Potential of Rice Husk in Methyl Parathion Removal: Preliminary Study

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ABSTRACT: The concern for the removal of pesticides in water resources is grown in recent years, which justifies the search for alternative technologies to those applied in conventional water treatment processes. The use of agricultural wastes directly for the preparation of the adsorbents, is a viable method, combining removal efficiency, low cost, and biodegradability of the material applied. The present work was carried out to evaluate the employability of Rice Husk (RH), a waste from rice agriculture, in Methyl parathion (MP) removal from aqueous solutions. To find the optimum removal yield of (MP) onto (RH), the effects of various experimental factors: adsorbent dosage, pH, and the effect of contact time were studied by using the batch experiments mode. The adsorption kinetic data were analyzed using the Pseudo First Order (PFO), Pseudo Second Order (PSO), and Elovich models. For the kinetic study, the adsorption process fitted the PSO model. Three adsorption isotherms namely the Langmuir, Freundlich, and Sips isotherms were applied to the adsorption equilibrium data. The results indicated that the parameters isotherm models are found to be suitable for fitting the present adsorption isotherms data in the following subsequent order: Sips > Langmuir> Freundlich. The Sips isotherm exponent n is near the unity indicating that the adsorption data were more of Langmuir form suggesting that the surface of RH is homogeneous for MP adsorption. The monolayer adsorption capacity, q_m , was found to be 4.38 mg/g. The present study showed that RH is a promising adsorbent for the removal of MP from an aqueous solution.

KEYWORDS: *Pesticide, Organophosphate, Methyl parathion, Rice husk, Adsorbent.*

INTRODUCTION

Pesticides have become indispensable in modern agriculture as a means of protecting crops and livestock,

both of which are essential to our food supply. However, the intensive use of pesticides in agriculture has had major

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health consequences [1]. For instance, oorganophosphates as the most extensively used pesticides in the agricultural industry and as chemical warfare agents [2].

Among the Organophosphates, Methyl parathion (o, odimethyl o-p-nitrophenol phosphorothioate, MP) is frequently used in cotton, wheat, rice and sugar crops. Some toxicological studies of MP have been applied [3]. The World Health Organization (WHO) classified MP as class IA "extremely hazardous" pesticides [4] and established the maximum contaminant level of MP in drinking water as 2 µg/L [5].

The concern over the potential hazards associated with MP residues makes it critical to develop an approach to effectively dispose of it. Many treatment technologies were introduced to remove MP from aqueous solution, including microbiological process [6], and physicschemical methods such as photocatalytic [7], electrochemical oxidation [8] as well as adsorption [9]. To date the adsorption process considering as one of the most efficient technologies for the treatment of pesticide polluted water. In addition, this technic has been used to study the removal of a variety of hazardous compounds such as the dyes [10,11] and heavy metals [12-16]. Thanks to his large surface area and numerous active sites in his surface, the commercial activated carbon is mainly used as adsorbent. However, the commercial activated carbon has high cost and is difficult to regenerate.

In order to overcome this problem, utilization of cheaper and indigenous waste material for the removal of pesticides from aqueous solutions has become a focal point of many studies and has gained much attention of the scientific searchers community. In this context natural adsorbents are preferred for their biodegradability, nontoxic nature, highly cost-effective nature and eco-friendly. As a result several agricultural waste materials, e.g., bagasse fly ash, rice husk, watermelon peels, orange peels, chestnut shells, coconut husk, mustard husk, and palm seed coat have been reported as low cost adsorbents on the removal of MP have been reported in the literature [17-21].

Rice is one of the major crops grown throughout the world, is the principal staple food and a provider of nourishment for the world's population [22]. Rice Husk (RH) is also reported as a good adsorbent for removal of pesticides from aqueous solution [23]. Therefore, in this study, a local agricultural, cheap, ecofriendly, abundant and solid waste material rice husk selected for the removal

of MP from aqueous solution. The present study is therefore an attempt to explore the potential of RH for the removal of MP in aqueous media. Hence the influence of different operating parameters on the MP adsorption onto RH has been studied and optimized, such as the adsorbent dosage, pH and the effect of contact time. Moreover, an efficient and comparative study of the adsorption process was investigated based on kinetics and isotherms results with the non-linear methods.

EXPERIMENTAL SECTION

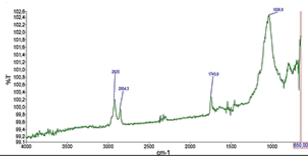
Adsorbate and adsorbent

All chemicals used in this study were of analytical reagent grade. MP in powder used in this study is given by the Chinese Cooperation in Mauritania. A stock solution containing 1000 mg/L of MP was prepared by dissolving 100 mg of MP in 100 mL of Methanol. MP solutions were prepared by diluting stock solution of MP to the desired concentrations in ultrapure water.

Raw rice husk collected from Rosso City, Wilaya of Trarza, Mauritania was crushed, sieved, and thoroughly rinsed with distilled water, and then dried at 105° C for 2 h until reaching a constant weight Finally, the RH was powdered, sieved (< 100 µm) and stored in dessicator until further use.

Batch experiments

The adsorption of MP using RH as adsorbent was conducted in batch experiments mode. In all sets of experiments, fixed concentration of MP (5 mg/L) was agitated at (70 rpm) with varying adsorbent doses for different time periods. In order to investigate the effect of contact time on MP removal by adsorption, 1 g of RH is added to 25 mL of MP at 5 mg/L, the whole is stirred at 70 rpm at pH 6.8 at ambient temperature. The effect of pH on the amount of MP removed was studied at pH of 4, 7 and 10. The study of Adsorption isotherms were obtained by varying the initial MP concentration from 5 to 100 mg/L. 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 micofiltered using micro filter (0.25 µm) and the residual concentration of MP was determined by High Performance Liquid Chromatography (HPLC) method. The adsorption capacity (qe) and percentage of MP removed R (%) was calculated using following equations (1) and (2), respectively:





$$q_e = \frac{(C_i - C_e)V}{m} \tag{1}$$

$$R(\%) = \frac{C_i - C_e}{C_i} \times 100$$
(2)

Where q_e is the amount of MP adsorbed by RH (mg/g), C_i is the initial liquid-phase concentrations of MP (mg/L), C_e is the liquid-phase concentration of MP (mg/L), V is the solution volume (L) and m is the mass of RH used (g).

RESULTS AND DISCUSSION

Characterization

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. The moisture, ash and loss mass ignition values of the RH are 5.8%, 19.9%, and 21.1%, respectively. The cellulose, lignin, and hemicellulose values of the RH are 29.1%, 19.7%, and 20.1 %, respectively [24]. To determine the surface functional groups of RH, Fourier transformation infrared (FT-IR) was used under the following conditions: 1 mg of sample RH was mixed with approximately 200 mg KBr to get pellets. The range of Measurements was (4000–400 cm⁻¹), with a spectral resolution of 4 cm⁻¹. Fig. 1.

As shown in Fig. 1. The band at 2925 cm⁻¹ can be corresponding to the C-H of the methylene groups. The peak at 2854.3 cm⁻¹ was assigned to C–H asymmetrical stretching of methyl groups on the surface of the RH adsorbent. Peaks at 1743.9 cm⁻¹ (C =O stretching of COOH). The peak lying in the region of 1036.6 cm⁻¹ shows the presence of Si-O-Si linkages. The analysis of the FT-IR spectrum showed the presence of carboxyl and hydroxyl groups.

The surface morphology of RH adsorbent was evaluated according to the SEM image obtained in Fig. 2. SEM slide examination of RH (Fig. 2) revealed a stick shape of the prepared powder with a predominance of white spots

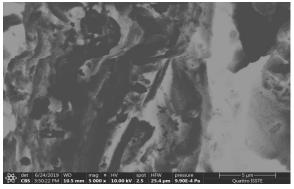


Fig. 2: Scanning electron microscopy images for the RH sample magnified ×5000

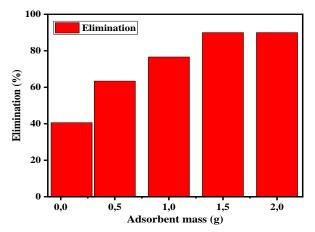


Fig. 3: Effect of adsorbent dosage on percentage adsorption of MP

(corresponding to silica, like grafted component), on RH surface as confirmed by FT-IR analysis. Similar observations are previously reported [24,25].

Effect of adsorbent dosage

Biomass dosage is an important parameter in adsorption studies as it gives the optimum dose at which maximum adsorption occurs. The amount of adsorbent on the efficiency of adsorption was also studied. Fig. 3 shows the removal of MP by RH by varying doses in the range from 0.1 g to 2 g at fixed MP concentration 5 mg/L. The results shown in Fig. 3 indicated that increase in the dose of adsorbent results in an increase in adsorption elimination which could be due to an increase in the surface area. However, further increase after a certain dose kept the adsorption capacity at a constant level, which may be due to the interference between binding sites of adsorbent doses. Finally, in the rest of the work to determine the kinetic models and adsorption isotherms we have chosen 1 g of RH doses.

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Removal

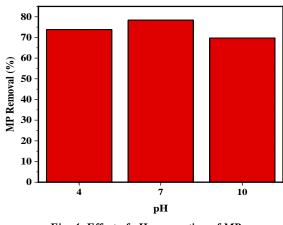


Fig. 4: Effect of pH on sorption of MP

Effect of pH

In Biosorption, pH is an important parameter as it affects both the ionization degree of the sorbate and the surface charge of the sorbent during the biosorption process [25]. The initial pH of the adsorbate solution influences the adsorption process by modifying the functional groups of the adsorbent. The adsorption of MP under different pH was determined in the pH (4, 7, and 10) for 5 mg/L of MP concentration with fixed dose (optimum) of RH (1 g) shown in Fig. 4.

In Fig. 4, the results of the pH effect on MP removal indicate that the removal efficiency of MP increased when the pH of the solution increased from 73.8 % at pH 4 to 78.4 % at pH 7. At pH 10, the percentage of the MP removed showed a significant decrease (69.7 %). The highest removal efficiency of MP adsorption obtained at pH 7 is evaluated at 78.4 %. The results are similar to those found by [26,27] who showed that the removal of other pesticides from aqueous solution, by using the agricultural wastes as adsorbents, is maximum at a pH value of around 7.

Kinetic study

The contact time between adsorbent-adsorbate in the adsorption process was a crucial parameter that indicted the necessary time to reach the equilibrium. Fig. 5 shows the effect of contact time (10 - 60 min) on removal adsorption of MP for RH. We can notice that the equilibrium can be achieved at 30 for the adsorption of MP with the adsorption experiments results showing the maximum removal efficiency was 77.6 %. The same results were reported by [9] who found that the equilibrium time of the biosorbent was short.

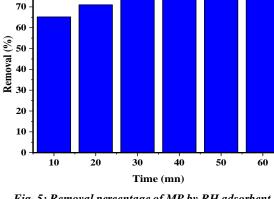


Fig. 5: Removal percentage of MP by RH adsorbent

The rate and mechanism of the adsorption process were evaluated using three different kinetic models, namely Pseudo-First-Order (PFO) model, Pseudo-Second-Order (PSO) model, and Elovich model [28]. The non-linear kinetics PFO, PSO, Elovich and simplified Elovich models are expressed by equations (3), (4), and (5), respectively:

$$q_t = q_e (1 - exp^{-k_1 t})$$
(3)

$$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t} \tag{4}$$

$$q_t = \left(\frac{1}{\beta}\right) Ln(1 + \alpha\beta t) \tag{5}$$

Where q_t is the amount of MP adsorbed per unit mass of RH (mg/g) at time t, k_1 is the PFO rate constant (L/min), k_2 (g/mg.min) is the rate constant for adsorption, q_e (mg/g), t is the contact time (min), α and β , known as the Elovich coefficients. α represents the initial adsorption rate (mg/g.min) and β is related to the extent of surface coverage and activation energy for chemisorption (g/mg).

The adsorption data kinetics were analyzed using the PFO, PSO, and Elovich models. The kinetic parameters constants of experimental data were calculated using nonlinear regression with the help of Solver Excel. Fig. 6 shows the experimental equilibrium data and the predicted theoretical kinetics for the adsorption of MP by RH for 5 mg/L. The values of model parameters k_1 , k_2 , α , β , and R^2 are presented in Table 1.

Based on the summary of kinetic models shown in Table 1, it was noticed that the three kinetics models are found to be suitable for fitting well the present adsorption kinetics data in the following subsequent order: PSO > PFO > Elovich. It could be noted that the mechanism of adsorption is PSO

 Table 1: PFO, PSO and Elovch models constants for the adsorption of MP by RH

Model	Parameters	Value
	q _{exp}	0.097
PFO	q _e	0.096
	K1	0.177
	R ² (%)	98.3
PSO	q _e	0.102
	K2	3.75
	R^{2} (%)	99.9
Elovich	α	5.86
	β	106
	R ² (%)	87.7

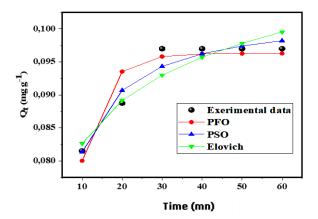


Fig. 6: PFO, PSO and Elovich non linear for RH adsorbent

reaction, suggesting that the adsorption process was predominantly controlled by chemical reactions between the MP and the adsorption sites of the RH adsorbent.

On the other hand, Pearson's correlation matrix was used to explore the relationships between the kinetic models (Table 2). The results showed that the strong and positive correlation obtained between the PSO and the Elovich (r=0.980) is superior to the positive correlation obtained between PFO and Elovich models (r=0.884) (Table 2). The results confirmed that the equilibrium data were best represented by the PSO and Elovich models. The best fit of PSO and Elovich kinetic models to the experimental data indicates the chemisorption of pesticide on the adsorbent surface [29].

Adsorption isotherms

Adsorption isotherms play an important role in the determination of the maximum adsorption capacity and the

Table 2: Correlation matrix of the kinetic models

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Variables	PFO	PSO	Elovich
PFO	1		
PSO	0,958	1	
Elovich	0,884	0,980	1

identification of the type of adsorption to occur. In this work, the three isotherm models equations, namely Langmuir, Freundlich, and Sips were tested for describing the experimental results. The Langmuir adsorption isotherm assumes that adsorption takes place at specific homogeneous surface sites within the adsorbent [30]. The Freundlich isotherm is an empirical equation employed to describe the heterogeneity of the adsorbent surface [30]. The Sips isotherm is a combination of the Langmuir and Freundlich isotherms, which represent systems for which one adsorbed molecule could occupy more than one adsorption site [31]. The non-linearized two-parameter isotherm Langmuir, Freundlich, and Sips models are expressed by Equation (6), (7), and (8), respectively:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \tag{6}$$

$$q_e = K_F C_e^{1/n} \tag{7}$$

$$q_e = q_m \frac{K_S C_e^n}{(1 + K_S C_e^n)} \tag{8}$$

Where q_e is the amount of MP adsorbed per unit mass of RH adsorbent (mg/g), k_L is the Langmuir constant related to the adsorption capacity (L/g), C_e is the concentration of MP in the solution at equilibrium (mg/L), q_m is the maximum uptake dose per unit mass of RH adsorbent (mg/g). K_F (mg/g) (L/mg)ⁿ and 1/n are the Freundlich constants related to adsorption capacity and adsorption intensity, respectively. q_m the Sips maximum adsorption capacity (mg/g), K_S the Sips equilibrium constant (L/mg) and *n* the Sips model exponent describing heterogeneity.

The isotherm constants of experimental data were calculated using nonlinear regression with the help of Solver Excel. The isotherms constants related to Langmuir, Freundlich, and Sips models determined from the plots shown in Fig. 7 are listed in Table 3.

As can be clearly seen from Table 3, the Sips, Freundlich and Langmuir models gave the highest R^2 values showing that the adsorption isotherms of MP by RH were best described by these three models, nevertheless, Langmuir

Table 3: Parameters of Langmuir, Freundlich and Sipsisotherm models for MP retention on the RH

(Parameters	Value
Langmuir	q _m	4.38
	K _L	0.018
	R ² (%)	99.76
Freundlich	1/n	0.78
	K _F	0.11
	R ² (%)	99.44
	q _m	3.70
Sips	Ks	0.019
	n	1.07
	R ² (%)	99.78

 Table 4: Correlation matrix of the isotherm models

Tuble II Confetation man at of the isotherm mouels				
Variables	Experimental data	Langmuir	Freundlich	Sips
Experimental data	1			
Langmuir	0,999	1		
Freundlich	0,997	0,999	1	
Sips	0,999	1,000	0,999	1

and Sips models seems to provide slightly better fittings. Pearson's correlation matrix confirmed that both the Sips and Langmuir isotherms could well represent the experimental adsorption data (r=1) (Table 4).

On the other hand, according to the literature that the Sips isotherm constant (n) is nearly 1, this means that the equilibrium isotherm behaves as Langmuir, not as Freundlich isotherm [32]. In fact, that in this study, the suitability of the Langmuir isotherm to fit the data was confirmed by the exponent value of the Sips model, n, which was near to 1. It should be noted that most of the isotherm adsorption studies of MP on various adsorbents follow the Langmuir isotherm model [1; 33].

Based on the Langmuir isotherm model, the adsorption of MP on RH adsorbent is assumed to have homogeneous surface-active sites which are all identical and energetically equivalent. The monolayer adsorption capacity, q_m , was found to be 4.38 mg/g.

Values of the adsorption capacity of other adsorbents for MP from the literature are given in Table 5 for comparison. The result clearly showed that the RH adsorbent developed is much more effective and comparable for the removal of MP pesticide from an aqueous solution compared to some adsorbents previously reported. It is important to note that the preparation of RH adsorbent required less energy and has not undergone any

 Table 5: Comparison of adsorption capacity of RH with other

 adsorbents for MP adsorption

Adsorbents	$q_m (mg g^{-1})$	References
Chitosan blank gel beads	3	[1]
Chestnut shell	5.92	[18]
Water melon peel	6.39	[19]
Sandy soil	0.84	[34]
Red soil	2.28	[34]
Clayey soil	3.49	[34]
Vermicompost	0.17	[35]
RH	4.38	Present study

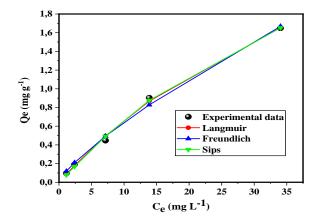


Fig. 7: Non-linearized parameter isotherm models for MP adsorption by RH

chemical modification, which makes it a very ecological adsorbent without danger to human health.

CONCLUSIONS

In conclusion, the adsorption of MP from aqueous solutions using RH as low-cost adsorbent has been studied in a batch system. The non-linear kinetic analysis showed that the PSO model is suitable for molding the adsorption of MP onto RH, suggesting a chemisorption limiting rate step. The adsorption isotherm study showed that the Langmuir model well fitted the experimental data compared to the Freundlich model, suggesting the formation of a monolayer during the adsorption of MP. The suitability of the Langmuir isotherm to fit the data was confirmed by the exponent value of the Sips model, n, which was near to 1. The maximum adsorption capacity was found to be 4.38 mg g⁻¹. This investigation indicates that RH could be considered as a potentially low-cost adsorbent for MP removal from an aqueous solution. The experimental results indicated that the RH adsorbent can be successfully used for the removal of MP from aqueous solutions. For future studies, the usability of RH for pesticides removal from real water will be tested and as a comparison, a fixed bed column will be employed to investigate the effect of reactor design.

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