



Faculty of Resource Science and Technology

## **The Composition of Organic Geochemicals in Merit Pilla Coal**

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Bachelor of Science with Honours  
(Resource Chemistry)  
2011

# **THE COMPOSITION OF ORGANIC GEOCHEMICALS IN MERIT PILLA COAL**

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A Project Report Submitted In Partial Fulfillment Of The Final Year Project II (STF 3015)

Course

Resources Chemistry

Faculty of Resource Science and Technology

UNIVERSITI MALAYSIA SARAWAK

2011

## DECLARATION

I declare that every part of the project work “THE COMPOSITION OF ORGANIC GEOCHEMICALS IN MERIT PILLA COAL” that I have submitted is genuinely my own work done by me except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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Date: 26 April 2011

## **ACKNOWLEDGEMENT**

First of all I would say Alhamdulillah praise to Allah for giving me the strength and health to do this project work until it done. I owe my deepest gratitude to my supervisor, Prof. Dr. Zaini Asim, whose encouragement, guidance and support from the initial to final level enabled me to develop an understanding of the study. It is a pleasure to thank those who made this study possible especially to Mr. Azzudin Shebli and Ms. Irna Syairina whose has made available their support in a number of ways and shared their personal experienced. Not forgotten to Benedict Samling and Puan Dayang Fatimah for their understanding and assistance throughout this study. I would like to acknowledge and extend my heartfelt gratitude to the following members for their vital encouragement and support: Mr. Nizam Ramli and Mr. Jeffery Lahang Belong. I am indebted to my colleague to support me through out the project progress. Thank you for all cooperation given. Last but not least, I would like to thankful to my family who always gives me support for doing my project.

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## LIST OF ABBREVIATION

DCM	dichloromethane
GCMS	gas chromatography mass spectrometer
GCFID	gas chromatography flame ionization detector
MEOH	methanol
PAHs	Polyaromatic hydrocarbons
PNAs	polynuclear aromatics
Rf	retention factor
TLC	thin layer chromatography
RRF	relative response factor

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# **The composition of organic geochemicals in Merit Pilla coal**

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## **ABSTRACT**

Coals are predominantly originated from plant matters and the formations are associated with the influence of temperature and pressure actions on the plant debris. Coals are varying significantly according to the geographical location and also times taken upon the formation which may result in the different physical and chemical properties of the coal. Merit Pilla which is located in Kapit, Sarawak is one of the coal resources in Malaysia. It is classified under sub-bituminous coal rank. Sub-bituminous coal is the second stage in the coal's formation over time. The objectives of this study are to sequentially extract the organic compounds from Merit Pilla coals by using different polarity of the solvents (n-hexane, dichloromethane, ethyl acetate and methanol; increasing in polarity) which then yields the crude extracts with different polarity (based on the solvents). The crude extracts were then fractionated by using silica gel column chromatography with the applications of different solvent systems. Chemical compositions of each fractionated was then analyzed by using gas chromatography; flame ionization detection and mass spectroscopy (GC/FID and GC/MS). Hydrocarbons obtained from GC/FID on crude hexane and DCM extract are ranges from C<sub>21</sub>-C<sub>33</sub> indicate the terrestrial origin together with waxy coating of grasses and leaves through alkane chain ranges between C<sub>14</sub>-C<sub>20</sub>. Algae also contribute in the formation of coal which represent by carbon C<sub>10</sub>- C<sub>14</sub>. Compounds detected on crude methanol on the other hand such as sulfur compounds can be associated with marine environment. Whereas ester compounds which dominantly detected in the sample may indicate Merit Pilla coal is still in the early process of diagenesis and can be categorized as immature sub-bituminous coal rank.

Key words: geographical location, Merit Pilla coal, geochemistry, hydrocarbon, ester

## ABSTRAK

*Proses pembentukan arang adalah hasil daripada sisa-sisa tumbuhan dan juga dipengaruhi oleh suhu dan tekanan pada sisa-sisa tumbuhan tersebut. Arang adalah berbeza berdasarkan lokasi geografi pembentukan arang tersebut berlaku dan tempoh masa yang diambil untuk pembentukannya. Faktor-faktor ini akan mempengaruhi perbezaan dari segi sifat fizikal dan kimia pada arang tersebut. Salah satu sumber yang membekalkan arang yang terdapat di Malaysia ialah di Merit Pilla, Kapit, Sarawak. Ia diklasifikasikan sebagai arang sub-bituminous. Arang sub-bituminous merujuk kepada tahap kedua yang terlibat dalam pembentukan arang dari semasa ke semasa. Objektif kajian ini adalah untuk mengekstrak komponen organik daripada arang Merit Pilla dengan menggunakan pelarut yang berbeza mengikut kepolaran (n-hexana, dichlorometana, ethyl acetate dan methanol; kepolaran meningkat) yang kemudiannya akan menghasilkan hasil ekstrak yang berbeza kepolarannya. Hasil ekstrak kemudiannya akan dipisahkan dengan menggunakan kolum silika gel dengan menggunakan system pelarut yang berbeza. Komposisi kimia dalam setiap pecahan akan dianalisis menggunakan gas kromatografi jenis flame ionization detection (GC/FID) dan mass spectroscopy (GC/MS). Didapati sebatian daripada kumpulan ester, hidrokarbon bukan aromatik dan hidrokarbon aromatik hadir secara dominan di dalam sampel. Hidrokarbon yang diperolehi daripada sampel hexana dan dichlorometana melalui GC/FID adalah dalam lingkungan  $C_{21}$ - $C_{23}$  yang menunjukkan arang batu tersebut berasal daripada tanaman terrestrial juga mengandungi lapisan lilin dari rumput dan daun melalui rangkain rantai alkane antara  $C_{14}$ - $C_{20}$ . Algae juga turut terdapat dalam proses pembentukan arang batu dan ia diwakili oleh karbon  $C_{10}$ - $C_{14}$ . Sebatian kerosin pada metanol mentah seperti sebatian sulfur dan klorin dapat dikaitkan dengan persekitaran laut. Manakalasebatian ester yang terdapat di dalam sampel menunjukkan senyawa yang menunjukkan arang batu Merit Pilla masih dalam proses awal diagenensis dan boleh dikategorikan sebagai arang batu peringkat sub-bituminous yang belum matang.*

*Kata kunci: lokasi geografi, arang Merit Pilla, geokimia, hidrokarbon dan ester*

## **1.0 INTRODUCTION**

### **1.1 General Introduction**

Coals are forms due to the transformation of a remaining complex mixture of plants which accumulated in the water log environments by microbial and other diagenetic activities. Various types of plants which involved in this process may contribute the different of chemical compounds to the peat and the degree of degradation (Hatcher and Clifford, 1997) which will then influence the chemical and physical properties of the coal. Coal is important in life especially in generating electricity. The energy obtained from the coal today comes from energy that the plant absorbed from the sun million of years ago. This proven by the photosynthesis process which helps in stored solar energy. When plants die, their energy is usually released as the plant decay. However, under favorable conditions of the coal formation will interrupt the decay process and preventing the released of solar energy. This will lead the energy to be locked in the coal. Besides, steam coal is also used for process and comfort heating in many industries and in the residential area and commercial sectors. Coal is burnt in isolated stoves or industrial boilers for central heating systems, while cooking coal is used in the steel industry. In various developing countries, coal plays a small role in transport, either directly in old steam locomotives or as a source for liquid fuels. It is also a source of gaseous fuel which is also known as synthetic gas. There are many reasons in performing a study on coal. First and foremost is due to the importance of coal in world emissions where it can determine the successfulness of the collaboration technology and also experiences providing lessons for other areas. Some of the lessons might have direct implications on coal with large implications for future global

carbon dioxide (Philibert and Podkanski, 2005). Through this way, it enables us to control the environmental pollution which may produce from the usage of coal.

## **1.2 Objective of the Project**

The objectives of the project were:

1. To sequentially extract the organic compounds from coals by using different solvents
2. To fractionate the crude extracts by using silica gel column chromatography
3. To identify the chemical composition of each fraction
4. To predict the source of organic matter in coals based on distribution of organics compounds in coal.

## **2.0 LITERATURE REVIEW**

### **2.1 Coals**

Coals is a fossil that has physical and chemical properties which determined by its geographical location. Different coals vary significantly based on their geographical location and age. It is believed to originate predominantly from plant matter and result from the action of temperature and pressure on plant debris. The relative amounts of remaining plant parts may lead to different types of coal, which are lignite, sub-bituminous, bituminous and anthracite. Different types of coals are formed due to the coalification process. This process takes place in two stages which are biochemical degradation and geochemical degradation. Biochemical degradation involves the chemical decomposition of botanical matter or plants. Geochemical degradation is the stage that follows the biochemical degradation stage (Ludere, 2006).

### **2.2 Chemicals Composition**

Elements such as carbon (C), hydrogen (H), nitrogen (N), and sulfur (s) are types of functional groups found in coal. Whereas the significant oxygen containing groups found in coals are carbonyl, hydroxyl, carboxylic acid and methoxy. The nitrogen containing groups include aromatic nitriles, pyridines, carbazoles, quinolines and pyroles. Sulfur is primarily found in thiols, dialkyl and alkyl-alkyl thioethers, thiophene groups and disulfides. Elemental sulfur is observed in oxidized coal (Stock *et al.*, 1989). Coal rank and maceral type may influence the relative and absolute amounts of the various groups. For example, the principal oxygen containing functional groups in vertrinites of mature coals are phenolic hydroxyl and conjugated carbonyls as in quinones. Spectroscopic evidence exists

for hydrogen bonding of hydroxyl and carbonyl groups such as ketone (Stock *et al.*, 1989). For aromatic of coal molecules, it is increases with coal rank. For example, calculation based on several models indicate that the number of attachment per cluster varies from 9 for lignite to 20 for volatile bituminous coal, and the number of attachment per cluster varies from 3 for lignite to 5 for sub bituminous through medium bituminous coal. The value is 4 for low volatile bituminous (Solum *et al.*, 1989). Reaction of coals and mild selective oxidizing agents such as benzoquinone may cause the coals to lose much of the H content. It is similarly to palladium catalyst where it can be cause the evolution of molecular hydrogen (Stock *et al.*, 1989). This method may be given an indication of the minimum amount of H in the coal that is involved in hydroaromatic rings. This amount is close to the total nanoaromatic hydrogen determined for lower rank coals. Despite, dehydrogenation using Sulfur and using halogens are another types of method in H determination. The values obtained by these methods are lower than that of benzoquinone (Raymond *et al.*, 1962). High resolution mass spectrometry (ms) is one of the methods which have been used by Vorres (2010) to indicate the association of different heteroatoms.

### **2.3 Coals Deposits in Malaysia**

Malaysia has a coal mining history dating back as far as 1851. The coal resource in Malaysia currently stands at about 1, 050 million tones of various coals ranging from lignite to anthracite, but bituminous to sub-bituminous coal forms the bulk of this amount. The resource of coal in Malaysia can be further divided into 231.8 million tones of proven reserve, 171.8 million tones of indicated reserve and 646.4 million tones of inferred reserve (Table 1). 69% of the coal reserves in Malaysia are found in Sarawak while 29% are found

in Sabah and the remaining 2% are found in Peninsular Malaysia. Generally, the coal reserves in Malaysia have heat values range between 21,000 to 30,000 kJ/kg with low ash and sulfur levels (Rahman and Teong, 2004).

**Table 2.1:** Coal resources in Malaysia (in million tones) (Rahman and Teong, 2004)

Location	Reserve			Coal type
	Measured	Indicated	Inferred	
<b>Sarawak</b>				
Silantek	7.25	10.60	32.40	Coking coal semi-anthracite, anthracite
Merit-Pilla	176.20	107.08	121.84	Sub bituminous
Bintulu	-	-	120.00	
Mukah-Balingian	43.60	8.30	98.10	
Sub-total	227.05	125.98	372.34	
<b>Sabah</b>				
a. Salimponon	4.80	1.50	7.70	Sub bituminous
b. Labuan	-	-	8.90	Sub bituminous
c. Maliau	-	-	215.90	Bituminous
d. Malibau	-	17.90	25.00	
e. SW Malibau	-	26.00	-	
	4.80	45.40	257.5	
<b>Peninsular</b>				
a. Batu arang	-	-	17.00	Sub bituminous
Sub-total	-	-	17.00	
<b>Grand total</b>	<b>231.85</b>	<b>171.38</b>	<b>646.84</b>	

## 2.4 Formation of Coal

The conversion of peat to bituminous coal is the result of the cumulative effects of temperature and pressure over a long period of time. The pressure and insulation are provided by the sediment covering the peat which causes the earth's internal heat to be applied to the conversion. The temperature increase is about 2-5 °C for each 100m of depth. The changes that occur in plant matter are termed as a normal coalification. During the process, moisture is lost and the chemical composition is changed. It will contribute to the decrease in oxygen and hydrogen whereas the carbon will increase. The compositional changes are accompanied by the decrease in volatile matter and increases in calorific value. For commercial classification in the United States and for the International classification, volatile matter and calorific content are the main criteria used for the purpose. The changes in rank from bituminous coal to anthracite on the other hand will involve the application of significantly higher pressures. The more distant the coals from the disruption, the less proportionate the alteration. Tectonic plate movement which involved in mountain building will provide pressure for some changes to anthracite. It can be concluded that the older the coal deposit, the more complete the coalification and the higher rank of coal. However, age alone does not determine the rank. Further coal conversions require enough heat and pressure (Othmer, 2004). According to Kentucky Geology Survey (2006) overall coal ranks with increasing order of alteration are including lignite, sub-bituminous, bituminous and anthracite. Coal starts off as peat. It is then metamorphosed from peat to lignite after a considerable amount of time, heat and burial pressure. At this stage, lignite is considered as immature coal due to the colour which is light hue and it remains soft. As time passes, lignite increases in maturity by becoming darker and harder and is then classified as sub-

bituminous coal. Continuous burial process and alteration lead to the occurrence of more chemical and physical changes forming bituminous coal type which is dark and hard in nature. When the coal reached ultimate maturation, anthracite coal type will be formed. Anthracite coal is very hard and shiny in nature. Every class of coal categorized in different coal rank. Rank of coal is the degree of alteration or metamorphism that occurs as a coal matures from peat to anthracite. There are two types of coal ranks which are low rank coal and high rank coal. Lignite and sub-bituminous coal are categorized under low rank coals. These coals have lower energy content because of low carbon content in it. They are lighter and have higher moisture levels. Whereas bituminous and anthracite are categorized under high rank coals. They have more carbon content than lower rank coals which lead to much higher energy content. The appearance of high rank coals is more vitreous (shiny) and lower moisture content than lower rank coals.

## **2.5 Coal Quality**

Coal quality is a term to describe coal chemical and physical properties that influence its utilization. There are a number of laboratory tests that can be used in order to determine the quality of coal such as test for ash, moisture, sulfuric acid and calorific value of the coal. It is important to know quality of the coal as it helps to predict on the behavior and the applications of the particular coals. More over, based on coal quality parameters, effect and implication from the usage of the particular coal to the environment can be determined (Warwick, 2005).

## **2.6 Organic Geochemistry and Organic Matter of Coals**

Geochemistry is a study of chemical changes on the earth. It accounted for the absolute and relative abundances of chemical elements in the minerals, soils, ores, rock, water, and atmosphere of the earth, the distribution and the movement of those elements from one place to another as a result of their chemical, abundance and stability in the universe (Krauskopf, 1967).

Compositional variations of sedimentary organic matter both at bulk as well as the molecular levels are useful palaeoenvironmental indicators. In particular, biomarkers may provide qualitative and quantitative information on biological sources of the organic matter, environmental conditions under which source of organism lived, and the fate of organic matter in the water column and sediment. Organic geochemistry is the studies of hydrocarbon accumulation and content, its subsurface movement and its alteration impacted by temperature and pressure. For nearly 150 years it has been known that the precursor of the hydrocarbons is organic matter deposited in sedimentary basins. Phytoplankton, bacteria zooplankton and terrestrial plant are the main supplies of organic matter. Organic matter and mineral particles are deposited together in aquatic environment. Due to the organic content in water column, organic matter will completely decomposed or there is also a possibility of its preservation (Wilkes *et al.*, 1999).

## **2.7 Inorganic Matter of Coal**

Coal is largely composed of organic matter. However, it is the characterization of the inorganic material in coal, where both mineral and trace elements, that have important ramifications in the technological aspect of coal used and in understanding the

environmental and health problems that may result from coal utilization. Some examples of potentially environmentally harmful trace elements that can be found in coal are such as mercury, lead, arsenic and selenium. These elements may cause environmental damage if concentration is great enough and if there is improperly management taken during disposal and treatment of the ash and smoke which result from the burned coal. Prolonged exposure of the coal to weathering effects or groundwater runoff (acid mine drainage) is another possible mode of release for the potentially environmentally harmful trace elements. For the most part, the concentration of these elements are generally is too small in most power plant feed stocks to cause significant short term damage power plants may result in accumulation of mercury and possibly other harmful trace elements in the environment sufficiently to be regulated in the surface (Warwick, 2005).

## **2.8 Chemicals Contribution of Coal**

According to Hatcher and Clifford (1997) some of the major components of plants that can be recognized in ancient coal seams for example as wood (mainly xylem), leaf cuticular material, pollen /spores and algal materials may contribute to the chemical compositions in coal. Both vascular and non vascular, growing in coal-forming environments each contributed different chemical compounds to the peat and the degree of degradation of the compound varied widely. Thus, coal is a complex assemblage of transformed plant remains whose structure and reactivity can be characterized as the sum of the chemistry of all contributors. (2.7.1-2.7.5)

### **2.8.1 Coalification of Wood (secondary xylem)**

The first reaction involves is the cleavage of aryl ether bonds in lignin; these include the hydrolysis of methoxyl groups and the cleavage of b-O-4 aryl ethers. Both reactions produce a phenolic-OH resulting in the formation of catechol-like structures (1,2-dihydrox-yphenols). The latter reaction leads to the pro-duction of a carbocation on the three-carbon side-chain of lignin, and this carbocation is believed to alkylate adjacent catechol-like aromatic rings. This reaction is an important step in the overall coalification scheme, because cleavage of the aryl ethers would form small, water-soluble phenols which would be expelled from the coal during burial by water. The alkylation reaction preserves the macromolecular integrity of the lignin-derived structures, thereby maintaining the physical integrity of the coal. Alkylation of the C-5 aromatic carbon is easily envisioned in gymnosperm lignin where the main monomer units are guaiacyl (mono-methoxyphenols). In angiosperm wood containing equal or greater amounts of syringyl units (dimethoxyphenols) in the lignin, alkylation at C-5 is blocked by the presence of a methoxyl (Hatcher *et al.*, 1989). Thus, cleavage of the b-O-4 bond in angiosperm lignin will likely produce water soluble monomers which can be removed from the remaining wood.

### **2.8.2 Resinites**

Resinites or also known as amber are the fossil remains of higher plant resins. Two major classes of Resinites are frequently encountered in the geosphere, those derived from the polymerization of labdatriene diterpenoids and those derived from polymerization of labdatriene diterpenoids and those derived from the polymerization of cadinene

sesquiterpenoids. Maturation of polycadienes leads to the formation of monomer (cadinane), dimer (bicadinane), and oligomers (tricadinane, tetracadinane) which can be observed in many oils from southeast Asia and these are formed by cracking of the biopolymer and/or reductive process. It is also clear that alkylated naphthalenes found in oils, coals and sediment extracts also derived from the biopolymers via aromatization.

### **2.8.3 Cuticles of Leaf Material**

Cuticles are petrographic components of many coals which is believed to contribute to coal structure. This biologically and chemically resistant biopolymer is thought to survive nearly intact during coalification by a selective preservation process as most of the other cuticle components are degraded. Structure suggested by GC/MS shown that cellulosic backbones to which the long chain aliphatic structures are linked through ether bonds. The existence of cellulosic structures comes from the identification of levoglucosan in pyrolyzates and the polymethylenic structures derive from the observation of a homologous series of such as n-alkane and n-alkene. The cellulosic components are eventually disappearing during coalification, leaving the polymethylenic structures to form the dominant components of fossil cuticles. Increasing of maturation also may lead to pyrolytic degradation of the polymethylenes, producing a homologous series of alkanes that are typical of the oil extracts of many tertiary coals and may even be involved in the formation of terrestrial oils.