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Characterization Of NLP, TW &SCB Using FTIR &SEM Analysis

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ABSTRACT:
In today's industrial revolution, many industries are generating different types of waste impurities which are harmful & toxic for environment as well as for human life. Many researchers had showed interest in overcoming these by using various types of biosorbents with adsorption process to extract the hazardous metals from the industry wastewater. Below paper mainly focuses on three biosorbents namely Neem leaves powder (NLP), Tea wastes (TW) & Sugarcane bagasse (SCB) with their preparation, composition & analysis for use in accomplishing adsorption process to be more effective. Current paper reveals the numerous functional groups responsible for adsorption process & to be detected on exterior surface of biosorbents i.e. Neem Leaves Powder, Tea wastes & Sugarcane bagasse by performing FTIR analysis. It also depicts biosorbents surface morphology at different magnifications by performing SEM analysis. Also, describes Physico-chemical analysis of all biosorbents taken into consideration for analyzing the effectiveness as well as suitability as a biosorbents in doing adsorption to be more conducive.

Keywords— *FTIR, SEM, NLP (Neem Leaves Powder), TW (Tea Waste), SCB (Sugarcane Bagasse)*

I. INTRODUCTION

Adsorption process mainly depends on adsorbent & adsorbates. Different biosorbents are used for adsorption process for removal of heavy metals from wastewater. Different biosorbents have different adsorption capacities. The adsorption capacity of biosorbents mainly depends on their surface area, bulk density, moisture content & pore size [4]. Therefore their characterization is of immense significance & plays vital role. Different techniques are involved to find the root cause of adsorption process to be done effectively as well as responsible functional groups for the said process.

II. PREPARATION OF BIOSORBENTS (NLP, TW & SCB)-

2.1 NLP BIOSORBENT PREPARATION:-

Neem leaves were collected from Neem trees located in nearby locality Mhasrul, Nashik.

1. Firstly, Neem leaves were washed thrice times with water to remove dust & water soluble impurities.
2. After washed out, Neem leaves were exposed to sun for a period of 24 hours until the leaves became crispy.
3. Then it was grinded by using domestic mixture. Sorbent was soaked & sun dried prior to use so that moisture can be removed.
4. Once the Neem leaves were converted into powdery form were sieved using sieve sizes ranging from 100-200 micro meter.
5. The resulting Neempowder was stored in plastic bottle & used as biosorbents for column adsorption studies.

Figure 1. Conversion of Neem Leaves into (NLP) Neem Leaves powder

Table 1: Chemical Composition of Fresh Neem leaves

Moisture	59.4%	**Proteins**	7.1%
Fat	1.0%	**Fiber**	6.2%
Carbohydrates	22.9%	**Minerals**	3.4%
Calcium	510 mg/100 g	**Phosphorous**	80 mg/100 g
Iron	17 mg/100 g	**Thiamine**	0.04 mg/100 g
Niacin	1.40 mg/100 g	**Vitamin C**	218 mg/100 g
Carotene	1998 microgram/100 g	**Calorific value**	1290 Kcal/Kg
Glutamic acid	73.30 mg/100 g	**Tyrosine**	31.50 mg/100 g acid
Aspartic acid	15.50 mg/100 g	**Alanine**	6.40 mg/100 g
Proline	4.00 mg/100 g	**Glutamine**	1.00 mg/100 g

(Source: - Neem Foundation, 1997)

2.2 TW BIOSORBENT PREPARATION:-

Tea wastes were collected from the tea wastes generated from the residence itself as well as from Sad-guruTea stall,Nashik which were usually discarded as waste in the surrounding sides.

1. Firstly, collected tea wastes were soaked & sun dried to remove moisture content, dust & other solid as well as soluble impurities.
2. Then tea wastes weresieved through a set of sieve shakers till the size of particles were reached between 150-250micro meters.
3. The resulting tea waste powder was stored in plastic bottle & used as biosorbents for column adsorption studies.

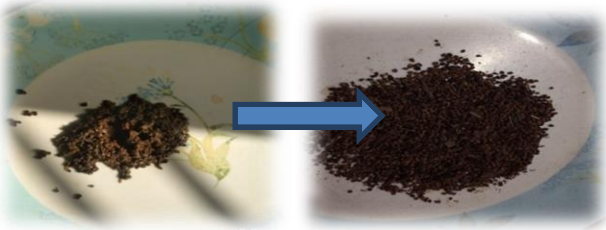


Figure 2. Conversion of Tea wastes (TW) into Tea waste powder

2.3 SCB BIOSORBENT PREPARATION:-

Sugarcane bagasse wastes were collected from the Gajanan-SaiRaswanti, sugarcane juice hawker, Mhasrul, Nashik which were usually discarded as wastes in the surrounding sides.

1. Firstly, sugarcane bagasse wastes were exposed to sun-light until the bagasse gets dried & soaked to make it moistureless. After drying, it was crushed into small pieces for use.
2. Thereafter, it was grinded by using domestic mixture to make it in the powered form.
3. Once the sugarcane bagasse waste was converted into powdery form which was sieved using sieve sizes ranging from 100-200 micro meter.
4. The resulting SCB powder was stored in plastic bottle & used as biosorbents for column adsorption studies.

Figure 3. Conversion of Sugarcane bagasse into (SCB) Sugarcane bagasse powder

Table 2: Chemical Composition of Sugarcane bagasse waste

Sr. No.	Characteristic	Percentage
1	Aplha cellulose	56.54
2	Protein	15.47
3	Lignin	10.75
4	Ash	4.87
5	Magnesium	0.08
6	Calcium	1.40
7	Potassium	0.07
8	Phosphrous	2.86

(*Source:Anil Kumar & Omprakash Sahu, DOI: 10.12691/wjce-1-1-5)

III. FTIR (FOURIER TRANSFORM INFRARED) ANALYSIS-

FTIR (Fourier Transform Infrared) analysis is a technique used to identify and characterize the functional groups present in a sample which can be useful in understanding the chemical composition & potential applications of biosorbents.

3.1 NEEM LEAVES POWDER BIO-SORBENT (NLP):-

NLP have a variety of functional groups, which may be involved in the potential binding of metal ions onto the adsorbents.

Neem leaves powder FTIR analysis can provide information about the presence of various bioactive compounds. Table3 gives presence of numerous functional groups to be detected on the exterior of NLP.

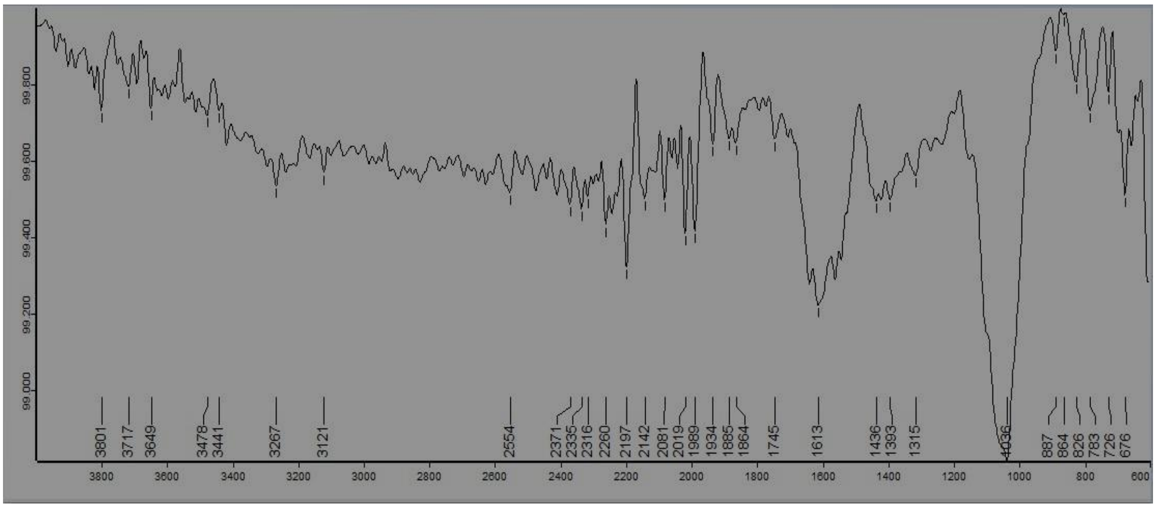


Figure 4. FTIR spectrum of (NLP) Neem Leaves powder

The specific peaks & their intensities may vary depending on the sample preparation, processing & storage conditions.

Table 3. Some fundamental FTIR frequencies of Neem Leaves powder (NLP)

Band position (cm^{-1})/ Wave-number (cm^{-1})	Possible assignments/Bond Type	Type of Compound/ Functional groups identified in NLP bio-sorbent
2142 to 2260	C≡C bond	alkynes
1613	C = C bond	alkenes
1036	C—O bond	alcohols, ethers, carboxylic acids
676 to 864 & 1393 to 1436	C—H stretching	Alkenes
1745	C = O bond	Aldehydes, ketones,
3267 to 3650	O—H stretch vibration	Phenols

In this spectrum (Fig.4), the peaks at 2142 to 2260 cm^{-1} indicate the presence of alkynes groups. The peak at 1613 cm^{-1}suggest the presence of alkenes groups. Peak at 1036 cm^{-1} refers to presence of carboxylic acid groups.Peaks from 676 cm^{-1} to 864cm^{-1}& 1393 to 1436 cm^{-1} indicates alkenes groups (C-H bend). Similarly, aldehydes, ketones & phenols groups observed for different possible bonds as shown in Table 3.

3.2 TEA WASTES BIO-SORBENT (TW):-

TW have a variety of functional groups, which may be involved in the potential binding of metal ions onto the adsorbents.

Table4 gives presence of numerous functional groups to be detected on the exterior of TW.

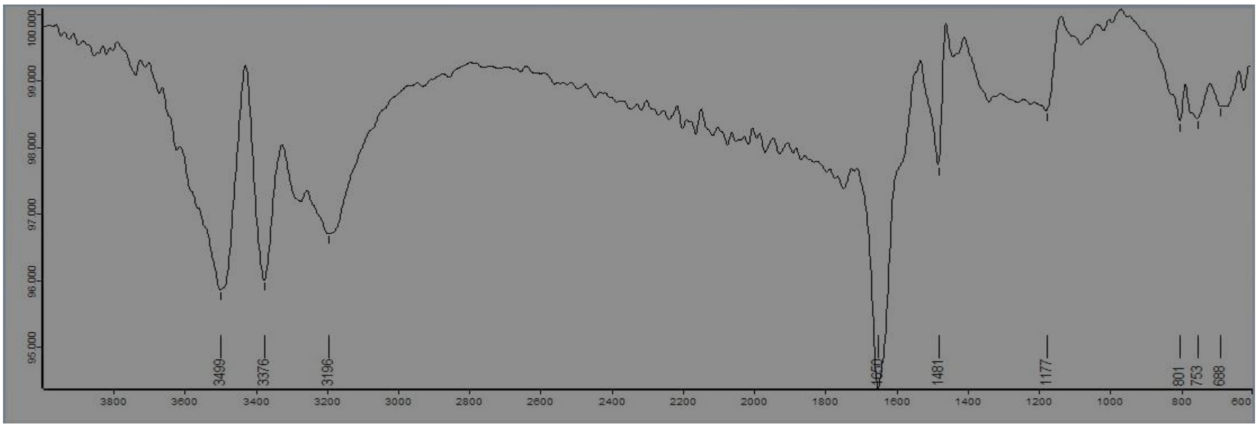


Figure 5. FTIR spectrum of (TW) Tea wastes powder

The specific peaks & their intensities may vary depending on the type of tea, processing conditions & storage methods.In this spectrum(fig.5), the peaks at 688 to 801 cm^{-1} indicate the presence of aromatic rings. Peaks at 1177 cm^{-1} shows presence of amines & amides groups. Similarly, phenols, alkanes & alkene groups identified for different possible assignments as shown in table 4.

Table 4. Some fundamental FTIR frequencies of tea wastes (TW)

Band position (cm^{-1})/ Wave-number (cm^{-1})	Possible assignments/Bond Type	Type of Compound/ Functional groups identified in TW bio-sorbent
688 to 801	—CH—stretching	Aromatic rings
1177	C—N	Amines, amides
3196 to 3499	O—H bend	Phenols
1481	C—H bend	Alkanes
1650	C = C bend	alkenes

3.3 SUGARCANE BAGASSE BIO-SORBENT (SCB):-

SCB have a variety of functional groups, which may be involved in the potential binding of metal ions onto the adsorbents.

Table 5 gives presence of numerous functional groups to be detected on the exterior of SCB.

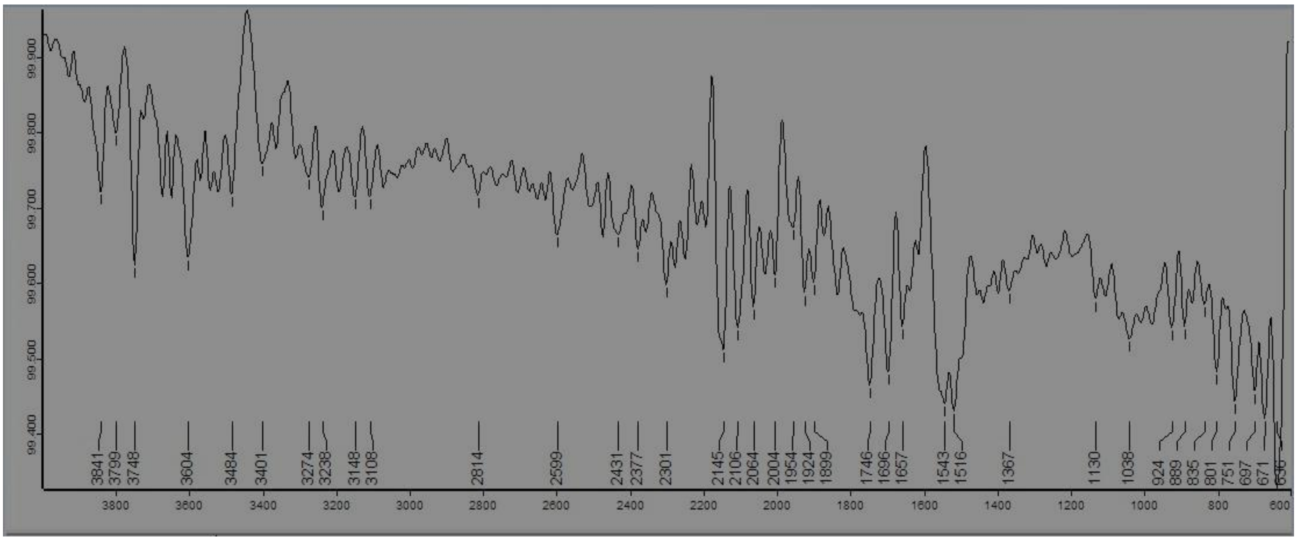


Figure 6. FTIR spectrum of (SCB) Sugarcane Bagasse powder

The specific peaks & their intensities may vary depending on the processing conditions, lignin content & cellulose crystallinity. The FTIR spectrum (fig.6) of sugarcane bagasse exhibit peaks in the regions as shown in table 3. In this spectrum, the peaks 636 to 924 cm^{-1} indicates alkenes groups. Table 5 show different groups with possible assignments based on peaks for SCB powder.

Table 5. Some fundamental FTIR frequencies of Sugarcane Bagasse powder (SCB)

Band position (cm^{-1})/ Wave-number (cm^{-1})	Possible assignments/Bond Type	Type of Compound/ Functional groups identified in SCB bio-sorbent
636 to 924	C—H stretching	Alkenes
1038 to 1130	C—O bond	alcohols, ethers, carboxylic acids
1516 - 1543	C = C bend	Aromatic rings
1657	C = C bend	alkenes
1696-1746	C = O bond	Aldehydes, ketones, esters
2106-2145	C≡C bond	Alkynes
3238 to 3841	O—H bend	Phenols, Carboxylic acids,alcohols

IV. BIO-SORBENT SEM (SCANNING ELECTRON MICROSCOPE) ANALYSIS-

To understand surface morphology of biosorbents, scanning electron microscopic (SEM) micrographs taken using FEI Ltd Model: Nova NanoSEM 450. Adsorbent particles were first gold coated to make the sample conductive, and then SEM was taken at various magnifications. It captures various images by focusing a very high energy beam of electrons on the surface of nano-porous materials.Result gives surface morphology at different magnifications. SEM analysis can provide valuable information about the physical properties and surface characteristics of biosorbents, which can be useful in understanding its behavior in various applications such as: Biofuel production, Composting, Landfill management, Cosmetics and personal care products, Pharmaceutical applications

The surface characteristics and morphology of biosorbents can influence its reactivity, solubility, and interactions with other materials, making SEM analysis a useful tool for characterizing the material.

4.1 NEEM LEAVES POWDER SEM ANALYSIS (NLP):-

SEM analysis can provide valuable information about the physical properties & surface characteristics of Neem leaves powder, which can be useful in understanding its behavior in various applications such as pharmaceuticals, cosmetics & agriculture.

Figure 7, shows the particles appear irregularly shaped and aggregated, with visible surface ridges and pores. The image also shows a crystalline structure, indicating the presence of azadirachtin.

Here are some possible observations from an SEM analysis of Neem leaves powder:

1. Particle shape and size: The SEM images may show irregularly shaped particles with varying sizes, ranging from a few micrometers to tens of micrometers.
2. Surface texture: The surface of the particles may appear rough, with visible ridges, grooves, and pores.
3. Aggregation: The particles may be observed to be aggregated, forming clusters or clumps.
4. Crystalline structure: The SEM images may reveal crystalline structures, such as needles or plates, indicating the presence of crystalline compounds like azadirachtin.
5. Porosity: The particles may exhibit porosity, with visible holes or cavities on the surface.

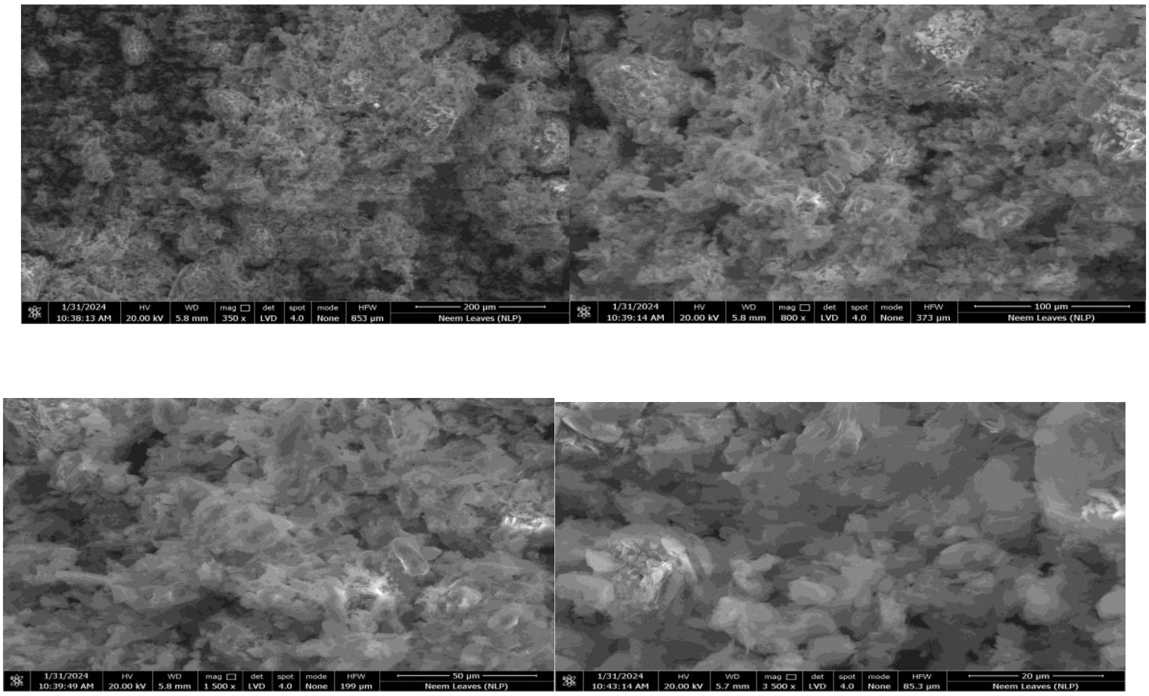


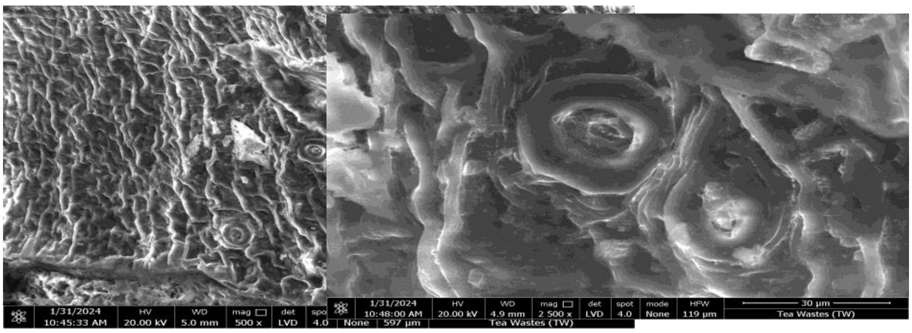
Figure 7. Scanning electron micrograph (SEM) of NLP

4.2 TEA WASTE SEMANALYSIS (TW):-

SEM (Scanning Electron Microscopy) analysis of tea waste powder can provide information about the morphology and surface characteristics of the particles. Here are some possible observations from an SEM analysis of tea waste powder:

1. Particle shape and size: The SEM images may show irregularly shaped particles with varying sizes, ranging from a few micrometers to tens of micrometers.
2. Surface texture: The surface of the particles may appear rough, with visible ridges, grooves, and pores.
3. Aggregation: The particles may be observed to be aggregated, forming clusters or clumps.
4. Fibrous structure: The SEM images may reveal fibrous structures, indicating the presence of cellulose and lignin.
5. Porosity: The particles may exhibit porosity, with visible holes or cavities on the surface.

In figure 8, the particles appear irregularly shaped and aggregated, with visible surface ridges and pores. The image also shows a fibrous structure, indicating the presence of cellulose and lignin.



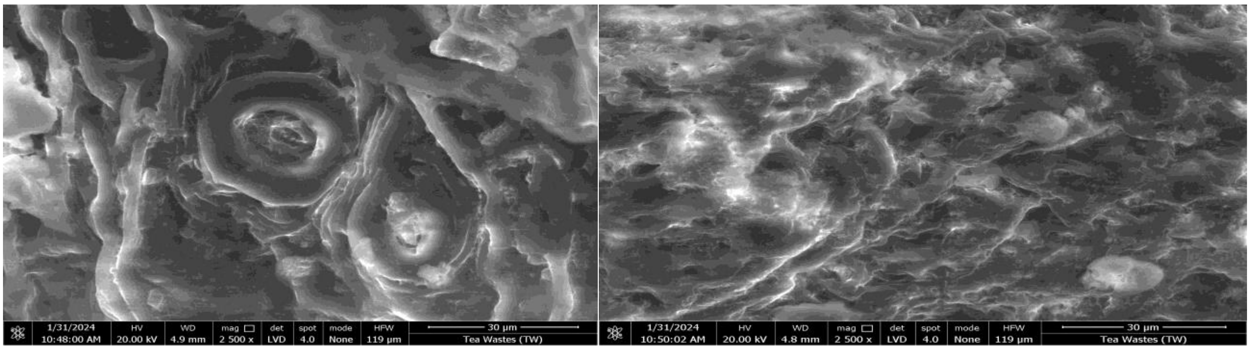


Figure 8. Scanning electron micrograph (SEM) of TW

4.3 SUGARCANE BAGASSE SEM ANALYSIS (SCB):-

Here are some possible observations from an SEM analysis of sugarcane bagasse powder:

1. Particle shape and size: Irregularly shaped particles with sizes ranging from 10-200 micrometers.
2. Surface texture: Rough surface with visible ridges, grooves, and pores.
3. Aggregation: Particles may be aggregated, forming clusters or clumps.
4. Fibrous structure: SEM images may reveal fibrous structures, indicating the presence of cellulose and lignin.
5. Porosity: Particles may exhibit porosity, with visible holes or cavities on the surface.
6. Crystal-like structures: SEM images may show crystal-like structures, indicating the presence of sucrose or other sugars.
7. Surface deposits: Particles may have surface deposits, such as dirt, dirt, or other impurities.

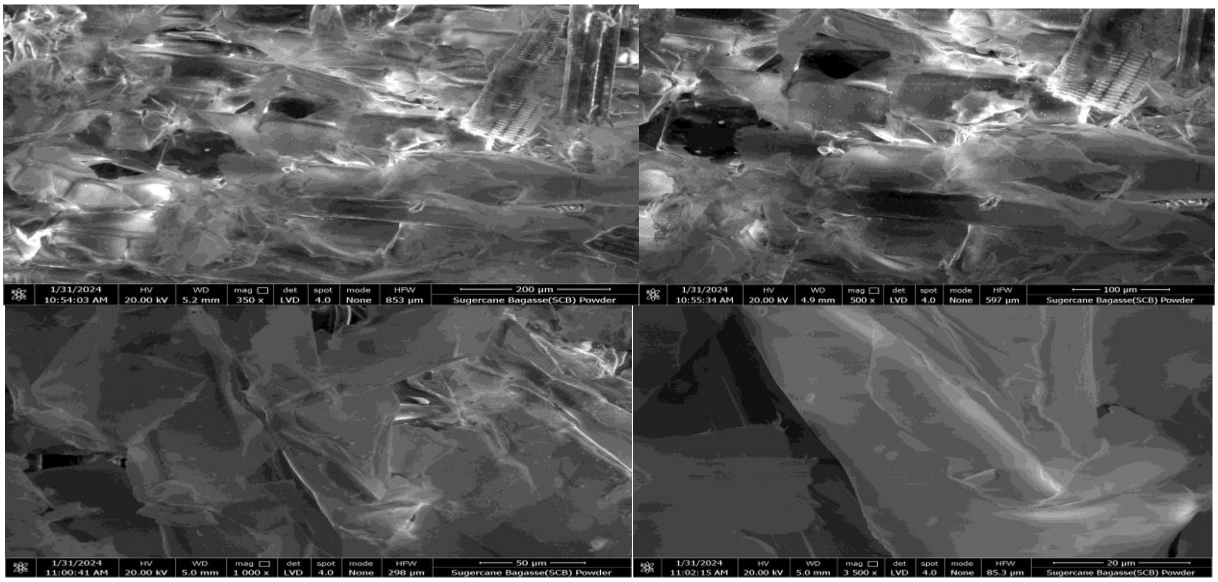


Figure 9. Scanning electron micrograph (SEM) of SCB

Figure 9 shows, the particles appear irregularly shaped and aggregated, with visible surface ridges and pores. The images also showed a fibrous structure, indicating the presence of cellulose and lignin.

V. BIO-SORBENTS PHYSICO-CHEMICAL ANALYSIS-

Table 6 Physico-chemical analysis

Sr. No.	Parameter	NLP Sorbent	SCB Sorbent	TW sorbent	Unit	Method
1.	pH (@25°C)	5.26	5.14	5.73	--	IS 4752: 1994
2.	Moisture Content	3.03	2.34	5.93	%	IS 286:1978
3.	Electrical Conductivity (@25°C)	5.14	0.746	2.21	mmhos/cm	FAO, Sec. III, 5, Page no. 85 :1976
4.	Ash Content	21.2	4.05	2.79	%	IS 10158:1982
5.	Bulk Density	0.8163	0.7427	0.8736	g/cm³	AEC/C/SAP/INS/S-27
6.	Carbon (as C)	84.4	94.1	97.1	%	IS 10158:1982
7.	Volatile Matter	66.3	80	75	g/100g (%)	IS 1350 (Part I) :1984

The Physico-chemical properties of all the three biosorbents are summarized in table 6. The biosorbents were characterized with the purpose of assessing its physical & chemical properties such that a better interpretation of the mechanism involved during the adsorption process can be known.
Biosorbents were analyzed for various parameters like pH, conductivity, moisture content, volatile matter and ash content by applying standard analytical methods as indicated in Table 6. From the Table-6, Following can be inferred.

1) Higher value of bulk density of biosorbents serves better filterability of the biosorbents as an adsorbent with values of 0.82, 0.74 & 0.87 is sufficient to retain substantial amount of liquid. Sorbents having less bulk densities may create operational problems on the column based study.
2) Among all biosorbents, the ash contents of tea waste biosorbents is less compared to others. Its value affects the adsorbent & it can reduce the overall activity of biomaterials, the lower the ash content better the overall activity of the biosorbents.Outof the three biosorbents, ash would be more in case of NLP which would require laborious efforts for cleaning the same, when adsorption is carried out.
3) Percentage of carbon yield of all biosorbents is in close agreement with the ASTM D-3175 standard of 72% when compared. Tea waste contained highest percentage of carbon by virtue of which the carbonization of the preparation of sorbent pertinent to it took less time & sorbent formed readily.
4) Biosorbents which are more soluble in moisture causes colossal problems in maintenance as well as operation. From above table tea waste biosorbents having more moisture content value.
5) The highest volatile matter generated in SCB because of the presence of highest volatile & combustible substance in it, which makes the medium iconic & it also increase the rate of adsorption with increase in volatile matter.

VI. RESULTS & DISCUSSION-

Moisture content of 3.03 (%) of Neem leaves powder (NLP)biosorbent is comparatively lesswith that of investigated by Biswajit Singha et al. (8.33 %) [7] & K. Gopalakrishnan et al. (2.47%) [8] Which indicates less maintenance & colossal problems in operation as well as adsorption process more effective. Also, ash content for NLP is 21.2% which is quite higher than 13.58 % [7] & 14.57% [8]which indicates laborious efforts to be taken for cleaning when the adsorption study is carried out. Bulk density of 0.82 g/cm^3 is also in close agreement with a value of 0.84 g/cm^{3}investigated by K. Gopalakrishnan [8].
The value of moisture content for tea waste (TW) biosorbents is 5.93% which is less compared to value of 10.5 % depicted by Mehrdad Cheraghi et al.[9]. Thus, indicating lower colossal problems in

maintenance as well as operation for adsorption process. The value of bulk density is 0.87 g/cm^3 as compared to 0.353 g/cm^3 that of shown by M. Cheraghi et al. for tea waste which indicates higher filterability of the biosorbents. Ash content of tea waste sorbent is 2.79% as compared to 2.85% in case of M. Cheraghi et al. indicating better activity of tea waste sorbent while carrying out adsorption studies.

The value of moisture content for SCB biosorbent is 2.34% which is lesser compared to value of 8.85 % depicted by Chitaranjan Dalai et al. [12]. Thus, indicating lower colossal problems in maintenance as well as operation for to be used as a biosorbent for adsorption process. Ash content of SCB sorbent is 4.05% as compared to 2.93% in case of Chitaranjan Dalai et al. indicating better activity of SCB sorbent while carrying out adsorption studies. Also, Bulk density of 0.74 g/cm^3 is also in close agreement with a value of 0.82 g/cm^3 investigated byChitaranjan Dalai et al.

Based on FTIR characterization,different functional groups are identified at different wavelengths. SEM images conclude different surface morphology to be observed on exterior of all the three biosorbents at different magnifications so that effective adsorption process is carried out.

VII. CONCLUSIONS-

NLP, TW & SCB biosorbents FTIR characterization results showed presence of different functional groups such as hydroxyl (-OH), carbonyl (-CO), carboxyl (-COOH), and aliphatic (-CH2) groups, which are involved in the binding of the metal ions onto the adsorbents. The SEM images of the NLP, TW &SCB indicated an irregular pattern and porosity of all there biosorbents. The comparative analysis of physico-chemical properties of all three (NTSB-Neem leaves, Tea wastes & Sugarcane bagasse) biosorbents showed that they are useful for column adsorption study.

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