

PHYSICAL AND THERMOCHEMICAL CHARACTERISATION OF MALAYSIAN BIOMASS ASHES

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W. A. Wan Ab Karim Ghani^{1,4}, M.S.F. Abdullah¹, K. A. Matori², A. B. Alias³ and Gabriel da Silva⁴

¹ Department of Chemical and Environmental Engineering, Faculty of Engineering,
Universiti Putra Malaysia, 43400 UPM Serdang, Selangor

² Department of Physics, Faculty of Science,
Universiti Putra Malaysia, 43400 UPM Serdang, Selangor

³ Faculty of Chemical Engineering,
Universiti Teknologi MARA Malaysia, 40450 Shah Alam, Selangor

⁴ Department of Chemical and Biomolecular Engineering,
The University of Melbourne, Victoria 3010, Australia

ABSTRACT

The agriculture sector in Malaysia has been growing rapidly over the years and this leads to large amount of agricultural waste. However, sustainable development and increasing fuel demand necessity had identified biomass ashes can be exploited into many commercial industrial products (i.e. energy, carbon storage, ceramics, construction materials, etc). In this paper, pyrolysis of biomass from four sources (rice husk, palm kernel shell, coconut shell and wood sawdust) has been performed to investigate its correlation with their physical and thermochemical properties in order to exploit their potential application as commercial products in the industry. Generally, biomass ashes have average bulk density between 0.61 and 1.21 gcm⁻³ characterised with high porosity (up to 80%) but low thermal conductivity (below 1 W m⁻¹ K⁻¹). Thermogravimetric analysis on the raw biomass revealed that the heating profile of the biomass governed by three major decomposition of hemicellulose, cellulose, and lignin at respective temperatures of 325°C, 375°C, and 400 to 500°C. Inductively coupled plasma (ICP) detected insignificant amounts of toxic metals in the biomass ashes samples. Silica has found to be major compounds in most biomass ashes samples especially rice husk ashes which explain its highest water absorption properties. Other compounds found include calcium, magnesium and potassium. The large variation observed in biomass ashes properties need to be considered when applying these materials to the environmental systems or designing a commercial product, where outcomes may be correlated with these features.

Keywords: Biomass Ashes; Characterisation; Lignin; Porosity; Thermal

1.0 INTRODUCTION

In Malaysia, more than 2 million tones agricultural wastes are produced annually and potentially an attractive feedstock for energy production as it contributes little or no net carbon dioxide to the atmosphere. Agricultural production in Malaysia continued to record positive growth from 2000 to 2005 and expected to expand more in 2010 consistent to the government policies [1]. There are significant amount of wastes and residues also called as agricultural wastes produced from the post-processing of these products. Major agricultural products are palm oil, sawlogs, paddy and tropical fruits. Table 1 report the production, residues generated and potential energy for selected biomass. In Malaysia, rice husk and paddy straw are among major agricultural wastes that can be applied as biomass- based

power generation. Rice husk referred to an outer cover of rice and in form of hull. The husk account for 22% of the weight of paddy and 78% of the weight is received as rice, broken rice and bran throughout the milling process [2]. While for oil-palm sectors, oil-palm solid wastes (including shell, fibre and its EFB) are cheap and abandoned materials produced during palm oil milling process. For every ton of oil-palm fruit bunch being fed to the palm-oil refining process, about 0.07 tons of palm shell, 0.146 tons of palm fiber and 0.2 tons of EFB are produced as the solid wastes [3]. For bagasse, 3×10⁵T per year was reported in 1999 while rubber wood residues generate 11.32×10⁶m³ in 2000, respectively. Bagasse is the matted cellulose fibre residue from sugar cane that has been processed in a sugar mill. Despite the decreasing acreage, coconut still plays an important role in the

Table 1: Oil palm, paddy and wood production in Malaysia in 2008[3]

Type of Industry	Production (10 ⁶ × Tone)	Residue	Residue product Ratio (%)	Residue Generated (10 ³ *Tone)	Potential Energy, (E) PJ*
Oil Palm (62707×10 ⁶ Tone)	62707	EFB	20.44	11698	57
		Fiber	14.63	8373	108
		Shell	6.58	3766	55
		Total solid		23837	15308
		Other (POME)		38870	
Paddy (214×10 ⁶ Tone)	2141	Rice	22	471	7.6
		Husk			
		Paddy	40	856	8.8
		Straw			
Wood	2937679	Sawn timber	0.5-0.6	1629718	5.2
	523336	Plywood and veneer	0.18-0.65	121000	0.374
	147813	Moulding	0.2-1	75600	0.232

* sample calculation is shown in appendix

socio-economic position of the Malaysian rural population that involves 80,000 households. About 63% of coconut production is for domestic consumption and 37% is for export and industrial processing. The domestic demand for coconut products takes in the form of fresh coconut, tender coconut, coconut oil and processed cream powders. In terms of exports, the country has seen an increase in the export of products of coconut such as desiccated coconut, coconut milk powder and activated carbon. In 2007, exports of coconut and coconut-based products were valued at RM 485,771,416 an increase from the 466.2 million in 2006 [4]. This large amount of residues need to be disposed at landfill and required substantial area to be mounted. By converting these materials into commercial products, this not only will solve landfill problem but also create wealth to the country.

Thermal conversion of solid agricultural waste seems to have a feasible application and has been developed attractively for industrial applications [5]. Recently, biomass ashes have attracted a lot of research with a focus on the application of biomass ashes (biochar) to soils not only contribute to carbon storages but at the same time act as fertilisers [6]. Despite the range of feedstock and techniques available to produce biomass ashes, very little work has been reported on properties of biomass ashes. Thus, detail investigation to evaluate their thermal behavior and structural characteristics is important before they were applied. The aim of the present paper was to characterise the physico-thermochemical properties Malaysian biomass ashes produced via pyrolyses in a bench-scale furnace system. Physical properties such as bulk density, apparent porosity, water

absorption and thermal conductivity were determined using standard procedures. A scanning electron microscopy (SEM) were used to observe biomass ashes surfaces and their porosity. For thermal characterisation, thermogravimetric analyser (TGA) were used to measure the thermal degradation of the raw biomass. For chemical characterisation, the ultimate analyser and inductive coupled plasma (ICP) were used to characterise chemical compositions.

2.0 EXPERIMENTAL

2.1 Raw Material and Ash Preparation

Rice husk, palm kernel shell, coconut shell and sawdust from local (Selangor, Malaysia) mills are chosen for the study. The biomass samples were dried in crucibles in an oven at 110°C for 24 hours to remove moisture. The samples were then ground and sieved to 2 to 3 mm particle sizes. The biomass ashes were obtained by burning (pyrolysis) the raw biomass materials at 800°C for 2 hours in a closed furnace.

2.2 Biomass Ashes Characterisation

Several techniques were used to assess the physical and chemical properties of the ashes. Physical properties such as bulk density, apparent porosity, water absorption and thermal conductivity were determined using standard procedures. For example, the apparent porosity was determined according to ASTM C 134/95, apparent density according to ASTM C 773/88. A scanning electron microscope (SEM) was used to observe the

char surfaces in order to verify the presence of porosity. For SEM studies, the sample were mounted in gold tabs and examined with a HITACHI S 3400 N by different accelerations voltages, depended on the roughness and conductivity of the specimen surface. A thermogravimetric analyser (Metler ToledoSDRA15e) was used to measure and record the mass change with temperature for biomass samples. Tests were conducted at heating rate of $10^{\circ}\text{C min}^{-1}$ over the temperatures 100 to 950°C . For all the pyrolysis runs, 30 mg of sample was used and nitrogen flow was around $30\text{cm}^3\text{min}^{-1}$. The chemical characterisation was performed in order to determine ash content and heavy metal content for each biomass ashes samples. Leaching of heavy metal ions was estimated by the toxicity characteristics leaching procedure (TCLP) method of the US Environmental protection Agency (EPA) by using inductively coupled plasma (ICP).

3.0 RESULTS AND DISCUSSION

3.1 Fuel Properties

Biomass ashes, being derived from a variety of biological feedstock that has thermally degraded exhibit a correspondingly large range in composition and chemistry. Table 2 presents the proximate and ultimate analyses of studied raw biomass samples. In the proximate analyses, biomass in general was characterised by high moisture content and high ash content. The moisture and ash content of the fuel is essential for the choice of the appropriate combustion and gas cleaning technologies. The lower moisture content fuel is preferable in power generation as this parameter will influenced the amount of heat required for the pyrolysis or combustion technologies. Furthermore, the lower ash content also would benefits power or energy production as fly ash formation,

ash deposit formation as well as logistics concerning ash storage and ash utilisation/disposal depend on the ash content of the fuel [7].

Fuels with low ash content are therefore preferable. Agricultural waste usually contains relatively high amount of ash (coconut shell in this case) as compared to coal. However, this problem can be solved by using grate or fluidised bed boilers which are suitable for ash-rich fuels. Furthermore, the composition, density, size and amount of the fly ash emissions formed are influenced by the amount of ash-forming elements in the fuel as well as by the combustion technology and process control applied. A more detailed insight into ash content is given in section 3.4.

The elemental compositions of Carbon(C), hydrogen (H) and oxygen (O) are the main componens found in the ultimate analyses (Table 2). C and H are oxidised during combustion by exothermic reaction (formation of CO_2 and H_2O). The content of C and H contributes positively to the calorific value, the content of O negatively. The C contents of coal and palm kernel shell are higher than those of coconut and rice husk, which explain the slightly higher calorific value. During thermal decomposition the fuel N is almost entirely converted to gaseous N_2 and nitric oxides (NOx (NO , NO_2)). Like nitrogen, the sulfur (S) contained in the solid fuels forms mainly pollutant gaseous sulfur dioxides (SO_2) and alkali as well as earth-alkali sulphates.

Furthermore, the calorific value of the biomass material was lower as double compared to coal. This mean the amount required biomass to produce the equivalent energy as coal is doubled. This phenomenon may contribute logistic problem such as storage when the biomass is utilised.

Table 2: Main characteristics of Malaysian Raw Biomass samples

	Rice Husk	Palm kernel shell	Coconut shell	Sawdust	Coal
Proximate analysis					
(wt% db)					
Volatile matter	60.68	72.47	30.62	51.39	29.2
Fixed carbon	15.02	18.56	26.41	14.29	54.0
Ash	24.30	8.97	42.98	22.67	14.8
Ultimate analysis					
(wt% db)					
Carbon	34.9	51.63	45.24	53.4	70.0
Hydrogen	5.50	5.52	5.04	6.7	8.5
Nitrogen	0.10	1.89	1.46	3.1	2.21
Sulfur	0.00	0.05	0.06	0	0.8
Oxygen(by different)	59.5	40.91	48.2	36.8	18.5
Calorific values	13.5	22.97	16.07	18.3	29.4
(MJ/kg)					

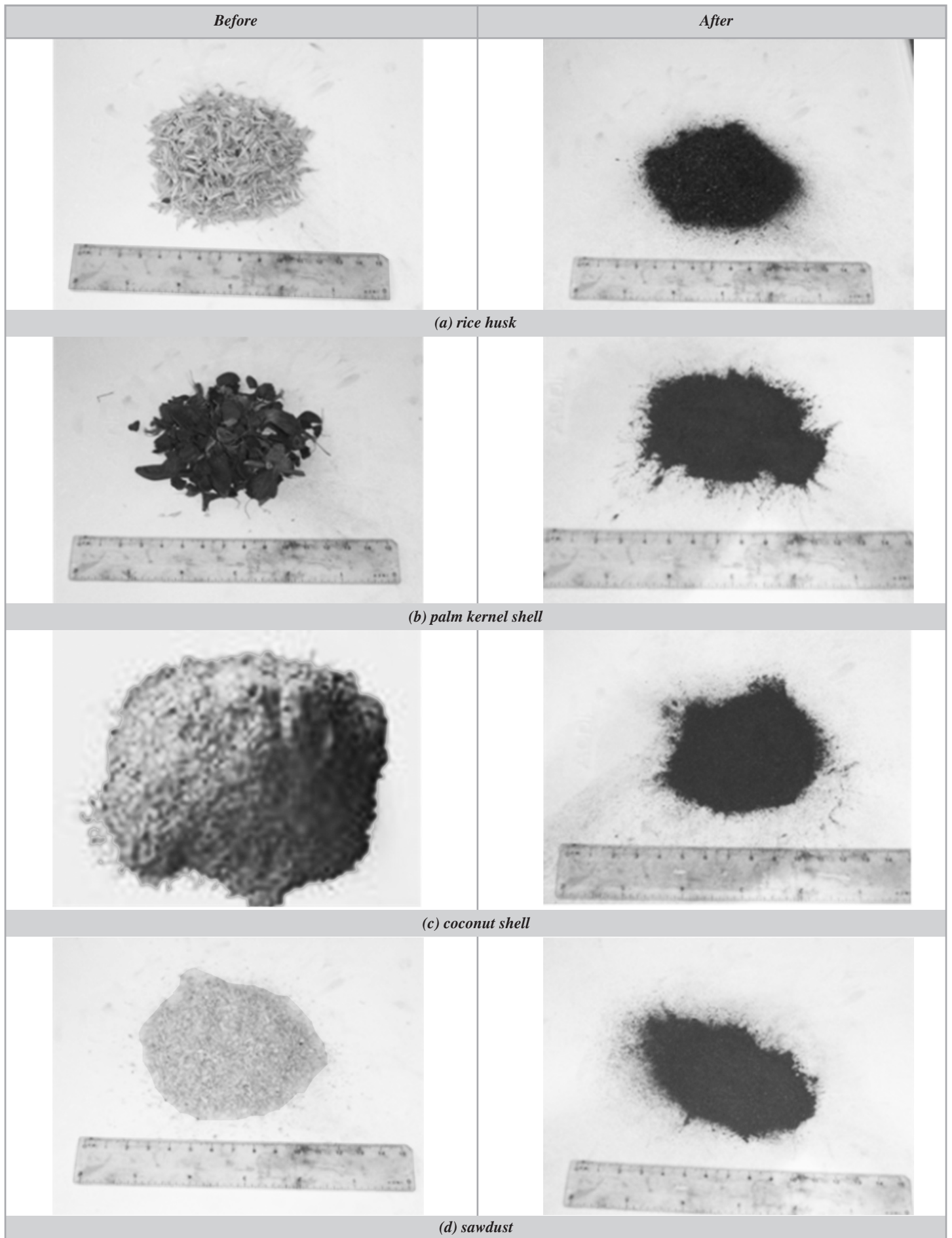


Figure 1: Pictures of biomass samples before and after the thermal treatment

3.2 Physical and Surface Analyses

This section focuses on the physical and macrostructure of biomass ashes. Figure 1 shows the biomass samples before and after thermal treatment while Table 3 lists the physical properties of biomass ashes. As summarised in this table, rice husk ashes showed the lowest bulk density and thermal conductivity but the highest water absorption as compared to other biomass ashes. On the contrary, palm kernel shell had a high thermal conductivity compared to other but very low percentage of porosity. The amount of the entrapped air will increase with increase porosity of the refractory. Hence, the thermal conductivity decreases.

Microscopic observation shows particle shape and structure variation in all raw ashes after heating. Figure 2 illustrates the scanning electron microscope (SEM) that described a fundamental macrostructure of the produced biomass ashes (biochar) for different biomass samples. The presence of aligned honeycomb-like groups of pores most likely the carbonaceous skeleton from the capillary structure of the botanical origin confirmed this structure [8]. As anticipated from the regular size and arrangement of plant cells in most biomass from which biochars are derived, the macro-pore size distribution is composed discrete groups of pores sizes rather than a continuum [13].

Table 3: Physical properties of biomass ashes

	Rice husk	Palm kernel shell	Coconut shell	Wood
Bulk density (gcm ⁻³)	0.61	0.78	1.21	0.98
Apparent porosity (%)	79.7	25.9	28.9	33.7
Water absorption (%)	37.41	19.75	19.99	20.3
Thermal conductivity (W K ⁻¹ m ⁻¹)	0.15	0.97	0.85	0.80

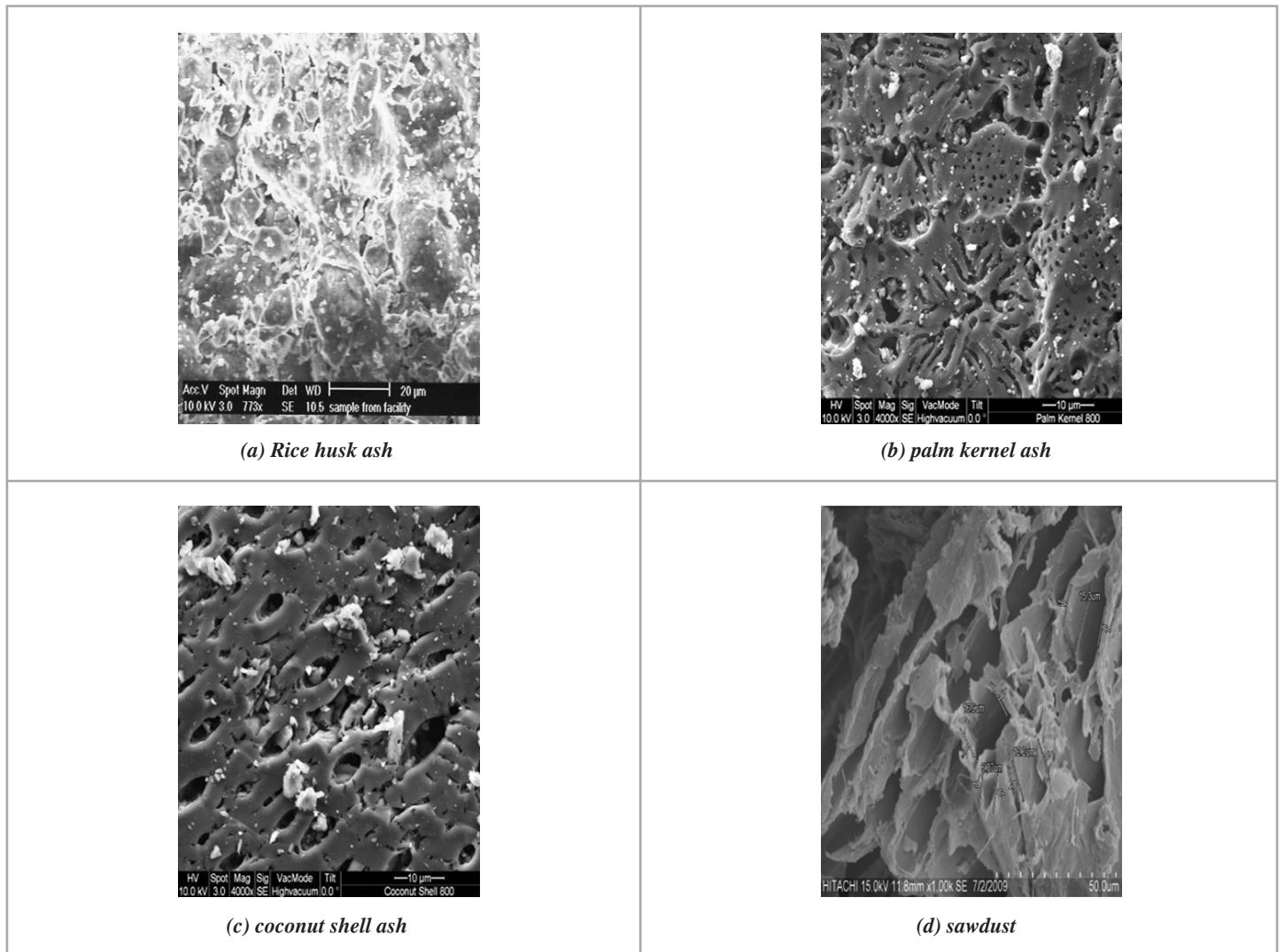


Figure 3: Scanning Electron Microscope (SEM)

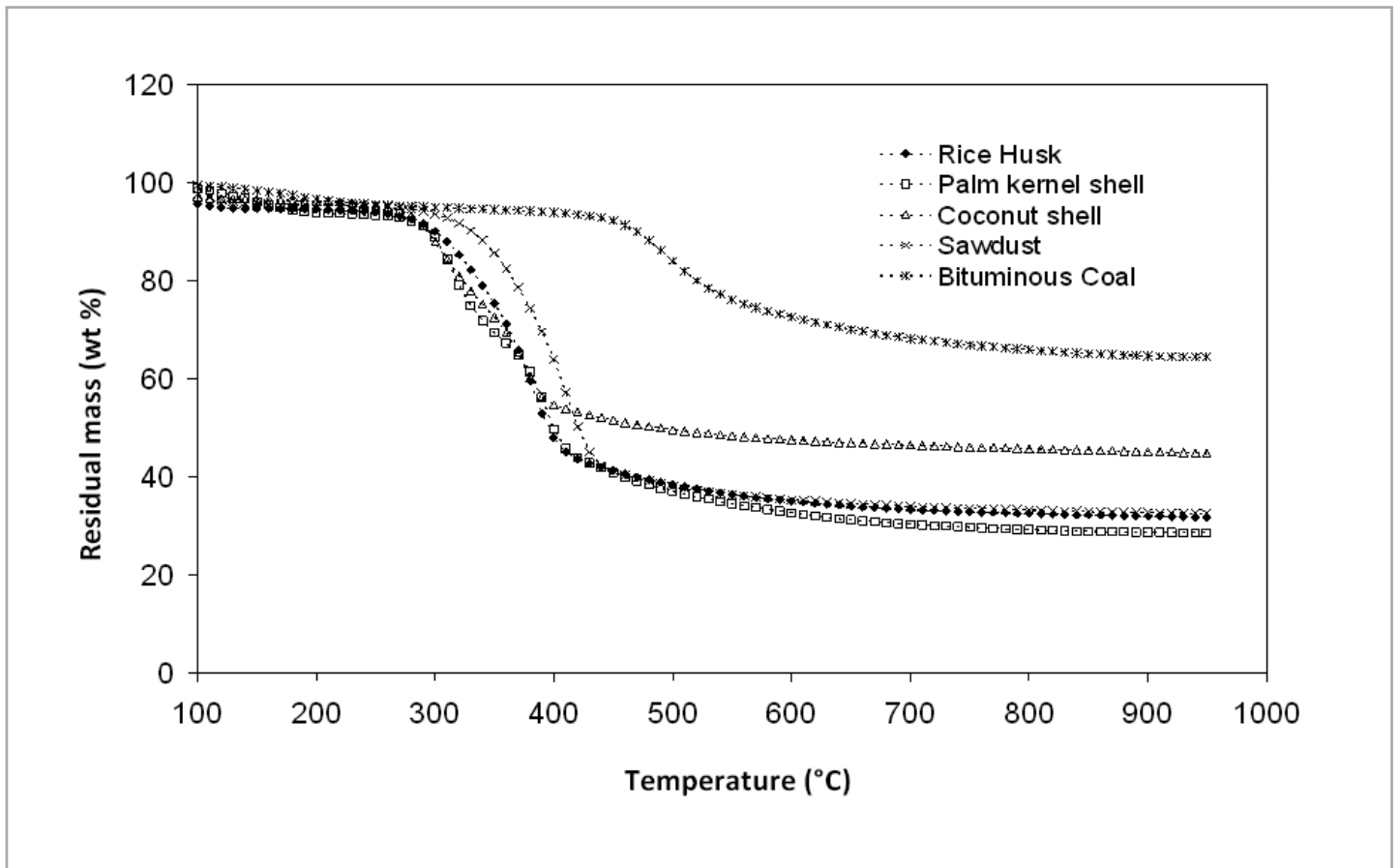


Figure 4: Thermogravimetric analysis (TG) of the pyrolysis of biomass samples at constant heating rate ($10^{\circ}\text{C min}^{-1}$) with N_2 sweep gas at 120 mL min^{-1}

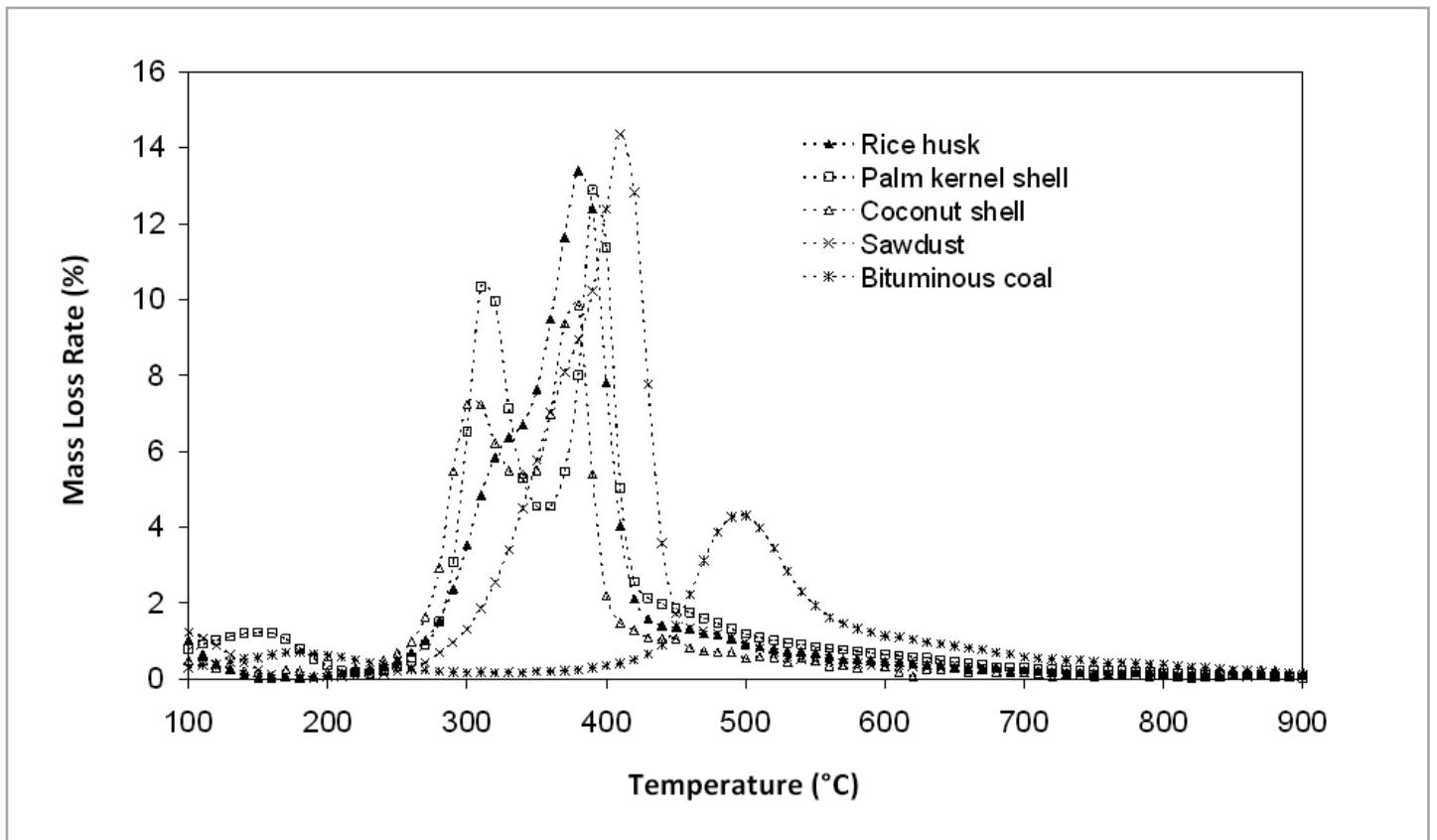


Figure 5: Differential thermogravimetric analysis (DTG) of the pyrolysis of biomass samples at constant heating rate ($10^{\circ}\text{C min}^{-1}$) with N_2 sweep gas at 120 mL min^{-1}

3.3 Thermal Analyses

The thermogravimetric (TG) and derivative of thermogravimetric (DTG) curves for coal and studied raw biomass undergoing pyrolysis in a nitrogen atmosphere at a heating rate of $10^{\circ}\text{C min}^{-1}$ is shown in Figures 4 and 5, respectively. The thermal decomposition starts at lower temperature approximately 200°C for agricultural waste compared to 350°C for coal. Furthermore, agricultural waste samples showed two overlapping peaks and a flat tailing section. It has been debated by other researchers that the lower temperature peak represents the decomposition of hemicellulose in the material and the higher temperature peak represents the decomposition of cellulose [8-11]. The flat tailing section of the DTG curves at higher temperature represents lignin, which is known to decompose slowly over a very broad temperature range [11]. The TGA shows about 17 wt% losses of physically bound water at 100°C . This could be due to volatilisation of unburned carbon and other organic matter present due to incomplete combustion as well as SiO_2 present in agricultural waste ash samples. On the contrary, in case of rice husk ash the weight loss is insignificant. This can be attributed to the fact that rice husk comprises primarily of silica with negligible amounts of volatile oxides. On the other hand, for the TGA curves of bituminous coal, the decomposition starts at about 350°C , which is significantly higher than the one

corresponding to the biomass samples. The maximum pyrolysis rate occurs at 500°C , at a level of $3 \times 10^{-2} \text{ min}^{-1}$ which is 5 to 7 times lower than that of the biomass materials, thus indicates that bituminous coal is less reactive. This phenomenon is reflective in the DTG curves which represent by peak height and peak temperature. Hence, the higher potential for energy production would be suggested for pyrolysis and gasification [12].

3.4 Chemical Characterisations

Table 4 shows the selected major elements formed in the ash components of the biomass ashes (biochar). From the results it is observed that SiO_2 is predominant in most of the ashes except for wood samples. Other compounds of phosphorus, magnesium, aluminium, calcium, iron and potassium are also present. These elements are of relevance for ash melting, deposition, formation, emissions as well as corrosion. A high content of potassium (K) and silica (Si) in all of the agricultural wastes are observed. This usually will decrease the ash melting point which may cause sintering or slag formation in the combustion chamber. In addition, melts occurring in fly ash particles may cause hard deposit formation on cooled furnace walls or heat exchanger tubes [6]. However a low content of aluminium and iron are observed in coconut shell sample. It tends to suggest that the origin of the ash is mostly inherent, rather than adventitious material from sand, clays and soil [13].

Table 4: Ash composition of Biomass Ashes (biochar)

Compound	Percentage (%)			
	Rice husk	Palm kernel shell	Coconut shell	Sawdust
SiO_2	89.57	31.73	42.45	8.78
Al_2O_3	1.32	3.46	3.31	13.89
Fe_2O_3	1.43	1.78	2.57	2.35
P_2O_5	1.04	2.57	2.33	19.32
CaO	0.77	20.27	1.21	2.72
TiO_2	1.01	12.39	1.33	35.72
MgO	0.76	1.01	21.36	0.35
Na_2O	1.15	1.38	1.97	1.04
K_2O	1.65	1.51	2.01	1.93
Cl	1.3	0.08	0.98	0.07
MnO	-	1.27	1.91	-
C	-	12.55	19.67	13.83

Table 5: Results of TCLP tests for selected biomass ashes

Heavy metals	Rice husk ash (ppm)	Palm kernel shell (ppm)	Coconut shell (ppm)	Limit (ppm) [15]
Cd	0.084	0.099	0.082	1.0
Cr	0.702	0.069	0.049	1.75
Cu	0.962	0.108	0.281	100
Pb	0.149	0.215	0.016	0.25
Ni	0.083	0.058	0.035	100

Generally, much of the mineral content in the biomass is carried over into the biomass ashes (biochar) where it is concentrated due to loss of C, H and O during pyrolysis. The amount and distribution of mineral ash in the biochar is influenced by its feedstock and process conditions. However, Table 5 reveals that all the toxic metals of selected biochar samples by TCLP tests were found to be below than regulation limit. Bridgewater suggested that the partial pressure of O₂, steam and carbon dioxide (CO₂) controls the amount of mineral ash in the biochar [14].

3.5 Potential Application of Biomass Ash

Based on the above properties, it appears that the presence of porosity and high water absorption, low thermal conductivity and immobilised toxic metals of the most biomass char (biochar) will make them suitable to be recycled for ceramic products such as insulators, filters, structural ceramics, adsorbent materials, etc. These applications have not been explored for biomass ashes. For instance, ceramic membranes can be used for clarification, separation and decontamination in several industries such as food processing, petrochemical and wastewater treatment [2]. In perspective with typical soil application, these structures vital soil functions such as aeration and hydrology [14], soil water holding capacity and adsorption capacity [16-18]. Furthermore, food-processing industries in Malaysia (dairies, beverage) where rice husk is commonly used in the boilers, can be use ceramic membranes for separation (e.g. removal of whey protein from whey) and clarification (fruit juice clarification). The presence of unburnt carbon in biomass ash could enhance its adsorbent properties. There is also the possibility to separate and activate the unburnt carbon as has been done for coal fly ash. The advantages of using unburnt carbon includes high yields of activated carbon since the carbon has already undergone devolatilisation and simplified process since only activation is required. Recently, biomass ashes (biochar) also claimed can be use to mitigate a greenhouse gases especially carbon dioxide (CO₂) from atmosphere or from power plant.

4.0 CONCLUSION

Physico-chemical properties of the biomass ashes are found to be varies and greatly influenced by the origin of their raw materials. Physically, biomass is characterised with high moisture and ash content but lower calorific value as compared to coal. These properties indirectly contributed to high water absorption and low thermal conductivity to the materials. Morphologically, SEM images reveal the presence of irregular shape and porous structure in all biomass ashes. Furthermore, thermal analysis shows most of the weight loss is in the range of 320-600°C which mainly governed by decomposition of cellulose, hemicelluloses and lignin in the raw biomass samples. These findings not only will gave some temperature profile of the raw materials but also will provide useful appropriate operating temperatures to deal with biomass samples depends on the target products. Furthermore, biomass-derived ashes exhibits a correspondingly large range in composition and chemistry over the studied pyrolysis temperature. Generally, SiO₂ is predominant in most of the biomass ashes except for wood samples. A high content of potassium (K) and silica (Si) in all of the agricultural wastes are observed. Apart from high ash content, insignificant amount of metals were found in the biochar samples except for Ca, Fe and Zn, albeit in very low concentration (less than 100 ppm). In conclusion, the properties of biomass ashes (biochar) products may affect many of the functional roles that they may play in environmental management applications. The large variations of their physical, thermal, and chemical characteristics observed in the biomass-ashes (biochar) will be more effective than others in certain applications. Hence, it is important that these characterisations are undertaken before they were experimentally applied to environmental systems and variations outcomes maybe correlated with these features.

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APPENDIX

a) Calculation of potential energy

$$\text{Potential Energy (E)} = \text{Mass of biomass (tonne)} \times \text{Calorific Value of biomass (kJ/kg)}$$

Example : energy potential from palm fibre residues (refer Table 1 for values)

$$\begin{aligned} E &= 11698 \times 10^3 \text{ Tonne} \times 4.87^* \text{ (kJ/kg)} \\ &= 11698 \times 10^3 \text{ Tonne} \times 4.87 \times 10^9 \text{ (J/Tonne)} \\ &= 56.9 \times 10^{12} \text{ J} \\ &= 57 \text{ PJ} \end{aligned}$$

* from experimental data / literature data

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PROFILES



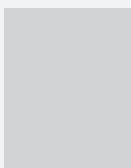
DR WAN AZLINA WAN AB KARIM GHANI

Dr Wan Azlina has a B Eng in Chemical Engineering from Universiti Teknologi Malaysia and a PhD in the area of Biomass combustion technology from University of Sheffield under supervision of Dr. Keith Cliffe. Later, she applies her knowledge as a lecturer cum active researcher towards biomass conversion technologies, particularly thermal treatment such as combustion, gasification and pyrolysis technologies. She further enhanced her interest in biomass pyrolysis product (biochar) for carbon capture study to mitigate global climate change in her fellowship research in University of Melbourne, Australia. Currently, she is a senior lecturer in Department of Chemical and Environmental Engineering, University Putra Malaysia. She had published several articles in high citation index journals, refereed journals and also manuscripts in conference proceedings.



AZIL BAHARI ALIAS

Mr Azil Bahari Alias has a B. Eng in Chemical Engineering from Universiti Teknologi Malaysia, Master of Science from University Technology MARA (UiTM) and pursuing his PhD in Energy and Environment in University of Melbourne, Australia. He currently a lecturer in Faculty of Chemical Engineering, University Technology MARA Malaysia. His research interest includes biomass combustion characterization, co-combustion of biomass with coal, coal cleaning/treatment and municipal solid waste management. From his excellencies in research, he has won several awards national and international exhibitions namely Invention, Innovation and Technology Exhibition (ITEX 2008) and British Invention Show (BIS 2008). Apart from that, he had published several articles in high citation index journals, refereed journals and also manuscripts in conference proceedings.



MOHD. SYAFIK FIRDAUS ABDULLAH

Mohd Syafik has a B. Eng in Chemical Engineering from Universiti Putra Malaysia and continuing his Master degree entitled "Vitrification of Biomass ashes". He managed to publish several articles in conference proceedings during his study period.



DR GABRIEL DA SILVA

Dr Gabriel da Silva studied PhD and B. Eng. in Chemical Engineering in University of Newcastle, Australia. During his bachelor study, he held a position as an Engineering Research Assistant at BHP Billiton, Biohydrometallurgy Research Group (2000 – 2002), Postdoctoral Research Fellow, New Jersey Institute of Technology, Chemistry and environmental Science (2005 – 2007) and later as a Lecturer in The University of Melbourne, Chemical and Biomolecular Engineering, (2007 – present). Dr da Silva's research interests are broadly in the area of energy and the environment, using computational chemistry techniques and reaction rate theory to model complex reaction networks relevant to combustion and atmospheric chemistry, as well as environmentally important organic reaction mechanisms. The current research focus is directed toward understanding and modeling reaction processes related to the use of biofuels (ethanol, biodiesel, and biomass), allowing us to optimize their combustion and thereby support the uptake of these green, renewable energy-sources. Studies are also underway to model the formation and atmospheric reactions of pollutants like soot, persistent free radicals, and ozone depleting substances. A further area of interest is bioleaching, where naturally occurring bacteria are used to leach base and precious metals from sulphide minerals.



DR KHAMIRUL AMIN MATORI

Dr Khamirul Amin Matori joined the Department of Physics, Universiti Putra Malaysia in 2000 as a Lecturer followed by eventual promotion to Senior Lecturer in 2008. He attained his PhD from the University of Sheffield under supervision of Professor W.E. Lee and Dr M.I. Ojovan in 2007 in the area of glass and glass ceramics. He has wide ranging research interests in glass and ceramic science, with particular emphases on the processing, microstructures and properties as well as waste immobilisation. Current research topics include development of nanostructured mullite-base ceramic composites from waste, design of phase change material composites for high-temperature ceramics. Other work includes fabrication of willemite-based glass and glass ceramics, preparation of oxide/carbide coatings and novel armour materials and waste-treatment.