

ADSORPTION OF ASCORBIC ACID ON ORGANO-BENTONITE CLAY



BY

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DECEMBER, 2022.

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**A RESEARCH WORK SUBMITTED TO THE DEPARTMENT OF
CHEMISTRY, UNIVERSITY OF BENIN, BENIN CITY IN PARTIAL
FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF
BACHELOR OF SCIENCE DEGREE IN INDUSTRIAL CHEMISTRY.**

DECEMBER, 2022.

CERTIFICATION

This is to certify that this work was carried out by **OSARETIN NELSON** of the department of chemistry, Faculty of Physical science, University Of Benin, with the matriculation number **PSC1707374**, in partial fulfillment of the requirements for the award of the Bachelor of Science degree in chemistry.

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DATE

DEDICATION

This project work is graciously dedicated to God Almighty who saw me through my study years. Also to my parents and siblings who gave me support financially and morally throughout this period.

ACKNOWLEDGMENT

I could not have find the right word to express how grateful i am to God for giving me the grace for successfully finish my year of studies in this great citadel of learning and to successfully undertake and finish this project

First of all I will like to thank my project supervisor Dr (Mrs) Omonmhenle for her unwavering support and guidance during the course of this research work, I would also like to thank my course adviser for his word of encouragement and helping hand during this process.

I will also want to thank my project mate for her relentless effort and contribution to make sure this was a success

A big thanks to my mother and my sisters for their support and word of encouragement during my say in university of Benin

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ABSTRACT

This study was design to investigate the adsorption capacity of ascorbic acid on organo-bentonite clay by studying the adsorption capacity of organo-bentonite clay. Kinetic parameters such as time and p^H from the result obtained, amount adsorbed with time increase with increase with time until equilibrium was attained, while for p^H increase in p^H leads to increase in amount adsorbed while equilibrium was attained at p^H 8 to 10.

From the result obtained it will be seen that adsorption capacity varied with different parameter.

CHAPTER ONE

1.1 INTRODUCTION

Due to such superior characteristics, montmorillonite has been applied as carrier of drugs, vitamins and amino acid. The interaction between nanoclay and biomolecules is fundamentally important in different area of research, such as preparation of newly modified nanomaterials for biomedical food and pharmaceutical application (joshi, patel, kavadija and bajaji 2009). Among different type of clay minerals montmorillonite has higher potential in biologically related application because of higher surface area and ion exchange capacity (carreto, Gomes and Toteo . 2006 , chap 115 joshi et al .,2014) .Montmorillonite is a natural clay of the mectite group which has potential in food and pharmaceutical application (joshi et ai.,2009. In addition bentonite clay is not absorbed by the human body during the digestion process and does not have side effect on the intestine's performance digestion and food *intake* (Miles- and Hamilton 1994 Patel Somani, Bajaji and jasra 2006)

Result have proven that intercalation of drugs into montmorillonite layer can lead to improve of delivery procedure, controlled and released (Ambrigo, latterini, Noccheti, *Pegano and Ricco 2012*). Various food supplement and pharmaceutical biomaterial such as DNA and RNA vitamin B1 and B6 have been loaded into montmorillonite. the mechanism of the adsorption monomolecule into

montmorillonite have been reported as hydrophilic intercalation (van der waal hydrogen bonding protonation, (Aguzzi, cerezo, viseras and Caramella 2007).

In this study nanoclay (derive from montmorillonite was used as adsorbent for ascorbic acid (vitamin C). The aim of study was to investigate the adsorption mechanism in order to achieve this goal, it was investigated kinetically.

1.1.1 BACKGROUND OF STUDY.

1.1.1.1 ORIGIN OF CLAY MINERALS

Clay minerals are hydrous aluminium phyllosilicate (e.g kaolin, $Al_2 Si_2 O_5(OH)_4$) sometimes with variable amount of iron, magnesium, alkali metal alkaline earth and other cations found on or near some planetary surface clay minerals form in the presence of water (Kerr2009 formation and occurrence of clay minerals) and have been important to life, and many theories of abiogenesis involves then they are important constituent of soil and been useful to human since ancient time in agricultural and manufacturing.

1.1.1.2 Origin of clay.

On the basic level clay soil is compose of millions of clay particles which are 0.02millimeteer (0.0000787 inches) in diameter or smalle . These particle are tightly spaced, which is why clay is notorious for having poor water or air movement throughout. further, clay particle have a very strong affinity for water.

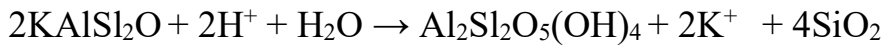
Clay is a type of fin-grained natural soil material containing clay mineral (Olive et al., 2000) clay develop plasticity when wet due to a molecular film of water surrounding the clay particles, but becomes hard, brittle and non plastic upon drying or firing (science leaning Hub 2010) (Breuer2012) most pure clay minerals are white or light- colored but natural clay show a variety of colour from impurity, such as a reddish or brownish colour from small amount of iron oxide. (Nesse 2000). Clay is the oldest known ceramic material, prehistoric human discovered the useful properties of clay use it for making pottery some of the earliest pottery sherd have been dated to around 14,000BC(Scarre2005) and clay tablet were the first known writing medium (Ebert 2011)

1.1.2 FORMATION OF CLAY

Clay most commonly form by prolonged chemical weathering of silicate bearing rocks. They can also be form locally from hydrothermal activity (Foley 1999). Chemical weathering take place largely by acid hydrolysis due to low concentration of carbonic acid. For example CO₂ gas can dissolve in water and form carbonic acid which will becomes ion H⁺ and bicarbonate ion and make water slightly acidic



The acidic water will react with the rock surface and tend to dissolve the K^+ ion and silica from feldspar. Finally, the feldspar is transformed into kaolinite + hydrogen ion + water \rightarrow clay (kaolinite) + cation dissolved + silica



1.1.3 CLASSIFICATION OF CLAY MINERALS

Clay minerals can be classified as 1:1 or 2:1, a 1:1 clay would consist of one tetrahedral sheet and one octahedral sheet and examples would be kaolinite and serpentite

A 2:1 clay consist of an octahedral sheet **sandwiched** between two tetrahedral sheet and example are talc, vermiculite and montmorillonite

1.1.4 CHARGE ON A CLAY MINERAL.

The layer in 1:1 clay are uncharged and are bonded by hydrogen bonds between layers but 2:1 layer have a net negative charge and may be bonded together either by individual cation (such as potassium in illite or sodium or calcium in smectites) or by positively charged octahedral sheet (as in chlorite)

1.1.5 EXCHANGABLE CATION IN CLAY.

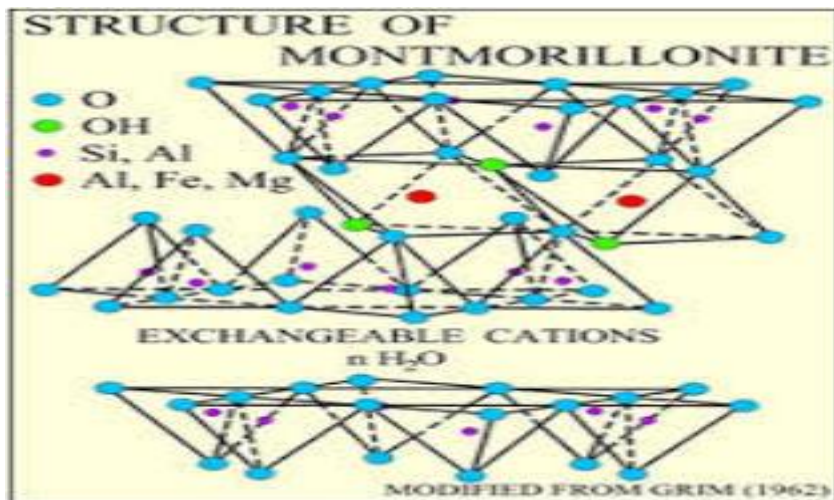
Cation exchange capacity (CEC) is a measure of how many cation can be retained on soil particle surfaces (Brady, Nyle C., WeilRay R .2008) the nature and properties of soil) . Negative charge on the surface of the particle

bind positively charged atom or molecule (cation), but allow these to exchange with other positively charged particle in the surrounding soil water (Birkeland, Peter w 1999). This is one of the ways that solid material in soil alter the chemistry of the soil. CEC affect many aspect of soil chemistry and is used as a measure of soil fertility, as it indicates the capacity of the soil to retain several nutrient (K^+ , NH_4^+ and Ca^+) plant available form, it also indicates the capacity to retain pollutant cation (e.g Pb^{2+})

1.1.6 TYPES OF CLAY MINERALS

Bentonite (montmorillonite)

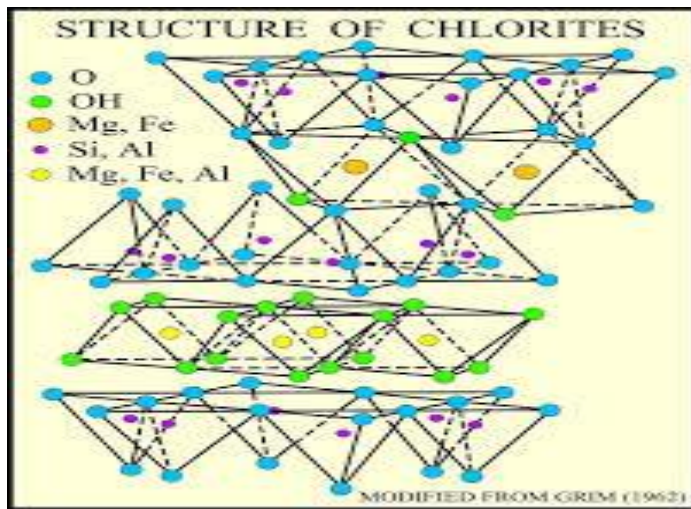
Bentonite is absorbent swelling clay mostly of the montmorillonite (a type of smectite) which can mostly be Na- montmorillonite or Ca-montmorillonite. Na-montmorillonite has the a considerable greater swelling capacity than Ca-montmorillonite. Bentonite usually forms from the weathering of volcanic ash in seawater, or by hydrothermal circulation through the porosity of the volcanic ash bed



1.1.7 CHLORITE CLAY

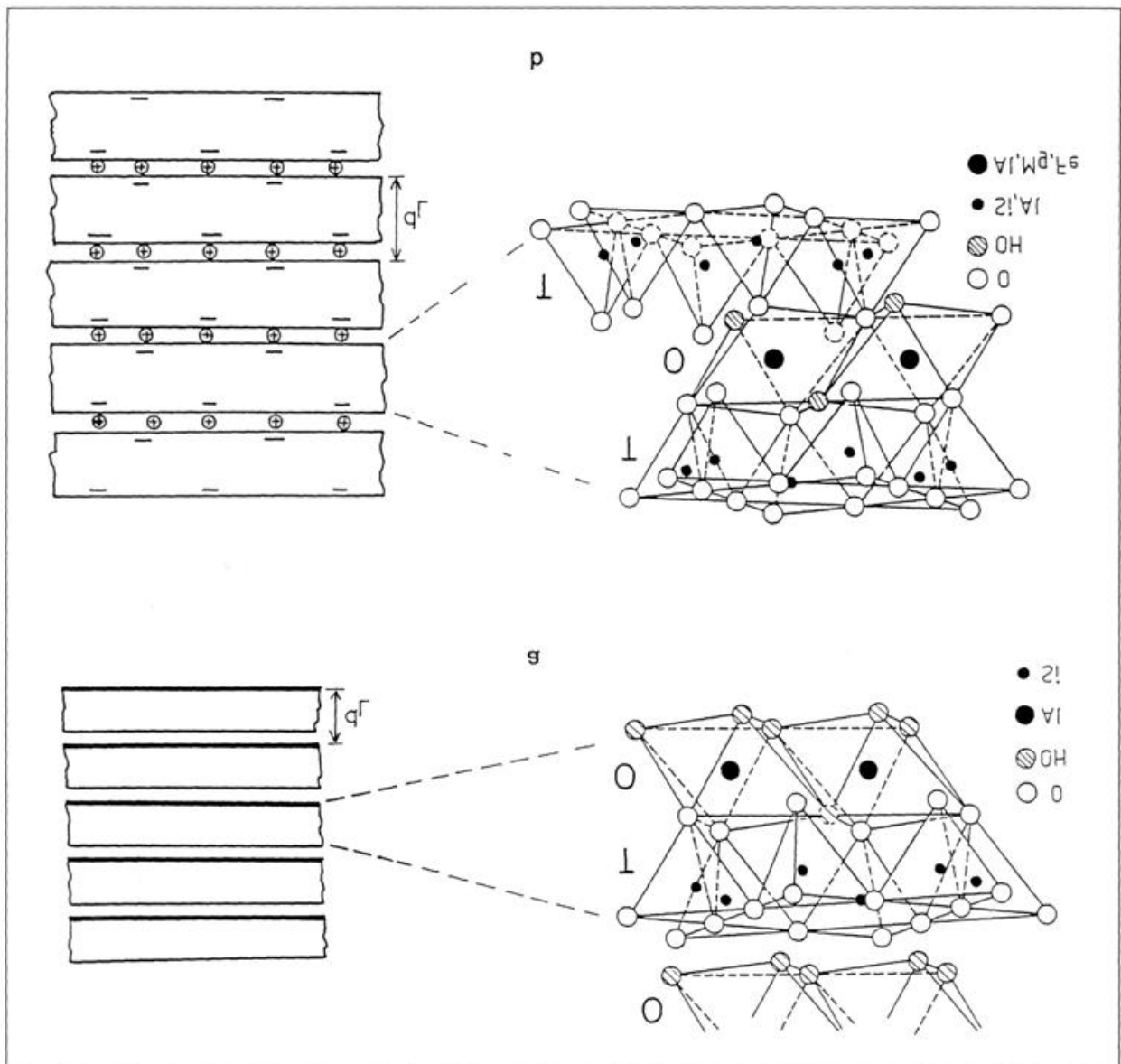
The chlorites are groups of phyllosilicate minerals common in low-grade metamorphic rock and in altered igneous rocks. Greenschist, formed by metamorphism of basalt or other low-silica volcanic rock, typically contain significant amount of chlorite

Chlorite minerals show a wide variety of compositions, in which magnesium, iron, aluminium, and silicon substitute for each other in the crystal structure. A complete solid solution series exist between the two most common end member, magnesium-rich clinochlore and iron-rich chamosite. In addition, manganese, zinc, lithium and calcium species are known. It is a 2:1 clay with interlayer space occupied by a stable, positive charged octahedral sheet, it is a nonexpandable clay and it has a minimum swelling



1.1.8 KAOLINITE

Kaolinite is a clay mineral with a chemical composition $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ it is an important industrial mineral. It is a 1:1 layer silicate mineral with one tetrahedral sheet of silica (SiO_4) linked through oxygen atom to one octahedral sheet of alumina (AlO_6) octahedral (Deer WA, Howie RA, Zussman J 1992). Rocks that are rich in kaolinite are known as kaolin or china clay. Kaolinite is a 1:1 phyllosilicate mineral, is platy in shape. The bonding between the layer are van der waal force and hydrogen bonds (stronger bonding) there is no interlayer swelling, the width ranges from 0.1- 4 μm , thickness 0.05-2 μm . It is nonexpensible clay with low cation exchange capacity (CEC). Kaolinite is use for making paper and pharmaceutical industries



1.1.9 STATEMENT OF PROBLEM

Due to the high cost of some drug carrier, the quest to look for a cheaper more efficient way to deliver to deliver drugs has become a major priority to scientist.

1.1.10 JUSTIFICATION / RELEVANCE OF STUDY.

To prove that clay can be used as a drug delivery agent.

1.1.11 SCOPE OF STUDY

The goal of the study is to determine the adsorption capacity of organo-bentonite clay using vitamin c as adsorbate .

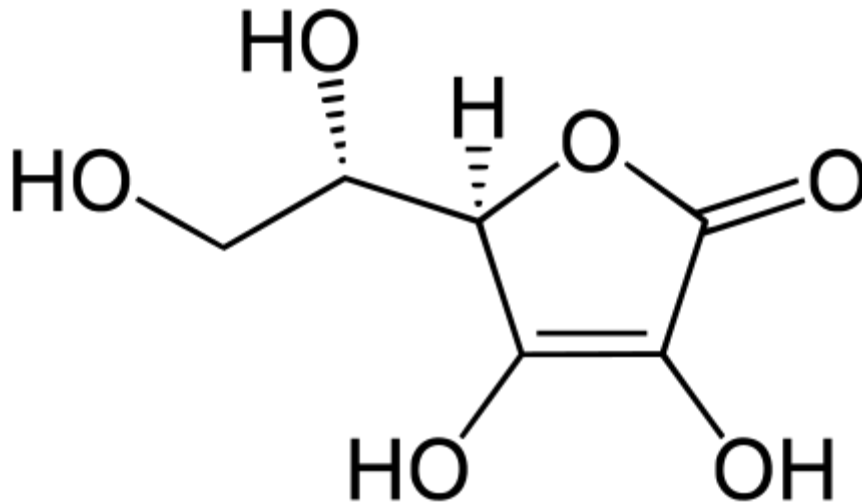
1.1.12 AIM

- The aim of this research is modified the bentonite clay from (MOLY CHEM Convert the clay to Na-bentonite clay and organo- bentonite clay using HDMTA as a surfactant
- Then reduce the size of the clay for adsorption.
- Compare the result to other research

1.2 LITERATURE AND REVIEW

1.2.1 (VITAMIN C)

Vitamin C (also known as ascorbic acid and ascorbate) is a water soluble vitamin found in citrus and other fruit and vegetable, it is also sold as a dietary supplement (the American Society of Health-System Pharmacists 2016) and as a popular serum ingredient to treat melasma (dark pigment spot) and wrinkles and b (Nathan, Patel P 2021) it is used to prevent and treat scurvy. Vitamin C is an essential nutrient in the repair of tissue, the formation of collagen and the enzymatic production of certain neurotransmitters (Institute of Medicine U.S.A. Panel on Dietary Antioxidant-Related Compounds 2000). It is required for the general function of several enzymes and is important for immune system function. It also functions as an antioxidant. Most animals are able to synthesize their own vitamin C, but however, apes (including humans and some primates), most bats, some rodents and certain other animals must require it from dietary



1.2.2 HISTORY

Vitamin C was discovered in 1912, isolated in 1928, and 1933, was the first vitamin to be chemically produced (Squires V.R, 2011) it is on the world Health organization list of essential medicine (world health organization) vitamin C is available as an inexpensive generic and over-the-counter drug party for its discovery, Albert szent-Gyorgyi and walter Norman Hasworth

was awarded the 1937 Nobel prizes in physiology and medicine and chemistry respectively (Zetterstron. R 2009)

1.2.3 CHEMICAL PROPERTIES OF ASCORBIC ACID

Ascorbic acid is vinylogous carboxylic acid and forms the ascorbate anion when deprotonated on one of the hydroxyls. This property is characteristic of reductones: enediols with a carbonyl group adjacent to the enediol group namely with the group – C(OH)=C(OH)-C(=O)-. The ascorbate anion is stabilized by electrons delocalization that result from resonance between two forms

Formula	C ₆ H ₈ O ₆
Molar mass	176.124/mol
Density	1,6949/cm ³
Melting point	190 to 192°C
Boiling	552.7°C

1.2.4 FOOD THAT CONTAIN VITAMIN C

Citrus fruit, kiwifruit, quava, broccoli Brussels sprout, bell papper, potatoes and strawberries.

1.2.5 CHEMISTRY OF ASCORBIC ACID

Ascorbic acid (vit C) is an organic compound with formula $C_6H_8O_6$ originally called hexuronic acid. It is a white solid but impure sample can appear yellowish, it can dissolved well in solvent like water to give mildly acid solution. It is a mild reducing agent.

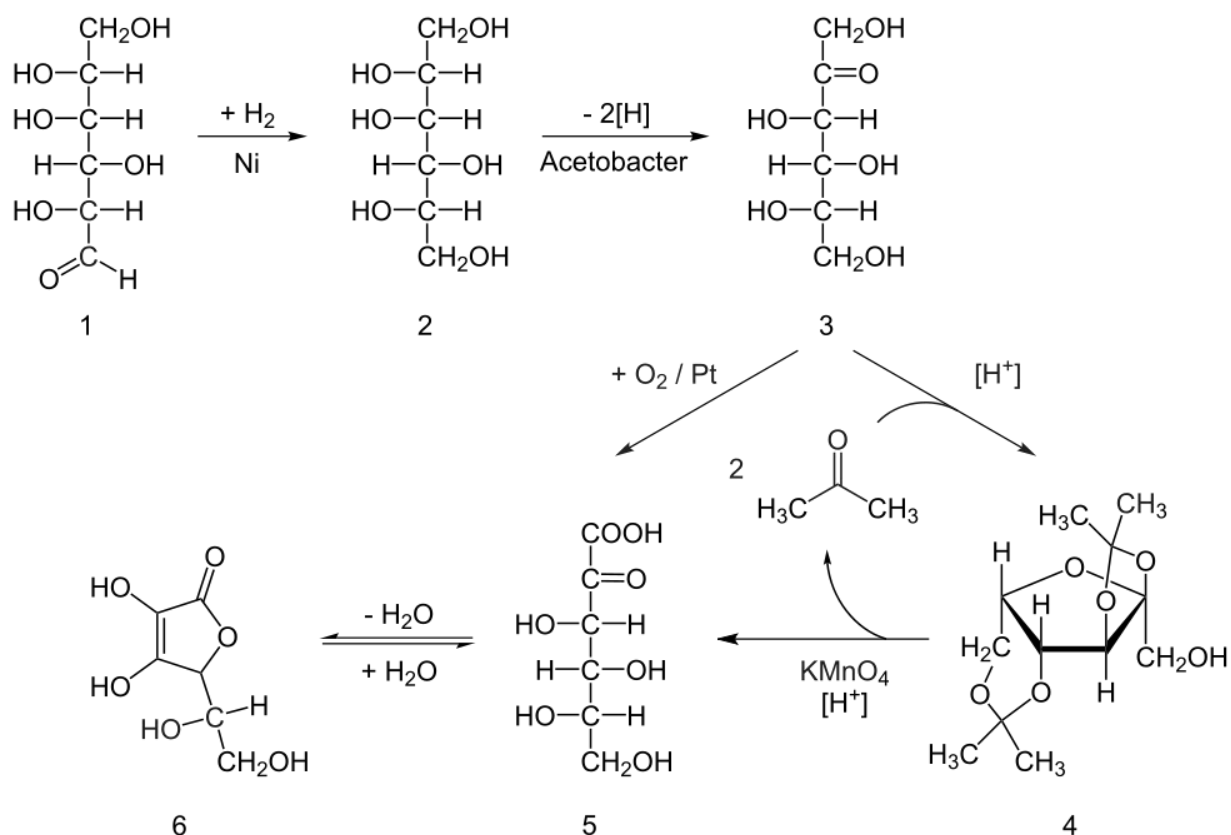
Acid exist as two enantiomers (mirror image isomers) commonly denoted as L (for levo) and D (for dexro) the two isomers is the most often encountered: it occur naturally in many food foods and is one form (vitamier) of vitamin C, an important nutrient for humans and many animals.

For it antioxidants properties the D form can be made via chemical synthesis but has no significant biological

1.2.6 SYNTHESIS

Natural biosynthesis of vitamin c occurs in many plant, and animals by a variety of process

1.2.7 INDUSTRIAL PREPARATION



Eighty percent of the world supply of ascorbic acid is produced in China (Weiss R may 20 2007). Ascorbic acid is prepared in industry from glucose in a method based on the historical Reichstein process. In the first of five-step process, glucose is catalytically hydrogenated to sorbitol, which is then oxidized by the microorganism *Acetobacter suboxydans* to sorbide. Only one of the six hydroxyl groups is oxidized by this enzymatic reaction. From this route are available. Treatment of the product with acetone in the presence of an acid catalyst converts four of the remaining hydroxyl groups to acetals.

1.2.8 DEFICIENCY OF VITAMIN C

Scurvy is a disease resulting from lack of vitamin c (ascorbic acid). Early symptoms of deficiency include weakness, feeling tired and some arms (Agarwal A, Shaharyar, A; Kumar, A, Bhat, MS; Mishra, M 2015) without treatment, red blood cells, gum disease, changes of hair and bleeding from the skin may occur.

As scurvy worsen there can be poor wound healing, personality changes and finally dearth.

1.2.9 SIDE EFFECT OF ASCORBIC ACID

Vitamin cis a water soluble vitamin (vitamin c medline plus medical july 28 2016) with dietary excesses not absorbed, and excesses in the blood rapidly excreted in the urine, so it exhibit remarkably low acute toxicity. More than two to three grams may cause indigestion, particularly when taken on an empty stomach. However, taking vitamin c in the form of sodium ascorbate and calcium ascorbate may minimize this effect (pauling 1976) other symptoms reported for large dose include nausea, abdominal cramps and diarrhea.

1.2.10 ADSORPTION

Adsorption is the adhesion of atoms, ion or molecules from a gas, liquid or dissolved solid to the surface (Glossary. The Brownfield 2009). The process create a film of the adsorbate on the surface of the adsorbate . This process is different from absorption .

1.2.11 DIFFERENCE BETWEEN ADSORPTION AND ABSORPTION

Adsorption and absorption mean quite different things. Absorption involves the whole volume of the material been soaked up by the absorbent while adsorption is mainly a surface phenomenon , which refer to individual molecule, atom or ions gathering on the surface. (Atkins, PW, De. Puala, Julio Keeler 2018)

1.2.12 TYPES OF ADSORPTION

are two types of adsorption., there are

- Physisorption
- Chemisorption

Physisorption:

is adsorption by van der waal force which is a weak force, intermolecular

attraction that occur below the critical temperature of the adsorbate and can lead to development of a monolayer or multilayer

chemisorption

This is a type of adsorption which involves a chemical reaction between the surface and the adsorbate. New chemical bonds are formed at the adsorbate surface. Examples are microscopic phenomena that can be very obvious like corrosion and subtler effects associated with heterogeneous catalysis where the catalyst and the reaction are in different phases. Strong interaction between adsorbate and substrate surface creates new kinds of electronic bonds (Oura K; Lifshits, V.G SARANIN, A.A Zotov, A.V Zatoiyama).

1.2.13 ADSORPTION KINETICS

Adsorption kinetics is the measure of the adsorption uptake with respect to time at a constant pressure and concentration. Adsorption kinetics describe the rate at which solute is adsorbed and the residence time of the adsorbate on the solid-liquid interface

The adsorption of gas and solutes is usually described through isotherm, that is amount of adsorbate on the adsorbent as a function of its pressure (if gas) or concentration (for liquid phase solute) at constant temperature. Quantity adsorbed is always normalized by the mass of the adsorbent to allow comparison of different materials (Foo, K, Y; Harneed B.H. 2010)

1.2.14 FREUNDLICH ADSORPTION ISOTHERM.

To show the variation of the amount of gas adsorption per unit mass of the adsorbent with pressure at constant temperature, Freundlich suggested an empirical equation which is known as Freundlich adsorption isotherm. The equation is:

$$\frac{x}{m} = Kp^{1/n}$$

Where x is the mass of the gas adsorbed, m the adsorbent, p is the equilibrium pressure and k and n constant which depends on the nature of the adsorbate and adsorbent and on the temperature. This equation is applicable only at low pressure.

1.2.15 THE LANGMUIRE ADSORPTION ISOTHERM

Langmuir derive an expression from theoretical consideration for type I isotherm. He was first to point out that in the chemical adsorption of a gas on a solid surface a layer single molecule in thickness is formed. He proposed that adsorption process consist of two opposing action- (i) adsorption of molecule from gas phase on the surface and (ii) desorption of molecule from the surface back into the body of the gas. At equilibrium the two rate become equal.

He derive an equation base on the following assumption:

- 1 an adsorbent side adsorbs single molecule I thickness.
- 2 adsorption of other molecule on this side is restricted
- 3 there is no interaction between the adsorbed molecules

CHAPTER TWO

MATERIALS AND METHOD

2.1 MATERIALS

- Bentonite Clay (from MOLY CHEM)
- Sulfactant (hexyl decyltrimethylammonia bromide)
- Measuring Cylinders
- Beakers (500ml and 250ml)
- Filter paper and funnel
- Erlenmeyer Flask.
- Ascorbic Acid. (from LOBA CHEMIE)

2.2 REAGENTS

- Distilled water
- Deionized water
- Sodium Chloride NaCl
- Silver Nitrate AgNO₃
- Nitric acid HNO₃

2.3 EQUIPMENT

- UV Spectrophotometer
- Centrifuge
- Mechanical Shaker
- pH meter
- Oven
- Water bath
- Weigh Balance (Produce Mettler pm 2000)

2.4 METHOD

2.4.1 PREPARATION OF SODIUM CHLORIDE (NaCl)

Preparation 0.5M of Sodium Chloride.

To prepare 0.5M of sodium Chloride

$$\text{Molarity} = \frac{\text{mass}}{\text{mass mass}}$$

$$0.5 = \frac{x}{58.44}$$

$$X = 0.5 \times 58.44$$

$$X = 29.22\text{g}$$

Then 29.22g of NaCl was measure and dissolved in 1L to prepare 0.5M of NaCl

2.4.2 PREPARATION OF CALCIUM CHLORIDE

Preparation 0.5M of Calcium Chloride.

To prepare 0.5M of Calcium Chloride

$$\text{Molarity} = \frac{\text{mass}}{\text{molar mass}}$$

$$0.5 = \frac{x}{55.5}$$

$$X = 0.5 \times 55.5$$

$$X = 27.75\text{g}$$

Then 27.75g of cacl was measure and dissolved in 1L to prepare 0.5M

2.4.3 PREPARATION OF SODIUM BENTONITE

Sodium bentonite was prepared by stirring raw bentonite for 24h with 1M NaCl.

The suspension was filtered through a filter paper (Watman 47). The filtration cake was rinsed with deionized water to remove NaCl and other exchangeable cation excess from the sample. Rinsing was repeated until the filtrate was Cl⁻ free (tested with 0.1M AgNO₃)

The same process was carried out using calcium chloride (CaCl). But the difference is that the calcium bentonite did not swell compare to the sodium bentonite.

Therefore, the sodium bentonite was chosen for further preparation

The sodium bentonite was dried and ground to obtain a particle size of 200 μ m in order to increase the surface area.

2.4.4 PREPARATION OF ORGANO BENTONITE USING HEXYL DECYLTRIMETHYLAMMONIUM BROMIDE

Organo-bentonite was prepared by the slow addition of HDTMA-bromide solution into a suspension of Na-bentonite in water. The procedure was adopted according to Baskaralingam et al; and consisted of 24h stirring at room temperature followed by washing with deionized water until a negative bromide test with 0. 1M AgNO₃ was obtained. The sample was dried at 80°C , denoted HDTMA-bentonite, and used for further adsorption studies. organo clay was dried and ground to a particle size of 200 μ m to increase the surface area for adsorptio

2.5 DETERMINATION OF MAXIMUM ABSORPTION WAVELENGTH OF ASCORBIC ACID.

A solution of ascorbic acid was prepared at different concentration (300, 350, 400, 450, 500, 550, 600,650, and 700mg/l) for UV visible full wavelength scanning, in order to determine the maximum absorption wavelength spectral scanning of ascorbic acid solution at different concentration were carried out with a UV- visible spectrophotometer (model 752)

Ascorbic acid solution was selected as reference and the characteristic absorption of ascorbic acid at about 280nm was selected for monitoring the absorption of ascorbic acid by organo bentonite. A significant decrease in the transmittance at about 280nm can be assigned to absorption of light caused by the excitation of electron from the valence band to the conduction band to the ascorbic acid solution (A Mittal et al., 2007)

Show plotted graph.

2.6 OPTIMUM P^H DETERMINATION

Absorption experiment were carried out by using batch technique.

To determine the optimum P^H values where the maximum absorption was achieved.

1g of ascorbic acid was dissolve in 1000ml erlenmeyer flask which is equivalent to 1000mg/l, then 500mg/l was prepared from the stock solution of 1000mg/l using dilution formula. 1g of organo clay was measured into five beaker and 20ml of the 500mg/l was added into the four beaker. And the P^H was adjusted from 2, 4, 6, 8 and 9. NaOH and HNO₃ was use to adjust the p^H and agitated for 2hours at constant speed of 200rpm then follow by centrifugation, which was carried out at 400rpm for 30minutue

2.7 OPTIMUM WEIGHT DETERMINATION.

500mg/l of ascorbic acid was prepared by weighing 1g of ascorbic acid into 1000ml then 50ml was taken and making it to 100ml, and 20ml was taken and 0.2g, 0.4g, 0.6g, 0.8 and 1g of the organo bentonite clay was added to 20ml of the ascorbic acid, and then added into five sample and agitated for one hour 30 minute and then followed by centrifugation for 30mintues. Then the absorbance was taken to get the maximum weight.

Amount adsorbed at different time at 500mg/l

500mg/l of ascorbic acid was prepared by weighing 1g of ascorbic acid into 1000ml of distilled water. Then dilution formula was used to get 500 mg/l. 1g of organo clay was weighed into five erlenmeyer flask and 20ml of the 500mg/l of ascorbic acid was added and was agitated at different time of 10, 20, 30, 40 and 60 minutes. Then it was centrifuge and the absorbance was taken.

CHAPTER THREE

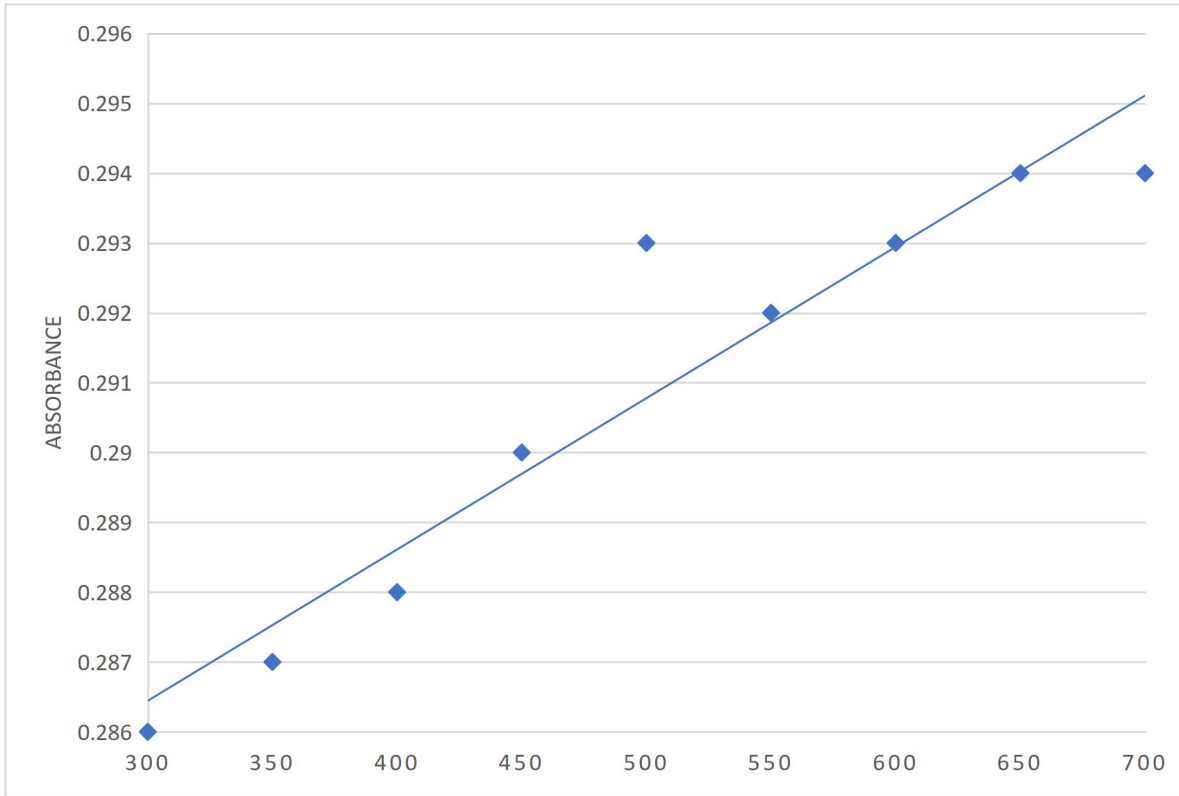
RESULT AND DISCUSSION

3.1 Calibration Curve

Concentration (500mg/l mg/l) absorbance(at 280nm)

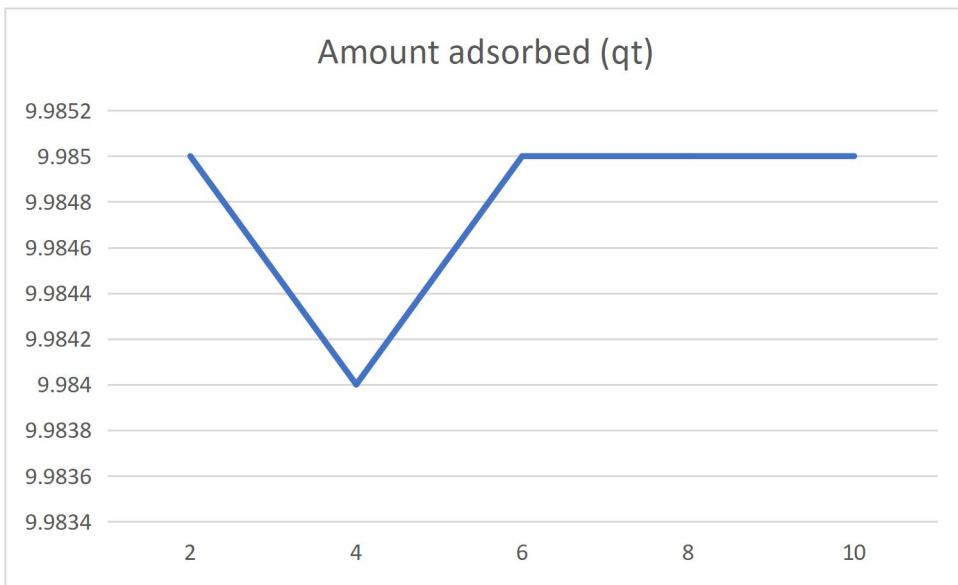
Concentration (mg/l)	Absorbance (nm)
700	0.294
650	0.294
600	0.293
550	0.292
500	0.293
450	0.290
400	0.288
350	0.287
300	0.286

From the table above, it could be seen that increase in concentration leads to increase absorbance which is in accordance with beer lambert law



3.2 Amount adsorbed at different pH at 500mg/l

pH	Amount adsorbed
2	9.985
4	9.984
6	8.695
8	7.545
10	7.545

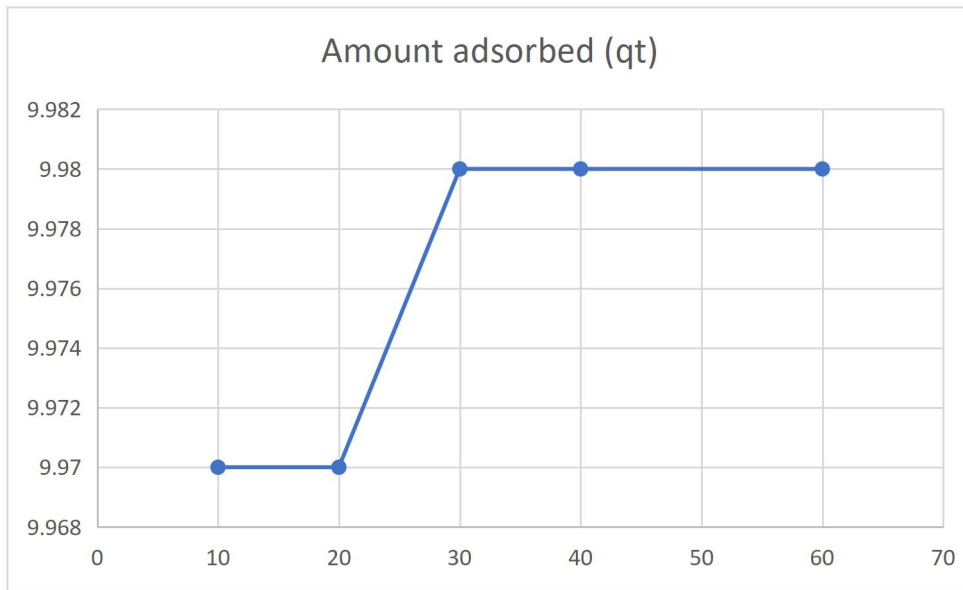


DISCUSSION

Amount adsorbed increase with increase in p^H , but maintain an equilibrium at p^H 8 and 10

3.3 Amount adsorbed different time at 500mg/l

Time (min)	q_t
10	9.97
20	9.97
30	9.98
40	9.98
60	9.98



DISCUSSION

It can be observe from the table, the amount ascorbic acid adsorbed at different p^H at 500mg/l. p^H 2 give the highest amount adsorbed of 9.985 and 9.984, and there was a gradual decrease from p^H 2 to 10 of the table which shows that the amount adsorbed increase with decrease in p^H

The amount adsorbed at different time at 500mg/l, time 60minute give the highest amount of ascorbic acid adsorbed and equilibrium was attain from time 10 to 20 and from time 30 to 60. From the effect of time there was no much effect on the amount of ascorbic acid adsorbed on the organo-bentonite clay.

CONCLUSION

From the study carry out the effect of p^H , p^H 2 give the highest amount of ascorbic adsorbed on the organo-bentonite clay. Increase in p^H leads to decrease in the amount adsorbed.

While the effect of time at time, time 10 to 20 give the same amount of ascorbic acid adsorbed, while time 30 to 60 also give the same amount of ascorbic acid adsorbed . Further parameter are to be investigated to know their effect .

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