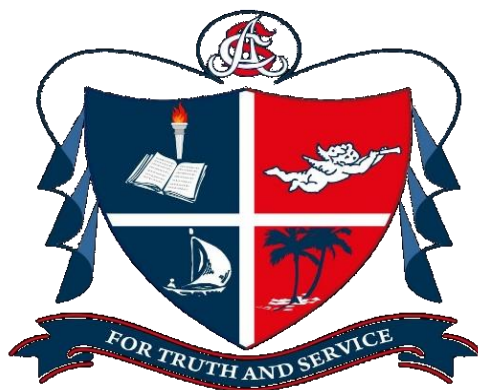A PROJECT REPORT ON

PREPARATION OF CELLULOSE NANO FIBER FROM RICE STRAW AND CORN STRAW AND IT'S USE IN REMOVING Ni^{2+} FROM LAB EFFLUENTS

SUBMITTED TO

ST. ALBERT'S COLLEGE (AFFILIATED TO MG UNIVERSITY) IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF SCIENCE IN CHEMISTRY

SUBMITTED BY

FRANCIS TINO

REG NO: 1901060021

DEPARTMENT OF CHEMISTRY

ST. ALBERT'S COLLEGE AUTONOMOUS ERNAKULAM

CERTIFICATE

This is to certify that the project entitled 'Preparation of cellulose from nano fiber from rice straw and corn straw and its use in removing Ni^{2+} from lab effluents' is a bonified record of the work carried out by Mr. Francis Tino in partial fulfilment of the requirement for the award of Degree in Bsc. Chemistry under the faculty of science of the M.G university during the year 2019-2022.

DECLARATION

I Francis Tino hereby declare that the project entitled 'Preparation of cellulose nano fiber from rice straw and corn straw and its use in removing Ni^{2+} from lab effluents' submitted to Mahatma Gandhi University in partial fulfilment of the requirement for the award of Degree of Bachelor of Science in Chemistry is a record of original project done by us during the period 2019-2022.

Francis Tino

Reg. no: 1901060021

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I express my sincere thanks to Dr. Vijay John Gerson, The Head of Department of Chemistry for giving the permission to undertake the project in the laboratory.

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I also express my sincere thanks to Dr. V. M Bijoy, The Principal of our college. I am also thankful to my parents, teachers, who helped me throughout the endeavour. I also express my sincere thanks to Jijoy T.J, Thomas John, and Jaison Dhas for their support and co-operation as team members for the success of this project.

PREPARATION OF CELLULOSE NANO FIBER FROM RICE STRAW AND CORN STRAW AND ITS USE IN REMOVING Ni^{2+} FROM LAB EFFLUENTS

CONTENTS

INTRODUCTION

Water Pollution due to heavy metals

Water pollution is one of the global challenges that society must address in the 21st century aiming to improve water quality and reduce human and ecosystem health impacts. Industrialization, climate change, and expansion of urban areas produce a variety of water pollutants. The environment and its compartments have been severely polluted by heavy metals. This has compromised the ability of the environment to foster life and render its intrinsic values. Among all the pollutants, heavy metals have received a paramount attention to environmental chemists due to their toxic nature. Heavy metals are usually present in trace amounts in natural waters but many of them are toxic even at very low concentrations. Metals such as arsenic, lead, cadmium, nickel, mercury, chromium, cobalt, zinc and selenium are highly toxic even in minor quantity. Heavy metals are dangerous because they tend to bioaccumulate. Bioaccumulation means an increase in the concentration of a chemical in a biological organism over time, compared to the chemical's concentration in the environment. Compounds accumulate in living things any time they are taken up and stored faster than they are broken down (metabolized) or excreted. Heavy metals can enter a water supply by industrial and consumer waste, or even from acidic rain breaking down soils and releasing heavy metals into streams, lakes, rivers, and groundwater. Heavy metal toxicity can result in damaged or

reduced mental and central nervous function, lower energy levels, and damage to blood composition, lungs, kidneys, liver, and other vital organs. Long-term exposure may result in slowly progressing physical, muscular, and neurological degenerative processes that mimic Alzheimer's disease, Parkinson's disease, muscular dystrophy, and multiple sclerosis. Allergies are not uncommon and repeated long-term contact with some metals or their compounds may even cause cancer.

SOUCES OF HEAVY METAL - Environmental pollution from hazardous metals and minerals can arise from natural as well as anthropogenic sources.

Natural sources are: seepage from rocks into water, volcanic activity, forest fires etc. Pollution also arises from partitioning of polluting elements (which are concentrated in clay minerals with high absorption capacities), between sedimentary rocks and their precursor sediments and water. With rapid industrialization and consumerist life style, sources of environmental pollution have increased. The pollution occurs both at the level of industrial production as well as end use of the products and run-off.

Chromium (Cr)-Mining, industrial coolants, chromium salts manufacturing, leather tanning • Lead (Pb) lead acid batteries, paints, E-waste, Smelting operations, coal- based thermal power plants, ceramics, bangle industry • Mercury (Hg) Chlor-alkali plants, thermal power plants, fluorescent lamps, hospital

waste (damaged thermometers, barometers, sphygmomanometers), electrical appliances etc. • Arsenic (As) Geogenic/natural processes, smelting operations, thermal power plants, fuel • Copper (Cu) Mining, electroplating, smelting operations • Vanadium (Va) Spent catalyst, sulphuric acid plant • Nickel (Ni) Smelting operations, thermal power plants, battery industry.

HUMAN EXPOSURE THROUGH FOOD, AIR AND WATER : Heavy metal pollution of surface and underground water sources results in considerable soil pollution and pollution increases when mined ores are dumped on the ground surface for manual dressing (Garbarino et al., 1995 INECAR, 2000). Surface dumping exposes the metals to air and rain thereby generating much AMD. When agricultural soils are polluted, these metals are taken up by plants and consequently accumulate in their tissues (Trueby, 2003).Animals that graze on such contaminated plants and drink from polluted waters, as well as marine lives that breed in heavy metal polluted waters also accumulate such metals in their tissues, and milk, if lactating. In summary, all living organisms within a given ecosystem are variously contaminated along their cycles of food chain.

HEAVY METAL POISONING AND BIOTOXICITY: The biotoxic effects of heavy metals refer to the harmful effects of heavy metals to the body when consumed above the bio-

recommended limits. Although individual metals exhibit specific signs of their toxicity, the following have been reported as general signs associated with cadmium, lead, arsenic, mercury, zinc, copper and aluminium poisoning: gastrointestinal (GI) disorders, diarrhoea stomatitis, tremor, hemoglobinuria causing a rust–red colour to stool, ataxia, paralysis, vomiting and convulsion, depression, and pneumonia when volatile vapours and fumes are inhaled (McCluggage, 1991). The nature of effects could be toxic (acute, chronic or sub-chronic), neurotoxic, carcinogenic, mutagenic or teratogenic.

Heavy metals are important in many respects to man, especially in the manufacturing of certain important products of human use, such as accumulators (Pb), mercury-arch lamps and thermometers (Hg), utensils (Al) and a wide range of other products (Yaw, 1990; McCluggage, 1991). But the biotoxic effects, when unduly exposed to them could be potentially life threatening hence, cannot be neglected. While these metals are in many ways indispensable, good precaution and adequate occupational hygiene should be taken in handling them. Although heavy metal poisoning could be clinically diagnosed and medically treated, the best option is to prevent heavy metal pollution and the subsequent human poisoning.

ADSORPTION

The phenomenon of adsorption was discovered over two centuries ago: The uptake of gases by charcoal was studied by C. W. Scheele in 1773 and by F. Fontana in 1777. In 1785, charcoal was found to decolorize' solutions by a surface adsorption mechanism.

The term adsorption was first coined in 1881 by a German physicist named Heinrich Kayser. Adsorption is often described as a surface phenomenon where particles are attached to the top layer of material. It normally involves the molecules, atoms or even ions of a gas, liquid or solid in a dissolved state that is attached to the surface. Adsorption is mainly a consequence of surface energy. Generally, the surface particles which can be exposed partially tend to attract other particles to their site. Interestingly, adsorption is present in many physical, natural, biological and chemical systems and finds its use in many industrial application. Adsorption is a process that involves the accumulation of a substance in molecular species in higher concentrations on the surface. If we look at Hydrogen, Nitrogen and Oxygen, these gases adsorb on activated charcoal. Meanwhile, we have to note that adsorption is different from absorption. The two processes involve totally different mechanisms.

For the adsorption process, two components are required. Adsorbate which is a substance that is deposited on the surface of another substance for eg: H_2, N_2 and O_2. Adsorbent surface of a substance on which adsorbate adsorbs eg: charcoal, silica gel , alumina

Mechanism of Adsorption

It is an exothermic process which means that energy is liberated during this process. The amount of heat that gets evolved when one mole of the adsorbate is adsorbed on adsorbent is known as enthalpy. The change in enthalpy is denoted to be negative. The reason behind this is that when adsorbate molecules are adsorbed on the surface, freedom of movement of molecules become restricted and this results in a decrease in entropy. At constant temperature and pressure, adsorption occurs spontaneously.

There are two types of adsorption physisorption and chemisorption.

Physical Adsorption or Physisorption This phenomenon involves the use of weak Van der Waal forces by means of which gas molecules get adsorbed on a solid surface. The characteristics of physisorption are there is no specificity as any gas can be adsorbed onto the surface .It has been observed that highly liquefiable gases are physically adsorbed more

strongly. It is reversible in nature and is dependent on pressure as well as temperature. An increase in pressure decreases the volume of gas and thus increases the adsorption of gas molecules. Conversely, a decrease in pressure will cause the removal of gas molecules from the solid surface. Since the adsorption process is exothermic, the physical adsorption occurs readily at low temperature and decreases with increasing temperature (Le-Chatelier's principle).Porous substances are better adsorbents as an increased surface area promotes adsorption. Does not require energy for activation.

Chemical Adsorption or Chemisorption We can characterize it by the involvement of chemical bonds between the gas molecules and the adsorbent surface Moreover, it results in a unimolecular layer. The characteristics of chemisorption are. The process specific in character means that it will occur only if there is a chemical bond formation between the adsorbent and adsorbate .The process is irreversible in nature .It is an exothermic process and the process is accompanied by an increase in temperature. It occurs slowly at low temperature and occurs at a higher rate with increase in pressure. Just as in case of physisorption, chemisorption is directly proportional to surface area and thus increases with increase in surface area. Since the process involves chemical bond formation, the enthalpy in high. It requires a certain energy of activation.

Physical adsorption   Chemical adsorption
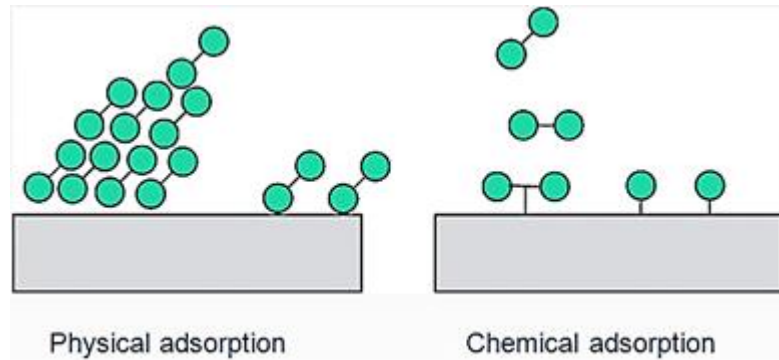
Adsorption constants are basically equilibrium constants. What it means is that they also follow or obey the van 't Hoff equation:

$$\left(\frac{\partial \ln K}{\partial \frac{1}{T}}\right)_\theta = -\frac{\Delta H}{R}.$$

In this formula, the variation of K must be isosteric, that is, at constant coverage. Starting from BET isotherm and assuming that the entropy change is the same for liquefaction and adsorption, we obtain

$$\Delta H_{ads} = \Delta H_{liq} - RT\ln c$$

Precisely, we can say that adsorption is more exothermic than liquefaction.

Adsorption is usually described by isotherms. It is due to the fact that temperature plays an important role or that it has a great effect on the whole process. Moreover, there are several isotherm models that are used to describe the adsorption technique.

FREUNDLICH THEORY

Freundlich adsorption isotherm is obeyed by the adsorption where the adsorbate forms a monomolecular layer on the surface of the adsorbent.
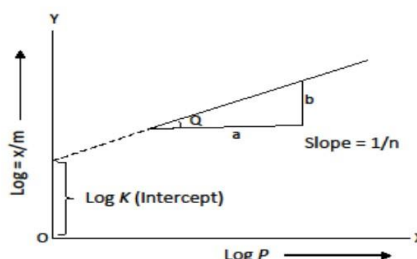
$$\frac{x}{m} = K.p^{\frac{1}{n}}; \log\frac{x}{m} = \frac{1}{n}\log p + \log k$$

x represents the amount of gas adsorbed on the m gram of adsorbent, K and n are adsorption constants, 'p' is pressure n always greater than one. A major drawback of Freundlich adsorption isotherm it fails at high pressure. It could not explain the multi-layered adsorption process.

LANGMUIR THEORY

In 1916, Langmuir proposed the theory of adsorption of a gas on the surface of the solid to be made up of elementary sites each of which would adsorb one gas. It is assumed that all adsorption sites are equivalent and the ability of a gas molecule to get bound to any one site is independent of whether or not the neighbouring sites are occupied. Additionally, it is also assumed that dynamic equilibrium exists between adsorbed and non adsorbed gas molecules. The gas adsorbed behaves ideally in a vapour phase. Only monolayer adsorption takes place. The surface of the solid

is homogeneous.There is no lateral interactive force between the adsorbate molecule. The adsorbed gas molecules are localized.

BET THEORY

The BET theory was proposed by Brunauer, Emmett and Teller in the year 1938. This theory explains the formation of multilayer adsorption during physisorption. This theory also talks about the uniformity in the sites of adsorption of solid surfaces. It assumes that when adsorption occurs at one site it will not affect adsorption at neighbouring sites.

APPLICATIONS OF ADSORPTION

Air pollution masks consist of silica gel or activated charcoal powder, when dust or smoke are paused through them, those particles get adsorbed on the surface of these materials. Separation of noble gases by Dewar's flask process a mixture of noble gases of Ne, Ar, Kr is passed through Dewar's flask in presence of heated coconut charcoal. Argon and Krypton gels adsorbed leaving Neon .By the addition of alum stone to the water, impurities get adsorbed on the alum and water gets purified. Moisture in the air is removed by placing silica gel on which water molecular gets adsorbed. It is used to separate pigments and hormones. In this method of removing the hardness of water, calcium and magnesium ions get adsorbed on the surface of ion exchange resin in the froth floatation process of concentration of ore, the particle gets adsorbed on the froth.

PHOTO CHEMISTRY

Photo Chemistry is a branch of science that deals with the study of chemical effects caused by the radiations those lie in the region of ultra violet and visible light and it includes the phenomenon like absorption and emission of radiations. In photo chemistry the reactions that are induced by the absorption of chemical radiations from our surroundings are termed as photochemical processes. Processes other than chemical reactions induced by absorption of chemical radiations are termed as photo physical processes.

Laws regarding Photo physical process

In the case of pure substance the relationship between the thickness of the absorbing material and extend of visible light absorbed is given by **Lambert's law**. If the absorbing material is a solution, the influence of its concentration on extend of light absorbed is given by **Beer's law**. On combining the above laws termed as **Beer-Lambert Law**, a useful relationship emerges for a solution

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Lambert's Law

"When a beam of monochromatic radiation passes through a pure homogeneous medium, the rate of decrease of intensity of

radiation with thickness of the absorbing medium is directly proportional to the intensity of the incident radiation"

Beer's Law

"When a beam of monochromatic radiation passes through a solution containing an absorbing substance, the rate of decrease of intensity of radiation with the thickness of the absorbing medium is directly proportional to the molar concentration of the solution"

Beer-Lambert Law

"When a beam of monochromatic radiation passes through a solution of an absorbing substance, the rate of decrease of intensity of radiation with thickness of the solution is directly proportional to the intensity of incident radiation as well as to the concentration of the solution."

Application of the Beer-Lambert Law

Beer-Lambert law can be applied in the estimation of a substance in a solution by determining the absorbance of light by the substance in a solution of known concentration. Colorimeter and spectrophotometer are two examples of the instruments that function on the basis of Beer-Lambert law. These are analytical systems that calculates the absorption of a certain wavelength of

light as a method to calculate concentration of an unknown solution.

A series of dilute solutions of known concentration is prepared and their absorbance is measured using colorimeter. A graph is plotted with absorbance against concentration and thus we obtain a straight line graph. Absorbance of a given solution of unknown concentration is measured then the concentration is determined by interpolating the graph.

$$y = 2.00x$$
$$A = 2.00c$$
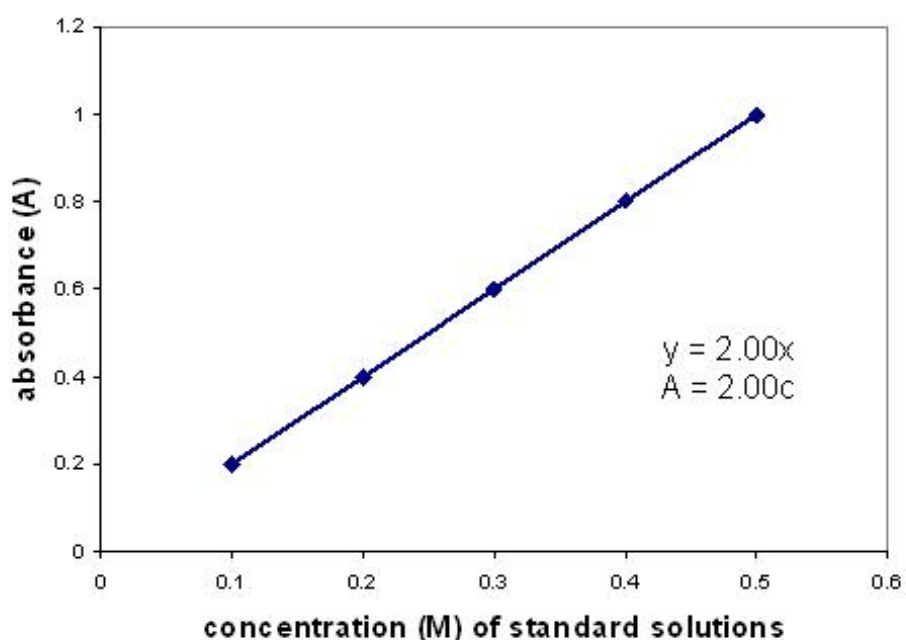
What is colorimetry

Colorimetry is the measurement of the concentration of a particular compound (solute) in a colored solution (solvent). It is used extensively for identification and determination of concentrations of substances that absorb light.

During the experiment, scientists often need to measure quantities of a particular compound in a mixture or the concentration of a solution. It is impossible to determine the concentration in colored solutions through our naked eyes, that's why the **colorimetry** is a quality and important part of the experiment.

For such investigations we need a certain device that is called a **colorimeter**.

What is Colorimeter

Colorimeter is a device that is used to measure light absorbance and transmittance of light that is, how much light is absorbed and how much light passes through a liquid by analyzing color intensity.

The earliest colorimeters relied on the human eye to match the color of a solution with that one of a series of colored discs. The results were too subjective and not particularly accurate.

During colour measurement by the colorimeter the change in the intensity of electromagnetic radiation in the visible wavelength region of the spectrum after transmitting or reflecting by an object or solution is measured. Such a measurement can help to find the concentration of substances, since the amount and colour of the light absorbed or transmitted depends on the properties of the solution, including the concentration of particles in it. A colorimeter is an instrument that compares the amount of light

getting through a solution with the amount that can get through a sample of pure solvent. A colorimeter contains a photocell which is able to detect the amount of light passing through the solution under investigation. The current produced by the photocell depends on the quantity of light hitting it after passing through the coloured solution. The higher the concentration of the colorant in the solution, the higher is the absorption of light; less light passing through the solution means less current created by the photocell. A colorimeter takes three wideband readings along the visible spectrum to obtain a rough estimate of a colour sample. Traditionally, the word 'colorimeter' is used for a device, having three filters, that simulates human vision. Colorimeters can be classified into two types:

•Visual
•Photoelectric.

Visual colorimeters are of two types:

•Visual absorption meters/colour comparators
•True visual colorimeter or tristimulus colorimeter.

The former type compares the colour of the test sample, usually liquid, with that of standard and finds a match between the two. Such instruments are employed for chemical analysis, concentration determination, and grading on the basis of colour.

The tristimulus colorimeter emphasises visual equivalence or psychophysical estimation. In this instrument, radiant power from the light source is incident onto the object. The reflected radiant power passes through one of the three tristimulus filters and falls onto the photo-detector, causing it to give a response proportional to the corresponding tristimulus value of the object-source combination. This raw data is then transferred to a microprocessor for the computation of the absolute CIE tristimulus values. It is a useful tool for monitoring the production of a coloured object. Most commercial tristimulus colorimeters are satisfactorily precise, but their measurements may not agree with the tristimulus values obtained by spectrophotometry.

The oldest and simplest colour comparator is the Nessler tube, which was developed into the Duboscq colorimeter. This type of colorimeter can compare only the optical properties of solutions of a particular colouring substance, but this is all that is required in many colour-assessment tests. There are two vertical cells which accommodate the reference and test solutions, having the same colorant but of different concentrations. Two movable glass plungers can be operated to vary the path lengths L1 and L2 of the absorbing solutions until the colours in both fields in the eyepiece appear the same. Applying the Beer–Lambert law, the concentration of the unknown solution can be determined by multiplying concentration of the known solution with the ratio of path lengths. According to the above law, when the colour of both

solutions appears equal, each of the light beams must have passed through the same number of molecules, and the number is directly related to the concentration of the solution multiplied by the path length , i.e,

$$C_1 \times L_1 = C_2 \times L_2 \quad or \quad C_1 = C_2 \times \frac{L_2}{L_1}$$

If the concentration C_2 is known, we can easily calculate the other concentration. The accuracy of the measurement depends on the visual perception of the observer. Hence in Hilger–Spekker absorption meter, visual assessment was replaced by measurement with photoelectric cells. A calibrated light gate was adjusted till the electrical output matched that for the test light. The equality was assured when there was no deflection of the galvanometer.

True colorimeters define colours in terms of their own primaries. A number of colorimeters were specially developed for colour vision research. These were very elaborate, costly and highly specialised to serve one or a limited number of purposes. The earliest true colorimeter was Clerk Maxwell's colour box (1860), consisting of a prism unit with adjustable slits in the appropriate parts of a light path to control independently the amounts of red, green and blue light beams viewed as a homogeneous colour in an optical viewing unit to match the colour of the sample shown in the other half of the optical unit. The relative aperture areas x, y and z were recorded as the amount of the three primaries.

Three famous visual tristimulus colorimeters used in Great Britain for research on various aspects of normal colour vision were those of Guild (1925), Wright (1927) and Donaldson (1935). Guild used an incandescent source and three colour filters. Donaldson used similar colour filters. Wright used an elaborate optical system to separate three wavelengths, namely 460, 530 and 650 nm, from white light to use as primaries. However, they produce a metameric match, and the results vary from observer to observer. Donaldson (1947) modified the instrument by using six primaries to overcome the problem of metamerism. The instrument was used for field trials on the $2°$ and $10°$ colour-matching functions (Wyszecki, 1964). It lost its popularity owing to the difficulty in calibration and its poor field illuminance. However, some of its basic features have been retained in the designs of other instruments.

MacAdam's Binocular Colorimeter (MacAdam, 1950) provided a large bipartite field for simultaneous viewing by both eyes. The instrument consisted of two symmetric parts, each of which could be used for spectral match with the colour stimulus by the other part. Wyszecki's seven-field colorimeter (Wyszecki, 1965) was designed with an array of seven visual fields for viewing with both eyes. The instrument was developed mainly for research where more than two visual fields were necessary, such as study on colour difference matching, colour-matching ellipses, hue matching, etc.

None of the above colorimeters was commercialised, because they were not very attractive in terms of cost, time and skill required. The Burnham colorimeter (Burnham, 1952) is relatively simple in construction and utilises additive mixing of primary stimuli, made up of coloured filters and a light source. A transparent disc (Fig. 6.3) is divided into three sectors bearing coloured filters, red (R), green (G) or blue (B). The disc is free to rotate about a central axis, and the axis can move horizontally, varying its position with respect to a stationary circular beam of white light shown by a small circular aperture plate, the centre of which has the same vertical position as the centre of the disc. After passing through the disc, light from the beam is mixed by multiple reflections:
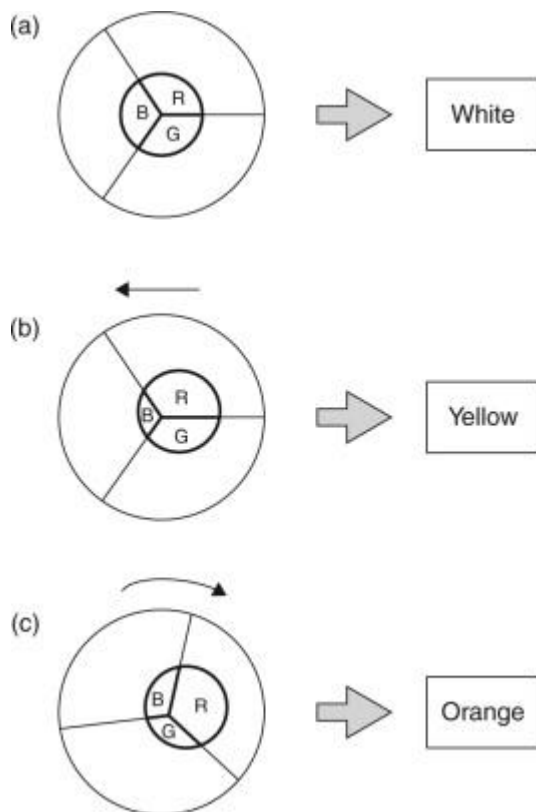
Burnham colorimeter with red, green, blue filters and aperture plate. (Different quantitites of R, G and B in mixture give white, yellow, orange and other colours.)

When the beam and disc are concentric, rotation of the disc is without effect. The transmission of the filters and their relative angular size can be adjusted so that the mixture has the coordinates of a suitable reference white.

❖ As the disc is moved horizontally within the beam, the relative portions of the three primary colours change.

❖ Rotation of the disc changes colour.

❖ The saturation of the colour varies monotonically with the eccentricity of the disc.

The Lovibond comparator is a type of colorimeter made in Britain by The Tintometer Ltd. It was invented in the nineteenth century by Joseph Williams Lovibond and updated versions are still available. The Lovibond colorimeter (1870–1880) is still a popular commercial visual colorimeter, even after 100 years of use and development (Lovibond, 1887; Chamberlin and Chamberlin, 1980). Lovibond colorimeters are used in the analysis of products such as edible and industrial oils, oil derivatives, liquid chemicals, paint vehicles and coatings. They are based on subtractive colour mixing of coloured glass filters. There are 250 Lovibond glass filters for each of three primaries, namely magenta, yellow and cyan, of a very permanent nature.

The filters are graduated in such a way that two '1.0' glasses match a '2.0' glass plus a colourless glass. Equal values of all three together give a grey series down to black.

Putting suitable Lovibond filters in the light filters in the light path, nearly nine million colours of varying brightness can be matched. In fact, the whole visible colour gamut can be covered, except the highly saturated green area. This area can now be covered using a cyan illuminant in the matching field instead of normal north daylight light source. The colour is assessed by visual matching of samples such as surface colours or transparent samples, including liquids kept in the sample field and the coloured filters on the path of illuminating light in the reference field. Automatic Lovibond instruments, which overcome the subjectivity of visual methods, are now available. The menu system guides operators through the selection of operating parameters. Thereafter, measurements are initiated by just a single key press and take less than 25 s to complete. Use of sample cells up to 6" path length ensures precise colour measurement, without multiplying errors, even with unsaturated samples. Sixteen interference filters are used for measurements in some automatic machines.

Visual colorimeters are simple, but slow and tedious in operation. To increase the speed and reproducibility of measurement, photoelectric colorimeters were developed which measure colours directly in colorimetric quantities for one illuminant and

observer (typically illuminant C and secondary standard observer) with the help of broadband filters and photoelectric cells. A photoelectric colorimeter uses a phototube or photocell, a set of colour filters, an amplifier, and an indicating meter for quantitative determination of colour. The principle of construction of a three-filter photoelectric colorimeter is shown in Fig. 6.4. Light reflected (or transmitted) from the object passes through the filters R, G, B successively, due to rotation of the filter-containing disc, and are measured separately by the photoelectric photon detectors. The results will be misleading unless the three filters provide direct reading in terms of CIE tristimulus values or similar standard specification and represent three response curves of CIE standard observer. This type of colorimeter is also known as tristimulus colorimeter. It is almost impossible to obtain such a filter exactly, but available filters may be adequate for routine work. The method may be useless for metameric pairs, i.e. identical in colour under a particular light, but different when seen under a second light source. The Lovibond automatic colorimeter combines the simplicity of a visual system of nomenclature and the speed and accuracy of a photoelectric colorimeter

How does a colorimeter work?

Colorimeters rely on the **Beer-Lambert's Law concept**, which postulates that the absorbance of a substance is proportional to its

concentration. For example, the more concentrated solution gives the higher absorbance reading. Many experiments in chemistry and biology are based on this concept. To obtain a Beer's law curve, several standards (solutions of known concentration) are prepared, and their absorbance values are determined by using a Colorimeter.

Colorimeter has to be calibrated first by using the standard solutions of the known concentration of the solute that is to be determined in the test solution. The standard solutions are poured into the cuvettes which are then placed in the sample holder.

A beam of light of a certain wavelength specific to the assay is directed towards the test solution. Before it reaches the test solution, it passes through a series of lenses and filters. The lens helps in accurate navigation of the beam of light. The filters split the incoming light into different wavelengths and allow the required wavelength to reach the cuvette containing the test solution.

The monochromatic light (light of one wavelength) reaches the test solutions and some of the light gets reflected, some would get absorbed and the remaining would pass through the test solution and falls onto the photodetector. The photodetector sends the pulses to the galvanometer. The galvanometer reads the electrical signals from the detector and displays them in digital form. The reading corresponds to the absorbance or the optical density of the test solution.
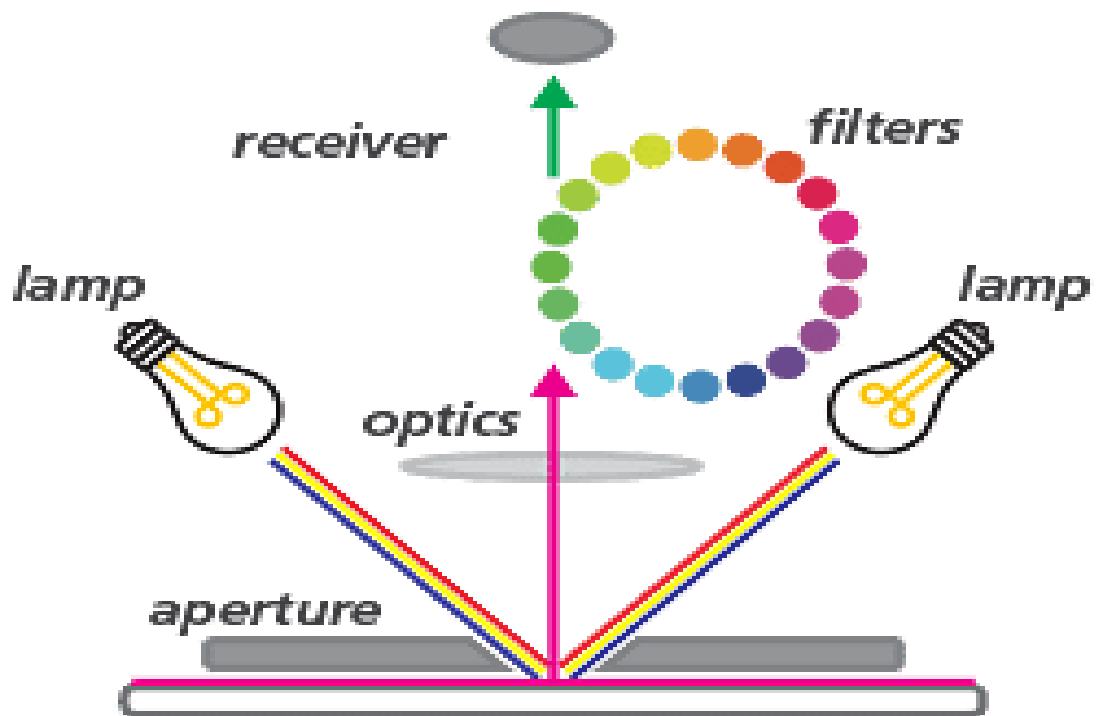
Uses of Colorimeter

The uses of colorimeter are as follows:

❖ Colorimeter is widely used in the medical industry to estimate biochemical samples such as blood, urine, cerebral spinal fluid, plasma, serum, etc.

❖ They are used to analyse the colour contrast and brightness in mobile, computer and television screens to provide users with the best viewing experience.

❖ It also finds its application in the paints and textile industries.

❖ Colorimeter is used in the food and food processing industry.

- ❖ It is used in the printing industry to measure the quality of print paper and printing ink.
- ❖ They are also used to test the water quality and screen for the identification of chemical substances such as chlorine, fluorine, cyanide, iron, molybdenum, etc.
- ❖ They are used in jewellery to measure diamond quality.
- ❖ Colorimeter is used to measure the concentration of haemoglobin in blood samples.
- ❖ It helps to monitor the nutrient concentration in the soil for plant growth.
- ❖ Colorimeter is also used in the pharmaceutical industry to identify substandard products and drugs.

AIM AND SCOPE

AIM AND SCOPE

Aim of this project is to prepare cellulose nano fibers from agricultural waste and find out the adsorbing capacity of the cellulose nano fibers (chemically treated agricultural waste) and other traditional adsorbents such as alumina, charcoal, chitosan and silica gel in removing heavy metals form water. Hence, these adsorbents can be used in reducing water pollution due to heavy metals.

The effluents or pollutants which are discharged into different water bodies are as variable as the human activities. Laboratories and various industries such as chemical plants, dying textiles, paper and pulp mills, iron and steel plants, pharmaceuticals etc. discharged a wide variety of both organic and inorganic pollutants. These pollutants include inorganic compounds like mercury, lead, arsenic, cadmium, chromium, and ions like Ni^{2+} nitrate, Fe^{3+}, cyanide, phosphate etc. The inorganic chemicals when reach in higher concentrations affect the aquatic life adversely and disrupt the natural ecosystem. They can interfere with the suitability of water for drinking and other industrial and domestic purposes.

Water contamination by antibiotics is becoming a worldwide environmental issue. Antibiotic compounds, belonging to

emerging pollutants, are consumed for therapeutic prevention. These contaminants cause toxicity mainly to aquatic fauna and flora even at low concentrations, such as micrograms per liter to nanograms per liter. Although sewage disposal is known as the main source of toxic materials, eco-toxicological effects caused by pharmaceutical compounds present in wastewater and sewage are not completely understood

Heavy metals are ubiquitous pollutants which are introduced into the environment through anthropogenic activities. These metals have the property of biomagnifications and accumulate in the food chain from non-specific compounds inside the cell causing toxicity at cellular level. Through the food chain, it can affect the terrestrial animals including human beings. The presence of heavy metals can create many diseases in man. Mina-mata disease and ouch-ouch disease are examples for diseases result s from heavy metal contamination in water bodies. Our laboratories contribute a major part in water pollution. After the experiments, all the water wastes from the lab reach the nearby water bodies. There are many techniques which have been developed for the removal of heavy metals from the water. Adsorption, being simple in nature and having the advantage of no sludge generation, is used widely in waste water treatment process. If the heavy metals such as iron, chromium, nickel etc. are removed from lab effluent using adsorbents such as charcoal, alumina,

Silica gel and chemically treated agricultural waste, we can protect our water bodies from heavy metal contamination.

After this investigation, the adsorption behavior of Ni^{2+} on the different adsorbent especially on the cellulose nano fibers (chemically treated agricultural waste) and its scope in waste water treatment can be studied.

MATERIALS AND METHODS

MATERIALS AND METHODS

In the first part of this project, the cellulose nano fiber is prepared from rice straw and corn straw. In the second part the adsorption of Ni^{2+} from its aqueous solution was found out using cellulose from rice straw and corn straw, Alumina, Charcoal, Silica Gel, and Chitosan was studied.

MATERIALS REQUIRED

1. Rice straw and corn straw
2. Pure nickel ammonium sulphate
3. Bromine water
4. Concentrated ammonia solution
5. Dimethyl glyoxime
6. Ethanol
7. NaOH
8. H_2SO_4
9. Adsorbents-Alumina, Charcoal, Silica Gel, Chitosan

Preparation of Cellulose Nano Fiber from Rice Straw and Corn Straw

Alkaline–acid pretreatment is the most common method used for lignin, hemicellulose, and pectin solubilization before mechanical isolation of cellulose nano fibers.

The rice straw and corn straw were soaked in 17 % solution for 2 hours in two separate 250 mL beakers. This process is used to raise the surface area of cellulosic fibers and eases the hydrolysis. It was then filtered using ordinary filter paper and washed with water. Then, these fibers were soaked in 1M HCl solution at 60–80°C for 30 minutes, in order to solubilizes the hemicelluloses. Finally, these fibers were transferred into two RB flasks and treated with 2% solution of NaOH for 2 hours at 60–80°C with constant stirring. This process, disrupts the lignin structure, and breaks the linkages between carbohydrate and lignin. The cellulose thus formed is filtered using Whatman filter paper and dried. Alkaline pretreatment is an effective method that can improve cellulose yield from 43% to 84%.

Study of the Adsorbing Capacity

The adsorption of Ni^{2+} from its aqueous solution was found out using Cellulose, Alumina, Charcoal, Silica Gel and Chitosan was studied using colorimetric method.

When dimethyl glyoxime is added to an alkaline solution of a nickel salt which has been treated with an oxidizing agent such as bromine or sodium hypochlorite, a red colour is produced. The red complex soluble in water contains nickel in a higher oxidation state. The complex has absorption maximum at 445 nm.

COLORIMETRIC ESTIMATION OF NICKEL

Standard nickel solution is prepared by dissolving 0.673 g of pure nickel ammonium sulfate, accurately weighed, in distilled water and making up to 1L. Make up 10 mL of this solution to 100 mL. One cubic centimeter of this solution contains 0.01 mg of nickel.

Exactly 2, 4, 6, 8 and 10 mL each of the standard nickel solution are pipetted into five different 50 mL volumetric flasks. 10 mL distilled water is taken in another 50 mL volumetric flask to be used as blank to set the spectrophotometer. Add about 20 mL of distilled water, 2 mL of saturated bromine water and 2 mL of ammonia solution to each flask and mix well. Then add 2 mL of 1% dimethyl glyoxime solution to each flask, mix and make up using distilled water. Keep for 5 minutes and measure the absorbance at 445 nm. Plot the absorbance against volume of standard nickel solution to obtain the calibration curve.

Take 1g each of alumina, charcoal, silica gel, chitosan and 0.2g of the two cellulose samples are taken in six different 250mL conical flask. Add 20mL of the standard Ni^{2+} solution to it and shaken for 30 minutes. After shaking, it is filtered through an ordinary filter paper. Transfer 5mL of the filtrate into two different 100mL volumetric flask and develop color as above. The absorbance is measured at 540nm. Find the corresponding volume equivalence to the standard iron solution from the calibration curve. From that the mass of nickel in this volume and

mass of Ni^{2+} adsorbed can be computed. In addition to that the absorbance is measured using UV- Visible Spectrophotometer.

RESULTS

A) Preparation of Cellulose Nano fiber

RICE STRAW

CELLULOSE NANO FIBER
FROM RICE STRAW

CORN STRAW

CELLULOSE NANO FIBER
FROM CORN STRAW
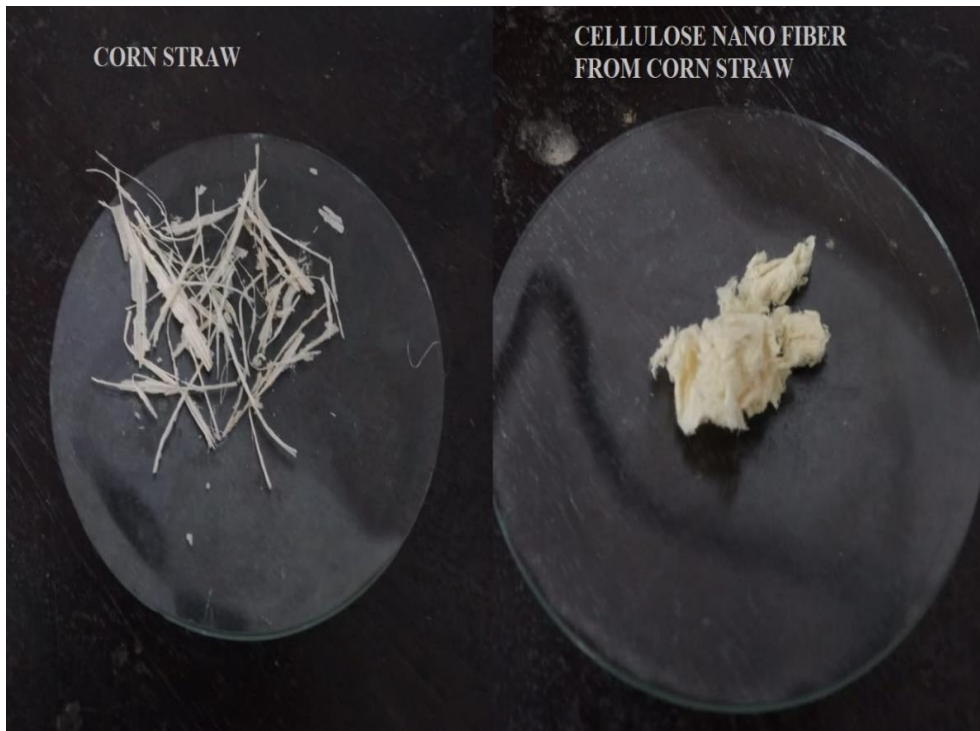
B) Adsorption of Ni^{2+} on various adsorbent

| SL. No	Material	Initial concentration of Ni^{2+}	Conc. Ni^{2+} After adsorption (g)	$\dfrac{x}{m}$	Percentage of Ni^{2+} removed
1.	Charcoal	1.9951×10^{-3} g	0.136×10^{-3} g	1.8591×10^{-3} g	93.20 %
2.	Alumina	1.9951×10^{-3} g	0.352×10^{-3} g	1.6431×10^{-3} g	82.30 %
3.	Chitosan	1.9951×10^{-3} g	0.125×10^{-3} g	1.8701×10^{-3} g	93.70%
4.	Silica Gel	1.9951×10^{-3} g	0.853×10^{-3} g	1.1421×10^{-3} g	57.24 %

Plot of Absorbance against Volume of Nickel Ammonium Sulphate solution
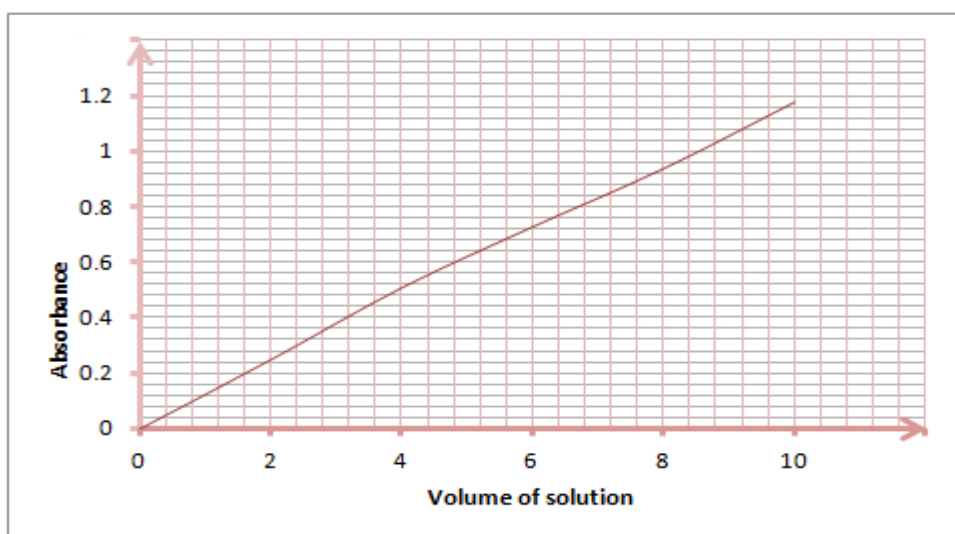
C) **Study of adsorption using UV- Visible Spectrophotometer**

Absorbance of Ni^{2+} solution

Volume of Ni^{2+} solution	Wavelength	Absorbance
2 mL	465.5 nm	0.006
4 mL	465.5 nm	0.098
6 mL	461.5 nm	0.164
8 mL	463.0 nm	0.215
10 mL	462.0 nm	0.281
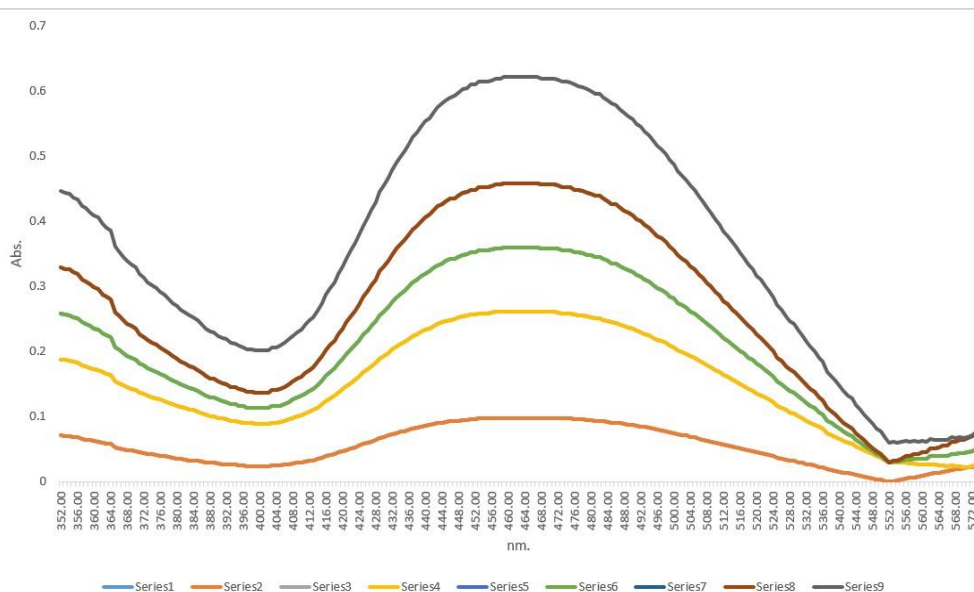
Absorbance of Ni^{2+} solution after adsorption

Volume of Ni^{2+} Solution	Adsorbent	Wavelength	Absorbance
5 mL	Charcoal	465.5 nm	0.000
	Chitosan	465.5 nm	0.000
	Alumina	466.5 nm	0.003
	Silica gel	459.0 nm	0.010
	Cellulose from rice straw	465.5 nm	0.000
	Cellulose from Corn straw	465.5 nm	0.000

DISCUSSION

DISCUSSION

The main objective of this project is to prepare cellulose nano fiber from agricultural waste and find out the adsorbing capacity of the cellulose nano fiber (chemically treated agricultural waste) and other traditional adsorbents such as alumina, charcoal, chitosan and silica gel in removing heavy metals form water. Hence, these adsorbents can be used in reducing water pollution due to heavy metals. It is better to remove the waste from the source itself rather than to treat or clean up waste after it has been discharged into the water bodies.

1. Cellulose nano fiber is prepared from Rice straw and Corn straw

   Using alkaline–acid pretreatment method, we could prepare cellulose nano fiber from rice straw and corn straw. It can be characterized by IR Spectroscopy, scanning electron microscopy, tunneling electron microscopy etc.

2. Removal of Ni^{2+} from lab effluent using various adsorbent

   It is observed that all of them are having good adsorbing power. Since the particle size studies were not carried out, we can compare the adsorbing power of each adsorbent. Firstly, we studied the adsorbing capacity of adsorbent in removing Ni^{2+} from the lab effluents using colourimeter.

   The adsorbing ability of these adsorbents in removing Ni^{2+} was studied using a UV- Visible Spectrophotometer. It is clear that

Nickel – Dimethyl Glyoxime complex shows a characteristic peak at 465.5nm. After adsorption, such peaks are absent, when chitosan, charcoal, cellulose from rice straw and cellulose from corn straw were used as adsorbents. Similar to charcoal and chitosan, cellulose prepared from rice straw and corn straw is also an efficient adsorbent in removing heavy metals from lab effluents and from water bodies. By using them, we can prevent the water pollution to a greater extent.

CONCLUSION

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From the above study it is clear that,

➢ Cellulose nano fibers can be easily prepared by alkaline- acid pretreatment method

➢ Charcoal, Chitosan, Cellulose nano fibers, alumina and silica gel are good adsorbents

➢ Similar to the conventional adsorbents such as charcoal and alumina, cellulose nano fibers are also an efficient adsorbent in removing heavy metals, antibiotics from the effluents from laboratories.

➢ It is not convenient to shake the lab effluents with adsorbent during our lab work. Therefore, we can make a chromatographic column using Cellulose nano fibers, Charcoal and chitosan. By using such a chromatographic column, we can prevent the pollutants from reaching the nearby water bodies or by placing the packets of powdered water melon seeds and other adsorbents in the sink of our laboratories, the extent of pollution of water bodies can be reduced.

➢ It is better to remove the waste from the source itself rather than to treat or clean up waste after it has been discharged into the water bodies.

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