



**ACTIVATED CARBON FROM PALM
KERNEL SHELL AS AN ADSORBENT OF
PARAQUAT**

BY

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**A dissertation in fulfilment of the requirement for the
degree of Master in Pharmaceutical Science
(Pharmaceutical Chemistry)**

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ABSTRACT

Highly Efficient Activated Carbon (HEAC) as an adsorbent of toxins has been successfully produced from palm shell through chemical activation process using phosphoric acid as an activating agent. Palm Kernel shell used as the main raw material for activated carbon production, was purchased from a local oil palm mill in Pahang, Malaysia. Temperature range 550 °C – 650 °C was used during the activation process. The effect of temperature variation on the pore size and surface morphology of the activated carbon were studied. Well developed pore size and low number of functional groups observed on activated carbon at 600 °C were determined by Scanning Electron Microscope (SEM) and Fourier- Transform Infrared (FTIR) spectroscopy, respectively. The surface area and pore volume were determined by Brunauer, Emmett and Teller (BET) method using N₂ gas adsorption. The highest surface area (1287 m²g⁻¹) and pore volume (0.74 cm³ g⁻¹) was observed with sample HEAC-2. The adsorption efficiency of HEAC-2 was studied *in vitro* for paraquat as toxin using distilled water and NaCl (0.9%) solution. These study shows paraquat was adsorbed more on HEAC-2 in the presence of sodium chloride solution (4.68 mgL⁻¹) than in distilled water (3.62 mgL⁻¹). Furthermore, a comparison was done between HEAC-2 (4.68 mgL⁻¹) and commercially available activated carbon (4.18mg L⁻¹) which proved HEAC-2 is more effective than commercially available activated carbon.

خلاصة البحث

لقد تم انتاج فحم فعال عالي الكفاءة من قشور ثمرة النخيل ليستخدم لادمصاص المواد السامة وتم انتاج هذا الفحم من خلال تفاعل كيميائي تنشيطي باستخدام حمض الفوسفور كعامل منشط. قمنا بشراء قشور ثمار النخيل والتي استخدمت في انتاج الفحم الفعال من مزودين محليين في ولاية باهانغ في ماليزيا. تراوحت درجة الحرارة المستخدمة في هذه الدراسة بين ٥٥٠ و ٦٦٠ درجة مئوية. درسنا تأثير تغييرات درجة الحرارة على ابعاد المسام وشكل سطح الفحم الفعال. لوحظت ابعاد مسام محسنة وعدد اقل من المجموعات الوظيفية على الفحم الفعال في درجة حرارة ٦٠٠ سلسيوس باستخدام المجهر الالكتروني الماسح ومطيافية الاشعة تحت الحمراء التحويلية الفورية بشكل متتالي. لقد تم تحديد سطح الفحم وحجم المسام بطريقة BRUNAUER, EMMETT AND TELLER (BET) باستخدام ادمصاص غاز النيتروجين. وجدت مساحة السطح الاعلى والمقدرة ب ١٢٨٧ م^٢ /غرام وحجم مسام ٠,٧٤ م^٢ /غرام في العينة HEAC-2. لقد درسنا كفاءة ادمصاص العينة HEAC-2 في الزجاج على الباراكوات كمادة سمية باستخدام محاليل الماء وملح كلور الصوديوم. لقد اظهرت هذه الدراسة ان ادمصاص الباراكوات كان أعلى بوجود محلول كلور الصوديوم (4.68 MGL-1) منه بوجود الماء (3.62 MGL-1). علاوة على ذلك اجرينا مقارنة بين الفحم الفعال المنتج والفحم الفعال المتوفر في الاسواق (التجاري) وكان الفحم المنتج HEAC-2 أفضل من الفحم التجاري حيث كانت نسبة ادمصاص ٤,٦٨ ميلي غرام /ليتر للفحم المنتج مقارنة مع ٤,١٨ ميلي غرام / ليتر للفحم التجاري.

APPROVAL PAGE

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DECLARATION

I hereby declare that this dissertation is the result of my own investigations, except where otherwise stated. I also declare that it has not been previously or concurrently submitted as a whole for any other degrees at IIUM or other institutions.

Mohosina Bintay Shahjahan.

Signature Date

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Signature

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*To my beloved parents
and my sisters*

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TABLE OF CONTENTS

Abstract.....	ii
Abstract in Arabic.....	iii
Approval Page.....	iv
Declaration.....	v
Acknowledgements.....	viii
List of Tables.....	xi
List of Figures.....	xii
CHAPTER ONE: INTRODUCTION.....	1
1.0 Activated carbon.....	1
1.1 Activated carbon as an antidote.....	2
1.3 Raw material for activated carbon.....	5
1.4 Problem statements.....	7
1.5 Objectives of the research.....	7
CHAPTER TWO: LITERATURE REVIEW.....	8
2.0 Introduction.....	8
2.1 Activated carbon from carbonaceous material.....	8
2.1.1 Chemical activation.....	9
2.1.2 Physical Activation or Steam Activation.....	9
2.2 Classification of activated carbon.....	10
2.2.1 Powdered activated carbon (PAC).....	10
2.2.2 Granular activated carbon (GAC).....	11
2.2.3 Bead activated carbon (BAC).....	11
2.2.4 Extruded activated carbon (EAC).....	11
2.2.5 Impregnated carbon.....	12
2.2.6 Polymer coated carbon.....	12
2.2.7 Amorphous forms of carbon.....	12
2.3 Properties of activated carbon.....	13
2.3.1 Porosity.....	13
2.3.2 Surface area.....	14
2.3.3 Surface Functional Groups.....	15
2.3.4 Adsorption.....	17
2.3.5 Adsorption Isotherm.....	19
2.3.6 Langmuir's isotherm.....	21
2.3.7 BET Isotherm for Multilayer Adsorption.....	22
2.3.8 Adsorption on Activated Carbons.....	22
2.3.9 Iodine number.....	23
2.3.10 Apparent density.....	24
2.3.11 Hardness/abrasion number.....	24
2.3.12 Ash content.....	24
2.3.13 Carbon tetrachloride activity.....	25
2.3.14 Particle size distribution.....	25
2.4 Mechanism of action.....	25
2.5 Activated carbon as an antidote for toxins.....	26

2.6	Indication of activated carbon.....	30
2.6.1	Intestinal disorders.....	30
2.6.2	Poisons and drug overdoses	30
2.7	Side effects of activated carbon	31
2.8	Precaution of activated carbon use.....	31
2.9	Characteristics of Activated carbon for pharmaceutical products	32
2.10	Dosage forms of activated carbon for pharmaceutical purposes	33
2.10.1	Charcoal tablet.....	34
2.10.2	Charcoal suspensions.....	35
2.10.3	Charcoal capsules	37
2.11	Palm shell as the raw material to prepare the activated carbon	37
CHAPTER THREE: METHODOLOGY.....		39
3.0	Preparation of activated carbon.....	39
3.1	Materials	39
3.2	Instruments.....	39
3.3	Manufacture of activated carbon.....	39
3.3.1	Raw material	39
3.3.2	Pre-treatment of raw material.....	41
3.3.3	Chemical activation with H ₃ PO ₄	41
3.4	Characterization of activation carbon.....	43
3.4.1	Determination of surface morphology.....	44
3.4.2	Determination of surface area	45
3.4.3	Determination of surface functional groups.....	46
3.5	Activated carbon as an adsorbent of toxin in suspension form.....	47
3.5.1	Preparation of paraquat solution.....	48
3.5.2	Determination of equilibrium adsorption isotherm of paraquat onto activated carbon.....	50
CHAPTER FOUR: RESULTS AND DISCUSSION.....		52
4.1	Surface morphology of the activated carbon by SEM:.....	52
4.2	Bet Surface Area, Pore Diameter, Pore Volume and Adsorption Isotherm of Produced Activated Carbon	51
4.2.1	Surface area analysis:	55
4.2.2	Pore diameter and pore volume analysis:	56
4.2.3	Adsorption isotherm of the activated carbon:	57
4.3	The functional group of activated carbon.....	60
4.4	Adsorption isotherm of paraquat as a toxin onto activated carbon:.....	62
CHAPTER FIVE: CONCLUSION.....		67
5.0	Conclusion	67
BIBLIOGRAPHY.....		69
PUBLICATIONS.....		76

LIST OF TABLES

<u>Table No.</u>		<u>Page No.</u>
1.1	A summary of some research work on activated carbon using coconut shell and palm shell.	6
2.1	Distribution of categories of substances implicated in poisonings 1996 to 2000.	27
2.2	General categories of poisons implicated	28
4.1	Surface area, total pore volume, t-plot micropore volume and average pore diameter of treated activated carbon at different temperatures.	56
4.2	List of functional group observed in the FTIR spectra of treated activated carbon at different temperatures.	60
4.3	Amount of adsorbed paraquat (PQ) onto activated carbon.	64
4.4	Comparison of surface area properties between activated carbon produced in this study and activated carbon produced by other researchers.	65

LIST OF FIGURES

<u>Figure No.</u>	<u>Page No.</u>
2.1 PAC as viewed under light microscope	10
2.2 Activated carbon viewed under scanning electron microscope	13
2.3 Functional group of activated carbon (a) carboxyl group, (b) phenolic hydroxyl group, (c) quinone-type carbonyl group, (d) fluorescein-like lactones, (e) cyclic esters, (f) carboxylic acid anhydrides, (g) cyclic peroxide	16
2.4 Types of adsorption isotherm (Graph: adsorbed volume against	20
2.5 Charcoal tablets	34
3.1 Raw material, palm kernel shell for preparation of activated carbon	40
3.2 A flow diagram in the preparation of activated carbon	43
3.3 EVO 50 Scanning Electron Microscope	44
3.4 LEICA EM SC005 Auto Fine Coater	45
3.5 Activated carbon as suspension form.	47
3.6 Innova 4000 incubator shaker	49
3.7 UV Spectrophotometer	50
4.1a SEM micrograph of produced carbon at 550 °C	53
4.1b SEM micrograph of produced carbon at 600 °C	53
4.1c SEM micrograph of produced carbon at 650 °C	54
4.2 Surface area of produced activated carbon at different temperatures.	55
4.3a Isotherm plot of activated carbon at 550 °C	58
4.3b Isotherm plot of activated carbon at 600 °C	59
4.3c Isotherm plot of activated carbon at 650 °C	59
4.4 FTIR spectrum of treated activated carbon at 550 °C in absorbance mode.	61
4.5 FTIR spectrum of treated activated carbon at 600 °C in absorbance mode.	61

- 4.6 FTIR spectrum of treated activated carbon at 650 °C in absorbance mode. 62
- 4.7(a) Adsorption isotherm of paraquat onto commercial activated carbon at 25 °C
in NaCl (0.9%) solution and distilled water. 63
- 4.7(b) Adsorption isotherm of paraquat onto produced activated carbon at 25 °C
in NaCl (0.9%) solution and in distilled water. 63

LIST OF ABBREVIATIONS

AC	Activated carbon
PAC	Powder activated carbon
GAC	Granular activated carbon
BAC	Bead activated carbon
EAC	Extruded active carbon
HEAC	Highly Efficient Activated carbon
PS	Palm shell
DW	Distilled water
BET	Brunauer, Emmett and Teller method
SEM	Scanning Electron Microscope
FT-IR	Fourier Transform Infrared Spectroscopy
PQ	Paraquat
KBr	Potassium bromide
NaCl	Sodium chloride
H ₂ S	Hydrogen sulphide
NH ₃	Ammonia
HCOH	Formaldehyde
TCAs	Tricyclic antidepressants
TVFM	Theory of volume filling of micropores
AWWA	American Water Works Association

CHAPTER ONE

INTRODUCTION

1.0 ACTIVATED CARBON

Activated carbon is a fine black odourless and tasteless powder made from wood or other materials such as palm shell, coconut shell etc, that have been exposed to very high temperatures in an unventilated environment. It is then treated, or activated, to increase its capability to adsorb various substances by reheating with oxidizing gas or other chemicals to break it into a very fine powder. So, active carbon is a processed carbon material with a highly developed porous structure and a large internal specific surface area. It consists principally of carbon (87 to 97%), other elements such as hydrogen, oxygen, sulphur and nitrogen, and many compounds generated during its manufacturing. Active carbon has the ability to adsorb various substances both from the gas and liquid phases and it can arrest different molecules at the inner surface of its large surface area that justifies it as a very powerful adsorbent (Jankowaska H. et al., 1991). It has a high degree of microporosity. One gram of activated carbon has a surface area in excess of 500m^2 , as determined typically by nitrogen gas adsorption. Sufficient activation for useful applications may come solely from the high surface area, through further chemical treatment often enhances the adsorbing properties of the material. Activated carbon is usually derived from charcoal. It can adsorb poisonous substances before they can cause harm. Activated carbon (charcoal) works by adsorption which is an electrical phenomenon that attracts toxins to the surface of the fine carbon particles.

1.1 ACTIVATED CARBON AS AN ANTIDOTE

Activated carbon, has a well-earned reputation of being a universal antidote. As an antidote, activated carbon can be used in drug overdoses and poisoning arising from chemicals, household products, medicinal substance, etc. The efficacy of activated carbon for the primary treatment of a number of poisonings caused by swallowing of drugs or poisons has been recognized (Donovan J. W, 1987). Activated carbon has been administered for the inhibition of absorption of drugs or poisons in the digestive tract and its efficacy in acute poisoning has been reported (Javaid K. A. and KI-Mabrouk B. H., 1983; Neuvonen P. J. et al., 1983). Chemical poisoning cases that were recorded in Malaysia are commonly related to pesticides used in the agriculture. In Malaysia, published epidemiologic data during the period 1987 to 1995 showed that hospital admissions due to pesticide poisoning nationwide averaged approximately 1000 cases every year (Ministry of Health, Malaysia 1989; 1990; 1992; 1994). The Ministry of Health, Malaysia, reported that from 1995 to 2000 there was an average of about 750 cases per year of pesticide and chemical poisoning (Ministry of Health, Malaysia 1997; 1999). It was also, reported that in Malaysia the most commonest poisons used was a weed killer containing paraquat compound and insecticides containing organophosphates.

In 1997, the Ministry of Health Malaysia introduced a surveillance programme for occupational and work related diseases including poisonings for cases seen in government health facilities. The programme notified 95 cases of poisoning by chemicals and pesticides and reported the commonest causes of occupational poisonings were paraquat (19%), organophosphates (16%), agro-chemicals excluding pesticides (15%) and gases (10%). The mortality from paraquat poisoning is higher

than that from any other type of poisoning associated with agricultural chemicals (Ukai, S. and Kawase, S., 1985).

Paraquat is the common name for the 1,1'-dimethyl-4,4'-bipyridylium chloride and it is a non-hormone, non-selective herbicide. Because of its superior herbicidal effect it has been used all over the world and can produce serious health problems when not properly used (Swan, A. A. B., 1969; Howard J. K., 1980). Paraquat is potentially lethal when accidentally or intentionally ingested. The LD₅₀ (oral) of paraquat in man is assumed to be 40 mg kg⁻¹ (Natori, H., 1979). Paraquat poisoning mostly affects the pulmonary system. Its toxicity is primarily pulmonary, later causing severe lung damage with pulmonary fibrosis over the weeks following exposure (Smith P., 1976 ; Haley, T. J. 1979) which may lead to respiratory defects and hypoxia (Konradsen F. et al., 2003). The gastrointestinal tract prevention of paraquat absorption is critical due to the limited means to significantly increase its elimination from the body (Mascie-Taylor BH, 1983). Moreover, in animals, mortality increased due to delay in beginning gastrointestinal decontamination (Clark DG., 1971) and therefore, in the treatment of paraquat poisoning, early gastrointestinal decontamination is a critical step. The fundamental treatment of paraquat poisoning is by means of gastrointestinal lavage and the selective excretion of toxic substances out of body following the administration of adsorbents. Clay minerals (Smith LL. Et al., 1974), cation exchange resin (Nokata, M. et al., 1984; Staiff, D. C. et al., 1973; Yamashita, 1987), fuller's earth and bentonite used as adsorbents for the primary treatment of paraquat poisoning bind paraquat effectively and prevent its absorption (Clark DG., 1971; Smith LL. Et al., 1974) but in most hospitals their widespread use is limited due to lack of availability. Activated carbon, an adsorbent, is readily available and effective for the primary treatment of paraquat poisoning. Moreover,

among adsorbents, activated carbon has been evaluated as a reliable, safe and inexpensive antidote and is recommended for use in the treatment of acute poisoning (Neuvonen PJ., 1982; Neuvonen PJ. and Olkkola KT., 1988; Palatnick W. and Tenebein M., 1992). *In vitro*, studies have shown that paraquat adsorbs onto activated carbon more rapidly and effectively in normal saline (0.9% sodium chloride solution) than in distilled water (Nakamura, M. et al., 1989; Kitakouji, M. et al., 1989). Normal saline is the most suitable solvent for paraquat removal by activated carbon.

1.2 HISTORY

Activated carbon as an adsorbent was probably first applied in medicine. It has often been used since ancient times to cure a variety of ailments including poisoning. Its healing effects have been well documented since as early as 1550 B.C by the Egyptians. The first identified use of activated carbon dates back to the Ancient Egyptians who make use of its adsorbent properties for purifying oils and medicinal purposes. Physicians treated epilepsy and anthrax with charcoal, during the time of Hippocrates (400 B.C). The students of Hippocrates recommended the dusting of wounds with charcoal as a means to take out their unpleasant smell. In the 1700's, charcoal was often prescribed for bile. During that time the adsorption properties of certain types of charcoal, such as charred animal bones, were discovered. By the early 19th century, for the decolorization and purification of cane sugar, wood and bone charcoal were in large use. From 1870 to 1920, after the development of charcoal activation process, many reports appeared in the medical journals about activated carbon (charcoal) as an antidote for a cure of intestinal disorder and for poisons (Cooney D. O., 1983). By turn of the 20th century, methods for deriving activated charcoal, also called activated carbon from coal became more effective and more

familiar. Systematic research on the antitoxic properties of activated carbon began at the beginning of the twentieth century. Adsorption on activated carbon of such toxic substances as heavy metal salts, alkaloids, barbiturates, phenols and alcohols as well as insecticides and defoliants has been considered (Lichwitz, L., 1908; Joachimogly, G., 1916; Dingemanse, E., 1929; Andersen, A. H., 1948; Chin, L. et al., 1969; 1970; Decker, W., 1968a).

1.3 RAW MATERIAL FOR ACTIVATED CARBON

In Malaysia, for the production of the activated carbon, there are several potential raw material resources like palm shell, coconut shell, etc. In this research, palm shells were selected as a raw material to produce activated carbon due to their easy availability and inexpensive material with high carbon and low inorganic content. A number of researchers have been reported (Nurul, A. B. Z., 2007; Ghafari, S. et al., 2009; Yin, C. Y. et al., 2009; Allwar et al., 2008; Rahman, M. M. and Yusof, A. M., 2011; Che, A. B. C. M., 2006; Sartape, A. S. et al., 2012) in the literature using coconut shell and palm shell as raw materials, as shown below in Table 1.1.

Table 1.1

A summary of some research work on activated carbon using coconut shell and palm shell.

Author	Year	Raw material	Method	Application
Lua and Guo	2001	Oil palm stones	CO ₂ activation	SO ₂ removal
Hu and Srivinasan	2001	Coconut shell and Palm shell	Zncl ₂ and CO ₂	Phenol, Methylene blue
Guo and Lua	2003	Palm shell	H ₃ PO ₄	Ammonia adsorption
Mozammel et al.	2002	Coconut shell	ZnCl ₂	Iodine
Hua et al.	2001	Coconut shell and Palm seed	ZnCl ₂	Phenol and dye
Daud and Ali	2004	Palm shell and Coconut shell	Physical activation (N ₂ gas)	Nitrogen adsorption
Nurul Ain	2007	Palm shell and Coconut shell	H ₃ PO ₄ and ZnCl ₂	Cyanide removal
M. Aroua et al.	2009	Palm shell	NA	Used as cathode material for Nitrate remediation.
M. Aroua et al.	2009	Palm shell	NA	Adsorption of metal ions.
Allwar et al.	2008	Oil palm shell	Zncl ₂	Nitrogen adsorption.
Rahman M.M. et al.	2011	Oil palm shell	NA	Cr adsorption.
C. M. C. Adnan et al.	2012	Palm shell	H ₃ PO ₄	Water filtration.
Kolekar S.S. et al.	2012	Coconut shell	NA	Bi (III) removal.

NA: Not available

Activated carbon (charcoal) is a 100% natural product, obtained from the carbonization of organic matter (hardwood, coconut, bamboo, olive pits, coconut shells, palm shells etc.) as regular charcoal and activated with oxidizing gases, such as steam or air at high temperatures. Its vast system of microscopic pores traps toxic chemicals and speed up their elimination from the digestive system. Since the last century, activated charcoal has been used by physicians to treat various intestinal complaints and because of that, activated carbon have been manufactured by numerous companies. They produced activated carbons, each of which have different adsorptive capacities. Different source materials and manufacturing procedures give each brand of activated carbon its own pore diameters and internal volume that determine its adsorption capacity (Beers, Mark H. MD. and Robert Berkow, MD.,

2004; 2002; Cooney, D. 1999). The United States Pharmacopoeia (U.S.P) standard for activated carbon specifies an internal surface area of $1000 \text{ m}^2\text{g}^{-1}$. Recently, many companies produced super activated carbons which have greater adsorption capacity than standard activated carbon.

1.4 PROBLEM STATEMENTS

1. What is the extent of specific toxicant removal by highly efficient activated carbon?
2. What is the efficacy of highly efficient activated carbon with the increasing level of toxicant?

1.5 OBJECTIVES OF THE RESEARCH

1. To use a local waste material (palm kernel shell) as the source of main raw material for produce highly efficient (pharmaceutical grade) of activated carbon for removal of paraquat.
2. To determine the effectiveness of the activated carbon produced as suspension form through *in vitro* test on paraquat and to compare it with activated carbon that is commercially available in the market.

CHAPTER TWO

LITERATURE REVIEW

2.0 INTRODUCTION

Activated carbon is used in medicine to remove poisons from the body. It is a most powerful adsorbent. It is mainly used as an antidote both for drug overdoses and chemical poisoning. Due to its amazing ability to attract poisons to itself, activated carbon acts to purify and cleanse the body. Activated carbon has been used to purify different products since Roman times. The phenomenon in which molecules of liquid or gas are trapped by either an external or internal surface of a solid is called adsorption and carbon treatment is primarily based on this phenomenon.

2.1 ACTIVATED CARBON FROM CARBONACEOUS MATERIAL

Activated carbon is carbon produced from carbonaceous source materials like nutshells, palm shells, peat, wood, coir, lignite, coal and petroleum pitch. Almost all materials containing a high fixed carbon content can potentially be activated (cameroncarbon.com). Most of the carbonaceous materials do have a certain degree of porosity and an internal surface area in the range of 10-15 m²g⁻¹. During activation, controlled oxidation of carbon atoms is usually achieved using steam at high temperature and the internal surface becomes more highly developed and prolonged. Then, the carbon will have acquired an internal surface area depending on the plant operating conditions (cameroncarbon.com). The internal surface area must be accessible to the passage of a fluid or vapour in order for adsorption to occur. Thus, it is necessary that an activated carbon has not only a highly developed internal surface

but that surface is connected through a network of pores of different diameters. Therefore, to remove the inorganic with dilute acidic solution before carbonization, a new pre-treatment method has been established. Activated carbons with high surface area and high porosity can be produced by two principal methods:

2.1.1 Chemical activation

Prior to carbonization, the raw material is impregnated with certain chemicals. The chemical is typically an acid, a strong base, or a salt (phosphoric acid, potassium hydroxide, sodium hydroxide, zinc chloride, respectively). Then, the raw material is carbonized at lower temperatures ranging from 450–900 °C in the absence of oxygen. So, in chemical activation process, carbonization and activation are passed in a single step through thermal decomposition of raw material impregnated with chemical agent. It is believed that the carbonization/ chemical activation step proceed simultaneously (en.mimi.hu).

2.1.2 Physical Activation or Steam Activation

Physical activation or steam activation is also known as thermal activation. The use of steam for activation can be applied to almost all raw materials. A variety of procedures have been developed but all of these used the same basic principle of initial carbonization at 500-600 °C followed by activation with steam at 800-1100 °C (cameroncarbon.com). The whole reaction (of converting carbon to carbon dioxide) is exothermic. This method involves heating a previously charred material at high temperatures in the presence of an oxidizing gas such as CO₂, N₂, steam etc.

Activated carbon is commonly produced by physical activation and chemical activation. However, chemical activation is preferred over physical activation due to

the lower temperatures and shorter time involved.

2.2 CLASSIFICATION OF ACTIVATED CARBON

Different forms of activated carbon have been employed for various different applications, such as for water filtration, decolourization, detoxifying agents, etc. It is difficult to classify activated carbon on the basis of their behaviour, surface characteristics and preparation methods. However, based on their physical characteristics, activated carbon are classified broadly into

2.2.1 Powdered activated carbon (PAC)

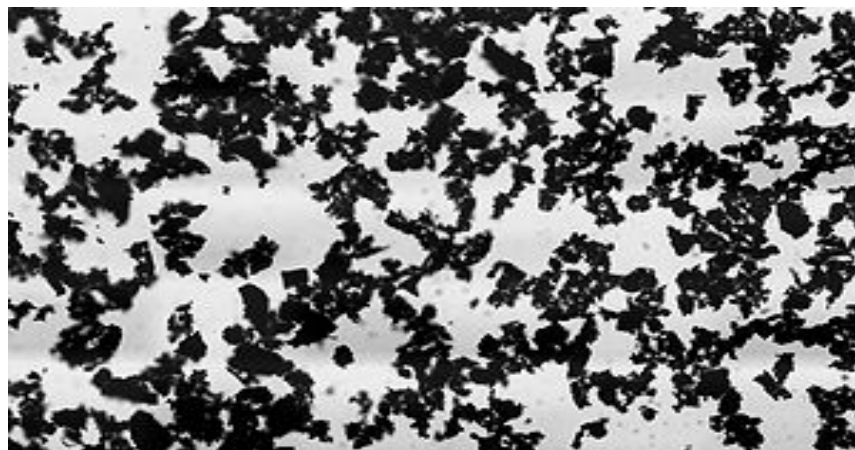


Figure 2.1: PAC as viewed under light microscope

Figure 2.1 shows a micrograph of activated charcoal under bright field illumination on a light microscope. For activated carbon, the fractal-like shaped particles indicate their enormous surface area. Each particle shown in this image, despite being only around 0.1 mm wide, has a surface area of several square meters. Traditionally, active carbons are made in particulate form as powders or fine granules less than 1.0 mm in size with an average diameter between 0.15 to 0.25 mm