

SYNTHESIS AND CHARACTERIZATION OF CHITOSAN - CELLULOSE-NANO COMPOSITE AND ITS APPLICATION

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Engineering degree in
Chemical Engineering

By

MOHD SALIM (Reg. No. 38190012)



DEPARTMENT OF CHEMICAL ENGINEERING

SCHOOL OF CHEMICAL ENGINEERING

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

Accredited with grade "A" by NAAC

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APRIL 2022



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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **MOHD SALIM (Reg. No. 38190012)** who carried out the project entitled "SYNTHESIS AND CHARACTERIZATION OF CHITOSAN -CELLULOSE-NANO COMPOSITE AND ITS APPLICATION" under our supervision from September 2021 to April 2022.

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Submitted for Viva voce Examination held on _____

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DECLARATION

We **MOHD SALIM (Reg. No. 38190012)** hereby declare that the Project Report entitled **“SYNTHESIS AND CHARACTERIZATION OF CHITOSAN - CELLULOSE-NANO COMPOSITE AND ITS APPLICATION”** done by us under the guidance of **Dr. S SATHISH** is submitted in partial fulfilment of the requirements for the award of Bachelor of Engineering degree in Chemical Engineering.

DATE: 30.04.2022

SIGNATURE OF THE CANDIDATE

PLACE: CHENNAI

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ABSTRACT

Nowadays, one of the major environmental problems faced by humanity is the increasing contamination of freshwater systems. The factors include effluent-containing dyes, heavy metals, ions, and other waste pollutants. One of the most effective methods for copper removal from wastewaters is the process of adsorption. Chitosan is gaining interest as one of the sorbents. In this project first, we have chitosan, from that chitosan we have prepared nano chitosan, after that we have prepared cellulose from banana fiber. Now we will prepare nanocomposites beads from nano chitosan solution, sodium alginate solution, and microcrystalline cellulose solution. Chitosan is characterized by the high effectiveness of copper sorption from an aqueous solution. Its sorption capacity may result from many processes including ionic exchange, formation of H₂ bonds, and physical & chemical adsorption. The naturally occurring polysaccharides such as alginic acid and chitosan have been recognized as the most effective adsorbent to eliminate low levels of heavy metals ions from the wastewater streams. The adsorption capacity can be controlled by sorbent particle size and the nano chitosan were produced from chitosan by an experimental approach. The agricultural by-products (banana, jute, coir, etc) are used for the production of adsorbents because they are renewable available in large quantities and inexpensive. Chemical modification such as steam explosion is done with cellulosic raw material which renders them more effective for the collection and binding of various metal ions. This applying steam explosion method the cellulose was extracted from banana stream fiber in microcrystalline form.

Key words: chitosan, nano chitosan, cellulose, nanocomposite beads, adsorption, sorbent

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CHAPTER - 1

INTRODUCTION

Water is a vital compound for supporting the assemblage on earth. There are numerous sources of water to support life in fresh ecosystems. Fresh ecosystems play a vital role as they supply services to support life methods in living creatures. But, the growing population, urbanization, industry place forceful pressure on the fresh ecosystems, with the result altering the standard state of affairs of freshwaters by adding immense quantities of contamination. Water contaminations not solely degrade the standard of fresh, but at the same time create harmful risks to the full surroundings. The chemical substances in a fresh scheme can't be neutral simply because of their advanced structure and have nice potential to stay intact in any reasonable environment. These substances these days are unceasingly intercalary into the fresh scheme on day to day by approach of discharging untreated domestic, industrial, and agricultural waste products. Most of those substances get accumulated within the bottom sediments and extremely minute concentrations within the variety of organic and inorganic constituents stay either in suspended kind or resolution in liquid medium of the fresh scheme. The presences of those forms of pollutants in the fresh scheme have long impacts on aquatic and associated assemblage. Therefore, would like of associate hour is to watch the standard of the fresh scheme on regular basis and the focus ought to lean to the treatment of effluents before its discharge into the fresh scheme. The rising pollution of freshwater systems is currently one of humanity's greatest environmental challenges. Dye-containing wastewater, heavy metals, ions, and other waste contaminants are among the culprit. Adsorption is one of the most successful methods for removing Copper from wastewaters. As a sorbent, chitosan is gaining popularity. Because of qualities such as faster deposition rate and higher removal effectiveness for COD (organic matter), SS (suspended solids), and metal ions, chitosan is a useful flocculant for tap water treatment. The high-quality flow allows treatment systems to operate more effectively, which has advantages both upstream and downstream. Time to settle down is quick. Filtration and backwashing are simple. Costs of transporting and disposal are reduced. Removes fine sediments, heavy metals, and PCBs, among other substances. Improve the purity of the water. Agricultural by-products (banana, jute,

coir, and so on) are utilized to make adsorbents because they are renewable, available in huge quantities, and inexpensive. Cellulosic raw materials are chemically modified, for as by steam explosion, to make them more effective at collecting and binding various metal ions. The cellulose was recovered from banana stem fiber in microcrystalline form using the steam explosion method.

Chitosan is a linear polysaccharide made up of N-acetyl-D-glucosamine and -(14)-linked D-glucosamine (deacetylated unit) that are randomly dispersed (acetylated unit). It's manufactured by treating shrimp and other crustacean chitin shells with an alkaline material like sodium hydroxide.

Chitosan has a variety of commercial and biological applications. It can be used as a seed treatment and biopesticide in agriculture to assist plants fight fungal infections. It can be used as a fining agent in winemaking, as well as a preservative. It can be utilised in a self-healing polyurethane paint coating in industry. It can be used to assist distribute medications through the skin and is beneficial in bandages to prevent bleeding and as an antibacterial agent in medicine.

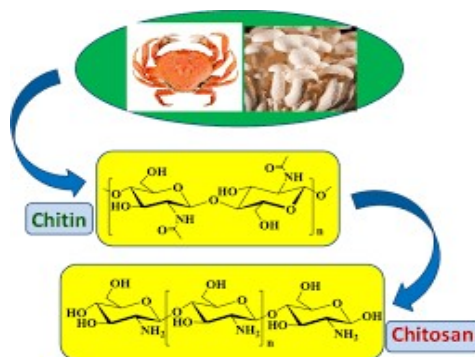


Fig 1.1

Chitosan is a sugar found in the skeletons of shellfish such as crab, lobster, and shrimp. It's utilised in medicine and drug production. Chitosan is a fibrous material that may help the body absorb less fat and cholesterol from diet. When administered to wounds, it also aids in blood clotting. People use chitosan for a variety of reasons, including high blood pressure, high cholesterol, obesity, wound healing, and so on, however there is no scientific evidence to back up many of these claims.

CHAPTER - 2

LITERATURE REVIEW

CELLULOSE BASED PACKAGING FILMS CONTAINING NATURAL ANTIMICROBIAL AGENTS

Kamile Nazan Turhan; Department of Food Engineering, Faculty of Agricultural Technology and Food Science, Izmir University of Economics, Turkey

Cellulose is one of the most abundant biopolymers on earth, occurring in wood, cotton, hemp and other plant-based materials and serving as the dominant reinforcing phase in plant structures. It is one of the most plentiful renewable resources. As a consequence of its chemical structure, it is highly crystalline, fibrous, and insoluble. Cellulose based edible films are very good barriers to aroma, oxygen and oil transfer like other hydrophilic films.

The Adsorption Study of Copper Removal by Chitosan-Coated Sludge Derived from Water Treatment Plant.

Meng-Wei Wan, Chan-Ching Wang, and Chien-Min Chen

Sludge, chitosan and CCS bio adsorbents where *C. Equilibrium Isotherms Studies Adsorption* isotherm models describe the relationship between adsorbates and adsorbents at equilibrium. The two most well-known isotherm models are the Langmuir and the Freundlich. In this study, equilibrium isotherms were used to determine the adsorption mechanism of CCS for Cu (II) ions. The adsorbents were combined with fixed volumes (30 mL) characterized and among these three, the CCS illustrates higher surface area and pore volume when compared to the other two adsorbents. Obviously, the metal uptake capacity of this adsorbent is higher due to its free adsorption sites. SEM/EDS analysis was exploited to illustrate the variation of morphology, mineralogical and chemical composition for each adsorbent.

Effect of Fiber Cross-Sectional Shape and Nano ZnO/Modified Chitosan Loading on Antibacterial Properties

Natthaphop Suwannamek, Nanjaporn Ruangpaisarn, Chureerat Prahsan

Melt spun antibacterial polypropylene cross-sectional form fiber's with nano ZnO and modified chitosan were used. The effects of nano ZnO and modified chitosan loading on two types of fiber cross-sections, round and worm cross-sectional shape, were investigated. The antibacterial characteristics, surface area, and fiber crystallisation were all looked into. The cross-sectional form has a direct relationship with the fiber's surface area and antibacterial capabilities. When compared to ZnO, lesser modified chitosan loading yielded better antibacterial activities.

A brief overview on chitosan applications

Ambore Sandeep, Kanthale Sangameshwar, Gavit Mukesh, Rathod Chandrakant, Dhadwe Avinash

Chitosan is made by the alkaline deacetylation of chitin, a significant component of the protective cuticles of crustaceans such as crabs, shrimps, prawns, and lobsters. A lot of research is being done on chitosan to see how it might be used in clinical, biomedical, food, agricultural, and horticultural settings. Chitosan is biodegradable, non-toxic, bacteriostatic, and fungistatic, making it useful in the pharmaceutical industry. The current review provides in-depth information on the properties, applications, and potential applications of chitosan in the pharmaceutical industry.

An Overview of Chitosan Nanoparticles and Its Application in Non-Parenteral Drug Delivery

Munawar A. Mohammed, Jaweria T. M. Syeda, Kishor M. Wasan, and Ellen K. Wasan

The goal of this review is to give a general overview of chitosan-based nanoparticles for non-parenteral applications, as well as to highlight current research on sustained release and mucoadhesive chitosan dosage forms. Chitosan is a biodegradable, biocompatible polymer that has been certified for wound treatment applications and is considered safe for human consumption. Chitosan has been utilised as a carrier in polymeric nanoparticles to deliver drugs via a variety of ways. Chitosan is a polymer with a wide range of possible

applications due to its chemical functional groups that can be changed to meet specific aims. Nanoparticles (NP) made from chitosan and chitosan derivatives have a positive surface charge and mucoadhesive characteristics, allowing them to stick to mucus membranes and release the therapeutic payload over time. The use of chitosan-based NP in non-parenteral drug administration for cancer, gastrointestinal illnesses, lung diseases, drug delivery to the brain, and ocular infections will be demonstrated in this review. In vitro and in some in vivo settings, chitosan has a low toxicity. Recent research on chitosan-based NP for non-parenteral drug delivery, as well as chitosan characteristics, modification, toxicity, pharmacokinetics, and preclinical trials, is discussed in this review.

FORMULATION AND EVALUATION OF CYCLOPHOSPHAMIDE LOADED CHITOSAN NANOPARTICLES CONJUGATED WITH FOLIC ACID FOR CANCER TARGETING.

Solanki, Gopi Shah, Nirmal Chauhan, Sachin Aundhia, Chintan Javia, Ankur Seth, A. K.

The use of nanotechnology for cancer therapy has grown in popularity during the last decade. The creation of smart targeted nanoparticles that can deliver drugs at a sustained rate directly to cancer cells could improve efficacy and reduce toxicity in the treatment of primary and metastatic tumours. Due to their numerous advantages, such as strong biocompatibility, biodegradability, and non-toxic qualities, Chitosan nanoparticles have recently been extensively explored as a potential drug carrier. The goal of this study was to create cyclophosphamide-loaded folate-chitosan conjugated nanoparticles (FA-CS NPs) that might be used to improve tumor-targeted medication delivery. The anticancer drug delivery effectiveness of Chitosan nanoparticles was improved by ionic cross linking, and folic acid (FA) was conjugated with CS NPs through electrostatic contact between the cationic amino group of Chitosan and the anionic carboxyl group of FA. Different concentrations of polymer (Chitosan) and cross-linking agent were used to optimise the method's processing parameters (TPP). Entrapment efficiency, particle size, drug release for 24 hours, and stability for three months were all tested on the NPs. The percent EE was determined to be 89.742.36 and the drug release was found to be 92.42.15. The NPs were confirmed to be stable during the

stability study. As a result, it can be concluded that FA-CS NPs can be exploited for anticancer drug delivery targeting.

Quaternary ammonium salt of chitosan: preparation and antimicrobial property for paper

Pengtao Liu*, Weisheng Meng, Shuai Wang, Yonghui Sun, Muhammad Aqeel Ashraf

The reaction of chitosan with glycidyl trimethyl ammonium chloride yielded a series of 2-hydroxypropyltrimethyl ammonium chloride chitosan (HACC). FT IR and ¹H NMR spectroscopies were used to analyse the structure of HACC, and it was discovered that the substitution reaction occurred mostly on the N element. HACC was tested for antimicrobial activity against *S. aureus*, *E. coli*, and *A. niger*. The inhibitory effects of HACC solutions varied with HACC concentration, quaternization degrees, pH values, metal ions, and heat treatment, according to the findings. The inhibitory zone method was used to investigate the antibacterial capabilities of hand sheets made from HACC. The sheets had high antimicrobial activities against *S. aureus* and *E. coli*, but a low inhibition rate against *A. niger*.

Fat resistance properties of chitosan-based paper packaging for food applications
F.Ham-Pichavanta, G.Sèbebe, P.Pardona, V.Coma

The goal of this research was to see if chitosan coating could replace fluorocarbon treatment of paper-based materials for oil barrier packaging. The nature of interactions between fatty acids chosen as model lipids (such as oleic acid, which is widely used in the food industry) and chitosan was examined in this study. The impact of fatty acid concentration as well as the effect of pH were investigated. Chitosan's ability to interact with lipid has been demonstrated to be pH dependent. The polycationic chitosan stabilised the fatty acid emulsion in acidic conditions, which was attributed to its ability to connect with anionic lipid molecules, as seen by the fast change in residual free oleic acid content with a little pH change.

Because calcium absorption from food reduces the nutritional value of the product and because chitosan has chelating capabilities, the interaction of chitosan and calcium was examined at the same time. Chitosan adsorption of Ca²⁺ ions was found to be minor no matter what pH was utilised.

CHAPTER - 3

AIM AND OBJECTIVE

3.1 AIM

The important aim is to Synthesis and characterization of chitosan -cellulose-nano composite and its application

3.2 OBJECTIVE

The main objective of this project is to Dye removal adsorption studies, Crab shells are used to make nano chitosan. Banana fibre is used to make cellulose.

CHAPTER – 4

MATERIALS AND METHODS

This is a list of the many materials used to make chitosan.

Nashik HCl gathered crab shells (protinudae sp) (2N), NaOH(3,15N), H₂O₂(0.1N), H₃COCH₃, CH₃COOH(3N), STPP STPP STPP STPP STPP ST (sodium tripolyphosphate), C₂H₂O₄.2H₂O is a chemical compound with the formula C₂H₂O₄. (Oxalic acid) Fish scales were acquired from the Nashik Fish Market. Stem of a banana, NaOH, Blue dye that reacts.

Grinding and polishing specimen surfaces to an ultra-smooth surface is typically required for backscattered electron imaging, quantitative X-ray analysis, and X-ray mapping. Carbon-coated specimens are frequently used in WDS or EDS analysis. Metals are not often coated prior to SEM imaging since they are conductive and provide their own path to ground.

Fractography is the investigation of shattered surfaces using a light microscope or, more typically, a SEM. The shattered surface is trimmed to size, cleaned of any organic residues, and mounted on a specimen holder for SEM observation. For SEM viewing, integrated circuits can be sliced with a focused ion beam (FIB) or other ion beam milling device. In the first scenario, the SEM may be integrated into the FIB, allowing for high-resolution imaging of the process outcome. Chemically polished metals, geological objects, and integrated circuits can all be viewed in the SEM. High-magnification imaging of inorganic thin films necessitates the use of specialised high-resolution coating processes.

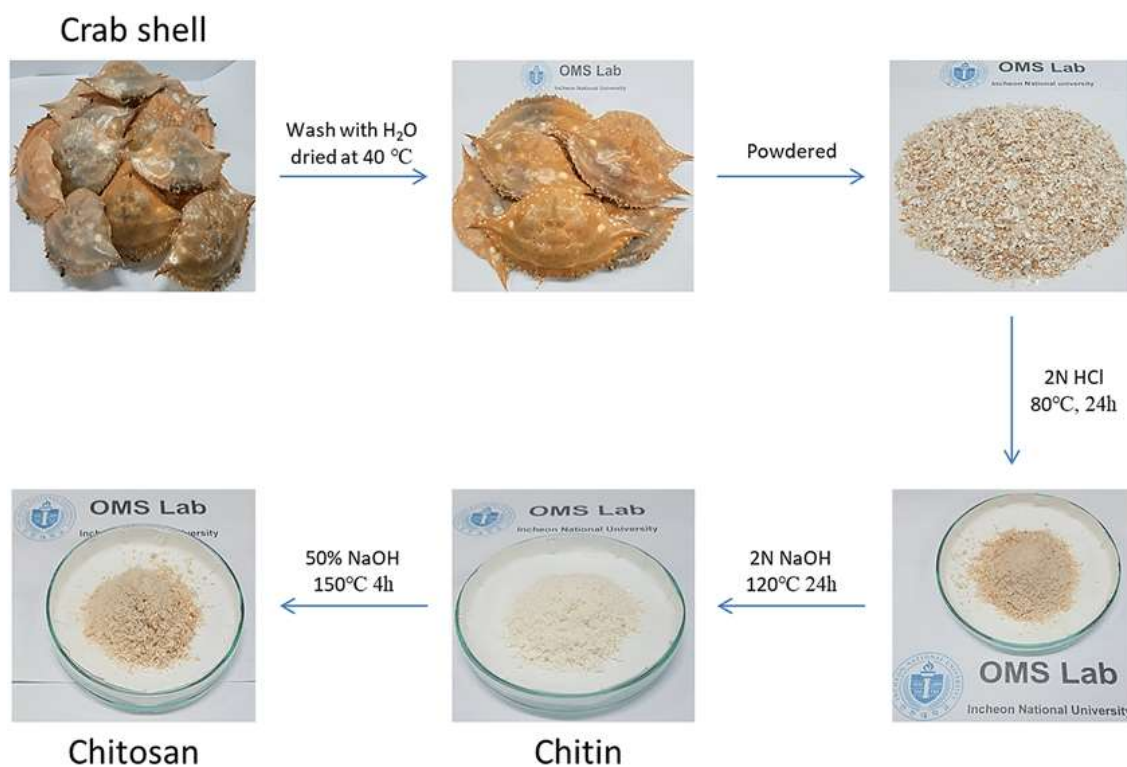


Fig 4.1

4.1 PREPARATION OF CHITOSAN

Chitosan is a generic word for a group of polymers formed by various degrees of chitin deacetylation. In fact, chitin and chitosan are distinguished by the degree of acetylation, which reflects the balance between the two types of residues (Figure 1). The result is called chitosan when the DA (given as a molar percentage) is less than 50 mol% and it becomes soluble in acidic aqueous solutions [94].

Deacetylation not only removes acetyl groups, but it also causes depolymerization, as evidenced by changes in chitosan MW. Enzymatic preparations or a chemical procedure can convert chitin to chitosan. Chemical methods are widely employed in the commercial preparation of chitosan due to their low cost and adaptability for mass production.

The following procedure is used for the preparation of Chitosan: -

- Crab shells that had been frozen were thawed at room temperature and washed under running water to remove organic and other impurities.
- The tissues are then removed by boiling them for 30 minutes at 1000 degrees Celsius.

- After that, it is dried for 120 minutes at 80 degrees Celsius.
- After that, they're crushed and blended to make powder.
- The resulting powder was demineralized for 150 minutes at 60°C with 2M HCL.
- Shells were then deproteinized with 3M NaOH at 80°C for 120 minutes.
- Heat at 2000°C was used to remove pigment remnants, which were then rinsed and dried for 2 hours at room temperature.
- Under vacuum and continual stirring, chitin was deacetylated with NaOH at 110°C.
- After that, it is allowed to cool for 30 minutes at room temperature.
- Last but not least Chitosan was washed with DM water until the filtrate reached a neutral pH, then dried for 24 hours at 600°C.



Fig 4.2

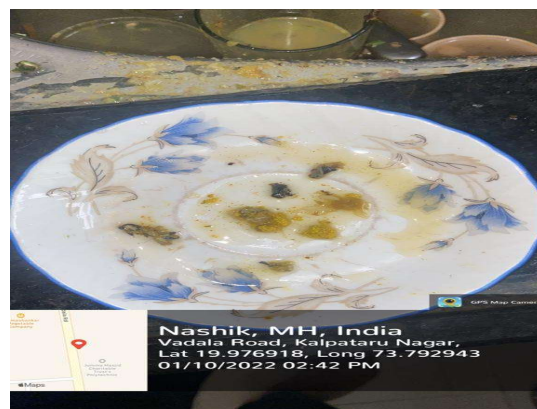


Fig 4.3



Fig 4.4



Fig 4.5



Fig 4.6



Fig 4.7



Fig 4.8

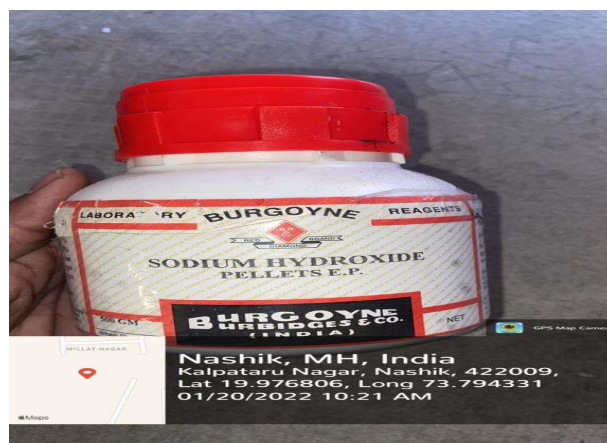


Fig 4.9



Fig 4.10



Fig 4.11

4.2 PREPARATION OF NANO CHITOSAN

- The solution was agitated for 20 minutes after 1 g of chitosan was dissolved in 2 percent acetic acid.
- In 100ml of DM water, dissolve 0.8 g sodium tri-polyphosphate.

- Then, drop by drop, this solution is added to the chitosan solution and agitated for 30 minutes.
- Because of ionic cross linking, a milky white emulsion of chitosan micro particles developed between STPP and chitosan solution.
- The solution is then stored overnight to allow nano chitosan to settle at the bottom.
- The solution was decanted, and the thick suspension of nano chitosan that had settled at the bottom was kept in the fridge.

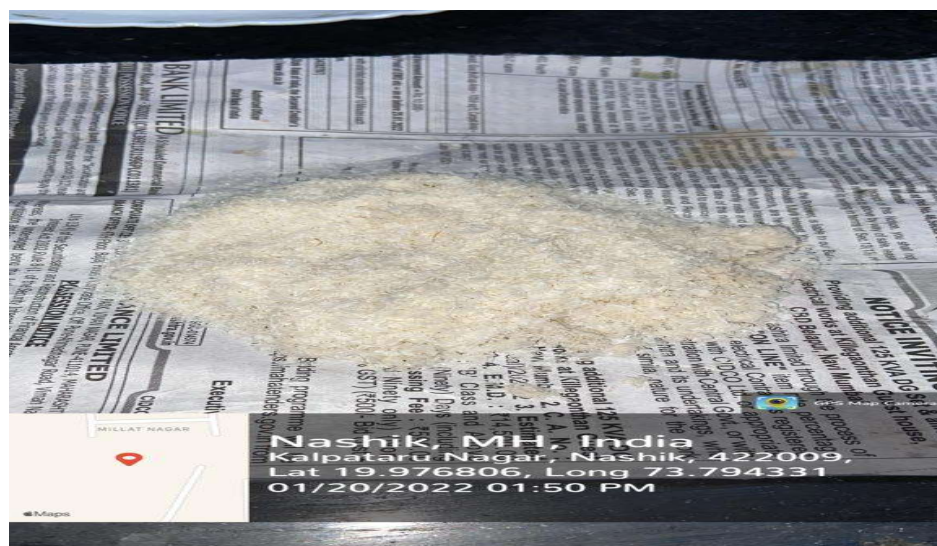


Fig 4.12

1g Chitosan

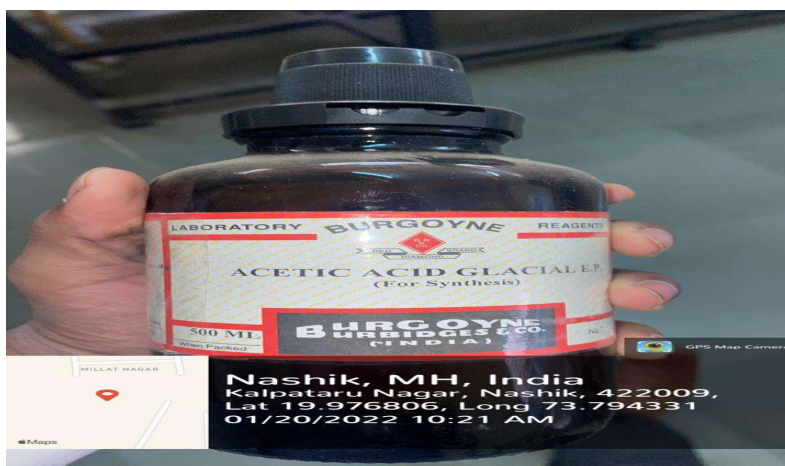


Fig 4.13

Acetic acid



Fig 4.14

STTP

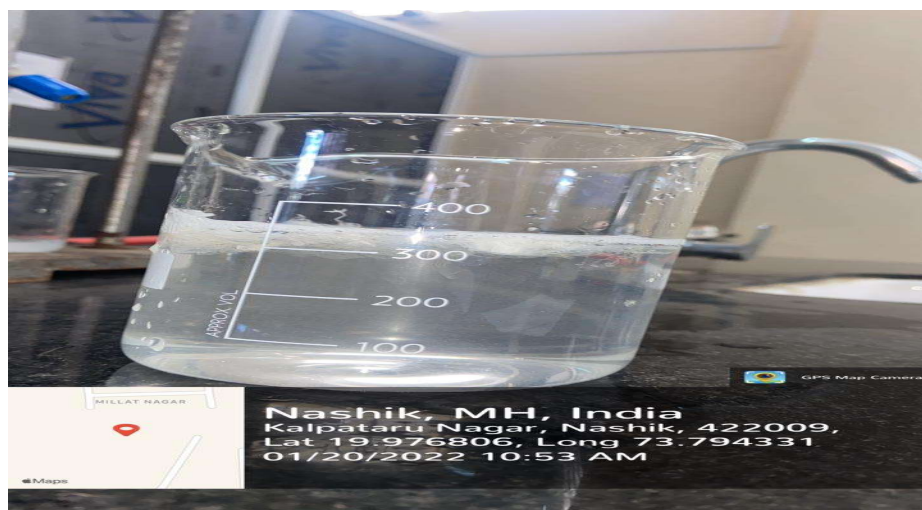


Fig 4.15

Chitosan + Acetic Acid

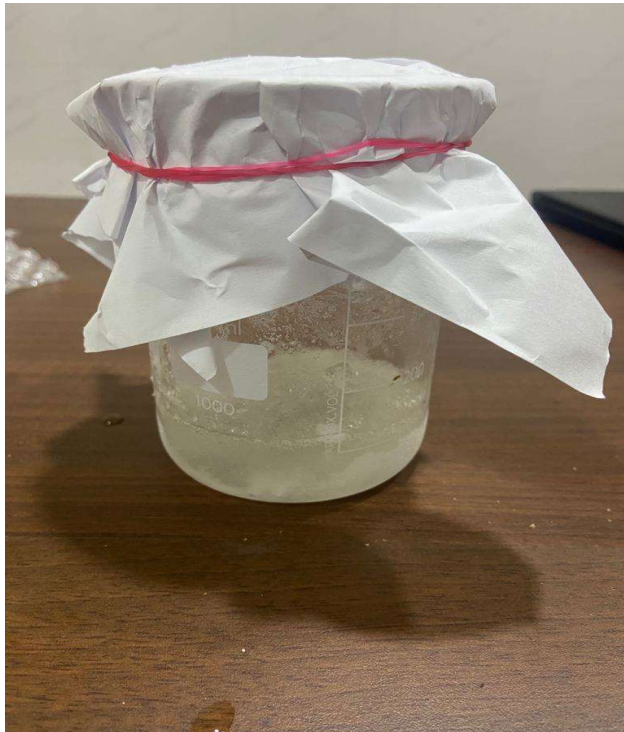


Fig 4.16

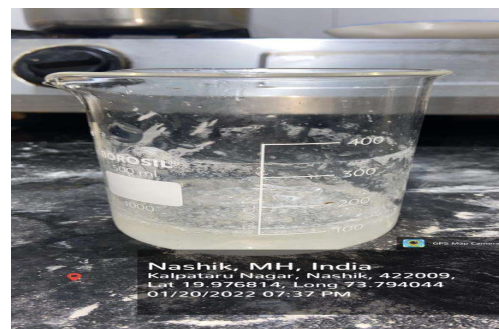


Fig 4.17



Fig 4.18

4.3 FTIR OF NANO CHITOSAN

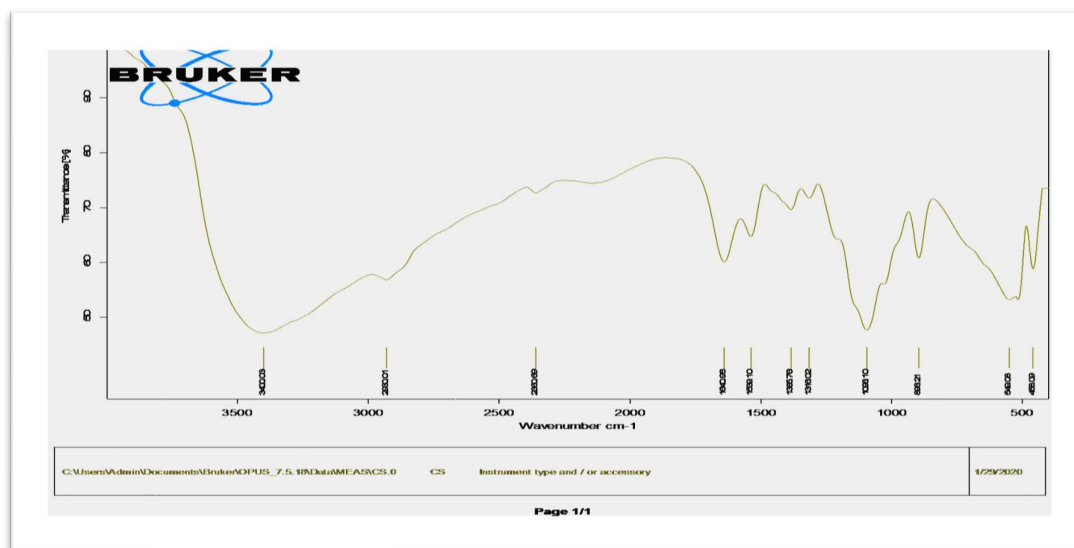


Fig 4.19

FTIR (Fourier transform infrared spectroscopy) is a technique for obtaining an infrared spectrum of a solid, liquid, or gas's absorption or emission. An FTIR spectrometer obtains high-resolution spectral data over a large spectral range at the same time. This gives it a big advantage over a dispersive spectrometer, which only measures intensity over a small range of wavelengths at a time. The term "Fourier transform infrared spectroscopy" comes from the fact that raw data must be converted into a spectrum using a Fourier transform (a mathematical technique). An FTIR spectrometer with an attenuated total reflectance (ATR) attachment is seen here.

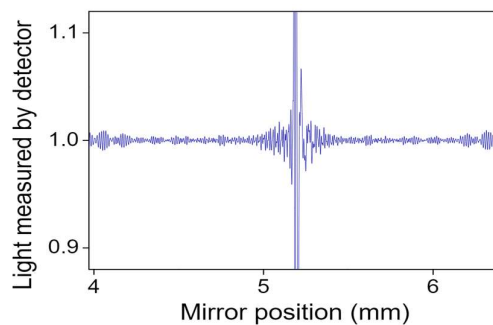


Fig 4.20

Introduction to the concept: Absorption spectroscopy techniques (FTIR, ultraviolet-visible ("UV-Vis") spectroscopy, and so on) are used to determine how much light a sample absorbs at different wavelengths. The simplest method, known as "dispersive spectroscopy," involves shining a monochromatic light beam at a sample, measuring how much of the light is absorbed, and repeating the process for each different wavelength. (Some UV-vis spectrometers, for example, operate in this manner.) The use of Fourier transform spectroscopy to derive the same information is less apparent. Rather than flashing a single wavelength of light at the sample, this technique shines a beam containing multiple frequencies of light at the same time and analyses how much of that beam is absorbed by the sample. The beam is then changed to contain a different frequency combination, yielding a second data point. This technique is performed several times in a short period of time. After that, a computer uses all of this information to infer absorption at each wavelength by working backwards. Starting with a broadband light source—one that contains the entire spectrum of wavelengths to be measured—the beam described above is created. The light shines through a Michelson interferometer, which is a set of mirrors with one of them being moved by a motor. Due to wave interference, the interferometer periodically blocks, transmits, blocks, transmits each wavelength of light in the beam as it advances. Different wavelengths are modulated at different rates, resulting in a different spectrum for each instant or mirror position in the interferometer. As previously stated, computer processing is required to convert raw data (light absorption for each mirror position) into the desired outcome (light absorption for each wavelength). The required processing turns out to be the Fourier transform, which is a standard procedure. The Fourier transform transforms one domain (in this case, the mirror's displacement in cm) into its inverse domain (wavenumbers in cm^{-1}). An "interferogram" is the term for the raw data.

Interferogram measurement and processing: The interferogram must be measured from zero path difference to a maximum length that is determined by the needed resolution. The scan can be on either side of zero in practise, producing a double-sided interferogram. Due to mechanical design constraints, the scan may only run to the maximum OPD on one side of zero for the highest resolution. A spectrum of the signal at a succession of distinct wavelengths is

the outcome of Fourier transformation. The separation of the data points in the interferogram limits the range of wavelengths that can be employed in the calculation. The shortest wavelength that can be recognised is the distance between these data points multiplied by two. For example, if a HeNe reference laser has one point per wavelength of 0.633 m (15800 cm⁻¹), the shortest wavelength is 1.266 m (7900 cm⁻¹). Any energy at shorter wavelengths is seen as originating from longer wavelengths due to aliasing, and hence must be minimised optically or electrically. [More information is required]. The maximum OPD determines the spectral resolution, or the separation between wavelengths that can be identified. The wavelengths employed in the Fourier transform are chosen in such a way that an exact number of wavelengths fit into the length of the interferogram from zero to maximum OPD, making their contributions orthogonal. As a result, a spectrum with identical frequency intervals separates the points. In the interferogram, adjacent wavelengths 1 and 2 will have n and $(n+1)$ cycles, respectively, for a maximum path difference of d . ν_1 and ν_2 are the corresponding frequencies:

$$d = n\lambda_1 \quad \text{and} \quad d = (n+1)\lambda_2$$

$$\lambda_1 = d/n \quad \text{and} \quad \lambda_2 = d/(n+1)$$

$$\nu_1 = 1/\lambda_1 \quad \text{and} \quad \nu_2 = 1/\lambda_2$$

$$\nu_1 = n/d \quad \text{and} \quad \nu_2 = (n+1)/d$$

$$\nu_2 - \nu_1 = 1/d$$

The inverse of the maximum OPD is the separation. A maximum OPD of 2 cm, for example, resulting in a separation of 0.5 cm⁻¹. The spectral resolution is defined as the independence of a value at one location from the values at nearby sites. By selecting different OPDs, most devices can be controlled at different resolutions. Spectrometers have been built with resolutions as high as 0.001 cm⁻¹, corresponding to a maximum OPD of 10 m. Instruments for routine analyses typically have a best resolution of around 0.5 cm⁻¹, while spectrometers have been built with resolutions as high as 0.001 cm⁻¹, corresponding to a maximum OPD of 10 m. The zero-path difference point in the interferogram must be found, which is usually done by assuming it is where

the highest signal occurs. In real-world spectrometers, the so-called center burst is not always symmetrical, hence a phase adjustment may be required. As the path difference grows, the interferogram signal decays, with the rate of decay being inversely proportional to the breadth of the spectral features. There will be undesirable oscillations or sidelobes associated with the features in the resulting spectrum if the OPD is not large enough to allow the interferogram signal to decay to an insignificant level. The interferogram is frequently multiplied by a function that approaches zero at the greatest OPD to reduce sidelobes. This so-called apodization lowers the amplitude of any sidelobes as well as the noise level, albeit at the cost of some resolution loss. The number of points in the interferogram must be a power of two in order to calculate quickly. To do this, a series of zeroes can be added to the measured interferogram. Although there is no enhancement in resolution, more zeroes can be added in a process called zero filling to improve the aesthetic of the finished spectrum. Interpolation following the Fourier transform, on the other hand, produces a similar effect.

Advantages: When compared to a scanning (dispersive) spectrometer, an FT spectrometer has three major advantages. Fellgett's advantage, or the multiplex. This is due to the fact that data from all wavelengths is captured at the same time. For observations constrained by a fixed detector noise contribution, it results in a greater signal-to-noise ratio for a given scan time (typically in the thermal infrared spectral region where a photodetector is limited by generation-recombination noise). This increase is equivalent to the square root of m for a spectrum with m resolution elements. It also provides for a faster scan time for a given resolution. Multiple scans are frequently averaged in practise, which increases the signal-to-noise ratio by the square root of the number of scans.

Jacquinot's advantage, or throughput. This is due to the fact that the monochromator in a dispersive instrument contains entrance and exit slits that limit the amount of light that enters through it. Only the diameter of the collimated beam originating from the source determines the interferometer throughput. FTIR spectrometers require an aperture to limit the convergence of the collimated beam in the interferometer, even though no slits are required.

Because the route difference varies, convergent rays are modified at different frequencies. A Jacquinot stop is the name for such an aperture. This circular aperture allows lighter through than a slit for a given resolution and wavelength, resulting in a greater signal-to-noise ratio. Connes' advantage or wavelength accuracy A laser beam of known wavelength passes through the interferometer, calibrating the wavelength scale. This is far more stable and accurate than dispersive instruments, which rely on the mechanical movement of diffraction gratings to determine scale. In practise, the accuracy is restricted by the divergence of the beam in the interferometer which depends on the resolution.

Another modest benefit is reduced sensitivity to stray light, which is light of one wavelength that appears at a different wavelength in the spectrum. This is caused by flaws in the diffraction gratings and unintended reflections in dispersive devices. The apparent wavelength in FT instruments is determined by the modulation frequency in the interferometer, therefore there is no direct equivalent. The length dimension is where the interferogram belongs. The interferogram's Fourier transform (FT) inverts the dimension, therefore it belongs in the reciprocal length dimension ($[L^{-1}]$), which is the dimension of wavenumber. The reciprocal of the greatest retardation in cm is the spectral resolution in cm^{-1} . When the maximum retardation is 0.25 cm, a 4 cm^{-1} resolution is attained; this is typical of cheaper FTIR equipment. By increasing the maximum retardation, much greater resolution can be produced. This is difficult because the travelling mirror must follow a near-perfect straight path. The use of corner-cube mirrors in place of the flat mirrors is helpful, as an outgoing ray from a corner-cube mirror is parallel to the incoming ray, regardless of the orientation of the mirror about axes perpendicular to the axis of the light beam. In 1966 Connes measured the temperature of the atmosphere of Venus by recording the vibration-rotation spectrum of Venusian CO_2 at 0.1 cm^{-1} resolution.[6] Michelson himself attempted to resolve the hydrogen H α emission band in the spectrum of a hydrogen atom into its two components by using his interferometer. A spectrometer with a resolution of 0.001 cm^{-1} is currently commercially available. Because the monochromator in a dispersive instrument with the same resolution would have very narrow

entrance and exit holes, the throughput advantage is crucial for high-resolution FTIR.

4.4 SEM (NANO- CHITOSAN)

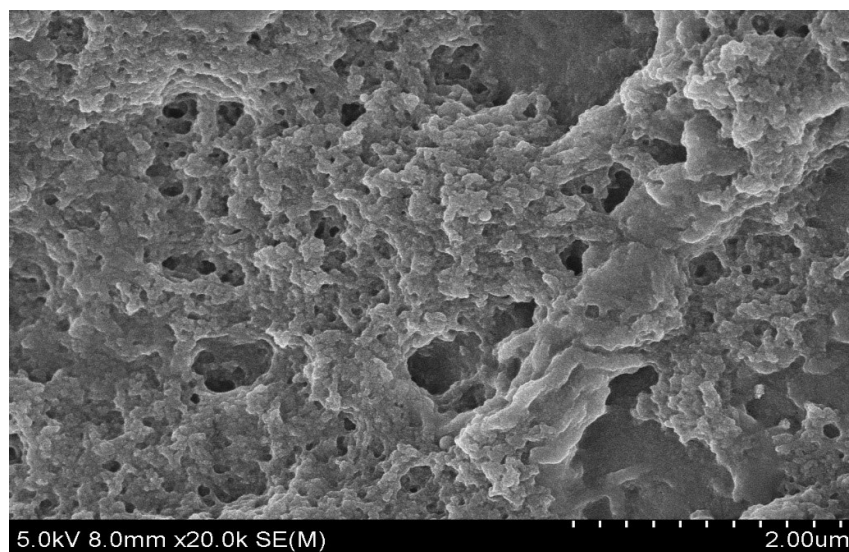
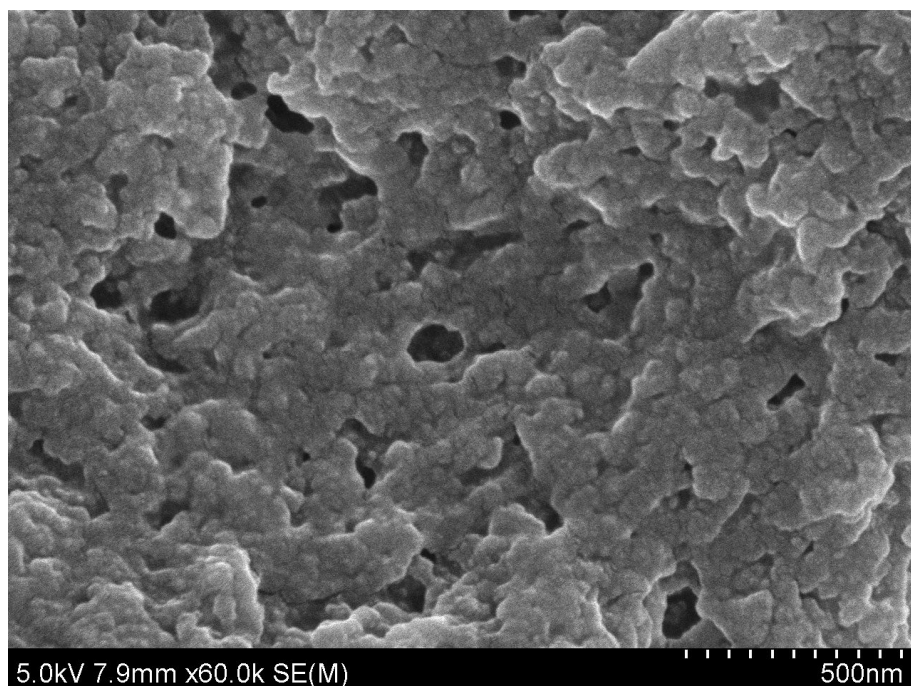


Fig 4.21

A scanning electron microscope (SEM) is a type of electron microscope that uses a concentrated beam of electrons to scan the surface of a sample to obtain images. When electrons interact with atoms in a sample, they produce a variety of signals that carry information about the sample's surface topography and composition. A raster scan pattern is used to scan the electron beam, and the position of the beam is combined with the intensity of the detected signal to create an image. Secondary electrons generated by atoms stimulated by the electron beam are detected using a secondary electron detector in the most common SEM mode (Everhart-Thornley detector). The number of secondary electrons that can be detected, and thus the signal intensity, is influenced by specimen topography, among other factors. Some SEMs can achieve resolutions of 1 nm or more. Specimens are viewed in a high vacuum in a typical SEM, in a low vacuum or moist environment in a variable pressure or environmental SEM, and at a wide range of cryogenic or higher temperatures in specialised devices.

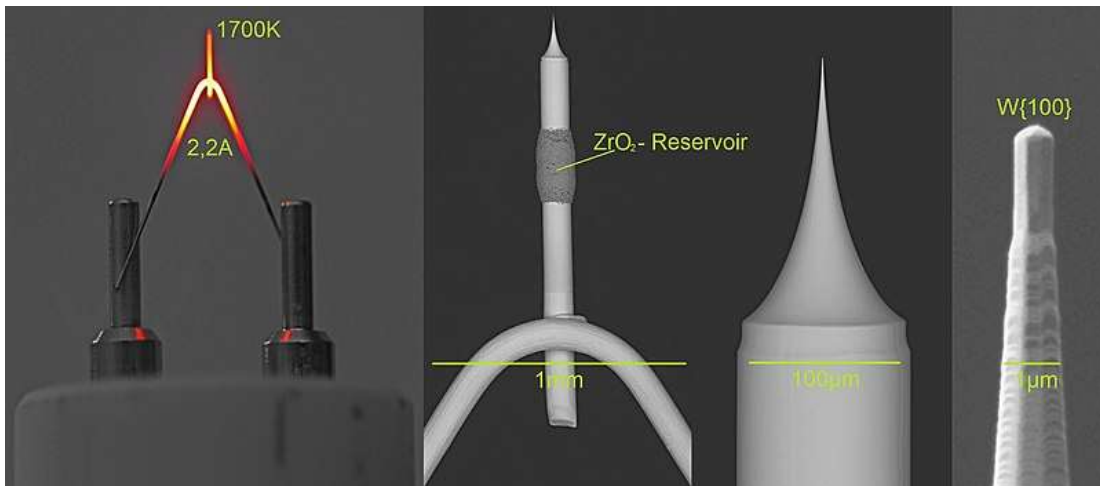


Fig 4.22

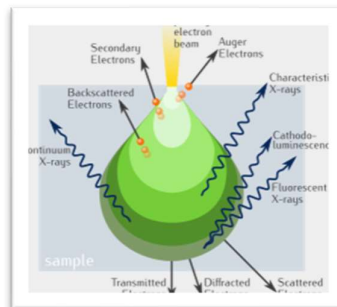


Fig 4.23

The interactions of the electron beam with atoms at various depths within the sample provide the signals needed by a SEM to produce an image. Secondary electrons (SE), reflected or back-scattered electrons (BSE), distinctive X-rays and light (cathodoluminescence) (CL), absorbed current (specimen current), and transmitted electrons are among the signals produced. Although secondary electron detectors are commonplace in all SEMs, it is uncommon for a single machine to incorporate detectors for all other conceivable signals.

The low energies of secondary electrons, on the order of 50 eV, limit their mean free path in solid matter. As a result, SEs can only escape from the top few nanometres of a sample's surface. Because the signal from secondary electrons is extremely localised at the site of impact of the original electron beam, pictures of the sample surface with a resolution of less than 1 nm can be collected. Back-scattered electrons (BSE) are beam electrons that are reflected by elastic scattering from the material. Because BSEs have a significantly larger energy than SEs, they arise from deeper regions inside the object, resulting in lower resolution than SE pictures. However, because the intensity of the BSE signal is highly connected to the atomic number (Z) of the material, BSE are frequently utilised in analytical SEM, along with spectra derived from the characteristic X-rays. BSE pictures can reveal the distribution of distinct elements in the sample, but not their identities. BSE imaging can scan colloidal gold immuno-labels of 5 or 10 nm diameter in samples mostly made of light elements, such as biological specimens, which would normally be difficult or impossible to detect in secondary electron pictures. When the electron beam removes an inner shell electron from the sample, a higher-energy electron fills the shell and releases energy, characteristic X-rays are generated. Energy-dispersive X-ray spectroscopy or Wavelength-dispersive X-ray spectroscopy can measure the energy or wavelength of these unique X-rays, which can be used to identify and measure the number of elements in the sample and map their distribution.

SEM micrographs have a significant depth of field due to the relatively narrow electron beam, giving them a distinctive three-dimensional look important for analysing a sample's surface structure. The micrograph of pollen displayed above exemplifies this. Magnifications range from about 10 times (about equivalent to a

powerful hand lens) to more than 500,000 times, or about 250 times the magnification limit of the greatest light microscopes.



Fig 4.24

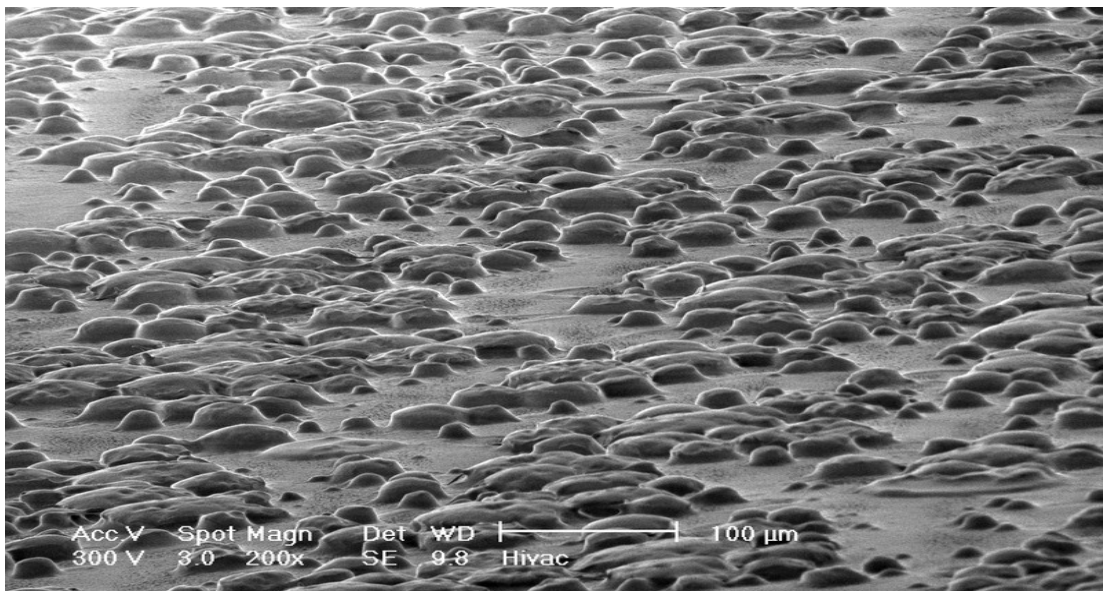


Fig 4.25

SEM samples must be small enough to fit on the specimen stage, and they may require specific preparation to improve their electrical conductivity and stabilise them so that they can endure the high vacuum and high-energy electron beam. A conductive adhesive is used to adhere samples rigidly to a specimen holder or stub. Manufacturers make machines that can inspect any portion of a 300 mm semiconductor wafer, and SEM is widely used for defect analysis of semiconductor wafers. Many instruments feature chambers that can tilt a large object to 45 degrees and rotate it continuously 360 degrees.

When scanned by an electron beam, nonconductive objects gather charge, resulting in scanning defects and other picture distortions, especially in secondary electron imaging mode. Specimens must be electrically conductive, at least at the surface, and electrically grounded to avoid electrostatic charge accumulation in the SEM for conventional imaging. Except for cleaning and conductively mounting a specimen stub, metal objects require little specific preparation for SEM. Non-conducting materials are often coated with an ultrathin layer of electrically conducting material, which is deposited on the sample using either low-vacuum sputtering or high-vacuum evaporation. Gold, gold/palladium alloy, platinum, iridium, tungsten, chromium, osmium, and graphite are some of the conductive materials now used for specimen coating. For samples with a low atomic number, coating with heavy metals may improve the signal/noise ratio (Z). The improvement is due to increased secondary electron emission in high-Z materials. For some biological samples, increasing the bulk conductivity of the material by impregnation with osmium utilising versions of the OTO staining procedure is an alternative to coating (O-osmium tetroxide, T-thiocarbohydrazide, O-osmium).

An environmental SEM (ESEM) or a low-voltage mode of SEM operation can be used to photograph nonconducting materials without coating. The specimen is placed in a relatively high-pressure chamber in ESEM apparatus, and the electron optical column is differentially pumped to keep the vacuum at the electron gun low enough[clarification needed]. In the ESEM, the high-pressure zone around the sample neutralises charge and amplifies the secondary electron signal. [requires citation] Low-voltage SEM is usually performed in a field emission guns (FEG) device, which can provide high primary electron brightness and small spot sizes even at low accelerating potentials. Operating settings must be adjusted so that

the entering beam current equals the sum of outgoing secondary and backscattered electron currents, which is most typically met at accelerating voltages of 0.3–4 kV. [needs citation] When imaging in backscattered electrons or doing quantitative X-rays microanalysis, embedding in a resin and polishing to a mirror-like sheen can be used for both biological and materials specimens. The primary preparation processes are not necessary in the environmental SEM described below, but fixation can be beneficial for some biological materials. Because the specimen chamber is at a high vacuum, a specimen for SEM must be entirely dry. Hard, dry materials like wood, bone, feathers, dried insects, or shells (even egg shells) can be inspected without chemical fixation, but living cells and tissues, as well as complete, soft-bodied creatures, require chemical fixation to maintain and stabilise their structure. Incubation in a solution of a buffered chemical fixative, such as glutaraldehyde, occasionally in combination with formaldehyde and other fixatives, and optional postfixation with osmium tetroxide are the most common methods of fixation. After that, the fixed tissue is dehydrated. Because air-drying causes the cells to collapse and shrink, this is usually accomplished by replacing the water in the cells with organic solvents like ethanol or acetone, and then replacing these solvents with a transitional fluid like liquid carbon dioxide via critical point drying. While in a supercritical condition, the carbon dioxide is finally eliminated, leaving no gas–liquid contact within the sample during drying. Before observation in the microscope, the dry specimen is commonly mounted on a specimen stub with an adhesive such as epoxy resin or electrically conductive double-sided adhesive tape, and sputter-coated with gold or gold/palladium alloy. If information about the organism's internal ultrastructure has to be exposed for imaging, samples can be sectioned (using a microtome). Freeze-fracturing, also known as freeze-etching or freeze-and-break, is a technique for analysing lipid membranes and their associated proteins in a "face-on" manner. The proteins contained in the lipid bilayer are revealed by the preparation technique.

4.5 PREPARATION OF CELLULOSE FROM BANANA FIBRE

- Banana fibre (30 g) is combined with 2% NaOH at a ratio of 1:10 in an autoclave and steam exploded for 1 hour at a pressure of 20 lb.
- Steamed alkali-treated fibres were then bleached with sodium hypochlorite,

then treated with oxalic acid and physically agitated to extract cellulose.



Fig 4.26



Fig 4.27



Fig 4.28



Fig 4.29



Fig 4.30



Fig 4.31

4.6 PREPARATION OF NANOCOMPOSITES BEADS

- Nano chitosan, sodium alginate, and microcrystalline cellulose derived from banana stem fibre were mixed in a specific ratio to make ternary biopolymeric beads.
- An aqueous 2.5 percent nano chitosan solution;
- An aqueous 10 wt percent alginate solution; and
- An aqueous 1.25 wt percent microcrystalline cellulose solution
- For around 30 minutes, the above three solutions were mixed thoroughly at 500 rpm.
- NCS beads are formed when a homogeneous blended substance is injected as minute droplets into 0.2 M calcium chloride using a syringe. It was then immersed for a few hours in 0.2 M CaCl_2 , rinsed thoroughly with distilled water to remove any remaining CaCl_2 , and dried.

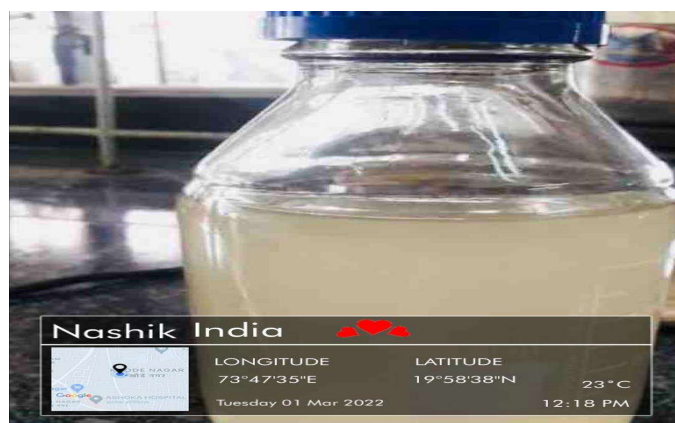


Fig 4.32

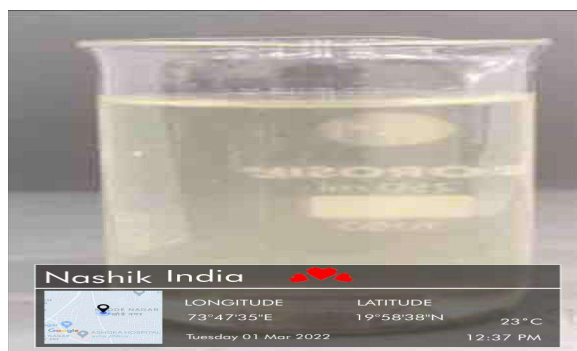


Fig 4.33



Fig 4.34



Fig 4.35

4.7 SEM OF BEADS (BEFORE ADSORPTION)

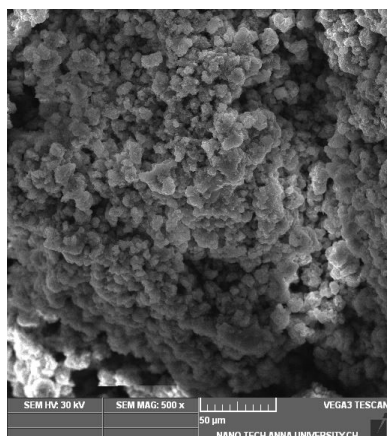


Fig 4.36

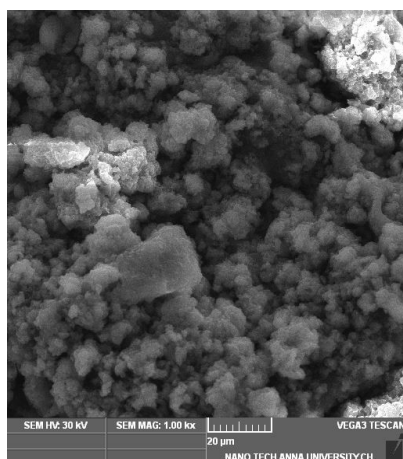


Fig 4.37

4.8 SEM OF NANO COMPOSITE BEADS (AFTER ADSORPTION)

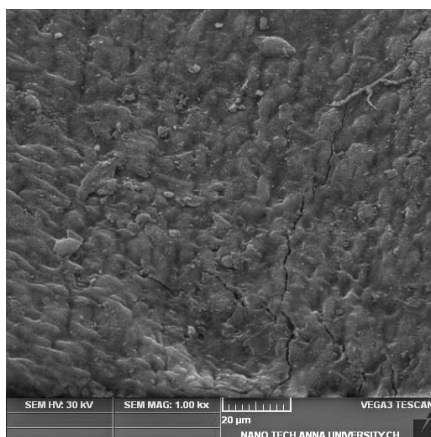


Fig 4.38

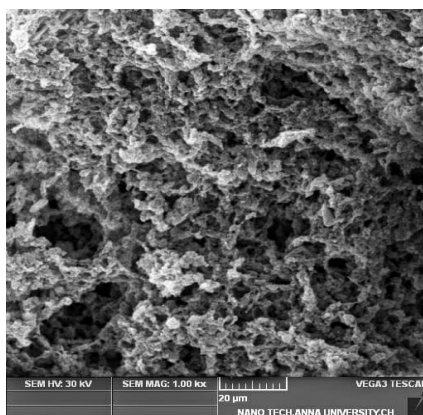


Fig 4.39

4.9 FTIR OF NANOCHITOSAN AND ALGINATE BEADS (BEFORE ADSORPTION)

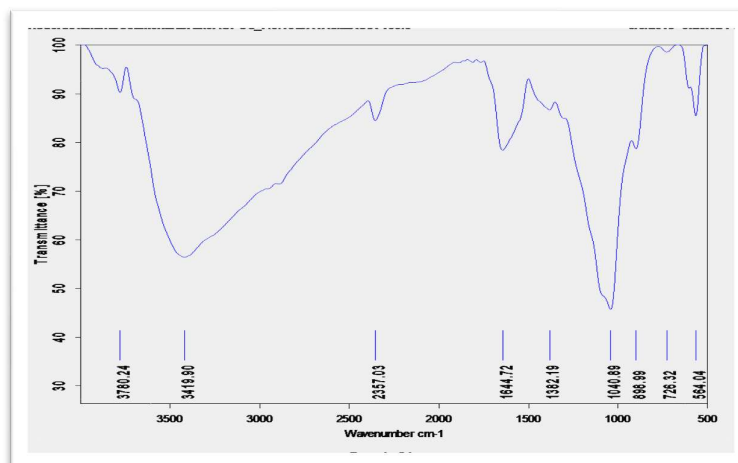


Fig 4.40

Due to overlapping of the OH and N-H stretching vibrations of functional groups engaged in hydrogen bond, the FTIR spectrum of chitosan exhibits a broad and strong bond at 3419cm⁻¹.

The symmetric and asymmetric -CH₂vibration linked to the pyranose ring causes the band at 2357cm⁻¹.

C=O stretching in the amide group band has a peak at 1644cm⁻¹.

The absorption band of P=O was discovered to be in the range of 1382cm⁻¹. The skeletal vibrations involving CO stretching, which are characteristic of the chitosan saccharide structure, peak around 1040 cm⁻¹.

4.10 FTIR OF NANOCHITOSAN AND ALGINATE BEADS (AFTER ADSORPTION)

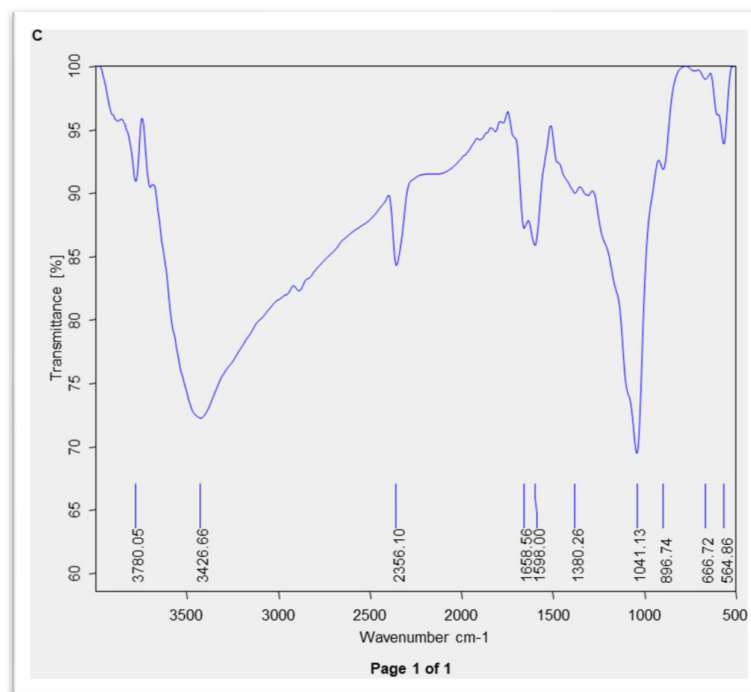


Fig 4.41

The peak of adsorption (3419cm⁻¹) shifts to 3426cm⁻¹ after adsorption with chitosan, and the peak for asymmetric deformation of the –NH vibration in the NH₃⁺ ion is 1658 & 1598 cm⁻¹.

Peaks are shifted and/or broadened in the FTIR spectra of composite nano particles before and after adsorption, showing that the functional groups on the composite nano particles are engaged in interaction with the RB9.

CHAPTER – 5

RESULTS AND DISCUSSION, PERFORMANCE ANALYSIS

The following Adsorption studies are the result of the project: -

1. Effect of dosage. (0.5-2g/L)
2. Effect of concentration. (50-250ppm)
3. Effect of time. (5- 30 min)
4. Effect of pH (2-10)
5. Effect of temperature. (30-60⁰c)

5.1 EFFECT OF DOSAGE. (0.5-2g/L)

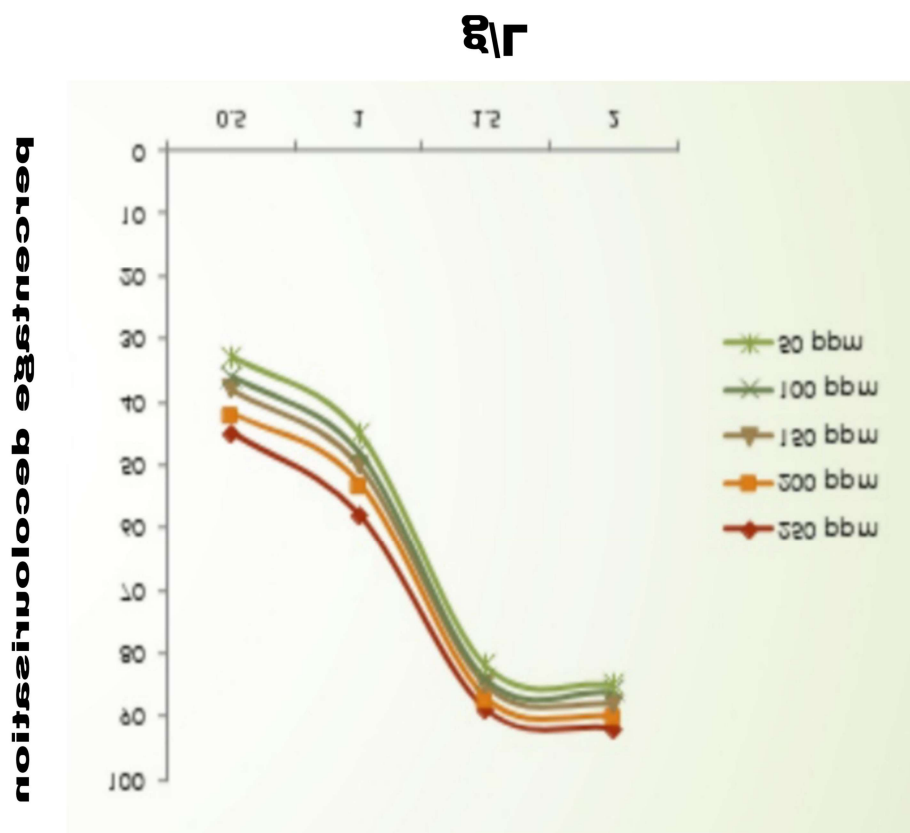


Fig 5.1

- Various dosages of our adsorbent (0.5-2.5g/L) were applied to varied amounts of dye solutions.
- It is determined that the optimal dosage was found to be 1.5g/L.

5.2 EFFECT OF CONCENTRATION

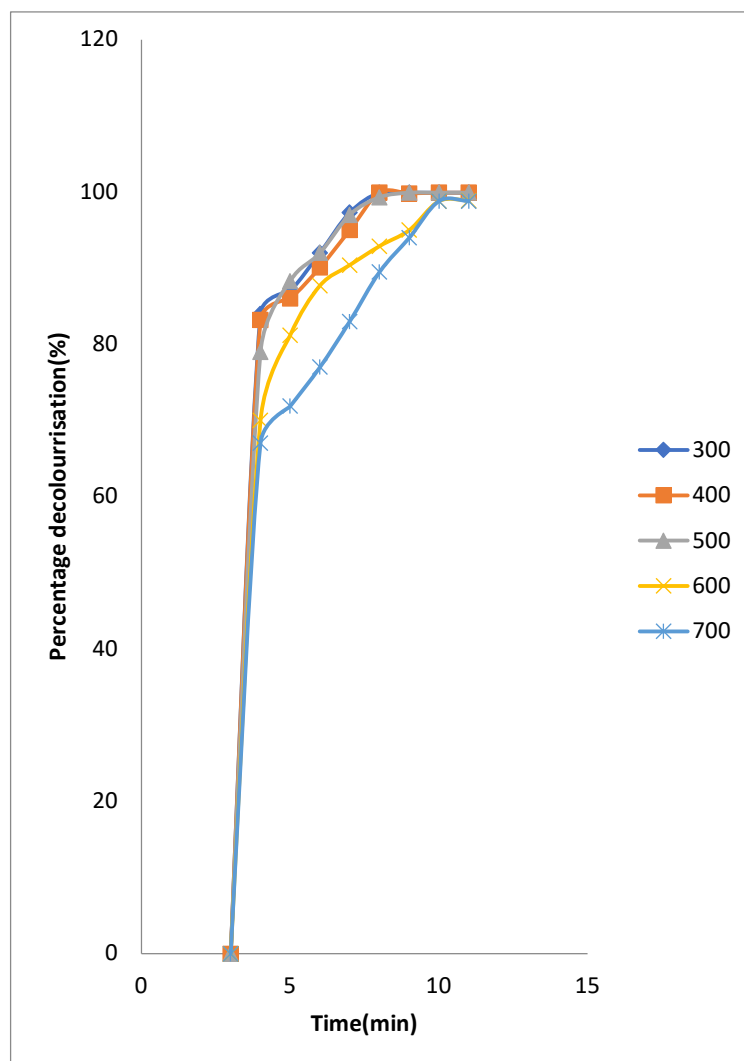


Fig 5.2

- The dye solutions were generated at various concentrations (ppm) and the adsorbent was added to determine the optimal ppm.
- The graph shows that maximum decolorization is achieved at 50, 100, and 150 ppm.
- As the concentration for the needed dosage increases, the percent decolorization tends to decrease.
- For a dosage of 1.5g/L, the best concentration was found to be 150ppm.

5.3 EFFECT OF TIME

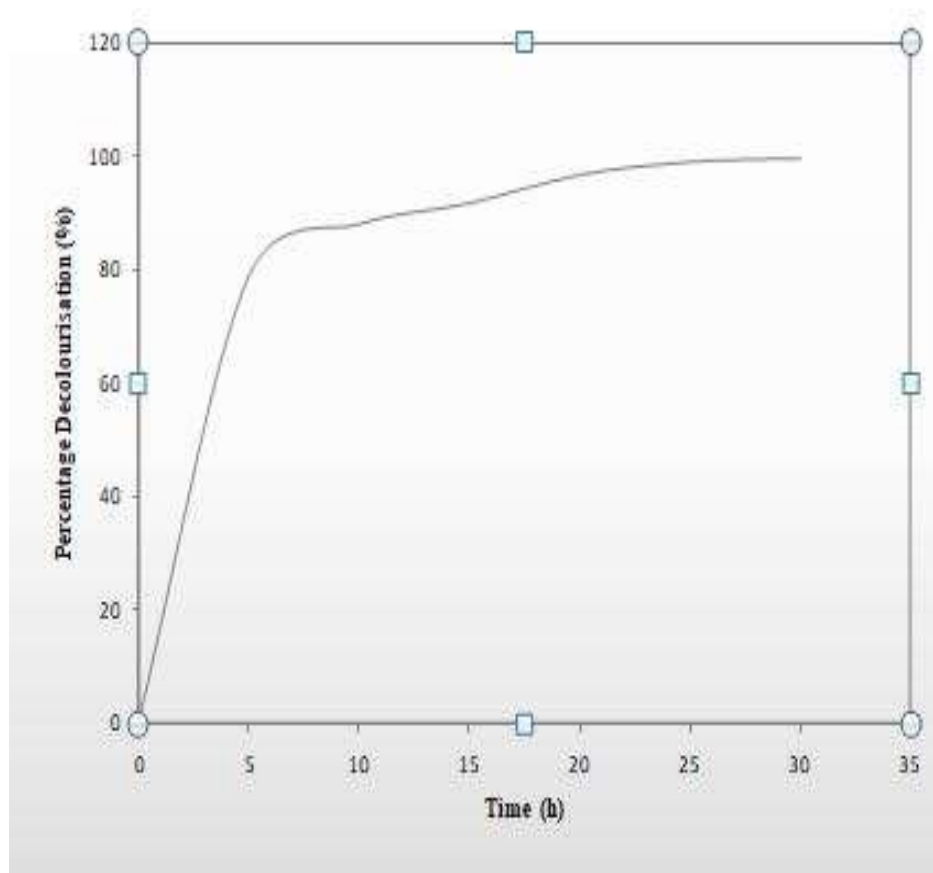


Fig 5.3

- The optimal dosage was applied after preparing several concentrations (ppm) of solution.
- The UV value of the solutions was recorded over a period of time.
- For most concentrations, 20 minutes was shown to be the optimal time.

5.4 EFFECT OF PH

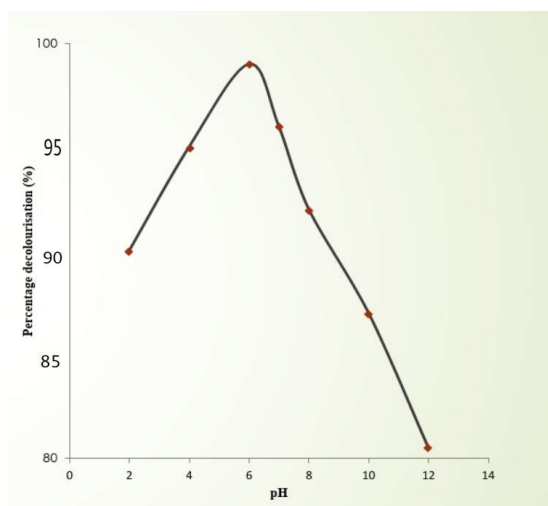


Fig 5.4

- The fixed concentration is tested at several pH levels (ranging from 1 to 12).
- The adsorbent was added and mixed for the best amount of time before recording the UV value.
- The graph shows that percent decolorization is greatest at pH 6.

5.5 EFFECT OF TEMPERATURE

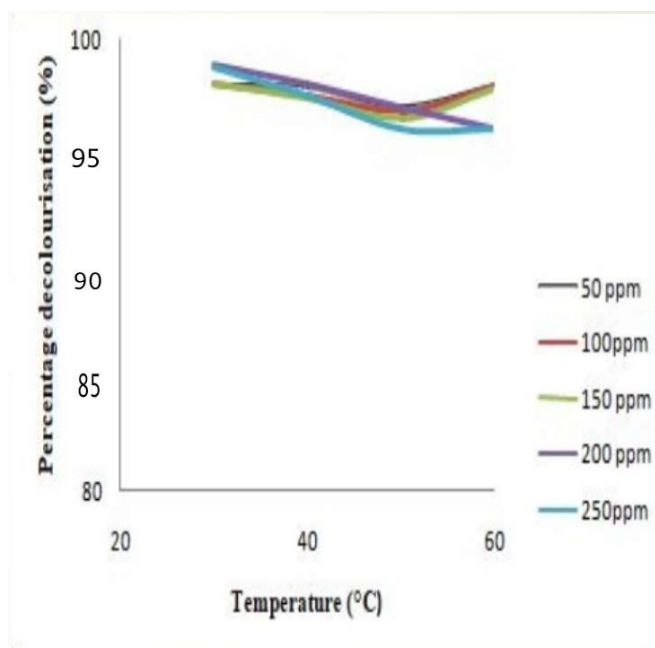


Fig 5.5

- For the required dosage, concentration, time and pH various temperature was studied.
- It is found that, as temperature increases the % decolorization decreases.
- The best temperature obtained is 20°-30°c.

5.6 ADSORPTION ISOTHERMS

5.6.1 LANGMUIR

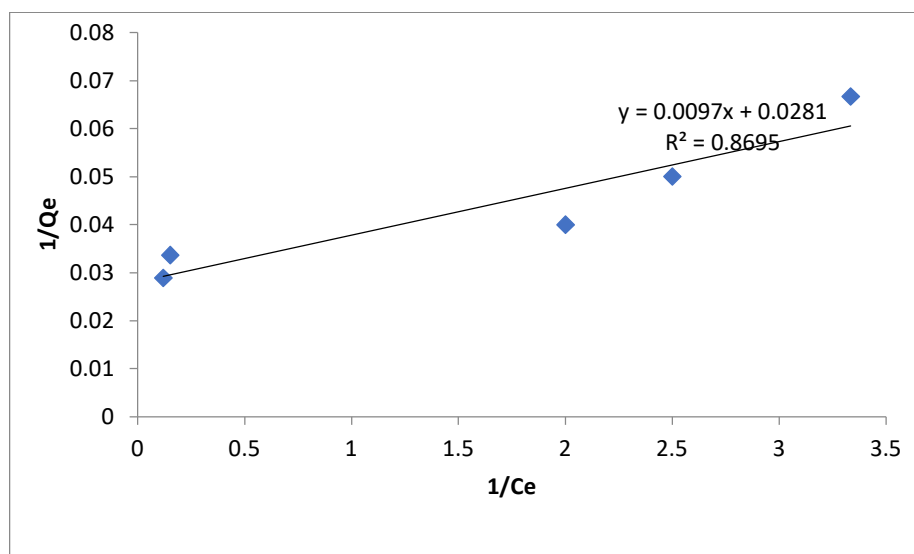


Fig 5.6

- Monolayer adsorption is represented by the Langmuir isotherm.
- Where C_e denotes the equilibrium concentration, K_l is the constant associated to binding site affinity, and is the maximum quantity of material ion per unit weight of adsorbent required to create a full monolayer on the surface.
- Until it is desorbed, the adsorbed molecule stays at the adsorption site.
- Only a monolayer form at maximum adsorption: adsorbate molecules do not deposit on other, already adsorbed, adsorbate molecules, but only on the free surface of the adsorbent.

5.6.2 FREUNDLICH

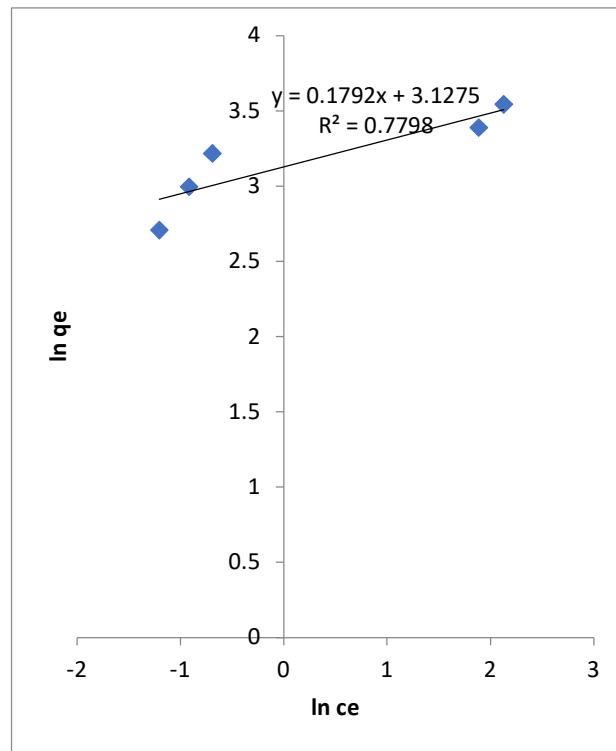


Fig 5.7

- Multilayer adsorption is represented by the Freundlich model.
- Different binding sites are considered in this isotherm.
- Adsorption capacity is represented by K_f , and adsorption intensity is represented by n . On both sides, use the logarithm.
- In this study, the Langmuir model outperforms the Freundlich model in terms of adsorption capacity.

5.6.3 TEMKIN

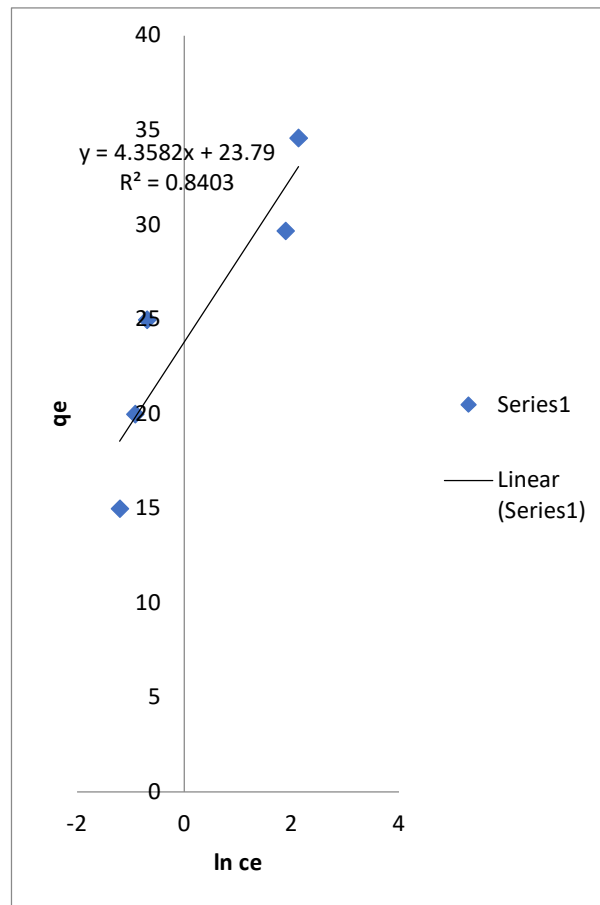


Fig 5.8

$$q_e = B \ln C_e + B \ln A$$

$$A = 234.8451 \quad \ln A = 5.48926$$

- This isotherm has a component that specifically takes adsorbent–adsorbate interactions into consideration.
- The constants were obtained from the slope and intercept of the plot of the quantity sorbed q_e against $\ln C_e$.

5.7 COMPARISON

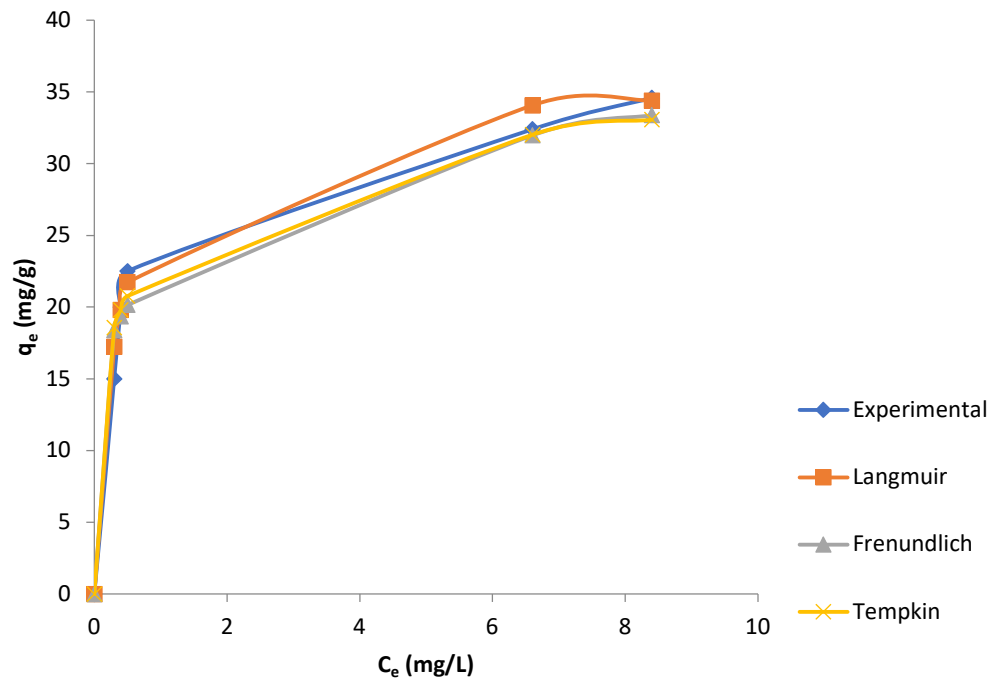


Fig 5.9

- The Freundlich isotherm was discovered to be the most effective for adsorption investigations.
- This isotherm allowed the most dye to be absorbed.

CHAPTER – 6

CONCLUSION

The properties of chitin and chitosan are presented in this review. Following that, there was a debate about the solubilization required to transform polysaccharides into new materials. Their fibre and film-forming properties are recognized based on the production of H-bonds networks in the solid state, which could be valuable for new applications.

The most essential applications, however, stem from their hydrophilic and antibacterial properties, which are particularly desirable for the development of novel biomaterials.

Chitosan, unlike chitin, is soluble in acidic conditions, making it useful for improving processing processes. In fact, chitosan can be made into a fibre, film, sponge, bead, gel, or solution with ease. Its cationic charge also allows for the formation of electrostatic complexes and/or multilayer structures.

The availability of free -NH₂ groups along the chitin and chitosan chains allows for particular alterations (on the C-2 position of the D-glucosamine unit) to be carried out under relatively benign conditions (even in aqueous conditions with chitosan). Chitin and chitosan can also be combined with synthetic or natural polymers (proteins, DNA, alginate, hyaluronan, etc.).

The adsorbent's properties were investigated, and the removal of reactive dyes was discussed and analyzed.

After reviewing the adsorption studies, it was discovered that 1.5g/L is the optimal dosage for adsorption.

At 150 ppm and 1.5 g/L, the optimum concentration was discovered.

For most concentrations, 20 minutes was shown to be the optimal time.

At pH 6, the highest percentage of decolorization was discovered.

Temperatures of 20-30° C resulted in the highest percentage of decolorization.

The Freundlich isotherm was discovered to be the optimum adsorption isotherm.

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