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Sorption of ivermectin in Nigerian Oxisols and Alfisols: quantitative insights on environmental fate in tropical soils

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Abstract: Because of the toxicity of ivermectin, the study of its environmental fate is necessary. Sorption experiments were done in laboratory batch experiments using two tropical soils. There was initial high sorption in the first 120 min, followed by steady continuous desorption. The pH of the solution had a reciprocal effect on the sorption of ivermectin in both soils. Sorption increased with an increase in ivermectin concentration for the Oxisol and Alfisol. The pseudo-second-order (PSO) kinetic model best describes ivermectin sorption. The adsorption on both soils was explained better by the Freundlich isotherm. An initial increase in the ambient temperature from 25°C to 40°C led to more significant adsorption. The study showed that ivermectin would have high bioavailability once in the environment.

Keywords: pharmaceuticals; Langmuir isotherm; Freundlich isotherm; solution pH; Oxisol; Alfisol.

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Biographical notes: Caleb Oluwaseun Ojo obtained a BSc in Industrial Chemistry and MSc in Analytical Chemistry from the University of Ibadan and another Master's degree in Environmental Science from the University of Aberdeen. He is interested in the chemistry of the environment, which involves the origin and fate of pollutants and how these pollutants can be eliminated with low-cost methods. He derived toxicity thresholds for Cu and Zn in agricultural soils, which can serve as soil guideline values. He is a recipient of various prestigious awards and is a sought-after speaker.

Bamidele Iromidayo Olu-owolabi is a Professor of Analytical/Environmental Chemistry in the Department of Chemistry at the University of Ibadan. Her work focuses on developing low-cost, sustainable alternative materials for the remediation of contaminated/polluted water and soil. Furthermore, her research group has extensively worked on and is still working on environmental monitoring, modelling and remediation with the application of fuzzy logic, an emerging global concept to interpret the result. As a mentor, she has supervised to date over 100 master's thesis, one (1) MPhil and twelve (12) PhD students. She is also a regular peer reviewer for several Elsevier journals.

1 Introduction

Ivermectin, an isolate from *Streptomyces avermectinius*, has been termed the most successful antiparasitic with many applications (Omura, 2008). Its success against internal and external parasites made it the first-ever 'endectocide'. This macrocyclic lactone is made up of two homologs, which are 22,23-dihydro avermectin B1a (\geq 80%) and 22,23-dihydro avermectin B1b (\leq 20%) (Figure 1). The discovery of ivermectin has contributed to the economic prosperity of animal production and the welfare of people in developing countries (Rath et al., 2016). Because of its robust anthelmintic and insecticidal behaviour, it is used as a veterinary drug for livestock (Horvat et al., 2012; Rath et al., 2016). It has also been used to cure onchocerciasis (river blindness), which was endemic in sub-Saharan Africa and South America (Lakwo et al., 2013).

However, because ivermectin undergoes little metabolism, it is excreted almost unchanged in feces depending on the animal and the route of administration (Bernal et al., 1994). Lifschitz et al. (2000), in their study on ivermectin distribution in different tissues known to harbour parasites, discovered that the drug is disproportionately concentrated in bile and feces. Because of this high fecal concentration, it is readily transferred to the soil and affects earthworms and other non-target organisms due to the long degradation half-life in soil or soil-feces mixture (Iglesias et al., 2018). This half-life is about 7–14 days in summer and 91–217 days in winter (Lumaret et al., 2012). In a study of the fecal excretion profile of ivermectin in sheep, the drug was discovered in feces as early as 8 h post-administration. It could still be detected for about 13–17 days post-administration (Vokral et al., 2019).

Ivermectin binds strongly to soil and manure and is persistent depending on the soil type, the climate and the frequency of administration (de Souza and Guimarães, 2022). In addition to its persistence, ivermectin exhibits potent toxicity at low concentrations (Verdú et al., 2015). Initially, it was thought that ivermectin and other drugs in the avermectin family do not penetrate the plant body and, as a result, are not taken up from contaminated soil in significant quantities (Bai and Ogbourne, 2016). However, Iglesias et al. (2018) found that plants that grew close to experimental fecal pats had a very high concentration of ivermectin. Another study also reported a 20 and 24% reduction in root growth of white mustard by 50 nM and 500 nM concentrations of ivermectin, respectively (Vokřál et al., 2019). The highest acute toxicity has been found in crustaceans, and considerable effects were observed even in concentrations as low as 0.001 ng/L (Horvat et al., 2012). Its deleterious effect on fauna present in feces can lead to the retardation of dung decomposition (Beynon, 2012). Consequently, ivermectin falls

under the class of emerging contaminants due to its recalcitrant nature, low environmental concentration and diffuse sources (Olu-Owolabi et al., 2021). Due to its toxicity, ivermectin has been pointed out for further environmental monitoring and risk assessment (Boxall, 2018).





Adsorption and desorption are vital processes that influence the transport, transformation, bioavailability and ecotoxicity of pharmaceuticals in soils (Strawn, 2021). Due to the importance of sorption in soil chemistry, the scarce data on this subject must be supplemented (Heinrich et al., 2021). In addition to sorption processes, the prevailing soil and environmental conditions also determine the fate of the chemical (Olu-Owolabi et al., 2016). Ivermectin partitions into run-off rain and irrigation water and eventually into surface water, and this can be potentially harmful to aquatic invertebrates and humans because ivermectins resist conventional water treatment (Rath et al., 2016).

Nigerian soils vary widely due to differing parent material and prevailing environmental conditions (temperature, precipitation, and biota) (Isong et al., 2022). Most studies on ivermectin mobility involve soils in temperate regions, with few studies on tropical soils (Krogh et al., 2008). To date, no study has reported the sorption of ivermectin in Nigerian soils, which are likely to be impacted due to the prevalent use of this antiparasitic agent in Nigeria (Suraka et al., 2018). Therefore, this work aims to examine the sorption characteristics of ivermectin in Oxisol and Alfisol while varying environmental variables to predict the fate of the antiparasitic in the environment. The objectives are to

- a study the sorption characteristics of ivermectinon the two Nigerian soils
- b use kinetics and adsorption isotherm models to explain the sorption data.

2 Materials and methods

2.1 Soil sampling, pre-treatment, characterisation

The Oxisol was collected from Teaching and Research farm Obafemi Awolowo University, Ile Ife, while the Alfisol was sampled from the University of Ibadan. The soil samples were air-dried, crushed, and sieved with a 2.0 mm sieve. The physicochemical parameters of the soils were determined by the agronomy department of the University of Ibadan using standard methods listed as follows: particle size by the hydrometer method (Bouyoucos, 1962), organic carbon by Walkley Black method (Walkley and Black, 1934), Cation Exchange Capacity (CEC) by base saturation method (Papanicolaou, 1976), pH by calibrated pH meterin 0.01 M CaCl₂ (Maderova et al., 2010) and the Point of Zero Charge (PZC) was determined by the pH drift method without any modification (Yang et al., 2004).

2.2 Adsorption studies

Ivermectin adsorption onto soil surfaces was studied using batch experiments. Brown glass vials of 50 mL sealed with Teflon-lined screw caps and wrapped with aluminium foil in the dark to reduce possible losses from photochemical degradation were used in these experiments. All used apparatus was thoroughly pre-cleaned with distilled water. The distilled water used in these experiments was ultrapure water with a resistance of 18.2 M Ω .cm, obtained from the pharmaceutical chemistry department, University of Ibadan. Ivermectin stock solution of 1000 µg/mL was prepared by weighing 100 mg of ivermectin in an analytical balance and transferred to a 100 mL conical flask which was made to the mark by dilution solution (a mixture of acetonitrile and distilled water in the ratio 1 : 4). Working solutions were prepared by taking the corresponding volume of stock solutions and made up to mark in a 100 mL volumetric flask. A concentration of 0.01 M CaCl₂ was used as the background electrolyte.

Sorption experiments were based on 20 mL of relevant ivermectin solution added to the brown glass vials containing 1 g of the soil sample. Vials were sealed, wrapped and equilibrated at 25°C by horizontal shaking (100 rpm). Experiments on the rate of sorption were carried out from 10 min to 1440 min, and effects of initial concentration and temperature were investigated at concentrations of 5, 10, 30 and 40 mg/L and temperatures of 25°C and 40°C.

All studies were conducted at the ambient pH of 7.24 except for the effect of pH, which was done from pH 2–10 (with an increment of 1) using 20 μ g/mL of ivermectin. The supernatants were separated after centrifugation at 3000 rpm for 15 min using a centrifuge. Then the supernatants were filtered, and the concentrations were analysed using a UV spectrophotometer. All measurements were done in duplicate. All experiments contained nine samples except those that varied the initial concentrations and reaction temperature.

2.3 Instrumentation

Absorbance readings of ivermectin were detected using General Scientific GS - UV 12Spectrophotometer single-beam spectrophotometer with a 200–800 nm wavelength range. The UV spectrophotometer was used due to the unavailability of the more sophisticated HPLC-Fluorescence detector. The concentrations used in this study were high, and the sensitivity of the adopted method was comparable to those of state-of-theart equipment (Dionisio and Rath, 2016). The pH was determined using a Philips PHS-3C pH meter. Centrifugation was also done using the Galkshemp centrifuge. The wavelength of maximum absorption (λ_{max}) was determined by running a standard (20 ppm) of ivermectin through the spectrophotometer at various wavelengths, and the λ_{max} was found to be 243 nm. The instrument was calibrated, and the r^2 was 0.9946. All weights were taken with an analytical balance.

2.4 Data treatment and statistical analysis

The amount of adsorption at equilibrium, qe ($\mu g/g$), was calculated by the following equation:

$$q_e = (C_o - C_e) V/W \tag{1}$$

where C_o and C_e (µg/mL) are the concentrations of pesticides in the liquid phase initially and at equilibrium, respectively, V is the volume of the solution (mL), and W is the mass of dry adsorbent used (g). The data were fitted to Langmuir (1916) (equation (2)) and Freundlich (1906) (equation (3)) isotherms to evaluate the adsorption parameters.

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o}$$
(2)

$$\log q_e = \log k_f + \frac{1}{n} \log C_e \tag{3}$$

where Q_o (µg/g) is the soil's maximum adsorption capacity; *b* is the binding energy constant; k_f and *n* are Freundlich isotherm constants.

The sorption kinetics data were described using the Lagergren (1898) pseudo-firstorder (PFO) (equation (4)) and pseudo-second-order (PSO) (equation (5)) models, the Elovich (equation (6)) (Olu-Owolabi et al., 2014), and the Weber and Morris (1963) intra-particle diffusion (IPD) (equation (7)) kinetics models.

$$\log (q_e - q_t) = \log q_e - \frac{k_1}{2.303} \tag{4}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{5}$$

$$q_t \frac{1}{\beta} \ln(\alpha \beta) + \ln \frac{1}{\beta}(t) \tag{6}$$

$$q_t = k_i(t^{1/2}) + C \tag{7}$$

where q_e and q_t are the amounts adsorbed (µg. g⁻¹) at equilibrium and time *t*, respectively; k_1 and k_2 are the rate constants of the PFO and PSO, respectively. The q_e and rate constants were calculated from the slope and intercept of the plots of log (q_e-q_t) vs. *t*; and t/qt vs. *t* for PFO and PSO, respectively; α is the initial sorption rate (mg g⁻¹ min⁻¹), β is the desorption constant (g mg⁻¹) during the experiment, and k_i (mg g⁻¹ min^{1/2}) is the rate parameter of the intraparticle diffusion control stage. Error bars displayed on graphs are the standard errors of duplicate data and represent the precision of the results. The t-test tests for statistical differences among results and significant levels were quoted at the 95% confidence level ($p \le 0.05$).

3 Results and discussions

3.1 Soil properties

Table 1 details the physicochemical properties of both soils used in the experiments. The pH of soils is near neutral, with the Oxisol being slightly more acidic due to the high clay content. The pH_{pzc} (point of zero charge: which is the pH at which the surface charge of the sorbent is zero) was higher than the pH for both soils showing that both soils had positively charged surfaces. The Alfisol had a higher cation exchange capacity than the Oxisol, possibly due to the type of clays, the pH or unquantified exchangeable acidity (Young and Young, 2005). Greater homogeneity was associated with the Alfisol due to the high percentage of sand (~80%), while the Oxisol was more heterogeneous.

3.2 Kinetics of ivermectin sorption in soil

Studying the kinetics of ivermectin sorption on soils is vital because the fate of this chemical in the environment depends on its sorption onto soil constituents. Results (Figure 2) showed that there was initially high adsorption of ivermectin on the Oxisol and Alfisol soil between the 10th and 180th minutes. However, this was followed by a steady desorption until the final minute of the reaction. The initial high adsorption and subsequent desorption over time show that the soils do not firmly retain ivermectin and would migrate significantly to water. A similar initial high adsorption at 60 min was observed in a study of ivermectin adsorption by pristine kaolinite. However, afterwards, the adsorption decreased continuously with time, as seen in our experiments (Olu-Owolabi et al., 2021). In another study, it took 24 h to attain equilibrium (Rath et al., 2016), which clearly explains why equilibrium was not attained in this study. The low adsorption can be explained by the low organic matter content of both soils since ivermectin binds strongly to organic matter (Krogh et al., 2008). This initial adsorption can be attributed to hydrogen bonding and multiple unoccupied adsorption sites with attraction for ivermectin. The subsequent desorption is due to the negative surface charge on the soils (since the solution pH of 7.24 is higher than the pH_{nzc}) repelling the partial negative charge on the oxygen atoms of the ivermectin. The soil with higher CEC (Alfisols) had a slightly higher adsorption capacity than the Oxisols, and the same pattern was observed by Rath et al. (2016). The CEC of the soils contributed more to the adsorption capacity than the organic matter content. A higher quantity of OM is expected to lead to higher ivermectin sorption due to the high K_{oc} (1.26 × 10⁴ to 1.57 × 10⁴) of ivermectin (Krogh et al., 2008). In this study, the organic matter content does not differ considerably between the two soils, and the difference in adsorption capacity of the two soils is not statistically significant (p > 0.05).

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sou properties	Oxisois	Alfisols
рН	5.75	6.17
pH _{pzc}	6.2	6.4
Cation exchange capacity (cmol/kg)	7.92	9.29
% Organic carbon	0.665	0.547
%Silt	11.4	13.4
%Sand	31.2	77.2
%Clay	57.4	9.4
%Organic matter	1.144	0.941

 Table 1
 Physico-chemical properties of the soil samples

Figure 2 Effect of time on the adsorption of Ivermectin on Oxisol and Alfisol (intra-particle diffusion plot would be the same trend with $t^{1/2}$ as the x-axis) (see online version for colours)



Once transferred to water, it would bind strongly with sediment with high organic matter due to its high organic-carbon-water partition coefficient (K_{oc}) (Heinrich et al., 2021) and persist long, thus being exposed to sediment-dwelling aquatic organisms (Slootweg et al., 2010).

The data obtained from the effect of time experiments were fitted into four kinetic models: the Lagergren pseudo-first and pseudo-second order (PFO and PSO) and IPD kinetics and Elovich models (Table 2). These models predicted the mechanism of ivermectin sorption, a complex process due to the soil's heterogeneous nature. As a result, to comprehensively predict the dominant mechanism, one must understand the role that factors such as ivermectin-soil interaction, non-specific complex interactions, and ivermectin partitioning within soil phases play in the sorption process (Olu-Owolabi et al., 2016).

Kinetics model	Model parameters	Oxisol	Alfisol
Pseudo-first order	$q_e (\mu \mathrm{g} \mathrm{g}^{-1})$	0.689	0.681
	K_1 (min ⁻¹)	-0.0023	-0.0023
	r^2	0.8324	0.8329
Pseudo-second order	$q_e (\mathrm{\mu g} \ \mathrm{g}^{-1})$	43.5	53.8
	$K_2 (g \mu g^{-1} \min^{-1})$	$-2.5 imes 10^{-4}$	-0.0002
	r^2	0.9285	0.9637
Intra-particle diffusion	$C (\mu \mathrm{g} \mathrm{g}^{-1})$	184.05	191.68
	K_{id}	-3.9063	-3.669
	r^2	0.8446	0.7871
Elovich	$\beta (g \mu g^{-1})$	0.0478	0.043
	r^2	0.459	0.5695

 Table 2
 Kinetic model parameters for ivermectin sorption

When the correlation coefficients of PFO and PSO were compared, it was observed that ivermectin sorption is better described by the PSO kinetic models (Table 2). In their study on ivermectin, Rath et al. (2016) and Olu-Owolabi et al. (2021) obtained similar results in their study on ivermectin. This indicates that there might be electrovalent or covalent bonding between the ivermectin molecules and the available adsorption sites of the soils. Thus, the process is fast and more dependent on the number of sorption sites on the soils than on the concentration of the adsorbate (Olu-Owolabi et al., 2012). The K values indicated that desorption was the dominant phenomenon for most of the processes. Hence the negative value and the desorption rate were similar for both soils.

The r^2 value showed that ivermectin sorption on both soils could not be explained using the Elovich kinetic model. The IPD model plot is not linear (r^2), which means that a single mechanism does not facilitate ivermectin sorptions on Oxisol and Alfisol. A linear plot indicates that the primary mechanism of the reaction is through IPD and is the ratelimiting step when the regression line passes through the origin (Olu-Owolabi et al., 2016). Single mechanisms usually occur with homogenous adsorption surfaces but not with heterogeneous surfaces like soils. The multistep curves shown in Figure 2 represent the various stages of the adsorption process. So, the sorption of ivermectin on the soils was due to two rate-determining steps: a rapid external mass transfer diffusion and a lengthier IPD which occurs later around equilibrium at which sorption and desorption are in a state of dynamic equilibrium (Diagboya et al., 2014).

3.3 Effect of pH

Soil properties such as pH affect the sorption behaviour of any adsorbate. This is because pH affects the nature of the soil surface and the charge density of the adsorbate and adsorbent (Nkutha et al., 2020). In contrast to the observations made by Krogh et al. (2008) about the independence of ivermectin adsorption on pH changes, in this study, the adsorption of ivermectin was affected by the pH of the solution, and we attribute this to the purity of the sample used.

The effect of pH on the sorption of ivermectin in these soils was studied from pH 2–10 (Figure 3). Sorption for both soils was high at lower pH, and the sorption capacity decreased as the pH was increased. Both soils showed no significant difference in the adsorption capacity at all pH (p > 0.05). In addition, there appears to be little change in sorption after the neutral pH for both soils. The same pattern of decreased adsorption withincreasing pH was shown with kaolinite but not with the surface-modified kaolinite species (Olu-Owolabi et al., 2021), indicating that pH change modifies the surface of the adsorbent and not the adsorbate. The high adsorption at low pH is due to the increased positive charge of the soil surfaces and a corresponding bonding with the oxygen of the OH group, which has a partial negative charge. At higher pH, the electronegativity of the soil surface is enhanced, and ivermectin molecules are repelled. The molecules adsorbed at these high pH values are due to pi-pi interactions that have decreased with increasing alkalinity.



Figure 3 Effect of solution pH on ivermectin sorption (see online version for colours)

The high adsorption at low pH could also be attributed to the reduced polarity at the charged surface by the hydrogen ion substitution, thus reducing ionisation and increasing association, making the soil attract hydrophobic compounds (Olu-Owolabi et al., 2014). However, when the pH is increased, polarity increases, and there is less attraction for hydrophobic compounds.

3.4 Adsorption studies

Equilibrium adsorption of ivermectin was carried out on the two soils at 25°C and 40°C with concentrations of 5, 10, 30 and 40 mg/L (Figure 4). The general trend for the Oxisol was increased adsorption of ivermectin with increasing concentration (Figure 4(a)). This trend could be explained by the transfer of ivermectin molecules into the pore after the adsorption surfaces had been saturated. There is also a possible π - π facilitated multilayer adsorption on top of the initial layer of ivermectin molecules, thus increasing adsorption (Olu-Owolabi et al., 2014). For Alfisol, adsorption also increased with increasing

concentration at room temperature, but at 40°C increasing the concentrations led to lower adsorption (Figure 4(b)). The phenomenon at 40°C for Alfisol could be explained by the saturation of the adsorption surfaces, hence limiting the uptake. At this higher temperature, there was likely a re-dissolution of the ivermectin molecules or increased entropy, dislodging them from internal pores and external adsorbent surfaces.

Figure 4 Adsorption trends as ivermeetin concentration and ambient temperature increased for: (a) Oxisol and (b)Alfisol (see online version for colours)



The adsorption of ivermectin on Oxisol at 40°C was higher than at room temperature. The same was observed for Alfisol except at 5 mg/L and 10 mg/L. This endothermic process was facilitated by removing barriers to adsorption by the energy supplied. Langmuir and Freundlich isotherm models were used to evaluate the mechanism of adsorption for equilibrium data at 25°C (Ravindra and Mahesh, 2016). The models' correlation coefficient (r^2) indicates that the adsorption on both soils could be explained by the Freundlich isotherm $(r^2 = 1)$ and not the Langmuir isotherm (Table 3). This noncorrelation with the Langmuir model indicates the absence of monolayer sorption of ivermectin molecules (Ravindra and Mahesh, 2016), usually associated with homogeneous surfaces (Table 1). Correlation with the Freundlich isotherm indicates both soils interact with ivermectin through a complex process that involves sorption on heterogeneous surfaces of differing energy and unequal affinities for ivermectin and the possibility of multilayer adsorption upon the initial layer of ivermectin fostered by a π - π bonding (Olu-Owolabi et al., 2021). The surface adsorption site energy distribution index (n), which indicates the linearity of the process, revealed that surface adsorption was a linear process (n = 1) and ivermeetin is favourably adsorbed (Ravindra and Mahesh, 2016). The result for the soils used in this study is similar to other studies that report fitting the ivermectin sorption process to the Freundlich isotherm (Krogh et al., 2008). The value of *n* indicates strong interaction with the soil binding sites, partly explained by forming adducts/complexes with static inorganic moieties (Rath et al., 2016). However, comparison between soils is difficult due to varying soil properties. In addition, it has also been found that contact time can affect the parameters derived from the fitted isotherm (Krogh et al., 2008). As a result, the ability to compare different studies is limited due to the different contact times studied.

Langmuir sorption model			Freundlich	sorption model			
Soil	$Q_o (\mu g g^{-1})$	b (L/mg)	r^2	Ν	Kf	r^2	
Oxisol	_	20 (*)	0	1	19.99	1	
Alfisol	-2E-17	-1.51	0.5	1	19.99	1	

Table 3Adsorption isotherm model parameters at optimum temperature (25°C) for the two
soils

**Qob* value reported due to constant C_e/Q_e value.

4 Conclusion

To determine the fate of ivermectin sorption, experiments were carried out. The sorption of ivermectin was explored concerning the physicochemical properties of two Nigerian soils (Oxisol and Alfisol). Both soils initially showed high adsorption followed by steady desorption, and no equilibrium was attained until the final time studied. The PSO kinetic model better described the sorption on both soils, indicating that the process depended more on the number of sorption sites than the adsorbate concentration. The pH of the solutions had a reciprocal effect on adsorption for both soils. An increase in concentration caused an increase in adsorption for the Oxisol at all temperatures, while there was a decrease in adsorption for Alfisol as concentrations increased at 40°C. An increase in surrounding temperature led to increased adsorption (endothermic reaction). Both soils displayed their heterogeneity by correlating well with the Freundlich isotherm. A range of bonding is implicated in ivermeetin adsorption on both soils. They involve electrostatic and different van der Waals interactions, pore-filling processes, hydrogen bonding, and π - π facilitated multi-layer ivermectin adsorption. Based on this study, ivermectin would be more available to sediment-dwelling biota and get to the aquifer faster in cooler temperatures or during raining season in tropical regions.

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