

**SOLVENT SWELLING AND PRE-HEATING ON
SUB-BITUMINOUS MALAYSIAN COALS –
THE EFFECT ON COAL THERMAL STABILITY
AND MICROSTRUCTURE**

BY

**ASSOC. PROF. DR. HJ. KHUDZIR HJ. ISMAIL
MOHD AZLAN MOHD ISHAK**

AUGUST 2006



UNIVERSITI
TEKNOLOGI
MARA
PERLIS
KAMPUS ARAU
02600 Arau Perlis
Tel : 04-9874289
Fax : 04-9874255

Rujukan : 600-UiTMCPs (URDC-5/1/109)
Tarikh : 16 Mac 2004

Unit Penyelidikan, Pembangunan
& Pengkomersilan

Prof. Madya Dr. Hj Khudzir Hj Ismail
Pensyarah
UiTM Perlis
Kampus Arau

Tuan,

Projek : Solvent Swelling And Pre-Heating On Sub-Bituminous Malaysian Coals-The Effect On Coal Thermal Stability And Microstructure

Perkara di atas adalah dirujuk.

Sukacita dimaklumkan bahawa Mesyuarat Jawatankuasa Induk Penyelidikan pada 19 November 2003 telah membuat keputusan :

- i) Bersetuju meluluskan cadangan penyelidikan yang telah dikemukakan oleh tuan dan En. Mohd Azlan Mohd Ishak.
- ii) Tempoh projek penyelidikan ini ialah 12 bulan, mulai 1 April 2004 hingga 31 Mac 2005.
- iii) Kos yang diluluskan ialah sebanyak RM 20,000 sahaja.
- iv) Penggunaan geran yang diluluskan hanya akan diproses setelah perjanjian ditandatangani.
- v) Semua pembelian peralatan yang kosnya melebihi RM 500.00 satu item perlu menggunakan Pesanan Jabatan UiTM (LO). Pihak tuan juga dikehendaki mematuhi peraturan penerimaan peralatan.
- vi) Pihak tuan juga dikehendaki mengemukakan Laporan Kemajuan Projek Penyelidikan setiap 6 bulan. Laporan Akhir perlu dihantar sebaik sahaja projek penyelidikan disiapkan.

Bersama-sama ini disertakan Perjanjian untuk ditandatangani oleh pihak tuan. Sila penuhkan perjanjian berkenaan dengan menggunakan pen berdakwat hitam dan kembalikan ke pejabat ini untuk tindakan selanjutnya.

Sekian, terima kasih.

Yang benar,

PROF. MADYA DR. MAHADZIR HJ. DIN
Ketua URDC

- s.k. 1. Penolong Naib Canselor (Penyelidikan), UiTM Shah Alam
2. Timbalan Bendahari UiTM Perlis

Surat Penyerahan Laporan Akhir Penyelidikan

Tarikh : 30 Ogos 2006
No. Fail : 600-UiTMCPs (URDC-5/1/109)

PROF. DR. AZNI ZAIN AHMED
Penolong Naib Canselor (Penyelidikan)
Institut Penyelidikan, Perundingan dan Pengkomersilan (IRDC)
Universiti Teknologi MARA
40450 Shah Alam
Selangor.

Assalamu'alaikum wbt,

YBhg. Prof.

**LAPORAN AKHIR PENYELIDIKAN "SOLVENT SWELLING AND
PREHEATING ON SUB-BITUMINOUS MALAYSIAN COALS – THE EFFECT
ON COAL THERMAL STABILITY AND MICROSTRUCTURE"**

Perkara di atas adalah dengan segala hormatnya dirujuk.

Bersama-sama ini disertakan dua (2) naskhah laporan akhir penyelidikan tersebut.

Sekian, terima kasih.

Yang benar,



PROF. MADYA DR. HJ. KHUDZIR HJ. ISMAIL
Ketua Projek Penyelidikan
Fakulti Sains Gunaan
Universiti Teknologi MARA Perlis
02600 Arau
Perlis.

TABLE OF CONTENTS

	Page
Surat Perlantikan/Kelulusan Menjalankan Penyelidikan	iii
Surat Penyerahan Laporan Akhir	v
Daftar Ahli Penyelidik	vi
Penghargaan	vii
Table of Contents	viii
List of Tables	x
List of Figures	xi
List of Plates	xii
Glossary	xiii
Abstract	xiv
CHAPTER 1 : INTRODUCTION	1
1.0 World Energy Demand Scenario	1
1.1 Energy Demand Scenario in Malaysia	3
1.2 The Origin and Formation of Coal	5
1.3 The Macromolecular Structures of Coal	8
1.4 Solvent Swelling Properties of Coal	11
1.5 Coal Pyrolysis Background	17
1.6 Pyrolysis and Kinetic Studies on Solvent Swelled Coal	19
1.7 Scope and Objective of the Research	21
CHAPTER 2 : RESEARCH METHODS	23
2.0 Materials and Methods	23
2.1 Chemicals	23
2.2 Coal Sample Selection and Preparation	24
2.2.1 Pre-heated Treatment Procedure	25
2.2.2 Determination of Swelling Ratio	25
2.3 Coal Pyrolysis via Thermogravimetric Analyser (TGA)	26
2.4 Sample Analyses	27
2.4.1 Ultimate Analysis using CHNS Elemental Analyser	27
2.4.2 Proximate Analysis using Thermogravimetric Analyser (TGA)	28
2.4.3 Calorific Value Analysis using Bomb Calorimeter	28
2.4.4 Petrographic Analysis	29
2.4.5 Coal Microstructure Analysis using Scanning Electron Microscopy (SEM)	30
CHAPTER 3 : RESULTS AND DISCUSSION	31
3.0 Characterisation of Malaysian Coal Samples	31
3.1 Swelling Properties of Malaysian Coal Samples	33
3.1.1 Effect of Pure Solvents	33
3.1.2 Effect of Mixed Solvents	35

ABSTRACT

Swelling properties of Mukah Balingian coal with various H-bonding (i.e. pyridine, THF, acetone, ethanol and methanol), non-hydrogen bonding (i.e. tetralin, hexane and toluene) were investigated using volumetric swelling ratio method. A higher rank Silantek coal was used as a comparison study. Besides, the swelling properties on to the pre-heated treated coal samples at 100 and 200 °C also been investigated. In all cases, H-bonding solvents exhibit higher swelling ratio in the coal than the non-H-bonding solvents with pyridine being the best swelling solvent. Swelling ratio of MB coal in all solvents gave higher values than SL coal due to low cross-link density and smaller size of aromatic ring cluster that exhibit in low rank coal. In addition, the swelling ratios of the MB coal with mixed of tetralin with the other H-bonding solvents at volume ratio of 20:80, 40:60, 60:40 and 80:20 were decreased with increased in the volume ratios of tetralin. This is probably attributed to the less interaction of the coal reactive sites with the solvent due to its non-hydrogen bonding characteristics. A high swelling ratio however also being observed with the pre-heated coal samples at 100 °C whereas the swelling ratios were decreased for pre-heated coal at 200 °C. An increase in the swelling ratio for pre-heated coals at 100 °C indicates that heat treatment had caused a decrease in the apparent cross-link density with an increased in open structure of the coal macrostructure. Furthermore, the thermal behaviour of pre-swelled, pre-heated and pre-heated swelled samples that were represented by DTG curve showed some changes on coal macrostructure had occurred during pre-treatment processes. In general, the overall activation energy and pre-exponential factor of all pre-treated samples at second stage pyrolysis (i.e. 200 to 700 °C) that were determined using the first-order kinetic reaction model showed lower values in comparison to the untreated coal due to the bond weakening of the coal macromolecular interaction network of the former. Interestingly, in all cases, the mixed solvent swelled samples exhibit much lower activation energy and higher organic volatile matter in comparison to untreated and pure solvent-swelled coal samples indicating an increased in reactivity of the coal during swelling. In addition, the MB pre-heated pyridine-swelled sample showed a further decrement in activation energy indicating an event weakened of the coal-coal macromolecular interactions with less energy being required during the pyrolysis process. The coal macrostructure of the swelled MB coal samples showed some changes when swelled with H-bonding solvents as showed through SEM micrographs where the swelled coal appeared to be more porous and brittle with the surfaces were being plain and smooth. The coal microstructure also showed some glueing and plastic deformation with formation of smaller particles due to the pores improvement during swelling pre-treatment process. Hence, pre-swelling treatment might give a future approach in increasing coal conversion and oil yield during coal liquefaction processes at less severe condition.