

**PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBONS
FROM BAMBOO FOR ADSORPTION STUDIES
ON THE REMOVAL OF SURFACTANTS**

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Dedicated to my beloved mother and father

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ABSTRACT

Activated carbon is an adsorbent material which is widely used in pollutant removal and water purification processes. The main characteristics of activated carbon are its high surface area and high adsorption capacity with certain adsorbates. Various materials are reported can be used for the preparation of activated carbon, but the use of bamboo is rarely reported. In this study, four activated carbons were produced using bamboo cane. Three of the mentioned samples were activated chemically by using zinc chloride and concentrated hydrochloric acid as the activating agent. These samples were activated at 700 °C in nitrogen, carbon dioxide and water steam flow respectively. The fourth sample was physically activated in nitrogen flow at 700 °C. The prepared activated carbons were characterized by Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetry Analysis (TGA), Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray Spectroscopy (EDX) and nitrogen adsorption at 77 K. The moisture, carbon and ash content of the activated carbons were determined. The results showed that the activated carbon which was activated with zinc chloride in nitrogen flow is the best activated carbon. It has the highest surface area ($1258.99 \text{ m}^2 \text{ g}^{-1}$), the highest thermal stability and the highest adsorption capacity in the adsorption of non-ionic surfactant (Triton-X 100) and cationic surfactant (cetyl trimethyl ammonium bromide). Its adsorption capacity is higher for non-ionic surfactant compared to the cationic surfactant. All chemically-activated carbons exhibit ability to adsorb both surfactants but none of them had higher adsorption capacity than the commercial activated carbon. The activated carbon prepared from bamboo exhibits good surface characteristics and porosity properties. This indicates that bamboo has the potential to be a promising precursor for the production of activated carbon.

ABSTRAK

Karbon teraktif ialah sejenis bahan penjerap yang luas penggunaannya dalam proses pengasingan bahan pencemar dan pembersihan air. Ciri-ciri karbon teraktif adalah luas permukaan yang tinggi dan kapasiti penjerapan yang tinggi terhadap zat terjerap tertentu. Pelbagai bahan telah dilaporkan dapat digunakan untuk menyediakan karbon teraktif, tetapi penggunaan buluh jarang dilaporkan. Dalam kajian ini, empat karbon teraktif telah dihasilkan dengan menggunakan batang buluh. Tiga daripada sampel tersebut telah diaktifkan secara kimia dengan menggunakan zink klorida dan asid hidroklorik pekat sebagai bahan pengaktif. Sampel-sampel tersebut telah diaktifkan pada 700 °C masing-masing dalam aliran nitrogen, karbon dioksida dan wap air. Sample keempat diaktifkan secara fizikal dalam aliran nitrogen pada 700 °C. Karbon teraktif yang disediakan telah dicirikan dengan Spektroskopi Inframerah Transformasi Fourier (FTIR), Analisis Termogravimetri (TGA), Mikroskopi Elektron Imbasan (FESEM), Spektroskopi Sinar-X Penyerakan Tenaga (EDX) dan penjerapan gas nitrogen pada 77 K. Kandungan lembapan, karbon and abu bagi karbon teraktif telah ditentukan. Hasil kajian menunjukkan bahawa karbon teraktif yang diaktifkan dengan zink klorida dalam aliran nitrogen merupakan karbon teraktif yang terbaik. Ia mempunyai luas permukaan tertinggi ($1258.99 \text{ m}^2 \text{ g}^{-1}$), kestabilan terma tertinggi dan kapasiti pejerapan tertinggi dalam penjerapan surfaktan tak-ionik (Triton-X 100) dan surfaktan kationik (setil trimetil ammonium bromida). Kapasiti penjerapannya adalah lebih tinggi bagi surfaktan tak-ionik berbanding dengan surfaktan kationik. Semua karbon yang diaktifkan secara kimia menunjukkan keupayaan untuk menjerap kedua-dua surfaktan tetapi tiada daripada mereka mempunyai kapasiti penjerapan yang lebih tinggi daripada karbon teraktif komersial. Karbon teraktif yang disediakan daripada buluh menunjukkan ciri-ciri permukaan dan keliangan yang baik. Ini menunjukkan bahawa buluh mempunyai potensi untuk menjadi bahan pemula yang sesuai bagi penyediaan karbon teraktif.

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LIST OF ABBREVIATIONS

APE	-	Alkylphenol ethoxylate
BAC-N2	-	Activated carbon produced by physical activation and activated in nitrogen
BAC-ZC-CO2	-	Activated carbon produced by chemical activation and activated in carbon dioxide
BAC-ZC-H2O	-	Activated carbon produced by chemical activation and activated in water steam
BAC-ZC-N2	-	Activated carbon produced by chemical activation and activated in nitrogen
BDDT	-	Brunauer, Deming, Deming and Teller
BET	-	Brunauer, Emmett and Teller
CAC	-	Commercial activated carbon
<i>CMC</i>	-	Critical micelle concentration
CTAB	-	Cetyl trimethyl ammonium bromide
EDX	-	Energy Dispersive X-ray Spectroscopy
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared Spectroscopy
IUPAC	-	International Union of Pure and Applied Chemistry
LAS	-	Linear alkylbenzene sulphonate
RB	-	Raw bamboo
TGA	-	Thermogravimetry Analysis
TX100	-	Triton-X 100
UTM	-	Universiti Teknologi Malaysia
UV-Vis	-	Ultraviolet-Visible Spectroscopy

LIST OF SYMBOLS

A	-	Absorbance
A_m	-	Average area of one adsorbate molecule in a complete monolayer
b	-	Langmuir constant related to the affinity of the binding site
b_B	-	Path length / sample cell length
C	-	BET constant
c	-	Concentration
C_e	-	Equilibrium concentration
C_0	-	Initial concentration
i	-	Intercept of a linear graph
K_F	-	Empirical constant in Freundlich equation
M	-	Sample weight (in the determination of moisture, carbon and ash content)
m	-	Sample weight (in the surfactant adsorption study)
n	-	Empirical constant in Freundlich equation
N_0	-	Avogadro number
P	-	Equilibrium pressure
P_0	-	Saturation pressure
q_e	-	Ratio of the weight of adsorbed adsorbate to the weight of adsorbent
q_{max}	-	Maximum adsorption at monolayer coverage
R_L	-	Separation factor
s	-	Slope of a linear graph
S_{BET}	-	BET surface area
S_{ext}	-	External surface area
S_{MP}	-	Micropore surface area
t	-	Statistical thickness
V	-	Volume

V_{ads}	-	Volume of gas adsorbed
V_m	-	Volume of gas adsorbed when monolayer coverage is formed
V_{MP}	-	Micropore volume
W	-	Pore width or diameter
x	-	Weight of adsorbed adsorbate
ε	-	Molar adsorptivity
λ	-	Wavelength
λ_{max}	-	Wavelength at which the absorbance is maximum

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Surface active agent (surfactant) is a group of chemicals that are designed to have cleaning or solubilization properties. They are extensively used in household products, detergents, industrial processes, pesticides formulations and pharmaceutical industry. The global production and consumption of surfactants reveal the importance of surfactants in human lives. In 1998, the global consumption of the most popular surfactant, linear alkylbenzene sulphonates (LAS), was estimated to be 2.8 million tons [1]. In 1997, the worldwide production of alkylphenol ethoxylates (APE), a non-ionic surfactant, was 500,000 tons [2].

After use, residual surfactants are discharged to sewage treatment plants or directly to surface waters, and then dispersed into different environmental compartments. Due to their widespread use and high consumption, surfactants have been detected at various concentrations in surface waters, sediments and sludge-treated soils [3]. Types of surfactants have been detected in sewage effluents, for example LAS was detected at concentrations up to 1090 $\mu\text{g/L}$ [4] and APE at 332 $\mu\text{g/L}$ [5].

Some surfactants are recalcitrant while some can self-degrade in the environment. The presence of surfactants and their degradation products may impose serious impacts to the environment, aquatic life and human health. The negative

influences caused by surfactants include toxicity towards microorganisms, invertebrates, crustaceans and vertebrates [6], estrogenic effects, ability to bioaccumulate in aquatic organisms and later be transferred to human bodies [3], and ability to increase the solubility of toxic organic compounds such as trichlorobenzene in water [7].

As the use of surfactants in modern world is unavoidable, scientists are attempting to minimize the effects of surfactants to the environment. Several methods are developed to remove surfactants from wastewater. These methods include adsorption technology, microfiltration [8] and biological degradation. However the latter method may cause other pollution problems as the biodegradation processes can produce products which are even more toxic than the parent surfactants. For example the degradation of APE produces alkylphenols. Alkylphenols exhibit strong estrogenic effects and are more recalcitrant than APE [3]. Therefore adsorption technology is gaining ground in recent years in the study of surfactants removal.

Activated carbon is one of the most popular adsorbents used for surfactant removal. As the consumption of surfactants is expected to increase in the future, there is a need to discover new material and develop optimized preparation methods for the production of activated carbon. The purpose of doing so is to reduce the production cost of activated carbon and at the same time increase the effectiveness of activated carbon. Many studies on activated carbon are reported [9-15] but only a few of them employed bamboo as the source of carbon [16-18]. Owing to the high carbon content of bamboo, we believe that bamboo is a potential material for the production of effective activated carbon.

1.2 Objectives of Study

The objectives of this study are:

- i. To prepare activated carbon by using bamboo cane as a source of carbon.
- ii. To characterize the prepared activated carbon by physical and chemical characterization.
- iii. To investigate the adsorption capacity of the prepared activated carbon in the adsorption of surfactants.
- iv. To compare the adsorption capacity of cationic and non-ionic surfactant onto the prepared activated carbon.

1.3 Scope of Study

In the preparation of activated carbon, many factors or parameters may affect the yield and quality of the product. These factors include type of chemical activating agent [9, 19], amount of activating agent [9, 11-13], activation temperature [10-13, 15], activation time [11-12, 14-15] as well as washing method [9].

In this study, we are interested in only the effect of activating gas. Therefore all other parameters were maintained constant in every preparation process. The chemical activating agent was zinc chloride and hydrochloric acid, with the bamboo-zinc chloride ratio of 1:1. The activation temperature was fixed at 700 °C. However for activating gas, three gases namely nitrogen, carbon dioxide and water steam were used respectively.

For adsorption capacity study, two adsorbates were chosen, namely cetyl trimethyl ammonium bromide (CTAB) and Triton-X 100 (TX100). CTAB is a cationic surfactant while TX100 is a non-ionic surfactant. The adsorption capacity of bamboo activated carbon in both surfactants were determined and compared. This study is to find out whether bamboo activated carbon is a suitable adsorbent for either cationic or non-ionic surfactants.

1.4 Activated Carbon

1.4.1 Characteristics of Activated Carbon

Activated carbon is an amorphous form of elemental carbon. Unlike diamond, graphite or fullerenes, activated carbon has no regular atomic structure. Activated carbon shows a very disordered micro-crystalline structure. In this disordered structure, graphitic microcrystals are randomly oriented. The graphitic layers therefore exist in a disordered manner. This then results in the occurrence of gaps between graphitic microcrystals. Such gaps are known as pores [20].

Due to the existence of such pores, activated carbon is well-known with its high surface area ($500 \text{ m}^2\text{g}^{-1}$ to $2000 \text{ m}^2\text{g}^{-1}$) and complex porosity. According to International Union of Pure and Applied Chemistry (IUPAC), the pores in activated carbon are classified to four types, as shown in Table 1.1. The classification is based on the width of the pores (W). Generally the pores exist in activated carbon are micropores and narrow-sized mesopores.

Table 1.1: Classification of pore width according to IUPAC [21].

Pore type	Pore width, W
Ultramicropore	$W < 0.7 \text{ nm}$
Micropore	$W < 2 \text{ nm}$
Mesopore	$2 \text{ nm} < W < 50 \text{ nm}$
Macropore	$W > 50 \text{ nm}$

In the production of activated carbon, the parameters that are concerned most are the surface area, the pore size distribution as well as the adsorption capacity. Surface area refers to the area occupied by adsorbates when a monolayer (single layer) of adsorbates is adsorbed on the activated carbon surface. An effective activated carbon usually exhibit surface area ranging from 500 to $2000 \text{ m}^2\text{g}^{-1}$. However it is important to know that surface area is not an absolute value like a weight or a geometric volume. It is a variable function dependent on how it is measured and how adsorption data are interpreted. The value of surface area is a normalized value so as to create a basis for both comparison and number sense [22].

Thus other characteristics are important too to tell the real characteristic of activated carbon.

In pore size distribution analysis, the total pore volume of micropores, mesopores and macropores in activated carbon are estimated. By such, we are able to find out which type of pores that is dominant in the activated carbon structure. We can prepare or select suitable activated carbon with appropriate pore size distribution depending on the type of targeted adsorbates.

The adsorption capacity reflects the effectiveness of an activated carbon in adsorbing and removing a certain adsorbate. Adsorption capacity can have units of mg g^{-1} or mmol g^{-1} or $\text{cm}^3 \text{g}^{-1}$ of adsorbate. The adsorption capacity of activated carbon may vary from one adsorbate to another. For example, an activated carbon can have high adsorption capacity in anionic surfactant solution but low adsorption capacity in cationic surfactant solution. Therefore any statement of adsorption capacity must be accompanied by the name of adsorbate used in the analysis.

1.4.2 Preparation of Activated Carbon

The preparation process of activation carbon consists of two stages, namely carbonization and activation. Carbonization process involves pyrolysis of the precursor material at temperature as high as 100 – 400 °C in the absence of air. In carbonization process, almost all elements except carbon are burnt away as volatiles. The carbonization process results in carbon char of which the carbon content is enriched and unwanted elements are eliminated. The carbonization process is also responsible for the development of porosity and the ordering of carbon structure [10].

Activation process is a process in which the porosity especially the surface area of carbon is increased. Activation can be carried out by physical or chemical means. In physical activation process, the carbon char from carbonization process is heated at temperature 400 to 900 °C in inert gas flow [10-13, 15]. Many inert gases can be used as activating gas, and among those dry nitrogen is the most common [9-

12, 14, 23]. Other activating gases include carbon dioxide [26], water steam [10, 15] and vacuum condition [11, 23]. Compared with chemical activation, physical activation usually requires higher activation temperature. But the problem of contamination is not encountered as no chemical agent is involved.

In chemical activation process, chemical activating agent is incorporated into the carbonaceous material. The incorporation of chemical activating agent is carried out by impregnation, physical mixing or acid-wash with impregnation [9, 24]. Some of the common activating agents are zinc chloride [9, 11, 19], phosphoric acid [13], potassium hydroxide [9, 12] and sulfuric acid [19]. The function of these activating agents is as a dehydrating agent to accelerate the pyrolytic decomposition in order to produce higher porosity. With the presence of chemical activating agent, carbon can be activated at lower temperature. However chemical activation is always accompanied by contamination problem. The residue of activating agent is retained in the activated carbon structure and subsequently lowers the adsorption capacity of activated carbon.

In chemical activation process, the activation and carbonization processes are usually carried out at the same time [24]. The raw material, impregnated with chemical activating agent, will undergo activation and carbonization process simultaneously at high temperature in inert gas flow. In our work, activated carbon was prepared with this simultaneous activation-carbonization method.

In the preparation of activated carbon, a few factors may influence the yield and quality of the product. These factors include type of chemical activating agent [9, 19], amount of activating agent [9, 11-13], activation temperature [10-13, 15], activation time [11-12, 14-15] as well as washing method [9]. Therefore the optimization of the preparation condition may be a time-consuming process that requires much effort and patience.

1.4.3 Source of Carbon

Despite the availability of activated carbon from commercial manufacturers, scientists have shown great interests to produce activated carbon using low cost materials, among which industrial and agricultural waste are most widely studied. Table 1.2 summarizes some of the materials that were used to produce activated carbon. The BET surface area and pore volume of the activated carbon are tabulated in the same table as well.

Table 1.2: Examples of materials used to produce activated carbon.

Material	BET surface area (m²g⁻¹)	Micropore volume (cm³g⁻¹)	Reference
Pistachio-nut shell	1635	Not reported	Lua, A.C. <i>et al.</i> [11]
Cassava peel	1605	0.513	Sudaryanto, Y. <i>et al.</i> [12]
Cane	1333	0.67	Vernersson, T. [13]
Teak sawdust	1150	0.43	Ismadji, S. <i>et al.</i> [15]
Corn cob	2700	Not reported	Cao, Q. <i>et al.</i> [25]
Tunçbilek lignite	1000	0.44	Önal, Y. <i>et al.</i> [26]
Pecan shell	917	Not reported	Bansode, R.R. <i>et al.</i> [27]
Coconut shell	1828	Not reported	Iwasaki, S. <i>et al.</i> [28]
Oil-palm shell	1022	0.49	Mohd Salih, S. [29]

From Table 1.2, it is observed that biomass resource has become a main source of carbon for the production of activated carbon. This is due to the low cost of those materials as they are usually unwanted waste or by-products in commercial activities. By re-using or recycling these low cost materials to produce activated carbon, we are actually providing another environmental-friendly alternative to dispose those waste and by-products.

Another reason for using these materials is that they contain high percentage of carbon. High carbon content in precursor is important in maximizing the yield of production. In many reported studies, the materials used for activated carbon preparation contain at least 40 % carbon. Table 1.3 summarizes some of the carbonaceous materials that were reported. The carbon content of these materials

ranges from 48 % to 62 %. As bamboo contains more than 50 % of carbon, we believe that bamboo is a suitable and feasible material for the preparation of activated carbon.

Table 1.3: Carbon content of some carbonaceous materials.

Material	Carbon content (%)	Reference
Palm shell	50.01	Wan Daud, W.M.A. <i>et al.</i> [10]
Cassava peel	59.31	Sudaryanto, Y. <i>et al.</i> [12]
<i>Arundo donax</i> cane	49.30	Vernersson, T. <i>et al.</i> [13]
Coconut shell	48.63	Wan Daud, W.M.A. <i>et al.</i> [14]
Teak sawdust	56.31	Ismadji, S. <i>et al.</i> [15]
Bamboo tar	62.00	Qiao, W.M. <i>et al.</i> [16]

1.4.4 Application and Importance of Activated Carbon

Due to its high surface area and high adsorption capacity, activated carbon is widely used as adsorbent material in many sectors. Among those, the use of activated carbon in wastewater treatment process is gaining ground in the past two decades. Industrial wastewaters, especially those from the textile industries, agricultural activities and raw material processing industries, contain high volume of dissolved pollutants. Some examples of pollutants are dyes, pesticides, trace toxic metals and non-biodegradable surfactants. As the conventional secondary wastewater treatment is not sufficient enough to remove those hazardous substances, new treatment method based on adsorption was developed. Adsorbents such as activated carbon, zeolite, and synthetic polymer are used to adsorb and separate pollutants from wastewater. However due to the high cost of zeolite and synthetic polymer, activated carbon is becoming the most widely used adsorbent in wastewater treatment application [30].

Activated carbon is also widely employed in many purification processes, such as removal of chloroform [31] and nitrate-nitrogen [18] in drinking water.

Besides, activated carbon is employed to remove undesired odour in consumer products. It is reported too that activated carbon is employed to remove the brown-coloured compounds that are formed during the manufacturing and storage of fruit juices and purees. The formation of such brown-coloured compounds is a result of the polymerization reaction of reactive intermediates in the fruits [32].

In catalysis study, activated carbon is employed as an activating catalyst or catalyst support. Due to its high adsorption capacity, it accelerates the formation of activated intermediates on its surface. As a result, the reaction is activated and the rate of reaction is increased. The use of activated carbon as catalyst is mostly in organic or organometallic reactions, for example in the production of vinyl fluoride by gaseous reaction of acetylene and hydrogen fluoride [33].

1.5 Bamboo

Bamboo is a perennial woody plant belonging to the subfamily *Bambusoideae* of the family *Graminae*. A bamboo plant often has a tree-like habit and can be characterized as having woody and hollow culms, complex rhizome and branch systems, petiolate leaf blades and prominent sheathing organs. Out of more than 1000 bamboo species found all over the world, about 200 species are found in South East Asia [34].

In Malaysia, bamboo is found in abundance although widely scattered in about five percent of the total forest reserved area [35]. Generally it grows from sea level up to 1000 m above sea level. Peninsular Malaysia has at least 63 bamboo species of which 25 are indigenous while the rest are known only in cultivation. All bamboo species are grouped under 11 genera namely *Bambusa*, *Chusquea*, *Dendrocalamus*, *Dinochloa*, *Gigantochloa*, *Melocanna*, *Phyllostachys*, *Racemobambos*, *Schizostachyum*, *Thyrsostachys* and *Yushania*. Among those, only about 13 are commercially utilized while the rest are left idle on their habitat with lack of knowledge on their properties and potential usage [36]. Thus it may be a wise

attempt to explore the potential of bamboo in industrial and technological applications.

Bamboo has long been exploited by mankind for various purposes in many developed and undeveloped countries. In the past two centuries, bamboo has been used as building material and it is now mainly used as structure scaffolding [17]. Bamboo is also the main material in the making of many products, for example baskets, musical instruments, paper, handicraft, and furniture [34]. In Malaysia, it is considered as a multipurpose plant with wide applications such as construction, paper, furniture, barbecue-sticks and chopsticks [36]. Besides, bamboo shoot is a common vegetable especially in rural community in Malaysia.

Owing to its abundance in Malaysia forests, bamboo is considered a readily available and comparatively cheap raw material in various industries. At the same time, bamboo wastes are being produced by the industries. In regions where bamboo is widely utilized as construction scaffolding material, for example Hong Kong, over 50,000 tons of bamboo scaffolding is dumped as construction waste annually [17]. Therefore, there may be a need to develop a method to reuse or recycle these bamboo wastes. In our work, we have investigated the potential of bamboo as the precursor for the preparation of activated carbon.

The main chemical components of bamboo are cellulose, hemicellulose and lignin. The minor constituents consist of slime, tannins waxes and inorganic salts. Table 1.4 summarizes the chemical properties of five bamboo species. It is obvious that all of them contain more than 60% of cellulose. And as known, carbon is the major element in cellulose. Therefore the high cellulose content in bamboo is an important indicator of its high content of carbon. The claim of high carbon content is supported by the elemental analysis done by Choy, K.K.H. *et al.* [17]. The result of the elemental analysis shows that bamboo cane contains approximately 47.6 % of carbon (Table 1.5). As such, bamboo has shown its potential as the precursor or raw material for activated carbon. Due to such high carbon content in bamboo, we can expect high yield of production from the preparation of activated carbon with bamboo.

Table 1.4: Chemical composition of some Malaysian bamboos [35].

Species	Ash (%)	Lignin (%)	Cellulose (%)	
			Holo-	Alpha-
<i>Gigantochloa Scortechinii</i>	1.1 – 1.4	24.9 – 27.9	66.8 – 68.0	40.5 – 41.4
<i>Gigantochloa levis</i>	1.4 – 1.9	23.3 – 26.6	63.5 – 67.2	36.6 – 42.5
<i>Bambusa vulgaris</i>	1.8 – 2.1	22.7 – 23.9	67.8 – 69.6	37.9 – 43.2
<i>Bambusa blumeana</i>	-	20.5 – 22.7	65.7 – 72.6	40.3 – 45.1
<i>Schizotachyum zolligneri</i>	-	20.1 – 22.7	68.8 – 74.3	48.7 – 52.6

Table 1.5: Elemental composition of bamboo cane [17].

Element	Content (%)
Carbon	47.6
Oxygen	43.9
Hydrogen	6.5
Nitrogen	0.3
Sulfur	0.3
Ash	1.4

1.6 Surface Active Agents

Surface active agents, more commonly known as surfactants, are a large class of molecules which have technological importance in various industries. The term “surface active” originates from the surfactant ability to adsorb at surfaces or interfaces. The surface activity of surfactants is due to their amphiphilic molecule structure. A surfactant molecule must consist of two parts. The first part is polar or electrically charged and soluble in water. This behaviour is called hydrophilic. The

other part is nonpolar and insoluble in water. This behavior is termed hydrophobic. The hydrophilic part is often referred to as the head group and the hydrophobic part as the tail group [37]. The general structure of a surfactant molecule is illustrated in Figure 1.1.

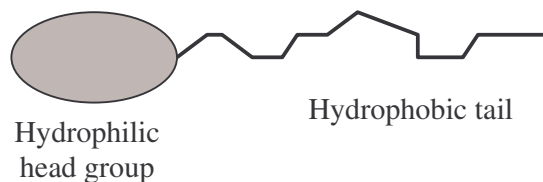


Figure 1.1: Schematic illustration of a surfactant molecule [37].

In dilute solution, surfactants exist in its unimer form. As the concentration of surfactant increases, the surfactants tend to form associate and form aggregates, the so called micelles. A micelle is a group of surfactants arranged in a cluster. In a micelle, the surfactant hydrophobic group is directed towards the interior of the cluster while the hydrophilic head is directed toward the solvent. The micelle is therefore a polar aggregate of high water solubility and without much surface activity. The concentration at which the surfactants begin to form micelles is termed the critical micelle concentration (*CMC*). Different surfactants have their own *CMC*.

Surfactants can be categorized by the type of their hydrophilic head group. Surfactant with positively charged head group is called cationic surfactant. The vast majority of cationic surfactants are based on the nitrogen atom carrying the cationic charge [37]. Examples of cationic surfactants are fatty amine salt in Figure 1.2 (a) and quaternary ammonium compound in Figure 1.2 (b).

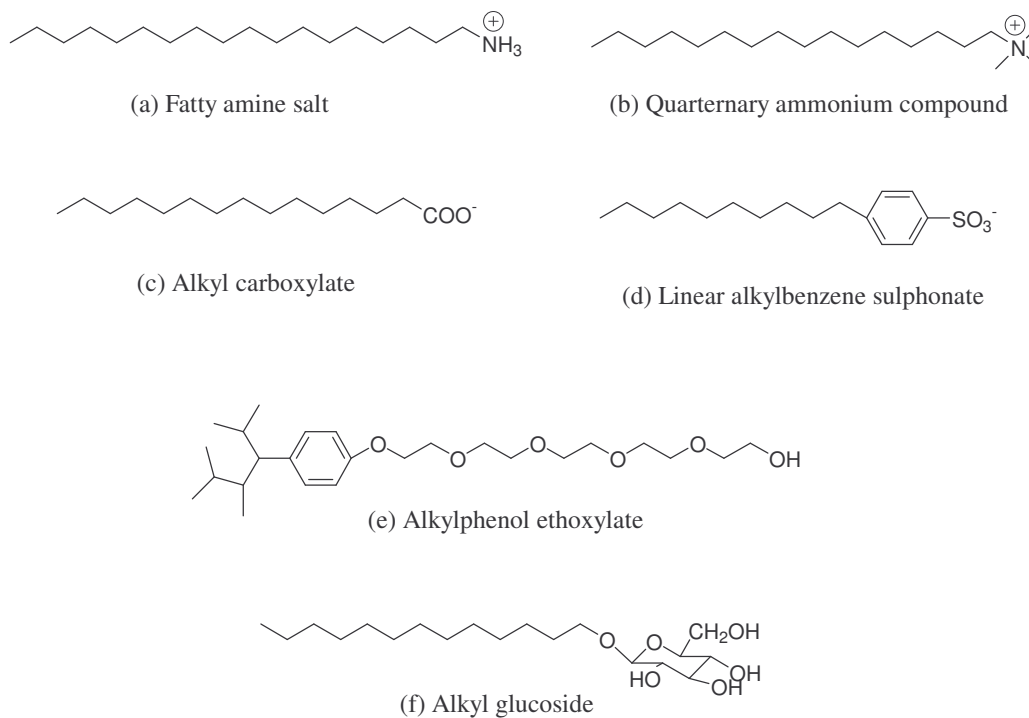


Figure 1.2: Examples of (a) & (b) cationic, (c) & (d) anionic and (e) & (f) non-ionic surfactants [37, 38].

When the hydrophilic head of a surfactant is negatively charged, it is called an anionic surfactant. Some examples of anionic surfactants are alkyl carboxylate in Figure 1.2 (c), alkyl sulphonate, alkyl phosphate and linear alkylbenzene sulphonates in Figure 1.2 (d) [21, 37]. Conventional soaps in early days utilized alkyl carboxylate as surfactants. However when detergency technology was developed, alkyl carboxylate was gradually replaced by linear alkylbenzene sulphonates (LAS). And in recent practice, LAS is being replaced by other cationic or non-ionic surfactants which are more environmental friendly.

Non-ionic surfactants have either polyether or polyhydroxyl as the polar group. In the vast majority of non-ionic surfactants, the polar group is polyether consisting of oxyethylene units, made by polymerization of ethylene oxide. Some examples of non-ionic surfactant are alkylphenol ethoxylate in Figure 1.2 (e), fatty alcohol ethoxylate, alkyl glucoside in Figure 1.2 (f) and fatty amine ethoxylate [37].

1.6.1 Cetyl Trimethyl Ammonium Bromide

Cetyl trimethyl ammonium bromide (CTAB), also known as hexadecyl trimethyl ammonium bromide, is a cationic surfactant. The molecular formula of CTAB is $\text{CH}_3(\text{CH}_2)_{15}\text{N}(\text{CH}_3)_3\text{Br}$. Its structure is illustrated in Figure 1.3. Its molecular weight is $364.46 \text{ g mol}^{-1}$. The *CMC* value of CTAB is $9.2 \times 10^{-3} \text{ mol L}^{-1}$ which is equivalent to 335 ppm [37]. CTAB is used as germicides, disinfectants and sanitizers. It is used together with alkaline inorganic salts and non-ionic surfactants for dishwashing in restaurants and pubs. CTAB is also used as textile softener for home laundry washing machine. Besides, it is used as emulsifier in bitumen emulsion for road building [38].

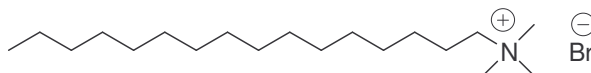


Figure 1.3: Molecular structure of cetyl trimethyl ammonium bromide (CTAB).

1.6.2 Triton-X 100

Triton-X 100 (TX100) is a non-ionic surfactant. It belongs to the class of alkylphenol ethoxylates surfactant. This class of surfactant is also known as alkylaryl polyether alcohols. The Triton series of surfactants are prepared by the reaction *tert*-octylphenol with ethylene oxide [39]. The molecular formula of TX100 is $\text{C}_5\text{H}_{11}(\text{CH}_3)_2\text{C}(\text{C}_6\text{H}_4)(\text{OCH}_2\text{CH}_2)_n\text{OH}$ with $n = 9 - 10$. The molecular structure of TX100 is illustrated in Figure 1.4. Its molecular weight is $476.56 \text{ g mol}^{-1}$. The *CMC* value of TX100 is $3.4 \times 10^{-4} \text{ mol L}^{-1}$ which is equivalent to 162 ppm [37]. In textile industry, TX100 is used as detergent, wetting agents and emulsifiers for processing wool, cotton and synthetics. It also makes excellent heavy-duty solvent-type cleaners for floors and general cleaning. However it is not used widely in the Western world because of biodegradation properties [38].

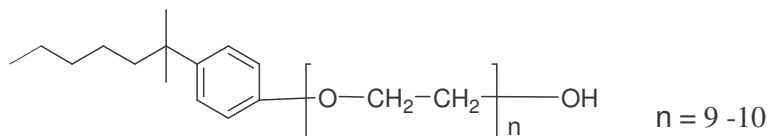


Figure 1.4: Molecular structure of Triton-X 100 (TX100).

1.7 Adsorption

Adsorption is a phenomenon that takes place at the surface or interface between two immiscible phases. Adsorption is defined as the process where the concentration of a certain component increases at the surface or interface, relative to its concentration in the bulk of the phase [21, 40]. The phase where the substance is accumulated is called the adsorbent and the adsorbed substance is termed adsorbate. In this study, the adsorption phenomena at solid-gas interface and solid-liquid interface were studied. The following discussions include the basic theories and principles of adsorption phenomenon.

1.7.1 Adsorption at Solid-Gas Interface

A solid-gas interface refers to the region where a solid phase is in contact with a gas phase. At the solid-gas interface, adsorption can happen in two ways namely physical adsorption (physisorption) and chemical adsorption (chemisorption). In physisorption, there is a van der Waals interaction between the adsorbent and adsorbate. Van der Waals interactions have a long range but are weak, and the energy released when a particle is physisorbed is of the same order of magnitude as the enthalpy of condensation. Therefore physisorption process usually takes place at temperature near or below the condensation point of the gas. The enthalpy of physisorption is typically in the region of 20 kJ mol^{-1} [41]. Physisorption processes are usually exothermic.

In chemisorption, the adsorbates stick to the surface of adsorbent by forming a chemical bond. The formation of the chemical bond, usually covalent bond, involves substantial rearrangement of electron density. Therefore the enthalpy of chemisorption is typically in the region of 200 kJ mol^{-1} . Chemisorption processes must be exothermic except in special cases [41]. The main differences between physisorption and chemisorption is summarized in Table 1.6.

Table 1.6: Differences between physisorption and chemisorption [21].

	Physisorption	Chemisorption
Temperature range	Near the condensation point of gas	Virtually unlimited
Adsorption enthalpy	Typically $5\text{-}40 \text{ kJ mol}^{-1}$	Typically $40\text{-}800 \text{ kJ mol}^{-1}$
Nature of adsorption	Non-dissociative Reversible	Often dissociative May be irreversible
Saturation uptake	Multilayer uptake possible	Limited to monolayer coverage
Kinetic of adsorption	Fast, non-activated process	Very variable, often an activated process

1.7.1.1 Adsorption Isotherms

When a solid is exposed in closed space to a gas or vapor at some definite pressure, the solid begins to adsorb the gas by increase of weight of the solid and a decrease in the pressure of the gas. The amount of gas uptake is proportional to the gas pressure, the temperature, the specific surface area, the solid porosity and the system nature. For a given gas adsorbed on a particular surface maintained at a fixed temperature, the temperature, the specific surface area, the solid porosity and the system nature are constant. The amount of gas uptake becomes a function of the gas pressure. The plot of adsorbed gas amount (weight, volume or mol) versus the gas pressure is termed the adsorption isotherm. The shape of adsorption isotherm reflects the adsorbent surface structure. Therefore the adsorption isotherm is very frequently

used to characterize surface structure of an adsorbent. In the laboratory practice, nitrogen gas is the most commonly used adsorbate [21].

In nitrogen adsorption analysis, dry nitrogen gas flows through sample. The volume of nitrogen adsorbed on the sample will be monitored versus the gas pressure. The result will be presented as a plot of volume of gas adsorbed (V_{ads}) versus relative pressure (P/P_0). Such plot is known as adsorption isotherm. From the shape of the isotherm, we will be able to determine the type, shape and size of the pores in the sample.

According to the BDDT classification proposed by Brunauer, Deming, Deming and Teller, there are five types of isotherm shapes namely Type I, II, III, IV and V. The sixth isotherm, Type VI shape was discovered later. All six typical types of isotherm are illustrated in Figure 1.5.

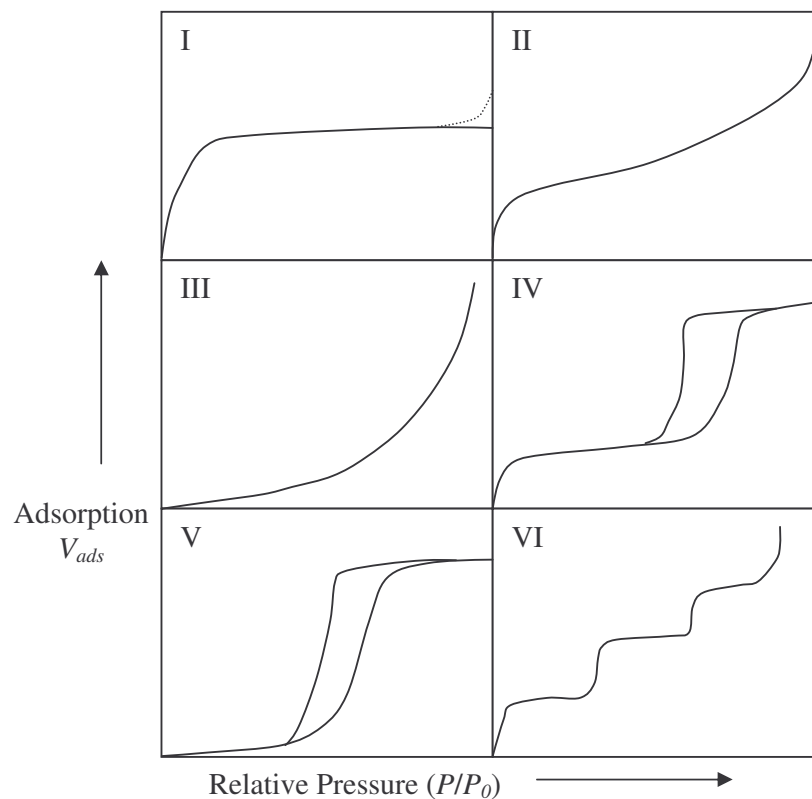


Figure 1.5: Types of the BDDT physisorption isotherm [21].

Type I isotherm indicates the formation of monolayer adsorption and that the adsorbent is a microporous material. Type II isotherm indicates the formation of multilayer adsorption and that the adsorbent is a non-porous or macroporous solid. In Type III isotherm, the parabolic curve is due to the weak adsorbent-adsorbate interaction. This implies that the adsorbent is a non-porous or macroporous material. In Type IV and V isotherms, hysteresis loops are observed. The hysteresis loop indicates the presence of mesopores in the adsorbent. Type IV isotherm signifies a mesoporous material while Type V signifies a mixture of mesopores and macropores. Type VI isotherm indicates that the adsorbent surface contains few groups of energetically uniform sites. The stepped isotherm is the result of stepwise multilayer adsorption of gas molecules [21]. However this behaviour is rather rare in the study of activated carbon.

1.7.1.2 Brunauer, Emmett and Teller (BET) Surface Area

The data of nitrogen adsorption is very useful in the determination of the Brunauer, Emmett and Teller (BET) Surface Area (S_{BET}) of the sample. The graph of adsorption data is plotted using BET equation (Eq. 1.1).

$$\frac{P}{V(P_0-P)} = \frac{1}{V_m C} + \frac{(C-1)}{V_m C} \frac{P}{P_0} \quad (1.1)$$

where

P = equilibrium pressure of nitrogen gas

P_0 = saturation pressure of nitrogen gas

V = volume of nitrogen adsorbed at pressure P

V_m = volume adsorbed when the entire surface is covered with monolayer of nitrogen molecules

C = BET constant

A plot of $P/[V(P_0 - P)]$ against P/P_0 will result in a straight line. By using the value of the slope (s) and intercept (i), we can calculate the value of V_m , using Eq. 1.2:

$$V_m = \frac{1}{s + i} \quad (1.2)$$

The value of V_m can then be used to calculate the value of BET surface area (S_{BET}), by using Eq. 1.3

$$S_{BET} = \frac{V_m}{22414} \times A_m \times N_0 \quad (1.3)$$

where

A_m = average area of one adsorbate molecule in the complete monolayer

N_0 = Avogadro number

When nitrogen is used as adsorbate, the value of A_m is $16.2 \times 10^{-20} \text{ m}^2$. By substituting the value of A_m and N_0 into Eq 1.3, we can get a simplified equation, Eq. 1.4, which can be used to determine the BET surface area of the sample.

$$S_{BET} = 4.35 \times V_m \quad (1.4)$$

1.7.1.3 The t -Plot

To determine the micropore volume and micropore surface area, the method of t -plot is frequently used. In the t -plot analysis method, the quantity of nitrogen adsorbed is plotted against the statistical thickness of the adsorbed film, t . Harkins-Jura equation (Eq. 1.5) is used to produce the t -plot.

$$t (\text{\AA}) = \left[\frac{13.99}{(0.0340 - \log P/P_0)} \right]^{1/2} \quad (1.5)$$

The intercept, i , of the t -plot graph can be converted to liquid volume to give the micropore volume, V_{MP} (Eq. 1.6). The constant 0.001547 tells the conversion factor of nitrogen gas volume to liquid.

$$V_{MP} = i \times 0.001547 \text{ (cm}^3 \text{ g}^{-1}\text{)} \quad (1.6)$$

The gradient, s , of the straight line within the range of 3.5 – 6.0 Å gives the external surface area, S_{ext} (Eq. 1.7).

$$S_{ext} = s \times 15.47 \text{ (m}^2 \text{ g}^{-1}\text{)} \quad (1.7)$$

The micropore surface area, S_{MP} is the difference between S_{BET} and S_{ext} (Eq. 1.8).

$$S_{MP} = S_{BET} - S_{ext} \quad (1.8)$$

1.7.2 Adsorption at Solid-Liquid Interface

The adsorption phenomenon at solid-liquid interface is rather complex compared with that at solid-gas interface. Two isotherm models, namely Freundlich isotherm and Langmuir isotherm, are widely employed to study the adsorption property at solid-liquid interface [21]. In this study, the solid-liquid interaction between bamboo activated carbon and surfactant solutions is investigated.

1.7.2.1 Freundlich Isotherm

Freundlich model is an empirical equation based on the distribution of solute between the solid phase and aqueous phase at equilibrium. The equilibrium relationship for adsorbent can be stated using Freundlich equation when two conditions are fulfilled. The first condition is that when molecules are adsorbed onto

the surface, no combination or dissociation of molecule occurs. The second condition is that the adsorption must occur by means of physical adsorption but not by chemical adsorption [21].

The basic Freundlich equation is:

$$x / m = q_e = K_F C_e^{1/n} \quad (1.9)$$

where

- x = weight of solute adsorbed
- m = weight of adsorbent
- C_e = equilibrium concentration of surfactant
- K_F and n = empirical constant

Eq. 1.9 can be rearranged into a linear form:

$$\log\left(\frac{x}{m}\right) = \log q_e = \log K_F + \frac{1}{n} \log C_e \quad (1.10)$$

A plot of $\log (x/m)$ versus $\log C_e$ will give a straight line of which the slope equals to $1/n$ and the intercept equals to $\log K_F$. The value of n and K_F will provide information about the capacity of the activated carbon or the amount required to remove a unit mass of adsorbate.

1.7.2.2 Langmuir Isotherm

The Langmuir model is the simplest model that can be used to describe monolayer adsorption. It assumes a uniform surface and a single layer of adsorbed molecules at constant temperature. If a system deviates from this model, the deviation may be due to the irregularity of the surface or maybe there are interactions between the adsorbed molecules. The adsorbed molecules on the surface can cause other molecules to be adsorbed more difficult or easier on the nearby sites [21].

The Langmuir equation is as follows:

$$\frac{x}{m} = q_e = \frac{bq_{max}C_e}{1+bC_e} \quad (1.11)$$

where

b = Langmuir constant related to the affinity of the binding site

q_{max} = maximum adsorption at monolayer coverage

Eq. 1.11 can be rearranged to a linear form:

$$\frac{m}{x} = \frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{bq_{max}C_e} \quad (1.12)$$

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{bq_{max}} \quad (1.13)$$

When C_e/q_e is plotted against C_e , a straight line graph is obtained with the slope of graph gives the value of $1/q_{max}$ and the intercept gives the value of $1/(bq_{max})$. By such, we can determine the value of b and q_{max} from the graph plotted. The value of b will be used to calculate the separation factor R_L later.

1.7.2.3 Separation Factor of R_L

Separation factor or equilibrium parameter, R_L , is a dimensionless constant that expresses the characteristics of Langmuir isotherm [21]. It is defined as:

$$R_L = \frac{1}{1+bC_o} \quad (1.14)$$

where

C_o = initial concentration of surfactant

The value of separation factor enables us to determine whether the adsorption system is “favourable” or “unfavourable”. Table 1.7 summarizes the effect of separation factor on the Langmuir isotherm shape.

Table 1.7: Effect of separation factor (R_L) on isotherm shape [21].

R_L value	Type of isotherm
$R_L > 1$	Unfavourable
$R_L = 1$	Linear
$0 < R_L < 1$	Favourable
$R_L = 0$	Irreversible

1.8 Methods of Characterization

In this study, Fourier Transform Infrared Spectroscopy (FTIR), Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray Spectrometry (EDX), Thermogravimetry Analysis (TGA) and nitrogen adsorption analysis were employed to characterize the prepared activated carbon. Ultraviolet – Visible (UV-Vis) Spectroscopy was used to determine the concentration of the surfactant after treated with activated carbon.

1.8.1 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a spectroscopy method that enables us to study the types of bonding in a molecule as well as the interaction of bonding in a solid matrix or a mixture. The infrared light refers to the electromagnetic wave within the frequency region of 3×10^{12} to 3×10^{14} Hz or the wavenumber of 10000 cm^{-1} to 100 cm^{-1} .

The basic principles of FTIR analysis is based on the fact that every molecule vibrates at a certain frequency. When the frequency of an infrared light is in

resonance with the frequency of the molecular vibration, the molecular vibration can absorb the infrared light. The frequency of the absorbed infrared light is characteristic of the bonds existing in the molecule. For example, the absorbance at region $1300\text{-}1000\text{ cm}^{-1}$ indicates the vibration of a C-O bond, while the absorbance at $3500\text{-}3200\text{ cm}^{-1}$ is characteristic of an O-H bond. Thus by obtaining infrared spectrum of the activated carbon, we can identify the chemical bonds that exist in the activated carbon. It can be determined whether the raw material is converted completely to pure carbon. We can determine too the functional groups present in the activated carbon.

1.8.2 Field Emission Scanning Electron Microscopy (FESEM)

Field Emission Scanning Electron Microscope (FESEM) is an instrument that can produce images of the sample surface up to $1000\times$ and even $10000\times$ magnification. In a scanning electron microscope, a beam of highly energetic electrons is emitted towards the surface of sample. The interaction between the electrons and the surface can produce signals which are in the form of electrons. The signals will be detected and be used to produce a point-by-point reconstruction of the surface image of the sample [42]. Thus SEM does not produce the real image of the sample surface. However the reconstructed image of the surface is good enough for scientists to study the surface morphology of their samples.

1.8.3 Energy Dispersive X-ray Spectroscopy (EDX)

Energy Dispersive X-ray Spectroscopy (EDX) is a spectroscopic technique that enables us to determine the main composition of a sample. In an EDX spectrometer, an X-ray beam is emitted to the surface of sample. The interaction of the X-ray with the surface electrons produces X-ray lines with different photon energies. The principle of EDX is the separation of X-ray lines based on their photon

energies [43]. Since the photon energies of X-ray lines produced are characteristics of the elements, we can determine the main composition of elements in the sample.

1.8.4 Thermogravimetry Analysis (TGA)

Thermogravimetry is an analytical technique in which the mass (weight) of sample is monitored versus time or temperature while the temperature of sample is programmed under certain atmosphere. The result of detection is presented graphically as a plot of weight % versus time or temperature. Such plot is known as a thermogram. From the thermogram, we are able to determine the steps of reaction when sample undergoes heating, the thermal stability, the temperature at which certain reaction takes place etc. In this study, TGA is important in determining the optimum activation temperature for the production of activated carbon, and also to ensure that the conversion of bamboo to activated carbon is complete.

1.8.5 Nitrogen Adsorption Analysis

In nitrogen adsorption analysis, dry gas nitrogen will be flowed through the sample. The volume of nitrogen adsorbed on the sample will be monitored versus the gas pressure. The result will be presented as a plot of volume of gas adsorbed (V_{ads}) at 77 K versus relative pressure (P/P_0). Such plot is known as an adsorption isotherm. From the shape of the isotherm, the type, shape and size of the pores in the sample can be determined. With the adsorption data obtained, the surface area, pore volume and pore size distribution of the sample can also be calculated. The details of the calculation are discussed in Section 1.7.1.

1.8.6 Ultraviolet-Visible Spectrometry (UV-Vis)

Ultraviolet-visible spectrometry is a very common analytical instrument used for analytical work. An ultraviolet light refers to the electromagnetic light of wavelength (λ) 100 nm to 380 nm, while visible light is within the wavelength of 380 nm to 800 nm.

The basic principle of UV-Vis is based on the excitation of electrons in a molecule. When an UV-Vis light is emitted and passes through a sample, electrons in the low-energy orbital tends to absorb energy of the light, and consequently are excited to high-energy orbital. By measuring the intensity of UV-Vis light before and after its passes through the sample, the light absorbance of the sample can be determined. Electrons in different molecules absorb at different wavelengths. Every molecule has its own unique wavelength at which the electrons absorption is at the maximum. This wavelength is commonly known as the maximum wavelength (λ_{max}). In all UV-Vis analysis, the absorbance of sample is measured at its own λ_{max} .

According to the Beer-Lambert Law (Eq 1.15), the light absorbance of a sample is linearly proportional to the molar adsorptivity of the analyte, the path length (which is usually the width of sample cell), and the concentration of the analyte solution.

$$A = \varepsilon b_B c \quad (1.15)$$

where

A = absorbance

ε = molar absorptivity of analyte

b_B = path length / sample cell length

c = concentration of analyte

In UV-Vis analysis, the value of molar absorptivity (ε) and path length (b_B) are always constant. Thus with the absorbance data of the analyte, we can determine the concentration of analyte in solution.

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