Methyl parathion pesticide removal from aqueous solution using Senegal River *Typha australis*

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ABSTRACT

The objective of this study is to investigate the sorption of methyl parathion (MP) pesticide from aqueous solution using *Typha australis* leaf powder as an alternative low cost biosorbent. The effects of various parameters such as adsorbent dosage and contact time are studied using the batch technique. Kinetics models (pseudo-first and pseudo-second orders) and isotherms models (Langmuir and Freundlich) are used to fit the experimental data by linear and non-linear methods. The results show that the pseudo-second order kinetics model is the best for describing the adsorption of MP by *Typha australis* for all initial MP concentrations. The equilibrium data fit well with the Langmuir model, suggesting the existence of monolayer adsorption of MP. *Typha australis* leaf powder can be considered as a new useful low cost natural biosorbent for pesticide clean up operations in aquatic systems.

Key words | low cost sorbent, methyl parathion, pesticide, *Typha australis*

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NOTATION

C	equilibrium pesticide concentration in solution
ΔG	free Gibbs energy
K	model coefficient
n	exponent
q	adsorbed amount of pesticide
t	time
y	data point value
$\bar{y}$	average value of all data points

L	Langmuir
max	maximum
MP	methyl parathion
PAC	powdered activated carbon
pr	prediction given by the model
TA	*Typha australis*

GREEK LETTERS

χ	determination coefficient

SUBSCRIPT

eq	equilibrium
F	Freundlich

INTRODUCTION

Pesticides are chemicals, which are commonly used in agriculture to protect crops from pest organisms. Methyl parathion (MP) (O, O-dimethyl O-(4-nitrophenyl) phosphorothioate) is an agricultural insecticide and acaricide. MP is an organophosphorus compound classified as highly toxic (class I) by the United States Environmental Protection Agency (USEPA) and extremely toxic (category Ia) by the World Health Organization (WHO) (Chávez- López *et al.*

2011). Some toxicological studies have showed that MP may appear as a pollutant in water sources and could threaten human health (Zhu *et al.* 2001).

There are many procedures available for MP removal from water, which include microbiological (Rani & Lalitha-kumari 1994), electrochemical (Alves *et al.* 2013), degradation (Pankaj & Parag 2012) and adsorption (Akhtar *et al.* 2007). In recent years, various natural adsorbents such as agricultural wastes including *Rhizopus oryzae* biomass (Chatterjee *et al.* 2010), bagasse fly ash (Gupta *et al.* 2002) and waste jute fiber carbon (Senthilkumaar *et al.* 2010) have been tried to achieve effective removal of pesticides. Some species of *Typha* are investigated for pollutants removal in aqueous media (Abdel-Ghani *et al.* 2009; Hegazy *et al.* 2011).

The focus of this research is to explore the possibility of using *Typha australis*, an abundant and available plant along the Senegal River, for removing MP from aqueous solution. The sorption of MP is found to be a spontaneous process ($\Delta G < 0$) and, depending on used adsorbent, this process may be endothermic (Memon *et al.* 2008) or exothermic (Aktar *et al.* 2007). The effects of various experimental factors such as adsorbent dose, initial concentration, and contact time are studied using the batch technique. For this purpose, kinetic and isotherm studies have been carried out with the linear and non-linear methods.

MATERIALS AND METHODS

Collection, preparation and characterization

Biomass of *Typha australis* growing along the Senegal River was collected from the city of Rosso, Wilaya of Trarza, in Mauritania. Before use, *Typha australis* leaves were washed with ultrapure water to remove dirt. The biomass was then air dried for 10 days followed by drying in an oven at 70°C for 24 h (Abdel-Ghani *et al.* 2009). The dried biomass was ground, sieved (<100 μm) and stored in a dessicator before use.

The point of zero charge (pH$_{pzc}$) of the *Typha australis* leaf powder adsorbent was carried out (Roudani *et al.* 2014) and the surface area was obtained using Sears method (Yadav *et al.* 2011).

For determination of the moisture content, 5 g of the material was weighed in a crucible. This was placed in the oven and heated for 5 h at a constant temperature of 105°C. The sample was then removed and put rapidly into a desiccator in order to prevent any moisture uptake from atmosphere. The difference in the mass constitutes the amount of moisture content of the adsorbent (Dada *et al.* 2013).

Concerning the determination of ash, this allows knowing the part of the mineral in the adsorbent used. 1 g of dry adsorbent was weighed and introduced into a crucible for calcination. The crucible was then placed in an oven at 600°C for 45 min. After cooling, the crucible was weighed again (Hamdaoui & Naffrechoux 2007).

The bulk density was determined using a picnometer (Moyo *et al.* 2013), and the surface acidic functional groups and basic sites of the *Typha australis* leaf powder were determined by the acid-base titration method proposed by Boehm (Evangelin *et al.* 2012).

Adsorbate preparation and analysis

All chemicals used in this study were of analytical reagent grade. A stock solution containing 1,000 mgL^{-1} of MP was prepared by dissolving 100 mg of MP in 100 mL of methanol. The MP molecule is about 9 Å radius with a quite heterogeneous surface charge distribution, as can be seen in Figure 1. MP solutions were prepared by diluting stock solution of MP to the desired concentrations in ultrapure water. All samples were filtered using a micro filter syringer and analyzed by High Performance Liquid Chromatography with a Diode-Array Detector (HPLC/DAD) using the ultimate 3000 system of Thermo Scientific with Chromeleon 7 Software. Ultra pure water (pH 6.8) and methanol (20:80 V/V) were used as a mobile phase at a flow rate of 0.5 mL min^{-1} at a selected wave length of 265 nm.

Batch experiments

MP batch adsorption using *Typha australis* as adsorbent was conducted in batch experiments.

In all sets of experiments, fixed concentrations of MP (5 mg L^{-1} and 10 mg L^{-1}) were stirred (70 rpm) with varying adsorbent doses for different time periods. The effects of process conditions and contact time (5–60 min) were
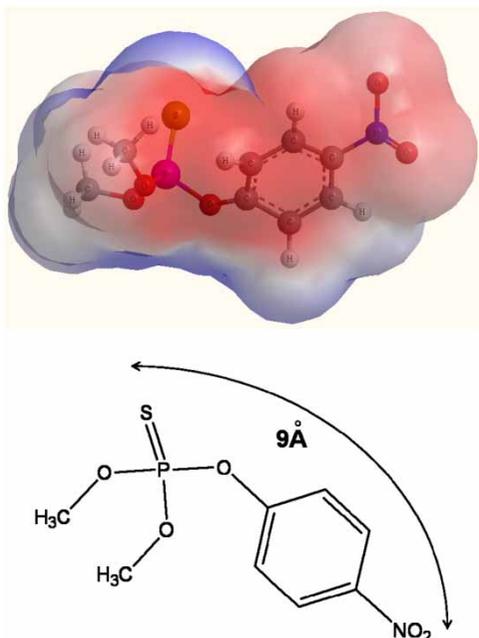
Figure 1 | MP chemical structure and solvent accessible potential surface distribution based on Extended Huckel Charge calculation.

evaluated for *Typha australis* adsorbent. At the end of each experiment the stirred solution mixture was microfiltered, and the residual concentration of MP was determined chromatographically. The amount of MP adsorbed (mg g^{-1}) at any time is computed using the following equation:

$$q_e = \frac{(C_i - C_e)V}{m}$$

The percentage of the removal of MP concentration in solution is calculated using the equation:

$$\text{Removal (\%)} = \frac{C_i - C_e}{C_i} \times 100$$

where q_e is the MP concentration in adsorbent (mg g^{-1}), V is the volume of the solution (mL), C_i and C_e are the initial and equilibrium solution concentrations (mg L^{-1}) and m is the mass of adsorbent (g). All experiments are conducted in triplicate and the mean values have been reported.

Kinetics and equilibrium adsorption modeling

The kinetic study was done for the *Typha australis* adsorbent in order to estimate the equilibrium time of adsorption and the best fitted pseudo-first order (PFO) and pseudo-second order (PSO) kinetic models. Different concentrations of MP (5 and 10 mgL^{-1}) spiked in an ultrapure water sample (25 mL) were taken in a conical flask and an adsorbent mass of 0.5 g of *Typha australis* was added. The mixtures were stirred at 70 rpm at pH 6.8 for 60 min at ambient temperature. At the end of each experiment, the stirred solution mixture was microfiltered and the residual concentration of MP was analyzed by HPLC/DAD.

The linear and non-linear mathematical expressions of PFO and PSO models are summarized in Table 1.

Batch isotherm experiments were conducted at different initial concentrations ranging from 5 to 100 mg L^{-1} taken in each conical flask. The adsorbent solution was stirred at ambient temperature at 70 rpm for a definite time period keeping initial pH at 6.8. At the end of each experiment, the stirred solution mixture was microfiltered and the residual concentration of MP was determined. The data were fitted to Langmuir and Freundlich isotherms to find the best fitted isotherm.

The linear and non-linear mathematical expressions of Langmuir and Freundlich models are summarized in Table 2.

Chi-square (χ^2) analysis is used to fit experimental data with kinetic and isotherm using the Excel® solver. The χ^2 value, relating the goodness of fit, is determined by:

$$\chi^2 = 100 \left(1 - \frac{\|y - y_{pr}\|^2}{\|y - \bar{y}\|^2} \right)$$

So $\chi^2 \leq 100$ – the closer the value is to the number 100, the more perfect is the fit.

RESULTS AND DISCUSSION

Characterization of *Typha australis* leaf powder adsorbent

The characterization of physical and chemical surface properties of adsorbent is one of the most important issues in an adsorption process because it evaluates its suitability for one or more of the application fields (Kumar *et al.* 2014).

The analysis of *Typha australis* leaf powder with physical and chemical properties is listed in Table 3.

Table 1 | Mathematical models of the kinetic adsorption models

Model	Equation	Parameter and dimension
PFO	$q_t = q_e(1 - \exp^{-k_1 t})$ (non linear form)	k_1, k_2: rate constant (min^{-1})
	$\log(q_e - q) = \log(q_e) - \dfrac{k_1}{2.30} t$ (linear form)	q_e: solute adsorbed at equilibrium state (mg g^{-1})
PSO	$q_t = \dfrac{k_2 q_e^2 t}{1 + k_2 q_e t}$ (non linear form)	q: solute adsorbed at any time (mg g^{-1})
	$\dfrac{t}{q} = \dfrac{1}{k_2 q_e^2} + \dfrac{t}{q_e}$ (linear form)	t: time (min)

Table 2 | Mathematical equation of the isotherm models

Model	Equation	Linear expression	Parameter and dimension
Langmuir isotherm	$q = q_{max} \dfrac{K_L . C_e}{1 + K_L . C_e}$	$\dfrac{C_e}{q_e} = \dfrac{C_e}{q_m} + \dfrac{1}{K_L q_m}$	q_{max}: maximum solute adsorbed at equilibrium state (mg g^{-1}) K_L: Langmuir coefficient $(\text{mg L}^{-1})^{-1}$
Freundlich isotherm	$q = K_F . C_e^{1/n}$	$\log q_e = \log K_F + \dfrac{1}{n} \log C_e$	K_F: Freundlich coefficient $(\text{mg g}^{-1}).(\text{mg L}^{-1})^{-n}$ n: model exponent (–)

Table 3 | Characteristics of *Typha australis* adsorbent

Parameters	Mean
pH_{pzc}	6.36
Moisture (%)	3.9
Bulk density (g mL^{-1})	0.48
Surface area $(\text{m}^2 \text{g}^{-1})$	0.91
Ash (%)	9.9
Loss of mass ignition (%)	16.9
Total surface acidity (mequiv/g)	0.744
Carboxyl (meq g^{-1})	0.380
Lactone (meq g^{-1})	0.340
Phenolic (meq g^{-1})	0.024
Total surface basicity (meq g^{-1})	0.376
Particle size (µm)	<100

To determine the surface functional groups of raw *Typha* leaf powder, its Fourier transform infrared (FTIR) spectrum is obtained as shown in Figure 2.

The broad band in the spectrum lies in the region of 3358.46 cm^{-1}, due to –OH stretching mode and shows the presence of free and bonded hydroxyl groups of cellulose.

The small peak observed at $2,913 \text{ cm}^{-1}$ denotes the presence of the stretching C–H vibration in the quinone group. Peaks at 1728.04 cm^{-1} (C $=$ O stretching of COOH) and 1638.14 cm^{-1} are assigned to C $=$ O (amide band/ OH). The band at 1519.64 cm^{-1} is assigned to C $=$ C stretching vibration of cycloalkenes. The absorption peaks at 1376.62 cm^{-1} and 1245.86 cm^{-1} can be attributed to the presence of (CH_2 and CH_3) and –C–O stretching, respectively. The peak lying in the region of $1,037 \text{ cm}^{-1}$ shows the presence of Si-O-Si linkages.

Effect of adsorbent mass

Biomass dosage is an important parameter in adsorption studies, as it gives the optimum dose at which maximum adsorption occurs.

The effect of the amount of adsorbent on the efficiency of adsorption was also studied. Variation of doses in the range 0.1–1 g at a fixed MP concentration (5 mg L^{-1}) for MP removal by *Typha australis* is shown in Figure 3.

The results suggest that the increase in the dose of adsorbent results in an increase in adsorption, probably due to increase in the retention surface area. However, further increase after a certain dose does not improve the adsorption; perhaps due to the interference between binding sites of the adsorbent at different doses. The optimal *Typha australis* adsorbent dose obtained is 0.5 g.
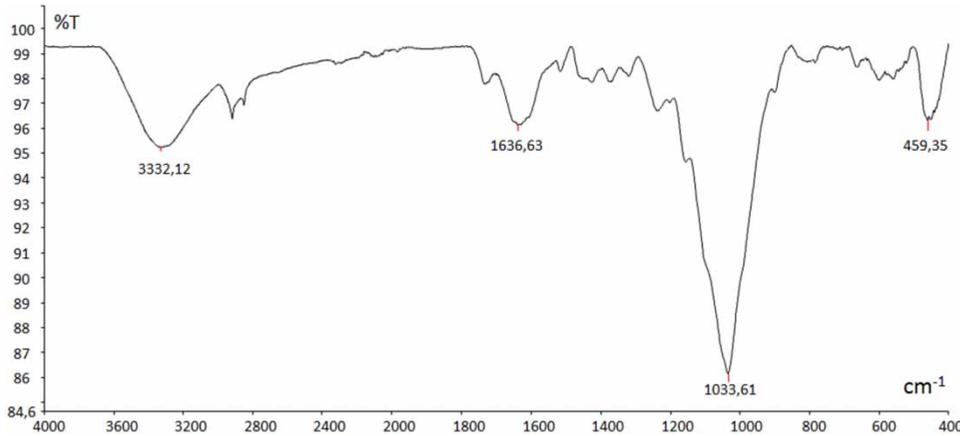
Figure 2 | FTIR-ATR spectra of *Typha australis*.

Kinetic study

Contact time is an important issue in adsorption, which is mainly an equilibrium process, and determining the equilibrium time is of real importance. The effect of contact time on removal of MP (5 and 10 mgL^{-1}) is shown in Figure 4. At equilibrium, 89.4% and 74% for 5 and 10 mg L^{-1}, respectively are obtained with a contact time of 20 min for *Typha australis*.

The kinetic study is very important in the adsorption study, which gives an idea of adsorbate uptake rate and efficiency of adsorption. The mechanism of adsorption depends upon the physical and chemical characteristics of the adsorbent as well as the mass transfer process (Kumar *et al.* 2014).

The adsorption kinetics data are fitted to the PFO and PSO kinetic models to evaluate the adsorption mechanism of the adsorption process.

Constants k$_1$ and q$_e$ for the PFO model could be calculated from the plot of ln (q$_e$ – q$_t$) versus time t, and are presented in Table 4. The correlation coefficient, χ^2 values for the PFO kinetic model are slightly lower at all initial MP concentrations. In addition, the PFO model gave a relatively large deviation between the calculated and experimental values of qe, indicating that the PFO model did not

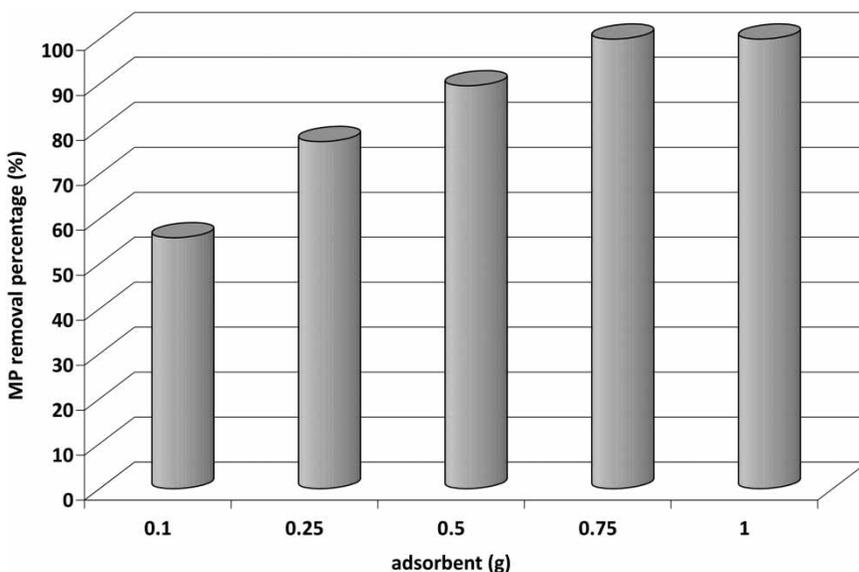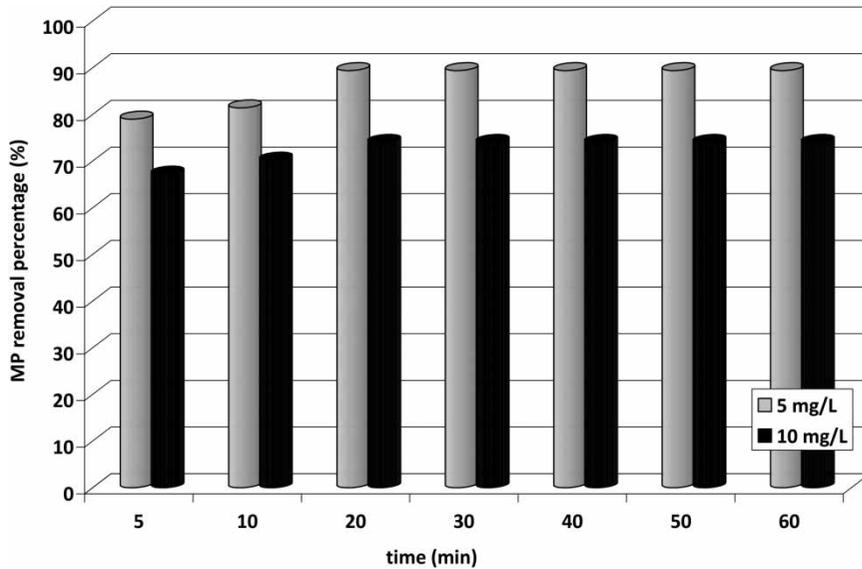**Figure 3** | Effect of *Typha australis* dosage on MP removal percentage.

Figure 4 | Removal percentage of MP by *Typha australis* adsorbent.

appropriately describe the adsorption process of MP *Typha australis* leaf powder adsorbent.

The PSO model predicted the adsorption behavior based on the agreement with chemisorption being the rate limiting step (Ho & McKay 1998). The linear plots of t/q_t versus t are plotted. The constants k_2 and q_e could be calculated from the plots and are also shown in Table 4. The PSO kinetic model yielded a better fit than the PFO kinetic model as the correlation coefficient, χ^2 values close to unity, indicating the applicability of the PSO kinetic model to depict the adsorption process of MP on *Typha australis* leaf powder adsorbent.

Figures 5 and 6 show the experimental equilibrium data and the predicted theoretical kinetics for the sorption of MP onto *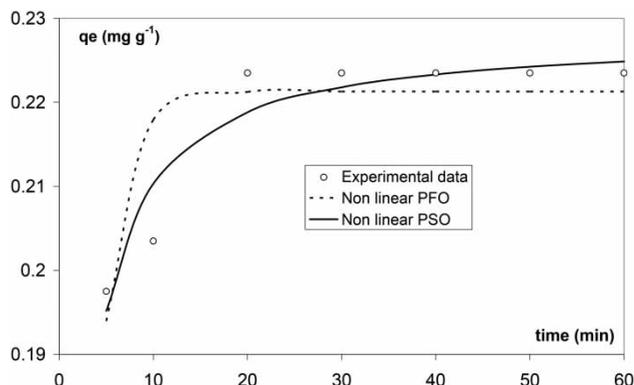Typha australis* for 5 and 10 mgL^{-1}, respectively. The values of model parameters k_1, k_2 and χ^2 are presented in Table 4.

The values of χ^2 are compared between kinetic models, the PSO kinetic model shows a lower value than the PFO kinetic model at all initial MP concentrations. In addition, the q_e calculated by the PSO kinetic model are close to those obtained from the experiments at all initial MP concentrations, indicating that the PFO kinetic model did not properly describe the adsorption process of MP on *Typha australis* adsorbent.

It could be concluded that the mechanism of adsorption is PSO reaction. A better fit to the PSO kinetic model suggested that the adsorption rate is dependent more on the availability of the adsorption sites rather than the MP concentration (Salman *et al.* 2011).

Table 4 | Linear and non linear kinetic model parameters

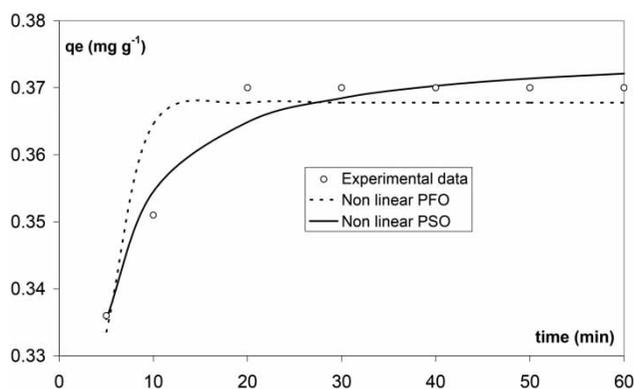| Model	C_i (mg L^{-1})	q_{exp} (mg g^{-1})	Parameter	Linear	χ^2 (%)	Non linear	χ^2 (%)
PFO	5	0.2235	K_1	0.108	97.2	0.418	99.9
			q_e	0.069		0.221	
PSO			K_2	6.30	99.9	5.21	99.9
			q_e	0.226		0.228	
PFO	10	0.370	K_1	0.035	99.2	0.470	99.9
			q_e	0.17		0.367	
PSO			K_2	5.96	99.9	4.42	99.9
			q_e	0.37		0.375	

Figure 5 | PFO and PSO non linear for *Typha australis* adsorbent with initial MP concentration of 5 mg L^{-1}.

Adsorption isotherms
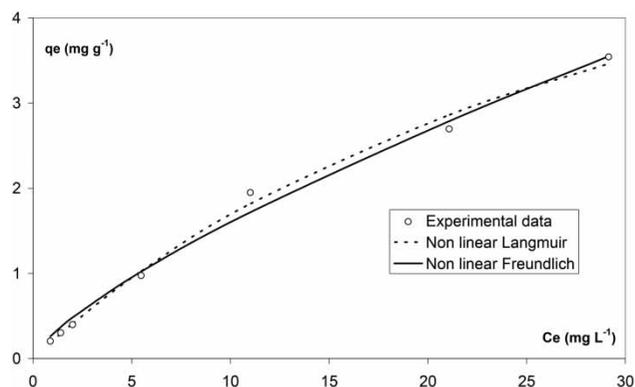
The adsorption isotherm gives an idea of the equilibrium behavior of an MP–*Typha australis* system. The Freundlich model is commonly used to describe the adsorption characteristics for a heterogeneous surface. The Freundlich constants K_F and intensity n could be calculated from the slope and intercept of the linear plot of ln q_e versus ln C_e, and the values are presented in Table 5. The adsorption capacity, K_F, and intensity n for adsorption of MP on *Typha australis* adsorbent are 0.26 and 1.23, respectively. The value of *n* is greater than unity, indicating that the sorption of MP pesticide onto *Typha australis* adsorbent is favorable.

The Langmuir isotherm is valid for monolayer adsorption onto a surface containing a finite number of identical sites. The values of q_m and K_L are determined from the

Table 5 | Linear and non linear isotherm model parameters

Model	Parameter	Linear	χ^2 (%)	Non linear	χ^2 (%)
Langmuir	q_m (mg g^{-1})	7.28	95.0	7.67	97.2
	K_L (mg L^{-1})$^{-1}$	0.030		0.028	
Freundlich	n (−)	1.22	99.6	1.34	92.7
	K_F (mg g^{-1}).(mg L^{-1})$^{-n}$	0.23		0.29	

Langmuir plots and are presented in Table 5. A lower K_L value of 0.030 indicates a high affinity of the MP for the *Typha australis* leaf powder adsorbent. The values of χ^2 are compared, Langmuir isotherms are shown to have higher values than Freundlich isotherms. The adsorption capacity of *Typha australis* leaf powder is found to be 7.67 mg g^{-1}. To compare the efficiency of *Typha australis* in removing aqueous MP, commercial powdered activated carbon (PAC) has been used and showed a capacity removal of 78 mg$_{MP}$/g$_{PAC}$. This result is comparable with the one found by Gupta *et al.* (2011), who reported a capacity removal of 88.9 mg$_{MP}$/g of activated carbon prepared from waste rubber tires.

Figure 7 shows the experimental equilibrium data and the predicted theorical isotherms for the sorption of MP onto *Typha australis* leaf powder. The values of isotherm parameters with the non-linear isotherm that are studied in this work are shown in Table 5.

The values of χ^2 are compared, Langmuir isotherm are shown to have higher values than Freundlich isotherms. The highest χ^2 value further confirmed the suitability of

Figure 6 | PFO and PSO non linear for *Typha australis* adsorbent with initial MP concentration of 10 mg L^{-1}.

Figure 7 | Langmuir and Freundlich non linear for *Typha australis* adsorbent.

Langmuir model in describing the equilibrium data, suggesting the existence of monolayer adsorption of MP onto *Typha australis* leaf powder.

In addition, the analysis of equilibrium data shows that it is not sufficient to use the coefficient of determination of the linear regression method for comparing the best-fitting isotherm. The non-linear curve fitting analysis method is found to be the more appropriate method to determine and confirm that the equilibrium data are best described by the Langmuir isotherm model.

Sensitivity analysis

A sensitivity analysis for the Langmuir and Freundlich models parameters using the contour curves was performed. The MODEST® (MODelisation ESTimation) software is used to carry out the estimation parameters, the sensitivity calculation as well as the contour plots (for the latter, MATLAB® software is used). The aim of parameter estimation is to find correct values for both Langmuir and Freundlich model parameters. The reliability and identifiability of the models' estimated parameters can be addressed using the objective function, f_{obj}, which is the solution point of the least squares problem. By plotting the two-dimensional contour lines of f_{obj}, one can study the identifiability of the problem. If the values of the objective function change rapidly in every direction from the peak point, the parameters are well defined (Figure 8(a)). Minimization of the objective function, f_{obj}, can be performed with a number of different optimization routines. When analyzing the objective function contour plots in Figure 8(a), one can see that the global minimum point is found. The true values of these coefficients, q_e and K_L (Figure 8(a)) and $(1/n)$ and K_F (Figure 8(b)), are to a high degree of probability in the immediate vicinity of the computed minimum, because the value of the objective function increases relatively rapidly when moving from the minimum in the directions of the parameter axis. By plotting the contour lines with varying parameters intervals for the χ^2 values of the fit, it was found that no crucial inter-correlations exist. So, the identifiability of the two parameters (Langmuir) proved to be good and precise.
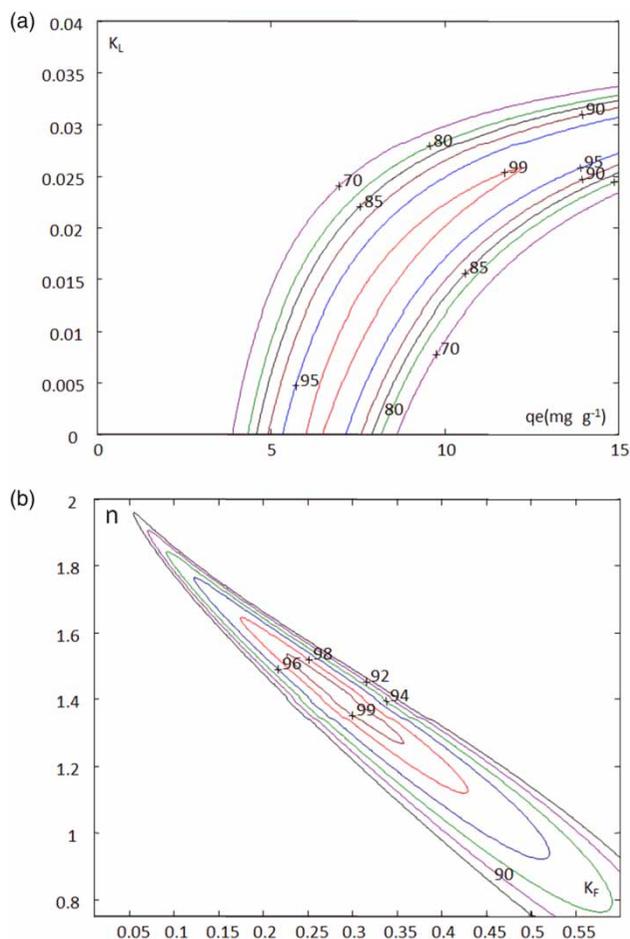

Figure 8 | Langmuir and Freundlich models sensitivity analysis.

CONCLUSION

The removal of MP pesticide from aqueous solution using *Typha australis* biomass collected from Senegal River banks as adsorbent has been investigated under different experimental conditions in batch process. In spite of its low accessible surface area, $0.91 \, m^2 \, g^{-1}$, the used biomass shows interesting pesticide uptake rate, more than $7 \, mg \, g^{-1}$. The linear and non-linear kinetic analysis with varying concentrations showed that the PSO model is applicable to *Typha australis* adsorbent used for MP removal, suggesting a chemisorption limiting rate step. Adsorption isotherm study showed that the Langmuir model well fitted the experimental data compared to the Freundlich model, suggesting the existence of monolayer adsorption of MP. These results indicated that *Typha*

australis sorbent can be successfully used for the removal of MP from aqueous solutions.

ACKNOWLEDGMENTS

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