



## RESEARCH PAPER

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## Synthesis and characterization of a corncob-based chitosan composite as an economical, ecologically friendly and effective sorbent for adsorption dye from aqueous solution

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### Abstract

A variety of materials are being researched to remove the dye molecules that give water its color. An adsorbent is the corn cob, a food waste that is left over after maize is consumed. The adsorbent prepared from corncob powder, an agricultural waste, was used to successfully remove malachite green dye from aqueous solution. Chitosan has emerged as an inexpensive, non-toxic, biodegradable and readily available option for removing dye molecules by adsorption. The functional groups of chitosan, such as hydroxyl and amino groups, are responsible for the adsorption of dyes. Therefore, the objective of this work was to prepare and evaluate a corncob composite based on chitosan as adsorbents using malachite green dye from aqueous solution. The binding of corncob composite based on chitosan was characterized by Fourier transform infrared spectroscopy, scanning electron microscopy, TGA, DTA, and Brunauer-Emmett-Teller analysis. The results showed that the removal efficiency increased with increasing adsorbent dosage and contact time, and was inversely proportional to increasing pH. The maximum removal of malachite green dye proved that the corncob composite based on chitosan as an adsorbent material has the ability to adsorb malachite green dye from aqueous solution. These studies have shown that the sample can be used as an inexpensive alternative when cleaning wastewater to remove the dye.

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## Introduction

Significant environmental issues have arisen as a result of the textile industry's extensive usage of synthetic organic dyes, especially in aquatic environments. There are currently about 10,000 different types of dyes in use, and their manufacture and use result in significant waste production roughly 700,000 tons per year (Mane *et al.*, 2007a). Because of their chemical makeup, toxicity, and resilience to degradation, these pollutants which are mostly produced by dyeing materials like cotton and polyester-can have a serious negative effect on aquatic life and water quality (Mane *et al.*, 2007b). The aquatic ecosystem is impacted by the colorful materials because they prevent sunlight from penetrating water bodies (Ismail *et al.*, 2018). Malachite green is a dye that has been employed as an antibiotic in aquaculture, which is utilized extensively in many other industries. However, because of its poisonous and persistent nature, it poses serious threats to the environment, animals, and human health (Tewari *et al.*, 2018; Swan and Zaini, 2019; Raval *et al.*, 2017). Dye removal from wastewater contaminants can be accomplished chemically, physically, or biologically. However, for textile wastewater, adsorption treatment is thought to be a better method that is more affordable, quicker, and more universal (Mall *et al.*, 2005). Materials that are abundant in nature and typically require little processing, or that are waste products or by-products of other operations, are considered low-cost adsorbents (Gupta, 2009). One of the world's most plentiful renewable resources is agricultural by-products, which are widely accessible (Demirbas, 2009). Structural modifications have been made to the biosorbents to improve their binding capacities, selectivity, and environmental stability in terms of their reusability because lignin cellulosic biomasses behave as good effective adsorbents but have low sorption efficiencies (Akar *et al.*, 2013; Foroughi-dahr *et al.*, 2015). In the past, chitin from crustacean sources was primarily chemically deacetylated to create commercial chitosan samples. More recently, chitosan from fungi is gaining interest in the market, driven by vegan demands and these are better in

terms of low viscosity (Ghormade *et al.*, 2017) and exhibit a very high deacetylation degree (Aranaz *et al.*, 2009; Rinaudo, 2006; Jiang *et al.*, 2017).

Dye adsorption dye from aqueous solutions using a variety of inexpensive adsorbents and unconventional sorbentes, including clay (Arellano-Cárdenas *et al.*, 2013), pine wood (Zhang *et al.*, 2011), bivalve shell-treated *Zea mays* L. (maize) husk leaf (Jalil *et al.*, 2012), carbon from spent tea leaves (Akar *et al.*, 2013), and walnut shell (Dahri *et al.*, 2014). This corn cob can be widely utilized as an adsorbent because it is a naturally inexpensive material that is simple to change (Palukurty *et al.*, 2014). A common annual cereal that grows quickly, has a large fibrous root structure, can resist harsh conditions, and yields a lot of seeds is maize. Several researchers have altered natural biopolymers with particular functional groups, such as carboxyl groups, to eliminate particular contaminants and enhance their adsorption capacities and efficiencies (Goyal *et al.*, 2009). The most often employed adsorbents are agricultural wastes, which may also be recycled at a minimal cost. In this regard, numerous studies have been conducted to find inexpensive adsorbents. For instance, corn straw is a naturally occurring lignocellulosic fiber that is accessible all over the world and has functional groups that can bond with dyes (Suteu *et al.*, 2011; Berber-Villamar *et al.*, 2018; Aljeboree *et al.*, 2019). Because of these qualities, corn straw has the potential to be an adsorbent.

A determined adsorbent's maximal adsorption capacity can be found using the batch studies parameters, which can also provide information regarding energetic, steric, and affinity views. The main objectives are to provide a summary of recent information concerning the use of corncob based chitosan composite as sorbent and to discuss the main interactions involved in the adsorption process. The present work clearly demonstrates the feasibility of utilizing Corncob based chitosan composite as a novel, effective, efficient, eco-friendly, and inexpensive sorbent for the removal of Malachite Green dye from aqueous solutions. The synthesized

adsorbents were investigated by different physicochemical tools such as TGA, SEM, FTIR, SEM and N<sub>2</sub> adsorption/desorption. The Malachite Green dye sorption performance of Corn cob based chitosan composite was affected significantly by solution pH, dosages, shaking contact time, and initial dye concentration.

## Materials and methods

### Materials

Sigma Aldrich provided the chitosan, acetic acid, NaOH, HCl and ethanol that were employed in this investigation. Without any additional purification, these analytical-grade compounds were utilized directly in this experiment.

### Preparation of chitosan

To dissolve the chitosan, 1M was added to 1% acetic acid and agitated for 60 minutes. The mixture was simultaneously mixed using the magnetic stirrer. The solution was stirred for 12 hours at room temperature, centrifuged for 45 minutes, and then heated in a hot air oven.

### Preparation of corn waste

Corn cob, an adsorbent material, was collected from adjacent fields. The gathered material was ground into a powder using a steel mill after being sun-dried. A 40–60 mesh screen was then used to filter the adsorbent. After soaking for 12 hours, the ground material was rinsed with both hot and cold distilled water until the wash water was colorless. The cleaned material was then stored in a glass bottle for use as an adsorbent without any pretreatment after being dried for 12 hours at 50°C in a hot air oven.

### Synthesis of corn cob based chitosan composites

A corresponding quantity of corn cob powder and chitosan was added to the solution along with 100 milliliters of distilled water and a synthesized corn cob based chitosan composite. For almost four to six hours, the reaction continued. Following that, it was centrifuged at 2000 rpm. For 30 minutes, the resulting composites were heated to 80 °C. To speed

up the drying process, ethanol was used to wash the modified composite material that was produced.

### Batch adsorption studies

Batch experiments for the adsorption were placed in a 250 mL round-bottom flask at different dye concentration (10,20,30,40,, 50 with 30°C, pH:7.0, 0.1g/L sorbent), sorbent dosages (0.02,0.04, 0.06, 0.08, 0.10g/L with 30°C, pH:7.0), Time (20, 40, 60 80, 100, 120, 140, 160, 180 mints with 30°C, pH:7.0, 0.1g/L sorbent), pH (2.0, 4.0, 6.0, 8.0, 10.0 with 30°C, pH:7.0, 0.1g/L sorbent) were investigated. A rotary orbital shaker was used to shake the mixtures at 160 rpm. The adsorbent was then centrifuged at 4500 rpm to remove it from the aqueous solution. To ascertain the remaining dye content, the absorbance value of the supernatant solution was measured using a Shimadzu UV-1601 Spectrometer at 617 nm both before and after treatment.

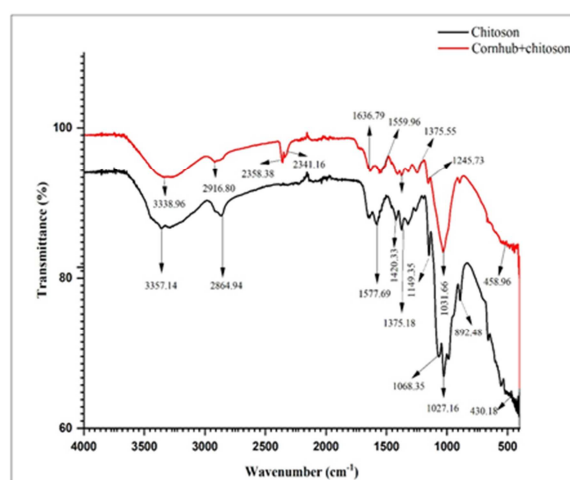
### Characterization of corn cob composite based on chitosan

The surface of the chitosan was altered to distinguish between dried and processed maize waste, and FTIR spectra at different wavelengths between 4000 and 500 cm<sup>-1</sup> were used to characterize the presence of functional groups. The TGA thermogram of Corn cob based chitosan composite showed the weight loss profile at various temperatures at a flow rate of 52.4 mL/min and at a heating rate of 46.8°/min in a nitrogen atmosphere. A differential thermal analyzer was used to characterize the physical properties of the Corn cob based chitosan composite, including heat absorption, phase transition, and crystallization. In this investigation, a scanning electron microscope was used to examine the surface morphology and particle size distribution of Corn cob based chitosan composite. The optical characteristics of Corn cob based chitosan composite were examined using the UV-vis Spectrophotometer in the wavelength range of 200 nm to 700 nm. The Brunauer–Emmett–Teller (BET) analytical method was used to assess the surface area of a Corn cob based chitosan composite utilizing nitrogen adsorption-desorption at 77.35°K.

## Results and discussion

### Fourier transform infrared spectroscopy (FTIR)

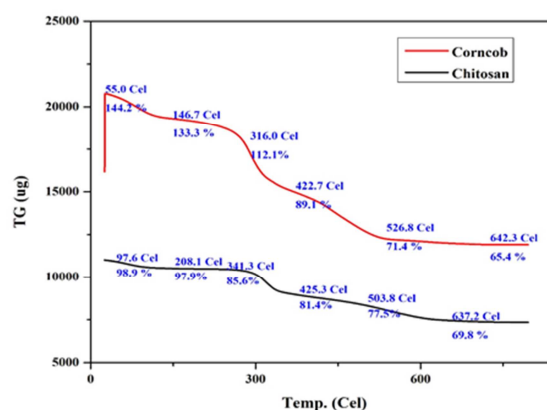
As illustrated in Fig. 1, the functional groups on the samples in the 4000–500  $\text{cm}^{-1}$  range were investigated using FT-IR spectrophotometry. The chitosan corn cob O-H stretching frequency may be created by intermolecular hydrogen bonds that bind the compounds found in corn, which is why the broadband's frequency range was 3338.96–2916.80. The cellulose's –OH stretching vibrations are linked to the strong absorption peak in the chitosan spectrum that appears at 1068 and 1027  $\text{cm}^{-1}$ .



**Fig. 1.** FTIR spectrum of corn cob composite based on chitosan

The C–H stretching vibrations of the methyl, methylene, and methoxy groups in chitosan peak at 1577 and 1420  $\text{cm}^{-1}$  were respectively. According to Chan and Koay (2019), compounds with considerable adsorption in chitosan-corn cob of around 1031.66  $\text{cm}^{-1}$  under the frequency of functional groups lasted aromatic C-H and C=O groups included aldehydes, ketones, esters, amides, and carboxylic acids. The absorption bands at 1375  $\text{cm}^{-1}$  and 1149  $\text{cm}^{-1}$ , respectively, reflected the C=O stretching present in hemicelluloses and the –C=C– stretching. The 1636  $\text{cm}^{-1}$  is due to the stretching of the C=C ring. The peak was seen at about 1245  $\text{cm}^{-1}$  due to the rise. The presence of glucosidic ( $\beta$ ) bonds between cellulose sugar units and hemicelluloses, as well as the C-O-C vibrational stretching of polysaccharides, were shown by the bands in the fingerprint region at 1027  $\text{cm}^{-1}$

and 892  $\text{cm}^{-1}$ , respectively. The adsorption maxima in the 3500–2000  $\text{cm}^{-1}$  range can be taken into account for angular deformation in the bonding plane of C-H aromatic rings. Furthermore, repeated peaks in the 1500–1000  $\text{cm}^{-1}$  range may be ascribed to the N-H bond. The surface of sorbents exhibits functional groups such as carboxyl and hydroxyl groups, which are necessary for the adsorption of dye molecules (Escamilla-García *et al.*, 2022; Yeng *et al.*, 2013; 2015).

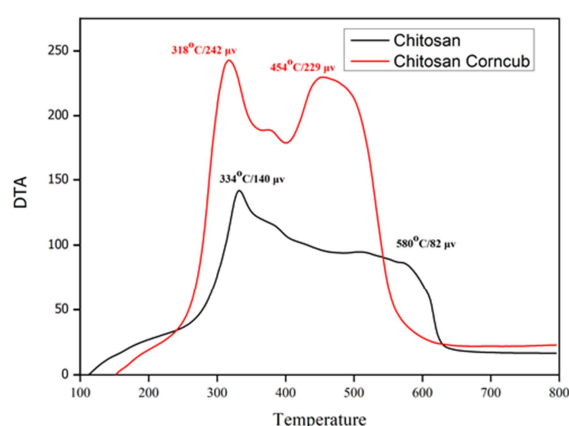


**Fig. 2.** Thermal gravimetric analysis of corn cob based chitosan composite

### Thermogravimetric analysis (TGA)

One method for measuring a sample's mass in relation to temperature is thermogravimetric analysis (TGA). Examining the thermal stability and purity of solid materials at temperatures between 0 and 700  $^{\circ}\text{C}$  is especially useful. Fig. 2 showed a chitosan composite made from corn cobs. TGA curves showed that both samples underwent three stages of heat deterioration. The breakdown of cellulose and hemicellulose is the main characteristic of the previously described stages, but lignin also decomposes concurrently within that temperature range; the last stage of lignin degradation is what causes the protracted weight loss that happens after 50  $^{\circ}\text{C}$ . Weight loss may occur gradually beginning at 200  $^{\circ}\text{C}$  due to the breakdown of the low molecular weight chitosan. The dehydration of the chitosan anhydroglucosidic ring is one of the complex processes that results in a more pronounced weight loss beginning at 300  $^{\circ}\text{C}$  (Jayakumar *et al.*, 2008).

Weight loss was accomplished as a result of the total breakdown of the adsorbents; however, at 280 °C, a larger mass reduction was brought about by the inclusion of thermally stable chitosan. The inclusion of chitosan with a high thermal withstanding capacity explains the larger residual masses of chitosan with corncob (Cardenas and Miranda, 2004).



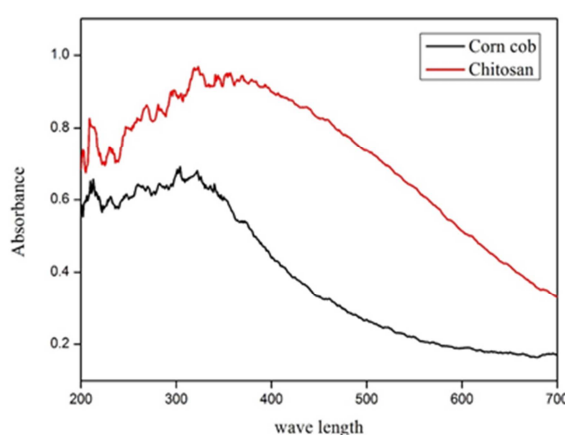
**Fig. 3.** DTA analysis of corncob based chitosan composite

The endothermic reactions range from ambient temperature to 800°C, as seen in Fig. 3. These temperature variations cause the evaporation of physisorbed water that has been adsorbed on the outer surface of the polymer as well as water from the interlamellar gap (Hong *et al.*, 2007). The curve's loss of definition of the peaks at about 200°C and 400°C is another feature that is noticeable when maize and chitosan are combined with corn cob. The DTA curves for chitosan and chitosan with corn do not significantly differ from the parent material; however these peaks were linked to interlamellar water loss and dehydroxylation from various settings (Chan *et al.*, 2018).

#### UV-visible spectroscopy

The optical characteristics of the particles, which are evaluated using UV-visible spectrosopes, were one of the most important studies for their applications. It contrasts the light intensity that strikes an object with the light intensity that travels through a sample of corncob based chitosan composite. The wavelengths of UV light, which are in the electromagnetic spectrum, range from 200 to 700 nm, which is too far

for the human eye to detect. The associated findings for the UV transmittance and absorbance spectra, which were measured between 200 and 700 nm in wavelength, are displayed in Fig. 4. All of the particles displayed more incoming radiation at a wavelength of 300 nm, which is ideal for dye removal applications. However, a minimum amount of light at a wavelength of 500 nm was absorbed, as seen in Fig. 4. The absorbance peak indicates that the particle was allowed to pass a specific amount of light, according to Bustamante *et al.* (2024). This result could be due to the fact that cellulose has a bigger surface area.



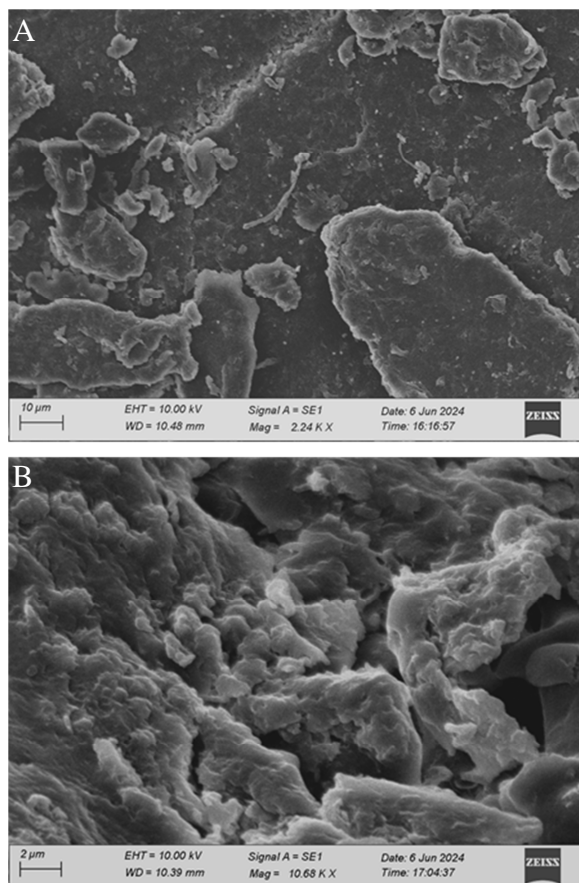
**Fig. 4.** UV-Vis spectrum of corncob based chitosan composite

In the visible light band between 210 and 350 nm, pure synthesized samples showed a discernible absorption, which is somewhat in line with the results of the previous study (Aswathy *et al.*, 2024; Silva Viana *et al.*, 2020). By serving as a sensitizer to absorb more photo-generated electrons and holes, corn enhanced the adsorption activity and caused a little shift in the longer wavelength region (Wang *et al.*, 2022). The produced composite exhibits broad background absorption with a higher absorbance at 340 nm, even though the absorption edge remains constant.

#### Scanning electron microscopy (SEM)

The surface morphology of the corncob based chitosan composite was investigated using a scanning electron microscope (SEM) the findings are displayed in Fig. 5. The images (Fig. 5A) showed that the

adsorbent had more uneven holes and rough surfaces than the raw material because of the reduction of volatile materials during the high temperature carbonization process. Interspatial holes in the matrix of the polymer indicated improved adsorption potentials (Iheanacho *et al.*, 2021).

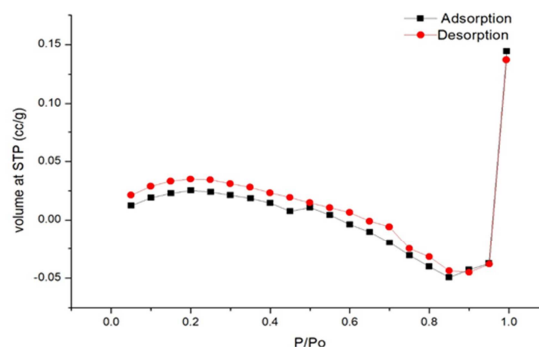


**Fig. 5.** SEM of chitosan (A) and chitosan-corn cob (B)

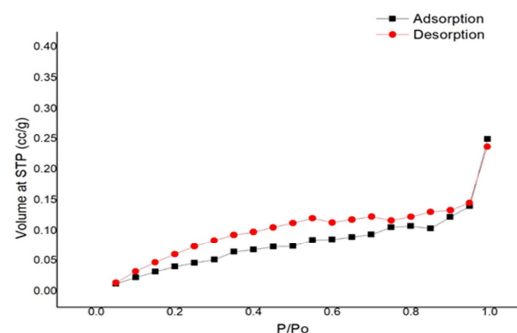
A subset of tubes that transport nutrients to the other plant parts make up the cellular structure depicted in Fig. 5A. The chitosan surface (Fig. 5A) is smoother than the corncob based chitosan composite (Fig. 5B), which showed rougher surfaces. This form has limited porosity, as is typical for raw biomaterials (Petrovic *et al.*, 2015). The majority of the ground tissue in maize corn stems has vascular bundles implanted in it, according to Vucurovic *et al.* (2014). The cell wall is composed of cellulose, while the primary component of the cell wall of vascular components is lignin. One can see the material's small pores and rough surface (Trujillo-Reyes *et al.*, 2012).

#### Brunauer–Emmett–Teller (BET) analysis

By using the gas adsorption technique, BET analysis is primarily utilized to ascertain the solid materials' specific surface area and porosity. The BET and BJH methods were used to measure the surface area and pore size of the processed corncob based chitosan composite. Fig. 6 and 7 show the nitrogen adsorption-desorption isotherm recorded at 77.350K to analyze the surface area, porosity size, and pore volume of synthetic corncob based chitosan composite. Fig. 7 shows the chitosan-incorporated corn cob's surface area and pore radius, which are 19.047 m<sup>2</sup>/g and 0.299 nm, respectively. According to Fig.6, the chitosan average surface area is 0.955 m<sup>2</sup>/g, and its pore radius is 6.12 nm. The chitosan-corn cob material's surface area increased to 5.094 m<sup>2</sup>/g.



**Fig. 6.** BET analysis of chitosan by nitrogen adsorption and desorption

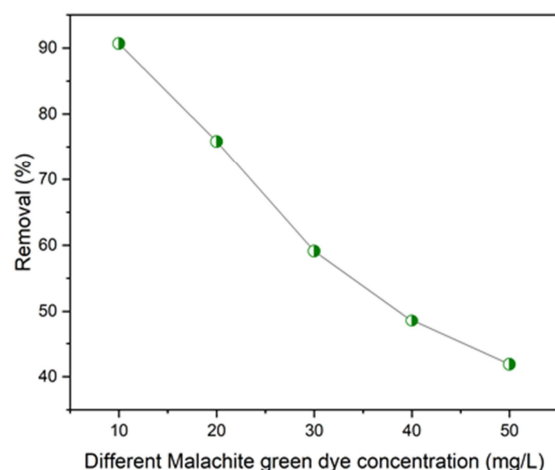


**Fig. 7.** BET analysis of corncob based chitosan composite by nitrogen adsorption and desorption

The homogeneous coating and integral penetration of a chitosan polymeric chain into the corn hub, which

tends to widen the interspacing and hence increase the surface area, is one explanation for the increased surface area of the corncob based chitosan composite (Oraon *et al.*, 2016). It was determined that the corncob based chitosan composite had a pore radius of 3.43 nm. The type I isotherm, which shows the presence of microporous structure, was found in the pore size distribution of the chitosan. The hysteresis loop was found in the pore size distribution of the corncob based chitosan composite.

This loop shows that the capillary condensation that takes place in a mesoporous range is consistent with the type IV isotherm. The total pore volume is determined by measuring the amount of vapor absorbed by a material at a relative temperature close to unity. Corn cob based chitosan composite were discovered to have pore volumes of 2.94 cc/g and 8.733 cc/g respectively. The chitosan-corn cob showed a larger surface area than the chitosan alone. According to (Da Silva Alves *et al.*, 2021), the adsorption of different colors and metals might be increased by increasing the material's surface area and decreasing its particle size.



**Fig. 8.** Effects of the concentration on the adsorption of malachite green

#### *The effect of dye concentration*

Fig. 8 demonstrated how the rate of adsorption on corncob based chitosan composite was impacted by the concentration of malachite green dye in the solution. The figure demonstrated that when the dye

concentration increased, the percentage of dye adsorption by chitosan-based maize cob decreased. The greatest amount of dye removal was noted at a particular contact time and dye concentration of 10 mg/L. The clearance of malachite green dye is approximately 90% at all dye concentrations, in contrast to high dye concentrations. Fig.9 illustrates the impact of a corncob based chitosan composite on the sorption of Malachite Green dye at pH 6.0. It is clear that the sorption rates and capacities of finer chitosan composite particles made from maize cobs are higher. The increased specific surface area and decreased intraparticle diffusion resistance of the corn corncob based chitosan composite, which improve internal site accessibility, account for this pattern (Schiewer and Patil, 2008; Tunc *et al.*, 2009; Dotto *et al.*, 2015; Nayda Karina Berber-Villamar *et al.*, 2018). Further studies employing the smallest particle size at different dosages were prompted by this discovery.

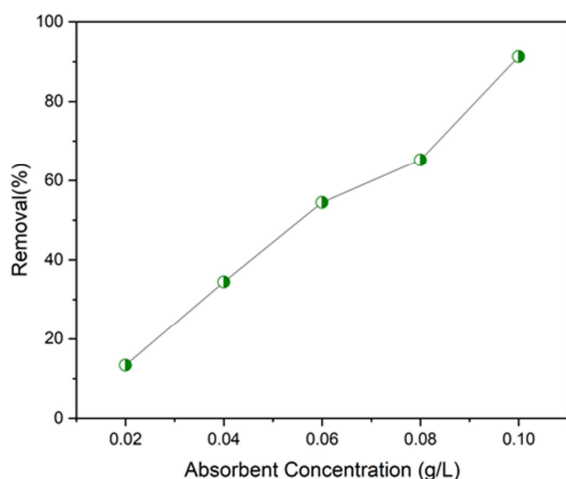
Additionally, regardless of the initial dye, the initial sorption rate was relatively quick due to the high driving force to move Malachite Green dye molecules from the aqueous solution to the corncob surface and the abundance of positively charged binding sites available for sorption (Dotto *et al.*, 2015; Moyo *et al.*, 2017; Nayda Karina Berber-Villamar *et al.*, 2018). The sorption rate then steadily dropped and eventually reached dynamic equilibrium as a result of a gradual decrease in the mass transfer driving force and the number of empty binding sites (Lim *et al.*, 2015; Puentes-ardenas *et al.*, 2016; Nayda Karina Berber-Villamar *et al.*, 2018).

Longer contact between the dye molecules and the adsorbent is made possible by the macro and micropores of the modified corncob. Consequently, the dye takes longer to cling to the surface of the adsorbent and diffuse within its porous structure (Abdullah *et al.*, 2005).

#### *The effect of adsorbent dosage*

Fig. 9 demonstrated that the percentage of malachite green dye clearance increased with the addition of

adsorbent dosage. The percentage of color removal for the corncob based chitosan composite increased from 15% to 94% when the adsorbent dosage was increased from 0.02 g to 0.10 g/mL. The availability of the adsorption active sites increased as the surface area attribution rose in tandem with the adsorbent dosage. The highest dye removal in this research was achieved at a dosage of 0.10 g/L, with malachite green in the aqueous solution being almost constant within 0.06 to 0.08 of adsorbent concentration. The effects of a chitosan composite based on corn cobs on the sorption of Malachite Green dye at pH 6.0 are shown in Fig.9. It is evident that finer chitosan composite particles based on corn cobs have higher sorption rates and capacities. This pattern is explained by the corncob based chitosan composite increased specific surface area and decreased intraparticle diffusion resistance, which enhance internal site accessibility (Schiewer and Patil, 2008; Tunc *et al.*, 2009; Dotto *et al.*, 2015; Nayda Karina Berber-Villamar *et al.*, 2018). This discovery led to additional research using the lowest particle size at various dosages.

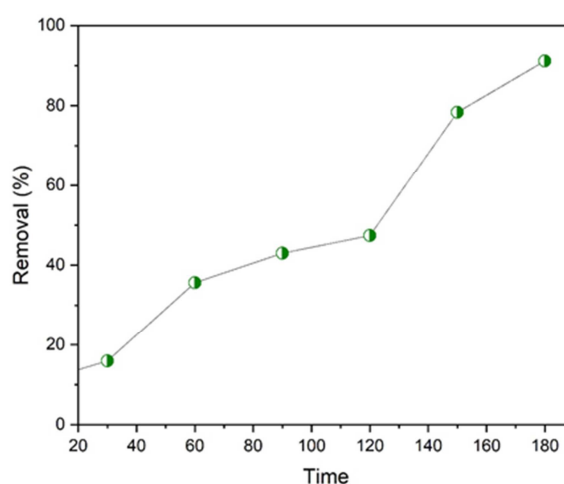


**Fig. 9.** Effects of the adsorbent dosage on the adsorption of malachite green

#### *The effect of contact time*

The impact of contact duration on malachite green dye adsorption by corncob based chitosan composite adsorbent is depicted in Fig. 10. We looked at the variation in contact time. It proved that 180 minutes is the ideal contact period for this experiment. corncob based chitosan composite absorb dyes more

rapidly; up to 80% of the pigment is eliminated after 160 minutes of equilibrium time. Otherwise, when the adsorbent dosage is increased and the dye's initial concentration is maximized at 180 minutes, 95% of the dye will be eliminated. As time increases, both the adsorption capacity and the removal efficiency increase. However, they eventually start to decline until they reach the equilibrium state. This can be explained by the fact that further adsorption is inhibited once the active sites achieve a saturation state. Similar behavior has also been reported (Azhar *et al.*, 2005).



**Fig. 10.** Effects of the time on the adsorption of malachite green

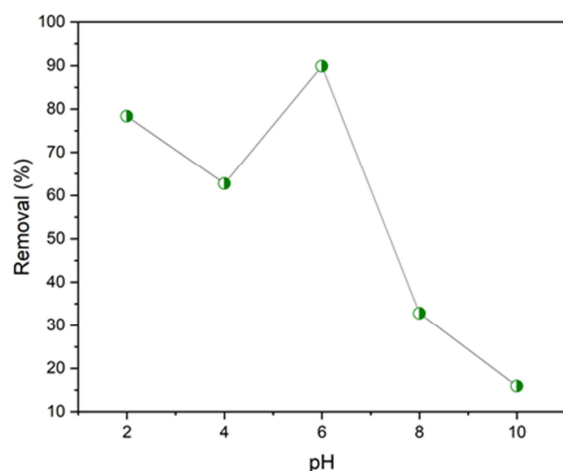
#### *Effect of pH*

Fig. 11 illustrates how pH affects malachite green dye adsorption. The effect of pH on adsorption was examined in the (2–10) range by altering the samples with HCl and NaOH solutions. It was shown that the adsorption capacity steadily increases to achieve a maximum adsorption percentage at pH 6 and then drops as the pH climbs from 2 to 10. Additionally, the removal efficiency drops from 80% to 59% before rising to 90%, which is at its highest at pH 6.

Additionally, the removal efficiency drops from 80% to 59% before rising to 90%, which is at its highest at pH 6. According to Parvin *et al.* (2018), this can be explained by the fact that malachite green is a cationic dye and that higher pH values have more OH<sup>-</sup> ions, which leads to enhanced adsorption due to the



electric attraction between charges. A sorbent's surface charge and sorption capacities are significantly impacted by the pH of the solution.



**Fig. 11.** Effects of the different pH on the adsorption of malachite green

The point of zero charge or isoelectric point can be used to qualitatively evaluate the surface charge (Vafahah *et al.*, 2014; Puentes-ardenas *et al.*, 2016; Nayda Karina Berber-Villamar *et al.*, 2018). The corncob based chitosan composite was found to have a pH of 6.0 (Fig. 10). Consequently, the corn cob-based chitosan composite particles contain positive charges for solution pH values below this threshold, and vice versa. According to Leyva-Ramos *et al.* [Leyva-Ramos *et al.*, 2005 pH (6.2) and Nayda Karina Berber-Villamar *et al.*, 2018 pH (6.83)], the natural corncob based chitosan composite weak acidity is caused by a slightly higher concentration of acid sites than basic ones. The value of 6.0 is comparable to their findings.

### Conclusion

One agricultural waste product produced in large amounts during maize processing is the corncob. This study demonstrates how agricultural waste can be used economically to remove water pollutants. When applying the physiochemical activation approach to prepare alternative adsorbents, corn cobs which are typically considered waste make valuable precursors. In this study, we used adsorption to remove the malachite

green from corncob based chitosan composite in order to examine their ability to purify water. The adsorbents were characterized using BET, Infrared, and UV-Vis spectroscopy, and their structure was verified using the SEM technique. Thermal stability was shown by thermogravimetric analysis. According to characterization, the chitosan composite made from corncobs contains big pores that improve its adsorption capacity. The initial dye concentration was inversely correlated with the amount of Malachite green dye removed by the corn cob-based chitosan composite. Batch adsorption tests produced the best-fitting adsorption isotherm for the elimination of malachite green color using a chitosan composite based on maize cobs. This implies that the adsorption process took place at the adsorbent's outer surface in a monolayer. According to batch adsorption tests, the adsorption effectiveness rapidly rises with increasing contact time and initial dye concentration and has strong inverse connections with the pH of the solution and the dosages of the corn cob-based chitosan composite. Several parameters were examined to optimize dye adsorption efficiency, and the results indicated that basic pH was optimal. The ability to adsorb dyes was associated with the number of OH-groups. Additionally, we employed maize cobs, an agricultural waste product that is inexpensive, easily accessible, environmentally benign, and an efficient adsorbent. Finally, chitosan derived from corn cobs might be a more effective adsorbent for eliminating dyestuff from an aqueous solution.

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