized from bagasse using of pyrolytic method (Sharon & Sharon 2006) [2] are used. Plants whether monocot or dicot used as precursor for synthesis of CNF display diverse structural and anatomical differences thus giving rise to complex structures that one may not be able to synthesize chemically Sharon & Sharon 202 [3, 6-11]. These structural differences hold immense potential as an adsorbent, as a carrier, as a facilitator for controlled release of nutrients etc. The present work is aimed at exploring the potential of biogenic CNF in holding water in soil and leaching from the soil. It was imperative for us to study not only the water holding capacity by CNF but also study the leaching of CNF into the waters after use. This study will open doors for use of biogenic CNF as nanofertilizer paving a way towards innocuous technology that addresses problems related to water scarcity, eutrophication, release of excess fertilizers in the nearby waters and thereby also promoting productivity of crops.

Materials and Methods

Biogenic Carbon Nanofiber (CNF)

CNF synthesized by standard CVD method under pyrolytic condition using bagasse as precursor (Sharon et al 2006) [2] was sourced from wcRnb Solapur. The bagasse was sun dried and subjected to pyrolysis at 850^{0}C in Argon gas atmosphere in a furnace, to obtain the CNF. The pyrolysis of plant material yielded CNFs which was characterized

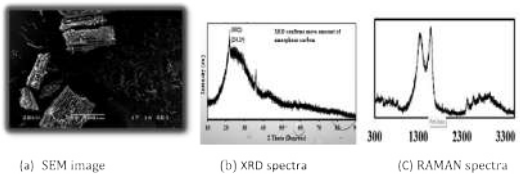


Figure 1: Characterization data of CNF obtained from bagasse - a: SEM; b: XRD; and c: Raman spectra

Table 1: pH, Conductivity, Cl^- ion, Ca^{2+} ions, Alkalinity, and Hardness of the Water Samples Adsorbed and released after 24 hours by CNF

Sample	Analysis Parameter of Water Sample After Being Leached Out From CNF							
	CNF mg/ 10G	ml H_2O Leached after 24 hrs	pH	Conductance mS	Cl- conc. mg/L	Alkalinity	Total hardness as $CaCO_3$ mg/L	
							Ca	Mg
D/W	0	50	7.5	0.18	237.582		66.66	
A	0	43	7.69	0.73	95.0328		96	
							60	36
B	15	34	7.75	0.8	142.549		96	
							60	36
C	25	32	7.78	0.84	142.549		96	
							60	36
D	35	31	7.81	0.86	142.549		96	
							60	36
E	45	31	7.84	0.88	190.065		108	
							72	36
F	55	36	7.86	0.89	190.065		108	
							72	36
G	65	32	7.89	0.9	95.0328		108	
							72	36
H	75	32	7.93	0.91	95.0328		108	
							72	36
I	85	34	7.96	0.93	142.549		120	
							84	36
J	95	33	8.01	0.95	142.549		120	
							84	36
K	105	33	8.08	0.96	142.549		120	
							84	36

No major change in Alkalinity was observed

prior to use by SEM, XRD, Raman spectroscopy and its yield as well as surface area was also measured.

Water Quality Analysis

Prior to using distilled water for water retention and leaching studies by CNF; the distilled water was analyzed for pH, hardness, chloride ion concentration, alkalinity, and conductance (Table 1).

Assessing Water Retention (Adsorption) and Leaching Capacity of CNF

A simple lab method was employed for the study of water holding and leaching out capacity of CNF. Throughout the study Soilrite was used. Biogenic CNF of different concentrations from 0 mg to 105 mg was mixed with 10 gm of soil respectively. Equal amount of all soil samples was set in a burette. 50 ml D/W was added in each of the burette followed by overlaying it with oil so as to prevent natural evaporation. The set up (Figure 1) was allowed to

stand for 24 hours. After 24 hrs. water from each burette was collected and analyzed for different parameters such as volume of water collected after 24 hrs., total hardness, chloride ion concentration, conductance, pH and so on (table 1). FTIR of the samples was carried out to confirm the findings (Table 2).

Analysis parameter of water samples studied after being leached out from CNF

i. ***Determination of Chloride ion concentration by Mohr's method***

In this method, 10cm^3 of sample water was pipetted into a 50cm^3 of volumetric flask and made up to mark with distilled water. A 10cm^3 of aliquot of the diluted sample solution was pipetted into a conical flask and 1cm^3 of K_2CrO_4 indicator solution (0.25M) was added which give faint yellow to the solution. This mixture was then titrated with 0.1M $AgNO_3$ solution and form pinkish brown coloured solution which indicate the end point of the solution. The titration process is repeated 3-4 times to achieve two concordant results.

Observations:

Volume of $AgNO_3$ used for distilled (Blank titration) = 0.5 cm^3

Normality of $AgNO_3$ = 0.134 N

Volume of sample taken = 10 cm^3

Table 2: %Water retention by biogenic Carbon Nanofiber

Concentration of CNF -mg/10g of soil	Water retention in %
0	**14**
15	**32**
25	**36**
35	**38**
45	**38**
55	**28**
65	**36**
75	**36**
85	**32**
95	**34**
105	**34**

Formula:

$$\text{Chloride ion concentration (mg/L)} = \frac{(A-B)\times N\times 35460}{V}$$

A= Volume of $AgNO_3$ for sample titration
B= Volume of $AgNO_3$ for blank titration
N= Normality of $AgNO_3$
V= Volume of water sample taken in cm^3

Calculation:

1. Volume of $AgNO_3$ used for Sample A = 0.7 cm^3

$$Chlorideionconcentration(mg/L) = \frac{(0.7-0.5)\times 0.134\times 35460}{10} = \frac{0.2\times 0.134\times 35460}{10} = 95.0328 \text{ mg/L}$$

2. Similarly, concentration of other sample solutions were calculated (table 1).

ii. ***Determination of total hardness (Calcium and Magnesium)***

It involved pipetting 10 cm^3 of sample water into a conical flask, then adding 2 cm^3 of buffer solution (pH 10) followed by 3 drops of Eriochrome Black T indicator. The mixture solution was then titrated with 0.01M EDTA solution until the solution changed from wine red to sky blue. The burette readings were noted in the observation table. The titration was repeated 3-4 times to achieve two concordant values, providing the volume of EDTA required for both calcium and magnesium ions and hardness for different concentrations were calculated (Table 1).

Determination of Calcium hardness

For Ca determination 10 cm^3 of sample water into a conical flask to which 30 drops of 50% w/v NaOH solution were added and the solution were swirled, allowing a few minutes for the complete precipitation of magnesium ions as $Mg(OH)_2$**(s)**. Then a pinch of Hydroxynaphthol Blue indicator was added, resulting in purplish red coloured solution. Subsequently, the mixture solution was titrated with 0.01M EDTA to get sky blue end point and the burette readings were noted in the observation table. The titration process is repeated 3-4 times to achieve two concordant results.

Observations:

Molarity of EDTA solution = 0.012M

Molarity of $CaCO_3$ = 0.01M

Volume of sample taken = 10 cm^3

$$\text{Total hardness as } CaCO_3 = \frac{V_1 \times C \times 1000}{V}$$

where, V_1 = volume of 0.01M EDTA required,

C = mg $CaCO_3$ equivalent to 1cm³ EDTA titrant $\left(1cm^3\, 0.01M\ EDTA \equiv 1.000\ mg\ CaCO_3\right)$ or

$$C = \frac{1 \times (\text{EDTA of Molarity})}{0.01M} = \frac{1 \times (0.012M)}{0.01M} = 1.2$$

V= Volume of water sample taken in cm³

Calcium hardness as $CaCO_3$, (mg /l)

$$\text{Calcium hardness as } CaCO_3 = \frac{V_2 \times C \times 1000}{V}$$

where, V_2 = volume of 0.01M EDTA required,
C = mg $CaCO_3$ equivalent to 1cm³ EDTA titrant,
V= Volume of water sample taken in cm³

Determination of Magnesium Hardness:

Magnesium Hardness = Total hardness as $CaCO_3$ (mg/l) – Calcium hardness as $CaCO_3$ (mg/l).

Calculations:

I. For sample A (Control)

$$\text{Total hardness as } CaCO_3 = \frac{V_1 \times C \times 1000}{V} = \frac{0.8 \times 1.2 \times 1000}{10} = 96 \text{ mg/L}$$

$$\text{Calcium hardness as } CaCO_3 = \frac{V_2 \times C \times 1000}{V} = \frac{0.5 \times 1.2 \times 1000}{10} = 60 \text{ mg/L}$$

Magnesium Hardness = Total hardness as $CaCO_3$ (mg/l) – Calcium hardness as $CaCO_3$ (mg/l). $= 96 - 60 = 36$ mg/L

II. Similarly, hardness of other sample solutions were calculated (table 1).

III. ***Determination of pH of the water sample by using pH meter***

The pH meter was switched on 10 minutes before starting the experiment and then it was standardized with the help of supplied connector cable which join the electrode terminal and pH terminal situated at the back of the instrument. After standardization, the combine glass electrode was dipped into the sample solutions to measure its pH. The pH measured was recorded and is represented in the figure 3.

IV. ***Determination of conductance of the water sample by using Conductivity Meter***

The conductivity meter was switched on 10 minutes before starting the experiment and then the range switch was kept at 2 millisiemens (mS/m) position and the standard conductance switch is kept on. The digital display reading was calibrated to show 1.000 for standardization. After that the conductivity cell were dipped in the sample solution of different concentrations of CNF water sample and the reading were recorded in millisiemens in the tabular form (Table 1) which is represented in the graph (Figure 4).

V. Determination of alkalinity in the water sample

In this method, 10 cm^3 of water sample was pipette out into a conical flask and 3 drops of phenolphthalein indicator were added. The solution remains colourless that indicate phenolphthalein alkalinity were absent. In that mixture, 3 drops of methyl orange indicator was added and the color of the solution became reddish pink which on titration with 0.01N H_2SO_4 remains reddish pink. Thus, Alkanity for all water samples for different concentrations of CNF was determined as follows:

Formula:

$$\text{Alkalinity as } CaCO_3\ (\text{mg/L}) = \frac{\text{Volume of } H_2SO_4 \times \text{Normality of } H_2SO_4 \times 50 \times 1000}{\text{Volume of water sample}(cm^3)}$$

Calculations:

$$\text{I. Phenolphthalein alkalinity (mg/L)} = \frac{P \times \text{Normality of } H_2SO_4 \times 50 \times 1000}{\text{Volume of water sample}(cm^3)}$$

Where, P (cm^3) = Volume of 0.01N H_2SO_4

$$\text{II. Methyl orange alkalinity (mg/L)} = \frac{M \times \text{Normality of } H_2SO_4 \times 50 \times 1000}{\text{Volume of water sample}(cm^3)}$$

Where, M (cm^3) = Volume of 0.01N H_2SO_4

VI. Fourier Transform Infrared (FTIR) Analysis of the Water Sample

FTIR analysis – Fourier Transform Infrared (FTIR) is an important analytical tool for researchers for the simultaneous determination of organic components, including chemical bond, as well as organic content (e.g., protein, carbohydrate, and lipid). In FTIR analysis procedure, samples are subjected to contact with infrared (IR) radiation. The IR radiations then have impacts on the atomic vibrations of a molecule in the sample, resulting the specific absorption and/or transmission of energy. This makes the FTIR useful for determining specific molecular vibrations contained in the sample (Kirk and Othmer, 1953) [4]. Here we have used FTIR to analyse the simple to complex organic materials in the sample, comprehended and interpreted FTIR data as represented in the figure 6 and table 3.

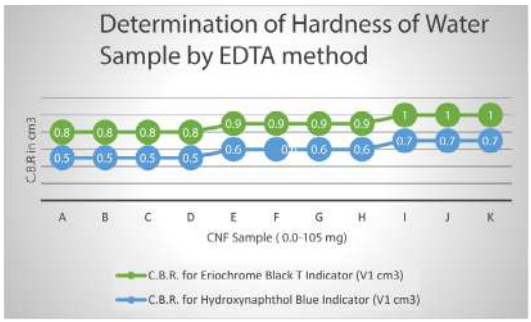


Figure 2: Determination of hardness of leached out water, by EDTA method (Values of A to K samples are given and same as mentioned in Table -1)

Results and Discussions

CNF - Morphology and Surface Area

Advantages of using plant derived precursors is that during pyrolytic carbonization their basic anatomical structures are not destroyed, resulting into some interesting channel types as well as porous structures. These structures are otherwise difficult to synthesize. These carbon materials possess suitable adsorption properties for both liquid and gases which we wished to explore. ***SEM images*** showed peculiar tubular fibrous structure (Pandey-Dubey; et al) [5]

XRD Spectrum

(Figure 2A) of CNF [4] obtained from Bagasse, shows a

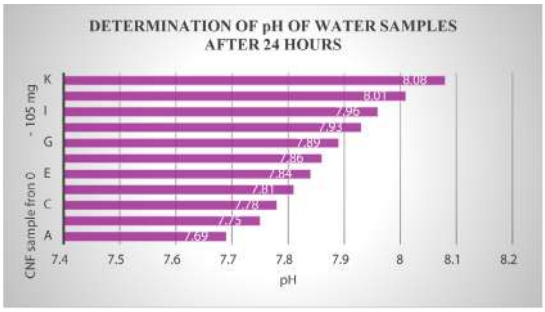


Figure 3: Determination of pH of water retained in CNF and leached out after 24 hours. (Values of A to K samples are given and same as mentioned in Table -1)

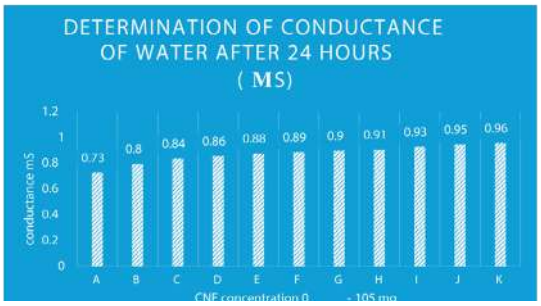


Figure 4: Determination of conductance of water leached out from CNF after 24 hours. (Values of A to K samples are given and same as mentioned in Table -1)

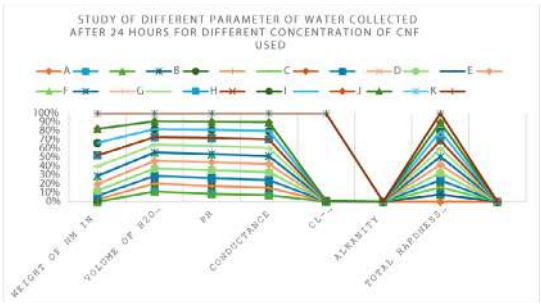


Figure 5: Representation of different parameters studied of leached CNF water samples after 24 hours

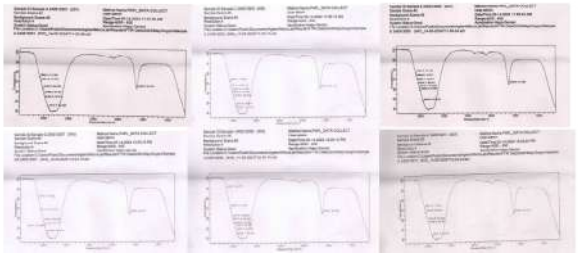


Figure 6: FTIR graphs of few samples (A,C,E,G,I,K) obtained and explained in table 3 are given here.

broad diffraction peak at 24.10 is that of (002), and 45 º of (100) plane of graphitic carbon respectively. The graphitic peaks are weak suggesting more amount of amorphous carbon i.e. lacking a defined ordered structure, they are chemically inert so not a good conductor of electricity, but it is suitable for present work where conductivity or hardness is not a concern. Amorphous carbon lacks a well-defined crystal structure. It is made up of disordered, randomly arranged carbon atoms [4].

Raman Spectra

(Figure 1B) shows D band as well as G band peaks at 1362 cm^{-1} and 1590 cm^{-1} respectively and thus confirming the graphitic nature (Figure 2). D band shows defect in graphitic carbon, whereas G band indicates typical Raman peaks of graphitic carbon. The peak at 1362 cm^{-1} is associated with vibrations of carbon atoms with dangling bonds in plane terminations of disordered graphite. The peak at 1590 cm^{-1} is attributed to the Raman active E_{2g} in plane vibrational mode and is related to the vibration of sp^2 bonded carbon atoms in a two-dimensional hexagonal lattice.

Methylene Blue Test showed that sample has a surface area of 94.61 m^2/g [4].

Water Retention (Adsorption) and Leaching Capacity of CNF after 24 hours

Table 1 reveals data about the amount of water retention after 24 hours of treatment. The water retention capacity was found to be different for varying concentrations of CNF. Initial CNF concentrations shows 32% retention followed by 36% and 38% at optimum concentrations followed by decrease at higher CNF concentrations i.e. up to 28 %; whereas for sample containing no CNF it was found out to be 14% (table 2). This provides valuable information about the role of CNF in holding water and its slow release into the soil thus justifying its utility as a *carrier of nanofertilizer.*

Alteration in Physicochemical Property of Leached water

The study of parameters like hardness, chloride content, pH, alkalinity, conductance of the water samples after 24 hours reveals, there is increase in conductance and hardness of water with increasing concentrations of CNF. The chloride concentration increases at the initial stage then decreases followed by increase again. The pH slightly shifts towards basicity at very high concentration of CNF. These results suggest optimizing the CNF concentration could alter the physico-chemical characteristics and thus

Table 3: FTIR analysis of water samples after 24 hours.

CNF mg Peak No.	OH Str	OH Str Dimeric	Tertiary alc. OH Str	Tertiary OH Str + Phenolic OH	OH Str Tertiary + Alc	HOH + Phenolic OH	HOH Dimeric OH str	C=C Str	C=O Str	NH Str	NH Heterocyclic amine	Sec Alc	Cyanide etc
0 mg (8)Pk	4	2								21 small 1str			
15mg (10)			2	1		2	1		1	1	1		1
25mg (9)			1			1		1	1	5			
35mg (8)	2		2	2				1	1				
45mg (9)	4		3					1	1				
55mg (9)	5			1				1	1			1	
65mg (8)	51 Non-bonded							1	1				
75mg (10)	5		3					1	1				
85mg (10)	61 Non-bonded			1				1	1				
95mg (7)	3			2				1	1				
105mg (7)	4		1					1	1				

prove beneficial for the soil.

FTIR Spectra

FTIR data provides deeper insights into the understanding of the bonding of water molecules with CNF. With varying concentrations of CNF, the bonding pattern of water molecules in the form of -OH (tertiary, secondary, phenolic, dimeric) is intriguing. Moreover, at certain concentrations NH stretch in the form of amide, amines and cyanides is clearly observed. The OH stretch increases with the increase in CNF concentration up to 85 mg and then shows a dip which is indicative of water bonding being optimum at certain CNF levels. The NH bonding is evident at initial concentrations of CNF i.e. at 15 mg and 25 mg. The C=C and C=O stretch indicative of alkenes and ketones, aldehyde, carboxylic acids, esters, amides respectively remains constant at all CNF levels. The FTIR data provides valuable information about the water holding capacity of CNF at particular concentrations; that will allow the slow release of H_2O into the soil and also paves way for more research needed to study the N, P, K bond formation with CNF and its holding capacity**.** The optimization study of CNF concentrations to be mixed with soil will aid in using CNF as a Carrier in the arena of Nano fertilizers.

Conclusion

Biogenic CNF has a good adsorption capacity, and our research findings prove that it has a good water holding capacity. The carbonaceous by product of lignocellulosic biomass produced by a variety of thermochemical processes is called biochar. In contrast to biochar, Biogenic CNF exhibits exceptional physico-chemical properties, such as increased stability, a distinct nanostructure, enhanced catalytic activity, a greater specific surface area, increased porosity, enhanced surface functionality, and surface-active sites. In addition to harmful pollutants (such as heavy metals (Sharon et al 2023) [6, 7-9], herbicides, and antibiotics), biogenic CNF effectively controls the transit and absorption of essential micro- and macronutrients and water retention. However, for large-scale applications, including development, physico-chemical characteristics, and targeted use, a thorough understanding and investigations is necessary. This research reveals -the CNF added to the soil exhibited good water adsorbing capacity. This unique property of biogenic CNF in holding water is promising in the near future as it facilitates slow release of water as required for crop growth in a judicious manner without harming the crop.

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Conflict of interest

The authors express no conflict of interest.

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