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Removal of methylene blue, bromocresol green and methyl red dyes from aqueous solutions by adsorption using *Bryophyllum pinnatum* (*Lam.*) *kurz* stem powder and its activated carbon.

Kindala T.J.[1*], Kayembe S.J.[2], Kifuani K.A.[2], Ilinga L.B.[2], Taba K.M.[1]

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Abstract

Adsorption of Methylene Blue (MB), Bromocresol Green (BG) and Methyl Red (MR) were carried out in batch on a low cost treated adsorbent, *Bryophyllum pinnatum (Lam.) Kurz* stem powder (BPP) and its activated carbon (ACBP). Adsorption capacities of the two adsorbents were compared to that of commercial activated carbon (CAC) and were also evaluated as function of pH and dose of adsorbent. Freundlich and Langmuir isotherms models were used to test the equilibrium data which were best represented with maximum monolayer adsorptions capacities of 255.754 (pH 11.15), 253.807 (pH 3.35) and 195.313 mg.g^{-1} (pH 3.25) for MB, MR and BG respectively on ACBP. On CAC their capacities were respectively 176.367 (pH 11.15), 342.466 (pH 3.35) and 108.342 mg.g^{-1} (pH 3.25) for MB, MR and BG; whereas, on BPP the adsorption capacities were respectively 61.013 (pH 11.15), 165.289 (pH 3.35) and 51.733 mg.g^{-1} (pH 3.25) for MB, MR and BG. The linear correlation coefficient R^2 was used to elucidate the best fitting isotherm model ($R^2 \approx 0.98$). The separation factor R_L values between 0.03 and 0.95 indicate a favorable adsorption. The adsorption kinetics showed that this process could be well described with the pseudo-second order model. The results have shown that, ACBP has excellent adsorption capacity compared to the values of BPP or with CAC. The equilibrium data were best represented by the used isotherms models of Langmuir and Freundlich.

[1] *Laboratoire de Chimie Organique et Energétique, Faculté des Sciences, Université de Kinshasa, B.P. 190 Kinshasa XI, Kinshasa, D. R. Congo.*
[2] *Faculté des Sciences, Université de Kinshasa, B.P. 190 Kinshasa XI, Kinshasa, D.R. Congo*

* To whom correspondence should be addressed: juniorkind@yahoo.fr

INTRODUCTION

Water pollution is a major problem in the global context and has even been suggested to be the leading cause of death and disease worldwide, especially in developing countries [ANONYMOUS, 2010; VERMA, 2010].

High production and use of dyes generate colored waste waters that pollute rivers; their presence in surface water blocks solar radiation from reaching aquatic organisms, thus affecting negatively the balance of aquatic ecosystems. In addition, the release of dyes into water streams may result in formation of toxic and carcinogenic degradation products. Hence, the removal of dyes from industrial effluent is an important and necessary process [BUNHU, 2011; LONGHINOTTI, 1998; PATIL *et al.*, 2011]. Discoloration of wastewater has become one of the major issues in wastewater treatment as many industries use dyes to color products, such as textiles, rubber, paper, plastics, leather, cosmetics, food and minerals [BINTI, 2004].

The most important technologies for organic pollutants elimination from water and wastewater are: chemical precipitation, electroflotation, reverses osmosis, adsorption, ion-exchange, low temperature

vacuum distillation, etc. [ADEKOLA *et al.,* 2005]. Adsorption is the most interesting from the point of view of large-scale application, simple technology and cost effectiveness. Adsorption has the additional advantages of applicability at very low concentrations, suitability for using batch and continuous processes, ease of operation, little sludge generation, possibility of regeneration and re-use of adsorbents and low capital cost [BUNHU, 2011; JADHAD *et al.,* 2004; JAYARAJ *et al.,* 2012; MAGHRI *et al.,* 2012; MECHEBBEK *et al.,* 2009; TSAI et al., 2009].

Many studies [GARCIA *et al.,* 2013; KAFIA, 2011; JIWALAK, 2010; SHRADDHA *et al.,* 2012; TAHER *et al.,* 2007] were carried out on adsorption by low cost alternative materials such as coconut shell, tropical grass shells, rice husk, sesame, pine bark to remove dyes from wastewater; instead of activated carbon (granular or powdered) which is the traditionally used adsorbent, and has been successfully applied for the removal of both heavy metals and organic pollutants from contaminated water. Activated carbon has attractive properties such as large specific surface, micro-porosity, and high adsorption capacity. However, its usage is limited due to high cost in terms of both capital and regeneration. The above factors and others have lent impetus to the continuing search for better; and low cost adsorbing materials [BAHADIR, 2004; BUNHU, 2011; JADHAD *et al.,* 2004].

Therefore, many efforts have been made to investigate the use of various low cost organic adsorbents easily available. These materials are derived from natural resources, agricultural wastes or industrial by-products such as peat, wood, barley and rice husk, sawdust, biomass, ... etc., most of them are cellulose based and can be used without any previous thermal or chemical treatment [JIWALAK, 2010; TAHER *et al.,* 2007].

Bryophyllum pinnatum (Crassulaceae), a common plant in D.R. CONGO which growths along rivers and in marshy places everywhere in the country, has been reported to possess many biological activities and finds applications in folk medicine [RUQAIYAH *et al.,* 2012; QUAZI *et al.,* 2011 ; UFELLE *et al.,* 2011].

The aim of this study was to evaluate the potential of *Bryophyllum pinnatum*'s stem powder as adsorbent in the removal of basic dye (Methylene blue: MB) and anionic dyes (Bromocresol green: BG and Methyl red: MR) from aqueous solutions. The effect of various parameters (contact time, amount of adsorbent, concentration and pH of dyes solutions) were investigated in batch experiments.

MATERIAL AND METHODS

Preparation of Adsorbents

Bryophyllum pinnatum is a small plant that was collected from the surrounding area of Kinshasa (N'djili Brasserie) in January 2014. The plant material was identified and authenticated in the Herbarium/Faculty of Sciences, University of Kinshasa. The samples (stem of this plant) were dried in an ALPHA I-4 LSC lyophilizer instrument. The dried material was powdered and impregnated with phosphoric acid 40 % for an hour (One advantage of using phosphoric acid in chemical activation is that, it can be cleaned from the activated carbon by rinsing with boiling pure water), and oven dried at 60°C. After drying, one part of stem powder was used directly in adsorption experiments as BPP adsorbent. In order to obtain activated carbon from this material, the other part of stem powder was carbonized at 800°C in a TDW sx-2.5-10 furnace; so prepared activated carbon was named ACBP (activated carbon of *Bryophyllum pinnatum*). The commercial activated carbon (CAC) was from Merck Co., USA.

Solutions and characterization of Dyes

Methylene blue (MB), methyl red (MR) and Bromocresol green (BG) used as adsorbates in this study were obtained from Merck Co., USA. Solutions of dyes (10^{-5}- 32.10^{-6}mol/L) were prepared in distilled water. The residual concentration of each dye was measured spectrophotometrically with CMC APL 02260187 UV-VIS spectrophotometer instrument.

Batch experiments

Adsorption of dyes was carried out by shaking (with THZ-92 C Desktop thermostatic oscillator) at 250 rpm, an amount of each adsorbent (10 – 100 mg) in 250 mL (*in a 500 mL Erlenmeyer flask*) aqueous solutions of dyes (10^{-5} – 32.10^{-6} mol/L) for different agitation times (2 to 700 minutes) at room temperature (25°C). For each dye sample, pH was maintained in an acidic or basic medium. After agitation, samples (BG and MR) were filtered (*Fisherbrand, Dia/Sise: 320mm filter paper*) to avoid turbidity interference. After investigation, the result showed that the filter paper adsorbs1.57% and 3.1% of MR dye at acidic and basic pH respectively. It also adsorbs 4.25% and 8.0% of BG at acidic and basic pH respectively. Results in this study were obtained after deduction of these percentages from experimental data. For Methylene Blue, after agitation time, adsorbent was removed by centrifugation at 2500 rpm and the supernatant was analyzed spectrophotometrically to determine its residual concentration. In both cases, the percentage of dye removal (R) was calculated using equation (1)

$$R(\%) = (Co\text{-}Ct).100/Co \qquad (1)$$

Where **Co** is the initial concentration of dye (mol/L), **Ct** (mol/L) is the instant concentration of dye at time **t** [KIFUANI *et al.*, 2012].

RESULTS AND DISCUSSION

Effect of initial dye concentration and contact time on the adsorption in acid and basic pH

The adsorption of MB, MR and BG at the initial dye concentration in acidic and basic pH was investigated as a function of contact time in order to determine the equilibrium time for a maximum adsorption and results are presented in **Figure 4-6**. It was found that the rate of dye adsorption in acidic and basic pH increased quickly until equilibrium was reached (**Table I**). The higher adsorption rate at the beginning of the process could be correlated to the large number of vacant adsorption sites available on the adsorbents surface (**Figure 1-3**).

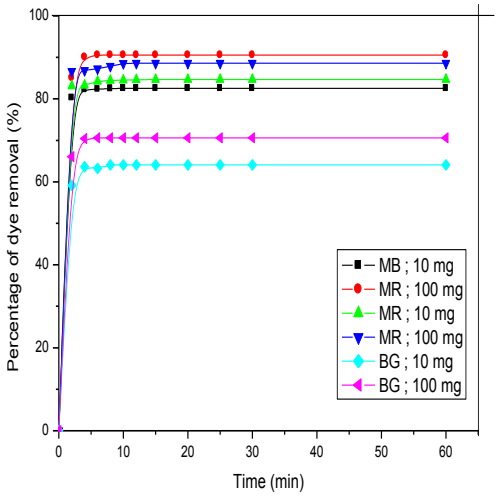

Figure 1. Effect of adsorbent dose of ACBP (MB in alkaline pH; MR and BG in acidic pH

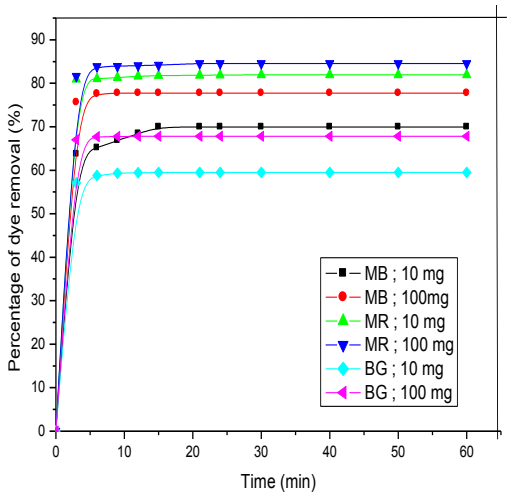

Figure 2. Effect of adsorbent dose of CAC (MB in alkaline pH; MR and BG in acidic pH)

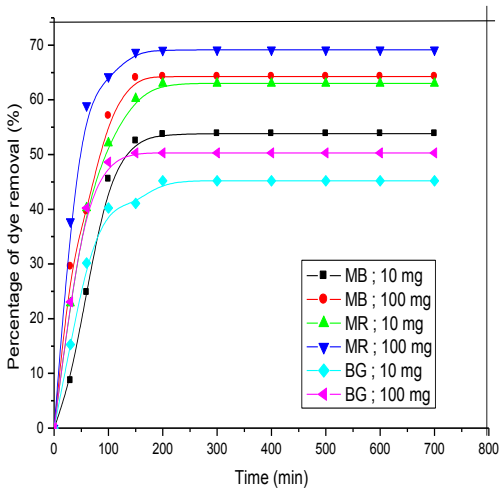

Figure 3. Effect of adsorbent dose of BPP (MB in alkaline pH; MR and BG in acidic pH)

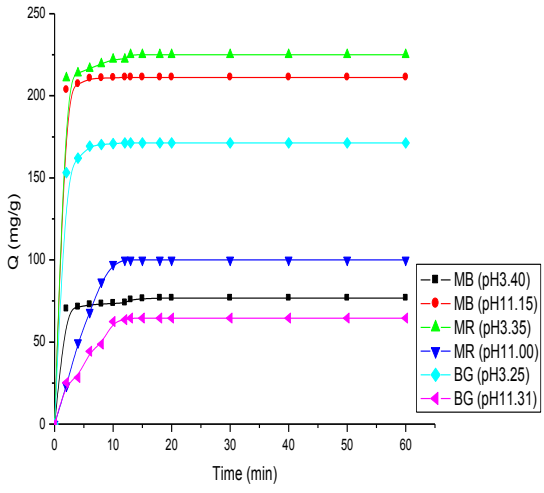

Figure 4. Dye adsorbed per mass unit of ACBP as a function of contact time (Co:32µmol/L and mACBP:10µg)

The effect of pH on adsorption of MB, MR and BG was investigated thoroughly. The results are shown in **Figures 4-6**, and it appears that adsorption of MB (a basic dye) on ACBP and on CAC was better in alkaline medium than in acidic (**Figures 4-6**). On the other hand, adsorption of MR and BG (anionic dyes) on ACBP and on CAC were better in acidic medium. Similar results were obtained by Azhar et al [2005], who removed the dye

from aqueous solution by using adsorption on treated sugarcane bagasse.

It appears that a change in pH of the solution results in the formation of different ionic species and surface charge. If the pH of the dye in solution is greater than pH_{PZC}, (pH_{PZC}: 7.30 for ACBP) the negative charge increase in the surface, and the uptake of basic dye increased but the uptake of the anionic dyes decreased. The increase or decrease of uptake is due to the attractive or repulsive electrostatic effect [AL-DEGS *et al.*, 2005].

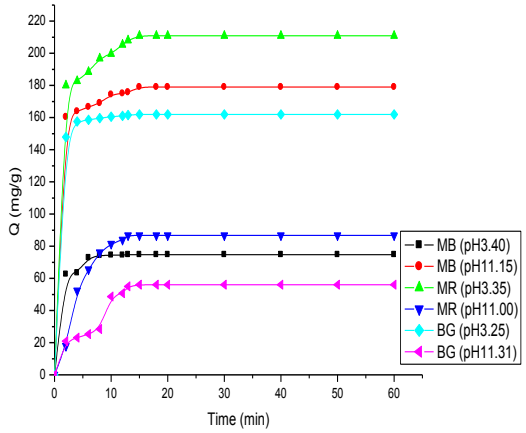

Figure 5. Dye adsorbed per mass unit of CAC as a function of contact time (Co:32µmol/L and mCAC:10mg)

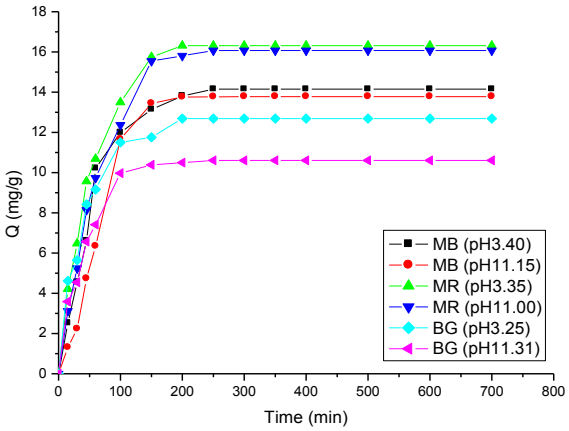

Figure 6. Dye adsorbed per mass unit of BPP as a function of contact time (Co:32µmol/L and mBPP:100mg)

For BPP absorbent, there is a slight difference in absorption of dyes (MB, MR, BG) in both acidic and alkaline media; which indicates that the electrostatic mechanism is not the only mechanism of dye adsorption. The adsorbent may also interact with the dye molecules through the hydrogen bonding or hydrophobic-hydrophobic mechanism [ABDELBAKI, 2010].

Table I. Equilibrium contact time (t_e) and the Equilibrium concentration (Q_e)

Dye	pH	ACBP		CAC		BPP	
		t_e (min)	Q_e (mg/g)	t_e (min)	Q_e (mg/g)	t_e (min)	Q_e (mg/g)
MB	3.40	20	76.77	13	74.69	300	14.16
	11.15	15	211.21	15	178.98	300	13.78
MR	3.35	13	224.96	18	210.89	200	16.31
	11.00	12	99.97	13	86.69	250	16.08
BG	3.25	12	171.30	15	162.02	200	12.68
	11.31	13	64.54	15	56.06	250	10.60

The adsorption of MB, MR and BG onto the adsorbents was investigated in varying the adsorbent amount for 32.10^{-6}mol/L of dye concentration. The adsorption yield increased with an increase in adsorbent amount (**Figure 1-3**). This was attributed to increasing of surface area and availability of more adsorption sites [ARIVOLI *et al.*, 2009].

Adsorption Kinetics

In order to examine the mechanism of adsorption process, pseudo first order and pseudo second order equations were used to test the experimental data. **Tables II-IV** list results of rate constant and other kinetic parameters studies by pseudo first order and pseudo second order models. The correlation coefficient (R^2), for the pseudo second order model showed value close to unity, suggesting that the dye adsorption process occurred predominantly by the pseudo second order adsorption mechanism.

Adsorption Isotherm

Many adsorption isotherms models have been used to describe experimental data such Langmuir and Freundlich models which are the most used [FASSI *et al.*, 2012; KARINE, 1999]. In this work, both models were used to elucidate the adsorption of MB, MR and BG onto

the different adsorbents and find out the concentration of adsorbates at equilibrium. The Langmuir isotherm is represented by equation (2):

$$\frac{1}{Q_e} = \frac{1}{Q_m.K_L.C_e} + \frac{1}{Q_m} \qquad (2)$$

Where C_e is the equilibrium concentration (mol/L), Q_e the amount adsorbed at equilibrium (mg/g), Q_m the adsorption efficiency and K_L is the Langmuir constants. The values of Q_m and K_L were determined from slope and intercepts of the plots and are given in **Tables V-VII**. The essential characteristics of the Langmuir isotherm can be expressed in terms of dimensionless constant separation factor R_L, defined as :

$$R_L = \frac{1}{1+K_L C_0} \qquad (3)$$

Where C_o is the initial dye concentration. The value of R_L indicates the type of isotherm which can be either unfavorable (R_L>1), linear (R_L=1), favorable (0 < R_L< 1) or irreversible (R_L=1) [SYAFALNI *et al.*, 2012].

The Freundlich equation used is shown in equation (4):

$$\ln Q_e = \ln K_f + \frac{1}{n_f} \ln. C_e \qquad (4)$$

Where Q_e is the amount of dye adsorbed (mg/g), C_e is the equilibrium concentration of dye in solution (mol/L) and K_f and n are constants incorporating the factors affecting the adsorption capacity and intensity of adsorption, respectively. These parameters can be calculated from the intercept and the slope of the linear plot. The values of K_f (mg/g)(mol/L)-1/n and nf are given in the **Tables V-VII** [ARIVOLI *et al.*, 2009].

Table II. Pseudo first and second order parameters for the adsorption of MB

		Pseudo First Order			Pseudo Second Order		
		Qe (mg/g)	k_1 (min^{-1})	R^2	Qe (mg/g)	k_2 ($g.mg^{-1}.min$)	R^2
ACBP	MB (pH3.40)	31.72	0.28	0.93	77.34	0.04	1.00
	MB (pH11.15)	49.34	0.54	0.96	211.42	0.15	1.00
CAC	MB (pH3.40)	53.95	0.53	0.98	75.08	0.06	1.00
	MB (pH11.15)	64.41	0.25	0.91	180.18	0.02	1.00
BPP	MB (pH3.40)	21.79	0.026	0.95	15.43	0.00	1.00
	MB (pH11.15)	26.69	0.03	0.98	16.62	0.00	1.00

Table III. Pseudo first-second order parameters for the adsorption of MR

		Pseudo First Order			Pseudo Second Order		
		Qe (mg/g)	k_1 (min^{-1})	R^2	Qe (mg/g)	k_2 ($g.mg^{-1}.min$)	R^2
ACBP	BG (pH3.25)	92.853	0.56812	0.97813	171.821	0.06253	0.99999
	BG (pH11.31)	100.600	0.34074	0.93710	68.120	0.66431	0.99677
CAC	BG (pH3.25)	46.654	0.36045	0.93196	162.338	0.05780	1.00000
	BG (pH11.31)	84.506	0.25378	0.88705	61.881	0.00378	0.98954
BPP	BG (pH3.25)	10.861	0.01831	0.97483	13.268	0.00343	0.99922
	BG (pH11.31)	10.902	0.02436	0.99005	11.121	0.00391	0.99889

Table IV. Pseudo first-second order parameters for the adsorption of BG

		Pseudo First Order			Pseudo Second Order		
		Qe (mg/g)	k_1 (min^{-1})	R^2	Qe (mg/g)	k_2 ($g.mg^{-1}.min$)	R^2
ACBP	MR (pH3.35)	22.026	0.18058	0.97749	225.734	0.02998	0.99999
	MR (pH11.00)	151.340	0.34134	0.94533	106.270	0.00385	0.99481
CAC	MR (pH3.35)	105.905	0.25938	0.94333	213.220	0.01023	0.99993
	MR (pH11.00)	109.289	0.29976	0.99418	91.912	0.00467	0.99430
BPP	MR (pH3.35)	18.1663	0.02155	0.98876	17.376	0.00187	0.99843
	MR (pH11.00)	19.972	0.02163	0.98580	17.547	0.00131	0.99690

Tables V-VII show Langmuir and Freundlich isotherm parameters for the adsorption capacities at 25°C. These adsorption isotherms showed good fitting results based on the high correlation coefficient (R^2) indicating good adsorption of MB, MR and BG onto ACBP, CAC and BPP. The values of n > 1 (Freundlich constant) obtained represent a favorable adsorption condition [FASSI *et al.*, 2012]. To confirm the favorability of the adsorption process, the separation factor (RL) was calculated and presented in **Tables V-VII**, the values were found between 0 and 1 confirms that adsorption process was much favorable [BACCAR, 2013].

Table V. Langmuir and Freundlich isotherm model parameters and correlation coefficients for adsorption of Methylene Blue at 25°C

		Langmuir				Freundlich			
Adsorbent	pH	Q_m (mg/g)	$K_L.10^{-4}$ (L/mol)	R_L	R^2	1/n	n	$K_f.10^{-3}$	R^2
ACBP	3.40	149.031	2.13694	0.59-0.82	0.89692	0.95578	1.04627	1500.456	0.89415
	11.15	255.754	43.71489	0.06-0.18	0.98713	0.53845	1.85718	131.835	0.99275
CAC	3.40	487.805	0.47786	0.86-0.95	0.92046	1.12936	0.88546	9384.876	0.91346
	11.15	176.367	81.27599	0.03-0.10	0.97755	0.35005	2.85673	9.920	0.99877
BPP	3.40	106.157	1.09028	0.74-0.90	0.98915	0.87185	1.14699	235.091	0.98683
	11.15	61.013	2.00367	0.60-0.83	0.98328	0.80077	1.24880	100.557	0.98073

Table VI. Langmuir and Freundlich isotherm model parameters and correlation coefficients for adsorption of Methyl Red at 25°C

		Langmuir				Freundlich			
Adsorbent	pH	Q_m (mg/g)	$K_L.10^{-4}$ (L/mol)	R_L	R^2	1/n	n	$K_f.10^{-3}$	R^2
ACBP	3.35	253.807	57.25604	0.05-0.14	0.98607	0.51928	1.92574	116.415	0.99534
	11.00	98.912	30.61308	0.09-024	0.89470	0.33831	2.95587	3.563	0.95058
CAC	3.35	342.466	25.14142	0.11-0.28	0.99533	0.63341	1.57876	450.498	0.99665
	11.00	128.205	7.57282	0.29-0.56	0.97880	0.58420	1.71174	44.695	0.98739
BPP	3.35	165.289	0.99018	0.75-0.90	0.98222	0.86900	1.15075	319.454	0.98090
	11.00	92.081	1.75444	0.64-0.85	0.97867	0.83995	1.19055	212.883	0.98115

Table VII. Langmuir and Freundlich isotherm model parameters and correlation coefficients for adsorption of Bromocresol Green at 25°C

		Langmuir				Freundlich			
Adsorbent	pH	Q_m (mg/g)	$K_L.10^{-4}$ (L/mol)	R_L	R^2	1/n	n	$K_f.10^{-3}$	R^2
ACBP	3.25	195.313	27.99681	0.10-0.26	0.97857	0.48696	2.05356	41.553	0.98570
	11.31	59.524	101.01557	0.03-0.09	0.82368	0.11840	8.44595	0.207	0.92247
CAC	3.25	-108.342	-5.59394	-8.41-9.52	0.94034	1.60529	0.62294	13927344	0.97113
	11.31	71.685	11.43443	0.21-0.46	0.99483	0.42223	2.36838	4.817	0.99736
BPP	3.25	51.733	2.03260	0.60-0.83	0.98211	0.78572	1.27272	72.811	0.97786
	11.31	32.321	2.52365	0.55-0.79	0.97505	0.72812	1.37340	28.146	0.96553

CONCLUSION

The adsorption of MB, MR and BG dyes using *Bryophyllum Pinnatum* (*Lam.*) *Kurz* powder (BPP) and its activated carbon powder (ACBP) was investigated. The results have shown that, ACBP has excellent adsorption capacity compared with the values of BPP or with CAC.

The results obtained with Langmuir and Freundlich isotherm models provided the best correlation constant. The adsorption kinetics was found to follow a pseudo second order kinetic model with the correlation coefficient close to unity.

The adsorbent surface characterization experiments are undergoing; the results will be published in the next paper.

RESUME

Absorption de la solution aqueuse du bleu de methylene, du vert du bromocrésol et du méthyl rouge sur la poudre et le carbone active des tiges du *Bryophyllum pinnatum* (Lam.) Kurz

L'adsorption du Bleu de Méthylène (MB), du Vert de Bromocrésol (BG) et du Méthyle Rouge (MR) a été effectuée sur un adsorbant à faible coût, la poudre des tiges de Bryophyllum pinnatum (Lam.) Kurz (BPP) et son charbon actif (ACBP). Les capacités d'adsorption de ces deux adsorbants ont été comparées à celle du charbon actif commercial (CAC) et ont été également évaluées en fonction du pH et de la dose d'adsorbant. Des modèles d'isothermes de Freundlich et de Langmuir ont été employés pour examiner les données d'équilibre qui sont mieux représentées par une adsorption en monocouche.

Les capacités d'adsorption ont été respectivement de 255.754 (pH 11.15), 253.807 (pH 3.35) et 195.313 mg.g -1 (pH 3.25) pour MB, MR et le BG sur ACBP. Sur CAC ces capacités étaient respectivement 176.367 (pH 11.15), 342.466 (pH 3.35) et 108.342 mg.g -1 (pH 3.25) pour MB, MR et BG; alors que, sur BPP les capacités d'adsorption étaient respectivement 61.013 (pH 11.15), 165.289 (pH 3.35) et 51.733 mg.g -1 (pH 3.25) pour MB, MR et le BG.

Le coefficient de corrélation linéaire R2 a été employé pour élucider le meilleur modèle d'isotherme (R2≈0.98). Les valeurs du facteur RL de séparation comprises entre 0.03 et 0.95 indiquent une adsorption favorable. La cinétique d'adsorption a prouvé que ce processus pourrait être mieux décrit avec le modèle de pseudo-second ordre. Les résultats ont montré qu'ACBP a une excellente capacité d'adsorption comparativement aux valeurs enregistrées avec BPP ou CAC.

Les données d'équilibre ont été mieux représentées par les modèles utilisés d'isothermes de Langmuir et Freundlich. La cinétique d'adsorption s'est avérée suivre un modèle de pseudo-second ordre avec un coefficient de corrélation proche de l'unité.

Mots clés : *Isotherme d'Adsorption, Bleu de Méthylène; Rouge de Méthyle, Vert de Bromocrésol, tige de Bryophyllum pinnatum, Charbon Actif*

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