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NATURAL POLYMER–BASED WASTEWATER TREATMENT USING PLANTAGO MAJOR SEED MUCILAGE: CHARACTERIZATION AND APPLICATION

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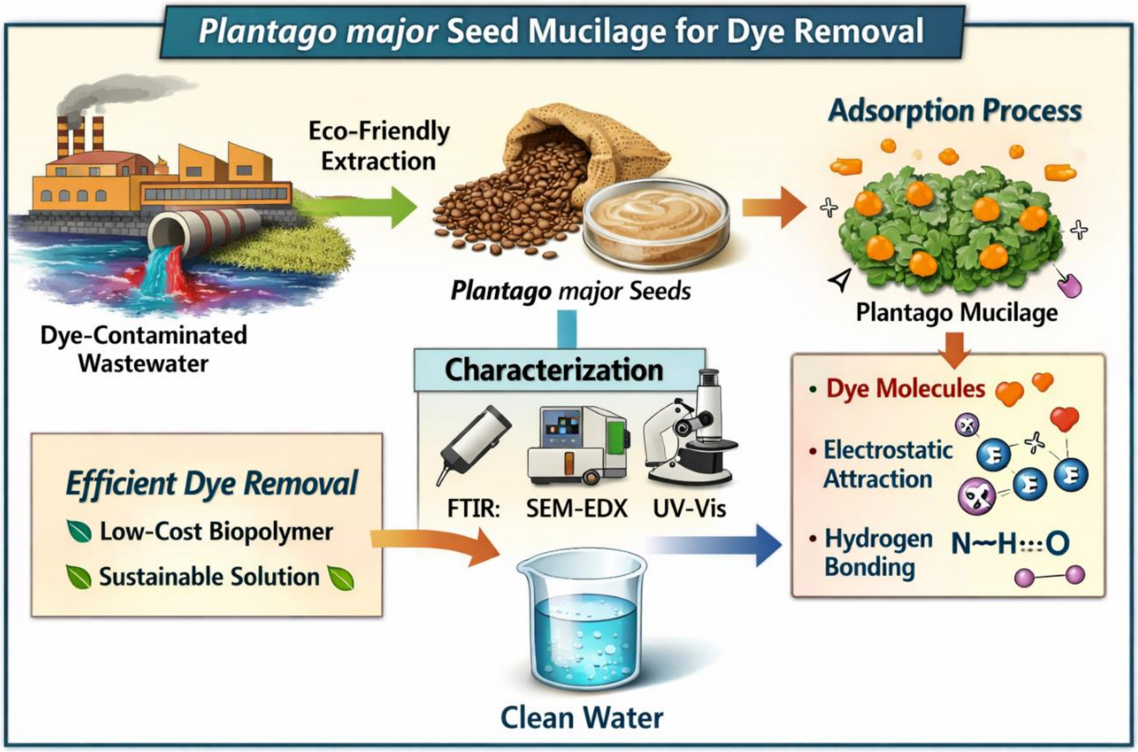
Abstract

The growing release of dye bearing effluents by textile industries and other related industries is presenting a significant danger to aquatic life and human health thus the need being on creating materials of sustainable and low cost in terms of treatment. This paper examined mucilage that had been extracted on the seeds of Plantago major as a natural polymer-based adsorbent to treat wastewater. Mucilage was prepared by following a green extraction path of aqueous and thoroughly characterized by FTIR, XRD, SEM, EDX, and UV- visible spectroscopy. The structural and morphological measurements showed that the biopolymer has large amount of hydroxyl, carboxyl, and ether functional groups, and has porous, layered and heterogeneous surface that is laden with organic and mineral compounds. Experiments on batch adsorption showed that there was an effective removal of dye in aqueous solution and that the intensity of absorbance reduced very fast with time, which indicates the high adsorption affinity. It is assumed that the adsorption mechanism is largely due to the electrostatic attraction and hydrogen bonding and surface complexation of the dye molecules with the functional groups on the mucilage matrix. The ion-exchange interactions also increase adsorption with the naturally occurring mineral ions. On the whole, the findings emphasize that Plantago major seed mucilage can serve as a sustainable, biodegradable, and cost-effective mucilage adsorbent that has a high potential of application in wastewater treatment technologies based on green technology.



INTRODUCTION

Population growth and anthropogenic activity are usually associated with the spread of pollutants in the ecosystem. This is a highly controversial topic in the global context because water resource pollution has long-term or even fatal effects on the living organisms[1-4]. More than third of renewable freshwater resources in the world are industrial, household, and agricultural use[5] [6] [7], and most of these activities contaminate water with numerous geogenic and synthetic chemicals, dyes, pesticides, fertilizers, radionuclides, and heavy metals[8, 9] [10]. Consequently, it is not astonishing that the water poisoning caused by numerous human actions has caused panic among people in terms of their health issues on a worldwide level[11] [12]. The textile industry followed by printing industries, then paper, paint, and leather production companies, were the first causes of the water pollution issue[13] [14].

More than third of renewable freshwater resources in the world are industrial, household, and agricultural use, and most of these activities contaminate water with numerous geogenic and synthetic chemicals, dyes, pesticides, fertilizers, radionuclides, and heavy metals[8] [15]. Approximately 15 per cent of overall world manufacture of dye is wasted throughout the dyeing process and is discharged by the form of liquid effluents. Textile finishing, dye manufacturing, pulp and paper industries are some of the industries that have one of the most challenging demands, which is the removal of color on such wastes[16] [17] [18] [19]. Some dyes, including methylene blue, are cationic dyes and are produced and used in dye, paint production and dyeing wool, among others[20] [21]. The dye molecule is typically made of the chromophores along with the auxochromes[22] [23], the former of which produces the color, and the latter enhance the solubility of dye as well as boosting their affinity to the fibers. Dyes have numerous structural types including acidic and basic, azo,anthroquinone based and metal complex dyes[24] [25]. Commercially available dyes are more than 100,000. Over the past decades, industries that have a large percentage of contaminant discharge have been relocated to the developing country. The largest number of dyes is produced nowadays in the developing countries, e.g., China, India, Thailand, Turkey, etc. Most of these emerging economies lack stringent laws or the discharge requirement is not fully implemented and this aggravates the dye contamination. And the developing countries are still struggling to do away with the environmental pollution in the textile and related industries because of the cost and technical difficulties[26] [27].

A number of methods have been established to remove dyes in wastewater and they include, adsorption, photo-oxidation, chemical oxidation, advanced oxidation processes (AOPs), biodegradation, filtration, coagulation among others[28]. However, these most popular methods also possess certain weaknesses and drawbacks. To provide an example, the adsorption is supposed to be a globally renowned method, but the conventional adsorbents will always portray the limitations of low adsorption capacity and adsorption rates, and the expensive regeneration and contamination of the column; therefore, the new adsorbents are still required[29] [30].

Seed mucilage is a natural polysaccharide-based biopolymer, which has received significant interest due to its strong water-absorbing capacity, non-toxicity, and a great number of functional groups which may react with contaminants. One of the promising plant-derived mucilage is Plantago major seed mucilage (PMSM) wherein availability is widespread, costs very low, and possesses the preferred physicochemical properties[31] [32]. The presence of high proportion of hydroxyl, carboxyl, ether functional groups permits the extraction, characterization and utilization of Plantago major seed mucilage as a natural polymer during wastewater treatment reactions via hydrogen bonding mechanism, electrostatic attraction and chelation[33] [34]. The work is aimed to evaluate its practical and electrolytic elimination and to join the process of creation of the sustainable and environmentally neutral alternatives to the traditional wastewater treatment materials. Hydrogen bonding and electrostatic interaction are the major mechanisms used to remove methylene blue in PMSM[35]. In aqueous solution, the carboxyl groups of the mucilage deprotonate, especially at the neutral to alkaline pH and the polymer matrix acquires a negative charge. This is because the strongly attractive positively charged methylene blue molecules are attracted to this negatively charged surface giving a good adsorption[36]. Additional enhancement of the dye uptake is hydrogen bonding between the nitrogen functional groups of methylene blue and the mucilage -OH functional groups. As such, an environmentally safe and efficient alternative to the removal of methylene blue in wastewater is the use of Plantago major seed mucilage, which involves the use of hydrogen bonding, polymer bridging reactions and electrostatic attraction. Not only does its use offer a cost-effective solution to dye polluted effluents, but also helps the broader goal of developing environmentally friendly materials with the aim of restoring the environment[37] [35].

Experimentation

Materials and reagent:

Plantago major seeds were chosen for their high purity after being found locally. Sigma-Aldrich provided analytical-grade methyl orange (MO) dye. All of the trials were conducted using distilled water. For pH correction, analytical reagent-grade sodium hydroxide (NaOH) and hydrochloric acid (HCl) were used. Every chemical was utilized just as supplied, requiring no additional purification. To reduce the possibility of contamination that could affect adsorption effectiveness, solutions were made with distilled water.

1. Preparation of P. major Seed Mucilage

To guarantee the highest yield and purity, the synthesis of P. major seed mucilage required a number of stages. The synthetic method is explained in detail below.

Seed Selection and Cleaning
To maintain consistency, seeds required to be the same size and maturity level. The seeds required to be thoroughly cleaned with distilled water to remove any surface contaminants, pesticides, or bacteria that might alter the mucilage's structure or prevent it from absorbing, as well as any foreign objects, stones, dust, or broken seeds.

Soaking Process
After being cleaned, the seeds were weighed and immersed in distilled water. A 1:4 seed to water ratio that is, 100g of seeds in 480mL of distilled water in a 500mL beaker—was employed. To ensure complete hydration, the soaking was done for 12 hours at room temperature (25 ± 2 °C). This process causes the polysaccharide-rich epidermal layer to expand, softening the seed coat and facilitating the release of mucilage.

Mucilage Extraction
To prevent the seeds from deteriorating, they were placed in a jacketed beaker that was connected to a water bath that was heated to 40 °C. For two hours, the mixture was steadily stirred at 200 rpm using a magnetic stirrer. Without severely harming the seed cells, a gentle stir releases the mucilage, which could contribute unnecessary proteins or lipids to the extract.

Separation of Mucilage
To remove the large chunks and seed husks, the thick, sticky sludge was poured through muslin fabric. To remove any leftover particles, the liquid was spun for 15 minutes at 5000 rpm after filtering. A thick mucilage solution was the sediment that was gathered.

Concentration and Drying
By gradually heating the mucilage solution at 40–50 °C with decreased pressure until it achieved the proper consistency, it was made thicker. To obtain powdered mucilage, the mucilage was dried for three to four days at 50 °C in a hot-air oven. To prevent it from absorbing any moisture, the dry mucilage was stored in sealed cases at room temperature.

Results and discussion
We thoroughly examined the connection between the colorant MO and the mucilage of the seeds of the species P. major using spectroscopic methods. XRD, FTIR, and UV-visible spectroscopy were used to confirm the mucilage's physical and chemical characteristics, and the interaction mechanism between its functional groups and MO in dye adsorption was clarified for the first time. It is vital to determine whether this eco-friendly substance can be utilized as a durable adsorbent. Understanding the cooperative behavior of the MO dye and, of course, the mucilage molecules is so essential. The printing and textile industries frequently utilize MO ($C_{14}H_{14}N_3NaO_3S$), an anionic azo dye. It is more stable due to its distinctive aromatic structure. The mucilage extracted from P. major seeds is a biopolymer with hydroxyl (-OH), carboxyl (-COOH), and ether (C-O-C) functional groups that can form H-bonds with dye molecules through electrostatic interactions.

Characterization
P. major seed mucilage's FTIR spectra showed several distinct bands of adsorption. It is evident that it included the functional groups required to bind with the MO dye. A wide band is visible around 3400 cm-1. This is caused by the H-bonds between carbohydrates and the stretching vibrations of hydroxyl groups. The aliphatic -CH2 groups in the mucilage backbone's C-H stretching vibration is responsible for a peak at about 2900 cm-1. There would be a distinct adsorption band at about 1850 cm-1. This is because C=O vibrations would cause the carbonyl and carboxyl groups to expand.Additionally, compounds with aromatic or conjugated stretching C=C bonds are associated with frequencies between 1500 and 1600 cm-1. The bending of amide or carboxylate groups may also be the cause, indicating that the mucilage contains protein-based components. A prominent band was visible around 1050 cm-1. It is composed of C-O-C and C-O stretching vibrations, just like the majority of polysaccharides and glycosidic bonds. The waves create a powerful band that is characteristic of glycosidic bonds and polysaccharides.

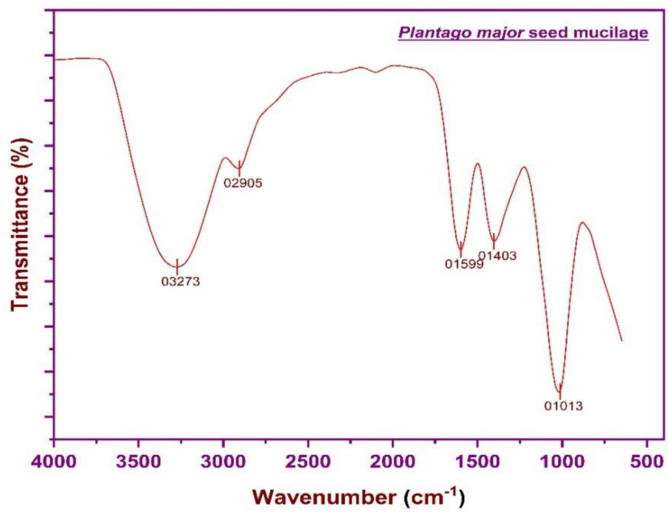


Figure 0-1 FTIR Spectra of P. major seed mucilage

Mucilage with a high concentration of polysaccharides and a poorly organized chain packing is characterized by a large amorphous hump in the XRD pattern at roughly 12.98° or 2θ. The semi-crystalline structure or grouping of somewhat larger crystalline particles, such as mineral salts or biogenic ash, is shown by modest reflections at 29.5° (110) and 38.26° (210) over this backdrop. These peaks' relative weakness in comparison to the humps suggests that the material is amorphous, which often promotes adhesion due to more open, discontinuous binding sites. The dye has been incorporated into the mucilage matrix if there is any change in the hump (12.98) or the (110)/(210) reflections upon loading with the dye.

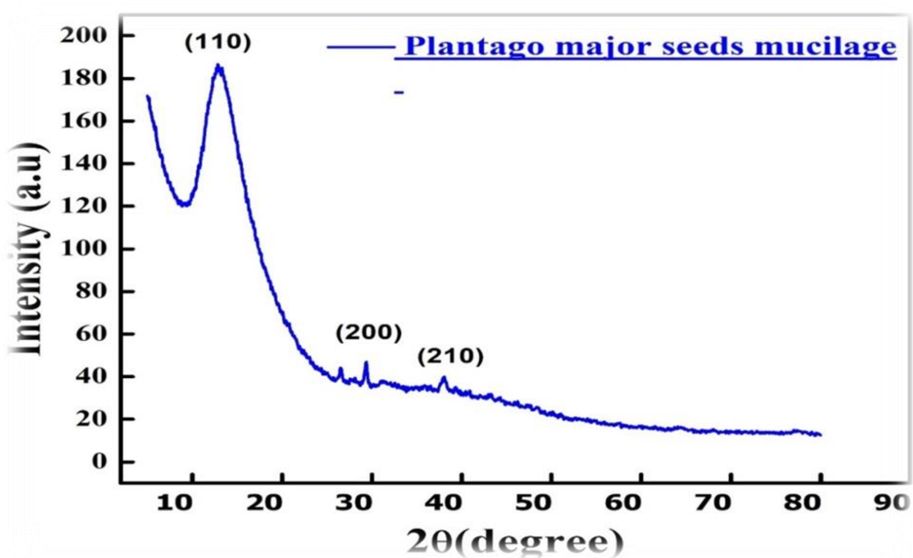


Figure 0-2 XRD spectra of P. major seed mucilage

The chemical makeup of the seed mucilage was investigated using SEM-EDX. The spectra and associated numerical findings are displayed in Figure 3a–b. The examination of EDX Figure 3a demonstrates that the substance is mostly composed of O (51.3 weight percent) and C (13.9 weight percent), indicating that it is rich in polysaccharides. Additionally, a significant amount of N (10.6 weight percent) was discovered, indicating the presence of protein components or amino-functional groups. Strong interactions between these and charged dye molecules are known to occur. The presence of minerals including Ca (5.2 weight percent), K (4.5 weight percent), Mg (2.6 weight percent), and Na (2.3 weight percent) is consistent with the natural mineral composition of biopolymers derived from seeds. These cations may improve the material's ability to absorb substances and aid in ion exchange. The flakes in Figure 3b's SEM image were layered and not flat. As a result, the surface became porous and rough, which is beneficial for adsorption. These findings collectively demonstrate that seed mucilage is an effective low-cost dye-removing absorbent due to its abundance of functional groups and mineral components.

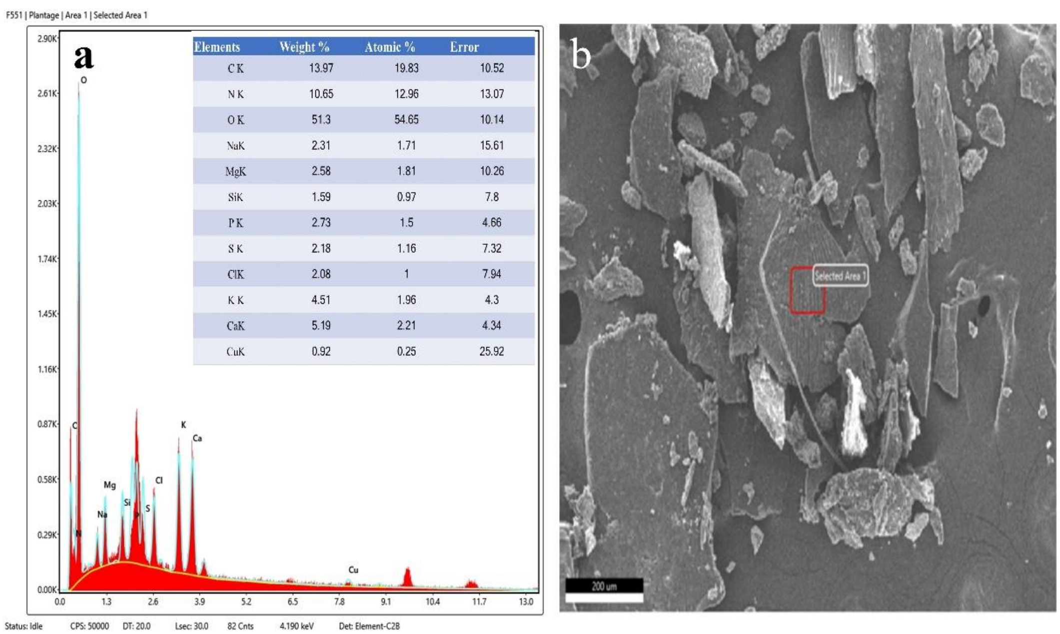


Figure 0-3 EDX Spectra of P. major Seed at (a) with table of elements atomic and weight % inset (b) SEM image at 200 μM

Figure 4 (a-i) shows the SEM analysis of the seed mucilage surface at various magnifications. Figure 4a (scale 200 nm, 20.0 kx): At very high magnification, the mucilage surface shows a thick sheet structure with tiny protuberances. The surface seems rough because to the closely packed layers of carbohydrates in the nanoscale structure. Figure 4b (300 nm, 30.0 kx): Because of the surface's deep flaws and fissures, the mucilage is thin and contains naturally occurring micro-pores. Chromophoric chemicals are transported through these pores. Figure 4c (scale 1μm, 10.0 kx): It is possible to identify larger spaces and interstices, indicating a confirmative porous architecture. By increasing the surface area, these micropores increase the efficacy of adsorption. Figure 4d (5.0 kx, scale 2μm): The morphology shows portions that resemble plates. The flake-like stratification suggests that the components of mucilage are not rigidly connected, making active areas freely accessible. The majority of the structures in Figure 4e (5.00 kx, size 2 μm) are smooth sheet-like structures with tiny surface granules in between, which is typical of a composite construction to balance open and compact space. Figure 4f (scale 10 µm, 1.00kx): Aggregated clusters and scattered fibrous pieces on the surface are visible in the micrograph. The roughness and more active binding sites are increased by these macroscopic features. Figure 4g (size 20 µm, 20 kx): The morphology is less uniform at low magnification, with flexible extensions and masses of aggregates. Surface protrusions suggest a non-uniform structure that is useful for sticking to phenomena. Figure 4h (scale 50 nm, 200 kx): Layers of mucilage particles are seen as broad sheet-like forms with noticeable ridges. A good surface to volume ratio is made possible by this flake-like appearance. Figure 4i (size 20 µm, 500 kx): There are long, linear ridges and grooves that may indicate that the biopolymer's organelles have been arranged in a fibrous shape or that fractures have been established in multiple directions. During adsorption, these channels aid in the dispersion of dye molecules. All things considered, SEM research shows that the mucilage surface is highly stratified, porous, and heterogeneous, with ridges, fissures, and pores at the nanoscale. The strong binding capacity and increased roughness of such a hierarchical structure are crucial for the adherence of dye molecules in aqueous solutions.

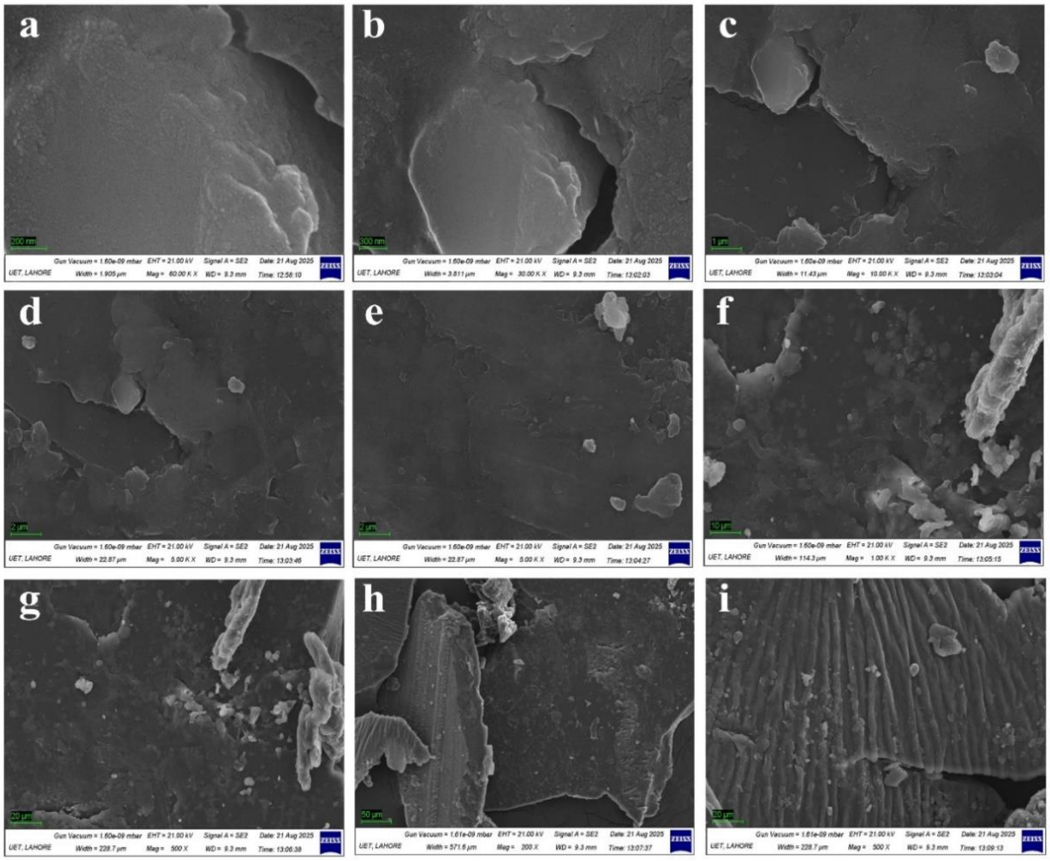


Figure 0-4 SEM pictures of P. major seed at different resolution (a) 200 nm (b) 300 nm (c) 1µm (d) 2µm (e) 2µm (f) 10 µm (g) 20µm (h) 50 µm (i) 20 µm

At various contact times (0, 20, 40, 60, 80, and 100 minutes), UV-Vis spectra were obtained. The absorbance at the maximum wavelength (λmax = 464 nm) exhibited a clear time dependence. The initial concentration of MO was determined by taking the maximal absorption at 0 minutes. After 20 to 100 minutes of contact, the maximum intensity steadily dropped. These findings suggest that the dye molecules were gradually adhering to the mucilage of P. major. The progressive loss of strength in the absence of any significant wavelength change suggests that adsorption, rather than chemical degradation, is the more significant mechanism. Consequently, the time-dependent decrease demonstrates that mucilage can be utilized as a potent natural adsorbent to eliminate MO.

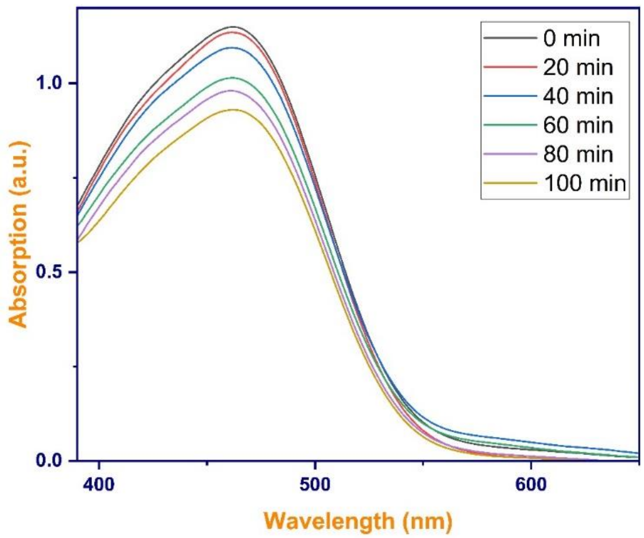


Figure 0-5 : UV-Visible Spectra at Different Time Interval from 0 to 100 Min

Conclusion

The current research proves that extracted seed mucilage is a highly efficient, sustainable and low cost, adsorbent of eliminating methylene blue (MB) cationic dye in aqueous solutions. Detailed characterization assured the heterogeneous and functionalized character of the mucilage which is most suitable in dye adsorption. Analysis of FTIR showed that the functional groups of hydroxyl, carboxyl, and amines are abundant and they are involved in adsorption by the forces of electrostatic attraction, hydrogen bonding and surface complexation. SEM analysis also revealed that the morphology of the surface was porous, layered, and irregular offering a high concentration of available adsorption sites. The EDX analysis was used to confirm the organic-mineral composition of the mucilage as oxygen, carbon, and nitrogen were the main elements with naturally present inorganic ions, including Ca^{2+} Mg^{2+} Na^{+}, and K^{+}. These mineral constituents help to increase the performance of adsorption by adding ion-exchange processes and balance of surface charges. The existence of organic functional groups and mineral ions together gives rise to a surface chemistry that is highly biased to the interaction with cationic MB molecules. Experiments of batch adsorption with methylene blue showed a high rate of reducing the concentration of the dye at the beginning, and a gradual approach to equilibrium. The rationale behind this adsorption behavior can be explained by the fact that the number of active sites at the initial steps of adsorption is high and the active sites become saturated with time. A combination of the surface activity, porosity and internal mineral framework of the mucilage is significant in the regulation of the adsorption process. In general, the results indicate the promising nature of seed mucilage as an economical and environmentally friendly adsorbent in the efficient extraction of cationic dyes like methylene blue in wastewater, which in turn can be used in efficient water treatment processes.

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