**Characterization of Activated Carbon sample: Cu+2 Adsorption Isotherm**

A Major Qualifying Project

Submitted to the faculty of the

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Worcester Polytechnic Institute

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Submitted by:

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# **1. Abstract**

Activated carbon serves as an excellent adsorbent and while we use carbon today in many of our everyday separation techniques such as filtering water and air in the home, are still unsure of the characteristics of most activated carbons. Considering that characteristics are different for every type of activated carbon made from different source materials through different methods, scientists have a lot of studying to do in order to characterize them all and learn what applications each type of activated carbon is best for.

In this paper, the adsorption of copper ions out of aqueous solution is studied using a 16hr glucose char in order to determine the specific surface area, the maximum capacity and the Langmuir constant for this char. Upon calculation of these values it was found that the 16hr glucose char has a large specific surface area on the order of magnitude of 1015 and can hold a maximum number of adsorbed ions of copper: 2.455\*10-4mol/g; also, the Langmuir constant was found to be 40.34. This study will only cover a small part of the characterization process, using the Langmuir Isotherm to determine the parameters mentioned above, and will pave the way for future teams to continue the characterization of this particular char.

# **2. Acknowledgements**

I would like to acknowledge the people that supported me and helped me drive this project to completion. Irreplaceable support came from one of my graduate student advisors, Avery Brown, who was always willing to work with me regardless of day or time. I’d also like to thank another graduate student advisor, Brendan McKeogh for his continued support and supply of research material at the beginning of this project. Finally, I’d like to thank my advisor, Dr. M. Timko, for being incredibly supportive and understanding of my circumstances and for allowing me to take the time I needed to complete this paper.

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# **4. Introduction**

Since the dawn of recorded history carbon has been used in a wide variety of disciplines for an even wider array of functions such as medicine for digestive issues in the days of Hippocrates, and being used in gas masks in World War 1 to protect soldiers from chlorine, phosgene and mustard gas.4 Today activated carbons are used for an even wider range of uses, most of which can be characterized under adsorption. Some examples of industries and processes that activated carbon adsorption is essential to include: food processing, decolorization of oils and sugars, chemical and pharmaceutical industries, filtration of toxic components from exhaust gases (particularly sulfides), nuclear technology and medicine.5

Scientist have studied the structure of carbons (activated or not) and the mechanisms by which adsorption occurs for decades. Before activated carbon adsorption can be applied to new processes, the structure and mechanisms by which it works must be studied and characterized. Today, scientists around the world are studying functional groups on the surface of activated carbons in order to determine the surface chemistry and thereby determine the best uses for this old but continuously developing technology.

Several methods of modelling and studying the characteristics of carbons have already been derived and are in use today. These models are packed with important information about the carbon in question such as the specific surface area, the adsorption capacity, average pore size and the heat of adsorption.5 They cannot, however, tell us more specific details such as pore distribution, pore size distribution, surface composition or different source carbon materials.4 There several different types of these models which will be described in the Theory section of this paper, but for the purposes of this study, the Langmuir isotherm is used to characterize the activated carbon studied.

The carbon studied in this paper is a thermal hydro char sample made from glucose sugars. These chars are made as a model for the eventual production of activated carbon from biomass material that could become an excellent product in developing countries used for filtration of water and air where current modern technology is difficult to appropriate. These chars are tested and characterized as a model for the characterization of biomass chars and in this paper, the Langmuir isotherm is used to determine the specific surface area, adsorption capacity and the adsorption coefficient.

# **5. Theory**

## **5.1 Adsorption**

### **5.1.1 What is Adsorption?**

Adsorption is a separation technique often used in industry and is one of three different types of sorption operations. Sorption is a generalized term describing the selective transfer of one substance (sorbate) to the surface of or into the bulk of another substance (sorbent). Adsorption occurs when the sorbate (adsorbate) molecules bond to the surface of the sorbent (adsorbent) through chemical bonds or weak intermolecular forces. In a typical adsorption process the adsorbent is a porous solid and the adsorbate is a fluid (gas or liquid mixture) that the adsorbent is submersed in. Efficient separation requires a large surface area per unit volume of adsorbent which can be achieved with highly porous solid particles. Adsorption may be classified as *chemisorption* or *physical adsorption* depending on the type of bonding between the adsorbent and adsorbate.2,5

#### **5.1.1.1 Chemisorption**

Chemisorption occurs as a chemical bond between the adsorbate and the adsorbent in a monomolecular layer on the surface of the adsorbent. This chemical reaction is typically exothermic and the enthalpy change typically ranges from 40 – 400 KJ/mol. Chemisorption is generally slow and irreversible and is the operation of separation observed in catalysis.5

#### **5.1.1.2 Physical Adsorption**

Physical adsorption of gaseous adsorbates occurs when the intermolecular forces between the gas molecules and the solid’s surface are stronger than the intermolecular forces between the gas molecules themselves. These forces can be dipole moments, polarization forces, dispersive forces, or short-range repulsive interactions i.e., Van der Waals forces. Physical adsorption of gas molecules onto the surface of the adsorbent is not unlike condensation and is similarly exothermic. The observed enthalpy change for this process is called the enthalpy of adsorption and is on the same order as the enthalpy of vaporization ranging from approximately 10 – 20 KJ/mol.2

For adsorption of a pure liquid adsorbate, there is no simple experimental method to measure the extent of adsorption, therefore, only liquid mixtures including dilute solutions are used.2 Comparative to gases, the theoretical analysis of adsorption from a solution and derivation of a fitting adsorption equation is much more difficult. This is because both components of the solution will compete for available surface area and also because thermal motion of molecules in the liquid phase is not understood as well as gas phase thermal motion is.5 The equilibrium states of liquid-solid systems can still be represented by isotherms by replacing instances of pressure with concentration within derived adsorption equations.2 The Langmuir isotherm will be used to represent the experiments in this project and will be discussed further on.

### **5.1.2 Adsorption Equilibrium**

Adsorption equilibrium is achieved when the rate of adsorption is equal to the rate of desorption. When an adsorption process starts, the rate of adsorption, defined as the rate at which molecules strike and stick to the surface of the adsorbent, is initially large because the entire surface area of the adsorbent is available for adsorption. As the surface of the solid becomes covered in adsorbed gas molecules, the rate of adsorption decreases. Simultaneously, the rate of desorption increases. Desorption is the process by which adsorbent molecules evaporate or rebound off the surface of the adsorbent back into the bulk fluid. As the rate of adsorption decreases and the rate of desorption increases, they reach a point at which they are equal. At this point the system has reached equilibrium. This type of equilibrium is called a dynamic equilibrium.2,5

It is in this state of equilibrium that adsorbate-adsorbent systems are typically observed and studied. Considering that the equilibrium of a system depends upon pressure and temperature there are several ways that this state may be represented: the adsorption isotherm, requiring constant temperature; the adsorption bar requiring constant pressure and the adsorption isostere requiring constant equilibrium adsorption. In practice, keeping an adsorbent-adsorbate system at constant temperature is simplest and so the adsorption isotherm is the most widely used method to represent the equilibrium state of an adsorbent-adsorbate system.

#### **5.1.2.1 Adsorption Isotherm**

The adsorption isotherm can be used to derive many different properties of the adsorbent and adsorbate such as the surface area, pore volume and size distribution, relative adsorbability and the enthalpy of adsorption. Many adsorption isotherm equations have been derived including the Langmuir, the Freundlich, the Brunauer-Emmett-Teller (BET) and the Dubinin equations. These are some of the most widely used isotherms but for the purposes of this project we will be using the Langmuir isotherm equations to represent the equilibrium state of our systems.

According to Bansal and Goyal, there are three theoretical approaches for the derivation of an adsorption isotherm:

* The kinetic approach
* The statistical approach
* The thermodynamic approach

Of these we will be using the kinetic approach to derive the Langmuir isotherm considering the kinetic approach uses the condition of equilibrium which states that the rate of adsorption must be equal to the rate of desorption.

#### **5.1.2.2 Langmuir Isotherm Derivation**

If it is assumed that the number of sites filled at equilibrium is *θ* and the number of sites not filled is *θo* such that *θ + θo = 1*, then rate of adsorption, denoted as *ra*bs can be written as,

equ. 5.1

where *a* is the adsorption coefficient, the fraction of molecules adsorbed onto the surface of the sorbent, *p* is pressure at equilibrium in the system and *k* is a constant written as,

equ. 5.2

The rate of desorption, *rdes* is written as,

equ. 5.3

where *Zm* is the number of adsorbed molecules per unit area of the surface of the adsorbent and is the oscillation frequency of molecules perpendicular to the surface. T is the temperature at equilibrium and R is the universal gas constant. When adsorbate condenses onto the surface of the sorbent, it is an exothermic process, therefore, E, the energy of adsorption must be negative.

If both rates are equated to each other, the resulting equation is,

equ. 5.4

equ. 5.5

Let,

equ. 5.6

*b* is also known as the Langmuir constant and can be rewritten as,

equ. 5.7

where is the rate constant for desorption and *k* is Henry’s constant. Substituting *b* into equation 2.5 thus gives,

equ. 5.8

equ. 5.9

Once equated, equations 2.1 and 2.3 ultimately reduce to equation 2.9 otherwise known as the Langmuir Isotherm. Θ, the fraction of sites occupied can be replaced by a number of different factors such as, the mass fraction of substrate adsorbed, *x/xm*, the ratio of volume of gas adsorbed to the monolayer volumetric capacity of the surface, *V/Vm,* or the the ratio of number of moles adsorbed to the surface to the monolayer molar capacity, *n/nm,* all at equilibrium. This allows for analysis of different forms of data collected from practical experiments. For this project, θ will be replaced with *n/nm* to give,

equ. 5.10

Finally, this equation considers a system where the sorbate is a gas, in order to correct for this, pressure, *p*, will be replaced by concentration, *Ce*.

equ. 5.11

Rearranging equ. 2.11 into a linear form will allow for a graphical analysis as well in which the slope of the graph will give *1/nm* and the intercept will give *1/nmb*.

equ. 5.12

#### **5.1.2.3Graphical Representation of a Langmuir Isotherm**

In order to obtain numerical values from equ. 2.12, experimental data must first be collected and manipulated into graphical representation. The experiment documented in this paper, produces light absorbance of the solution after being shaken for 24hrs with a certain activated carbon sample. Using an absorbance calibration curve for Cu2+, the concentration of the solution can be determined. This is the equilibrium concentration, *Ce*.

Knowing *Ce* will allow us to calculate *n* using the equation,

equ. 5.13

where *V* is the volume of coper solution and *m* is the mass of activated carbon used.3 Together *Ce* and *n* constitute on data point on a graphical representation of a Langmuir isotherm. In order to obtain more data points, more experiments must be conducted with varying initial concentrations of copper solution. Once several data points have been obtained, an isotherm can be constructed and will resemble a graph like this,

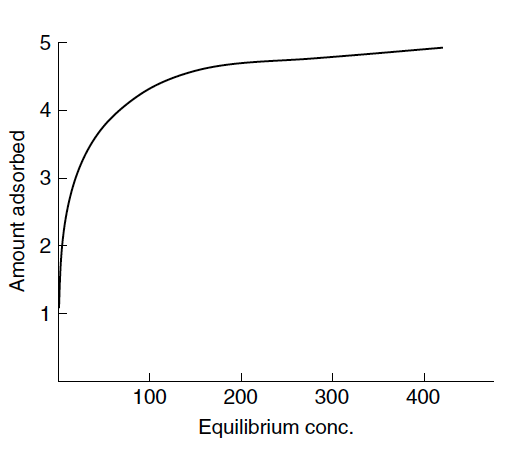


Figure - Example of a typical Langmuir Isotherm (Bansal & Goyal, 2005)

Once the graph is constructed, any point can be chosen and the slope taken at that point to find *1/nm* or the y-intercept found to determine *1/nmb*.

This graph can also be used to find the surface area per unit mass of the adsorbent using this equation,

equ. 5.14

where N is Avogadro’s number, S is the contact surface area of a single molecule of adsorbate and M is the molecular weight of the adsorbate.8

## **5.2 Activated Carbon**

### **5.2.1 What is Activated Carbon?**

The term activated carbon includes a wide range of carbon based materials that have been processed in order to achieve high pore density and extensive internal surface areas. They are created through the combustion, partial combustion or thermal decomposition of carbonaceous raw materials. Activated carbon is manufactured in all different shapes and sizes including spherical, fibrous and even cloth form for special applications.5,4 According to Bansal and Goyal the granular form of activated carbon has small pores and an extensive internal surface area and the fine powder form exhibits larger pores diameters and thus a smaller internal surface area. For this project, the granular form of activated carbon will be used.

The large surface area and porous nature of activated carbons make it a good adsorbent. As such, they are used in industry for a number of different applications such as the purification of waste water and air, solvent recovery and in the chemical industry. Activated carbons are even used in the home for removal of odor, color, taste and organic and inorganic impurities in air and drinking water. Whether in industry or in the home, activated carbon is used as an adsorbent in separation processes involving both gases and liquids. “Nearly 80% (~300,000tons/yr) of the total active carbon is consumed for liquid-phase applications, and the gas-phase applications consume about 20% of the total product.” (Bansal and Goyal, 2005).

### **5.2.2 How is Activated Carbon made?**

In theory, all carbonaceous raw materials could be made into activated carbon but in reality only some can be used commercially.4 There are certain requirements carbonaceous materials have to achieve before they can be used to make commercial activated carbon. The raw material should have low levels of inorganic impurities, should be cheap and easily accessible, should have a long shelf life and should be easily activated. There is a great effort in industry now to recycle waste materials from industrial processes such as waste water treatment sludge and agricultural waste to make activated carbon.

There are two main steps in the production of commercial activated carbon: carbonization and activation. The choice of raw carbonaceous material used has more than economic significance as it can also influence the properties of the end product. The first step, carbonization, or otherwise known as pyrolytic decomposition, breaks down the carbonaceous material at temperatures lower than 800˚C resulting in a material called char. Impurities such as oxygen, nitrogen and hydrogen would be eliminated as volatile gases and what remains is a material consisting of mostly carbon atoms. The carbon in the char now forms highly disorganized stacks of flat aromatic rings that are cross-linked forming free interstices between them. These interstices eventually give rise to pores but at this stage may be filled with ash or unreacted carbon and as a result the adsorption capacity of the char is lowered. In order to achieve a higher adsorption capacity, the interstices are developed into pores through an oxidative treatment. This is called the activation step.

During the activation step of the process the char is reacted with air, CO2, and steam at temperatures between 800˚C and 900˚C.4,5 This oxidizes the char and clears the interstices giving rise to an extensively porous structure consisting of mostly micropores. The product obtained, therefore, has a high surface area reaching up to several thousand m2/g and an excellent adsorption capacity.

# **6. Experimental**

## **6.1 Preparation of Reagents**

The reagents used in the model experiment were Cu2+ solutions at varying concentrations made with Cu(NO3)2 ∙ 2.5H2O, and a 16hr glucose char prepared before hand by a separate team within the lab. Eight 30ml Cu2+ solutions were mixed with concentrations varying between 0.01M and 0.08M. These concentrations were chosen in accordance with an absorbance calibration curve for Cu2+ solutions constructed by another team working on a separate project within the lab.6 The calibration curve can be seen in the appendices. Once the solutions were prepared, they were shaken with 0.4g of the 16hr glucose char for 24hrs.

## **6.2 Adsorbent-Adsorbate System Equilibrium**

All eight solutions were shaken while controls of their corresponding starting concentrations were stirred for 24 hrs. Two runs were conducted where all eight concentrations were shaken with the 16hr glucose char twice. According to a paper written by a previous team, the system is assumed to be in equilibrium after 24hrs and that data taken from the system at this time can be considered equilibrium data.6 Therefore, samples of the solutions were taken and examined in a UV-vis spectrometer. The absorbances of the solutions, before and after being shaken, were recorded and a calibration curve constructed. This calibration curve was then used to determine the equilibrium concentrations of the solutions after being shaken.

## **6.3 Constructing the Langmuir Isotherm**

Once the equilibrium concentration, *Ce*, was known, it was used to calculate the number of moles of Cu2+ adsorbed per gram of char, *n*. *Ce* and *n* where then used to construct the Langmuir isotherm graph. From the graph the slope and y-intercept were used to find the monolayer molecular capacity of the char, *nm*, and the Langmuir constant, *b*, respectively. Then, using *nm* and *b*, the surface area per unit mass of the char and the adsorption coefficient were calculated as well. All sample calculations can be found in the appendices.

# **7. Results and Discussion**

## **7.1 Copper II Nitrate Absorption Calibration Curves Comparison**

Two calibration curves were utilized in this paper. One derived from data obtained during the experiments described in this paper, and another provided by a previous team working in the lab. Both curves were constructed using the same methodology described above.



Figure - Absorption curve for Copper II Nitrate provided by Escapa et al.

Figure - Absorption curve for Copper II Nitrate derived from experimental data

Both graphs present a linear equation to describe a best fit line for the data and seem to vary slightly on the slope of the line and y-intercept by 0.02 and 0.05 respectively. When the experimental absorption data for the copper nitrate solutions post-adsorption were fed into both equations, the concentrations that were calculated differed by ~0.0005M. In order to reduce the error this might introduce into further calculations these concentrations were averaged and the averages used in equations from there on. The same was done for the initial concentrations of the copper nitrate solutions and the averaged calculated concentrations were plotted against their original absorbances to create a new calibration curve with greatly reduced error.

Figure - Calculated Average Copper II Nitrate Calibration Curve

This curve will not be used to determine concentrations for the follow up calculations and was constructed for the use of future teams in need of an accurate copper II nitrate calibration curve. However, the averaged initial concentrations calculated for this graph will be used in further calculations.

## **7.2 Constructing the Langmuir Isotherm for both experiments**

Using the averaged equilibrium concentrations calculated from the calibration curves the number of moles of copper adsorbed, *n*, was calculated for each system (each data point). They were then used to construct two graphs: *Ce* Vs. *n* and *Ce/n*Vs. *n*. Both of these graphs represent two different forms of the Langmuir isotherm.

Figure - Non-linear Langmuir Isotherm for the first set of experiments

Figure - Linear form of the Langmuir Isotherm for the first set of experiments

Figure - Non-linear Langmuir Isotherm for the second set of experiments

Figure - Linear form of the Langmuir Isotherm for the second set of experiments

Looking at the first data set in comparison with the second, it is clear that there are significant error concerns and these will be discussed later on in this section. Considering the drastic errors evident in the data collected from the first set of experiments, only the second set will be analyzed further. It is recognized that this discussion in and of itself could result in larger error margins than if the data sets were averaged, however, it is believed that the error margin associated with the second data set is smaller than the error margin would even if both data sets were merged.

Equ. 2.12 was used to construct the linear forms of the isotherms and as such, the slope and y-intercept of the linear trend line in fig. 7 in used to calculate *nm* and *b* respectively.

equ. 5.12

equ. 7.1

Therefore, mol/g. Now that *nm* has been calculated, *b* and the *surface area* can be determined. Equ. 2.12 is used in conjunction with the linear equation of the isotherm above in order to calculate *b*.

equ. 7.2

Therefore, *b* = 40.34. The equation for surface area, equ. 2.14,

give a *surface area* of 8.737E15 m2/g.

## **7.3 Uncertainty and Error**

During the course of these experiments, there were several instances where error could have been introduced into the results. Examples of such instances include, making of solutions, measuring the mass of char, transference of liquids to and from different containers and the specific incident of the ~0.02M vial breaking after being shaken for 24hrs which, if data had been converged, would’ve resulted in the use of only one data point at that concentration rather than two. However, since the data set obtained from the first set of experiments was not extensively studied, this particular source of error is over looked.

It is uncertain why the data from the first set of experiments produced such uncertain data points, especially considering that the second set of data produced much more reliable data points. This suggests that the error was introduced through something other than the methodology used to conduct the experiment. Most likely the error was introduced during the measurement of concentrations after the 24hrs.

# **8. Conclusions and Recommendations**

In conclusion, the specific surface area, the maximum monolayer capacity, *nm*, and the Langmuir constant, *b*, were calculated for this particular activated carbon sample, 16hr glucose char. The specific surface area was calculated to be 8.737E15m2g-1. The maximum capacity was calculated to be, 2.455E-4mol/g. And the Langmuir constant, *b*, was calculated to be 40.34.

For future teams it is suggested that they construct their own isotherm and merge it with these data to create a more accurate isotherm. It is also recommended that they calculate the adsorption coefficient of the char using data obtained from the isotherm.

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# **10. Appendices**

## **10.1 Appendix A**

### **10.1.1 Calibration Curve data**

|  |  |
| --- | --- |
| Measured Initial Concentration, Co | Absorption |
| 0.01 | 0.122002 |
| 0.02 | 0.238029 |
| 0.03 | 0.398396 |
| 0.04 | 0.454176 |
| 0.05 | 0.575434 |
| 0.06 | 0.694692 |
| 0.07 | 0.800689 |
| 0.08 | 0.938067 |
|  |  |
| Calculated Avg Initial Concentration, Co | Absorption |
| 0.009530492 | 0.122002 |
| 0.019719747 | 0.238029 |
| 0.033857726 | 0.398396 |
| 0.0387753 | 0.454176 |
| 0.049465423 | 0.575434 |
| 0.059979227 | 0.694692 |
| 0.069323678 | 0.800689 |
| 0.081435204 | 0.938067 |

### **10.1.2 Run 1 data**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Initial Conc, Co (M)[avg] | Equilib Conc, Ce (M) [avg] | Amount Adsorbed, n (mol) | Mass of Carbon, m (g) | Volume of sol'n, V (L) | Ce/n |
|  |  |  |  |  |  |
| 0.009530492 | 0.008771045 | 3.79439E-05 | 0.4003 | 0.02 | 231.1582845 |
| 0.019719747 |  |  | 0.4005 | 0.02 |  |
| 0.033857726 | 0.032161425 | 8.44982E-05 | 0.4015 | 0.02 | 380.6167696 |
| 0.0387753 | 0.037312117 | 7.31592E-05 | 0.4 | 0.02 | 510.0129922 |
| 0.049465423 | 0.047283352 | 0.000108777 | 0.4012 | 0.02 | 434.6806502 |
| 0.059979227 | 0.057889567 | 0.000104041 | 0.4017 | 0.02 | 556.4120255 |
| 0.069323678 | 0.066933244 | 0.000119402 | 0.4004 | 0.02 | 560.5691456 |
| 0.081435204 | 0.077052902 | 0.000218514 | 0.4011 | 0.02 | 352.6219666 |

### **10.1.3Run 2 data**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Initial Conc, Co (M)[avg] | Equilib Conc, Ce (M) [avg] | Amount Adsorbed, n (mol) | Mass of Carbon, m (g) | Volume of sol'n, V (L) | Ce/n |
|  |  |  |  |  |  |
| 0.009530492 | 0.008693733 | 4.17649E-05 | 0.4007 | 0.02 | 208.1590287 |
| 0.019719747 | 0.017664645 | 0.000128204 | 0.4002 | 0.02 | 137.7854435 |
| 0.033857726 | 0.031468234 | 0.000151119 | 0.4 | 0.02 | 208.2347951 |
| 0.0387753 | 0.03697789 | 0.000163372 | 0.4 | 0.02 | 226.341662 |
| 0.049465423 | 0.046768869 | 0.000174212 | 0.4 | 0.02 | 268.4595148 |
| 0.059979227 | 0.056869717 | 0.000169544 | 0.4001 | 0.02 | 335.427482 |
| 0.069323678 | 0.066497172 | 0.000188579 | 0.4 | 0.02 | 352.6223599 |
| 0.081435204 | 0.076955699 | 0.000165819 | 0.4006 | 0.02 | 464.094579 |

## **10.2 Appendix B**

### **10.2.1 Sample Calculations**

***Calculating mass of Cu(NO3)2∙2.5H2O needed to make solutions***

Need 100mL of 0.01M Cu(NO3)2∙2.5H2O.

# of moles of Cu(NO3)2 in solution, *n*

mass of Cu(NO3)2∙2.5H2O needed, *m*

# of moles of water present in *m*

Volume of water present in *m*

***Calculating concentrations from calibration graphs***

Calibration curve equation

***Calculating number of moles adsorbed***

Number of moles adsorbed, *n*

***Calculating maximum capacity from isotherm graph***

Langmuir Linear equation

From graph,

Maximum capacity, *nm*

***Calculating Langmuir constant, b***

***Calculating Surface Area***