The Ability of Closed-House Chicken Litter to Adsorb Dimethoate on Inceptisol

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***Abstract*—Applying animal-manure-based amelioration techniques to soil in an attempt to increase the soil's capacity to adsorb pesticide residues from excessive use in the recent past has been suggested. Since the clay content and mineral types in Inceptisol are insufficient to support the sorption of contaminants optimally, soil ameliorants are needed. Closed-house chicken litter is a common waste in the environment that has demonstrated potential for this role. This waste, which is produced in three to five tons every harvest (32 days) by a chicken farm with a cage capacity of 80000–100000 chickens, is a combination of sawdust from cage matting and chicken manure. Using the batch equilibrium adsorption method and the Freundlich and Langmuir equations, this study treated soil with closed-house chicken litter ameliorant at 40 tons/ha. It was discovered that adding closed-house chicken litter to the soil improved the soil's pH level, organic content, negative charge, and cation exchange capacity (CEC). Consequently, the soil's capacity for adsorption rose with the use of this ameliorant, with a distribution coefficient (Kd) value of 5.581 L g^{-1} (at a dimethoate concentration of 200 mg/L) and a Qmax of 769.23L/mg. The adsorption process tends to follow the isotherms for both the non-ameliorated and ameliorated treatments, suggesting that adsorption can occur physically and chemically.**

***Keywords*—amelioration, pesticide, waste, Freundlich, Langmuir**

I. Introduction

Pesticide adsorption on soil has recently become an environmental issue that needs to be considered, given the increasingly widespread soil contamination due to the use of pesticides that do not comply with the rules of use on agricultural land. The latent existence of some pesticides in soil after usage and their breakdown products has caused a major environmental concern, potentially disrupting microbial ecosystems, polluting the food chain, encouraging biomagnification, causing negative health consequences, and spreading by leaching and runoff to other environmental compartments like water [1].

Pesticides have been found to have a long-term impact on soil health, affecting microbial diversity, fertility, and ecosystem function [2]. Continuous pesticide usage reduces soil microbial diversity and biomass, which are essential for nutrient cycling and organic matter breakdown. Insecticides, among other pesticides, have an easy probability of getting into the food chain and producing major health impacts. When farmers increase the dosage of insecticide usage simultaneously, it will have consequences on both human and soil health. Herviyanti *et al.* [3] in Banuhampu District, Agam Regency, West Sumatra, Indonesia, found that the insecticide concentration in the region reached 0.499 mg/L.

Furthermore, global pesticide use has moved up by more than 40% over the last 20 years, and since 2017, it has stabilized at about 4.1 million tons per year [4]. Dimethoate is an organophosphate insecticide with a chemical structure that is highly soluble in water, in a pure form as a white crystalline solid. At room temperature, it is stable in aqueous solutions with pH of 2–7 and is unstable under alkaline conditions. It has a strong hydrophilic property against contamination in surface water and groundwater, which is important to consider. In addition, it has a low affinity for soil and a moderate affinity for organic matter. Research found that, although it has low persistence in soil, its retention in soil can be affected by organic matter [5].

Dimethoate is lost mainly through microbial hydrolysis and oxidation in aerobic soil, with a half-life of about 2–4 days. It is insignificantly photodegraded in water and soil, highly soluble in water, and weakly adsorbs onto soil particulates, indicating possible leaching. It is hydrolyzed rapidly under alkaline conditions (with a half-life and at pH 9 at 25ºC of about 4 days), leading to the production of O-desmethyl dimethoate and O,O-dimethyl thiophosphoric acid. Under neutral to acidic conditions, it is considered more persistent in soil [6]. Therefore, additional ameliorants are needed in soils with appropriate characteristics so that they can be used as adsorbents that can increase the adsorbability of soil particles to contaminants, especially dimethoate residues, thereby reducing their availability in soil solution and leaching to underground water.

Primary mechanisms such as structural incorporation, surface precipitation, partition, and surface adsorption can be used in adsorbing pollutants, including dimethoate. Surface adsorption, in particular, is the concentration of pollutants onto or around the surface or pores of an adsorbent. I this process, atoms, molecules, or ions accumulate on the surface of solid or liquid phase due to energy generated by the uneven forces of attraction on the solid or liquid surface. It is a mass transfer process that occurs when a solid or liquid surface adsorbs a gas or solute [7], in which case the adsorbate and adsorbent are interdependent.

Adsorption can happen through physical and chemical processes [8]. Physical adsorption is exothermic and reversible, where Van der Waals and electrostatic forces attract adsorbent and adsorbate molecules. Meanwhile, chemical adsorption occurs when adsorbate molecules form chemical bonds with the adsorbent surface [9]. An effective approach to decreasing pollutants is by employing adsorption with readily available ameliorants. Not only do they boost soil fertility, but these ameliorants act as adsorbents that adsorb pesticides that are heavily utilized on agricultural land.

Assessment of the adsorbent's performance is one of the most important aspects to research. Typically, the adsorption capability or pollutant uptake of several materials is compared. The latter is a parameter often determined in specific experimental settings using pre-defined quantities of pollutant and adsorbent. Otherwise, it can be derived from the adsorption isotherm. The specifications for an open pore structure and high specific surface area might be suggested as basic guidelines for designing novel adsorbents [10, 11]. Additionally, surface chemistry also matters when figuring out how a pollutant will interact with an adsorbent [12]. Carbon-based materials, especially nanostructured ones, have found a variety of uses as adsorbents for various contaminants because of their numerous attractive qualities, including their high surface area, configurable pore structure, adjustable surface chemistry, and relatively inexpensive price.

Closed-house chicken litter can be utilized as both an ameliorant and a suitable adsorbent to improve soil quality on land. It has the potential to enhance the adsorption of contaminants on soil particles. It can be used to address the weak adsorption of dimethoate on soil and its high solubility in water, which cause concerns regarding its potential uptake by plants and leaching into the environment, especially affecting groundwater and rivers, posing a risk of toxicity to ecosystems. Comprising sawdust from cage matting and chicken manure from the chicken farming industry, it is available in abundance [13]. The interaction between the functional groups and the surface area of the sawdust constituent of chicken litter and the pollutants will lead to the intended sorption. Sawdust in chicken-litter-based adsorbents has been found to have an adsorption capacity of 69.44–372mg/g for pesticides, herbicides, and agrochemicals [14].

However, research on chicken litter utilization for this purpose has been rare. There is a need to investigate the adsorption capacity of soils, particularly Inceptisols, which are developed in intensive horticultural farming. Physisorption and chemisorption. Besides, a study into the mechanism of chicken litter as an ameliorant against pesticide contamination, particularly dimethoate, on soil is required. Therefore, this research was carried out to address this gap.

II. Materials and Methods

This study was conducted based on laboratory analysis from June to September 2024 at the Laboratory of Chemistry and Soil Fertility, Faculty of Agriculture, University of Andalas.

A. Soil Sampling and Analysis

This research was conducted in Banuhampu District, Agam Regency, West Sumatra, Indonesia, at geographic coordinates 0°21'5.53"S and 100°21'38.87"E. The area is characterized by forest soils that are uncontaminated by pesticides or subjected to no prior cultivation. Soil sampling was based on maps of soil types and land use available at the horticulture crop production center of the research area. Meanwhile, chicken litter was collected from a chicken farm industry in Payakumbuh, West Sumatra.

Soil samples were collected compositely at depths ranging from 0 to 20 cm, with three replications. These soil samples were air-dried for 2×24 h before being crushed and sieved through a 2-mm sieve. The soil sample was then weighed to 500 g absolute dry equivalent (ADE), mixed with 40 tons/ha chicken litter based on research suggestions [15] regarding the optimal dose of incubation characterization results with soil and incubated, for 2 weeks.

After incubation, soil analysis was carried out at the laboratory. A soil assessment aimed to explore the surface charge activity of sorption (Inceptisols) and geo-biosorption (Inceptisols+chicken litter 40 ton/ha): clay content (Inceptisols treatment only), cation exchange capacity (CEC), pH (H_2O, KCl), point of zero charges (PZC), Organic matter (OM) [16] and soil mineral content using XRD Xpert Pro Panalytical.

B. Dimethoate Solution

Dimethoate solutions were prepared at three concentration levels (200, 2000, and 20000 mg/L) [17]. To achieve these concentrations of dimethoate active ingredients, dimethoate was dissolved at 0.5, 5, and 50mL of dimethoate insecticide L^{-1} to be comparable to 200, 2000, and 20000 mg/L dimethoate active ingredients. The raw materials utilized for the standard analysis of the dimethoate active ingredients 98% were obtained from Sigma-Aldrich®.

C. Adsorption Experiment

The analysis of dimethoate adsorption on soil was carried out by weighing 0.5g of incubated soil in an air-dried conditions (passing a 2mm sieve) and putting it into a 100mL Erlenmeyer tube, to which each concentration of dimethoate-active ingredient of 200, 2000, 20000mg/L (0.5; 5; 50mL dimethoate/L) [17] dissolved in 20 mL of 0.1 M $CaCl_2$ as it was added. Each sample was then shaken for 24 hours at 25°C using a shaker set at 300 rpm. The suspension was centrifuged at 4000 rpm for 30 minutes to separate the sample solution from the solids. The supernatant (liquid solution) was then transferred to a vial using a pipette in the amount of 1.5mL. The supernatant was analyzed using HPLC with a column C18 at 25°C with an injection volume of 20µL at a wavelength of 200 nm with the mobile phase consisting of distilled water and acetonitrile [18]

HPLC analyzed adsorption data and estimated solution concentrations to determine sorption capacity (Qe) in milligrams of ions per gram of adsorbent (Eq. 1).

$$Qe = ((C0\text{-}Ce)\times V)/\ W \quad (1)$$

$C0$ is the initial ion concentration (ppm), Ce is the ion concentration after adsorption (ppm), V is the solution volume (L), and W is the soil quantity (g). Data projection onto the dimethoate adsorption curve can be analyzed by comparing the applicability of two equations, the Langmuir and the Freundlich isotherms.

The adsorption efficacy (%R) and sorption coefficient (Kd) of dimethoate on Inceptisol were also calculated (Eqs. 2 and 3) [19].

$$\%\ R = (C0\text{-}Ce)/\ C0\times 100 \quad (2)$$

$$Kd\ (\mathrm{L\ kg^{-1}}) = Qe/\ Ce \quad (3)$$

The R^2 values of the Langmuir and Freundlich isotherm curves indicate the tendency of the alleged adsorption mechanism that occurs (López et al., 2021). Where the value of R^2 indicates that the behavior of the isotherm is described as unfavorable adsorption ($R^2>1$), linear adsorption ($R^2=1$), no adsorption ($R^2=0$), and favorable adsorption ($0<R^2<1$) [20].

To identify the adsorption mechanism, the dimethoate adsorption data relevant to each ameliorant were analyzed using linear regression with the Freundlich (Eq. 4) and Langmuir (Eq. 5) equations [21, 22].

$$\text{Log}\, Qe = \log Kf + 1/n\, \text{Log}\, Ce \tag{4}$$

$$Ce/Qe = (1/Q\text{max})\, Ce + 1/Q\text{max.Kl} \tag{5}$$

III. Result and Discussion

Soil characteristics greatly affect the presence of contaminants in the environment as they determine the chemical and physical interaction of soil as a sorbent with incoming toxic substances. The results of the analysis of the soil chemical properties of non-ameliorated soil (Inceptisol) and ameliorated soil (Inceptisol+chicken litter 40 tons/ha are presented in Table 1. The examination of soil samples collected in the field has revealed that the soil texture was composed of 27.23% sand, 42.36% silt, and 30.41% clay loam. Because clay possesses charges that actively integrate with pollutants, the amount of clay in soil significantly impact on its adsorption capacity. In addition, the mineral type in soil also affects the sorption ability. The mineral types in Inceptisols are shown in Fig. 1.

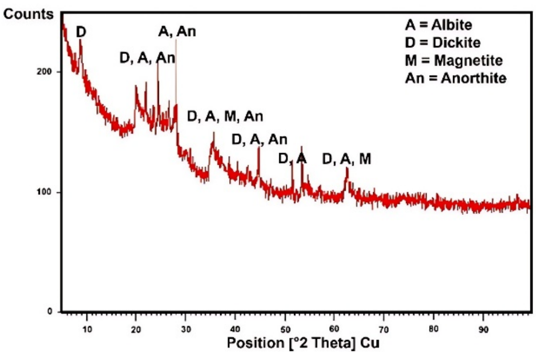


Fig. 1. XRD Analysis of Inceptisol to determine the type of minerals that are involved in adsorption.

Based on the results of XRD analysis (Fig. 1), it is known that Inceptisol in this study contains the minerals dickite, albit, anortite, and magnetite. Dickite is a 1:1 type clay mineral. Albit and anortite are skeletal silicates (tectosilicates) with the chemical formula $NaAlSi_3O_8$, whose structure consists of silica and alumina tetrahedrons interconnected in a three-dimensional framework. Unlike clay minerals (such as kaolinite, smectite, or illite), albite does not have a layer structure that allows expansion or interlayer space to store water or ions [23] therefore the minerals are not large enough to provide adsorption sites for contaminants. The mineral composition in Inceptisols demonstrates the soil's lack of ability to absorb pollutants, which can be addressed using organic matter.

Data show (in Table 1) that adding chicken litter to inceptisol increased soil chemical characteristics such as pH, H_2O, KCl, and PZC. In addition, the organic matter (OM) value reached 7.12%, while the cation exchange capacity (CEC) moved up from 61.99 to 63.18 Cmol Kg^{-1}. The application of chicken litter to Inceptisol raises pH (H_2O and KCl), enhances negative charge due to the disparity between pH H_2O and PZC values, increases organic matter, and augments cation exchange capacity (CEC), hence facilitating improved pollutant adsorption by the soil.

Table 1. Soil chemical properties of Inceptisol (unameliorated) and Inceptisol + Chicken litter 40 ton/ha (ameliorated)

Sample	pH H_2O	pH KCl	PZC	OM (%)	KTK (Cmol Kg^{-1})
Inceptisol	5.3	5	4.67	3.87	61.99
Incept+chicken litter 40 ton/ha	5.83	5.1	4.37	10.98	63.18

As research has revealed [5], the organic matter content of the soil enhances the dimethoate's sorption ability, reducing the likelihood of it being transported in runoff water or infiltrating groundwater. The functional groups (COO^- and OH-) found in organic matter cause H^+ to be released and replaced by Ca^{2+}, Mg^{2+}, and K^+ from chicken litter. Chicken litter has high pH and contains Ca, Mg, and K, hence increasing the soil pH [13]. The increase in pH in turn causes an increase in negative charges on soil due to the presence of functional groups (COO^- and OH^-), hence marking an increasing adsorption ability of the soil. This increase comes from electrostatic interactions and the formation of covalent bonds between dimethoate functional groups on the adsorbent (Inceptisol+chicken litter). The adsorption process is influenced by interactions between the dimethoate molecules and the soil surface. The hydrogen bonds help to strengthen the adsorption of dimethoate on the surface of soil particles, although hydrophobic or ionic interactions can also play a role, depending on the soil pH conditions and soil properties. Ionic interactions or electrostatic bonds can be a cause of dimethoate adsorption on soils due to the bonds between clay minerals and functional groups on organic matter from ameliorated and non-ameliorated soils. These hydrogen bonds occur when dipole-dipole interactions occur between O at P=O (δ-) and H at -OH (δ^+) in soil minerals or organic matter. The presence of different groups, such as O-H, N-H, and H-F indicates different functionalities with at least one electronegative agent combined with functional groups forming hydrogen bonds capable of removing pollutants such as pesticides, pharmaceuticals, and dyes [24].

Ionic or electrostatic bonding between dimethoate and soil is one of the important mechanisms in the pesticide adsorption process in the soil environment, taking the form of attraction between different charges on the adsorbent and adsorbate. Dimethoate, which is polar, has functional groups such as amine groups that can carry partial or full charges depending on the environmental conditions. On the other hand, soil particles, especially clay minerals and organic matter, generally have a negative charge due to the ionization of -OH and -COOH groups, enabling them to bind through electrostatic bonds. Weak acid functional groups such as carboxylic acids (-COO) and phenolic acids (-OH) are deprotonated, negatively charged, and capable of adsorbing cations through electrostatic forces [25, 26].

Table 2. The adsorption capacity of adsorbents

Sample	C0 (mg/L)	Ce (mg/L)	Qe (mg/g)	R (%)	Kd (L/g)
Inceptisol	200	9.150	7.63	95.42	0.834
	2000	79.532	76.82	96.02	0.966
	200000	5082.13	596.72	74.59	0.117
Incept+chicken litter 40 ton/ha	200	1.423	7.943	99.29	5.581
	2000	91.247	76.35	95.44	0.837
	200000	3450.36	661.99	82.75	0.192

The adsorption occurring between treatments differs in the adsorption mechanism, capacity, and distribution of adsorbate in the adsorbent. Pesticide molecules that are positively charged are attracted to negatively charged particles on clays and organic matter. The chemical reactions between unaltered pesticides or their metabolites often lead to the formation of strong bonds (chemisorption), resulting in an increase in the persistence of residues in the soil, while causing it to lose its chemical identity [24]. The adsorption mechanism elucidates the method by which the adsorbate (dimethoate) adheres to the adsorbent (Inceptisols and Inceptisols+chicken litter at 40 ton/ha) (Table 3 and Fig. 2).

Physical and chemical adsorption were estimated simultaneously according to the R^2 using the Freundlich and Langmuir equations. Subsequently, the data from the estimations were converted to obtain Kf, n, Kl, and Qmax. The results are presented in Table 3.

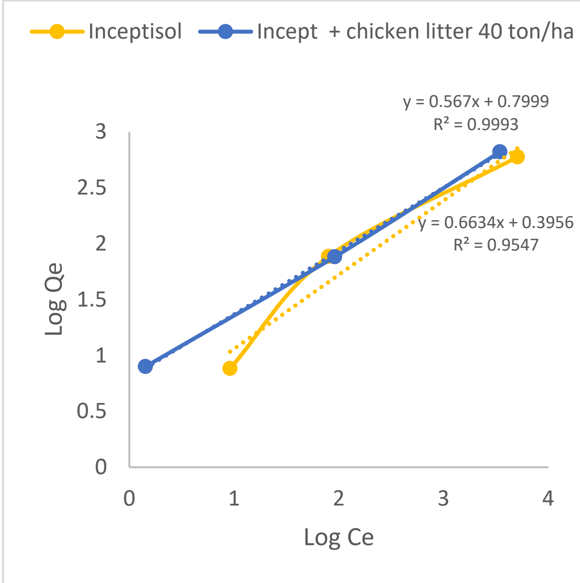
Table 3. Isoterm Freundlich and Langmuir properties of Inceptisol and Inceptisol + chicken litter 40 ton/ha

Isoterm Freundlich: Qe = log Kf + 1/n log Ce				
Perlakuan	**Linear regression**	**R^2**	**Kf (L/g)**	**n**
Inceptisol	0.6634*x*-0.3956	0.95	2.49	1.51
Incept + chicken litter 40 ton/ha	0.567*x*+0.7999	0.99	6.31	1.76
Isoterm Langmuir: Qe = 1/Qmax.Ce + 1/Qmax.Kl				
Perlakuan	**Linear regression**	**R^2**	**KL (L/g)**	**Qmax (L/mg)**
Inceptisol	0.0015*x*+1.0528	0.99	701.87	666.67
Incept + chicken litter ton/ha	0.0013*x*-0.6194	0.97	476.46	769.23

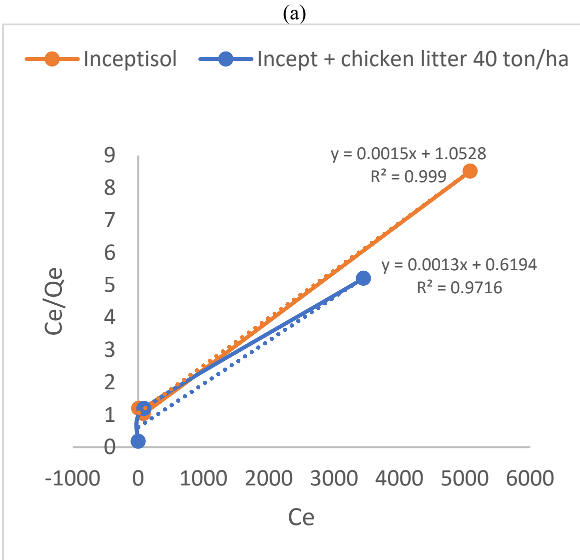
The Qe value demonstrated that the non-ameliorated Inceptisols and ameliorated Inceptisols differed in adsorption ability (Table 2), particularly at a dimethoate concentration of 20,000 mg/L. Since the Qe value of the ameliorated Inceptisols was higher than those of other treatments, it can be concluded that the ameliorating Inceptisols with chicken litter at 40 tons/ha would result in excellent adsorption ability, with a higher adsorption effectiveness value (%R) and sorption coefficient (Kd).

The highest dimethoate concentration of 20,000 mg/L showed lower effectiveness than the lower concentration treatments (200 and 2,000 mg/L), indicating that the higher the concentration of the adsorbate, the faster the contact would be due to the strong push of adsorbate molecules. However, it would eventually lead to a stable state reachinged saturation and equilibrium [27]. In other words, an increase in adsorption capacity occurred in the soil ameliorated with chicken litter until its effectiveness decreased or came to saturation when a dimethoate concentration of 20,000 mg/L was applied.

The addition of ameliorants to soil also increases the distribution coefficient (Kd) of a contaminant in the soil, thus reducing its occurrence underground. A higher Kd value indicates that dimethoate is more bound to the solid phase of the soil, while a lower Kd values indicate that dimethoate is more soluble in the aqueous phase and thus more easily leached. the fate and distribution of an insecticide in the waterways mostly depend on its leaching potential as well as sorption-desorption behavior in soil. The leaching potential, as determined by Kd values, is the most crucial factor in assessing the environmental risk of an insecticide [8].



(a)



(b)

Fig. 2. Freundlich (A) and Langmuir (B) isotherm curves of Inceptisol and Inceptisol + chicken litter 40 ton/ha.

In the case of this research, it can be explained that the effectiveness of dimethoate adsorption dropped with the increase in concentration in all treatments, until dimethoate experienced maximum adsorption or until the adsorbent became saturated when the dimethoate concentration of 20,000 mg/L was applied, causing the adsorbent to cease from adsorbing dimethoate effectively. The non-ameliorated soil and ameliorated soil treatments were recognized to follow both the Freundlich and Langmuir equations as indicated by their high R^2 values. These results suggested that the adsorption mechanism happened concurrently or alternately, depending on the chemical makeup of each adsorbate, in line with previous research findings [28].

An adsorption isotherm describes the pathway of a substrate's interaction with the bulk solution to the surface of the adsorbent. It represents a relationship between the amount of substrate adsorbed per unit mass of adsorbent and the substrate concentration or pressure in the bulk solution at a constant temperature. The Freundlich and Langmuir models, for physical and chemical adsorption, respectively, are two examples of models that use adsorption isotherms to explain the mechanism and characteristics of adsorption. The Freundlich model posits that adsorption takes place on a heterogeneous surface, with several adsorption sites of varying binding strengths. The pesticide molecules in solution bind to these locations unevenly, resulting in theoretically infinite multilayer adsorption [29]. The presence of pores formed from the mixture of sawdust and chicken manure on a given adsorbent allows contaminants to stick and get trapped, leading to the occurrence of a physical adsorption mechanism.

Meanwhile, the Langmuir isotherm indicates that the adsorption mechanism transpires through chemical processes. It implies that monolayer adsorption occurs on the surface, and adsorption is characterized by a homogeneous distribution of binding energy [29]. The pores, large surface area, charged surface, and functional groups of the sorbent greatly affect the adsorption mechanism and ability [28].

The values of Qmax and Kl in Table 3, indicate that ameliorated soil treatment was more effective at absorbing pesticides than the non-ameliorated. This is because the pores and functional groups of raw materials in the form of closed-house chicken litter constituents are thought to be capable of absorbing contaminants in the soil. The maximal adsorption capacity indicates the soil's ability to adsorb pesticides, where the amount of pesticide that the soil can absorb increases with the maximum adsorption capacity value. Meanwhile, adsorption energy represents the strength of pesticide interactions with soil active sites, where strongly bonded pesticides remain in the soil for extended periods, thus preventing leaching. Finally, the Kf value indicates an adsorbent's relative ability to absorb adsorbate; the higher the Kf value, the better the adsorbent's ability to adsorb [29]. Soil amelioration can sequester pollutants, thereby diminishing their availability and toxicity [30], which helps prevent undesirable effects such as soil degradation. The addition of chicken litter into the soil can enhance the adsorption of dimethoate, enabling microorganisms derived from the litter to degrade dimethoate promptly into less hazardous molecules and probably resulting in non-toxic substances. It is also considered a more cost-effective, easy-to-use way to reduce dimethoate contamination in the environment with a raw material that is available in abundance in nature. Thus, chicken litter amelioration technology can serve as a solution to the pollution problem on agricultural land, supporting the implementation of a sustainable agricultural system optimally despite challenges.

IV. Conclusion

Dimethoate adsorption increases in ameliorated soil due to the soil's improved chemical characteristics. Using the equilibrium adsorption method and the Freundlich and Langmuir equations in non-ameliorated soil (Inceptisols) and ameliorated soil (Inceptisols + chicken litter at 40 ton/ha) treatments, the study found that incorporating closed-house chicken litter to soil could enhance the soil's pH, organic matter content, negative charge, and cation exchange capacity (CEC). As a result, the soil's adsorption capacity increased with the soil amelioration using closed-house chicken litter, with a distribution coefficient (Kd) of 5.581L/g (at a dimethoate concentration of 200 mg/L) and a Qmax of 769.23L/mg. The adsorption process appeared to follow the isotherms of both non-ameliorated and ameliorated soil treatments, implying that adsorption could occur both physically and chemically. Thus, it is proved that closed-house chicken litter can increase the adsorption of dimethoate on Inceptisol so that its mobility and existence in soil solution and groundwater can be reduced.

Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

AM conceptualized the research; IP and MM conducted the research at the laboratory; MM and ID analyzed the data; TBP, MM and H wrote the paper and conducted administration project, review and editing; all authors had approved the final version.

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