International Journal of Development, Vol.8, No.(1) (2019): 81-93
ISSN: 2314-5536 **e-ISSN: 2314-5544 (Online)**
www. ijd.byethost13.com **e-mail: fas_ijd@yahoo.om**

Methylene blue removal from wastewater using silica/corncob nanocomposite

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ABSTRACT

Silica/corncob nanocomposite was prepared for remove of methylene blue. Fourier transform infrared (FTIR) spectrophotometry, X-ray diffraction (XRD), Scanning electron microscopy (SEM), dispersive energy x-ray spectrometer (EDX), High-resolution transmission electron microscopy (HR-TEM), and Zeta potential measurements were performed to reveal functionality, crystal structure, morphology, and point of zero charge of the synthesized sorbents. The effect of contact time, initial dye concentration, pH and sorbent dose were evaluated to study the removal efficiency and the adsorption capacity for silica/corncob nanocomposite. The equilibrium concentration of dye was measured by spectrophotometer. Adsorption isotherms and kinetics were also evaluated and reported. The removal efficiency of silica/corncob nanocomposite was (98%).

Keywords: Corncob, Silica nanocomposite, Methylene Blue, Sorption.

INTRODUCTION

Water is an important and rare commodity in all our life.Due to the growth of the world's populationand industries, many countries over the world face water shortage[1].Particularly, by 2025 Egypt may subject to water and energy shortages[2].Hence, all countries over the world adopting of water security strategy by treatment the wastewater and reuse it. Among the wastewater pollutants, dyesfrom various industrieshaveincreased and now become a serious environmental and human health problems [3, 4].Dyes are natural or synthetic coloredorganic compounds,although there are many naturaldyes available but generally, thesynthetic dyes are more widespread due to its availability and cheap price [5].

Synthetic dye discharge to wastewater from many industrial processes including textile, paper, plastics, printing, pigment, paints, food, and pharmaceuticals which generate large volumes of colored wastewater [6].

In recent years, the treatment of industrial wastewater contaminated with dye became more important. Chemical oxidation, membrane processes, coagulation, distillation, ion exchange, reverse osmosis, biological treatment, and solvent extraction were reported as various technologies for removing dyes from wastewaters [7-8]. Due to the high cost, low efficiency, and generation of toxic by-products of the above methods, the sorption process has been nominated as an efficient and cheaper technology for the dyes removal from wastewater[9].Several sorbents have been used such as the activated carbon, clays, chitosan, and zeolites[10].Due to its widespread availability and low cost of agricultural solid waste, many researchers use it as sorbent not only to treat the dye from wastewater but also to solve agriculture waste disposal problems to farmers[11, 12].

Nowadays, nanotechnology is as a wide application in the field of environmental science. The main advantage of this technology is to provide unique physiochemical properties such as high surface area, and high mechanical and thermal properties, which expected to influence the sorption capacity and increase the removal efficiency of dyes within a short time [13].

In this study, silica/corncob nanocomposite was prepared to remove methylene blue from wastewater. The characterization of the synthesized sorbent was investigated using different techniques. The factors that may affect the removal efficiency and the adsorption capacity of silica/corncob nanocomposite were studied. Adsorption isotherm and kinetic were also evaluated and reported.

MATERIALS AND METHODS

All chemicals employed in this study were of analytical grade purity and were used without further purifications.

1.Preparation of the silica/corncob nanocomposite

Corncob which collected from Menoufia, Egypt was cut into small pieces and washed with distilled water to remove any impurities. Thesis pieces were dried under sunlight and then dried at 70 °C for 24 hours. The samples were then grounded into powder using a grinder. The powdered corncob was soaked in 1.0 M hydrochloric acid(HCl) at 60 °C for 24 hours and then washed with distilled water and dried at 60 °C for 24 hours. In Parallel 30 ml of ethanol was added into 5.5 ml of distilled water and stirred for 10 min. 1ml tetraethyl orthosilicate (TEOS) was then added and again stirred for 20 min. 1.5 ml ammonium hydroxide (NH_4OH) was added and The mixture was stirred for 1 hour to obtain nanosuspension silica (Silica sol). 5 g of treated corncob was added to 25 ml of silica sol and shaken for 20 hours. The resulting silica/corncob nanocomposite was separated by centrifugation and then dried at 100 °C for 3 hours[14].

1.1Sorbent characterization

The functionality, crystal structure, morphology, and point of zero charge of the synthesized sorbent were investigated using different techniques.

FTIR spectra of sorbents were recorded over a range of 4000–400 cm^{-1} at room temperature using an FTIR spectrophotometer (FT/IR-6100 Jasco, Japan) after dispersing the samples in excess KBrto analyze the functional groups.

The crystal phase of synthesized sorbents was performed using XRD (X’Pert PRO PANalytical, Netherlands) at 45 kV and 30 mA with Cu Kα radiation (λ = 1.5404 Å) and the HighScore Plus software.

The surface morphology of Corncob, silica/corncob nanocomposite, and magnetic silica/corncob nanocomposite was analyzed using scanning electron microscopy(SEM, JSM 6360LV, JEOL/Japan)at an accelerating voltage of 10-15 kV. The microscope was attached to a dispersive energy x-ray spectrometer (EDX) to supporting the information of the prepared composite.

High-resolution transmission electron microscopy (HR-TEM, Tecnai G20, FEI, Netherlands) was used to characterize the shape and size of the silica/corncob nanocomposite by bright-field imaging at 200 kV using a LaB6 electron source gun.

To reveal the point of zero charge (PZC) of the silica/corncob nanocomposite, Zeta potentials (Zetasizer Nano S, MalvernInstruments, UK) of the silica/corncob nanocomposite aqueous suspensions of different pH (2–11) were measured.

2.Sorption study

2.1 Methylene blue sorption study

The batch equilibrium experiment was employed at room temperature to remove methylene blue dye from 100 ml aqueous solutions by silica/corncob nanocomposite. Different adsorption conditions were investigated including; (i) various time intervals (0–60 min) at pH = 7.5 using 0.15 g of sorbent and 20 mg/L of methylene blue, (ii) different concentrations of dye (5, 15, 20, 50, 100, 250, and 500 mg/L) at pH 7.5 and 0.2 g of sorbent for 30 min, (iii) the influence of pH (from 2.5 to 11) using 0.2 g of sorbent, where the pH of solution was adjusted by HCl and NaOH, (iv)effect of silica/corncob nanocomposite dose (0.01, 0.025, 0.05, 0.1, 0.2, and 0.3 g) in 20 mg/L of dye, and the solution was filtered and the remaining concentration was measured by spectrophotometer.

The removal efficiency percent (R) of the sorbent was calculated from equation (Eq. 1),

$$\boldsymbol{R = (C_o - C_t/C_o) * 100} \text{........... (Eq. 1)}$$

Where; C_o: the initial dye concentration (mg/L), C_t: the remaining dye concentration (mg/L) after a period of time (t). The amount of the methylene blue uptake (q) within the sorbent was calculated as follows:

$$\boldsymbol{q = (C_o - C_t) * (V/M)} \text{......... (Eq. 2)}$$

Where V: volume of solution (ml), and M: mass of the sorbent added (g).

2.2 Study of kinetic model

To identify the sorption kinetics of methylene blue removal using silica/corncob nanocompositeas sorbent, Pseudo-first-order, and pseudo-second-order have been applied.

a- Pseudo-first-order

The linear form of the pseudo-first-order kinetic model is represented by

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \quad \text{............. } \textit{(Eq. 3)}$$

Where; q_t: the sorption capacity (mg/g) at any given time (t, min), q_e: was the equilibrium sorption capacity (mg/g), and k_1: rate constant of the pseudo-first-order (min^{-1})which can be determined from the slope of the linear plot of log (q_e– q_t) versus t [15].

b- Pseudo-second-order

The pseudo-second-order reaction was used to determine the sorption rate constant.

$$\frac{t}{q_t} = \frac{1}{K_2 q_e} + \left(\frac{1}{q_e}\right)t \text{........... (Eq. 4)}$$

Where; K_2: rate constant of the second order reaction (g/mg min) which can be calculated from the intercept of the linear plot of t/q_t versus t. The following expression denoted the sorption rate h (mg/g min) [16]:

$$h = k_2 q_e^2 \text{.................... (Eq.5)}$$

c- Isotherm study

Adsorption isotherms illustrate how the adsorbate molecules interact with the sorbent particles and in the current study, the interaction of methylene blue with silica/corncob nanocomposite as sorbent were fitted by applying four isotherm models, namely Langmuir, and Freundlich.

d- Langmuir Isotherm

The Langmuir isotherm suppose a monolayer adsorption which represents by the following equation:

$$q_t/q_{max} = bC_t/\,(1 + bC_t) \text{........ (Eq. 6)}$$

Where; q_e: the equilibrium sorption capacity (mg/g), C_e: the equilibrium concentration (mg/L), q_{max}: the maximum sorption capacity (mg/g), and b: the Langmuir constant (L/mg), which correlates to the adsorption energy.

e-Separation factor

The essential characteristics of the Langmuir isotherm can be expressed in terms of separation factor R_L which is defined as:

$$R_L = 1/(1 + bC_o) \text{.......................... (Eq. 7)}$$

$R_L > 1$ represents an unfavorable adsorption, $R_L = 1$ represents linear adsorption, $R_L = 0$ translates into irreversible, whereas R_L values between 0 and 1 indicate favorable adsorption[17].

e-Freundlich Isotherm

The Freundlich isotherm is represented by the following equation:

$$\ln q_e = \ln k_f + \left(\frac{1}{n}\right) \ln C_e \text{......... (Eq. 8)}$$

Where; k_f: the Freundlich constant, and n: the strength of adsorption which can be calculated from the intercept and slope respectively, of the linear plot of ln q_e versus ln C_e.
n=1 represents linear adsorption; n<1 indicate chemical process; n>1 represents physical process [18].

RESULTS AND DISCUSSION

1.Characterization of sorbent

1.1Fourier transform infrared (FTIR) spectrophotometry

Figure (1) shows the FTIR spectra of silica/corncob nanocomposite. FTIR spectra of sorbent show characteristic peaks of cellulosic materials; a strong and broad peak of OH stretching at (3400 – 3440 cm^{-1}),low intensity of CH methyl group at (2920-2930 cm^{-1}), and the absorption peak of carboxylate group COO^- at (1535-1537 cm^{-1}) [19]. The FTIR spectra also showed absorption peak at(1030-1045 cm^{-1}) which indicate the presence of Si-O-Si[20].

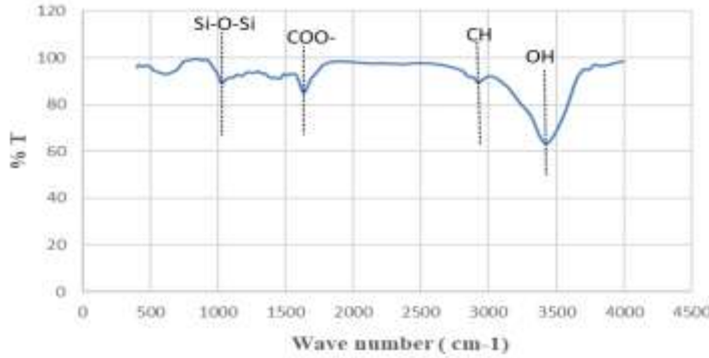

Fig.1. FTIR spectra of silica/corncob nanocomposite.

1.2 X-ray diffraction (XRD)

Figure (2) illustrates the XRD patterns of sorbent material. Peaks at 2Θ = 16.9°, 21.8° and 34.6° of silica/corncob nanocomposite are corresponding to (110), (200) and (004) planes of crystalline cellulose[21].

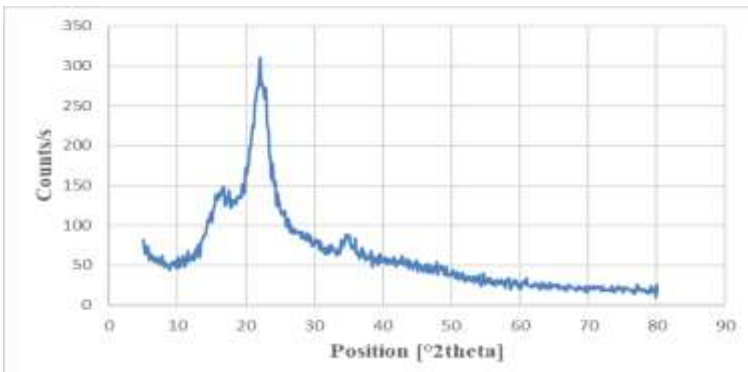

Fig.2.X-ray diffraction pattern of silica/corncob nanocomposite.

1.3 Scanning electron microscopy (SEM) for different nanocomposite

The surface morphology of silica/corncob nanocomposite (Fig. 3)), was characterized using scanning electron microscopy (SEM) with (EDX).In the SEM image of silica/corncob nanocomposite, spherical nature particles of nano silica are deposited on the surface of corncob structures. The presence of Si in the EDX spectra of silica/corncob nanocomposite (Fig. 3C)), confirmed the formation nanocomposite.

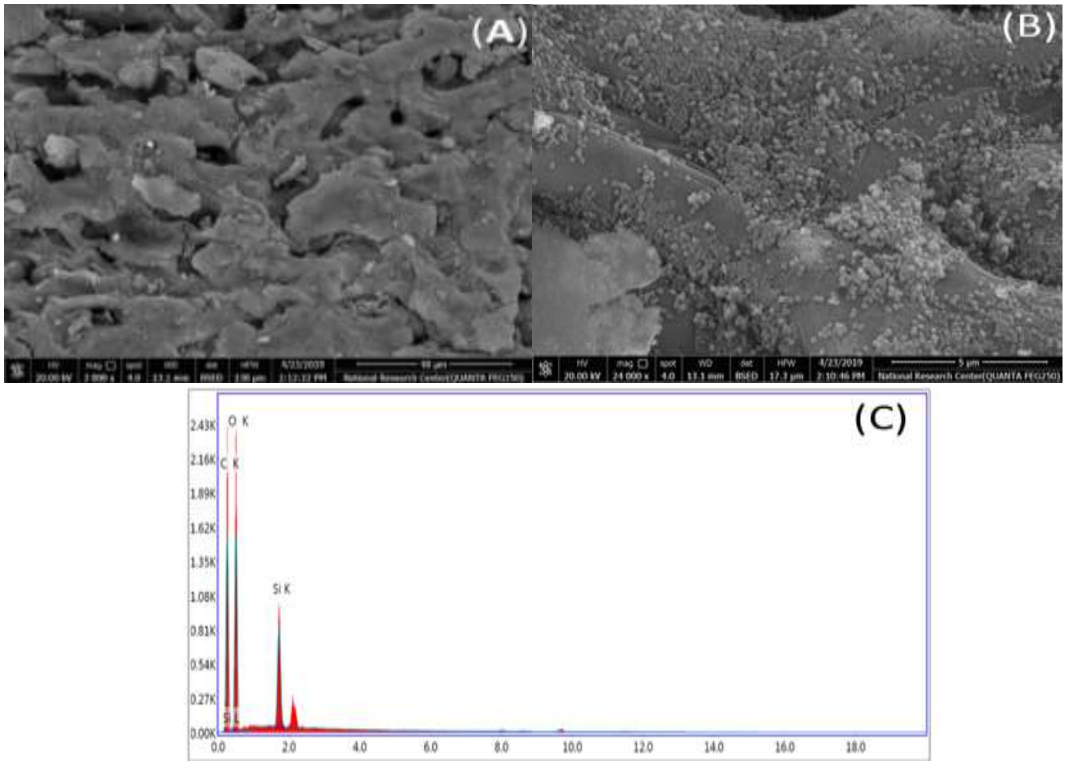

Fig. 3. SEM images of silica/corncob nanocomposite at low (A), higher (B) magnification, and EDX (C).

1.4 Characterization for silica/corncob nanocomposite using High-resolution transmission electron microscopy (HR-TEM)

Figure (4) explained the morphology of silica/corncob nanocomposite synthesized by sol–gel method using TEM imaging. Figure (4A) illustrated that the treated corncob formed nanofibrils network doped with silica nanoparticles. At higher magnification (Fig.4B), showed that the Nano-silica appeared spherical which matched with SEM and distributed well in nanofibrils network with no aggregation [22].

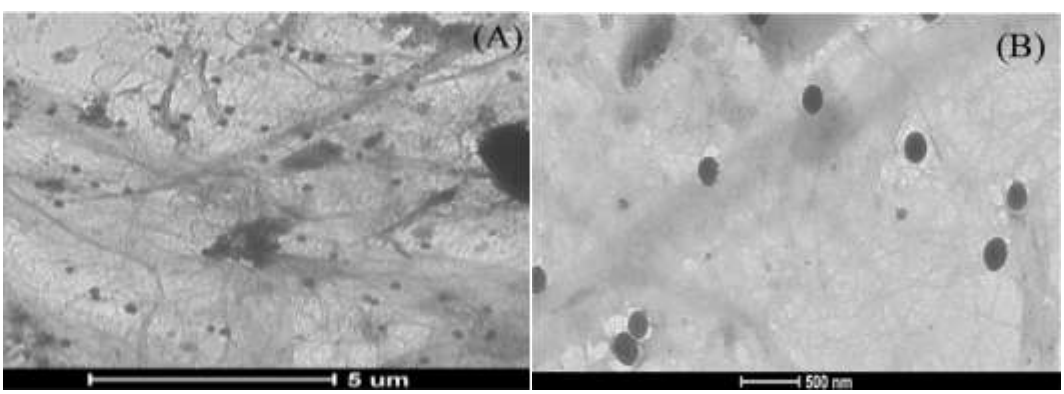

Fig. 4. TEM images for the obtained silica/corncob nanocomposite at low (A) and higher (B) magnification.

1.5.Determination of Zeta potential measurements for nanocomposite

The zeta potential and point of zero charge (PZC) for silica/corncob nanocomposite at pH range (3–11) were measured and presented in (Fig. 5) which revealed a PZC at pH of 3.8. This means that at pH lower than 3.8, silica/corncob nanocomposite will be positively charges and above pH of 8, it will carry a negative charge which enhance the removal of positively charged methylene blue[10].

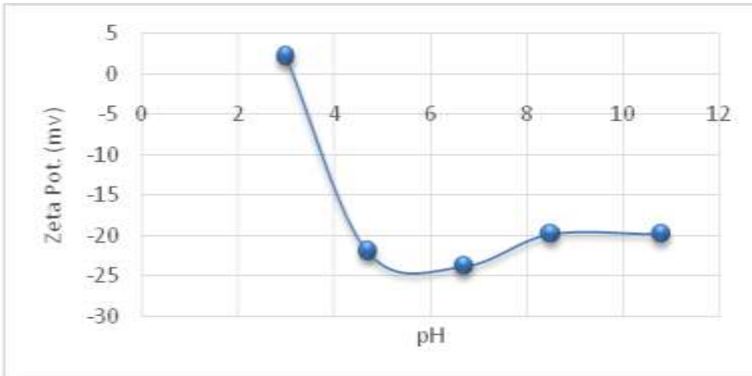

Fig. 5. Zeta-potential for silica/corncob nanocomposite suspensions at different pHs.

2. Sorption study

2.1Effect of contact time on the removal efficiency of silica/corncob nanocomposite

Figure (6) displaying the isotherm of methylene blue adsorption by silica/corncob nanocomposite at ambient temperature (20± 5ºC) and pH value 7.5. The data indicaterapid removal of dye within the first 5 min of adsorption before reaching equilibrium time. This may be due to the presence of efficient vacant sites in the initial of the reactionbut after this period, active sites became occupied [23].

In order to understand the kinetics of methylene blue sorption by silica/corncob nanocomposite, two kinetic models namely pseudo-first-order, and pseudo-second-order have been studied. The kinetic models constants and correlation coefficients were calculated and listed in the Table (1) which indicated that the kinetics of sorption reaction follows the pseudo-second-order model (Fig. 7)) due to the higher correlation coefficient (R^2 = 1) value and the matching between the experimental and calculated q_e.This may indicate that the removal process of methylene blue adsorption by silica/corncob nanocomposite was mainly controlled by the adsorption step [24].

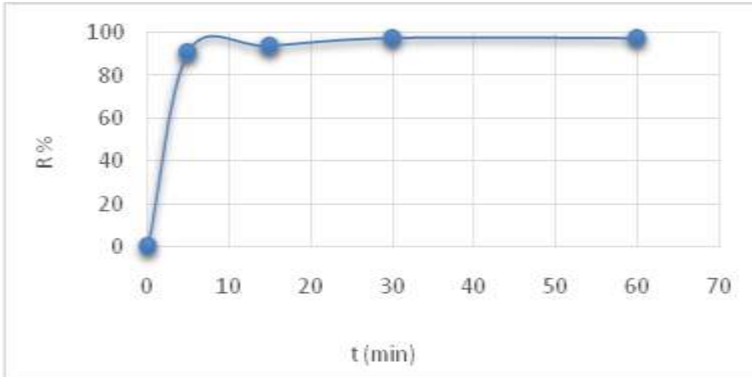

Fig. 6: Effect of contact time on the removal efficiency of methylene blue (pH= 7.5) by0.15 g silica/corncob nanocomposite

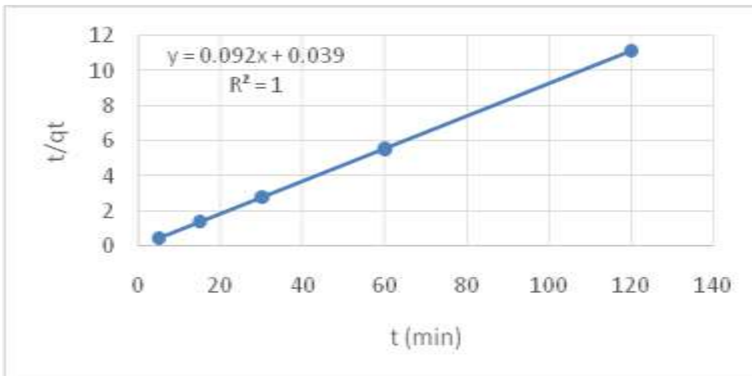

Fig. 7. Pseudo-second-orderfor the adsorption of methylene blue ontosilica/corncob nanocomposite.

Table 1:Constants of adsorption kinetics models for the removal of methylene blue onto silica/corncob nanocomposite.

Pseudo-first-order				Pseudo-second-order				
K_1 (min-1)	q_e (exp.) (mg/g)	q_e (cal.) (mg/g)	R^2	K_2 (g/mg min)	q_e (exp.) (mg/g)	q_e (cal.) (mg/g)	h (mg/g min)	R^2
0.097	10.787	1.38	0.9736	0.22	10.787	10.83	25.8	1

2.2Effect of methylene blue concentration on uptake efficiency of silica/corncob nanocomposite

As we know, the dye concentration may affect the sorption process. To evaluate this effect, a series of solutions containing different concentrations of methylene blue was prepared and the sorption efficiency and uptake of 0.2 g silica/corncob nanocomposite were estimated (Fig. 8).The data shows that as the initial concentration increase the removal efficiency decrease from 98 % to 25 % and this attributed to lacking sufficient active sites for adsorption[25]. On the opposite wise, the uptake increase from 4 mg/g to 55 mg/g with increasing concentration and this may be due to that the ratio between sorbent and sorbate is sufficient to providing a driving force to overcome the mass transfer resistance between them [26].

To discuss the reaction process between methylene blue and silica/corncob nanocomposite, two isotherm models namely Langmuir, and Freundlich have been evaluated. The isotherm models constants and correlation coefficients were calculated and listed in Table (2).The results indicate that the reaction of the sorption follows the Langmuir isotherm (Fig.9) due to the higher correlation coefficient (R^2 = 0.9903) value which assumes monolayer adsorption of the methylene blue onto active sites of the silica/corncob nanocomposite surface[27]. The value of n >1 in Freundlich indicating that the adsorption is a physical process[18].

R_L which is the essential characteristics of the Langmuir isotherm was found to be in the range from 0 to 1 (Fig. 10), suggesting favorable adsorption between methylene blue and silica/corncob nanocomposite.

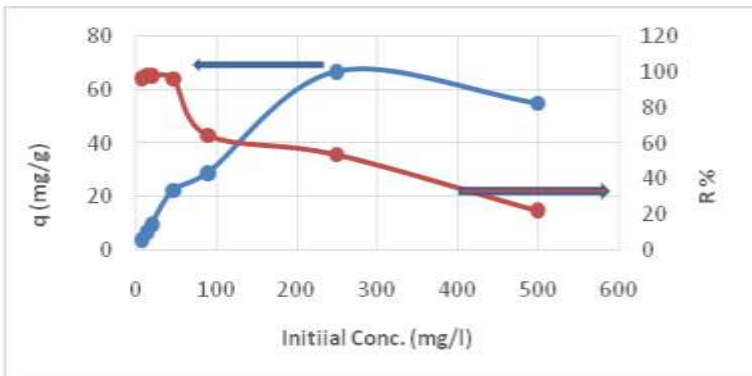

Fig.3.Effect of the methylene blue concentration on uptake efficiency of silica/corncob nanocomposite (adsorbent dose = 0.2 g, pH 7.5).

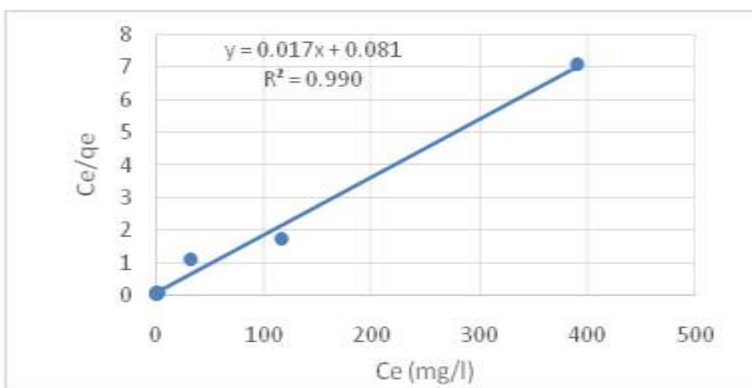

Fig. 9.Langmuirisotherm for the adsorption of methylene blue onto silica/corncob nanocomposite.

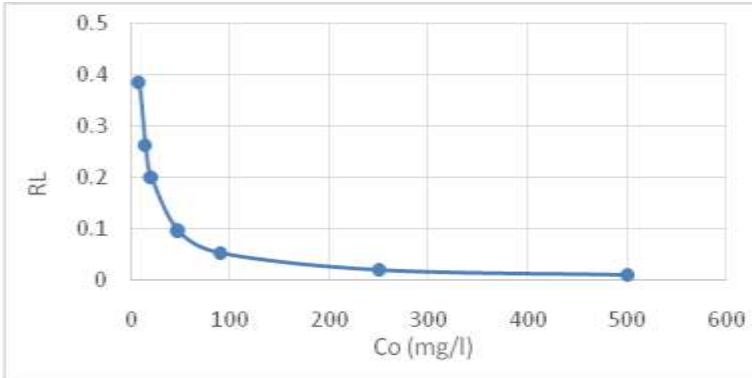

Fig. 10.Separation factor at different concentration of methylene blue.

Table 2: Constants of adsorption isotherms models for the removal of methylene blue onto silica/corncob nanocomposite.

Langmuir adsorption isotherm			Freundlich adsorption isotherm		
q_{max}(mg/g)	b (L/mg)	R^2	n	K_f	R^2
56	0.2	0.9903	3	10.5	0.8669

2.3Effect of pH on uptake efficiency of silica/corncob nanocomposite

One of the most important parameters which influence the removal of dye from wastewater is the pH. The sorption of methylene blue by 0.2 g of silica/corncob nanocomposite in 100 ml over the pH range of 2 to 11 was evaluated (Fig.11). The data illustrated that as pH increase the removal efficiency increase until equilibrium reaching and the optimum removal efficiency was obtained at pH near 7. This attributed to a competition between H^+ and positively charged groups of methylene blue at low pH which leave few sites available for methylene blue[28]. At high pH, the proton competition disappears and the silica/corncob nanocomposite gain a negative electrical charge that increases the attraction with methylene blue so the removal efficiency increase and this was very matched with zeta potential measurements[24].

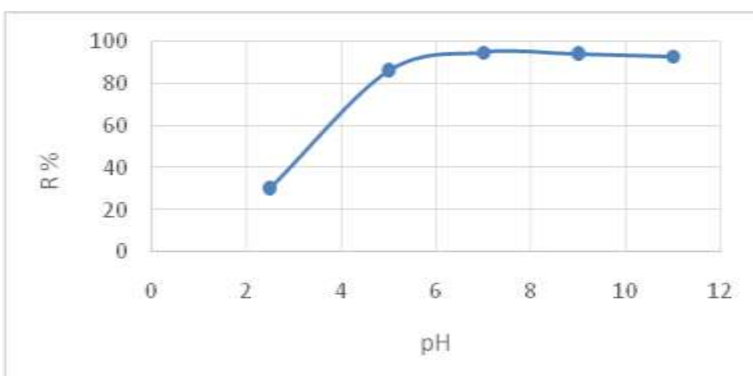

Fig. 11:Effect of pH on the removal of methylene blue by 0.2 g silica/corncob nanocomposite at 30 min.

2.4Effect of sorbent dosage on uptake efficiency of silica/corncob nanocomposite

Optimizing the sorbent dosage used in the sorption process is also important on the removal of dye from wastewater. Figure (12) illustrated the removal efficiency and the uptake of methylene blue by a different dose of silica/corncob nanocomposite. The results show that the removal efficiency increased from 20 % to 98.5 % with increasing the sorbent dosage from 0.01 g to 0.3 g before reaching equilibrium and this may be due to the increase of the active sites available for the methylene blue removal[29]. Although the removal efficiency of methylene blue increased, the uptake decreased from 43 mg/g to 7 mg/g with increasing the adsorbent dosage, this attributed to the overlapping of the adsorption sites which limits the availability of active sites during sorption [30].

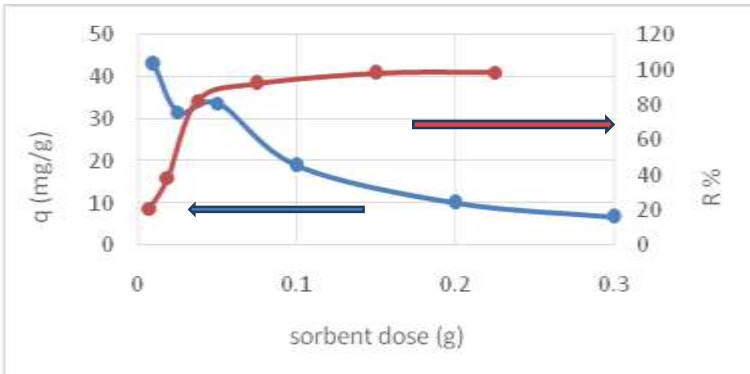

Fig. 12. Effect of adsorbent dosage of silica/corncob nanocomposite on removal efficiency and the uptake of methylene blue (pH=7.5).

Conclusion:

In this study silica/corncob nanocomposite was prepared for removal of methylene blue from aqueous solutions. The removal efficiency increased to 98 % and 93 % with sorbent dose and pH respectively, while decreased to 25 %with initial dye concentration. The higher correlation coefficient (R^2 = 1) indicated that the kinetics of the sorption reaction follows a pseudo-second-order model. The isothermal results indicate that the reaction of the sorption could be fitted to Langmuir isotherm due to the higher correlation coefficient (R^2= 0.9903).

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ازالة صبغة المثيلين الزرقاء من مياه الصرف باستخدام متراكب السليكا النانومتري

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المستخلص

تم تحضير متراكب السليكا النانومتري لإزالة صبغة المثيلين الزرقاء من مياه الصرف. وقد تم توصيفه كيميائيا وفيزيائيا وذلك بتحديد التركيب الكيمائي وشكل وحجم الجسيمات وشحناتها باستخدام أحدث الأجهزة المتخصصة في ذلك كاجهزة قياس الخواص الضوئية، جهاز حيود الأشعة السينية، الميكرسكوب إلالكتروني الماسح،و الميكرسكوب إلالكتروني النافذ. وكذلك تم تقييم قدرة المتراكب النانومتري لإدمصاص صبغة المثيلين الزرقاء تحت العديد من الظروف والعوامل المختلفة مثل: وقت التلامس بين المتراكب والصبغة، والتركيزات الأولية للصبغة، ودرجة الحموضة ، وكذلك كمية المواد الماصة المضافة لدراسة كفاءة الإزالة وقدرة للإدمصاص للمتراكب.تم قياس تركيزات صبغة المثيلين الزرقاء بواسطة مقياس الطيف الضوئي.

اظهرت النتائج ان كفاءة إزالة الصبغات عالية جدا باستخدام متراكب السليكا النانومتري حيث وصلت نسبة الازالة الي 98٪.