

Research Paper

Fate and transport of paraquat dichloride in corn and rubber plantation soils

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ABSTRACT

The rubber and corn plantation soils in Nan province were investigated for the fate and transport behavior of the paraquat dichloride including biodegradation, adsorption, photolysis and infiltration. All experiments were conducted in the batch scale in laboratory basis. Results showed the biodegradation and photolysis were the main factors in decreasing the paraquat dichloride concentrations in the corn soil and rubber soil. The adsorption and infiltration described the holding and the leaching of paraquat dichloride of the soils. The biodegradation rates of paraquat dichloride in corn and rubber soils were 0.0548 and 0.0213 kg/day-mg, respectively. In photolysis reaction, the kinetic rate constants for corn and rubber soils were 0.017 and 0.0133 kg/min-mg, respectively. Corn and rubber soils had strong and quick adsorption for paraquat dichloride due to the soil textures of both two soils are clay and the high amount of cation exchange capacity. Corn soil had higher adsorption rate constant than rubber soil. The adsorption behavior of paraquat dichloride in rubber soils is best described by Freundlich isotherm and that in the corn soil is best describe by Langmuir-II isotherm. The kinetic rate constants for photo-degradation of paraquat dichloride in corn and rubber soils were 0.0170 and 0.0133 kg/mg-min, respectively. From the mass balance, paraquat dichloride in leachates was detected in the lower amount than in the soil for both corn and rubber soils. Although paraquat dichloride can bound to organic matter or soil particles and adsorb in the soil, small amount of paraquat dichloride can be leached out to the groundwater.

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1. Introduction

Thailand is a tropical country in the peninsula of Southeast Asia covered with a tropical monsoon climate. Thailand has been known as agricultural country and one of the world's food exporter as corn, rubber, cassava, rice and tropical fruit. Currently, the crop production is threatened by pests and unwanted grass. Around 50 percent of the crops are lost to pests in the plantation without crop protection strategies (Oerke EC., et al., 2014). To increase crop production levels, product quality, and product appearance, Thai farmers strongly rely their products on the use of the pesticides. Recording to Office of Agriculture Regulation (OAR) 70,000 tons of pesticides comprised of 265 individual active ingredients was imported into Thailand in 2010. These pesticides were around 75% of the total amount imported (Panuwet, P., et al., 2012).

Nan Province is one of the main agricultural provinces for the national agricultural production. In 2012, the agricultural area was covered 19.72 % of Nan province area (180,800 ha). Mostly 53% of the agricultural area in Nan province was covered with the agricultural plants such as corn, tobacco, bean and cassava. Otherwise, the paddy field, the perennial plant (rubber), and vegetable and flower plants were 20%, 19%, and 0.27% respectively. From 2002 to 2012, the agricultural area increased 1,104 ha and most of it was corn and rubber plantation. At the same time, the paddy field decreased 10% of the agricultural area. Around 59% of 157,177 families in Nan Province are the farmers. Nan province is the high land that the pesticides needs are large among for many steps of corn growing. The corn plantation required 82% of the amount of total pesticides imported to Nan Province (Kitchaicharoen, J., et al., 2015). In 2011, herbicides, insecticides, and fungicides were applied on the agricultural field 1,172.7, 44.8 and 56.6 tons respectively. Herbicides made up the largest proportion of applied pesticides in province which is 92% of total applied pesticides. They are applied on the plantation to kill the unwanted grass, although they effect to the ecosystem and living organisms.

Paraquat dichloride (1, 1'-Dimethyl-4, 4'-bipyridinium dichloride) is a well-known kind of herbicides which is widely used in Nan Province and over agriculture area in Thailand. Paraquat dichloride is extensively used for several applications including weed control on orchard floors, pre plant weed killers for many crops, pre harvest desiccants on crops and aquatic weed control (Chuntib, P., et al., 2015). Although paraquat dichloride is available on the market, easy to use, many adverse health effects and environmental impacts have resulted from paraquat dichloride application. Paraquat dichloride is not only destroys targeted weeds and pests, but it also

contaminates soil, water and air as well as damages the surrounding ecosystem and other living organisms necessary for maintaining ecological balance, for example, insects, birds, worms, fishes etc. (Wongwichit, D., et al., 2012). paraquat dichloride is toxic to human and a cause acute poisoning and even death after human receive in high among of paraquat dichloride. It is also well-known to be the cause diseases of the liver, lung, and heart. It also kills algae, fish, and other aquatic organisms including crayfish and insects (Saad, B., et al., 1997).

This research described the available body of information concerning the fate and transport of paraquat dichloride. The biodegradation, adsorption, photolysis, and infiltration of paraquat in the corn and rubber plantation soils from Nan Province were intensively investigated. The adsorption isotherms and the kinetic rates also calculated and discussed.

2. Materials and methods

2.1 Materials

Paraquat dichloride with purity 27.6% (w/v) was purchased from AG-GRO (Thailand) Co.,Ltd, Thailand, calcium chloride (CaCl_2), sodium sulfate anhydrous (Na_2SO_4) were lab grade and obtained from Fisher Chemical. Hexane (C_6H_{14}) was analytical grade and obtained from RCI Labscan. All chemicals were used as received.

2.2 Soil sampling

Corn and rubber plantation were collected in Nan province. The coordinate is E700750 and N2091353 for rubber plantation soil. The corn plantation soil was taken at the coordinate E700606 and N2091353. The 20 depth of the soils were dug in each plantation. Then, the roots and rocks were removed. The soils were taken into the plastic sacks. They were brought to the laboratory. Table 1 shows the properties of both corn and rubber soils.

2.3 Methods

2.3.1 Biodegradation procedure

The biodegradation method is modified from Siripattanakul. (Siripattanakul, S. et al., (2009). The sieved soils were weighted $1557 \pm 0.2\text{g}$ for the rubber soil and $1550 \pm 0.2\text{g}$ for the corn soil. Both soils were put into the $23.5 \times 16 \times 8\text{ cm}$ plastic reactor. The sterile ID water was added to the soils and mix them to increase the moisture content around 30 to 40% (w/w) which is

Table 1. The soil properties of corn and rubber soils.

Source of soil sample	% Clay	% Sand	% Silt	Texture	Bulk density (g/cm)	% Nitrogen	Potassium (mg/kg)	Phosphorus (mg/kg)	% Ca	CEC (meq/100g)	% Organic content	Soil pH
Corn	70.26	8.86	20.88	Clay	1.15	0.1	17.62	11.99	0.004	35.4	4.74	5.4
Rubber	83.15	3.01	13.84	Clay	1.19	0.11	22.88	6.45	0.004	26.7	3.23	4.3

suitable for bacteria growth. Then, the 16 x 23 x 9.5 cm plastic reactor was cover the polyethylene film and leave them in the dark chamber in the room controlled the temperature at 25°C for 12 hours to let the bacteria growing. A plastic container with the water was put into the chamber to reduce moisture content loss in the soil.

After 12 hours, 100 mL of sterile DI water as the control and paraquat dichloride solution with the concentration in the range of 100 to 1,000 mg/L were poured into the soil and mixed them homogenously. The $15 \pm 0.02 \times 2$ g of soil samples were used for the paraquat dichloride extraction, 1 ± 0.01 g of soil samples were applied for the bacteria count and 3 ± 0.3 g of soil samples were used for moisture content measurement. The samples were taken for analysis during time interval for 33 days. The extracted solutions were measured with UV spectrometer in the wavelength 255 nm.

2.3.2 Adsorption procedure

The adsorption of paraquat dichloride in corn and rubber plantation soils from Nan Province was investigated using a batch experiment technique (Chefet, B., et al., 2004). The corn and rubber soils were dried and sieved with the 2 mm diameter in the room temperature. Adsorption was initiated by mixing 0.5 g of air-dried soil sample with 11.25 mL of 0.01 mole/L CaCl_2 in 50 mL brown bottles. Then, the brown bottles were shaken with the slurries for 12 hours. Solutions with different initial paraquat dichloride concentrations in the range of 10-80 mg/L were prepared in 0.01 mole/L CaCl_2 and added into the brown bottles. The slurries were mechanically shaken with 120 rpm at the room temperature $28 \pm 2^\circ\text{C}$. The soil samples in the brown bottles were taken for analysis during time interval. The soil slurries were filtrated with the G/FC 47 mm filter to obtain the liquid sample which further measured by UV spectrometer in the wavelength 252 nm.

2.3.3 Photolysis procedure

The photolysis experiment was modified from Xiaozhen. (Xiaozhen, F., et al., 2005) This experiment set was conducted under the strong radiation of the sun at Naresuan University (16.7483°N , 100.1923°E) from 1

PM to 3 PM. The aluminum tray (40 x 40 x 4 cm) containing the 1,200 gram of paraquat dichloride contaminated soils were used for this experiment. The 300 mL with 50 - 4,000 mg/L of paraquat dichloride solution was poured into the soils and mixed homogeneously. The tray was covered with polyethylene film to prevent the volatilization of paraquat dichloride and to let the sunlight passing through the film and get into the contaminated soils. For sample analysis, 15 ± 0.02 g were weighted for extraction and then, the extracted solutions were measured with UV spectrometer in the wavelength 255 nm.

2.3.4 Infiltration procedure

The infiltration method is adapted from Neurath. (Neurath, S. K., et al., 2004) The corn and rubber plantation soils were sieved through less than 2 mm screen and dry at the room temperature. The soils were packed into the body of the columns with an inner diameter of approximately 2.6 cm and 50 cm long. The soil depth in the column was 40 cm. The stock solution of 1,000 mg/L paraquat solutions were prepared in DI water. 0.560 mL of the solution was added onto the surface of the soil in the column. The 0.01 M of the calcium chloride drops to the column with the rate 18 mL/hour in 24 hours. The liquid samples that pass through the soil layer will be taken at 2, 4, 6, 8, 10, 13, 15, 24, and 48 hours. At the end of the experiment, every 5 cm depth of the soil layers were extracted to determine the paraquat dichloride residue in the soil layers. Then, the extracted solutions were measured with UV spectrometer in the wavelength 255 nm.

3. Results

3.1 Biodegradation result

The Plate Count Agar method was commonly used for counting the number of colony forming bacteria present in the soil. The total indigenous bacteria in the soil had been counted and plotted by logarithmic formational values, shown in CFU (colonies forming unit)

per one gram. The paraquat dichloride concentration was also observed and the moisture content was kept around 25-40% (dry soil).

According to **Fig. 1**, it shows that the biodegradation process in the soil took 17 days to decay paraquat dichloride in corn soil. The initial paraquat dichloride concentration in the corn soil was detected between 0.49 - 1.64 mg/kg. After 17 days, the paraquat dichloride concentrations were totally stable. The percentages of paraquat removal were around 61 - 81%. After plotting, the biodegradation rate range was between 0.0028-0.0620 day⁻¹.

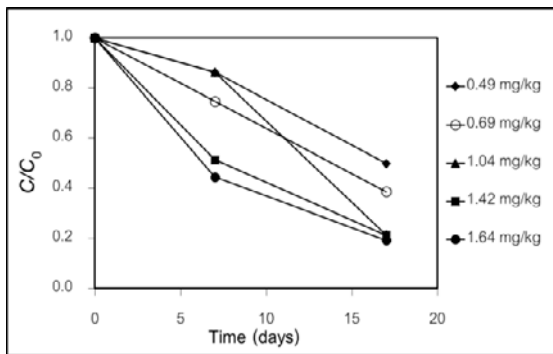


Fig. 1. Paraquat dichloride residue from biodegradation of corn soil.

Fig. 2 shows the microbial process in decreasing of paraquat dichloride during 17 days for rubber soil. The paraquat dichloride sharply decreased. The paraquat dichloride concentrations in the soil were ranging from 0.10-1.80 mg/kg. After 17 days, paraquat dichloride concentration stopped decrease. The percentage of paraquat dichloride removal was around 47-94%. Finally, the biodegradation rates are ranging from 0.0341 to 0.065 day⁻¹.

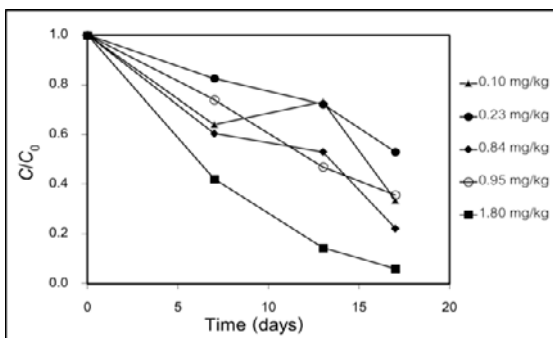


Fig. 2. Paraquat dichloride residue from biodegradation of rubber soil.

From **Fig. 3**, The biodegradation rates of paraquat dichloride for corn and rubber soils were 0.0548 and 0.0213 kg/day-mg, respectively.

Fig. 4 shows the results of a number of colonies as the logarithmic enumeration in corn soil with paraquat

dichloride treatment. Initially, the microbials in the soils were around 0.65 to 1.5 million CFU/g. It clearly shows that the growth of microbial colonies in each condition are in similar amount. The growth of total colonies in all paraquat dichloride concentration tended to be progressive dramatically within 7 days. However, the microbial colonies in the concentrations in the range of 1.04 and 1.64 mg/kg are slowly increased in population. Triplically, 17 days were required for bacteria growing up. Beyonds this point, the growth of colonies tended to be stable. At the end, the microbial colonies in corn soil were around 4 to 8.1 million CFU/g.

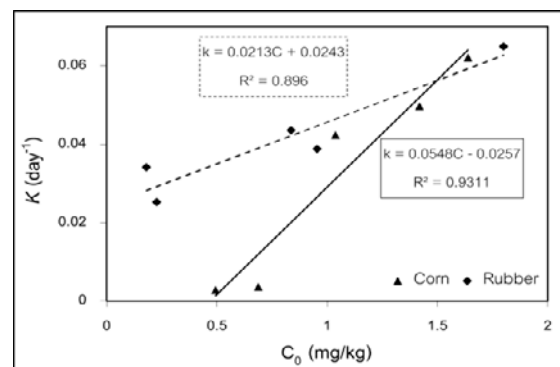


Fig. 3. The kinetics of the biodegradation of paraquat dichloride of corn and rubber soil.

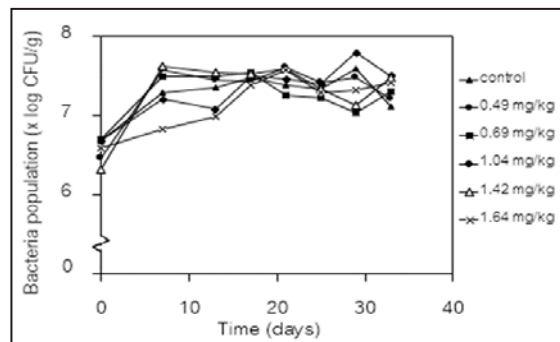


Fig. 4. Colonies of bacteria in the biodegradation experiment of corn soil.

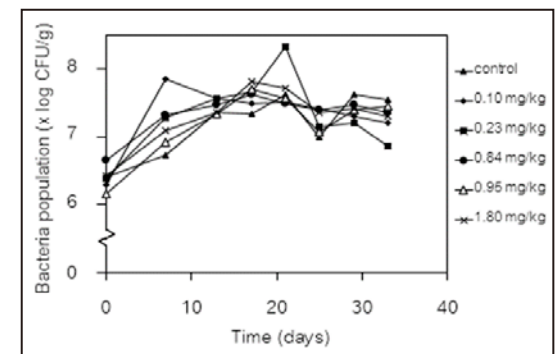


Fig. 5. Colonies of bacteria in the biodegradation experiment of rubber soil.

Fig. 5 shows the bacteria colonies in log CFU/g in rubber soil. It totally shows that all numbers of colonies were the relatively same growth at the varied time. The colonies at the initial time were around 0.45 to 0.14 million CFU/g. The microbial colonies in all concentrations in rubber soil tends to increase with 17 days. After 17 days they tended to decrease slowly. The number of microbial colonies at the end of experiment were around 5.0 to 11.1 million CFU/g.

3.2 Adsorption result

The adsorption of paraquat dichloride of corn and rubber soil occurred very quickly within only 2 minutes after starting the experiment. By plotting solid-phase concentration with residual liquid-phase concentration graphically, the equilibrium adsorption isotherms were described. In order to optimize the design of a sorption system to remove paraquat dichloride from CaCl_2 solution, it is necessary to establish the most appropriate correlation for the equilibrium curve. (Ncibi, M. C., et al., 2008). The adsorption isotherms were determined as linear, non-linear, Freundlich and Langmuir II.

As seen in table 2, it was observed that the linear isotherm parameters with K_d for the corn and rubber soils were 3,398.8 L/kg and 1,122.2 L/kg respectively. The K_{OC} values depended on the organic carbon in the soils were 34,717.1 L/kg and 26,404.7 L/kg for corn and rubber respectively.

Tab. 3 shows the Freundlich non - linear isotherm parameters such as the correlation coefficient (R^2), Freundlich constant K_F , and Freundlich exponent $1/n$. the K_F values are 5,371.0 and 1,438.4 L/kg for corn and rubber soils. The n values are 0.639 for corn soil and 1.412 for rubber soil.

Tab. 4 shows a high correlation between the adsorbed concentration in the soil and the adsorbed concentration in the solution which confirm a better fit to Freundlich isotherm than to linear isotherm. K_F value are 5,372 and 1,438 L/kg for corn and rubber soils. For n value, it was calculated resulting 0.64 for corn soil and 0.84 for rubber soil.

Additionally, Tab. 5 is determined on Langmuir II with K_a , q_m and R^2 . The K_a are -1.286 L/kg for corn soil and -0.05 L/kg for rubber soil. For $1/q_m$ in corn soil = -0.0009 and in rubber soil = -0.00002, whereby q_m described the sorption capacity of atrazine sorption on the soil.

3.3 Leaching experiment with soil column result

According to potential paraquat dichloride leaching risk in the soil, the leaching experiment with soil columns had been done to observe to potential transformation and

movement (leaching into the deeper soil layers and eventually into the groundwater). The experiment was also investigated on the paraquat dichloride residue in the soils.

Fig. 6 shows the percentage of leached paraquat dichloride and volume of leachate of corn and rubber soils with 10 mg and 40 mg mass of paraquat dichloride in the columns. At the same conditions, leaching characteristics of paraquat dichloride for the conducted experiments are similar in the both soils and provided relative values. For 10 mg of sprayed paraquat dichloride, the mass of paraquat dichloride in the leachate was around 4.6 to 5.3 % by volume in the total amount of 313-405 mL of leachate during 30 hours experimental time. For 40 mg of sprayed paraquat dichloride, the mass of paraquat dichloride was around 2.8 to 3.8 % by volume for the total amount paraquat dichloride leachate in the total volume of 289-434 mL within 30 hours experimental time.

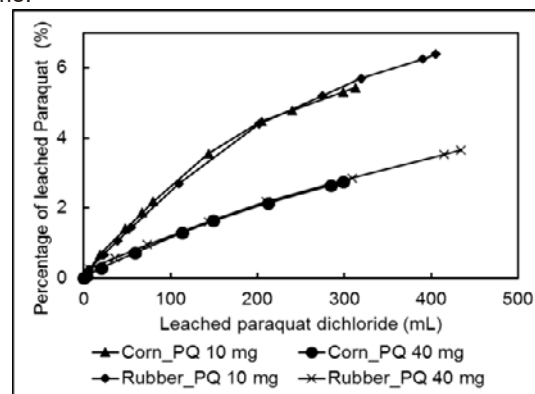


Fig. 6. The percent of Paraquat dichloride leaching and volume of water leaching.

Fig. 7 shows remaining of initial 10 mg of paraquat dichloride in varied corn and rubber soils layers. The concentration of paraquite dichloride in every 5 cm depth of column was determined. From the result, the paraquat dichloride was contaminated for 8 soil-layers of the whole column due to high solubility and strong adsorption of paraquat dichloride.

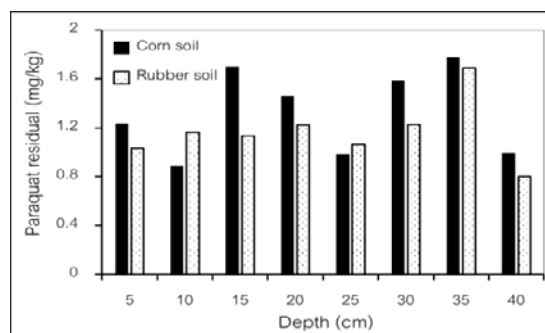


Fig. 7. The residue of 10 mg of paraquat dichloride in different layers of corn and rubber soils.

Table 2. The linear adsorption isotherms of Corn soil and Rubber soil.

Type	Equation	Graph equation	K_d	R^2
Corn	$q_e = K_d C_e$	$y = 3398.8x$	3398.8	0.8678
Rubber	$q_e = K_d C_e$	$y = 1122.2x$	1122.2	0.7991

Table 3. Non-linear adsorption isotherms of corn and rubber soils.

Type	Equation	Graph equation	K_F	1/n	n	R^2
Corn	$q_e = K_F C_e^{1/n}$	$y = 5371x^{1.566}$	5371.0	1.566	0.639	0.931
Rubber	$q_e = K_F C_e^{1/n}$	$y = 1438.4x^{0.7081}$	1438.4	0.708	1.412	0.930

Table 4. Freundlich adsorption isotherms of corn and rubber soils.

Type	Equation	Graph equation	1/n	n	$\log K_F$	K_F	R^2
Corn	$\log(q_e) = (1/n)\log C_e + \log K_f$	$y = 1.566x - 3.7301$	1.57	0.64	3.73	5372	0.931
Rubber	$\log(q_e) = (1/n)\log C_e + \log K_f$	$y = 0.7081x - 0.8421$	0.71	0.84	3.16	1438	0.953

Table 5. Langmuir II adsorption isotherms of corn and rubber soils.

Type	Equation	Graph equation	1/qm	1/(qm.ka)	K_a	R^2
Corn	$C_e/q_e = (1/q_m).C_e + (1/K_a.q_m)$	$y = 0.0007x - 0.0009$	-0.0009	0.0007	-1.286	0.943
Rubber	$C_e/q_e = (1/q_m).C_e + (1/K_a.q_m)$	$y = 0.0004x - 0.00002$	-0.00002	0.0004	-0.05	0.909

Fig. 8 shows remaining of initial 40 mg of paraquat dichloride in both soils and the concentrations of paraquat dichloride. Under the conditions, paraquat dichloride in both soils was transported into the deeper soil layers of the soil columns especially to the soil layer from 25 to 40 cm.

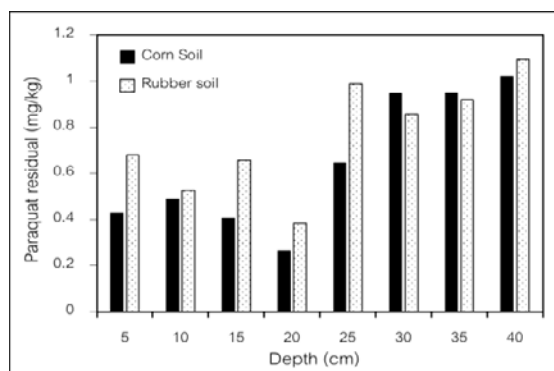


Fig. 8. The residue of 40 mg of paraquat dichloride in different layers of corn and rubber soils.

3.4 Photolysis of paraquat dichloride

Fig. 9 shows the photolysis of paraquat dichloride in corn soil. In this case, the initial paraquat dichloride concentrations prepared in corn soil are ranging in concentration from 0.11 to 0.70 mg/kg. The concentrations of paraquat dichloride tended to decrease within 20 minutes under the sunlight. Beyond this point, the concentrations of the paraquat dichloride are stable.

During 20 minutes photolysis, paraquat dichloride had been removed 33 to 54% of the total concentration of paraquat dichloride in corn soil. The photolysis rate constants are ranging from 0.0202 to 0.0306 min^{-1} for corn soil.

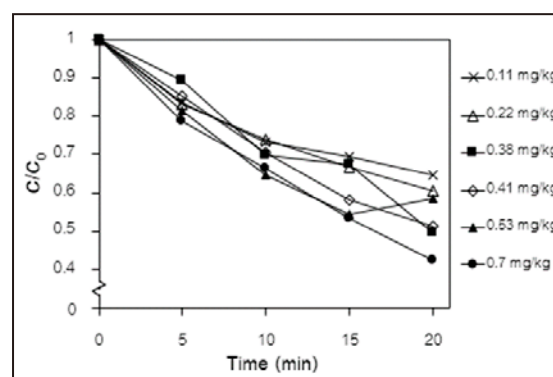


Fig. 9. The residue of paraquat dichloride from photolysis of corn soils.

Fig. 10 shows the photolysis of paraquat dichloride in rubber soil. The initial paraquat dichloride was in the range of 0.29 mg/g to 0.84 mg/kg in the rubber soil. The concentrations of paraquat dichloride tended to decrease within 20 min under the sunlight as same as corn soil. Beyond this point, the concentrations of the paraquat dichloride were stable. During photolysis, paraquat dichloride had been removed 35 to 48%. The photolysis rate constants are ranging from 0.016 to 0.023 min^{-1} .

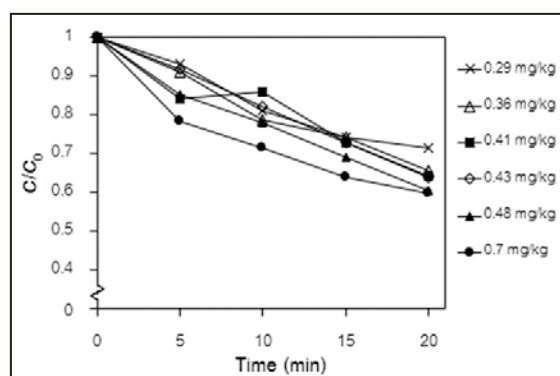


Fig. 10. The residue of paraquat dichloride from photolysis of rubber soil.

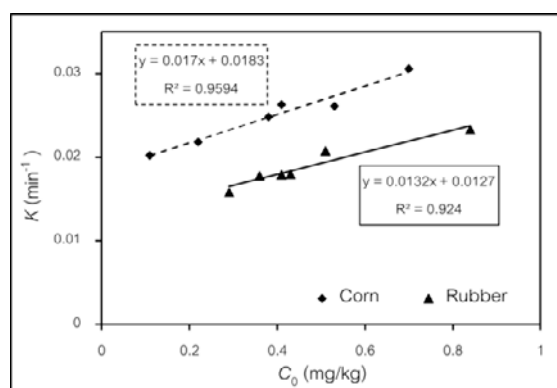


Fig. 11. The kinetics of the photolysis of paraquat dichloride of corn and rubber soils.

Fig. 11 shows the kinetics of the photolysis of paraquat dichloride of corn and rubber soils. After plotting the photolysis rate constants of corn and rubber soils, the photolysis rate constants increase with the increasing of paraquat dichloride concentration. The kinetic rate constants for corn and rubber soils were 0.017 and 0.0133 kg/min-mg, respectively.

4. Discussion

This current study shows that the biodegradation and photolysis are the main factors in decreasing the paraquat dichloride concentrations in the corn soil and rubber soil. The adsorption and infiltration described the holding and the leaching of paraquat dichloride of the soils.

4.1 Biodegradation discussion

From this work, the microbials have high tendency in consuming paraquat as food source. Results shows when microorganism growth in the soils stopped increasing, paraquat dichloride degradation stopped decreasing at the relative time. The microbials in both

paraquat dichloride contaminated soils have higher amount colonies than the control soils (without paraquat dichloride). The average microbial colonies in corn soil is trivially higher than in the rubber soil because of organic matter effects. Results from this work was in good agreement with the previous work (Tu, C., et al., 1968) which reported the detection a microbial degradation of paraquat dichloride in the soils. The carbon dioxide evolution as well as oxygen consumption was used as an index of metabolic activity of soil micro-organisms and paraquat dichloride had a stimulatory effect at higher concentrations. This is an evidence that ammonification of soil organic-matter nitrogen occurred in both soils.

4.2 Adsorption discussion

As the adsorption result, corn and rubber soils had strong and quick adsorption for paraquat dichloride due to the soil textures of both two soils are clay and the high amount of CEC (Cation Exchange Capacity) in both soils. Corn soil had higher adsorption rate constant than rubber soil because corn soil contains higher organic matter than rubber soil (Zabaloy, M. C., et al., 2011). The adsorption behavior of paraquat dichloride in rubber soils is best described by Freundlich isotherm which is the relationship between pressure and mass of paraquat adsorbed on surface of soils per unit of mass. The adsorption of paraquat dichloride in the corn soil is best describe by Langmuir-II isotherm.

4.3 Photodegradation discussion

From this work, the paraquat dichloride was possibly be degraded by the sunlight. In comparison, the kinetics rate of atrazine photodegradation in corn soil was slightly higher than that in rubber soil. The kinetic rate constants for photodegradation of paraquat dichloride in corn and rubber soils were 0.0170 and 0.0133 kg/mg-min, respectively. The percentage of clay in both soils played the major role to capture the pollutant in the soil. The results were in good agreement. (Zhen, et al., 2004) which reports that the photolytic rate in thin silt soil is higher than that in the coarse sand soil.

4.4 Infiltration discussion

During the infiltration process, paraquat dichloride was detected in both corn and rubber soils and water. Rarely low percentage of paraquat dichloride was detected in the leachates from both corn and rubber soils. From the mass balance, paraquat dichloride in leachates was detected in the lower amount than in the soil for both corn and rubber soils. Although paraquat dichloride can

bound to organic matter or soil particles and adsorb in the soil (Gevao. B., et al., 2000) (Paranjape. et al., 2015). So, small amount of paraquat dichloride can be leached out to the groundwater.

5. Conclusions

From investigating the behavior of the paraquat dichloride in corn and rubber soils, it indicates that the bacteria in the soils and the sunlight are the main factors that decrease paraquat dichloride. With strong adsorption of the soil, paraquat dichloride can bound to organic matter or soil particles and adsorb in the soil. Small amount of paraquat dichloride can be leached out to the groundwater.

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