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DISCOVERING THOUGHTS AND INVENTING FUTURE

HIGHLIGHTS

Issue 4

Crystallization Kinetics

Micronutrient Contents

Low-Cost Adsorbents

Desorption of Diazinon

Volume 12

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Contents of the Volume

- i. Copyright Notice
- ii. Editorial Board Members
- iii. Chief Author and Dean
- iv. Table of Contents
- v. From the Chief Editor's Desk
- vi. Research and Review Papers
- 1. Directional Crystallization Kinetics of Coconut Oil Under Temperature Gradient. *1-6*
- 2. Comparison of Adsorption of Dye Onto Low-Cost Adsorbents. 7-12
- 3. Micronutrient contents of under-Utilized Spices Common in Nigeria. 13-16
- 4. Kinetic & Thermodynamic Study for Adsorption– Desorption of Diazinon with Copper in The Presence of Surfactant. *17-31*
- vii. Auxiliary Memberships
- viii. Process of Submission of Research Paper
- ix. Preferred Author Guidelines
- x. Index



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Directional Crystallization Kinetics of Coconut Oil Under Temperature Gradient

By Jie Peng, Xue Bai, Yunzhu Zhang & Xingpeng Bai

Hainan University, Haikou, China

Abstract - The directional crystallization kinetics of coconut oil was studied under the temperature gradient from 13°C to 15°C. During the crystallization of coconut oil, the overall crystal growth rate Rg decreased over time, but it increased with the growth of temperature difference. While the change of crystallization yields Y first increased then decreased to constant. And the variation trend of crystallization yield with temperature difference was the same with crystal growth rate. The Avrami equation was basically fit for the analysis of coconut oil's crystallization. Some deviations also existed during the last stage of crystallization. What's more, the crystalline form and the methods of crystal growth both changed with different temperature.

Keywords : Coconut oil, temperature gradient, Avrami, crystallization kinetics. GJSFR-B Classification : FOR Code: 030502



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Jie Peng $^{\alpha}$, Xue Bai $^{\sigma}$, Yunzhu Zhang $^{\rho}$ & Xingpeng Bai $^{\omega}$

Abstract - The directional crystallization kinetics of coconut oil was studied under the temperature gradient from 13°C to 15°C. During the crystallization of coconut oil, the overall crystal growth rate Rg decreased over time, but it increased with the growth of temperature difference. While the change of crystallization yields Y first increased then decreased to constant. And the variation trend of crystallization yield with temperature difference was the same with crystal growth rate. The Avrami equation was basically fit for the analysis of coconut oil's crystallization. Some deviations also existed during the last stage of crystallization. What's more, the crystalline form and the methods of crystal growth both changed with different temperature.

Keywords : Coconut oil, temperature gradient, Avrami, crystallization kinetics.

I. INTRODUCTION

ats have many physical and chemical characteristics, such as the melt point, the crystallization behavior and the crystalline form. These special characteristics played an important role in the food quality control and food processing[1]. For some oil and fat food, such as cakes, chocolates, candies, ice creams, margarines, shortening, cocoa butter; the unique plastic grease can extend their shelflife, enhance their quality and stability, and provide special mouth-feel[2, 3].In order to improve the characters of oil and fat food, acquire some special products, making some studies about oil fractionation and crystallization is really essential.

Oil fractionation aims at separating solid lipid and liquid oil according to the temperature. Three main methods can be taken: fractional crystallization, liquidliquid extraction and distillation[4]. Dry fractionation, which belongs to fraction crystallization, is the simplest and the most economic method among oil fractionation.

Different types of triglycerides have different melting points under different temperature, so we can achieve the purpose of solid-liquid separation by cooling[4, 5].

Dry fractionation is a physical modification process, it can avoid the produce of trans fatty acid and decrease vanadium pollution. Low temperature brings the directional suspended crystal separation, which decreased the separating efficiency and purity of the products, mixed the crystal with a large number of lowmelting compositions. Dry fractionation contains three main stages: the produce of crystal nucleus, the growth of crystal and the separation and purification of crystal. In order to give an exact description of grease crystallization behavior, some relevant parameters should be determined.

Coconut oil is one of the Laurel acids grease, which contains about 90% saturated fatty acid. It's main compositions are lauric acid(C12:0,45.9%~50.3%) and myristic acid(C14:0,16.8%~19.2%). This fatty acid makes coconut oil much easier to oxidize, then provide energy to the body in a short time. What's more, it can also reduce the risk of atherosclerosis and heart disease, benefit to our health [6].

In order to acquire the solid lipid with the same characters of butter, a series of temperature gradients were built to give a step-by-step separation of coconut oil in this study. The experimental data was simulated by means of molecular diffusion Fick's Law and the Avrami equation. The fundamental crystallization parameters such as the crystal morphology (n), the crystallization rate (k) were obtained and the mathematical model was established. The purpose was to get the improved mathematical model of the crystallization behavior of coconut oil under the temperature gradient, and this model would be used to guide the experimental to get the ideal value of the crystallization parameters. The results will lay the foundation for exploring the effect of the temperature gradient on the nucleation mechanism and molecular orientation aggregation structure.

II. EXPERIMENTAL

a) Materials

Refined, bleached and deodorized coconut oil was used in this work. It was a kind of clear liquids and presented full-bodied coconut taste at room temperature(25°C).

b) Mpparatus

Fig.1 illustrates the apparatus used in this study. A cylindrical container (15cm in the typical radius) was put on the magnetic stirring apparatus (5).The container was double-layered and its temperature was controlled by the heating and temperature controlled system (4). Another temperature controlled cylinder (condenser pipe (6), 7cm in the typical radius) was put into the middle of double-wall beaker. It was refrigerated by the refrigeration and temperature controlling system (7).

Author $\alpha \ \rho \ \Theta$: College of Food Science and Engineering, Hainan University, Haikou, 570228, P.R. China. E-mail : xinpeng2001@126.com Author σ : College of Horticulture and Landscape Architecture, Hainan University, Haikou 570228, China.



Figure 1 : Schematic of the apparatus used: 1.Temperature controller 2.Container's interlayer 3.Temperature controller 4.Heating and temperaturecontrolling system5.Magnetic stirring apparatus 6.Condenser pipe 7.Refrigeration and temperature controlling systemFig

c) Methods

Two of the above apparatus were used to decrease the error and shorten the repetitive experimental period. The coconut oil (about 100-120g) was conditioned at 60°C for 10 to 20 min to destroy the crystal structures and subsequently poured into the double-wall breaker, the temperatures of condenser

pipe and double-wall breaker were set according to the desired crystallization temperature. When the system was stable, the condenser pipe was dipped into the melt, which was agitated by a magnetic stirrer. The examined process parameters are summarized in Table1. Weight the crystal of condenser every 2 hours until 22hours.

Table 1: Experimental conditions

1 19 2 17 32 minimum 25-27	»mperature/۲	Indoor temp	stirring rate	Temperature of double- wall breaker/℃	Temperature of condenser pipe/°C	Groups
2 17 32 minimum 25-27					19	1
	25-27	25-2	minimum	32	17	2
3 15					15	3

All the data we obtained were the mean values of six times' experiments. So the accuracy of the data was much higher. The data were simulated by means of the Avrami equation, with the help of Microsoft Excel Solver, some fundamental crystallization parameters were obtained, and a mathematical model of directional solidification was also established.

i. Determination of crystal growth rate

The overall crystal growth rate was calculated via Eq. (1) [6]:

$$Rg = \frac{dMc}{Adt} [gcm^{-2}h^{-1}]$$
(1)

Where Rg is the crystal growth rate (assumed as a constant crystal growth rate), Mc is the mass of crystal deposited on the surface of the condenser pipe [g], A is the surface area of the condenser pipe [cm2]

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 $(A=2\pi rL+\pi r^2)$ and t is the crystallization time [h].

ii. Yield of crystallization

The crystallization yield was calculated via the following Eq.(2) [6]:

$$\text{Yield} = \frac{M_{\text{solid}}}{M_{\text{melt}}} \times 100\% \tag{2}$$

Where Msolid is the mass of the solid fraction crystallized on the condenser pipe's surface[g] and Mmelt is the mass of the melt[g].

iii. Crystallization kinetics

Avrami equation (Eq.3) was used to simulate the course of crystallization[7]. The half period of the crystallization was measured by Eq.4:

$$Xt=1-\exp(-kt^{n})$$
(3)

Where Xt is the relative crystallinity, k is the crystallization rate, n is the Avrami index.

$$t_{1/2} = (0.693 / k)^{1/n} \tag{4}$$

III. Results and Discussion

a) Analyses of crystal growth rate and crystallization yield

Fig.2 shows the changes of crystal growth rate (Rg) under different temperature. The overall trend was decreasing. The greater the difference in temperature was, the more obvious of the downtrend, and the greater the crystal growth rate was. At the beginning of crystallization (about 2 hours), the crystal growth rate in 15°C was about 0.0224, while in 19°C was about 0.0449. 12hours later, the downtrend became mild. Fig.3 shows the change of crystallization yield(Y). Its main trend was first increasing then declining. The value of Y reached the maximum between 10 hours to 15 hours.



Figure 2: Curves of coconut oil 's crystal growth rate under different temperatures. The temperatures in the figure stand for the temperatures of the condenser pipe.



Figuer 3 : Curves of coconut oil's crystallization yield under different temperatures. The temperatures in the figure stand for the temperatures of the condenser pipe.

The degree of the saturation in solid fat changed with the temperature. That is to say, per unit of volume of the maximum dissolved solid fats varied with the change of temperature, Stable temperature gradient was built during the experiments, and then the concentration gradient of solid fats formed, because of the molecular diffusion, when the solid fats contacted with the cold wall, crystal formed. The higher the temperature was, the greater the degree of the saturation was and the more crystal we got, so both Rg and Y became bigger. After a long time of crystallization, the coconut oil trended to the saturated state, then Rg declined. When the oil reached the saturated state, some crystal would be dissolved again until it became stable, so at later stage of crystallization, Y dropped to stable.



Figure 4: Fitting curves of crystallization about coconut oil under different temperatures. The temparatures in the figure stand for the temperature of condenser pipe.

b) Analysis of crystallization kinetics

Avrami equation was used to stimulated the data we got.Fig.4 shows the fitting curves of the crystallization. Three curves were similar to S type, that suggested the crystallization were heterogeneous nucleation. At the beginning, the relative crystallinity (Xt) in 19°C was the biggest, and 15°C was the smallest; 8 hours later, the condition was just the reverse. All curves' variation trend was similar, slopes of the curves and Rg both increased with the increasing of temperature gradient, which was agree with the change in Fig.2. The greater the temperature gradient was, the shorter time was needed to reach the stable state during the crystallization. The time of 15°C was about 500min, but the times of 17°C and 19°C were much longer than 22hours. Fig.5 can be used to examine the fitness of the Avrami equation. to means the initial moments of the crystallization, it's equal to 0 in this experiment. The curves were straight line, so the Avrami equation was basically fit for the crystallization of coconut oil. And the curves also told us that the crystallization rate in 19°C was the max, but it was similar in 15°C and in 17°C, which could be examined by comparing the values of k in Tab.2.



Figure 5: The relationship between $\ln(-\ln(1-x))$ and $\ln(t-t0)$

Table 2 : The parameters of Avramiequation during the crystallization of coconut oil

Temperature of condenser pipe (T)	Crystal morphology (n)	Crystallization rate (k)	Half period of crystallization(t1/2)
292	0.714	0.252	4.130
290	1.105	0.131	4.509
288	1.425	0.152	2.901

Table 3: The relationship between Avramiparameters and crystallization behavior[10]

The Nucleation mechanism method of crystal growth	Homogeneous nucleation	Heterogeneous nucleation
One-dimensional growth (acicular crystal)	n=1+1=2	n=1+0=1
Two-dimensional growth (flat crystal) Three-dimensional growth	n=2+1=3	n=2+0=2
(spherical crystal)	n=3+1=4	n=3+0=3

Generally, the value of n varied with the temperature. It is the function of the number and the size of crystal, which reflects the mechanism of nucleation. Relevant data shows the value of n is generally integer, between 1 to 4.But in this experiment, the data we got were non-integer and all less than 2. There were following reasons suggested: (1) The production of fractal geometry during the crystallization; (2) Different crystallization mechanism exited at the same time; (3) The existence of secondary crystallization [9].

The non-integer value also showed the existence of heterogeneous nucleation, it also predicted 1. the fitness of the Avrami equation was not perfect.. Especially in the later stage of crystallization, some 2. deviations existed. According to the data in Tab.3, we could find that in 17°C and 15°C, the mechanism of crystal growth was heterogeneous nucleation and one-dimensional growth, the crystal was acicular crystal. But 3. in 19°C, the mechanism were not sure, and the crystal morphology was also different. The half period of crystallization is some contribution of both k and n.. In 4.

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19°C and 17°C, the value of t1/2 was much higher than in 15°C, that is, at this temperature (15°C), the time to produce 50% crystal was the shortest.

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Comparison of Adsorption of Dye Onto Low-Cost Adsorbents By Ibtissam Maghri, Fatiha Amegrissi, Mohamed Elkouali, Abdelkbir Kenz, Omar Tanane, Mohamed Talbi & M. Salouhi

Hassan II Mohamedia-Casablanca University, Morocco

Abstract - The adsorption of methylene blue onto Mytilus Edulis shells and corn stalks was studied under various conditions such as concentration of adsorbate, adsorbent dosage and granulometry. Batch adsorption experiments were conducted and the result showed that the adsorption was dependent to granulometry and adsorbate concentration, but was partly dependent on the adsorbent dosage. It was rapid, stable and occurs in less than 60 minutes. The objectives of this work were to compare the process of adsorption of methylene blue with two types of absorbents (Mytilus Edulis shells and Corn stalks) and to use isotherms (Langmuir and Freundlich) for modeling adsorption process. Results showed that Mytilus Edulis shells and Corn stalks are suitable for the adsorption of methylene blue dye and could be used as a low cost effective adsorbent in the treatment of the industrial wastewater.

Keywords : Adsorption, Methylene Blue, Mytilus Edulis shells, Corn Stalks, Modeling, isotherms, Langmuir model, Freundlich model, low cost adsorbent, treatment of industrial wastewater.

GJSFR-B Classification : FOR Code: 030301



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Comparison of Adsorption of Dye Onto Low-Cost Adsorbents

Ibtissam Maghri^α, Fatiha Amegrissi^σ, Mohamed Elkouali^ρ, Abdelkbir Kenz^ω, Omar Tanane[¥], Mohamed Talbi[§] & M. Salouhi^x

Abstract - The adsorption of methylene blue onto Mytilus Edulis shells and corn stalks was studied under various conditions such as concentration of adsorbate, adsorbent dosage and granulometry. Batch adsorption experiments were conducted and the result showed that the adsorption was dependent to granulometry and adsorbate concentration, but was partly dependent on the adsorbent dosage. It was rapid, stable and occurs in less than 60 minutes. The objectives of this work were to compare the process of adsorption of methylene blue with two types of absorbents (Mytilus Edulis shells and Corn stalks) and to use isotherms (Langmuir and Freundlich) for modeling adsorption process. Results showed that Mytilus Edulis shells and Corn stalks are suitable for the adsorption of methylene blue dye and could be used as a low cost effective adsorbent in the treatment of the industrial wastewater.

Keywords : Adsorption, Methylene Blue, Mytilus Edulis shells, Corn Stalks, Modeling, isotherms, Langmuir model, Freundlich model, low cost adsorbent, treatment of industrial wastewater.

I. INTRODUCTION

ater pollution is beginning to take alarming proportions for both terrestrial waters and seacoast. Several physicochemical methods have been proposed to treat contaminated waters. At the high cost of certain techniques, it seems appropriate to direct the activities of the national scientific research in the field of environment, to the development of economic techniques. Among these techniques, there is adsorption on activated carbon. This treatment was effective but in most cases expensive [1]. Severalworkers have reported on the potential use of agricultural by-products as good maritime and substrates for the removal of dyes from aqueous solutions and wastewaters such as bacteria [2], sawdust [3], algae [4], shells [5], clay minerals [6,7], china [8], bauxite [9], bagasse [10], molasses [11] and coconut [12]. This process attempts to put into use of waste to treat waste and become even more efficient

Author α : Laboratory of analytical chemistry and physico chemistry of materials, faculty of sciences, Casablanca, Morocco.

E-mail : maghri.ibtissam@gmail.com

because these maritime and agricultural by products are readily available and often pose waste disposal problems. Hence, they are available at little or no cost. This makes the process of treating wastewaters with maritime and agricultural by–product adsorbents more cost effective than the use of conventional adsorbents like activated carbon.

This work aims to study elimination of methylene blue using as adsorbent two materials: Corn stalks and Mytilus Edulis shells.

II. Theoretical and Experimental Part

a) Adsorption parameters

In order to optimize process conditions for adsorption of methylene blue on Corn Stalks and Mytilus Edulis shells, we studied the influence of some factors which may be involved in the process of this phenomenon such as concentration of adsorbate, adsorbent dosage and granulometry.

b) Modeling of adsorption isotherms

Freundlich model (van Bemmelen, 1988 [13] Freundlich, 1909 [14]) is the most commonly used. We consider that it applies to many cases, especially in the case of multilayer adsorption with possible interactions between the adsorbed molecules [15]:

$$q_{\rm e}{=}~K_{\rm F}.C_{\rm e}$$

The most common form used is the plot in logarithmic scale variations qe according to Ce:

$$Log q_e = log K_F + log C_e$$

The constant n (adimentionelle) gives an indication of the intensity of adsorption. It is generally accepted that:

- 0.1<n<0.5 characteristic of a good adsorption.
- 0.5<n<1 characteristic of a moderate adsorption.
- n>1 characteristic of a weak adsorption.

Langmuir model is based on assumptions well known. The initial assumptions are that the solid adsorbent has a limited adsorption capacity (qm), all active sites are identical, they can only complex solute molecule (monolayer adsorption) and there are no interactions between adsorbed molecules. This model can be expressed by equation (1):

$$q_e/q_m = \theta = KL .C_e / (1 + K C)$$
(1)

E-mails : m.elkouali@gmail.com^e, a.kenz@voila.fr[@],

o.tanane@gmail.com^{*}

Author §: Laboratoire des Sciences et d'ingénieur des Bio-systemes, EMI, Agdal-Rabat, Morocco. E-mail : maarifcentre@yahoo.fr

 $K_L,\,$ equilibrium constant of Langmuir, $\theta,\,$ recovery rate The development of equation (1) leads to the linear form of Langmuir isotherm. The ratio RL = 1 /

- (1 + KL.C0), unitless magnitude, indicates that:
 - The adsorption more favorable if R_L tends to 0.
 - Adsorption much worse if R₁ tends to 1.

Figure 2 : Principal equilibrum models.

Isotherms	Non linear expression	Linear expression
Langmuir	$q_e/q_m = \boldsymbol{\theta} = K_L . C_e / (1 + K_L . C_e)$	$1/q_e = 1/(C_e. K_L.q_m) + 1/q_m$
Freundlich	$q_{\rm e}=K_{\rm F}.C_{\rm e}.n$	$Log~(q_{e}) = log~(K_{F}) + n~log~(C_{e})$

III. Results and Discussions

a) Adsorbate concentration effect

The figure 1 shows the adsorption kinetics of methylene blue onto Mytilus Edulis (A) and Corn Stalks (B) at different initial concentrations. We notify a decrease in the residual concentration. After sixty minutes, it reaches a constant value whatever the initial concentration; this shows that the equilibrium time is independent of the initial concentration of the dye.

b) Effect of adsorbent dosage

The adsorption kinetics of methylene blue with three different masses of adsorbents is shown in figure 2. From these results, the biosorption is important for a mass of 4g.l-1 of adsorbent.

c) Adsorbent particle size effect

In this study we used different size fractions. The adsorption kinetics of methylene blue is shown in 3. The adsorption capacity is better for a size range between 0.08 and 0.2mm for Corn Stalks and for a size range 0,056mm for Mytilus Edulis Shells because the adsorption depends on the external surface of the adsorbent material increases with the fineness of its particles.

d) Modeling adsorption isotherms

The experimental isotherms of adsorption equilibrium and maximum adsorption capacity have been validated in detail by the Langmuir model (Corn stalks (Table 2), Mytilus Edulis shells (Table 3)) and Freundlich model (Corn Stalks (Table 4), Mytilus Edulis (Table 5)). The isotherms obtained were L-type according to the classification of Giles [16], which promotes a monolayer adsorption and the interaction between the adsorbate and the adsorbent is important.





30

25

20

15

10

0



(A) Mytilus edulis shells

(B) Corn Stalks





(A) Mytilus edulis shells

(B) Corn Stalks

Figure 3: Effect of adsorbent particle size, pH = 6,8; initial concentration 10 mg.l-1; adsorbent dosage 4 g.l-1; ambient temperature.

Table 2 : Parameters of Langmuir adsorption of methylene blue onto Corn Stalks.

C ₀ (mg.l ⁻¹)	K _L (l.mg⁻¹)	q _m (mg.g ⁻¹)	r²	RL
20	0,023	500	0,9858	0,684
40	0,1429	58,479	0,9718	0,149
50	0,313	29,154	0,9692	0,050

Table 3 : Parameters of Langmuir adsorption of methylene blue onto Mytilus Edulis Shells.

C ₀ (mg.l ⁻¹)	K _L (l.mg⁻¹)	q _m (mg.g⁻¹)	r²	RL
20	0,149	95,23	0,8271	0,251
40	0,91	5,66	0,8316	0,027
50	0,81	3,43	0,9083	0,024

Table 4 : Parameters of Freundlich adsorption of methylene blue onto Corn Stalks.

C₀ (mg.l ⁻¹)	K _F (mg ^(1- n) l ⁿ g ⁻¹)	n	r²	q _m (mg.g ⁻¹)
20	2,867	0,901	0,985	42,624
40	2,50	0,617	0,9706	24,336
50	3,389	0,134	0,889	5,866

Table 5 : Parameters of Freundlich adsorption of methylene blue onto Mytilus Edulis Shells.

C ₀ (mg.l⁻¹)	K _F (mg ^(1- n) l ⁿ g ⁻¹)	n	r²	q _m (mg.g⁻¹)
20	1,258	0,4586	0,8057	40,969
40	2,474	0,5245	0,844	17,12
50	1,845	0,4867	0,9758	12,38

The results show that the maximum adsorption capacity (q_m) obtained from Langmuir model decreases with increasing the concentration value of the Methylene Blue (C₀). It reaches its maximum value at C₀ = 20 mg. I⁻¹. The adsorption is favorable (R_L tends to 0) and moderate (0.5< n <1).

The low values of maximum adsorption capacities obtained from the Freundlich model, confirm that the molecule of Methylene Blue is not strongly adsorbed inside the pores because of its size.

IV. Conclusion

This review highlighted the capacities of Corn Stalks and Mytilus Edulis shells to pretreat raw wastewaters. The extent of dye removal increased with decrease in the initial concentration of dye and particle size of the adsorbents and also increased with increase in contact time and the adsorbents doses used. The equilibrium adsorption is practically achieved in 60 min. Adsorption data were modelled using the Freundlich and Langmuir adsorption isotherms. The adsorption capacities of Corn stalks and Mytilus Edulis shells reaches a maximum at C0=20 mg.l-1. We can say that Mytilus Edulis shells adsorb dyes better than Corn stalks with a high maximum adsorption capacity in comparison with corn stalks. The results indicate that both Corn stalks and Mytilus Edulis shells could be employed as low-cost alternative to commercial activated carbon in methylene blue wastewater treatment.

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Micronutrient Contents of Under-Utilized Spices Common in Nigeria

By Ogunka-Nnoka, CU & Jaja, G

Rivers State University of Science and Technology, Rivers State

Abstract - Vitamin and Mineral contents of five indigenous spices common in Nigeria were investigated. The spices include: 'bailo' (Uapaca guineense); 'atarko' (Zanthoxyllus zanthoxyloides); 'amilo' (Parinari excelsa); 'uburo' (Afromomum danelli) and 'clove' (Syzygium aromaticum). These samples are processed into fine flour, ashed at 5500c and later subjected to wet digestion using nitric sulphuric and perchloric acid. The mineral contents in mg/1 for iron, ranged from 0.99 (S.aromaticum) to 4.42 (U.guineense); zinc 1.24 (P.excelsa) to 3.81 (Uguineense); calcium 12.45 (Z.Zanthoxyloides) to 20.60 (A.danelli); magnesium 16.91 (P.excelsa) to 44.78 (A.danelli); potassium 48.97 (P.excelsa) to 153.66 (A.danelli); Sodium 64.77 (Z.Zanthoxyloides) to 155.70 (P.excelsa). The phosphorus contents for all the samples studied was insignificant. The data obtained for vitamin A showed that Z.Zanthoxyloides had the highest value (3.84 IU) and S.aromaticum the least (0.26 IU); while no value was detected for U.guineense and P.excelsa. Vitamin C contents also was highest for S.aromaticum (31.73/100g) and least for A.danelli (5.55mg/100g). These spices however can serve as mineral and vitamin supplements. The samples studied had good storage properties since they exhibited moisture content below **20%**.

Keywords : Indigenous spices, Vitamin, Mineral, Dry matter. GJSFR-B Classification : FOR Code: 030599



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2012

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Ogunka-Nnoka, CU ^a & Jaja, G ^o

Abstract - Vitamin and Mineral contents of five indigenous spices common in Nigeria were investigated. The spices include: 'bailo' (Uapaca guineense); 'atarko' (Zanthoxyllus zanthoxyloides); 'amilo' (Parinari excelsa); 'uburo' (Afromomum danelli) and 'clove' (Syzygium aromaticum). These samples are processed into fine flour, ashed at 550°c and later subjected to wet digestion using nitric sulphuric and perchloric acid. The mineral contents in ma/1 for iron, ranged from 0.99 (S.aromaticum) to 4.42 (U.guineense); zinc 1.24 (Uguineense): (P.excelsa) 3.81 calcium to 12.45 (Z.Zanthoxyloides) to 20.60 (A.danelli); magnesium 16.91 (P.excelsa) to 44.78 (A.danelli); potassium 48.97 (P.excelsa) to 153.66 (A.danelli); Sodium 64.77 (Z.Zanthoxyloides) to 155.70 (P.excelsa). The phosphorus contents for all the samples studied was insignificant. The data obtained for vitamin A showed that Z.Zanthoxyloides had the highest value (3.84 IU) and S.aromaticum the least (0.26 IU); while no value was detected for U.guineense and P.excelsa. Vitamin C contents also was highest for S.aromaticum (31.73/100g) and least for A.danelli (5.55mg/100g). These spices however can serve as mineral and vitamin supplements. The samples studied had good storage properties since they exhibited moisture content below 20%.

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I. INTRODUCTION

Spices are condiments of plant origin consisting of parts of trees, seeds shrubs and grass which abound in the tropical rainforest and savannah grass land zones (Pearson, 1976). They are often referred to as food accessories or adjuncts because of their ability to stimulate appetite and increase the flow of gastric juice (Diezak, 1989). Some common examples are garlic, ginger, piper nigrum etc. They are used principally to spice foods, drinks and as medication for various ailments (Achinewhu, 1996; Nwiruka et al 2005).

Indigenous spices and herbs in Nigeria are mostly obtained from the wild (Abib 1994) and little attempt has been made to domesticate and cultivate them despite the fact that they constitute a large proportion of the daily diets of rural dwellers (Achinewhu, 1996). Although, they are used nutritionally in insignificant quantities, some researchers have argued that they can also contribute to the nutrient content of the food (Ranjith and Rosabalch, 1995). Studies have been carried out on spices mostly on their flavours and aroma (Agooha, 1981., Iwu, 1989), medicinal values (Gammaniel and Akalu, 1986); antinutrients (Nwinuka et al 2005., Nwachukwu and Ukoha, 2006, Ogunka-Nnoka and Mepba, 2008); as well as in drinks and beverages and in the production of perfumes (Aldelany and Bavrakat, 1970., Purseglove, 1991).

Spices cannot be recognized as major sources of macronutrients, but they can be potential sources of some micronutrients (Ranjith and Rosabalch, 1995; Achinewhu, 1996). Some under-utilized spices in Nigeria namelv 'bafilo' (Uapaca guineense); 'atako' (Zanthoxyllus zanthoxyloides); 'amilo' (Parinari excelsia); 'uburo' (Afromonum danielli) and 'clove' (syzygium aromaticum) have been studied for their proximate and antinutritional factors and were found to contain antinutrients in low toxicity levels (Ogunka-Nnoka and Mepba, 2008). It will be necessary to expand the nutritional contribution towards knowing the amount of mineral and vitamin contents present visa vis their importance to health.

II. MATERIALS AND METHODS

a) Sample source

Samples of 'Vapaca guineense, zanthoxyllus zanthoxyloides, Parinari excelsa, Afromum danelli and syzygium aromaticum were purchased from local farmers from Ughelli at the fruit garden market in Port Harcourt, Rivers State Nigeria.

b) Sample preparation

Samples (300g each) were sorted and cleaned to remove rotten seeds and debris. The seeds were dehulled and oven dried at 600c for 12 hours. Dried samples were separately ground in a kenwood food processor, Model 967 England, then sieved to 300 min mesh. The processed spices were packaged in sealed low density polythene bags and stored in a desiccators for subsequent analysis.

III. CHEMICAL ANALYSIS

a) Mineral analysis

Metals such as sodium (Na) potassium (k) calcium (Ca), Magnessium (mg), 'iron (fe) and zinc (zn) were of analysed from the indigenous spices. Samples were digested following the procedure described by Salami and Non, (2002). Briefly, samples (1.0g each of

Author α : Department of Chemistry (Biochemistry Option), Rivers State University of Science and Technology, Nkpolu-Oroworukwo, P.M.B. 5080. Port Harcourt, Rivers State. E-mail : cogunkannoka@yahoo.co.uk.

oven dried flour were digested with 5ml concentrated nitric acid (HNO_3) and 1ml each of concentrated sulphuric acid ($H2SO_4$) and 60-62% *perchloric* acid ($HCLO_4$) and heated until white fumes of perchloric acid formed. The volume of the digest was reduced by heating but not to dryness. The flask was set aside to cool, after which the content was diluted with distilled deionized water and then filtered into a 50ml volumetric flask. The content was made up to mark with deionized water and stored until analyzed for mineral contents using Atomic Absorption spectrophotometer (AAS), phosphorous content of the digest was determined spectrophoto metrically according to method described by Nahapetain and Bassiri (1975).

b) Vitamin A and C analysis

Vitamin A content was determined using antimony trichloride as described by Kefford et al, 1974, while vitamin C content was determined using the EDTA/TCA extraction method as described by Baraket et al, 1973.

IV. DRY MATTER CONTENT

Dry matter content of the sample were determined by the AOAC, 1995 method.

a) Statistical analysis

The data obtained were subjected to analysis of variance (ANOVA) to determine any significant difference at 5% level using steel and Tornic (1980) method. Means were separated by Duncan's New multiple range test to establish if there were significant differences between the samples (Wahua, 1999).

V. Results and Discussion

Table 1 shows the moisture contents of the spices studied. The moisture contents obtained were 13.56, 12.16, 12.01, 11.83 and 10.67% for S.aromaticum, A.danielli U.guineese, P.excelsia and Z.zanthoxylloides, while the corresponding values of dry matter are 86.44, 87.84, 87.99, 88.17 and 89.33% respectively. The values shows that almost 90% of the spices consist of dry matter content although S.aromaticum had a significant moisture compared to the other samples.

The moisture obtained in this result slightly varied from the previous reports (Ogunka-Nnoka and Mepba, 2008). This may be attributed to seasonal variation or processing methods. The result generally corroborates the reports of Nwachukwu and Ukoha, 2006 for other indigenous spices.

Sample	Moisture contest(%)	Dry matter contents
U.guineense	12.01 ^b	87.99 ^{ab}
Z. zanthoxylloides	10.67 ^c	89.33 ^a
P.excelsia	11.83 ^b	88.17 ^a
A.danelli	12.11 ^b	87.84 ^{ab}
S. aromaticum	13.56ª	86.44 ^c

Values are means of triplicate determination. Means in the column not followed by the same superscripts differ significantly.

The results of mineral contents of the spices are shown in table 2. the Fe (4.42mg/1), and Zn (3.81mg/l) levels were significantly ($p \le 0.05$) high in U.guineese while S.aromaticum and P.excelsia were relatively low in Fe (0.99mg/l) and Zn (1.24mg/l) contents respectively. Ca (20.60mg/l) and Mg (44.78mg/l) were significantly ($P \le 0.05$) high in Adanielli; while Na (155.70mg/l) and K (153.66mg/l) were significantly high in P.excelsia and A.danielli respectively. Relatively low values of Ca (12.45mg/l) and Na (64.77mg/l) were obtained for Z.zanthoxylloides. P.excelsia had low values of Mg (16.91mg/l) and K (48.97mg/l). Phosphorus level was insignificant in all samples studied.

Potassium plays a large role in supporting the nervous system and natural heart rhythm. It stabilizes blood pressure and help in electrochemical transmission and has been shown to prevent strokes. It also works with sodium to maintain a proper water

balance in the body (Jennifer, 2009). These spices, especially *P.excelsia* and *A.danielli* being highly rich in Na and K respectively may be used therapeutically in the area of medicine and to meet the RDA in infants and adults (Bamishaiye et al., 2011). However the high levels of Na in these samples will not favour their incorporation in the diet of obese and hypertensive patients; based on the fact that the effect of high sodium intake on cardiac failure is well known (Sofola, 1981; Olowoyeye, 1981). The moderate level of iron and the relatively low Zn contents could still serve for medicinal purpose. Since iron is vital for the production of haemoglobin, formation of red blood cells and the oxygenation of red blood cells. Iron also improves circulation, digestion, elimination and respiration Zinc promotes a healthy immune system, taste, smell, joint, and connective tissue, cell division, repair and growth and helps in the proper functioning of insulin (Jennifer,

Global

2012

2009). The recommended daily intake is between 8-15 mg/ and dose larger than 25mg may cause anaemia and copper deficiency (Lenntech water treatment and B.V. purification, Holdings, 2005). The level of Zn in these samples meets the recommended dietary requirements and agrees with the result obtained by Bamishaiye et al., 2011 for Zobo drink. The concentration of Ca and Mg in these samples are moderate. Calcium is quantitatively most abundant mineral in the body and in ionic form it regulates transport across the cell wall. Also, the cells need calcium such that 99% of calcium in the body is used for healthy teeth, bones and muscles growth, while magnesium has a vital role in a varying range of biochemical and physiological process including binding to ATP to form active ATP, contributing to DNA

and RNA synthesis, nerve and heart function as it brings about decrease in blood pressure. It has been reported that mg and Ca serve as cofactors in a number of enzyme systems and are involved in neurochemical transmission and muscular excitability. Severe deficiency of Mg causes, tetany just like when calcium level falls (Ukoha, 2006). Consuming these species will definitely contribute to the required daily allowance (RDA) of infants and adults. The amount of mineral content reported in this study may vary based on factors like the time of harvest, method of processing and the type of planting soil. It is also possible that most of the moisture lost during processing may contain some mineral. These spices contain low toxic levels of antinutrients making it possible for the minerals to be easily absorped without any interference.

Mineral s (mg/l)	SAMPLES						
	U.guine	Z.	Ρ.	Α.	S.		
	ense	zanthox	excelsi	danielli	aromat		
		ylloides	а		icum		
Fe	4.42 ^a	2.22 ^b	1.14 ^b	3.92 ^a	0.99 ^{bc}		
Zn	3.81 ^a	1.61 ^b	1.24 ^b	1.84 ^b	1.62 ^b		
Ca	17.43 ^b	12.45 ^c	13.47 ^c	20.60 ^a	13.98 ^c		
Mg	29.24 ^b	21.59 ^c	16.91 ^c	44.78 ^a	17.55 ^d		
Na	121.60 ^b	64.77 ^e	155.70 ^a	110.22 ^c	76.36 ^d		
К	53.14°	60.46 ^b	48.97 ^d	153.66 ^a	60.38 ^b		
Р	< 0.01 >	< 0.01 >	< 0.01	< 0.01 >	< 0.01		

Table 2 : Mineral contents of the spices.

Values are means of triplicate determinations. Means in the same column not followed by the same superscripts differ significantly (P. ≤ 0.05).

The vitamin A and C contents are shown in Table 3 below. The vitamin A content was significantly (P. ≤0.05) high in Z.zanthoxylloides (3.84IU) and was not detected in U.guineense and P.excelsia. All samples with the exception of A.danielli had appreciable yield of vitamin C. Absence of vitamin A in some spices may not be clearly explained; however, one could suggest that the intrinsic lipid properties that identifies vitamin A as fat soluble vitamin may be lacking in these samples. The high vitamin C concentration is an added advantage in prophylactic control of malignant growths like cancer and the effects of free redical damage which leads to aging (Nwaoguikpe, 2009). As an antioxidant, it also help in the production and maintenance of collagen (Matters and Wildowson, 1992).

These results can be compared with the report by Okwu and Josiah, (2006) for A.Africana and B. Pinnatum. However, vitamin C content of these spices results are lower than what Nwachukwu and Ukoha, 2006 reported Zylopia aethiopica (89.76 mg/100g), and piper guineense (117.68mg/100g) The appreciable mineral and vitamin C contents of these spices and also the significant yield of vitamin A in Z.zanthoxylloides reveals the nutritional importance of these spices.

Table 3 :	Vitamin	contents	of the	spices.
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Sample	Spices Vitamin A	Vitamin C
U.guineense	ND	17.72 ^c
Z. zanthoxylloides	3.84 ^a	16.54 ^c
P.excelsia	ND	21.85 ^b
A.danelli	0.38 ^b	5.55 ^d
S. aromaticum	0.26 ^b	31.73 ^a

Values are means of triplicate determinations. Means in the same column not followed by the same superscripts differ significantly ($P. \leq 0.05$).

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Kinetic & Thermodynamic Study for Adsorption – Desorption of Diazinon with Copper in The Presence of Surfactant

By Rounak M. Shariff

University of Salahaddin-Erbil, Iraq

Abstract - In the present work change in the adsorption-desorption processes of Diazinon[O,Owhich diethylo-(2-isopropyl-6-methyl4pyrimidinyl) phosphorothioate] is nonionicorganophosphorous pesticide, preformed by using batch equilibrium experiments on six agricultural soil samples. The kinetics study for adsorption processes investigated that first order rate law and power function equation model provided the best correlation with experiment results. The isothermal models Linear, Freundlich, and Langmuir were applied to describe the adsorption-desorption affinities to the soils. Thermodynamic parameters (ΔG , ΔH and ΔS) were also calculated according to the values of binding Langmuir constant K₁ at 10, 25, 40 \pm 1°C. Linear coefficient K_d values for adsorption process of diazinon varied between 3.261 - 6.413 mlg-1. Freundlich coefficient KF values for adsorption process varied between 1.194 - 1.506 mlg⁻¹. Langmuir coefficient KL for adsorption process varied between 0.017 - 0.020 mlg⁻¹. The negative values for each of ΔG , ΔH and ΔS constants confirmed that diazinon adsorption processes more at lower temperature and done via enthalpy effect.

Keywords : Adsorption- desorption isotherms, Copper, Diazinon, HPLC, Surfactant. GJSFR-B Classification : FOR Code: 030505

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Kinetic & Thermodynamic Study for Adsorption– Desorption of Diazinon with Copper in The Presence of Surfactant

Rounak M. Shariff

Abstract - In the present work change in the adsorptiondesorption processes of Diazinon[O,O-diethylo-(2-isopropyl-6methyl & pyrimidinyl) phosphorothioate] which is nonionicorganophosphorous pesticide, preformed by using batch equilibrium experiments on six agricultural soil samples. The kinetics study for adsorption processes investigated that first order rate law and power function equation model provided the best correlation with experiment results. The isothermal models Linear, Freundlich, and Langmuir were applied to describe the adsorption-desorption affinities to the soils. Thermodynamic parameters (ΔG , ΔH and ΔS) were also calculated according to the values of binding Langmuir constant K₁ at 10, 25, 40 \pm 1°C. Linear coefficient K₁ values for adsorption process of diazinon varied between 3.261 - 6.413 mlg⁻¹. Freundlich coefficient KF values for adsorption process varied between 1.194 - 1.506 mlg⁻¹. Langmuir coefficient K₁ for adsorption process varied between 0.017 - 0.020 mlg⁻¹. The negative values for each of ΔG , ΔH and ΔS constants confirmed that diazinon adsorption processes more at lower temperature and done via enthalpy effect.

To investigate the presence of copper on the adsorption process of diazinon which enhance the adsorption, K_d , K_F and K_L values varied between 4.809 - 9.454 mlg⁻¹, 1.198-1.656 mlg⁻¹ and 0.008 - 0.023 mlg⁻¹ respectively. Application of nonionic surfactant at critical micelles concentration cmc on the desorption for the presence of copper on the adsorption process of diazinon, K_d , K_{Fdes} and K for desorption process in presence of the nonionic surfactant ranged varied between 3.729- 8.058, 0.800- 1.207 and 0.005- 0.011 mlg⁻¹ respectively. All results show a high hysteresis effect for Adsorption desorption processes.

Keywords : Adsorption- desorption isotherms, Copper, Diazinon, HPLC, Surfactant.

I. INTRODUCTION

iazinon [O,O-diethylo-(2-isopropyl-6-methyl-4-pyrimidinyl) phosphorothioate] insecticide for agricultural and indoor pest control, it used for the control of pest in cruciferous vegetables and for controlling insect pests of tobacco plants (1&2) Organophosphours compound tend to have the characteristic phosphoryl bond oxon P=O, and thiophosphoryl bond thion P=S (3&4). It degrades

photochemically produced hydroxyl radicals, degrading from thion to oxon compounds⁽⁵⁾. The detection of organophosphates based on the reaction of the unshared pair of electrons available at the nitrogen of a pyridine ring with the available pair of electrons at the positive site of the organophosphates. A displacement takes place, with the alcohol moiety being split off (6&7). Hydrolysis of diazinon is relatively slow at pH 7 and 9, but is faster at pH 5⁽⁸⁾. Diazinon has toxic degradation products, and is a clear threat to aquatic ecosystems and salmon at current use rates, it commonly detected in surface water ⁽⁹⁾. The rate of adsorption in soil pore governed by temperature which indicated a partly physical and partly chemical⁽¹⁰⁾. The correction of solubility- temperature effect on the standard enthalpy of the pesticide adsorption processes⁽¹¹⁾.

The presence of copper enhances adsorption due to strongly coordination and complex formation or through forming a bridge between the soil and diazonin. Finally it may be via the lowering negative charge, which easily adsorbed to the negatively charged soil surface ⁽¹²⁾.

The presence of nonionic surfactant with nonpolar end at cmc concentration enhances the apparent solubility of hydrophobic organic compounds when both coexist in soil. It represent an important tool for chemical remediation of contaminated soils^(13&14). The molecule character has a great effect on adsorption and mobility through soil⁽¹⁵⁾.

II. MATERIALS AND METHODS

a) Soils

Fresh soil samples were taken from six soil samples were collected from six main agricultural, representing a range of physico-chemical properties. Subsamples of homogenized soils were analyzed for moisture content, organic matter content, particle size texture, pН, loss on distribution, ignition and cations exchangeable basic the detail were characterized in previous article⁽¹⁶⁾.

b) Materials

Analytical grad substituted with following purities expressed in weight percent diazinon (purity 99%), were all purchased from Riedal-de Haen, Sigma-Aldrich company Itd. A nonionic surfactant TritonX 100 2012

Author : University of Salahaddin-Erbil, College of Science, Department of Chemistry, Kurdistan Region, Iraq.

(TX-100), its chemical name is [Octylphenol ethoxcylate] surfactant, its Empirical formula is $(C_8H_{17}C_6H_4O(CH_2CH_2O)_NH)$; where N=9.5, its molecular weight is 625 g mol⁻¹, and its critical micelles concentration cmc concentration 0.0002M was obtained from Fluka AG, Buchs SG, and were used without further treatments. All chemicals used were of analytical grade reagents and used without pre-treatments. Standard stock solutions of the pesticides were prepared in deionised water.

c) Adsorption-Desorption Experiments

Adsorption of the pesticides from aqueous solution was determined at temperature (25±)1 C employing a standard batch equilibrium method. An aqueous stock solution of diazinon of 100 mgL⁻¹ was prepared daily by diluting 1μ l in 100 ml de-ionized water and methanol as co-solvent⁽⁶⁾. The stock and working solution were stored in the dark at 5° Cor up to two days. Duplicate air-dried soil samples were equilibrated with different pesticide concentrations (25, 50, 75, and 100 μ g ml⁻¹) at the soil solution ratio 1:10. The samples plus blanks (no pesticide) and control (no soil) were thermostated and placed in shaker for 0.5, 1, 2, 2.5, 3, 3.5, 4, 6 and 24h. The tubes were centrifuged for 20 min. at 3000 rpm. One ml of the clear supernatant was removed and analyzed for the pesticide concentration. Pesticide identification was done by PerkinElmer series 200 USA family high performance liquid chromatography (HPLC) equipped with a changed loop (20µl), C₁₈ reversed phase column, flow rate 1.5 ml min⁻¹, and a variable wave length UV detector at wavelength 240 nm. Separation of diazinon in aqueous phase was achieved with a mobile phase of ratio 65:35 acetonitrile to water. Under these conditions the retention time was 4.49 min. To study the effect of temperature the same experiments done at temperature (10, 25, 40 \pm 1 C°) employing a standard batch equilibrium method⁽¹¹⁾.

The adsorption of diazinon-copper experiments done in the presence of 40 mgL⁻¹copper, the samples were shaken for 24h, and the amounts of Cu adsorbed were calculated from the difference before and after equilibrium. Each sample was injected twice to determine the pesticide content by integrating the obtained peak with the respective standard pesticides. pesticide content was average The of two measurements, with no more than 5% deviation between the measurements. Desorption processes were done at 25 ± 1 °C and done as each test tube was placed in a thermostated shaker after equilibration for 24 h with different pesticide concentrations the samples were centrifuged 5ml of supernatant was removed from the adsorption equilibrium solution and immediately replaced by 5ml of surfactant and this were repeated for four times (12&17).

III. DATA ANALYSIS

a) Adsorption Kinetics

The rate constants for adsorption of each pesticide on soils were calculated using the first order rate expression⁽¹⁸⁾:

$$Log(C_{\circ} - C_{t}) = \log C_{\circ} - \frac{k}{2.303}t$$
 (1)

Where *k* is the rate constant (hour⁻¹), *t* the time (hour), C_o the concentration of pesticide added(μ g ml⁻¹) and C_t the amount adsorbed (μ g ml⁻¹) at time t. In all cases, first order equation provided satisfactory fit for the data by a linear plots of log (C_o - C_t) against t as shown in (Table 1).

Power function equation used to describe the pesticides adsorption-desorption from soils is given as $^{\scriptscriptstyle (19)}$:

$$Ln(C_t) = \ln C_{\circ} - k \ln t \tag{2}$$

Where C_t is the amount of the pesticides released at time t. A plot lnC_t versus ln (t) should give a straight line with a slope (k) and the intercept is ln (C_o) where the value of slope of straight line is coefficient of release rate as shown in (Table 1).

b) Adsorption-Desorption Isotherms

i. Linear Adsorption Coefficient (Distribution Coefficient)

The distribution coefficient (K_d) was calculated using the equation ^(17&20):

$$C_s = K_d C_e \tag{3}$$

The distribution coefficient (K_d) was calculated by taking the ratio of adsorption concentration in soil (C_s) and equilibrium concentration in solution (C_e). The results were summarized in (Table 2, 3, 4, and 6).

ii. Freundlich Adsorption Isotherm

Adsorption isotherm parameters were calculated using the linearized form of Freundlich equation⁽²¹⁾:

$$LogC_s = \log K_F + \frac{1}{n}\log C_e \tag{4}$$

 C_s and C_e were defined previously, K_F is Freundlich adsorption coefficients, and n is a linearity factor, it is also known as adsorption intensity, 1/n is the slope and logK_F is the intercept of the straight line resulting from the plot of logC_s versus logC_e. The values of K_F and 1/n calculated from this regression equation showed that Freundlich adsorption model effectively describes isotherms for both pesticides in all cases. The results were summarized in (Table 2, 3, 4, and 6).

iii. Langmuir Adsorption isotherm

Data from the batch adsorption conform to Langmuir equation⁽²²⁾:

$$\frac{C_e}{C_s} = \frac{1}{C_m K_L} + \frac{C_e}{C_m}$$
(5)

 $C_{\rm m}$ is the maximum amount of pesticide adsorbed (adsorption maxima, μg ml⁻¹), it reflects the adsorption strength and K_L is the Langmuir adsorption coefficient, binding energy coefficient. The results were summarized in (Table 2, 3, 4, and 6).

The same equations (3, 4 and 5) used to describes the process of desorption in all experiments and on all soil samples ⁽²³⁾. Where k is k_{des} is the desorption rate constant (h⁻¹), Ct is the amount of released pesticides at time t and C is C_e is the amount of released pesticides at equilibrium the results was demonstrated in (Table 6).

c) Thermodynamic parameters

i. Standard free energy change

Values of binding Langmuir constant K_L , can be expressed in terms of the standard Gibbs or free energy for adsorption (ΔG) $^{(24)}.$

$$\Delta G = -RTLnK_L \tag{6}$$

The results were summarized in table 5.

ii. Standard enthalpy change

The standard enthalpy change of adsorption (Δ H) represents the difference in binding energies between the solvent and the soil with the pesticides. Values of Δ H determined graphically from the following equation⁽²⁵⁾:

$$LnK_L = \frac{\Delta H}{RT} + cons \tan t \tag{7}$$

Plotting $-lnK_L$ against 1/ T, a straight line is expected the standard enthalpy change (ΔH) of adsorption were determined from the slope as shown in Fig1. The results were summarized in table 5.

iii. Standard entropy change

The entropy change ΔS for each system were determined by using the equations bellow $^{(26)}.$

$$\Delta G = \Delta H - T \Delta S \tag{8}$$

The values of ΔS were summarized in table 5.

d) Hysteresis coefficient

Hysteresis coefficients (H), can be determined by using the following equation $^{(27)}$.

$$H_1 = \frac{n_a}{n_{des}} \tag{9}$$

Where n_a and n_{des} ratio for Ferundlich adsorption and desorption constants, respectively, indicating the greater or lesser irreversibility of adsorption in all samples, the highest values corresponding for which the highest adsorption constant was obtained. The coefficient H_1 is a simple one and easy to use, Data in table 7 demonstrated H_1 values the selected soil samples.

The extent of hysteresis was quantified by using hysteresis coefficient (ω), it was defined on the discrepancy between the sorption and desorption isotherms, and calculated by using Freundlich parameters estimated from sorption and desorption isotherms separately, (ω) expressed as⁽²⁸⁾:

$$\omega = (\frac{n_a}{n_{des}} - 1)x100 \tag{10}$$

Recently Zhu et. al $^{(23)}$ proposed an alternative hysteresis coefficient (λ) based on the difference in the areas between adsorption and desorption isotherms, they derived the following expression for the parameter λ for the traditional isotherms:

$$\lambda = (\frac{n_a + 1}{n_{des} + 1} - 1)x100 \tag{11}$$

e) Organic matter normalized adsorption coefficient

The linear or distribution coefficient (K_d) is related to soil organic carbon (OC) and soil organic matter (OM) by the following equations⁽²¹⁾:

$$\% OC = \frac{\% OM}{1.724} \tag{12}$$

$$K_{OM} = \frac{100K_d}{\% OM} \tag{13}$$

$$K_{OC} = \frac{100K_d}{\% OC} \tag{14}$$

IV. Results and Discussion

Data in Table 1 showed the values of rate constant for the adsorption of diazinon on different soil samples using two models first order rat law and power function equations, the values were in the range from 0.967 to 2.139 h⁻¹ and 0.104 to 0.244h⁻¹ for the two equations respectively, and the value of standard error (S.E.) from 0.129 to 0.185 and from 0.132 to 0.214 for the two equations respectively. Diazinon has a high octanol water partition coefficient (logK_{ow}= 3.81) exhibited the moderate rate of accumulation with 14.1 % adsorption on the soil solid matrix after 0.5 hours. The

adsorption of diazinon in all cases followed first order rate law as reported in literature ⁽²⁹⁾. The non-linear adsorption isotherms might be expected for diazinon which competes with a limited number of cation exchange sites contributes significantly to adsorption process. The magnitude of the Kd is moderate were ranged between 3.261 - 6.413 mlg⁻¹. as shown in Fig2_{-a}. These findings are in agreement with the hydrophobicity of the pesticide as represented by octanol-water partition coefficient value ⁽²⁹⁾.

The Freundlich adsorption model effectively describes isotherm for diazinon on all soil samples as shown in Fig2-b, values of $K_{\rm F}$ were varied from 1.194 1.506 mlg-1. Values of KF for diazinon was in the order of S3>S4>S5>S1>S6>S2, the difference in the behavior of diazinon toward the soil samples is due to the difference in the type of interaction with the soil which it is likely due to the binding of its phosphoric side moiety to cation on the clay or organic matter, adsorption is correlated with unoqupied phosphate adsorption capacity of a soil. The values of n all n>1 the variable slopes of the adsorption isotherm obtained for different soil systems studied reveal that diazinon adsorption on soil is complex phenomena involving different types of adsorption sites with different surface energies⁽²⁰⁾.

Langmuir adsorption model effectively describes isotherm for diazinon on all soil samples as shown in Fig2-c, with regression factor R² ranged between 0.704- 0.948, values of K_I ranged from 0.017 to 0.020 mlg⁻¹ the maximum amount of diazinon adsorption (C_m) ranged from 333.33 to 1000 mg g⁻¹ the high C_m values on the examined soil samples could be explained by the high affinity of diazinon to bind to soil organic matter and clay values of K_L for diazinon was in the order of $S_4 > S_1 > S_3 > S_6 > S_5 > S_2$. The different value of adsorption coefficients for the same pesticide with different soils is due to soil organic carbon and clay content. A significant differences in the chemistry of the soil organic carbon may be encountered from soil to soil in its polarity, elemental composition, aromaticity, and condensation evolution from loss polymer to condensed cool –like structures^(30&31). Therefore, soil organic carbon and clay are not adequate to estimate different soil adsorption capacity, but also their quality and their chemical nature are important.

Data in Table 2, 3, and 4 shows the effect of temperature on the adsorption of diazinon on different soil samples. The values of free energy change ΔG for adsorption of the studied pesticides on the selected soil samples at 283.15, 298.15 and 313.15K were summarized in tables 5. The negative value of ΔG and decreased with the rise in temperature, indicating that at all experimental temperatures; the interactions of diazinon on soils were spontaneous ⁽³²⁾. The ΔG values were in the range -5.888 to -11.742 KJmol⁻¹. The negative values of ΔH indicated the exothermic

behaviors of the reaction, as the temperature increases the negative values of Δ H is decreased. The linear nature of the plot indicates that the mechanism of adsorption is not changed as temperature is changed. The values of enthalpy change Δ H followed the range -7.892 to -47.887 KJmol⁻¹. The values of R² were in the range 0.816 to 0.996. The negative enthalpy of adsorption for partition coefficient, indicating an exothermic binding reaction, showing that the interaction of pesticides with the soil is an energetically stable exothermic process and the adsorption occurred through a bonding mechanism^(11&33). The negative values of entropy change Δ S followed the range -65.600 to -194.48 Jmol⁻¹ k⁻¹.

The presence of copper on the adsorption process of diazinon which enhance the adsorption as shown in table 6, K_d , K_F and K_I values for adsorption process varied between 4.809 - 9.454 mlg⁻¹, 1.198-1.656 mlg-1 and 0.008- 0.023 mlg⁻¹ respectively. The regression coefficient R² value ranging between 0.697-0.766, 0.853-0.993, and 0.713-0.987 for each model respectively, the standard error S.E. value between 0.118 - 0.372, 0.424-0.569, and 0.231-0.287. The regression equations relating that the highest values are the most fitted model, our results agreed with The desorption research⁽¹³⁾. experiments were conducted with a nonionic surfactant TritonX-100 at concentration 0.1cmc, cmc and 20cmc on diazinon sorbed soil^(17&34). The K_d values for desorption process in the presence of cmc concentration of the surfactant varied between 3.729- 8.058mlg⁻¹ while the value of R² ranging from 0.874 to 0.979 with standard error S.E. value between 0.165 - 0.630. Freundlich coefficient for desorption process K_{Edes} for diazinon in the presence of cmc concentration of the surfactant varied between 0.800- 1.207 mlg⁻¹, the R² value ranging from 0.982 to 0.998 with S.E. 0.404-0.607, the values of n_{Edes} 1.166-1.348. Langmuir desorption coeffecient K_L ranged from 0.005- 0.011 mlg⁻¹ the maximum amount of diazinon desorption (C_m) ranged from 500 to 1000 mg g⁻¹ the R² value ranging from 0.636 to 0.989 with S.E. 0.138-0.219.

Data in table 7 demonstrated H1 values for diazinon from the selected soil samples in the range from 1.236-1.714. The calculated values of hysteresis coefficient (ω) for adsorption-desorption for diazinon on the selected soil samples ranged from 23.6 to 71.4. Whereas hysteresis coefficient (ω) is only applicable for the traditional type isotherms of the successive desorption^(35&36).The hysteresis coefficient (λ) for diazinon from the selected soil samples were ranged from -22.3 to 47.8.

V. Conclusion

The batch kinetics experiments were used to investigate the behavior of diazinon in six agricultural soil samples. The soil OC and clay content and the chemical nature of both constituents determined the adsorption affinity of the soil. The using of pesticide diazinon may increase pesticides leaching to depth relative to the use on agricultural soil samples. The cmc concentration gave the best results in desorption. So the used surfactant solution is therefore fairly effective in desorption of diazinon from the contaminated soil. Thermodynamics and kinetic investigations of clay and soils are limited. Adsorption experiments were conducted at 10, 25, and 40°C to study the thermodynamic parameter, associated with the adsorption of the studied pesticides on the selected soil samples.

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Soil	Con	First or	First order rate equation			Power –function equation		
	c. ppm	(calc (h ⁻¹)		R₂	(calc (h ⁻¹)	S.E	₽²	
S ₁	25	1.288	0.148	0.884	0.244	0.135	0.963	
	50	1.372	0.144	0.824	0.121	0.174	0.932	
	75	1.500	0.143	0.967	0.108	0.197	0.998	
	100	1.320	0.153	0.813	0.123	0.208	0.918	
S ₂	25	1.442	0.144	0.978	0.107	0.142	0.917	
	50	1.566	0.143	0.958	0.157	0.179	0.801	
	75	1.605	0.144	0.910	0.135	0.200	0.984	
	100	1.083	0.138	0.997	0.131	0.214	0.892	
S ₃	25	1.071	0.129	0.848	0.172	0.133	0.980	
	50	1.329	0.129	0.939	0.185	0.175	0.973	
	75	1.454	0.130	0.959	0.170	0.197	0.898	
	100	1.429	0.130	0.984	0.186	0.211	0.858	
S ₄	25	0.967	0.131	0.986	0.152	0.132	0.839	
	50	1.197	0.134	0.937	0.180	0.173	0.946	
	75	1.395	0.134	0.930	0.146	0.198	0.707	
	100	1.411	0.139	0.985	0.182	0.211	0.913	
S ₅	25	1.092	0.135	0.863	0.136	0.135	0.994	
	50	1.166	0.133	0.974	0.185	0.172	0.934	
	75	1.422	0.139	0.979	0.171	0.197	0.831	
	100	1.518	0.145	0.950	0.184	0.212	0.952	
S ₆	25	1.117	0.130	0.985	0.160	0.138	0.928	
	50	1.596	0.145	0.969	0.106	0.179	0.960	
	75	2.139	0.185	0.945	0.109	0.202	0.908	
	100	1.579	0.142	0.991	0.104	0.214	0.931	

Table 1 : Adsorption rate constants for diazinon adsorption on the selected soil samples.

Table 2 : Adsorption of diazinon linear, Freundlich and Langmuir models isotherm parameters on the selected soil samples at 283.15 K.

Adsorpt Models	Parame	Soils					
lion	ter	S ₁	S ₂	S ₃	S ₄	S ₅	S ₆
Ads.	K _d (calc)	12.927	6.976	8.773	10.441	6.931	8.814
Distr.	S.E	0.376	0.279	0.329	0.286	0.132	0.127
coffi.	R ²	0.724	0.764	0.778	0.810	0.788	0.893
Freur	K _F (mL/g)	1.678	1.601	1.641	1.567	1.288	1.288
ndlich	S.E	0.639	0.499	0.551	0.592	0.509	0.558
	n _F	1.730	2.079	1.969	1.647	1.412	1.311
	R ²	0.991	0.938	0.951	0.998	0.963	0.987
Lang	K _L (ml/g)	0.024	0.076	0.061	0.018	0.011	0.082
gmuir.	S.E	0.205	0.263	0.242	0.219	0.282	0.225
coffi.	C _m (µg/g)	1000	500	1000	1000	1000	1000
	R ²	0.999	0.833	0.841	0.956	0.937	0.965

Table 3 : Adsorption of diazinon linear, Freundlich and Langmuir models isotherm parameters on the selected soil samples at 298.15 K.

Adsorpt Models	Parame	Soils	oils				
ion	ter	S ₁	S ₂	S ₃	S ₄	S ₅	S ₆
Ads	K _d (calc)	6.413	3.261	5.503	6.107	5.200	3.559
.Distr.	S.E	0.161	0.899	0.224	0.229	0.140	0.117
coffi.	R ²	0.862	0.737	0.725	0.784	0.720	0.730
	K _{oc} (mL/g)	229	314	172	259	272	236
	K _{om} (mL/g)	3.949	5.411	2.968	4.469	4.682	4.066
Freu	K _F (mL/g)	1.353	1.194	1.506	1.494	1.357	1.291
ndlich	S.E	0.487	0.347	0.449	0.473	0.441	0.362
	n _F	1.572	1.754	2.045	1.912	1.724	1.893
	R ²	0.977	0.947	0.871	0.900	0.990	0.954
Lan	K _L (ml/g)	0.019	0.017	0.019	0.020	0.018	0.019
gmuir.	S.E	0.257	0.313	0.279	0.269	0.282	0.309
coffi.	C _m (µg/g)	1000	333.3	500	500	500	333.3
	R ²	0.855	0.796	0.704	0.708	0.948	0.854

Table 4 : Adsorption of diazinon linear, Freundlich and Langmuir models isotherm parameters on the selected soil samples at 313.15 K.

Adsorp Models	Parame	Soils					
tion	ster	S ₁	S ₂	S ₃	S ₄	S ₅	S ₆
Ads	K _d (calc)	3.326	1.757	2.299	2.585	3.019	1.719
.Distr.	S.E	0.141	0.399	0.602	0.369	0.725	0.335
coffi.	R ²	0.712	0.741	0.741	0.812	0.671	0.756
Freur	K _F (mL/g)	1.363	0.865	1.067	0.747	1.096	0.805
ndlich	S.E	0.346	0.249	0.284	0.317	0.335	0.248
	n _F	2.173	1.608	1.773	1.261	1.628	1.531
	R ²	0.777	0.934	0.957	0.952	0.917	0.940
Lan	K _∟ (ml/g)	0.016	0.011	0.017	0.007	0.015	0.015
gmuir.	S.E	0.316	0.343	0.333	0.321	0.317	0.345
coffi.	C _m (µg/g)	333.3	250	250	500	333.3	200
	R ²	0.745	0.761	0.879	0.776	0.778	0.905

Table 5 : Free energy change, standard entropy change and standard enthalpy change at three temperatures for adsorption of diazinon on the selected soil samples.

Temp(Parame	Parame				Soils		
\$	eter	S ₁	S_2	S ₃	S ₄	S_5	S ₆	
283.1	ΔG	0.700	6.067	6 504	0.457	10 617	5 000	
Οī	(KJ/ITIOI	-8.780	-0.007	-0.384	-9.457	-10.017	-0.888	
	ΔS							
	(J/mol. K)	-66.260	-190.55	-135.57	-113.60	-65.600	-169.89	
298	ΔG							
5.15K	(kJ/mol	-9.824	-10.100	-9.824	-9.697	-9.958	-9.824	
	ΔS							
	(J/mol. K)	-66.429	-194.48	-139.62	-108.69	-59.872	-174.56	
313	ΔG							
3.15K	(kJ/mol	-10.766	-11.742	-10.608	-12.918	-10.934	-10.934	
	ΔS(J/							
	mol. K)	-66.254	-190.42	-135.43	-113.77	-60.119	-169.74	
	H (kJ/mol)	-9.9812	-47.887	-31.803	-22.709	-7.892	-42.219	
	R ²	0.996	0.924	0.836	0.839	0.816	0.872	

Table 6: Adsorption of diazinon in the presence of 40ppm of copper and desorption in the presence of TritonX-100 at cmc concentration, the linear, Freundlich and Langmuir models isotherm parameters on the selected soil samples.

Models		Parame	Soils					
		ster	S ₁	S ₂	S ₃	S_4	S ₅	S ₆
(ad	lir	K _d (calc)	9.454	4.809	6.623	8.578	6.274	5.410
sorptic	iear.Di coffi	S.E	0.257	0.131	0.291	0.372	0.143	0.118
on)	istr.	R ²	0.766	0.750	0.697	0.733	0.762	0.766
	F	K _F (mL/g)	1.553	1.328	1.599	1.656	1.340	1.198
	reund	S.E	0.569	0.424	0.487	0.544	0.482	0.452
	lich cc	n _F	1.689	1.727	2.137	2.045	1.567	1.441
	offi.	R ²	0.991	0.993	0.890	0.853	0.955	0.907
		K _L (ml/g)	0.017	0.017	0.023	0.015	0.011	0.008
	angm	S.E	0.231	0.287	0.267	0.245	0.265	0.275
	uir. co	C _m (µg/g)	1000	500	500	1000	1000	1000
	ffi.	R ²	0.987	0.945	0.766	0.713	0.941	0.820
(des	Des	K _d (calc)	8.058	3.739	3.729	4.532	4.371	4.001
sorptic	.Distr.	S.E	0.165	0.630	0.583	0.620	0.465	0.349
on)	coffi	R ²	0.932	0.874	0.968	0.979	0.979	0.975
	Ē	K _{Fdes} (mL/g)	1.207	0.921	0.842	0.865	0.857	0.800
	reundl	S.E	0.566	0.404	0.426	0.607	0.436	0.414
	ich cc	n _F	1.348	1.328	1.248	1.193	1.191	1.166
	offi.	R ²	0.982	0.983	0.991	0.990	0.996	0.998
	Lan	K _L (ml/g)	0.011	0.011	0.010	0.005	0.005	0.005
	gmuir.	S.E	0.138	0.219	0.200	0.173	0.189	0.201
	. coffi.	C _m (µg/g)	1000	500	500	1000	1000	1000
		R ²	0.767	0.963	0.731	0.636	0.851	0.989

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Table 7 : Hysteresis effect for Adsorption of diazinon in the presence of 40ppm of copper and desorption in the presence of TritonX-100 at cmc concentration on the selected soil samples.

S	Diazinon					
Oil	H ₁	ω	λ			
S ₁	1.252	25.3	-22.3			
S_2	1.300	30.1	-30.7			
S ₃	1.712	71.2	-47.3			
S_4	1.714	71.4	-47.8			
S_5	1.316	31.6	-36.0			
S ₆	1.236	23.6	-33.2			



Figure 1 : Variation of log K_L with 1/T for adsorption of diazinon on the six soil samples (\blacklozenge S₁, \blacksquare S₂, \blacktriangle S₃, x S₄, * S₅ \bullet S₆).





Figure 2 : Fitted models for diazinon adsorption (a) Linear(b) Ferundlich (c)Langmuir, isotherms on selected soil samples (\blacklozenge S₁, \blacksquare S₂, \blacktriangle S₃, x S₄, * S₅, \blacklozenge S₆).



Figure 3 : Fitted Ferundlich model for diazinon alone in presence of 40ppm of copper (a) adsorption (b) desorption in presence of nonionic surfactant, isotherms selected soil samples (♦ S₁, ■ S₂, ▲ S₃, x S₄, * S₅, ●S₆).

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Search engines for most searches, use Boolean searching, which is somewhat different from Internet searches. The Boolean search uses "operators," words (and, or, not, and near) that enable you to expand or narrow your affords. Tips for research paper while preparing research paper are very helpful guideline of research paper.

Choice of key words is first tool of tips to write research paper. Research paper writing is an art.A few tips for deciding as strategically as possible about keyword search:



- One should start brainstorming lists of possible keywords before even begin searching. Think about the most important concepts related to research work. Ask, "What words would a source have to include to be truly valuable in research paper?" Then consider synonyms for the important words.
- It may take the discovery of only one relevant paper to let steer in the right keyword direction because in most databases, the keywords under which a research paper is abstracted are listed with the paper.
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Keywords are the key that opens a door to research work sources. Keyword searching is an art in which researcher's skills are bound to improve with experience and time.

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References	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring

INDEX

Α

adimentionelle \cdot 9 aromaticum \cdot 18, 20, 21

D

diacylglycerol · 7 downtrend · 4

Ε

 $ehnomedicine\cdot 22$

F

Freundlich · 9, 11, 14, 16, 23, 24, 25, 26, 30, 31, 32, 34

L

Langmuir · 9, 11, 13, 14, 23, 25, 26, 30, 31, 32, 34, 36

Μ

margarines · 2, 6

Ν

nucleation · 2, 4, 5

0

Olowoyeye · 20 organophosphates · 23

Ρ

perchloric · 18, 20

T

triglycerides \cdot 2



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