Study of paraquat adsorption using different types of soil

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Title	Study of paraquat adsorption using different types of soil
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Field of Study	Petrochemical Engineering
Advisor	Asst. Prof. Dr. Nuttapol Lerkkasemsan

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Abstract

The paraquat is toxic agriculture chemical which is able to be found in crops, animal and water source. To reduce the paraquat in environment, the aim of this study is to compare the removal efficiency of paraquat from aqueous solution using bentonite and 3 levels of Lb-soil series including Lbtop, Lbmiddle and Lbbottom. The different types of soil were prepared to adsorb the paraquat solution at various concentration (10.78-345 mg/L). After the adsorption, the remaining concentration of paraquat solution was analyzed by UV-Visible spectrometry to calculate the amount of adsorbed paraquat on each soils. Then, the adsorption isotherm was constructed from the result of calculation. Finally, the results indicated that the order of adsorption efficiency of soils was depended on the concentration of paraquat solution. At low concentration of paraquat solution, the adsorption efficiency of soils sorted from high to low were bentonite, Lbmiddle, Lbtop and Lbbottom. At high concentration of paraquat solution, the adsorption efficiency of soils sorted from high to low were bentonite, Lbtop Lbmiddle and Lbbottom.

Keywords: Adsorption isotherm, Paraquat, Lb-soil series, Bentonite

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บทคัดย่อ

พาราควอตเป็นสารพิษจากการเกษตรซึ่งสามารถพบได้ในพืชสัตว์และแหล่งน้ำ จุดประสงค์ของการศึกษานี้คือเพื่อเปรียบเทียบประสิทธิภาพการกำจัดพาราควอตในน้ำโดยใช้เบนโทไนต์และชุดดินลพบุรีที่ระดับความลึก 3 ระดับได้แก่ ดินชั้นบน ดินชั้นกลาง และดินชั้นล่าง ในการทดลองดินชนิดต่างๆ จะถูกเตรียมเพื่อดูดซับสารละลายพาราควอตที่ความเข้มข้นต่างๆ (10.78 ถึง 345.00 มิลลิกรัมต่อลิตร) หลังจากนั้นความเข้มข้นที่เหลืออยู่ของสารละลาย พาราควอตจะถูกนำมาวิเคราะห์ด้วยเครื่องวิเคราะห์การดูดกลืนแสง เพื่อคำนวณหาปริมาณการดูดซับพาราควอตในดินแต่ละชนิด สุดท้ายผลการศึกษาพบว่าประสิทธิภาพการดูดซับของดิน ขึ้นอยู่กับความเข้มข้นของสารละลายพาราควอต กล่าวคือที่สารละลายพาราควอตความเข้มข้นต่ำ ประสิทธิภาพการดูดซับของการดูดซับพาราควอตของดินแต่ละชนิด สามารถเรียงลำดับได้ดังนี้ เบนโทไนต์ ดินชั้นกลาง ดินชั้นบน และดินชั้นล่าง ตามลำดับ แต่ที่สารละลายพาราควอตความเข้มข้นสูง ประสิทธิภาพการดูดซับของการดูดซับพาราควอตของดินแต่ละชนิด สามารถเรียงลำดับได้ดังนี้ เบนโทไนต์ ดินชั้นบน ดินชั้นกลาง และดินชั้นล่าง

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NOMENCLATURE

C_e = equilibrium solution concentration (mg/L)

C_o = maximum concentration of adsorbate that can exist in solution before precipitation (mg/L)

X = paraquat adsorbed per mass of soil or clay (mg/g)

X_m = maximum amount of paraquat that can be adsorbed (mg/g)

a = a term related to the free energy of transfer of adsorbate from the bulk solution to the surface of adsorbent

b = equilibrium constant of adsorption

k = rate constant of formation

n = order of formation

CHAPTER I
INTRODUCTION

1.1 Background

Nowadays bentonite is used as an adsorbent to reduce the amount of pesticide [1,2,3,4] in water and various plants. Bentonite is a volcanic rock that has been transformed into soil and has been immersed in water for 200 million years. Bentonite contains smectite or montmorillonite mineral group as a key component. Due to bentonite, when wet, can adsorb substances with its positive charge such as various pathogens so bentonite can effectively prevent the symptoms of rot in plants and soil such as fungal diseases, root rot, etc. In Thailand, there are 3 popular pesticides that are used in agriculture consisting of paraquat, glyphosate and chlorpyrifos. Paraquat is known as the trade name called "Grammoxone" which is the most popular herbicide in Thailand. The effect of paraquat when used with plants is growth inhibition of weeds especially the green part. In addition, it causes drying of the cell tissue without spreading into the wood tissue. There is not only bentonite that can adsorb paraquat but various soil also can adsorb too such as illite [3], sepiolite [3] and local soil [5]. The soil in Thailand can be divided into 20 great soil groups [6]. In order to classify the soil into different great soil groups, they are classified based on soil morphology such as the arrangement of the soil layer. It means that if materials origin, development of the soil profile and climatic conditions are similar, they will be arranged in the same great soil group. In each great soil group, there are further subdivisions called "soil series". In each soil series, it will be called as the name of the place where the soil was found for the first time for examples, Korat soil series, Lampang soil series, Yala soil series, Phuket soil series, etc.

In order to remove paraquat in environment, the study of paraquat adsorption efficiency on various soils including Lb-soil series and bentonite is the aim of this research.

1.2 Objective

1.2.1 To compare the removal efficiency of paraquat from aqueous solution using 3 levels of Lb-soil series and bentonite

1.3 Scopes of Work

1.3.1 Soil series that was brought to study is Lb-soil series which collects from Baan Pung Pattana, Kaeng Khoi district, Saraburi province.

1.3.2 Studying the paraquat adsorption process by using 3 levels of Lb-soil series and bentonite clay as adsorbents

1.3.3 Analyzing the results by using UV-visible Spectrophotometer

1.4 Expected Outputs

1.4.1 Understand the principles of paraquat adsorption in water by using 3 levels of Lb-soil series and bentonite as adsorbents

1.4.2 Can use this experiment to further develop to produce high-efficiency adsorbents for paraquat adsorption from animal feed such as pig feed and chicken feed

CHAPTER II
LITERATURE REVIEW

This chapter will cover the basic knowledge and theories related to this research, which consists of soil, pesticide, adsorption and related research. The detail are as follows.

2.1 Soil

2.1.1 Soil texture

In agronomy, the soil is classified into many types. It is determined by proportional of the mass of inorganic particles.

1) Sand: Sand or sand particles are classified as the largest group in the soil.

2) Silt: Silt or sediment sand particles or silt particles are classified as medium-sized groups.

3) Clay: Clay or clay particles are classified as the smallest group in the soil.

2.1.2 Characteristics of soil particles of each size group

2.1.2.1 Sand has the following characteristics.

1) A small grain of quartz crystal and mineral feldspar decomposed from the original stone.

2) Big size, visible to the naked eye (Except very fine sand) and hand irritation

3) Not agglomerate to form a soil and if there are no particles of other sizes, it will appear as single grain

4) The grain of sand when arranged together will form a large pore.

5) Good drainage and ventilated but low water retention

6) The sand particles group has a small specific surface area. Therefore it has a little surface to adsorb substances such as water and nutrients.

2.1.2.2 Silt has the following characteristics.

1) A group of medium particles

2) It has mineral composition like sand

3) The particles are small, invisible to the naked eye and slippery touch

4) Not agglomerate to form a soil like sand

5) The grain of silt when arranged together will form a pore which suitable for water retention

2.1.2.3 Clay has the following characteristics.

1) Clay particle often refers to secondary minerals that are synthesized from original minerals which have decayed and deposited in the soil.

2) The smallest particle group, invisible to normal microscopes, the particle is a sheet of aluminosilicates that are stacked together into a layer and touch when dry, will be hard and rough but if wet will be sticky and slippery

3) Clay particles, when arranged into a lump of soil, will create a small gap between the particles and have a large total volume of gap.

4) Because the clay has a high surface area and non-neutral particles. Therefore clay particles can adsorb various substances well.

2.1.3 Soil taxonomy

According to the soil taxonomy, soil classification is to divide the soil into different levels which consist of [7]

1) Order
2) Suborder
3) Great soil group
4) Sub soil group
5) Family
6) Soil series

2.1.4 Lb-soil series

Table 2.4: Properties of Lb-soil series [6]

Soil series	Lb (Lop buri)
Soil classification (USDA)	Very-fine, smectitic, isohyperthermic Typic Haplusters
Area condition	Quite smooth
Morphology	Riverbed
Source of soil	Soil sediment was deposited on the marl courtyard on a limestone mountain
Drainage	Medium to good
Water permeability	Slow
Afflux of water on soil surface	Slow to medium
Soil characteristics	A deep clay, top level of clay has black color or dark grey color, reaction of top level of clay is slightly acidic to medium alkaline (pH 6.5-8.0), bottom level clay has black color of very dark grey color up to grayish brown color, marl layer was found at a depth of 80 cm from the ground, reaction of bottom level of clay is medium alkaline to extremely alkaline (pH 8.0-9.0), It will split into grooves greater than 1 cm or more than at a depth of 50 cm in the dry season and soil surface has the nodular mass of lime which is accumulated everywhere.

Lb-soil series can be divided into 3 levels which depend on how depth from the soil surface consist of Lb top, Lb middle and Lb bottom.

Figure 2.2: Cross-section of Lb-soil series [6]

Table 5.2: Properties of Lb top, Lb middle and Lb bottom [6]

Lb-soil series	Lb top	Lb middle	Lb bottom
Depth (cm.)	0-25	25-50	50-100
Organic matter	high	high	high
CEC	high	high	high
Base saturation	high	high	high
Available Phosphorus	high	medium	high
Available Potassium	high	high	high
Soil fertility	high	high	high

2.1.5 Bentonite

Bentonite is a clay mineral that has high swelling properties. There are 2 types of bentonite that are Na-bentonite and (Ca-bentonite). Sodium bentonite, after adsorbing water, is able to swell 15-20 times more than its original volume. Sodium bentonite has lubricating properties and prevent spreading by commonly used as mud drilling head. Calcium bentonite has the ability to swell less than sodium bentonite, therefore is commonly used as a bleaching agent detergent. In addition, it can be modified by reacting with acids to get activated clays which can be used as catalysts or react with caustic soda to change calcium bentonite to sodium bentonite.

In geology, bentonite belongs to the family of soil minerals. There are proportions of the elements that vary according to the source. It creates unique physical and chemical characteristics, mainly from volcanoes.

In mineral resources, bentonite mainly consists of montmorillonite and has other minerals mixed in such as feldspar, calcite, silica, gypsum, etc. The physical structure of montmorillonite is a group of scales or crystals which has a wide base plane. The difference is in the range of 0.2-2.0 µm and thickness at 6-10 µm. In reality characteristics and formation of crystals can be different according to the origin and the specific characteristics of the mineral source.

Table 2.3: Composition of bentonite

Composition of bentonite	%w
SiO_2	69.00-74.55
Al_2O_3	11.22-14.99
CaO	0.44-0.51
MgO	2.53-2.70
K_2O	1.62-2.96
Na_2O	3.03-3.19
Fe_2O_3	2.50-2.70
FeO	0.02-0.04
MnO	0.00
TiO_2	0.11-0.15
P_2O_5	0.05-0.08

2.2 Pesticide

2.2.1 Pesticide

Pesticide are substance that are widely used today. Pesticides can be divided into many types which can be divided as follows.

2.2.1.1 Classified by internal and external activate of plant cells

1) Contact pesticide or non-systemic pesticides: This type of substance is coated on the surface of the plant cells and activate by contact with the target group.

2) Systemic pesticide: This type of substance will activate by absorbed into the plant cells.

2.2.1.2 Classified by a group of target

1) Insecticide: A substance use to inhibit growth or kill insects.

2) Fungicide: A substance use to prevent, destroy or stop the growth of fungi in plants.

3) Herbicide: A substance use to prevent or kill weeds.

4) Others such as rodent slaughter killing substance, nematode killing substance, etc.

2.2.1.3 Classified by structure and chemical composition of substance

2.2.2 Herbicide

Herbicide means any chemical substance which use to kill or inhibits the growth of weeds. Herbicide can be classified according to a basic chemical structure by using the characteristics of molecular structure and the position of atoms of elements or elements within the same molecule. It can be divided into 2 groups as follows.

2.2.2.1 Inorganic herbicide: An herbicide that no carbon atoms in the molecule such as ammonium sulfamate (AMS), copper sulfate, metaborate, sodium chlorate, etc.
In most cases, substances in this group are used to kill weeds in areas that do not grow plants such as roads, forests or water weeds like algae.

2.2.2.2 Organic herbicide: An herbicide that contains at least 1 atoms of carbon in the molecule as a component. In general, the molecules of organic consist of 12 different elements. The most common elements are carbon (C), Hydrogen (H) and oxygen (O). Some other elements that may be found are nitrogen (N), sulfur (S), phosphorus (P) and elements in the halogen group such as fluorine (F), chlorine (Cl), bromine (Br) and iodine

(I). Main structure of organic herbicide has an arrangement of atoms of carbon and other substances. Organic herbicide can divided into 2 types which consist of

1) Aliphatic hydrocarbons: The structure of molecules that are arranged atoms of carbon into a chain, linear or branching

2) Aromatic hydrocarbons: The structure of molecules that are arranged atoms of carbon into a ring-shaped.

2.2.3 Paraquat

Paraquat is an herbicide in the group of bipyridiliums used for spraying after weeding. Paraquat is classified as contact pesticide or contacts-membrane disrupters. Paraquat has many trade names such as Grammoxone, TriQuat or Dex Suron. Paraquat is a dangerous substance that is used to prevent and kill weeds. Paraquat has high solubility.

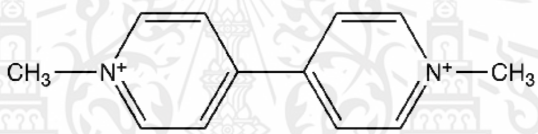

Figure 3.2: Structure of paraquat [8]

Table 2.4: Properties of paraquat

Molecular formula	$C_{12}H_{14}N_2^{+2}$
Molecular weight	186.25
Physical description	Off-white powder, odorless, hygroscopic solid
Density	1.24 g/cm^3
Boiling point	175-180°C
Melting point	300°C
Flash point	Nonflammable
Solubility	700,000 mg/L at 20°C
Vapor pressure	0 mmHg at 20°C

2.3 Adsorption

2.3.1 Adsorption mechanism

The adhesion or adsorption consist of 3 mechanisms, which are electrostatic mechanism, chemical mechanisms and physical mechanisms. Adsorption mechanism can be divided into 3 steps as follows.

1) External diffusion: External diffusion is a mechanism by which the molecules of the adsorbent reach the adsorbent. The surface of the adsorbent is liquid, which is wrapped with molecules and inserted through the layer of liquid to the surface of the adsorbent.

2) Internal diffusion: Internal diffusion is a mechanism by which the molecules of the adsorbate spread into the surface area of the adsorbent cavity for absorption.

3) Surface reaction: Surface reaction is a mechanism by which the molecules of the adsorbate are absorbed on the surface of the adsorbent, which is a very fast process. When comparing with the diffusion process, the resistance from surface reactions should also be considered.

2.3.2 Adsorption isotherm

Adsorption isotherm is the graph that shows the relationship between the amount of adsorbate and the concentration of the substance at a balanced point at a constant temperature. The most commonly used adsorption isotherms are Langmuir isotherm, Freundlich isotherm and Brunauer-Emmett-Teller (BET) isotherm.

2.3.2.1 Langmuir isotherm: It suitable to use for monolayer case.

$$X = \frac{bX_mC_e}{1+bC_e} \tag{2.1}$$

where, X = amount of adsorbate which is adsorbed per amount of adsorbent (mg/g)

C_e = equilibrium solution concentration (mg/L)

b = equilibrium constant of adsorption

X_m = maximum amount of adsorbate which is adsorbed per amount of adsorbent (mg/g)

2.3.2.2 Freundlich isotherm

$$X = kC_e^{n} \tag{2.2}$$

where, X = amount of adsorbate which is adsorbed per amount of adsorbent (mg/g)

C_e = equilibrium solution concentration (mg/L)

k = equilibrium constant of adsorption

n = constant from experiment

2.3.2.3 Brunauer-Emmett-Teller (BET) isotherm

$$X = \frac{aC_oC_eX_m}{[C_o+(a-1)C_e](C_o-C_e)} \tag{2.3}$$

where, X = amount of adsorbate which is adsorbed per amount of adsorbent (mg/g)

C_e = equilibrium solution concentration (mg/L)

X_m = maximum amount of adsorbate which is adsorbed per amount of adsorbent (mg/g)

C_o = maximum concentration of adsorbate that can exist in solution before precipitation (mg/L)

a = a term related to the free energy of transfer of adsorbate from the bulk solution to the surface of adsorbent

2.4 Literature review

D. Ait Sidhoum and group [1] studied the sorption–desorption of the cationic pesticide 1,1′-dimethyl-4,4′-bipyridinium dichloride (paraquat) on a bentonite from Maghnia (Algeria) desiccated at 110 °C (M), and calcined at 400 °C (M400) and 600 °C (M600) from aqueous solution at 25 °C by using batch experiments. The results show that the sorption capacity of the calcined samples greatly decreased with heat treatment. On the other hand, the sorption process is hardly affected by theworking temperature, whereas the higher electrolyte concentration, the lower sorption of this pesticide.

L. Hiera Da Cruz and group [2] studied 1.Adsorption of glyphosate (GPS) on clays and soils in different pHs and concentration of $CaCl_2$ and 2.Competitive adsorption between glyphosate (GPS) and inorganic phosphate (Na_2HPO_4) on clays and soils in different pHs. The results show that the adsorption of glyphosate (GPS) depends on surface area for clays and amount of clays and CEC for soils. Organic matter (OM) had a secondary role in the adsorption of GPS on soils. The adsorption of GPS on soils from Londrina and Floraí counties and clays (montmorillonite, kaolinite) decreased when pH increased, however, for bentonite clay and soil from Tibagi county was kept constant. For the soils, the competitive adsorption between GPS and phosphate showed that displace of GPS by phosphate was related to the amount of clays, CEC and pH. GPS was not easily displaced by phosphate on the clays.

Y. Seki and K. Yurdakoç [3] studied the comparable of sorption of paraquat from aqueous solution using clays and organoclays. Sepiolite (S), bentonite (B), and illite (I) were used as clay samples. Organoclays were prepared by the modification of the clays with nonyl- and dodecylammonium chlorides, denoted as NS, DS, NB, DB, NI, and DI, respectively. The result show that the adsorption data indicated that illite and NI are the most effective adsorbents among these clays and organoclay samples, respectively.

H. Muhamad and group [5] studied the adsorption equilibrium time and effects of pH and concentration of ^{14}C-labeled paraquat (1,1′-dimethyl-4,4′-bipyridinium dichloride) in two types of Malaysian soil which consist of clay loam and clay soil from rice fields. The result show that adsorption equilibrium time was achieved within 2 h for both soil types. The amount of ^{14}C-labeled paraquat adsorbed onto glass surfaces increased with increasing shaking time and remained constant after 10h. The paraquat adsorbed by the two soils was very similar: 51.73 (clay loam) and 51.59 µg/g (clay) at 1 µg/mL. The adsorption of paraquat onto both types of soil was higher at high pH and adsorption decreased with decreasing pH. At pH 11, the amounts of ^{14}C-labeled paraquat adsorbed onto the clay loam and clay soil samples were 4.08 and 4.05 µg/g, respectively, whereas at pH 2, the amounts adsorbed were 3.72 and 3.57 µg/g, respectively. The paraquat sorption by soil is concentration dependent.

CHAPTER III
RESEARCH METHODOLOGY

The experiment can be divided into 4 steps which consist of 1.step of preparing soil, 2.step of preparing paraquat solution, 3.step of adsorption and 4.step of analyzing the samples.

3.1 Step of preparing soil

3.1.1 Preparing Lb-soil series

3.1.1.1 Chemical

1) Distilled water

3.1.1.2 Experimental equipment

1) Zip lock bag

2) Hoe

3) Shovel

4) Rubber hammer

5) Auger

6) Measuring tape

7) Standard test sieve

8) Mortar

9) Plastic jars

10) Spoon

3.1.1.3 Experimental procedures

1) Gather information of Lb-soil series from website of land development department [6].

2) Choose the location to collect Lb-soil series.

3) Go to Baan Pung Pattana, Kaeng Khoi district, Saraburi province at coordinate N14°45′12″ E101°02′53″.

4) Use a hoe and a shovel to remove reclamation soil from the ground for a depth of 30 cm from the ground surface.

5) Use an auger to collect the Lb-soil series at 3 levels of depth which consists of 1. Lbtop: 25 cm start from the point which removes the reclamation soil,

2. Lbmiddle: 25 cm start from the previous point and 3. Lbtop: 50 cm start from the previous point.

6) Use rubber hammer to remove Lb-soil series from auger and pack Lb-soil series into zip lock bag (repeat step 5. and 6. until finish collect Lbtop, Lbmiddle and Lbbottom).

7) Drying Lb-soil series by sun bathing for 12 h.

8) Rough mashing with mortar.

9) Leaching with water.

10) Screening with standard test sieve 2 mm and 500 µm to remove rock and gravel.

11) Drying by sun bathing 2 times for 12 h per time.

12) Fine mashing with mortar.

13) Use a spoon to scoop the Lb-soil series out of the mortar and drop it into plastic jars.

3.1.2 Preparing bentonite

3.1.2.1 Experimental procedure

1) Buy from KRUNGTHEPCHEMI

3.2 Step of preparing paraquat solution

3.2.1 Preparing paraquat solution

3.2.1.1 Chemicals

1) Paraquat 27.6%w/v (Gallon)

2) DI water

3.2.1.2 Experimental equipment

1) Pipette 1 mL

2) Pipette filler

3) Volumetric flask 1000 mL

4) Cylinder 100 and 1000 mL

5) Beaker 400 and 1000 mL

6) Rubber gloves

3.2.1.3 Experimental procedures

1) Wear rubber gloves.

2) Prepared paraquat solution at concentration 345 mg/L (paraquatC1): Use pipette 1 mL to draw 1 mL of paraquat from a gallon of paraquat 27.6%w/v, drop to a volumetric flask 1000 mL, add DI water until volume equal to 800 mL by use cylinder 1000 mL and shake it.

3) Make paraquat solution at concentration 172.5 mg/L (paraquatC2): Pour 200mL of paraquatC1 into a cylinder 1000 mL, add DI water until volume equal to 400 mL, pour solution into a volumetric flask 1000 mL and shake it.

4) Make paraquat solution at concentration 86.25 mg/L (paraquatC3): Pour 200mL of paraquatC2 into a cylinder 1000 mL, add DI water until volume equal to 400 mL, pour solution into a volumetric flask 1000 mL and shake it.

5) Make paraquat solution at concentration 43.13 mg/L (paraquatC4): Pour 200mL of paraquatC3 into a cylinder 1000 mL, add DI water until volume equal to 400 mL, pour solution into a volumetric flask 1000 mL and shake it.

6) Make paraquat solution at concentration 21.56 mg/L (paraquatC5): Pour 200mL of paraquatC4 into a cylinder 1000 mL, add DI water until volume equal to 400 mL, pour solution into a volumetric flask 1000 mL and shake it.

7) Make paraquat solution at concentration 10.78 mg/L (paraquatC6): Pour 200mL of paraquatC5 into a cylinder 1000 mL, add DI water until volume equal to 400 mL, pour solution into a volumetric flask 1000 mL and shake it.

3.3 Step of adsorption

3.3.1 Adsorption of paraquat by use Lb-soil series as an adsorbent

3.3.1.1 Chemicals

1) Paraquat solution (C1-C6)

2) Lbtop

3) Lbmiddle

4) Lbbottom

3.3.1.2 Experimental equipment

1) Tubes (with cap)

2) Barrack

3) Beaker 100 mL

4) Erlenmeyer flasks 125 mL

5) Spatula

6) Funnel

7) Parafilm

8) Shaker incubator

9) Laboratory Centrifuge

3.3.1.3 Experimental procedures

1) Use a spatula to scoop 0.1 g of Lbtop and drop into an erlenmeyer flask 125 mL (make 6 flasks).

2) Poor 20 mL of paraquat solution into a flask (one concentration of paraquat solution per one flask).

3) Use parafilm to close mouth of flasks.

4) Use shaker incubator at condition 100 rpm, 25°C and 24 h.

5) Poor samples into tubes through a funnel (one sample per one tube).

6) Use laboratory Centrifuge at condition 4000 rpm, 25°C and 1 h.

7) Repeat step 1) to step 6) but change Lbtop to Lb middle and Lbbottom, respectively.

3.3.2 Adsorption of paraquat by using bentonite as an adsorbent

3.3.2.1 Chemicals

1) Paraquat solution (C1-C6)

2) Bentonite

3.3.2.2 Experimental equipment

1) Tubes (with cap)

2) Barrack

3) Beaker 100 mL

4) Erlenmeyer flasks 125 mL

5) Spatula

6) Funnel

7) Parafilm

8) Shaker incubator

9) Laboratory Centrifuge

3.3.2.3 Experimental procedures

1) Use a spatula to scoop 0.1 g of bentonite and drop into an erlenmeyer flask 125 mL (make 6 flasks).

2) Pour 20 mL of paraquat solution into a flask (one concentration of paraquat solution per one flask).

3) Use parafilm to close flasks.

4) Use shaker incubator at condition 100 rpm, 25°C and 24 h.

5) Pour samples into tubes through a funnel (one sample per one tube).

6) Use laboratory Centrifuge at the conditions of 4000 rpm, 25°C and 1 h.

3.3.3 Preparing Blank solutions

3.3.3.1 Chemicals

1) Bentonite

2) Lbtop

3) Lbmiddle

4) Lbbottom

5) DI water

3.3.3.2 Experimental equipment

1) Tubes (with cap)

2) Barrack

3) Erlenmeyer flasks 125 mL

4) Spatula

5) Funnel

6) Parafilm

7) Shaker incubator

8) Laboratory Centrifuge

3.3.1.3 Experimental procedures

1) Use a spatula to scoop 0.1 g of bentonite and drop into an erlenmeyer flask 125 mL.

2) Add 20 mL of DI water into a flask.

3) Use parafilm to close mouth of flask.

4) Use shaker incubator at condition 100 rpm, 25°C and 24 h.

5) Poor a blank solution into a tube through a funnel.

6) Use laboratory Centrifuge at condition 4000 rpm, 25°C and 1 h.

7) Repeat step 1) to step 6) but change bentonite to Lbtop, Lb middle and Lbbottom, respectively.

3.4 Step of analyze the samples

3.4.1 Analyze Lb-soil series sample

3.4.1.1 Chemicals

1) Lbtop samples (C1-C6)

2) Lbmiddle samples (C1-C6)

3) Lbbottom samples (C1-C6)

4) Blank solutions (Lbtop, Lbmiddle and Lbbottom)

5) DI water

3.4.1.2 Experimental equipment

1) Volumetric flasks 25 mL

2) Pipette 3 mL

3) Pipette filler

4) Quartz cuvettes 10 mm, 3.5 mL

5) UV-Visible Spectrophotometer

3.4.1.2 Experimental procedures

1) Use pipette 3 mL to draw 3 mL of Lbtop samples (C1-C6) and drop to volumetric flasks 25 mL (one sample per one flask).

2) Use pipette 3 mL to draw 3 mL of Lbmiddle samples (C1-C6) and drop to volumetric flasks 25 mL (one sample per one flask).

3) Use pipette 3 mL to draw 3 mL of Lbbottom samples (C1-C6) and drop to volumetric flasks 25 mL (one sample per one flask).

4) Use UV-Visible Spectrophotometer at condition 200-400 nm.

3.4.2 Analyze bentonite sample

3.4.1.1 Chemicals

1) Bentonite samples (C1-C6)

2) Blank solutions (bentonite)

3) DI water

3.4.1.2 Experimental equipment

1) Volumetric flasks 25 mL

2) Pipette 3 mL

3) Pipette filler

4) Quartz cuvettes 10 mm, 3.5 mL

5) UV-Visible Spectrophotometer

3.4.1.2 Experimental procedures

1) Use pipette 3 mL to draw 3 mL of bentonite samples (C1-C6) and drop to volumetric flasks 25 mL (one sample per one flask).

2) Use UV-Visible Spectrophotometer at condition 200-400 nm.

CHAPTER IV
RESULTS AND DISCUSSION

The results of the paraquat adsorption with Lb-soil series and bentonite shown in the Tables 4.1- 4.4 were analyzed by using UV-Visible Spectrophotometer to construct adsorption isotherm shown in the Figure 4.1. The adsorption isotherm was predicted from the combination of Langmuir (2.1) and Freundlich model (2.2). The model can be explained by the equation below;

$$X = \frac{bX_m C_e}{1 + bC_e} + kC_e^{\,n} \tag{4.1}$$

where, C_e = equilibrium concentration of paraquat in solution (mg/L)

X_m = maximum amount of paraquat that can be adsorbed (mg/g)

X = paraquat adsorbed per mass of soil or clay (mg/g)

b = equilibrium constant of adsorption

k = rate constant of formation

n = order of formation

Table 4.1: Experimental data and parameters value of Lbtop

<table>
<tr><th rowspan="2">Initial conc. (mg/L)</th><th rowspan="2">C_e (mg/L)</th><th rowspan="2">X (mg/g)</th><th colspan="2">Langmuir</th><th colspan="2">Freundlich</th><th rowspan="2">R^2</th></tr>
<tr><th>X_m (mg/g)</th><th>b (L/mg)</th><th>$k*10^{-5}$ $((L/mg)^{1-n})$</th><th>n</th></tr>
<tr><td>0.00</td><td>0.00</td><td>0.00</td><td rowspan="7">19.60</td><td rowspan="7">7.31</td><td rowspan="7">2.08</td><td rowspan="7">3.01</td><td rowspan="7">0.7621</td></tr>
<tr><td>10.78</td><td>0.00</td><td>2.16</td></tr>
<tr><td>21.56</td><td>0.00</td><td>4.31</td></tr>
<tr><td>43.13</td><td>0.00</td><td>8.63</td></tr>
<tr><td>86.25</td><td>0.00</td><td>17.25</td></tr>
<tr><td>172.50</td><td>55.92</td><td>23.32</td></tr>
<tr><td>345.00</td><td>108.55</td><td>47.29</td></tr>
</table>

Table 4.2: Experimental data and parameters value of Lbmiddle

Initial conc. (mg/L)	C_e (mg/L)	X (mg/g)	Langmuir		Freundlich		R^2
			X_m (mg/g)	b (L/mg)	$k*10^{-5}$ ((L/mg)$^{1-n}$)	n	
0.00	0.00	0.00	29.12	0.51	1.56	2.93	0.9414
10.78	0.00	2.16					
21.56	0.00	4.31					
43.13	0.00	8.63					
86.25	2.63	16.72					
172.50	32.89	27.29					
345.00	115.79	45.84					

Table 4.3: Experimental data and parameters value of Lbbottom

Initial conc. (mg/L)	C_e (mg/L)	X (mg/g)	Langmuir		Freundlich		R^2
			X_m (mg/g)	b (L/mg)	$k*10^{-5}$ ((L/mg)$^{1-n}$)	n	
0.00	0.00	0.00	15.98	1.80	0.12	3.40	0.9798
10.78	0.00	2.16					
21.56	0.00	4.31					
43.13	0.66	8.49					
86.25	7.90	15.67					
172.50	78.94	18.71					
345.00	140.13	40.97					

Table 4.4: Experimental data and parameters value of bentonite

Initial conc. (mg/L)	C_e (mg/L)	X (mg/g)	Langmuir		Freundlich		R^2
			X_m (mg/g)	b (L/mg)	$k*10^{-5}$ ((L/mg)$^{1-n}$)	n	
0.00	0.00	0.00	38.23	6.52	4.16	3.01	0.8384
10.78	0.00	2.16					
21.56	0.00	4.31					
43.13	0.00	8.63					
86.25	0.00	17.25					
172.50	1.32	34.24					
345.00	72.37	54.53					

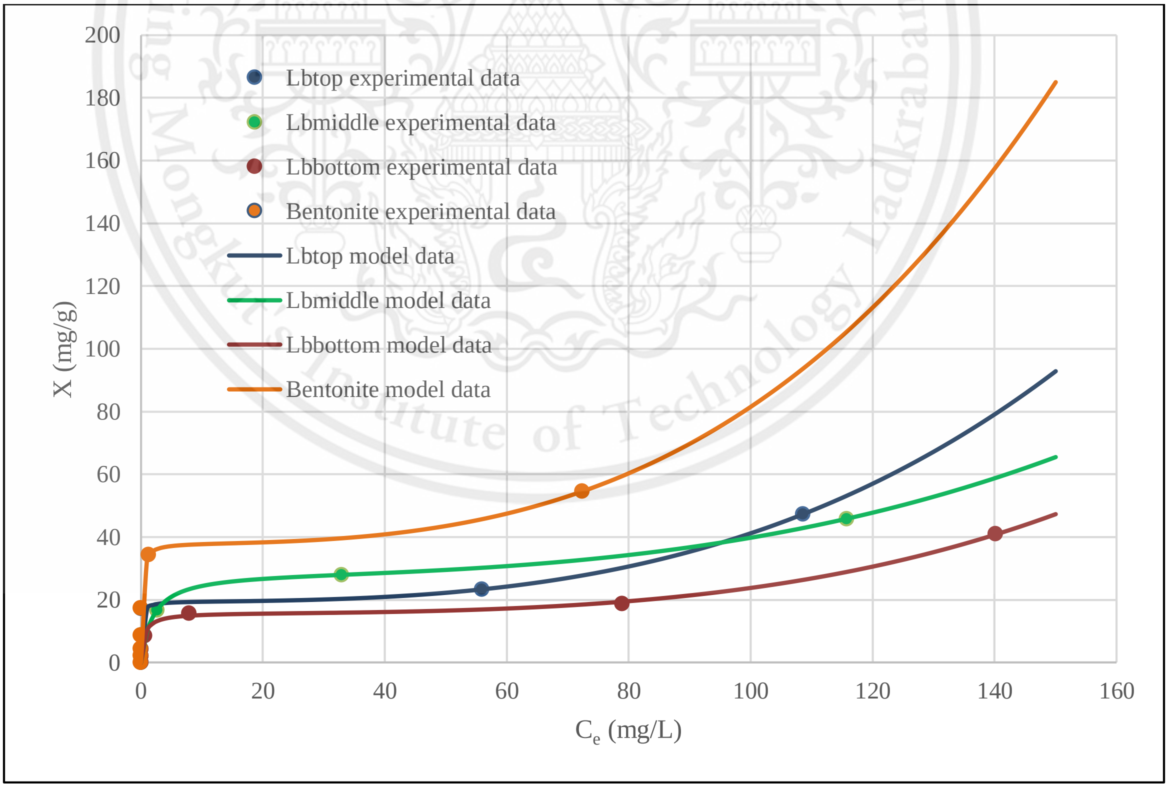

Figure 4.1: Adsorption isotherms of paraquat on different types of soils

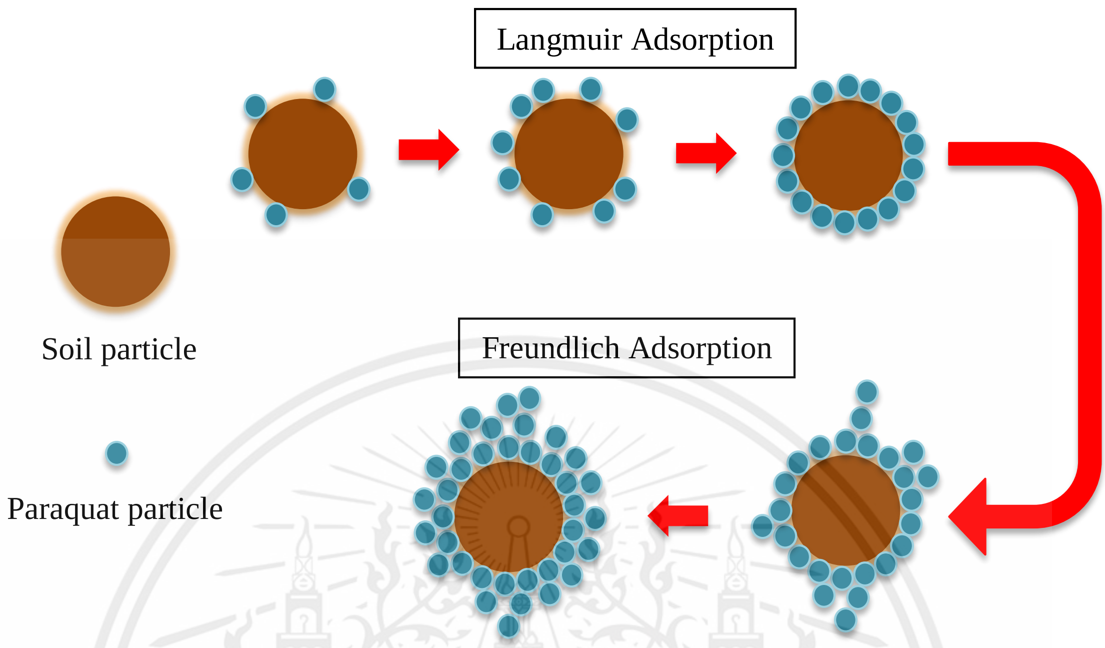

Figure 4.2: Adsorption mechanism of paraquat on soil

In this experiment, the results show that the behavior of paraquat adsorption on each soil were similar pattern. The pattern could be obtained from the curves shown in the Figure 4.1. Each curve indicated that it was divided into 2 sections depending on the equilibrium concentration of paraquat in the solution. In the first section, the pattern of paraquat adsorption was similar to Langmuir isotherm because there was low concentration of paraquat in solution. It could be described that the soil had enough surface area to be able to adsorb the paraquat at low concentration as a monolayer. In another section, the paraquat adsorption pattern behaved as Freundlich isotherm due to the remaining paraquat at high concentration could be reacted with each other to form multilayer after it had been adsorbed as a monolayer on the soil surface. As mentioned above, it can be clearly described by Figure 4.2.

Due to the adsorption mechanism of paraquat on soil consisting of 2 mechanisms including Langmuir and Freundlich. Therefore, the combination of Langmuir and Freundlich models shown in Equation 4.1 could describe this phenomenon. From Tables 4.1-4.4, it showed that X_m of bentonite is the highest value compared with other soils. The X_m of bentonite, Lbmiddle, Lbtop and Lbbottom were 38.23 mg/g, 29.12 mg/g, 19.60 mg/g and 15.98 mg/g, respectively. These values indicated that the paraquat could be adsorbed on the surface of bentonite higher than other soils. From theory, it describes that if soil contains high montmorillonite, it can well adsorb negative charge in solution. In previous researches, demonstrated that bentonite contained montmorillonite as a major component. Moreover, Lbtop, Lbmiddle and Lbbottom were selected in this study due to Land Development Department in Thailand indicated that these soils contain high montmorillonite.

Furthermore, Tables 4.1-4.4 showed that the value of n of each soils were equal to 3 approximately and they were not different significantly. However, the values of k of Lbtop, Lbmiddle, Lbbottom and bentonite were equal to 2.0780, 1.5570, 0.1239 and 4.1560, respectively. The value of k was refer to rate of reaction between paraquat with each other to form multilayer. From the data, it showed that the value of k of Lbtop, Lbmiddle and Lbbottom were less than bentonite. It might be a result of the desorption of some metal that was a positive charge from Lbtop, Lbmiddle and Lbbottom more than bentonite. The desorption of some metal to the solution might obstruct the formation of paraquat to form multilayer.

From experiments, it could be concluded that bentonite was the best soil for adsorption of paraquat from the solution. However, the price of this soil was higher than other soils, so Lbtop should be optional used as adsorbent at high concentration of paraquat because it could adsorb paraquat more than Lbmiddle and Lbbottom. On the other hand, Lbmiddle should be optional used as adsorbent at low concentration of paraquat because the value of X_m was higher than Lbtop and Lbbottom.

CHAPTER V
CONCLUSION AND FUTURE WORK

Conclusion

This aim of this study was to compare the removal efficiencies of paraquat from aqueous solution using 3 levels of Lb-soil series and bentonite. The experiment of this research was divided into 4 main parts**.** In each part, the different types of soil were prepared to adsorb the paraquat solution at various concentration including 345 mg/L, 172.5 mg/L, 86.25 mg/L, 43.125 mg/L, 21.5625 mg/L and 10.78125 mg/L. The types of soil used in this study consisted of Lbtop, Lbmiddle, Lbbottom and bentonite. After the adsorption, the remaining concentration of paraquat solution was analyzed by UV-Visible spectrometry to calculate the amount of adsorbed paraquat on each soils. Then, the adsorption isotherm was constructed from the result of calculation. The predicted model of adsorption isotherm was the combination of Langmuir and Freundlich model. Finally, the results indicated that at low concentration of paraquat solution, the order of paraquat adsorption efficiency on soils were bentonite, Lbmiddle, Lbtop and Lbbottom, respectively. In addition, at high concentration of paraquat solution, the order of paraquat adsorption efficiency on soils were bentonite, Lbtop, Lbmiddle and Lbbottom, respectively.

Future work

In order to improve the predicted adsorption model, BET isotherm was selected for study the pattern of paraquat adsorption. BET isotherm is an isotherm that takes account of the possibility that the monolayer in the Langmuir adsorption isotherm can act as a substrate for further adsorption. The equation can be written as in Equation 2.3. The calculation of Lbtop test showed that the BET model give the result better than Langmuir+Freundlich model so this model will be used in order to predict adsorption isotherm in the future work.

Table 5.1: The comparison of R^2 of Lbtop by using Langmuir+Freundlich and BET model

Model	R^2
Langmuir+Freundlich	0.7621
BET	0.9054

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APPENDIX

A.1 Step to collect Lb-soil series

1) Go to the website of land development department.

Figure A.1: Website of Land Development Department (1)

2) Click on the soil information section in the land development information section.

Figure A.2: Website of Land Development Department (2)

3) Click on the map of soil series, scale 1:100,000.

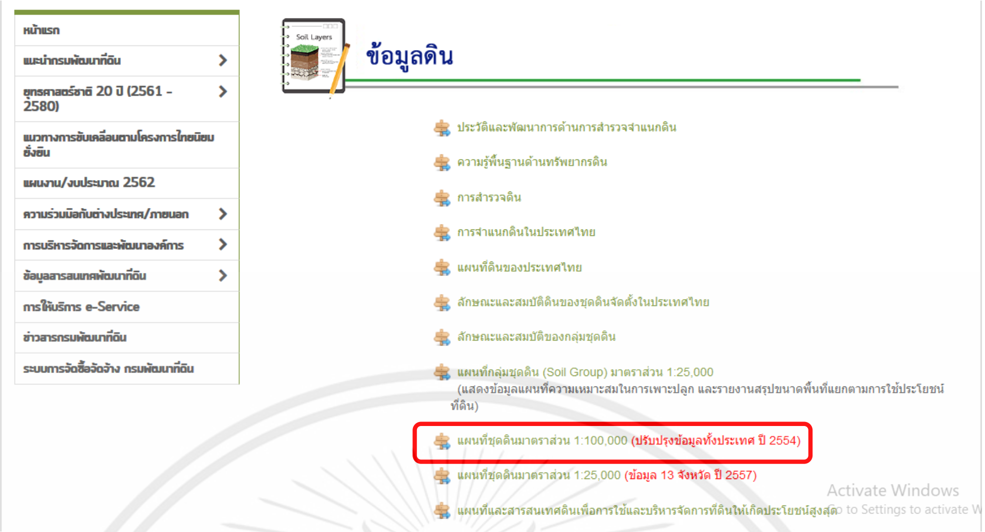

Figure A.3: Website of Land Development Department (3)

4) Select central region.

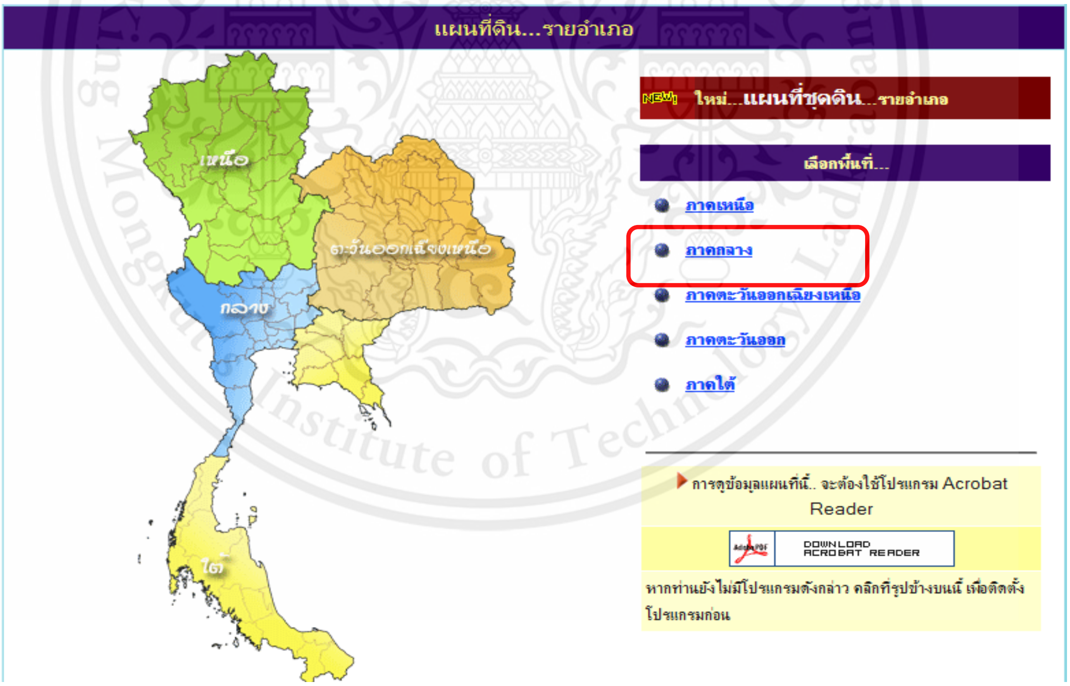

Figure A.4: Website of Land Development Department (4)

5) Select Saraburi province.

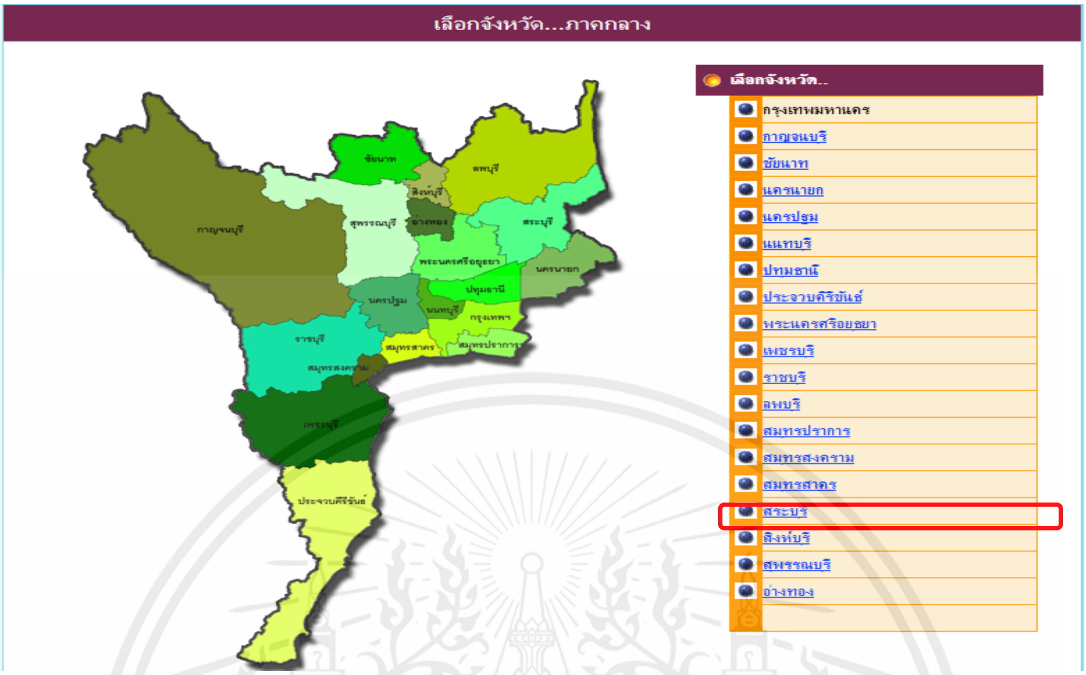

Figure A.5: Website of Land Development Department (5)

6) Select Kaeng Khoi district.

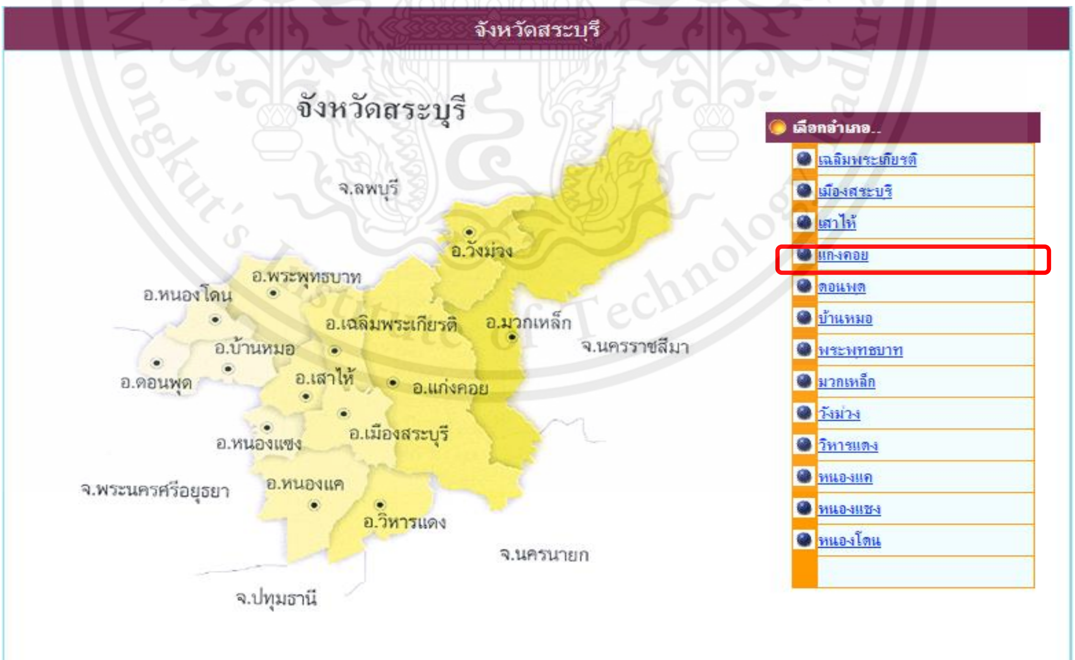

Figure A.6: Website of Land Development Department (6)

7) Compare the Kaeng Khoi district soil series map with Kaeng Khoi district satellite images.

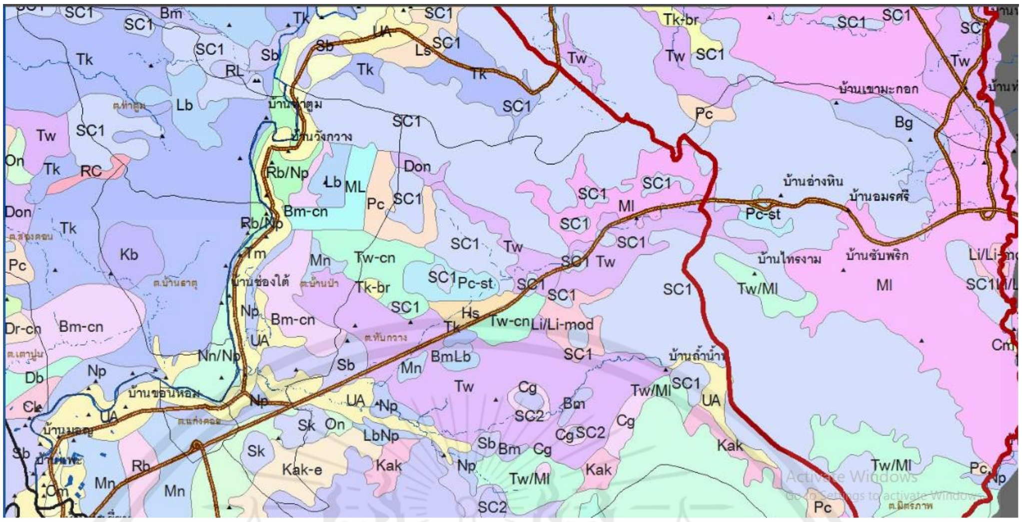

Figure A.7: Kaeng Khoi district soil series map

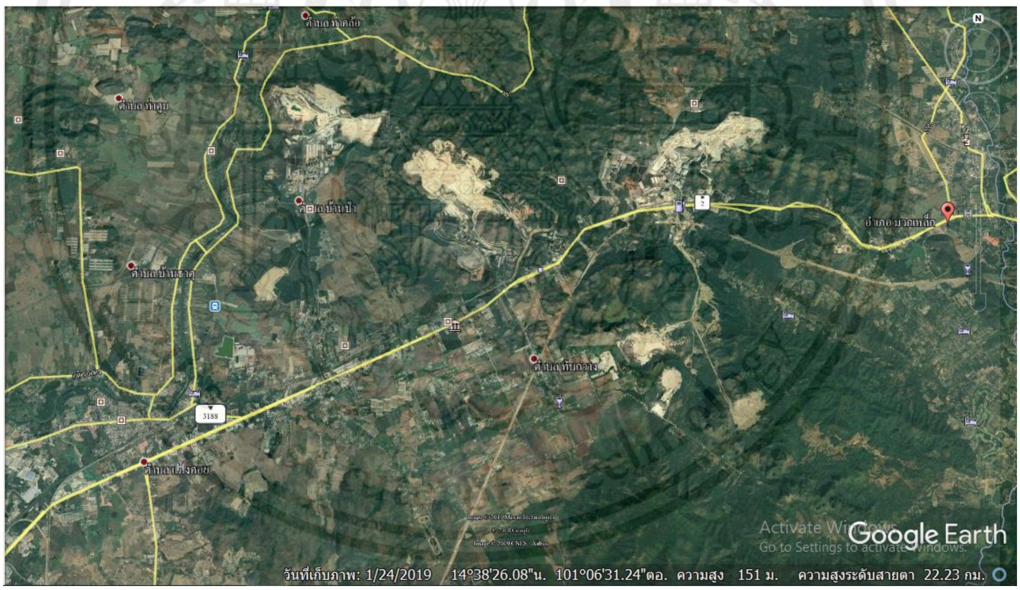

Figure A.8: Kaeng Khoi district satellite image by Google Earth

8) Locate the location of Lb-soil series which want to collect samples from Kaeng Khoi district soil series map.

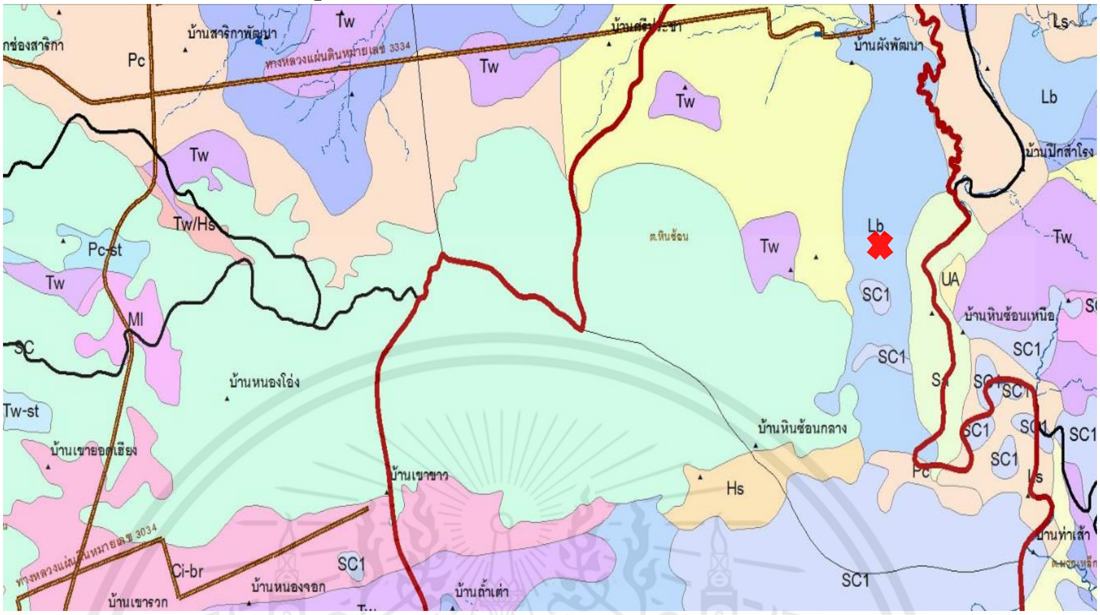

Figure A.9: Location of Lb-soil series from Kaeng Khoi district soil series map

9) Use Kaeng Khoi district satellite image to find coordinate of real location for collecting sample.

Figure A.10: Location of Lb-soil series from Kaeng Khoi district satellite image (N14°45′17.08″ E101°02′55.41″) by Google Earth

10) Use Street View to check the location for collecting Lb-soil series

Figure A.11: Location for collecting Lb-soil series from Street view Google Earth

11) Go to Baan Pung Pattana, Kaeng Khoi district, Saraburi province at coordinate N14°45′17.08″ E101°02′55.41″

Figure A.12: Real location for collecting Lb-soil series (17/7/2019)

12) Remove reclamation soil for a depth of 30 cm from the ground surface and collect the Lb-soil series at 3 levels consist of 25, 50 and 100 cm from the point which removes the reclamation soil.

Figure A.13: Images of collecting Lb-soil series samples (17/7/2019)

A.2 Data from experiment

1) Lbtop

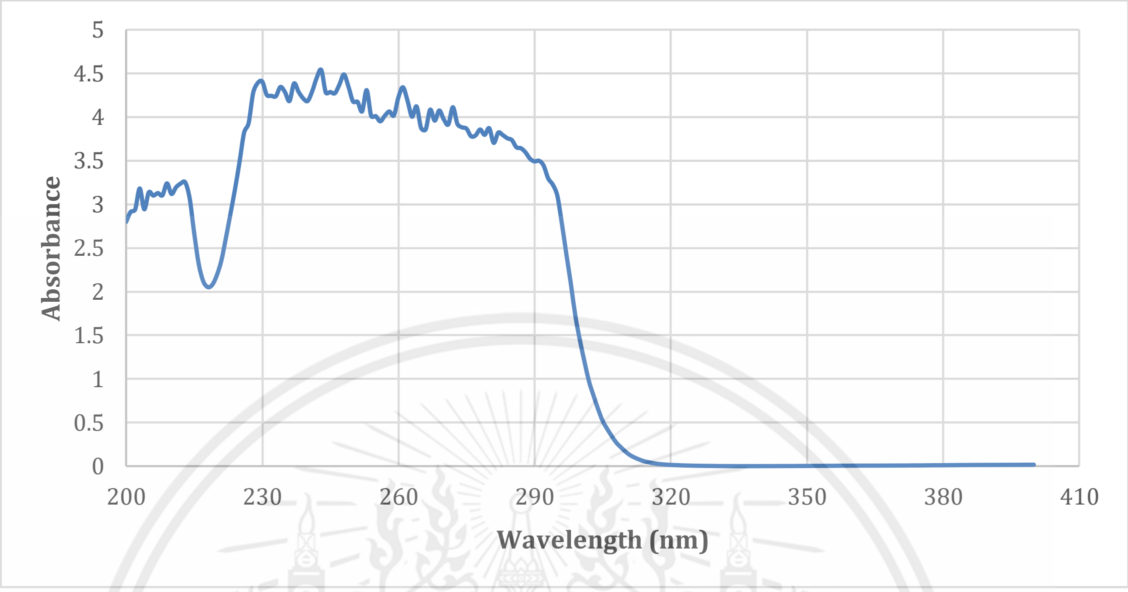

Figure A.14: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbtop and the wavelength of 200-400 nm when the initial paraquat concentration was 345 mg/L

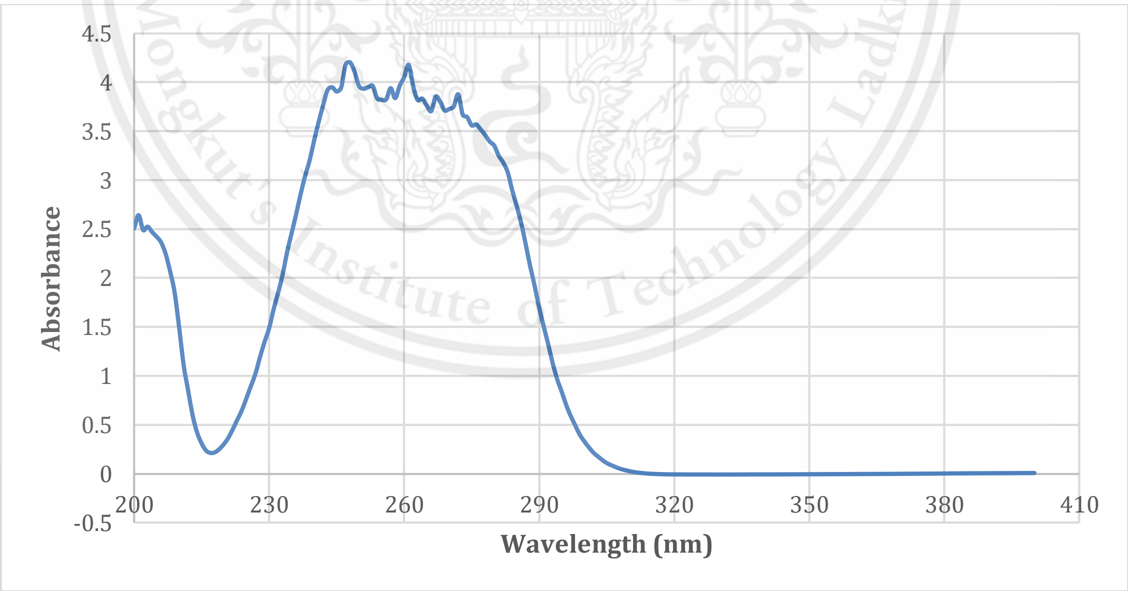

Figure A.15: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbtop and the wavelength of 200-400 nm when the initial paraquat concentration was 172.5 mg/L

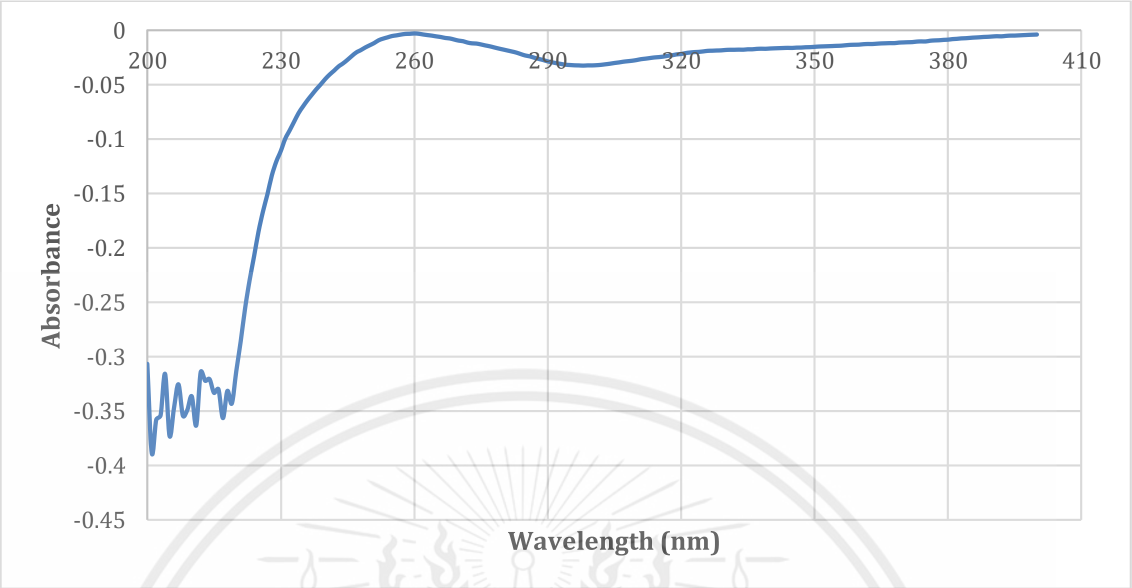

Figure A.16: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbtop and the wavelength of 200-400 nm when the initial paraquat concentration was 86.25 mg/L

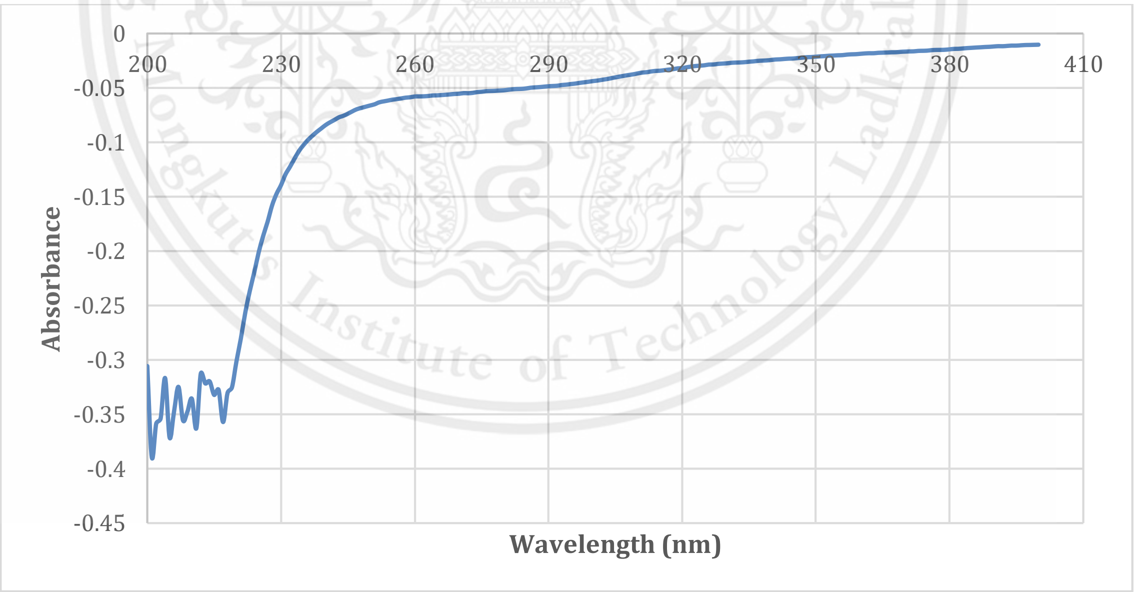

Figure A.17: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbtop and the wavelength of 200-400 nm when the initial paraquat concentration was 43.125 mg/L

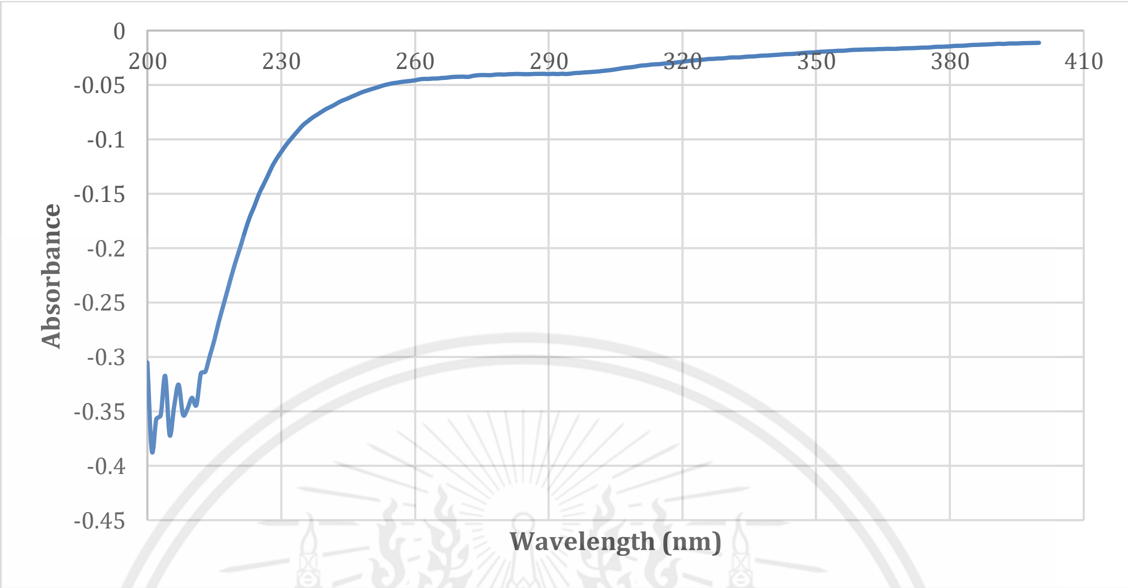

Figure A.18: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbtop and the wavelength of 200-400 nm when the initial paraquat concentration was 21.5625 mg/L

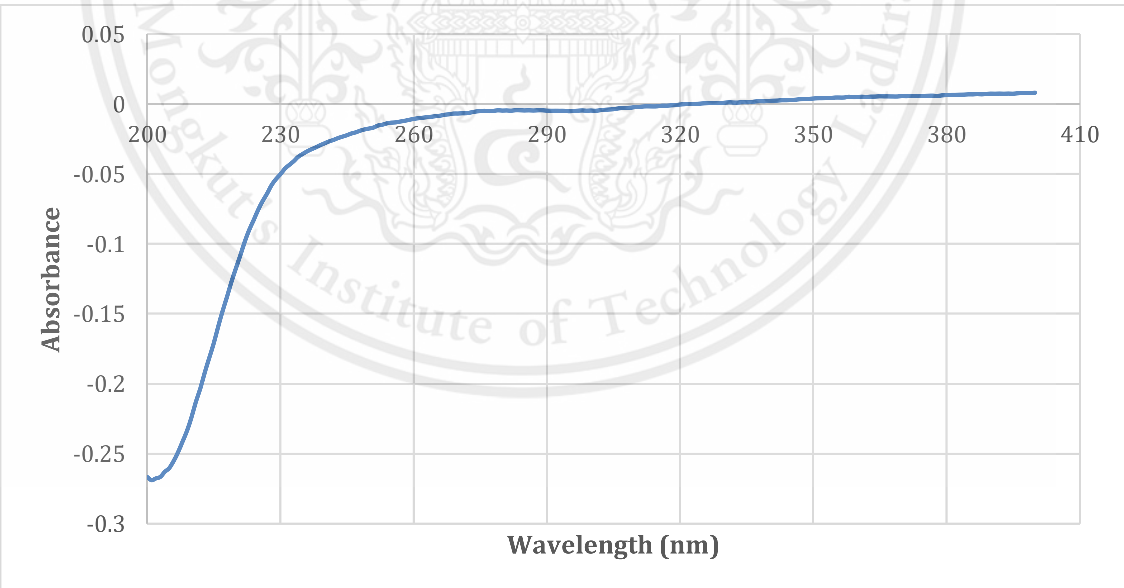

Figure A.19: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbtop and the wavelength of 200-400 nm when the initial paraquat concentration was 10.78125 mg/L

2) Lbmiddle

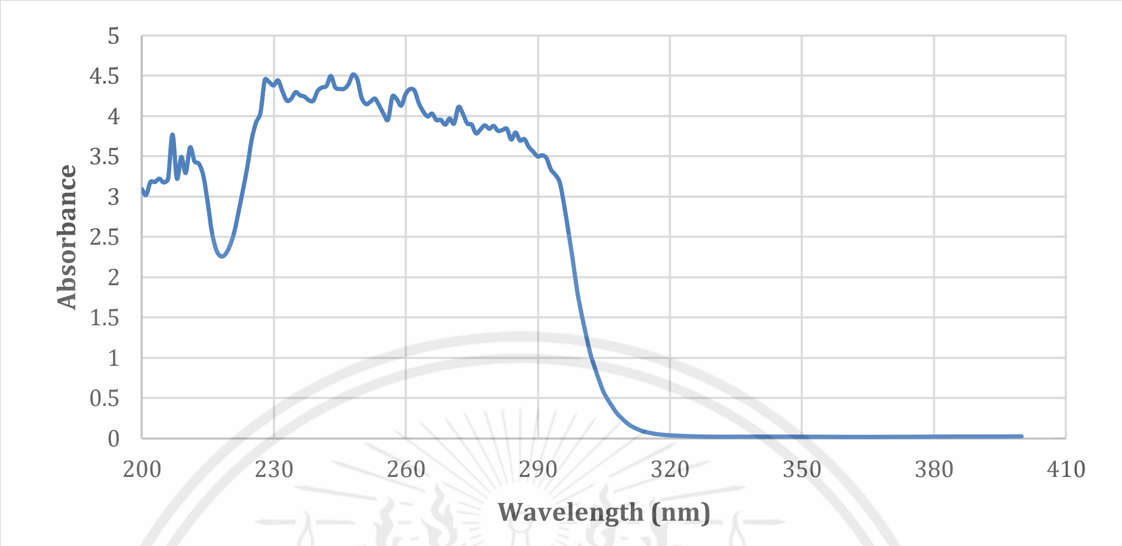

Figure A.20: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbmiddle and the wavelength of 200-400 nm when the initial paraquat concentration was 345 mg/L

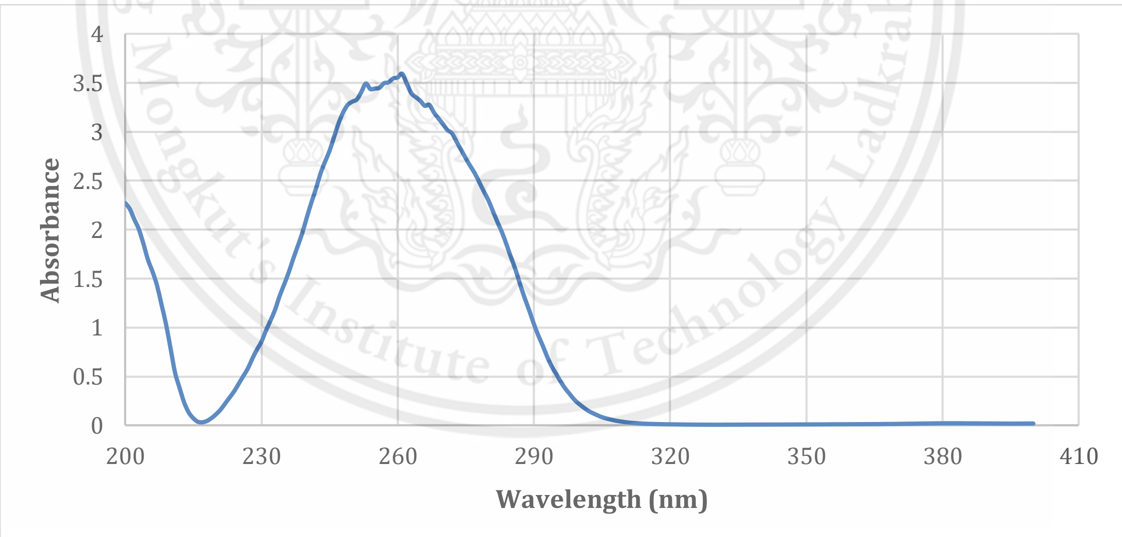

Figure A.21: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbmiddle and the wavelength of 200-400 nm when the initial paraquat concentration was 172.5 mg/L

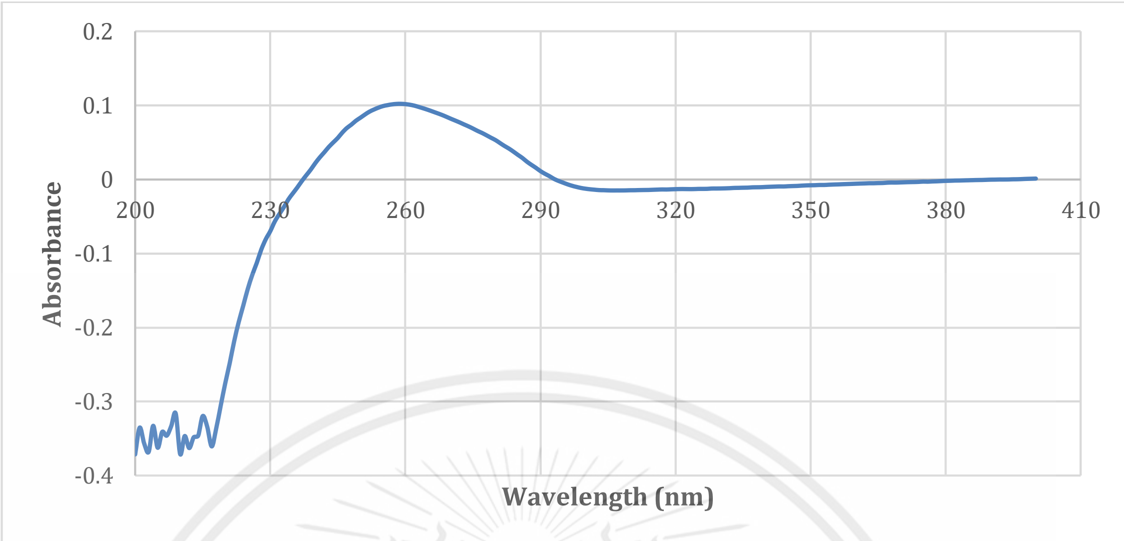

Figure A.22: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbmiddle and the wavelength of 200-400 nm when the initial paraquat concentration was 86.25 mg/L

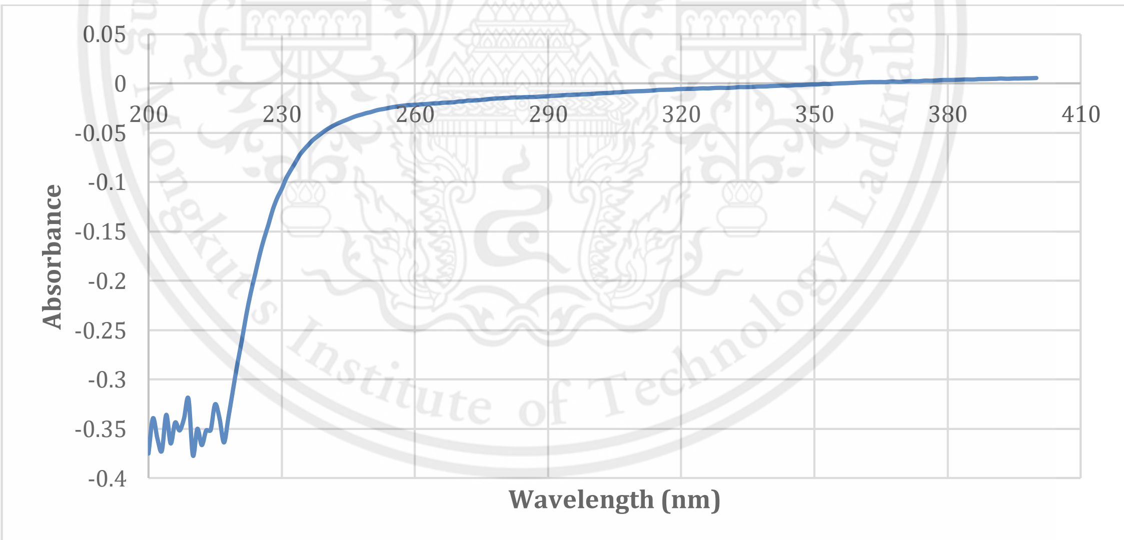

Figure A.23: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbmiddle and the wavelength of 200-400 nm when the initial paraquat concentration was 43.125 mg/L

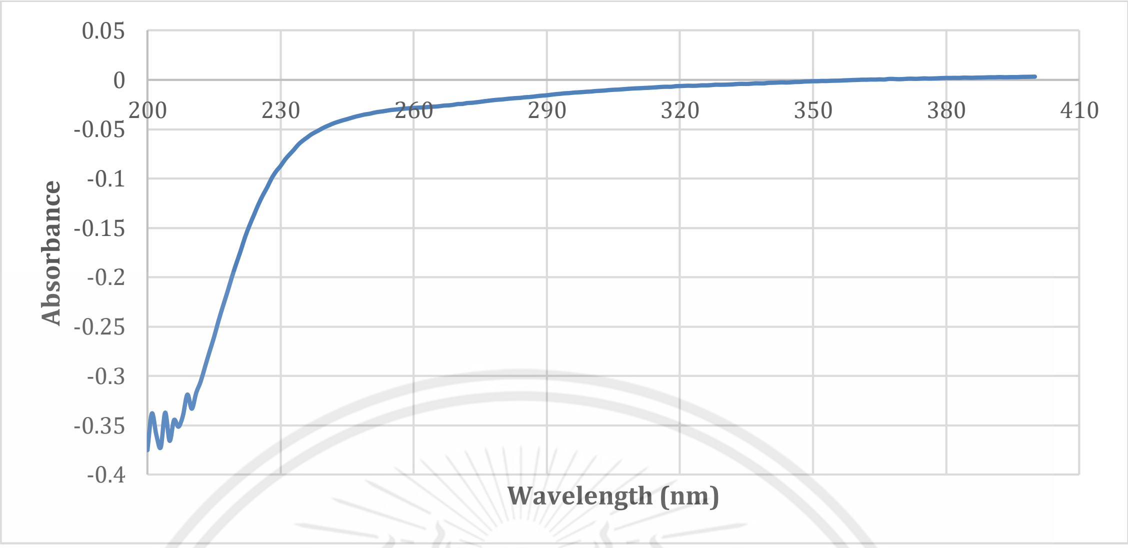

Figure A.24: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbmiddle and the wavelength of 200-400 nm when the initial paraquat concentration was 21.5625 mg/L

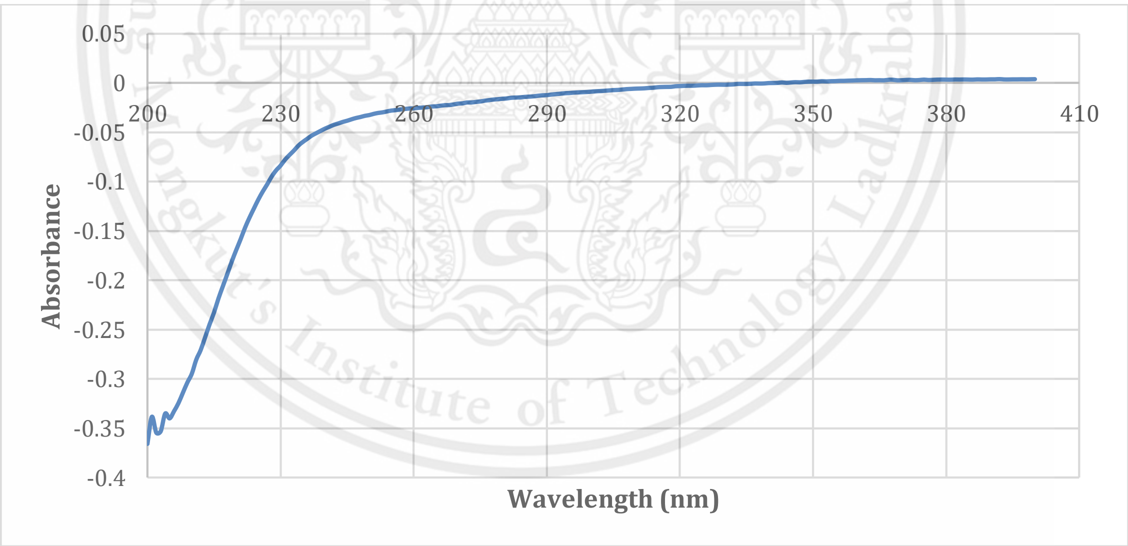

Figure A.25: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbmiddle and the wavelength of 200-400 nm when the initial paraquat concentration was 10.78125 mg/L

3) Lbbottom

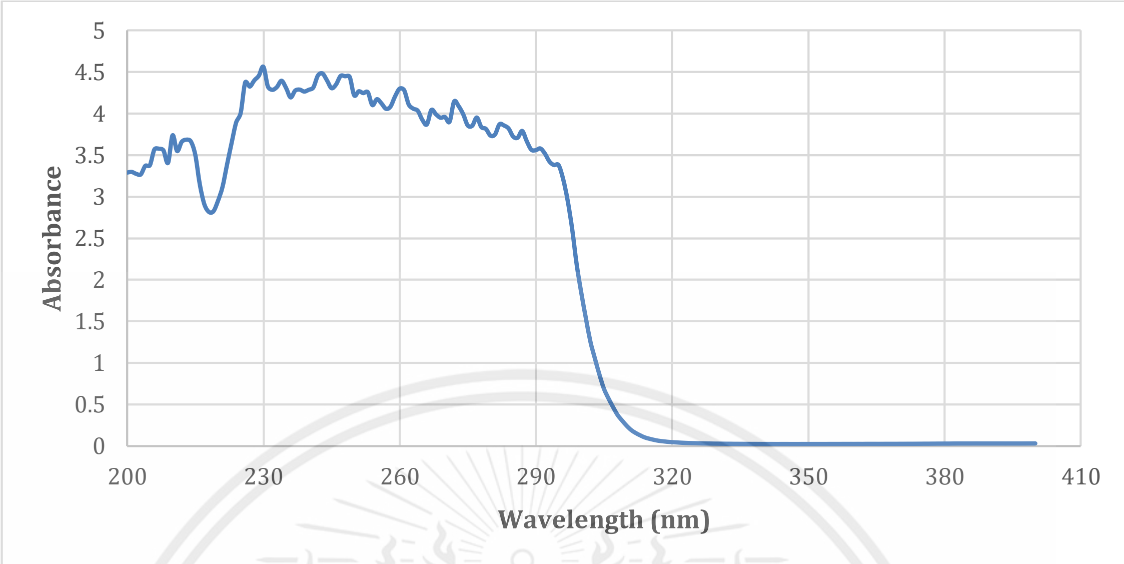

Figure A.26: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbbottom and the wavelength of 200-400 nm when the initial paraquat concentration was 345 mg/L

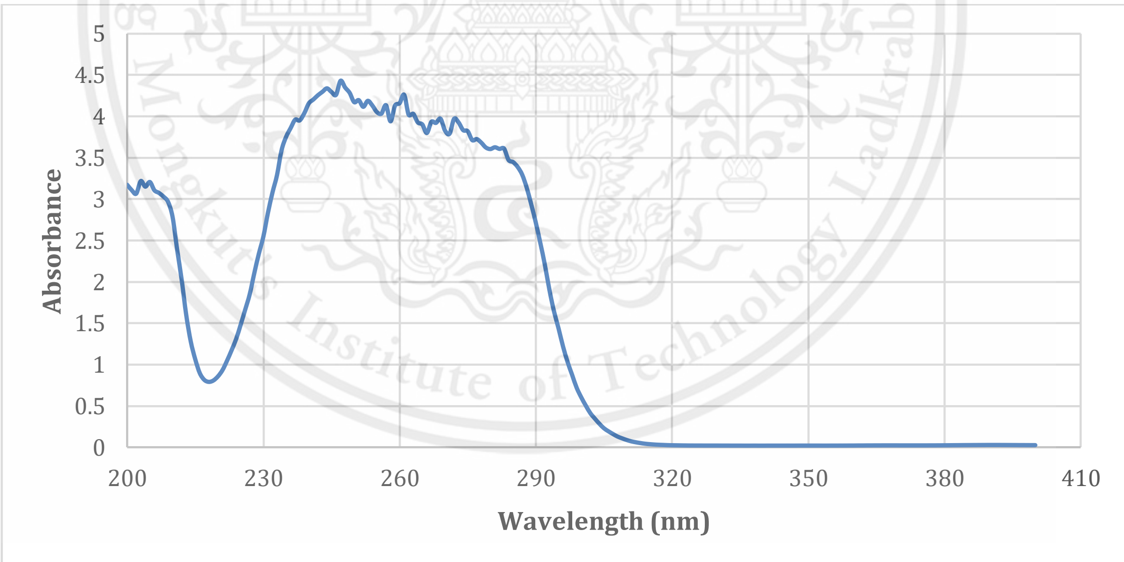

Figure A.27: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbbottom and the wavelength of 200-400 nm when the initial paraquat concentration was 172.5 mg/L

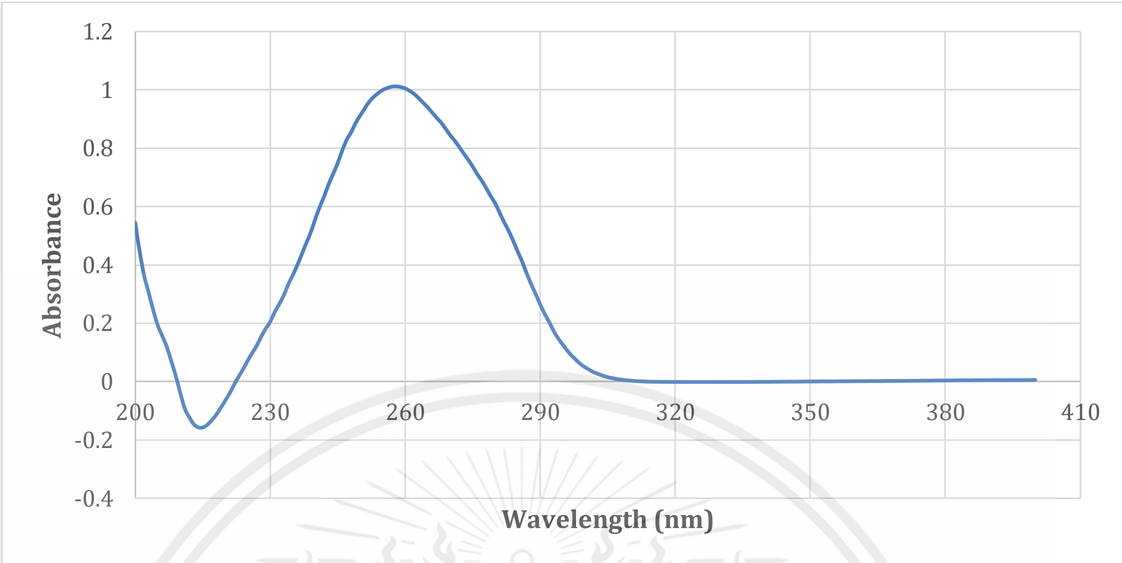

Figure A.28: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbbottom and the wavelength of 200-400 nm when the initial paraquat concentration was 86.25 mg/L

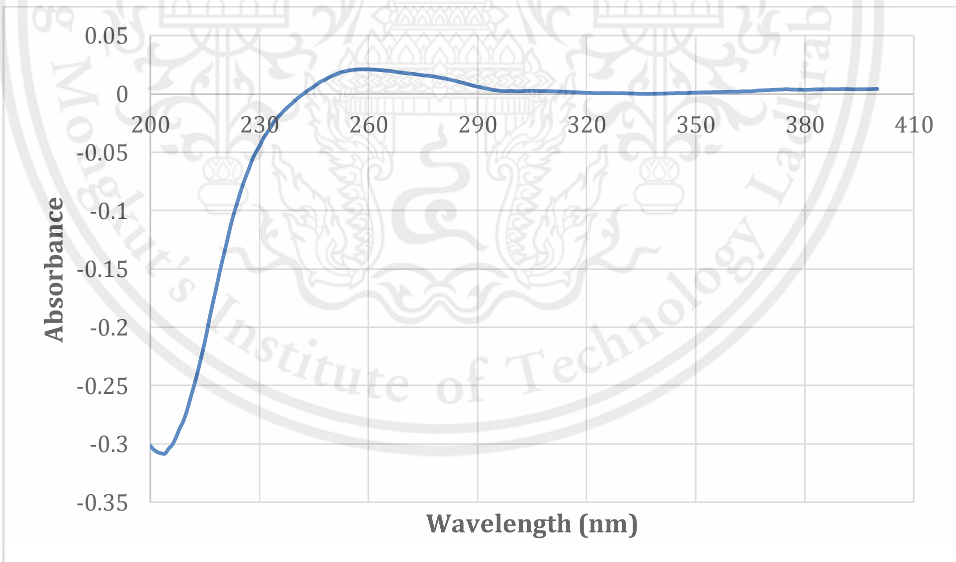

Figure A.29: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbbottom and the wavelength of 200-400 nm when the initial paraquat concentration was 43.125 mg/L

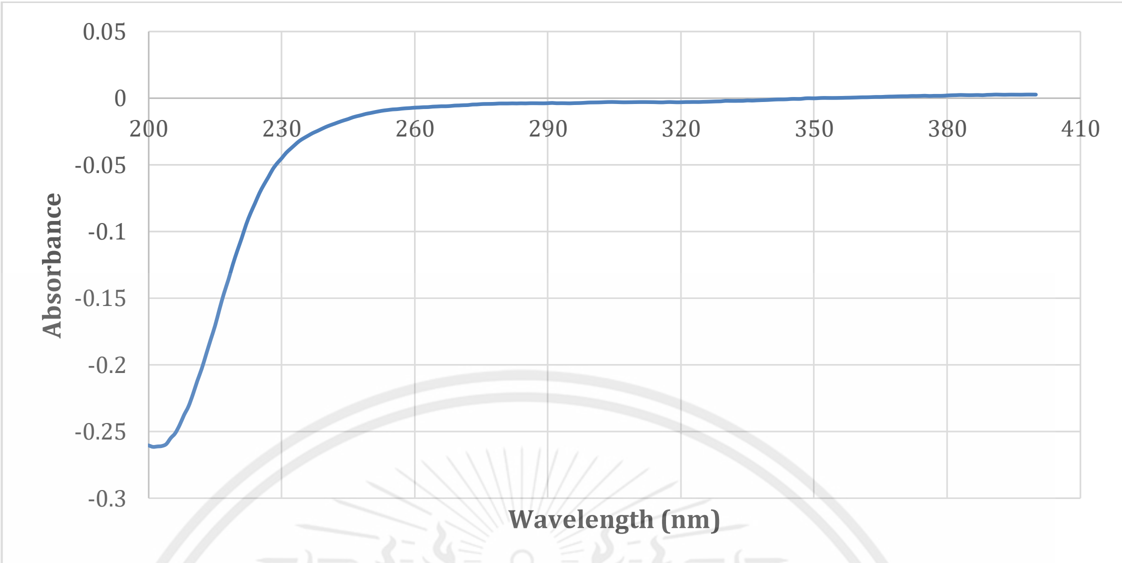

Figure A.30: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbbottom and the wavelength of 200-400 nm when the initial paraquat concentration was 21.5625 mg/L

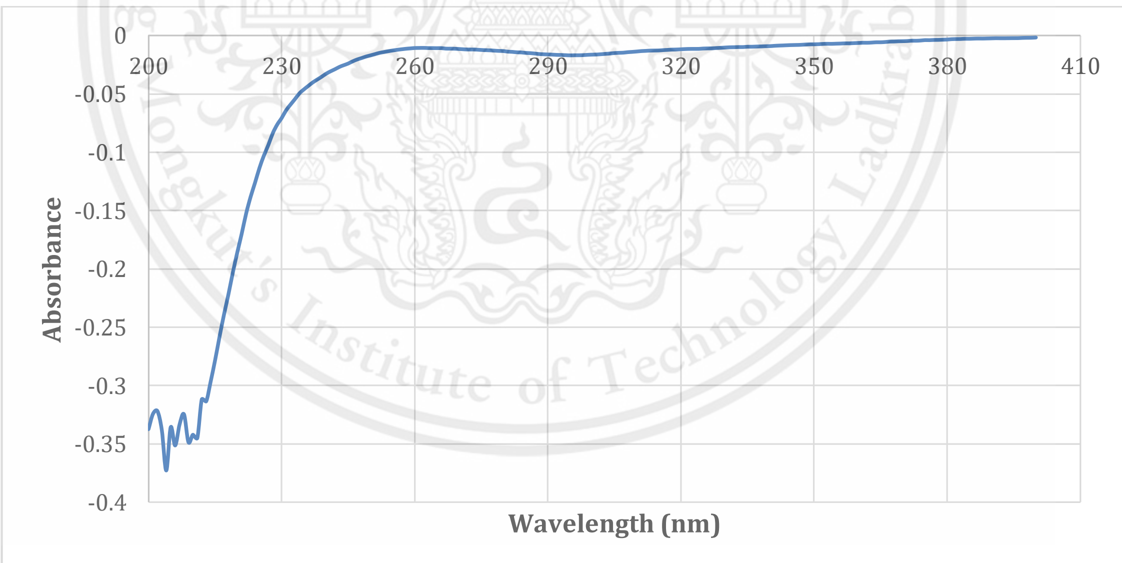

Figure A.31: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on Lbbottom and the wavelength of 200-400 nm when the initial paraquat concentration was 10.78125 mg/L

4) Bentonite

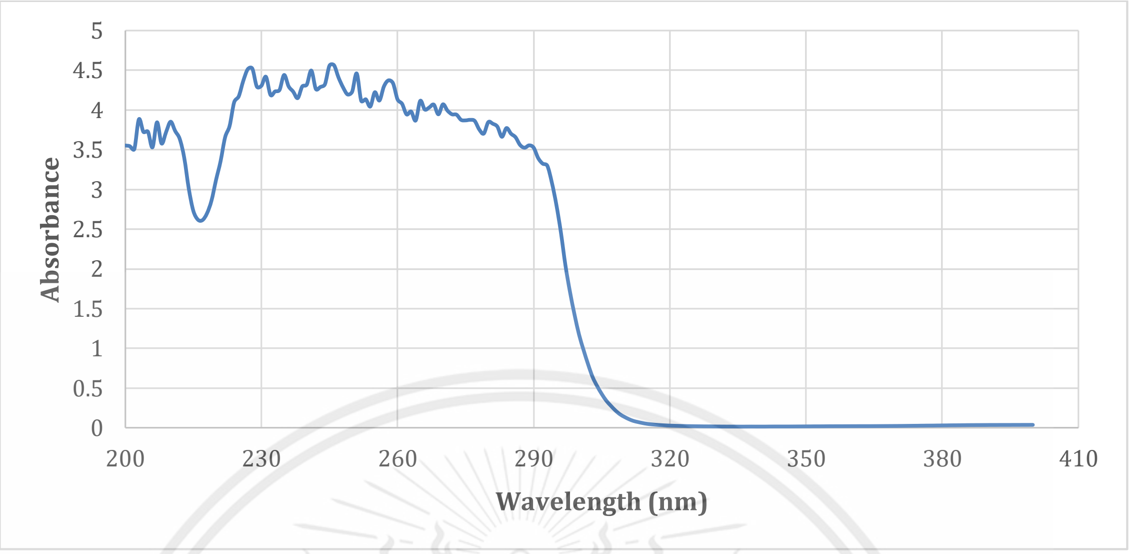

Figure A.32: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on bentonite and the wavelength of 200-400 nm when the initial paraquat concentration was 345 mg/L

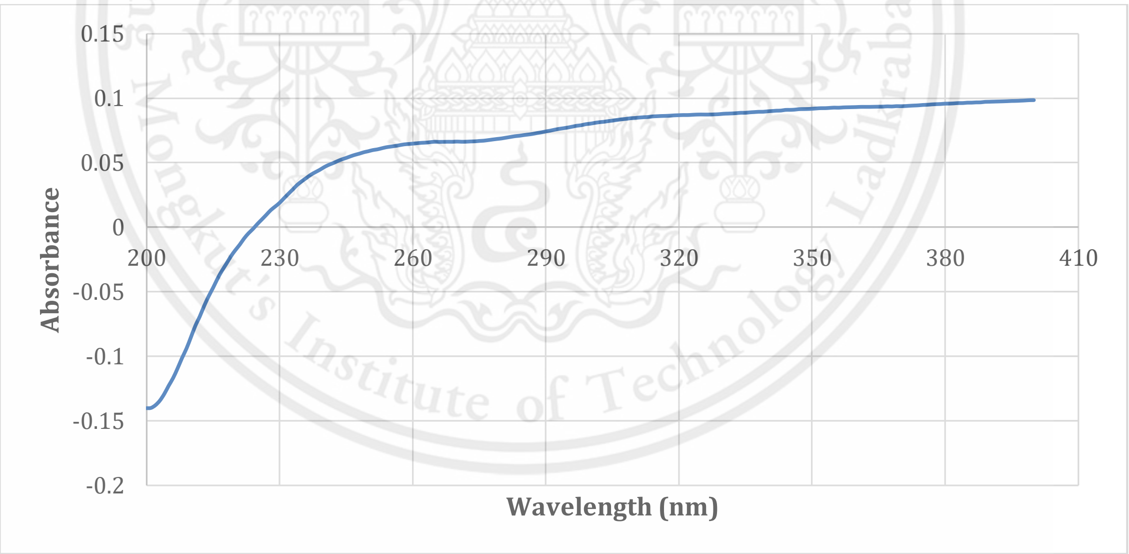

Figure A.33: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on bentonite and the wavelength of 200-400 nm when the initial paraquat concentration was 172.5 mg/L

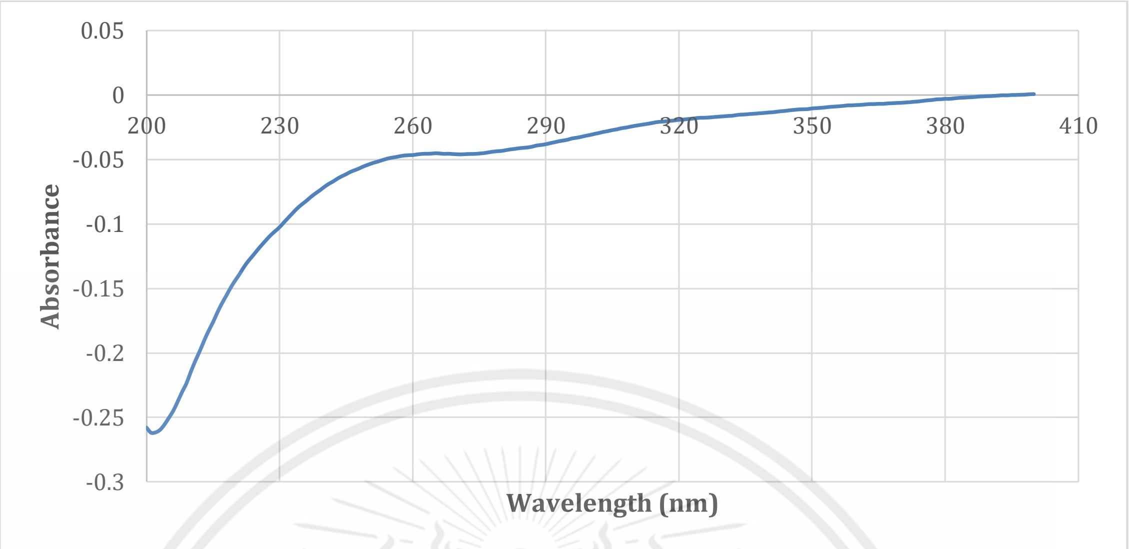

Figure A.34: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on bentonite and the wavelength of 200-400 nm when the initial paraquat concentration was 86.25 mg/L

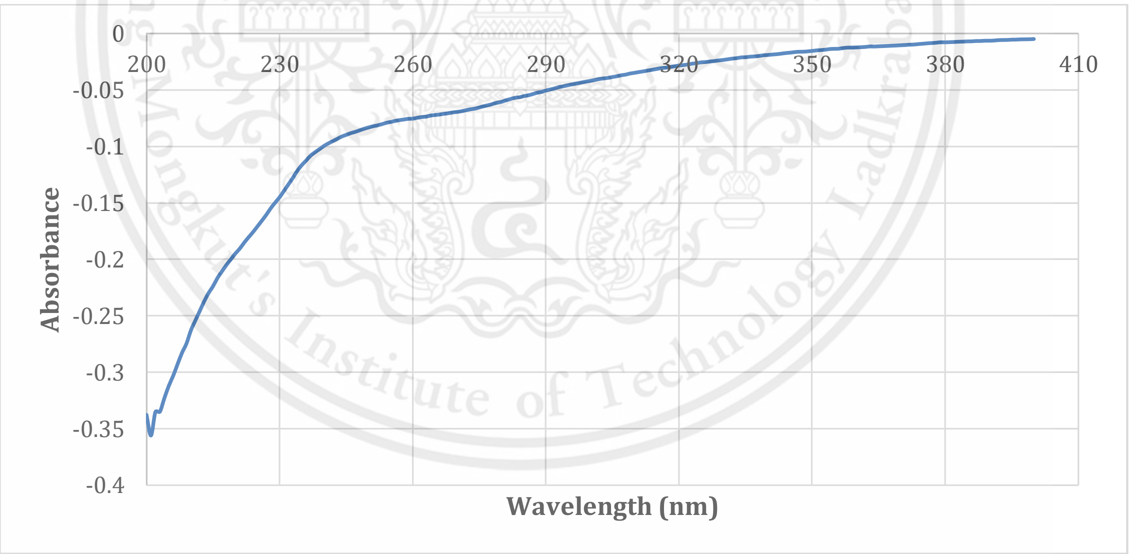

Figure A.35: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on bentonite and the wavelength of 200-400 nm when the initial paraquat concentration was 43.125 mg/L

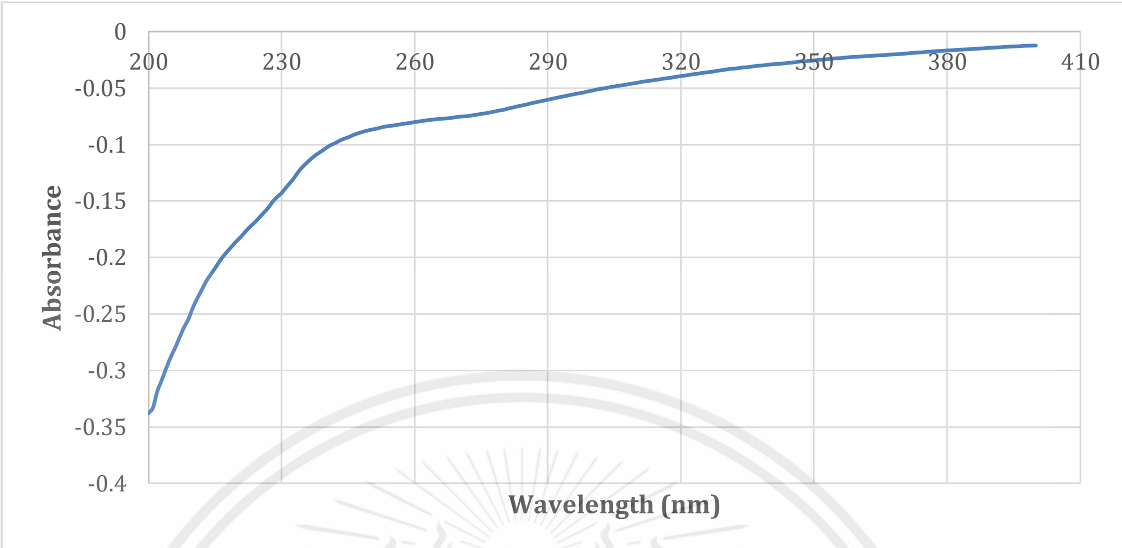

Figure A.36: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on bentonite and the wavelength of 200-400 nm when the initial paraquat concentration was 21.5625 mg/L

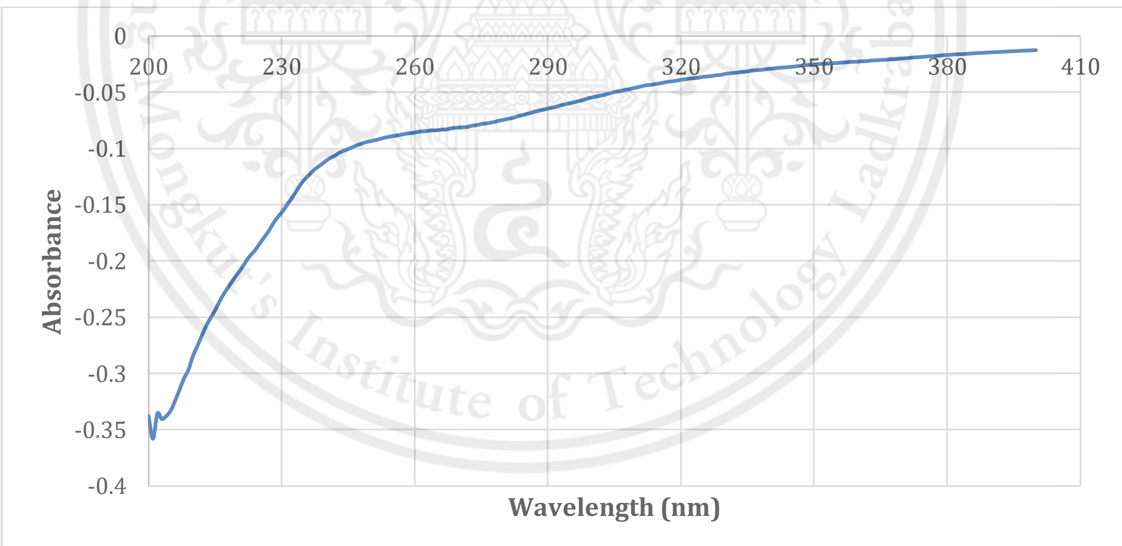

Figure A.37: The graph shown the relation between the absorbance of the paraquat solution at equilibrium adsorption on bentonite and the wavelength of 200-400 nm when the initial paraquat concentration was 10.78125 mg/L

5) Calibration curve

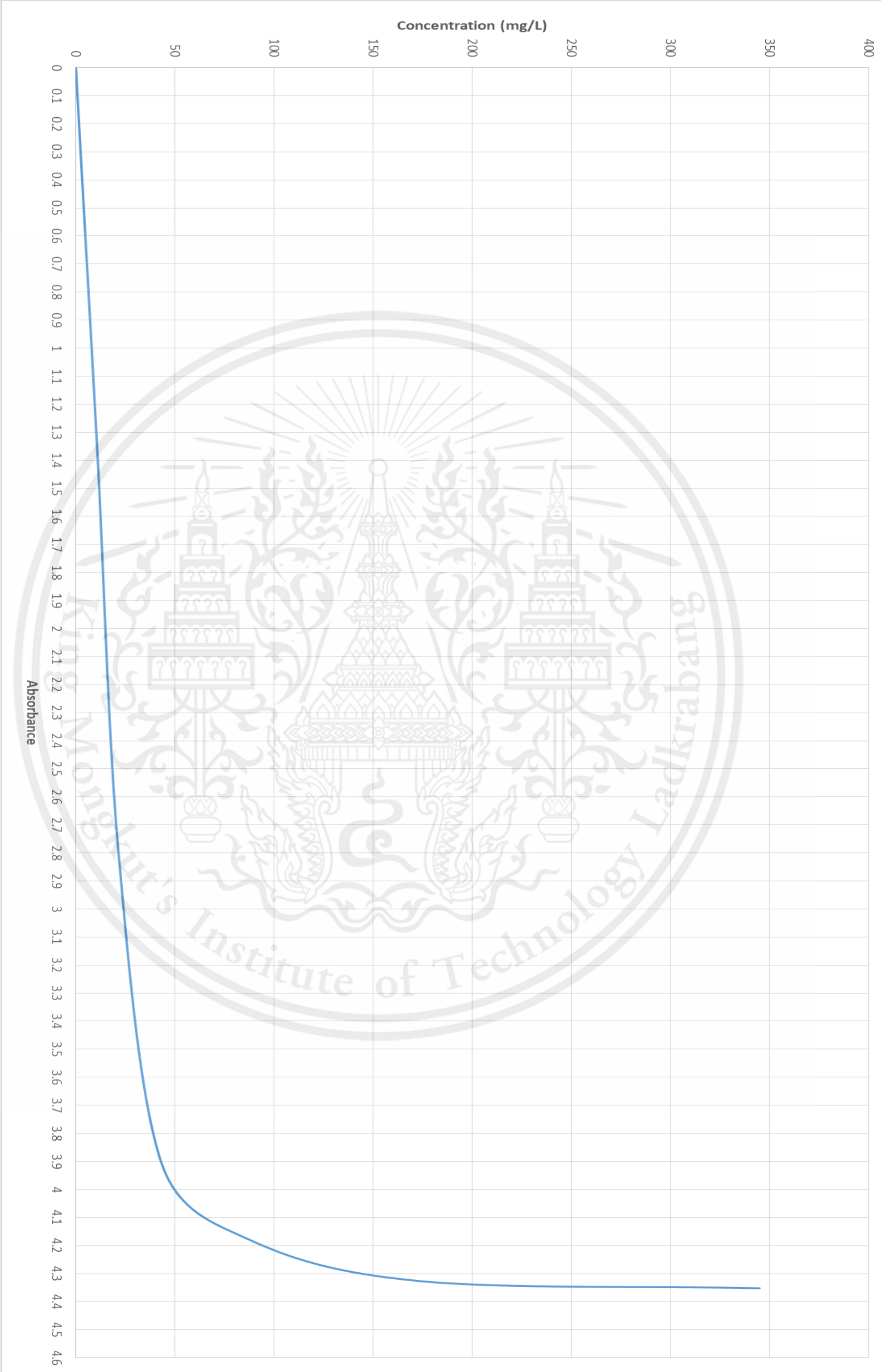

Figure A.38: The calibration curve shown the relation between concentration and absorbance at the wavelength of 260 nm

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