Biosorbentfrom Tomato Stems: Adsorption Properties Using Methylene Blue as Pollutant Test

ABSTRACT

Dye removal from wastewater using activated carbons was the subject of much scientific research in recent decades. However, high–quality and inexpensive adsorbents production using biomass has attracted more attention from researchers. In this study, tomato stem–based bio–adsorbent was prepared for methylene blue (MB) removal from aqueous medium using batch mode adsorption. Activated carbon performance for MB adsorption was evaluated by studying several parameters effect such as contact time, solution initial pH, dye solution initial concentration, temperature and adsorbent dose. High adsorption percentage of more than 80% of dye was observed with initial pH of 11, contact time of 120 min, adsorbent dose of 3 g L^{-1} and temperature of 60°C. Adsorption isotherm and kinetic models results predict that Langmuir isotherm as well as pseudo–second–order kinetic would better describe adsorption process. The maximum monolayer adsorption capacity obtained according to Langmuir model was 4.15 mg g^{-1}.

Keywords: *Tomato stem;Biosorbent; Activated carbon; Methylene blue; Adsorption; Modeling.*

1. INTRODUCTION

The current world population is estimated at more than 8 billion inhabitants while it was about 6 billion inhabitants in 2000. Based on this rapid growth, production plants in general, and particularly those of dyes, have increased day by day with technological progress in order to satisfy this population. However, wastewater resulting from extensive production of dyes presents dangers for aquatic environment due to the presence of acids, bases, organic, inorganic, dissolved and colored toxic solids (Harrache and Abbas, 2022). In recent years, water quality in the world has deteriorated considerably due to uncontrolled industrial discharges, intensive use of organic nitrogen compounds andwater resources disorderly exploitation.

Methylene blue (MB) is one of a large group of dye compounds that have many applications, including as dermatological agent, biological dye, veterinary medicine, to inhibit molds spread in poultry feed, fungi, and intestinal parasites (Nandi et al., 2009). MB is toxic when injected into humans and animals. In addition, commonly observed adverse effects of MB in humans are irritation of gastrointestinal tract that can lead to organ damage upon long–term exposure (Mittal et al., 2008; Kismir and Aroguz, 2011). Thus, MB concentration in industrial discharges must be strictly controlled by environmental protection authorities in order to minimize pollution of the latter.

Nowadays, several innovative and modern water treatment solutions were developed to manage hazardous contaminants discharge from wastewater. Several techniques such as coagulation, adsorption, solvent extraction, ion exchange, chemical precipitation, evaporation and membrane filtration technologies were used in wastewater treatment at different times (El-taweel et al., 2023; Allou et al., 2023; Mukhamediev and Bekchanov, 2019; Salman et al., 2022; Wang et al., 2024; Barat et al., 2009). All these methods are feasible on industrial scale, but among them, adsorption is economically viable approach that can simultaneously remove multiple contaminants using wide variety of low–cost adsorbents. Moreover, adsorption is an effective method, due to its characteristics of ease of use, environmental friendliness, cost–effectiveness, low health risk, sensitivity to harmful pollutants and non–destructive process (Reza et al. 2020). However, due to the high cost of activated carbons produced from non–renewable materials (coal and lignite), biomass wastes were targeted as precursor materials to produce activated carbon due to their abundance. In literature, various biomass materials were used to produce activated carbon for dye adsorption, such as *Lantana camara* stems

(Amuda et al. 2014), banana stems (Misran et al. 2022), *Cornulacamonacantha* stems (Sharma et al. 2019), *Catha edulis* stems (Abate et al. 2020), and cassava stems (Sulaiman et al. 2021).

Tomato is one of the vegetables produced in abundance in Côte d'Ivoire with an annual production of around 34,000 tons. Thus, large quantity of tomato stems is generated each year. However, after harvesting, farmers burn these stems, which constitute waste for them. However, high carbon content of tomato stems makes them good candidate for conversion into activated carbon. This study therefore aims to study the possibility of preparing activated carbon derived from tomato stems using zinc chloride ($ZnCl_2$) as activating agent. To this end, after materials synthesis, determining parametersstudy indicating adsorbent effectiveness to remove methylene blue, such as contact time, adsorbent concentration, pH, initial pollutant concentration and temperature was carried out.

2. MATERIALS AND METHODS

2.1. Materials and chemicals used

Tomato stems were collected from farmers after they had finished their harvest. Zinc chloride ($ZnCl_2$), hydrochloric acid (HCl), sodium hydroxide (NaOH) and methylene blue ($C_{16}H_{18}ClN_3S$) were chemicals used in this study and were used without any purification. Distilled water was used as solvent forsolutions preparation. **Figure 1** and **Table 1** give structural methylene blue molecule representation and its physicochemical properties, respectively.

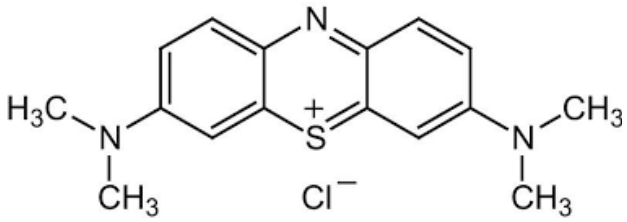

Fig. 1. Chemical structure of methylene blue (MB).

Table 1. Some physicochemical properties of methylene blue.

Characteristic	Indication
Molecular Formula	$C_{16}H_{18}ClN_3S$
Molar mass (g mol^{-1})	319.85
Dye family	Basic
Solubility in water at 20°C (g L^{-1})	20
Wavelength (nm)	663

2.2. Activated carbon based on tomato stems preparation

Tomato stems were cut into small pieces and then sun–dried until relatively dry stems were obtained. These stems were washed with distilled water to remove all impurities and sun–dried again for 72 h. Approximately 100 g of tomato stems were oven–dried during 24h at 110 °C. The dry stems were ground to obtain tomato stems powder. Tomato stems powder impregnation was done according to the work described by Jabar et al. (2022) with small modifications. 50 g of tomato stems powder was introduced into 500 mL of $ZnCl_2$ solution (30% wt) and heated at 70 °C for 5h with continuous stirring. Subsequently, powder thus impregnated is left to stand at laboratory temperature for 19 hours, then filtered and dried for 24 hours at 50 °C. Tomato stems powder was then subjected to carbonization by increasing temperature to 700 °C for 1 hour with heating rate of 10 °C min^{-1} under an inert atmosphere. The product obtained is washed with 0.5M HCl followed by washing with distilled water until water in the filtrate is neutral. Final product is then dried at 110 °C in oven for 24 hours and was coded TSAC.

2.3. Methylene blue adsorption studies

Adsorbent Samples (from 0.2 to 5 g L^{-1}) were brought into contact with certain volume of dye aqueous solutions at different initial concentrations (from 5 to 120 mg L^{-1}), various initial pH (from 2 to 13) at fixe temperature (from 29 to 60 °C), and subjected to mechanical stirring of approximately 250 rpm and samples aqueous phase were taken and filtered as time function for adsorption efficiency determination. Sample absorbance was measured using UV–visible spectrophotometer at the wavelength λ_{max} of 663 nm.In order to determine the point zero charge pH (pH_{pzc}), after each experiment on pH effect, the suspension was filtered and the final pH value was recorded. Then, the

plot of pH variation (difference between initial and final pH) versus initial pH, gives pH_{pzc}, at which there is equality between initial and final pH.Depending on the desired initial pH value, HCl (0.1M) or NaOH (0.1M) solution was added (few drops) to the solution.MB removal percentage, adsorption capacities at each time t (q_t) and at equilibrium (q_e), they were calculated using equations (1), (2) and (3), respectively.

$$\% \, MB \, removed = \frac{C_i - C_t}{C_i} \times 100 \quad (1)$$

$$q_t = \frac{C_i - C_t}{W} \times V \quad (2)$$

$$q_e = \frac{C_i - C_e}{W} \times V \quad (3)$$

2.4. Kinetics and isotherm modeling

Improving adsorbent derived from biomass performance necessarily requires knowledge of factors to be optimized, hence adsorption kinetics and isotherm modeling. Therefore, in this work, pseudo–first–order, pseudo–second–order, intraparticle diffusion and Elovich kinetics models on the one hand, and on the other hand Langmuir, Freundlich and Temkin adsorption isotherm models were applied to experimental data obtained. **Table 2** underlines the linear equation, plot as well as parameters to be calculated from the slop and intercept of thelinear plot. The major factor in Langmuir model indicating that the process is favorable,named separation factor (R_L), is calculated using equation (5).$C_0$is pollutant initial concentration expressed in mg L^{-1}.

$$R_L = 1/(1 + k_L C_0) \quad (5)$$

Table 2. Kinetics and isotherm models used equation, plot and parameter to be calculated (Allou et al., 2018; Tran et al., 2017).

	Model	Equation	Plot	parameter
Kinetic	Pseudo–first–order	$ln(q_e - q_t) = lnq_e - k_1 t$	$ln(q_e - q_t)$*vs.*t	k_1, $q_{e,cal}$
	Pseudo–second–order	$t/q_t = 1/k_2 q_e^2 + t/q_e$	t/q_t*vs.*t	k_2, $q_{e,cal}$
	Intraparticle diffusion	$q_t = k_{id} t^{1/2} + C$	q_t*vs.*$t^{1/2}$	k_{id}, C
	Elovich	$q_t = ln\,(\alpha\beta)/\beta + ln\,t/\beta$	q_t*vs.* $ln\,t$	α, β

Isotherm	Langmuir	$1/q_e = 1/q_m + 1/(q_m C_e k_L)$	$1/q_e$ vs. $1/C_e$	q_m, k_L, R_L
	Freundlich	$lnq_e = lnk_F + lnC_e \times 1/n$	lnq_e vs. lnC_e	k_F, n
	Temkin	$q_e = BlnA + BlnC_e$	q_e vs. lnC_e	A, B

3. RESULTS AND DISCUSSION

Physicochemical parameters influence such as contact time, adsorbent mass, solution pH, initial dye concentration and temperature was evaluated in this study. Different models application for adsorption experimental results description of kinetics and isotherms made it possible to specify validity and especially limits of models application in order to generate adsorption database in terms of adsorption capacities.

3.1. Contact time effect

Contact time between adsorbent and pollutant remains by far one of the critical factors affecting adsorption. Thus, MB removal percentage from aqueous medium using TSAC was measured at different contact times and experimental data are presented in **Figure 2**. This figure mainly indicates three major phases during MB adsorption. During the first five minutes, removal percentage increases rapidly reaching 45%. This rapid adsorption phase could be explained by active sites abundance and solute concentration gradient elevation. The second phase (between 5 and 120 min), on the other hand, indicates moderate increase in MB removal percentage due to active adsorption sites reduced availability. The last phase (from 120 min), marked by the lack of unoccupied active sites, indicates that adsorption equilibrium is reached (Gouamid et al., 2013) with maximum adsorption percentage around 90%. Therefore, the optimal time for MB adsorption on TSAC is 120 min.

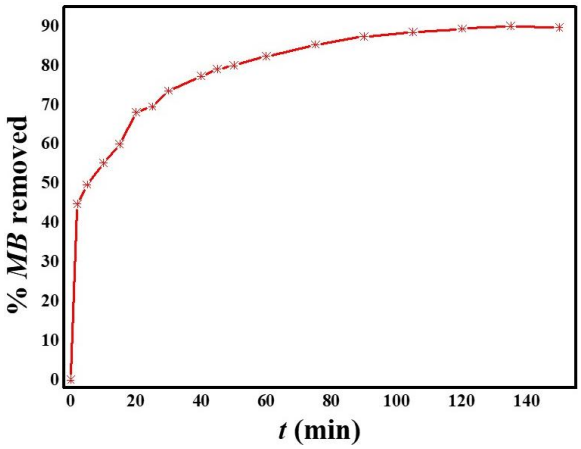

Fig. 2. Influence of contact time on MB removal from aqueous medium using TSAC.

3.2. Adsorption kinetics modeling

Adsorption kinetics would provide reaction mechanisms better understanding during adsorption (Abbasi et al., 2023). In this study, pseudo–first–order, pseudo–second–order, intraparticle diffusion and Elovich models, which are widely used in environmental applications, were chosen. Adsorption parameters were evaluated from the slope and intercept values of the kinetic model plots as shown in **Figure 3**. **Table 3** summarizes the calculated adsorption kinetics parameter data. The conformity between experimental data and expected values of a well–defined model was indicated by correlation coefficient. From **Table 3**, it can be noticed thatpseudo–second–order model correlation coefficient, which is 0.998, is much larger than those of the other models studied. Moreover, adsorption capacity ($q_{e,cal}$) calculated value is much closer to experimental value ($q_{e,exp}$). Therefore, MB adsorption on TSAC would be better suited to pseudo–second–order model which reflects chemical adsorption process governed by electrostatic attractive forces at adsorbent surface(Revellame et al., 2020).

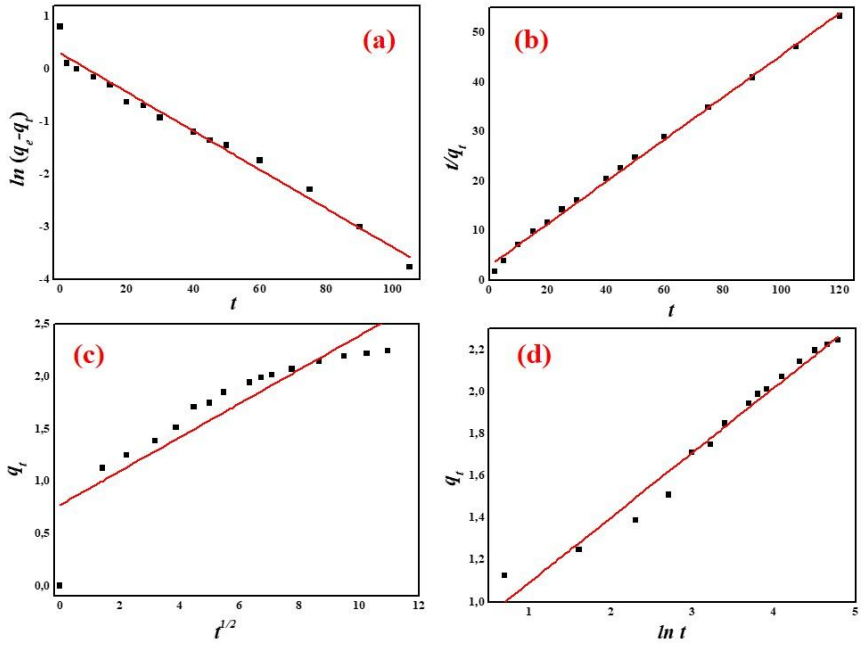

Fig. 3. Adsorption kinetics curve for MB removal from aqueous medium using TSAC: (a) pseudo–first–order, (b) pseudo–second–order, (c) intraparticle diffusion and (d) Elovich.

Table 3. Adsorption kinetics parameter data for MB removal from aqueous medium using TSAC.

Pseudo–first–order			Pseudo–second–order			Experimental data
$q_{e,cal}$ (mg g^{-1})	k_1 (min^{-1})	R^2	$q_{e,cal}$ (mg g^{-1})	k_2 (g mg^{-1} min^{-1})	R^2	$q_{e,exp}$ (mg g^{-1})
1.362	–0.037	0.978	2.347	0.062	0.998	2.248
Intra particular Diffusion			Elovich			
k_{di} (mg g^{-1} min$^{-1/2}$)	C (mg g^{-1})	R^2	α (mg g^{-1} min^{-1})	β (g mg^{-1})	R^2	
0.162	0.774	0.808	0.677	3.244	0.975	

3.3. Activated carbon mass effect

It is obvious that adsorbent dose has undeniable impact on pollutant removal at given initial concentration. Adsorption process is a phenomenon linked to mass transfer. However, adsorbent excessive use leads to unnecessary expenses making adsorbent/adsorbate contact less attractive. In **Figure 4**, TSAC dose influence on MB removal is shown. As indicated in the latter, MB adsorbed percentage increases from 7 to 88% by varying TSACmass from 0.01 to 0.25 g for 50 ml of pollutant. It emerges from this observation that MB removal percentage increases when active sites number oradsorbent dose increases. However, from the optimal dose, there is no significant change in adsorbed percentage. Beyond the optimal adsorbent dose, active sites overlap limits access to them (Basu et al., 2018), thus reducingadsorbed MB percentage. As result, an optimal adsorbent dose of 3 g L^{-1} was retained in the rest of this work.

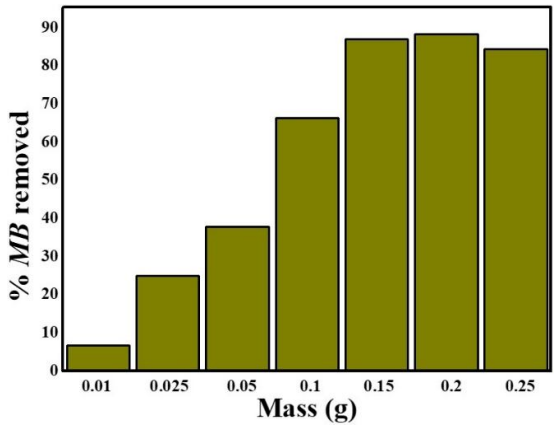

Fig. 4. Effect of TSAC mass on MB removal from aqueous medium.

3.4. Surface charge analysis and pH effect

Point zero charge pH (pH_{pzc}) is good indicator of chemical and electronic properties of functional groups on adsorbent surface (Khandaker et al., 2018). Indeed, when pH_{pzc}> pH then adsorbent surface is positively charged while it is negatively charged when pH_{pzc}<pH.**Figure 5(a)** presents result obtained for pH at point zero charge. pH_{pzc} value obtained is 9.51 thus suggesting that the prepared TSAC surface has basic character. Therefore, when solution pH is lower than pH_{pzc}, the functional groups on TSAC surface are protonated by H^+ protons excess in solution. On the other hand, solution pH is higher than pH_{pzc}, functional groups TSAC surface will be deprotonated by the presence of OH^- ions in the solution. Therefore, adsorbent will be attractive to any positively charged pollutant and thus will promote cationic dye adsorption by increasing electrostatic forces between TSAC negative charge and MB positive charge. Thus MB adsorption would be much more favored for pH above 9.51.

Due to its ability to dissociate functional groups from biosorbent surface, pH plays crucial role in adsorption process. **Figure 5(b)** shows pH influence on adsorption process. It was observed that by increasing pH from 2.05 to 13.18, MB adsorbed percentage increased from 18% to 81% indicating improvement in the electrostatic attraction between MB cations and TSAC surface when moving from acidic to basic medium. Low adsorption in acidic medium could be explained by H_3O^+ ions abundance competing with MB cations to bind to TSAC active sites. On the other hand, in basic medium, active sites deprotonation promotes better MB cations adsorption. Moreover, better MB adsorption is observed at pH values beyond point zero chargepH.

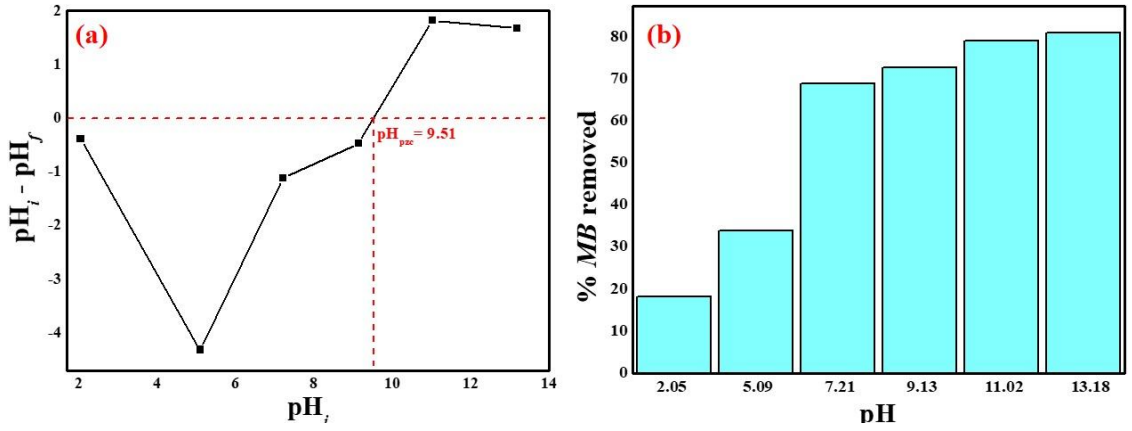

Fig. 5. Point zero charge curve (a) and pH influence (b) on MB removal from aqueous medium using TSAC.

3.5. Initial dye concentration effect

Initial dye concentration influences dye adsorption performance, which is related to process occurring on adsorbent surface. Thus, initial MB concentration effect on removal percentage is shown in **Figure 6**. As shown in this figure, when initial dye concentration increases from 2.5 to 25 mg L^{-1}, removal percentage decreases from 96 to 76%. This observation is thought to be due to the fact that high initial dye concentration causes an overload of available adsorbent sites, resulting in large amount of dye in solution.

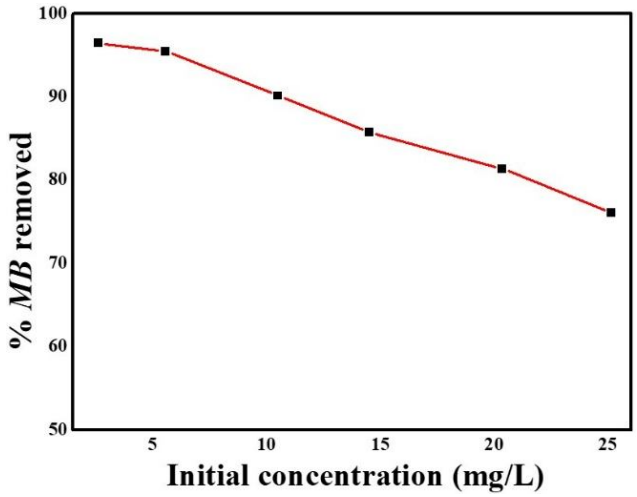

Fig. 6. Initial concentration influence on MB removal from aqueous medium using TSAC.

3.6. Adsorption isotherm modeling

Nature and mechanism of the interaction between MB and TSAC surface can be explained through adsorption isotherm study. In this study, equilibrium data were analyzed by Langmuir, Freundlich, and Temkin isotherm models. **Figure 7** shows Langmuir, Freundlich, and Temkin isotherms linear plot, and adsorption isotherms parameter values are reported in **Table 4**. Langmuir isotherm correlation coefficient (R^2) is 0.9942, much larger than those of Freundlich and Temkin which are 0.9845 and 0.9675, respectively. Therefore, Langmuir model would be better suited to describe MB adsorption on the prepared TSAC. Furthermore, considering separation factor calculated values ($0 < R_L < 1$), adsorption process is favorable. This observation would reflect the fact that MB fixation is carried out in monolayer on energetically homogeneous adsorption sites and without interaction between dye adsorbed cations (Postai et al., 2016). Furthermore, $n$calculated value, much higher than 1, indicates that adsorption phenomenon is dominated by chemisorption.

Table 5 lists, according to literature, MB removal maximum Langmuir adsorption capacities using various low–cost adsorbents. From this table, it is seen that TSAC has much higher maximum adsorption capacity than many other adsorbents, indicating that it could be promising adsorbent for dye removal from solution.

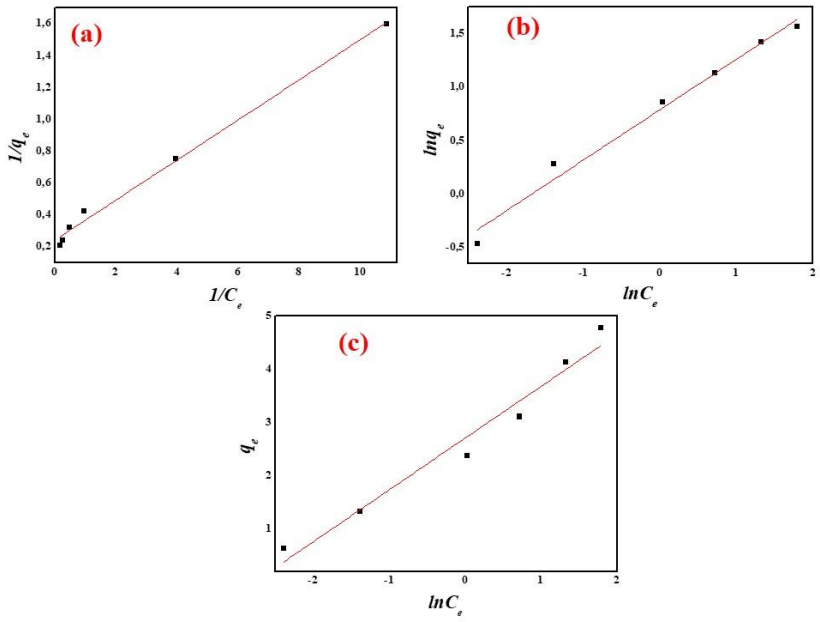

Fig. 7. Adsorption isotherm curve of MB removal from aqueous medium using TSAC: (a) Langmuir, (b) Freundlich and (c) Temkin.

Table 4. Adsorption isotherm parameter data of MB removal from aqueous medium using TSAC.

Isotherm model	Parameters	Values
Langmuir	$q_{max,cal}$ (mg.g^{-1})	4.1528
	k_L (L mg^{-1})	1.9157
	R_L	0.0203–0.1674
	R^2	0.9942
Freundlich	k_F (mg$^{1-1/n}$ L$^{1/n}$ g^{-1})	2.2021
	n	2.1204
	R^2	0.9845
Temkin	A	16.0534
	B	2577.3229
	R^2	0.9675

Table 5. Maximum MB adsorption capacity of Langmuir model from some low–cost adsorbents.

Adsorbent	Adsorption capacity(mg g^{-1})	Reference
Activated carbon derived from corn cobs	0.84	Tseng et al., 2006
Activated carbon derived from fir wood	1.21	Wu and Tseng, 2008
Zeolite/CeO_2nanocomposite	2.51	Nyankson et al., 2020
Fe_3O_4/activated carbon/ cyclodextrin/ sodium alginate	2.079	Yadav et al., 2020
Alginate–graftedpolyacrylonitrilebeads	3.51	Salisua et al., 2015
TSAC	4.15	This study

3.7. Temperature effect

Figure 8 shows temperature impact on MB adsorption using TSAC by varying it from 29 to 60°C. The results show that MB adsorption percentage increases with temperature from 86 to 98%. This observation would be due to MB molecules rapid mobility to interact with TSAC free active sites as temperature increases. In addition, increase in adsorption efficiency would suggest adsorption process endothermic nature.

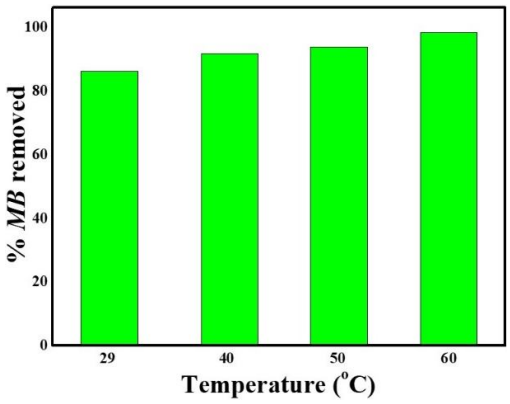

Fig. 8. Temperature influence on MB removal from aqueous medium using TSAC.

4. CONCLUSION

This study, which falls within wastewater decontamination framework, aimed to study activated carbon from tomato stems preparation possibility by chemical activation and to verify its effectiveness in methylene blue adsorption process. Study to identify the conditions (mass of the adsorbent, initial pH of the solution, concentration of the pollutant, temperature and adsorbate/adsorbent contact time) ensuring methylene blue adsorption better efficiency on activated carbon was carried out.

Study of factors influencing the adsorption process shows that dye retention by the support is rapid during the first five minutes of contact followed by more or less moderate retention until plateau is reached which corresponds to equilibrium after 120 min, which is relative to support active sites availability. The activated carbon optimal mass beyond which there is adsorption sites overlap is 3 g L^{-1}. Point zero charge pH being 9.51, adsorption was better at pH higher than the latter and mainly from 11. Given MB molecules rapid mobility to interact with free TSAC active sites as temperature increases, adsorption was much more important at high temperature. MB adsorption on TSAC isotherm is best described by Langmuir model with maximum adsorption capacity of 4.15 mg g^{-1} while adsorption kinetics obeys to pseudo–second–order kinetic model.

This internship project indicates that activated carbon from tomato stems could be used as an adsorbent for methylene blue adsorption from aqueous solution. It was noted that methylene blue adsorption potential from aqueous solution was affected by various independent variables. However, in order to offer these activated carbons to industrial customers, further studies should be carried out,

including in–depth characterization tests, adsorbent regeneration after use, and adsorption tests on real solutions containing several organic pollutants.

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COMPETING INTERESTS

Authors declared that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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DEFINITIONS, ACRONYMS, ABBREVIATIONS

C_e **(mg L^{-1}):** equilibrium concentration

C_i**(mg L^{-1}):** initial concentration

C_t**(mg L^{-1}):** concentration at time t

k_F**(mg$^{1-1/n}$ L$^{1/n}$ g^{-1}):** Freundlich constants related to adsorption capacity

k_L **(L mg^{-1}):** Langmuir constant

k_{id}**(mg g^{-1} min$^{-1/2}$):**intraparticle diffusion rate

k_1**(min^{-1}):**pseudo–first–order rate constant

k_2**(g mg^{-1} min^{-1}):** pseudo–second–order rate constant

q_e**(mg g^{-1}):** amount adsorbed at equilibrium

q_m **(mg g^{-1}):** maximum monolayer adsorption capacity

q_t**(mg g^{-1}):**amount adsorbed at time t

MB: methylene blue

TSAC: tomato stem activated carbon

A **(L g^{-1}):** Temkin equilibrium binding constant

$\boldsymbol{B}$**:** Temkin constant related to adsorption heat and $B = RT/b$

$\boldsymbol{C}$ **(mg g^{-1}):** constant indicating boundary layer thickness

$\boldsymbol{R}$ **(8.314 J mol^{-1} K^{-1}):**gas constant.

$\boldsymbol{T}$ **(K):**absolutetemperature

$\boldsymbol{V}$ **(L):**pollutant solution volume

$\boldsymbol{W}$ **(g):** adsorbent mass.

$\boldsymbol{b}$ **(J mol^{-1}):** Temkin constant

$\boldsymbol{n}$**:**Freundlich constants related to adsorption intensity

$\boldsymbol{\alpha}$ **(mg g^{-1} min^{-1}):** initial adsorption rate

$\boldsymbol{\beta}$**(g mg^{-1}):** related to the extent of surface coverage