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Utilization of durian biomass for biorenewable applications

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Utilization of durian biomass for biorenewable applications

by

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DEDICATION

اِنَّ صَلاَتِي وَنُسُكِي وَمَحْيَايَ وَمَمَاتِي لِلَّهِ رَبِّ الْعَالَمِينَ

To

Papa, Mama, Izzah,

Harizz, Ayla, Faizz and my unborn son.

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ABSTRACT

The utilization of tropical fruit biomass as feedstock for biorenewable resources is an attractive proposition due to its abundance and potential to reduce reliance on conventional sources as well as the overall economic gain to all stakeholders. This study aims to pave the way towards successful and viable utilization of this feedstock by exploring and examining key physical and chemical traits of durian biomass, a major tropical fruit crop in Malaysia.

The study was divided into three main parts: identification of durian biomass moisture test method, characterization of physical and chemical properties of durian biomass for thermochemical conversion, and composting of durian biomass. In the first section, HORT 2 method was found to be the best available moisture content test and depending on the process optimization need, ground samples can provide the fastest accurate result, while cubed or whole samples provide easier handling but require adequate drying time and temperature. Durian biomass was found to be comparable to most biomass studied in the literature. Its low ash value and high calorific value, especially when compared to palm oil biomass which is currently used in co-firing plants, makes it an attractive option. For other thermochemical applications, high moisture and oxygen content in durian biomass requires drying and pretreatment for optimal processing. In the composting section, nutrient content observed from the analysis of the compost indicates that durian rind is suitable for greenhouse media application. However, pH conditioning, nutrient supplement, and particle size reduction are needed to amend and improve its quality.

CHAPTER I. GENERAL INTRODUCTION

Research Motivation

Rising energy costs, reliance on depletable conventional energy sources and environmental conservation are main instigators driving most economies towards more sustainable alternatives. Under various policy impetuses (EPU, 2010), Malaysia is gearing towards incorporating more renewable alternatives to diversify its energy mix. The abundance of various biomass resources from agricultural activities is seen as a potential feedstock source that can be converted into various biorenewable resources.

Tropical fruit crops are one of the major agricultural production crops in Malaysia with an annual production of 1.6 million Mg (DOA, 2012). Current practices of discarding post-harvest agricultural waste in landfills or through open burning create obvious and grave ramifications to the environment and general health standards. Furthermore, supply glut and its adverse impact on the farmers' economic gain further magnifies the need to devise and introduce not only a better post-harvest management system but also an avenue to add value to these discarded waste streams.

Fruit crops research, production and marketing are under the purview of various governmental agencies that also oversee the implementation of governmental policies, dissemination of information to the stakeholders as well as logistical management of all key assets. This existing structure can be seen as a great advantage for the implementation of biorenewable conversion processes that are often plagued by logistical and public acceptance issues (Gold and Seuring, 2011; Savvanidou et al., 2010).

Research Overview

Biorenewable technology encompasses various conversion routes that involve conversion of various feedstocks into energy, chemicals and materials through thermochemical or biochemical processes, or a combination of both. The body of research into this area is vast and covers a myriad of different feedstocks. Before any biorenewable utilization of tropical biomass can begin, it is imperative that its fundamental chemical and physical characteristics be investigated and explored. This can be done by applying existing techniques established for widely researched feedstocks such as corn residues, soft and hardwoods, and perennial grasses. Being a major contributor to the tropical fruit industry, choosing durian biomass for biomass exploratory studies was an obvious choice and the knowledge garnered will then be extended to other tropical fruit types as well.

The key aspect of this study was to quantify the potential of utilizing durian biomass in thermochemical conversion processes. Moisture content is a vital aspect for this processing route that affects not only the processing parameters but also storage and logistical decisions. In order to avoid confusion or uncertainties as seen in moisture testing practices for dried distiller's grains (Thiex, 2000), establishing an accurate moisture method test must be carried out. This is done through two phases, where phase one is the comparison of existing methods of oven drying with reference to a Karl Fischer method. Phase two consists of the evaluation of moisture content measurement through drying at different temperatures, drying times and sample preparation methods.

In thermochemical conversion processes, physical and chemical characterization is integral in understanding the thermal degradation properties of biomass. Chemical composition also affects the pyrolysis process where high nitrogen and ash concentration negatively affect hydrocarbon yields (Sanderson et al., 1996). Here, proximate and ultimate analyses need to be done to determine key attributes and traits such as ash and volatiles, as well as elemental and metal fractions. These traits will lead to further analysis of the material such as yield prediction, mathematical models, process optimization and economic analysis.

Other than thermochemical conversion routes, composting is seen as a comparatively simple and low-cost waste treatment alternative that can be easily and quickly implemented in the industry. Compost can be utilized in various applications including greenhouse potting media, forest soil amendment (silvicultural), and land reclamation applications (Shiralipour et al., 1992), as well as highway embankment erosion control (Glanville et. al, 2004; Bakr et al., 2012) and as a treatment measure for bio-security purposes in response to large scale emergency livestock mortality (Glanville and Trampel, 1997; Glanville et. al., 2006). Application of compost as greenhouse media is an attractive option particularly due to increasing cost and environmental impact of peat extraction (Herrera et al., 2008; Benito et al., 2005). Therefore, evaluation of key composting characteristics such as temperature profiles and carbon dioxide production rates need to be observed during the composting of durian rind at different levels of carbon and nitrogen (C:N) ratios and sample preparation methods. The compost products will then be evaluated through relevant chemical and physical tests to gauge their viability for greenhouse media applications.

Thesis Organization

Understanding the need for renewable and sustainable energy alternatives in Malaysia can be fortified by viewing the energy scenario from a historical, present, and projected outlook in Chapter 2. It also includes a summary of both conventional and renewable energy alternatives including biomass utilization potential.

This thesis follows the thesis with journal papers format, where the following chapters will incorporate individual scientific reports related to the three focus areas of this research. Chapter 3 is the report on the determination of suitable air oven moisture content test for durian biomass. The physical and chemical characterization for thermochemical conversion and pyrolysis product identification will be discussed in Chapter 4. The third research focus, involving the study of durian rind composting, will be presented in Chapter 5. Lastly, a generalized conclusion based on the conclusions of all four chapters is presented in Chapter 6.

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CHAPTER 2. OUTLOOK OF ENERGY DEMAND AND RENEWABLE ENERGY POLICIES IN MALAYSIA

A paper to be submitted to *The Biomass and Bioenergy Journal*

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Abstract

Malaysia is a ‘newly-industrialized’ country that relies heavily on its exports of oil and is a major producer of palm oil and rubber. Due to favorable geographical attributes, renewable resources such as hydroelectric, solar and biomass provide attractive alternatives to dependence on fossil fuels. Fuel diversification policies were introduced in 1999 to promote the use of renewable energy, particularly in electricity generation. Diversification of energy resources are one of the main pillars in the new Malaysia plan. Other than hydroelectric and solar energy, biomass from palm oil and other agricultural practices including fruit crops can be utilized to produce energy and renewable resources. Although existing policies point towards integration of sustainable energy resources, a more aggressive approach is needed to substantially offset fossil-fuel consumption.

Introduction

Diversification of energy sources and achieving energy independence are seen as key steps in ensuring stable economic growth and societal development. Although blessed with

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relatively abundant natural resources, Malaysia is not immune to the challenges of dwindling energy resource supplies, environmental stewardship and sustainable development. Due to favorable climate for cultivation of various kinds of crops for food and energy, the potential of diversifying the energy mix through utilization of these renewable resources is very promising.

Beyond the promise of palm oil and its derivative, Malaysia is also home to a myriad of other crops that can be utilized as well. Within the existing framework of governmental agencies that governs the cultivation, collection, distribution and marketing, as well as research and development, a concerted effort between these entities will facilitate a smoother transition and implementation of sustainable initiatives. To envision an energy mix that incorporates all available natural resources as well as renewable options, it is essential to look at current and future energy demands as well as the important figures pertaining to existing and renewable energy resources. This paper will summarize these key facts as well as highlight important policies that may facilitate the implementation of sustainable initiatives and finally look into how biomass can contribute to the mix.

Historical overview

Historically, the Malaysian economy and its multicultural makeup were heavily influenced by its British colonial masters. During the latter part of the 19th century, tin mining and rubber plantations were introduced by the British and with them, the influx of foreign laborers from India and China. After gaining independence in 1957, mining and agriculture became the backbone of the economy. The 1970s saw the shift towards a more multi-sector economy with more emphasis in developing its industrial sector and its wealth in oil reserves. The energy crisis during the 1970s has not only greatly shaped the energy landscape of the world but in Malaysia as well. Oil was the main contributor to the energy mix with about 87.9 % in 1980 (MoE, 2010).

To reduce the dependency on foreign oil, several policy measures were taken by the government. The national oil corporation, PETRONAS, was formed under the terms of the Petroleum Development Act passed in 1974. The company was given exclusive rights to explore, develop and produce petroleum resources in Malaysia. In 1979, the National Energy Policy was introduced to ensure the provision and full utilization of cost effective and diversified energy. With regards to prolonging the lifespan of the oil reserves as well, two further acts were passed. They are the 1980 National Depletion Policy and the Four-Fuel Diversification Strategy 1981. These measures were taken to ensure future energy security and stability, and balancing the utilization of oil, gas, hydroelectric and coal.

Energy demand

Foreign and local investments, open market practices and state-influenced macro-economic planning in the preceding years saw the country bounce back from serious regional and global economic downturns, and emerge as a major economic presence in Asia. Malaysia is currently classified as a *newly industrialized country* and the major economic activities involve exports of oil and palm oil, as well as being a major exporter of semiconductor and electronic goods. Gross domestic production (GDP) averaged above 6% growth from 1990 to 2005 and it was as high as 9% yearly growth before the Asian Financial Crisis in 1997 (MoF, 2000). The corresponding total primary energy consumption climbed to 60.4 Mtoe[1] (2.5 x 10^9 GJ) in 2005

[1] Mtoe = million tonne of oil equivalent (1 toe = 42 GJ)

from 19.6 Mtoe (0.8 x 10^9 GJ) in 1990 (EPU, 2006).Although the country is seeking to move away from the manufacturing industry as a measure to counter uncertainties in global trade market and volatile oil prices, manufacturing will still play a big role in the economy and it figures heavily in various economic projection studies.

Based on simulations done by Gan and Li (2008), Malaysia's GDP is expected to average 4.6% growth rate from 2004 to 2030, with the total primary energy consumption expected to grow at an average of 4.3% annually in the same period tripling the projected energy demand to 160.9 Mtoe (6.7 x 10^9 GJ). In the same period, GDP share of agriculture sector is projected to drop further from 10.2% to 7.2%. Marginal increase of 1% is projected for industrial and service sectors from 50% and 41% GDP share respectively, within the same time period as well (Gan and Li, 2008). A similar study done by the Asia-Pacific Economic Cooperation (APEC) in 2006 projected the primary energy demand increase of 146.7 Mtoe (6.1 x 10^9 GJ) by 2030 with an annual rate of 3.5%. The increase in GDP also translates into better spending power and an increased standard of living. This results into increased car ownership, up to 12.8 million cars in 2030 from 6 million in 2004 (Gan and Li, 2008) and an increase in electricity demand as well.

The future outlook of the energy mix will depend heavily on the compelling need to cater to the demands from the industrial sectors and the public in the form of transportation fuel and electricity. Lau et al., 2009 summarizes the breakdown of energy users, where a sizeable chunk goes to the transportation and industrial sectors, which account for 40.3% and 38.6% respectively. Residential and commercial sectors account for 13%, as well as 0.3% for the agricultural sector. It will be interesting to view the energy scenario in Malaysia from both a chronological aspect as well as having an overview look at the policies taken in promoting and developing the alternatives to the current mix. Through these policies, it will be possible to gage the preparedness of the country to meet future energy challenges and adherence to international climate change regulations.

Current Energy Supply

Oil and natural gas

Malaysia's revenue and energy structure has been built around petroleum, where 40% of the revenues are estimated to come from oil and gas related activities. Looking at the energy mix, oil and natural gas accounted for more than 80% of the total mix from 1978 to 2000 (Figure 1). As of 2011, Malaysia has proven oil reserves of about 4 billion barrels, which comes mostly from offshore oilfields. Total oil production in 2011 was an estimated 630,000 barrels per day, of which 83% was crude oil (EIA, 2011). Malaysia is still a net exporter of oil, however with peak of production occurring in around 2003, increased consumption has reduced net exports. With the declining production and increasing demands, the government has turned to promoting enhanced output and advancing exploration in deepwater areas. Through new tax and investment incentives, the objective is to increase aggregated production capacity by 5 percent per year up to 2020. Blocks of potential oilfields were opened to foreign companies under joint ownership with PETRONAS as well as agreement with neighboring countries such as Brunei and Vietnam to jointly develop once disputed areas.

A more recent development saw PETRONAS acquiring Canada's Progress Energy Resources Corp in a US$5.5 Billion deal. This deal enables PETRONAS to tap into the unconventional gas reserves in northeast British Columbia and northwest Alberta, as well as shale gas assets in Montney (Dittrick, 2012). Taking advantage of its geographical location, straddling crucial shipping lanes that connect the Pacific Ocean in the east to the Indian Ocean to the west, the government is also focusing in becoming a major regional storage and trading hub as well as utilizing existing local refineries to decrease the dependency on foreign distillates particularly from Singapore. According to the Oil and Gas Journal (OGJ), Malaysia's refining capacity is around 538,580 barrels per day from seven major refining facilities across Malaysia and it will be boosted by the completion of a direct linked pipeline from the fields to the refinery in Sabah by 2013, which will add 300,000 barrels per day to the capacity.

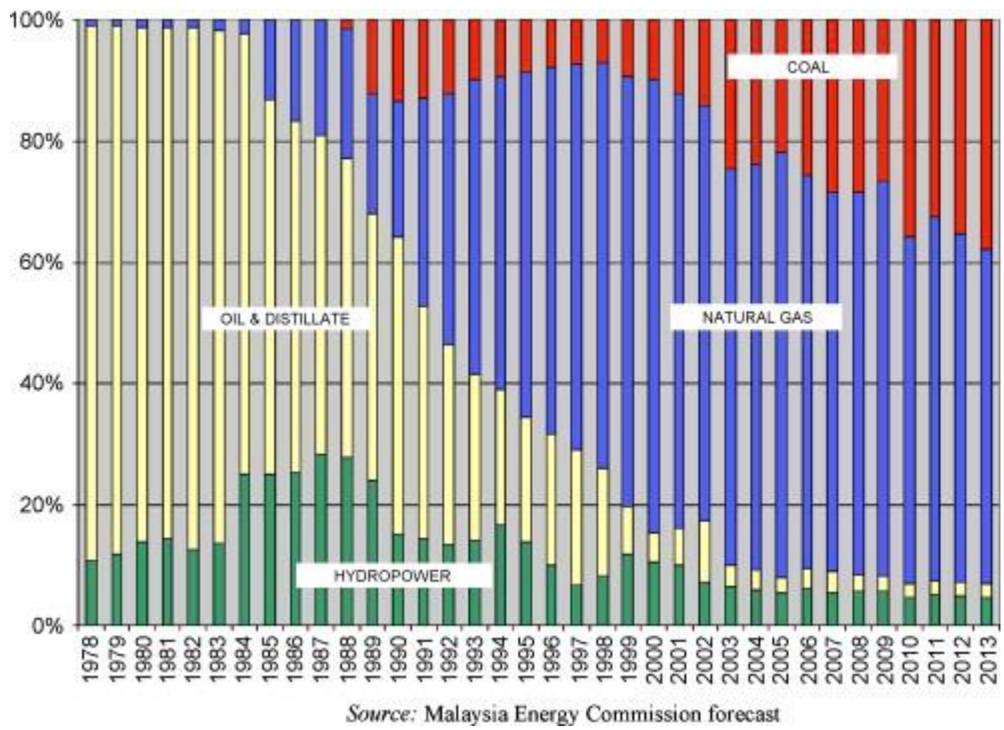

Figure 1. Energy mix trend in Malaysia.

In terms of natural gas reserves, Malaysia currently has 83 trillion cubic feet of proven gas reserves as of January 2011 (EIA, 2011). Unlike oil production, natural gas production has steadily increased in recent years. As seen in Figure 1, consumption of natural gas has increased from the year 1998 onwards in order to reduce the demand load on oil. Total production is currently around 2800 billion cubic feet with total consumption of around 1000 billion cubic feet (EIA, 2011). The most active areas of exploration and production are in the lower part of the Gulf of Thailand, where Malaysia and Thailand own equal percentages of the joint venture in operations. New oilfields off Sarawak in east Malaysia are also being developed with big investments from foreign companies such as Murphy Oil and Shell that would also add to the production capacity for at least the next 10 years. The Bintulu liquid natural gas (LNG) processing complex in East Malaysia is the largest in the world with a total liquefaction capacity of 22.7 million metric tons (1.1 trillion cubic feet) per year. Having this facility close to major wells in the region will be beneficial both in trade and also giving Malaysia an infrastructural edge when securing joint operations of oilfields with Thailand, Brunei and the Philippines.

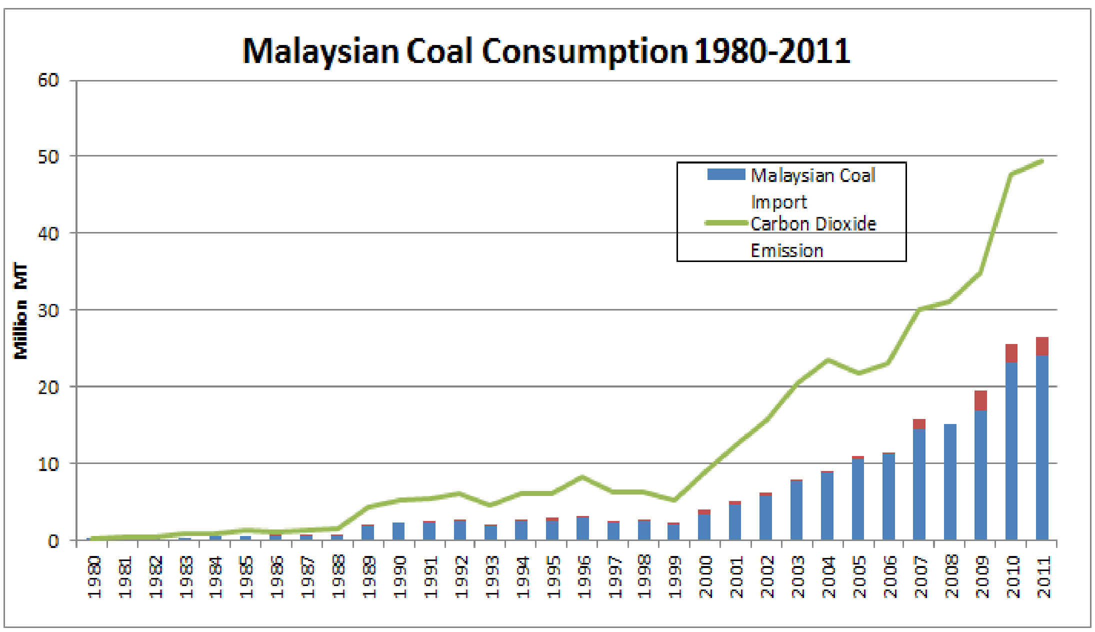

Figure 2. Malaysian coal consumption and carbon dioxide emission (Source: U.S. Energy Information Administration: www.eia.doe.gov).

Coal

Figures 1 and 2 also clearly reflect the policy change in terms of which resource is mainly utilized in the energy mix. Although still largely dominated by the utilization of oil and natural gas, since 1999, the policy has shifted into consuming less oil and increasing the usage of coal due to its relatively reliable worldwide reserve and stable price. Coal resource in Malaysia is relatively small and is estimated to be around 1724 million metric tons, of which 274 million metric tons are measured, 347 million tons indicated and 1102 million metric tons inferred (Thaddeus, 2002). 80% of these reserves are located in the interior regions of Sarawak in East Malaysia and are not easily accessible for current consumption. The demand is met by imports from Indonesia, Australia and China (Oh et al., 2010). Analyzing Figure 2, total coal consumption in 1980 to 2000 was about 32 million metric tons and this figure rose significantly in the following decade to about 142 million metric tons. In relation to the change in economic policies, this four-fold increase is due to the need to supply power generation plants as well as industrial sectors such as cement, iron and steel plants.

There are currently four coal-fired power plants in operation supplying about 7200 MW of power to the national electricity grid. As far as energy security is concerned, although there is considerable risk of relying on foreign supply of coal for around 40% of the total energy mix, these plants are supplied by long-term contracts with solid trade ties such as Indonesia and Australia. The Sarawak Corridor of Renewable Energy (SCORE) is a major development initiative to promote the development of the central region in the state where most of the coal reserve is located. Although the renewable energy targeted in this scheme is hydropower, this initiative seeks to capitalize on the abundance of natural resources especially coal and natural gas in the central region. These energy resources would then attract the influx of manufacturing industry to give the state the highest economic impact focusing on physical development, investment promotion and marketing planning (SCORE, 2011). However, this operation involves huge investments and interests from international entities.

Under the 10th Malaysian Plan (2011-2015), two 1000-MW coal plants in Tanjung Bin and Manjung are scheduled to be built and commissioned by 2016 (EPU, 2010). These plants will operate at supercritical levels which would increase steam cycle efficiency, reducing the amount of coal required thus lowering carbon dioxide and NOx emission per megawatt (Alstom, 2012).

Hydropower

Hydropower facilities in Malaysia are more than energy producing centers. Hydropower dams are also pivotal in socio-economic development such as irrigation to farmland, providing ample water supply especially during drought seasons, flood control during the monsoon season and improvement of crucial river navigation routes. Currently, hydropower is the only renewable energy that is commercially available on a large scale in Malaysia. The estimated hydropower resource and potential is around 29,000 MW (Leo-Moggie, 2002), with only 2,091 MW being utilized as of 2008 (NEB, 2009).

This low number is due to the very high capital investment and the issue of controversial encroachment into native land. However, through governmental policy such as SCORE, development of hydropower stations in Sarawak is projected to increase to 3500 MW by 2015 with the completion of the Bakun Dam and to 20 GW by 2030 (Aqua Media, 2010). The Bakun

Dam is one of the largest in South East Asia with a 207-m-high rock filled concrete dam creating a 70,000-hectare reservoir (roughly the size of Singapore). This project was downsized a few times and completion was delayed due to the Asian financial crisis and disputes with the native tribes on the building site. Despite these setbacks, the Bakun Dam will supply 2400 MW to the national grid and further projects are planned to begin upstream.

Renewable Energy Initiatives

Five-Fuel Diversification Policy and other energy policies

The Malaysian fuel diversification policy is reviewed on a regular basis to avoid dependency on a single source of energy. The latest version of this policy was in 1999 in which the Five-Fuel Diversification Policy was introduced to include renewable energy as the fifth fuel in the supply mix with the target to contribute 5% of the demand by 2005. Under the Third Outline Perspective Plan (OPP) for 2001-2010 and the 8th Malaysia Plan (2001-2005), the focus was to encourage the utilization of renewable resources such as biomass, solar and mini-hydroelectric stations to generate electricity. Under this plan, the Small Renewable Energy Power (SREP) program was launched to encourage private sectors to invest in small power generation projects utilizing biomass, biogas, mini-hydroelectric, solar and wind energy (Second National Communication to the UNFCC, 2010).

As of February 2010, a total of 43 projects were approved under SREP generating a capacity of roughly 290 MW of grid-connected power (Energy Commission, 2010). By 2005, the target of 5% was not met, where renewable energy only accounted for 0.3% of the total electricity supplied to the country. Relatively cheap fuel prices and low private investment into renewable energy initiatives were seen to be the main factors in this low number. Under the 9th Malaysia Plan, the establishment of the Ministry of Energy, Green Technology and Water to replace the Ministry of Energy, Communications and Multimedia was seen to reflect the government's drive to promote greater implementation of green technology and sustainability. This ministry is responsible in the planning and implementation of green policies and sustainable initiatives in Malaysia. In 2009, the National Green Policy was introduced with the objective of steering Malaysia towards energy independence and efficient utilization of natural resources with minimal impact to the environment.

The National Renewable Energy Policy was also introduced in 2010 as a step to rectify the failure of achieving the target of 5% renewable energy in the energy mix. This policy was introduced to enhance the utilization of indigenous renewable energy resources through increased contributions of renewable energy in the national power generation mix, to facilitate the growth of the renewable industry, ensuring reasonable renewable energy generation cost and to create public awareness of the importance of sustainability and clean technology (KETTHA, 2010). The impacts of this policy by 2020 are expected to be seen in a minimum of RM2.1 billion ($0.6 Billion) of external cost to mitigate CO_2 emissions (Total avoided 2011-2020 = 42 Mg x RM50/Mg), a minimum of RM19 billion ($6.3 billion) of loan values for renewable energy projects as new revenue sources for local banks, a minimum RM70 billion ($23 billion) of renewable energy business revenues generated that will subsequently generate RM1.75 billion ($0.5 billion) in tax to the government and a minimum of 50,000 jobs created to construct, operate and maintain these renewable energy power plants (Haris, 2010).

Under the 10th Malaysian Plan (2011-2015), providing reliable, high quality and cost effective energy is seen as a way to appeal to new investors thus enhancing the potential of the key development corridors. Here, the New Energy Policy (2011-2015) will focus on five strategic pillars to ensure energy security, economic efficiency as well as emphasis on improving environmental and social standards (EPU, 2010). Sustainability, diversification, flexibility and emission reduction are goals for the supply of energy that have been mapped out under the current plan. These goals can be achieved through development of renewable sources such as biomass, hydroelectric and biofuel (Ahmad, 2010).

Potential renewable energy sources in Malaysia are summarized in Table 1. Due to the geographical attributes of Malaysia, large rivers with suitable elevation encourage the development hydropower facilities. Being close to the equator also provides ample sunlight all year round that also makes solar power a viable option as a renewable energy source. Furthermore, being one of the world's largest exporters of palm oil also provides bountiful supply of biomass that can be utilized to produce both electricity and fuel. Although Malaysia ratified the Kyoto protocol in 1999, the country's status as a non-annex country does not bind the country to any CO_2 emission target. However, with the Five-Fuel Policy, the government is looking into the development of sustainable clean fuel to not only secure energy independence but also to maximize available natural resources.

Table 1. Renewable energy potential in Malaysia.

Renewable Energy	Potential (MW)
Hydropower	22,000
Mini-hydro	500
Biomass/biogas (oil palm mill waste)	1,300
Municipal solid waste	400
Solar PV	6,500
Wind	Low wind speed

Source: Malaysia Energy Centre's National Energy Balance.

Mini hydroelectric energy

Small-scale hydroelectric dams are attractive due to the relatively lower capital costs and maintenance. Smaller sized operations also reduce the environmental impact of clearing lands to build the dams and flooding forest areas for reservoirs. A mini hydroelectric dam can be defined as a facility that generates up to 10 MW of electricity. Currently, there are 26 approved applications under SREP with a total generation capacity of 102 MW and 97 MW grid connected capacity (KETTHA, 2009). Most of these projects are located in the interior regions of the two states in East Malaysia (Sabah and Sarawak), where total capacities are 8.3 MW and 7.3 MW respectively. The interior regions of Sabah and Sarawak are sparsely populated and some communities are not connected to any energy grids. An installed station in Long Lawen, Sarawak was very beneficial to the community as it eliminates use of 1000 gallons of diesel per year. An installation such as this benefits the communities that are located too far from established grid lines and at the same time provids a cleaner and more sustainable source of energy. The relatively high cost of building conventional hydroelectric stations will make this type of energy source more viable for these states and the targeted installed capacity from mini hydroelectric stations is 490 MW by 2020 (Khalib, 2010).

Solar

The daily average solar insulation in Malaysia is about 5.5 kWh/m^2 or 15 MJ/m^2 (Oh et al. 2010). Solar power in Malaysia through photovoltaic (PV) system is estimated to be four times the world fossil fuel resources (Kadir and Rafeeu, 2010). August and November are months with the highest solar radiation of 6.8 kWh/m^2, and the lowest is in December with 0.6 kWh/m^2. The states in the northern region of peninsular Malaysia and several places in East Malaysia have high solar radiation throughout the year. Similar to mini-hydroelectric stations, PV-powered systems can supply electricity to off the grid communities as well. Current application of PV systems is concentrated for domestic household uses to heat water only. Due to the very high cost to produce mass generation of electricity from PV systems and the fact that all the materials are imported from Japan, the commercial supply is not feasible yet. Currently, the majority of the PV heater system users are those from the higher income group, which is a very small percentage to the general population.

To generate more involvement from the private sector to invest in PV systems, in 2005, the government introduced the Malaysia Building Integrated Photovoltaic (MBIPV) project through funds from the United Nations Development Program (UNDP/GEF). The objective of the project is to lower the cost of systems through wider application of MBIV systems and the adoption of supportive regulatory frameworks to establish a sustainable BIPV market (Chen, 2009). After a five-year period, the cost of BIPV systems was reduced from RM28/kWp (kilowatt-peak) ($8.4/kWp) in 2005 to RM19/kWp ($6.1/kWp) in 2010 (Oh et al., 2010). The government also introduced SURIA 1000 in 2007 as a step to further develop the BIPV market, with special emphasis to provide the opportunity to the public and industrial sector to be directly involved in environmental and renewable energy initiatives. In this project, limited numbers of grid-connected PV systems are put up for auction, where the winning bidders will be allowed to install the PV system on their premises at a subsidized price. Under this program, a 5 kWp BIPV system costs about RM135,000 ($37,500) and to date the installations only totaled up to a marginal 0.4 MW of the total energy mix.

Biomass

The main focus of the National Energy Policy was to highlight the importance of shifting towards renewable resources that have the least amount of impact to the environment. Under these terms, biomass became a very attractive and viable option. Due to favorable climatic conditions and an economy that is still reliant on some form of mass agriculture, there is an abundant supply of feedstock ready for utilization. Malaysia's major agricultural crops are rubber (39.7%), oil palm (34.6%), rice (12.7%), cocoa (6.8%) and coconut (6.3%) from an estimated 4.9 million hectares (14.9% of Malaysian land mass) of agricultural land (Basiron, 2007). The main source of biomass will come from the palm oil and paddy plantations, which is mainly from post-harvest processing waste. Poh and Kong, 2002 estimated the amount of energy potential from biomass in Malaysia to be about 440 PJ/year (4.4×10^8 GJ) in 1996. From this number, the total biomass energy potential is about 90 PJ (9×10^7 GJ), of which 80% is from palm oil waste. Biomass from palm oil solid waste totaled to about 80 million tons (Mg) in 2010 and will rise to 100 million Mg by 2020 (MIGHT, 2011). Currently, the main use of palm oil biomass is in the form of cogeneration charcoal production, timber drying and electricity generation. Because of the large and constant supply of palm oil biomass as well as the mature logistical mechanism of the industry, palm oil biomass is seen as a very viable option to be utilized as feedstock in large-scale power generation (Sulaiman et al., 2011).

For the purpose of power generation, the oil palm industry generates the highest waste (59.8 million Mg), followed by paddy (2.14 million Mg) and sugar (1.11 million Mg) (UNDP, 2010). Palm oil wastes from post-harvest processing such as empty fruit bunches, fronds and trunks, fiber and palm kernels are currently being used directly as fuel for the boilers in the mills. Of the 63 projects approved under the SREP program, 25 projects are generators using palm oil based waste (Ludin et al., 2004). The government also introduced the BioGen project in 2002 with funds provided by the UNDP totaling to $14.7 million to promote and demonstrate the potential of utilizing these wastes in grid-connected power generation. Among the incentives given were favorable bank loans and tax exemptions. Currently, this initiative has generated a total of 700 MW of electricity for several oil mills around Malaysia (Mustafa et al., 2010). In 2006, Chubu Electric Power in Japan agreed to build two biomass plants in Sabah to generate 10 MW of electricity from palm oil waste. This project is also registered as a CDM (Clean Development Mechanism) project under the UN to generate CO_2 emission credits. The estimated reduction of CO_2 emissions from these two plants is expected to be about 2 million tons in 2012 (Chubu Electric Power, 2006). Recently, an agreement was signed with Envergent Technologies to convert palm oil biomass into renewable heat and electricity. This facility will be operational in 2013. Energy generation is not limited to palm oil biomass utilization exclusively and this trend is also seen in countries throughout South East Asia. Producers of wood and agricultural products such as sugar, rice and lumber in most of these countries have been relying on biomass to supplement their energy requirement. This is due to the large amount of residues generated, which is in the range of 20 to 70% of raw material input (Lacrosse and Shakya, 2004).

In this regard, fruit crops can also be viewed as a potential biomass resource stream. According to the latest Malaysian fruit crop statistic report (DOA, 2012), the total production for the year 2011 was 1.6 million Mg acquired from a total crop hectarage of about 230,000 ha. The Department of Agriculture (DOA) and the Federal Agricultural Marketing Authority (FAMA) are two state agencies that provide technical resource, marketing and logistical support to local fruit producers. Table 2 summarizes the top ten major fruit crops planted in Malaysia. In the top three of the ranking, durian and rambutan are seasonal fruits and are susceptible to market price fluctuations especially during supply glut. Value added product diversification is seen as a method to increase farmers’ income and overcome oversupply of fresh fruits. Conversion of waste stream from these post-harvest processing outlets into renewable energy or products is an attractive proposition, especially when this can also bring monetary benefits to the stakeholders. Although this amount is considerably smaller than the other major agricultural crops, the existing structure and mechanism is able to support bioconversion processing activities. DOA and FAMA serve as collection depots with stations across Malaysia and in some stations, processing of value added products is also carried out. Waste streams from both the processing facilities as well as culls from the marketing activities can easily be channeled to potential bioconversion facilities. Inline with the construction of newer and cleaner coal plants that are capable of co-firing with non-coal sources, biomass waste streams from these agricultural activities can be used to generate cleaner electricity and reduce coal imports as well.

Table 2. Summary of top ten major fruit crops (DOA, 2012).

Type of fruits	**Hectarage (ha)**	**Production (Mg)**
Durian	76,399	363,420
Banana (pisang)	29,132	306,283
Rambutan	18,650	63,345
Pineapple (nanas)	14,922	309,331
Watermelon (tembikai)	13,814	220,560
Dokong	10,934	107,334,330
Duku	9,921	35,113
Cempedak	7,894	32,749
Mango (manga)	7,176	21,022
Langsat	5,647	24,928
Total	194,489	108,711,081

Biofuels

The use of biofuels is relatively small in Malaysia. Although there were some efforts to include renewable energy source in electricity generation, renewable transportation fuel was not given any sort of priority despite the higher percentage in demand of transportation fuel over electricity generation (Lau et al., 2009) and the practice of heavily subsidizing fuel to the market. It wasn't until 2005 that the government introduced the National Biofuel Policy specifically to address the issue of using renewable biofuels. Under this policy, producing a 5% blend of biodiesel and providing incentives to the public for its use was the main agenda. In the same year, the government along with the Malaysian Palm Oil Research Institute introduced Envo Diesel, which is comprised of 95% diesel and 5% palm olein. This effort was ultimately unsuccessful due to very cheap oil prices and relatively high price of palm oil. There were also issues with the blend clogging up engines, low public interest and unclear policy and directive by the government (Oh et al., 2010). In an effort to improve its performance, the Envo Diesel was replaced with the B5, a palm based methyl ester variant in 2006 and the mandate for it to be used in all government diesel vehicles by February 2009. It was targeted that 70,000 Mg of this B5 diesel to be used by government vehicles annually.

Despite the advancement in second generation and advanced biofuel conversion techniques, and the high potential of utilizing the abundance of feedstock, there is currently no large-scale production of any type of biofuels in Malaysia. However, in 2005 a Dutch-based company, BTG-BTL, constructed a 2 Mg/h demonstration plant in Kluang, Johor to pyrolyze waste from palm oil mills or empty fruit bunch (EPB). The bio-oil produced is then co-fired in a waste disposal system to replace diesel (BTG-BTL, 2013). The potential of utilizing the various types of biomass as feedstock is very attractive and there are many conversion pathways available that can be tailored to suit the needs and the resources that are available in each region in Malaysia. Palm wastes in East Malaysia and some parts of the Peninsular can be utilized to produce bio-ethanol or drop-in fuels as well as using higher lignin compound of paddy waste for thermochemical conversion into bio-oils. In terms of biofuel production from palm oil biomass, production of useful sugars such as glucose and xylose from empty fruit bunch (EFB) is possible (Rahman et al, 2006; Lim et al., 1997). These sugars can then be converted into bio-ethanol and blended into regular gasoline.

The price of using an E10 blend was estimated to be RM1.37 / liter ($0.45 / liter) based on gasoline price of RM 1.35 / liter ($0.44 / liter) and the cost price of bioethanol at RM1.50 /

liter ($0.50 / liter) (DECP, 2005). Although oil prices have changed a lot since, the fact that Malaysia heavily subsidizes its fuel for public consumption makes blending with a slightly higher costing biofuel a feasible option to reduce its subsidizing bill.

Conclusions

Despite still being a net exporter of oil and with new oilfields being discovered and developed, the incentives for Malaysia to seek renewable and sustainable options are compelling. The annual cost of subsidizing fuel for public consumption amounts to about RM40 billion ($13 billion) in 2007 (Shuit et al., 2009). High fuel prices adversely affect food prices and other local macroeconomic elements and the government must seek better options in order to quell growing public discontent. Although ratifying the Kyoto accord as a non-annex country, there is still an obligation to reduce CO_2 emissions and to prepare its infrastructure for carbon trading with countries such as Japan, China and Australia. However, the overall future outlook of renewable energy seems uncertain, at least as far as governmental policies are concerned. While some policies seem to be very beneficial especially to the population in the interior regions, the overall outlook of these policies can be seen to have minimal impact. Sulaiman et al., 2011 highlighted the need for the government to take a more aggressive stance through incentives and encouraging joint efforts between governmental agencies and private institutions.

The current Malaysia plan (Tenth Malaysia Plan, 2011-2015) is targeting to increase renewable energy contribution to the electricity generation mix to 5.5%. This is a mere 0.5% increase from the previous plan and, looking closer at the mix, the decision to turn to coal when it is less environmental friendly seems to be a paradox against the objective of reducing emissions, as well as undermining energy security by relying on imported coal. The development of renewable resources in line with the guidelines of the current Malaysia plan can be seen as a step in the right direction, albeit a more aggressive stance in terms of developing potential renewable resources such as biomass. Implementation of biomass utilization methods can be facilitated by the existing framework within relevant governmental agencies such as FAMA, DOA, MARDI (Malaysian Agricultural Research and Development Institute) and PORIM (Palm Oil Research Institute Malaysia).

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CHAPTER 3. DETERMINATION OF A SUITABLE AIR OVEN MOISTURE CONTENT TEST METHOD FOR DURIAN WASTE

A paper to be submitted to *The Journal of Biomass and Bioenergy*

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Abstract

An accurate and reliable method of determining moisture content is integral to ensure viable and efficient thermochemical conversion of biomass. Utilization of new biomass waste streams such as durian biomass can be aided by a standardized method will also eliminate confusion and discrepancies. Existing oven-drying methods established for corn residues and other feedstock were compared with Karl Fisher moisture test. Clarification of the results was further explored by testing different drying temperatures, drying time and sample preparation methods. From the experiment, the HORT 2 method was found to be the easiest and simplest method with the highest accuracy. Depending on the process optimization requirements, the levels of the treatment factors can be adjusted to produce accurate results. Ground samples dried for 2 h (at 60°C, 80°C, 103°C, 135°C), whole rinds dried at 103°C (48 h) or 135°C (24 h) were found to most accurately measure moisture content.

Introduction

As prescribed in the New Energy Policy (2011-2015) under the 10th Malaysian Plan (2011-2015), identifying new energy feedstock is vital to the efforts of diversifying energy

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resources. Other than oil palm residues, fruit crop biomass has the potential to be utilized using the pathways established in the utilization of other biomass such as corn residues, hardwoods as well as palm oil residues. With total production of 1.6 million Mg from total crop hectarage of about 230,000 ha in 2011 (DOA, 2012), the role of fruit crop utilization is more in the view of socio-economic impact that goes beyond the fulfilment of national policy by potentially increasing the income of farmers and mitigating the effects of oversupply during peak/glut periods. Durian is one of the major fruit crops cultivated in Malaysia, and according to the latest consensus by the DOA (2012), total durian production amounts to about 360,000 Mg from a total plantation area of about 76,000 ha. With growing development of value added products, postharvest waste and residues are projected to increase and thereby benefit efforts of renewable energy utilization. Since the edible portion of the fruit only accounts for about 15-30% of the entire weight of the fruit (Brown, 1997), conversion of waste streams from these post-harvest processing outlets into renewable energy or products is an attractive proposition that can potentially bring monetary benefits to the stakeholders. In order to achieve this goal, understanding the physical and chemical characteristic of fruit crop waste is essential.

Moisture content of the feedstock or raw material is an integral attribute in any biomass utilization pathway, whether it is through thermochemical or biochemical conversion. Due to the heterogeneous nature of biomass, its physical characteristics including moisture content vary greatly. Moisture content affects the price, combustion optimization, storage management as well as the handling properties of a particular biomass (Mattsson, 1990; Evald and Jacobsen, 1993; Jensen et al., 2004; Jirjis, 2005). Kaltschmitt and Hein, 2005 also concluded that within an entire solid biofuel (biomass) supply chain, moisture content is a primary property that ensures successful utilization and this encompasses the transportation cost, storage management, calorific value and conversion at end use. Available moisture determination methods such as oven drying method and the Karl Fisher moisture test are commonly used for biological materials. However, both methods have unique relative benefits and disadvantages.

Previous studies on comparison and evaluation between methods of moisture determination have been carried out for rice (Chen, 2003), distillers dried grain with solubles (Thiex, 2009, Ileleji, 2010) and animal feed (Thiex and Richardson, 2003). Chen compared six oven methods and concluded that precise measurement is dependent on drying time and temperature, where adequate drying time and temperature are needed to drive off all moisture to give precise measurement. But at the same time high temperature can also drive off volatiles that may corrupt the results. Similarly, when comparing different oven methods with reference to the Karl Fisher test, Theix(2009), concluded that the procedure with the least variation to the reference indicated the best method to drive off the moisture and ensure none or very minimal volatile loss. Theix also found that the Karl Fisher test was the most accurate, however the labor (time and training) as well as the cost of instruments and reagents makes this method less accessible for implementation in industry. The Karl Fisher method should also be used as the primary method to evaluate all oven drying methods before they are applied for practice (Thiex and Richardson, 2003).

Therefore selection of a suitable method that is both reproducible and easy to implement is crucial especially for a non-established feedstock such as durian biomass. Due to the lack of a specified and standardized moisture determination test for durian as well as for other tropical fruit waste, identification of the best suitable method for durian can also be subsequently referenced and implemented for other fruit waste as well.

The objective of this experiment was to identify a suitable method for moisture content determination of durian waste (rind). This is done through two phases, where phase one is the comparison of existing methods of oven drying with reference to a Karl Fischer method. Phase two consists of the evaluation of moisture content measurement through drying at different temperatures and drying times. The results from this drying test are also compared to a reference Karl Fisher test.

Materials and methods

Material

Due to the unavailability of raw Malaysian durian in the United States, frozen durian fruit imported from Thailand were obtained from the local Asian market in Ames, Iowa. The specific name of the durian cultivar used in this experiment is *Mon Thong* or Golden Pillow (*Durio zibethinus* Murray). These fruits were harvested before maturity and post-harvest ripening was carried out in storage at room temperature. Once ripe, the fruits were frozen at -40°C (-40°F). Then each fruit was individually placed in plastic netting and packed in corrugated carton boxes. Each box weighs about 12 kg (25 lb) and contains four to five fruits. The fruits were refrigerated at -18°C (0°F) during transport and storage. Figure 3 illustrates the structural form of the rind and its cross section. The thorns on the rind are significantly harder and studier compared to the moist and soft inner section.

The materials used in this experiment were rinds collected from the fruits. The frozen fruits were thawed at room temperature for at least 12 hours before separation of the edible pulp and the rind was carried out. Separation was done manually using a sturdy cleaver or knife to pry open the fruit. Once open, the pulp was scooped out by hand and the rinds were then collected and stored in an -18°C (0°F) freezer and thawed before use. The rinds were then cut into 25-mm (1-inch) cubes or ground using a stone mortar.

Figure 3. Cross section of durian rind displaying the different structure and characteristics of the thorns and the inner section.

Moisture content methods comparison

Eight oven drying methods were chosen from a list of methods commonly used for biological materials and biofuels. These methods were distinctly different from each other and only air oven methods were chosen due to their frequent use in the industry. Table 3 summarizes the characteristics of the methods. Aluminum dishes were used for all methods except HORT 1, HORT 2 and KF, according to the respective prescribed procedures. The forced-convection oven (Heratherm 51028115H, 60L, 120 VAC 60 Hz, Thermo Fisher Scientific Inc, USA) was preheated at least two hours before the start of each experiment. Sample weights were measured on one of the two digital balances; Denver Instrument DI-4K (0.001g, Denver Instrument, Bohemia, NY) and Adventurer Pro AV114 (0.0001g, Ohaus Corporation, NJ) according to the required precision.

Table 3. Oven drying methods in use for biological and biofuel materials.

Method / Standard	ID	Procedure and conditions	Materials prescribed for the standard
Horticulture 1[a]	HORT 1	Whole rind piece in brown bag, dried in air oven 60°C, 24 h	Flowers, fruits
Horticulture 2[a]	HORT 2	Whole rind piece in brown bag, dried in air oven 60°C, 48 h	Flowers, fruits
ASABE/ASAE S358.2[b]	ASABE 1	25-mm (1-inch) cube, dried in air oven 103°C, 24 h	Forages
ASABE/ASAE S358.2[b]	ASABE 2	25-mm (1-inch) cube, dried in air oven 55°C, 72 h	Forages
BS EN 14774-2 (Simplified)[c]	BS 1	25-mm (1-inch) cube, dried in air oven 105°C, 48 h	Solid biofuels
BS EN 14774-3 (Oven dry method)[d]	BS 2	Ground samples dried in air oven 105°C, 2 h	Solid biofuels
NFTA 2.2.2.5[e]	NFTA	Ground samples dried in air oven 103°C, 3 h	Dry matter forages
AOAC 930.15[f]	AOAC	Ground samples dried in air oven 135°C, 2 h	Feeds
AOCS Ca 2e-84[g]	KF		Karl Fischer Reagent

[a] Epstein, 2005; Chapman and Pratt, 1961
[b] ASABE Standards S358.3: 2012. Moisture measurements for forages. American Society of Agricultural and Biological Engineers, St. Joseph, MI
[c] BS EN 14774-2 (Simplified): 2004. Solid biofuels - Determination of moisture content - Oven dry method - Part 2: Total moisture - Simplified method. European Committee for Standardization (CEN), Brussels
[d] BS EN 14774-3 (Oven dry method): 2004. Solid biofuels - Determination of moisture content - Oven dry method - Part 3: Moisture in general analysis sample. European Committee for Standardization (CEN), Brussels
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[f] Official Methods of Analysis of AOAC INTERNATIONAL (2000) 18th Ed., AOAC INTERNATIONAL, Gaithersburg, MD, USA, Official Method 930.15
[g] AOCS Ca 2e-84: 2009. Moisture Karl Fischer Reagent. Official methods of the American Oil Chemists' Society

Temperature, drying time and form experiment

The effects of temperature, time and sample form were tested and evaluated to obtain better definition of these effects on moisture content and to quantify their accuracy compared to the Karl Fisher test. The different levels for each independent variable were chosen based on values commonly found in other methods. Four levels of temperature (60°C, 80°C, 103°C, 135°C), three levels of drying times (2 h, 24 h, 48 h) and three sample preparation methods (whole, cubed, ground) were chosen in this experiment with three replications each. The oven and digital scale used in the previous setup were also used in this experiment.

Karl Fischer Moisture Test

Samples for the reference moisture content were sent to the University of Missouri Agricultural Experiment Station Chemical Laboratories (ESCL) for testing. The AOCS Ca 2e-84 method (AOCS, 2009) was used to determine the moisture content using Karl Fischer reagent.

Statistical Analysis

For the method comparison experiment, each method was replicated three times and one-way analysis of variance (ANOVA) was used to determine if the methods tested were statistically different. Analyses were performed in SAS v9.2 (SAS Institute, Cary, NC) using PROC GLM procedure and the different methods were then compared using Tukey's Studentized Range. Karl Fischer method was used as the reference or standard to measure the accuracy of each oven drying method. Accuracy is quantified by the percent of deviation from the Karl Fischer method and this is calculated using the formula below (Rosentrater and Muthukumarappan, 2006):

$$Percent\ Deviation, \% = \frac{MC_{Oven} - MC_{Karl\ Fischer}}{MC_{Karl\ Fischer}} \times 100$$

The coefficient of variation, CV was calculated for each method to measure the repeatability of the procedure. The coefficient of variation for each method is calculated by the following formula:

$$Coefficient\ of\ Variation, CV\ (\%) = \frac{Standard\ Deviation}{Mean} \times 100$$

Statistical analysis for the temperature, time and form experiment was also done on SAS v9.2 (SAS Institute, Cary, NC). A three-way ANOVA procedure was used to test effects of temperature, time, and form, as well as the respective interaction of the main effects.

Results and Discussion

Moisture content methods comparison

The results for the comparison of the nine methods used to determine moisture content are summarized in Table 4 and Figure 4. Overall, the highest measured moisture content was 81.5% (ASABE 1) and the lowest was 57.7% (HORT 1). The coefficient of variation for all methods was well below 5%, which indicated that the methods provide good repeatability. The results also indicate that moisture content measured using HORT 2 was found to have the least percent deviation (0.062%) compared to KF, whereas HORT 1 was the most different with the highest percent deviation of -22.87. While other methods required drying temperatures of at least 103°C, the drying temperature for HORT 1 and HORT 2 were set at 60°C. The significantly smaller percent deviation for HORT 2 indicates that low temperature drying is possible but must be done for a prolonged period, since the drying time for HORT 2 was 24 hours as opposed to 48 hours for HORT 1. Overall, five of the methods compared were found to be within less than 5% deviation from KF and all methods except HORT 1 were found to be within less than 10% deviation from KF.

When comparing the different methods based on Tukey's test, ASABE 2, BS 2, AOAC, and KF were found to not be significantly different and the moisture contents measured were close to those of the standard KF values. Since these three methods were carried out at different temperatures, drying times and at different forms, the need to further this experiment to test and further define the effects of these parameters on moisture content becomes important and is carried out in the next section. Furthermore, the similarity of almost all of the methods tested indicates that structural differences in the rind, with its hard thorns, and the soft and moist inner part does not impact the measurement of moisture content since methods that required drying of whole rind and ground rind produced similar results.

Table 4. Summary of results for the comparison of different moisture content measurement methods.

Method	% Moisture Content Mean	Standard Deviation	CV, %
HORT 1	57.7[a]	2.3	3.9
HORT 2	74.8[c]	1.3	1.8
ASABE 1	81.5[b]	1.9	2.4
ASABE 2	77.7[b,c]	2.2	2.8
BS 1	79.8[c]	0.7	0.9
BS 2	74.3[b,c]	0.3	0.4
NFTA	74.3[c]	0.3	0.4
AOAC	78.2[b,c]	0.6	0.8
KF	74.8[b,c]	2.7	3.6

[a,b,c] Mean with the same letters are not significantly different at $P \geq 0.05$

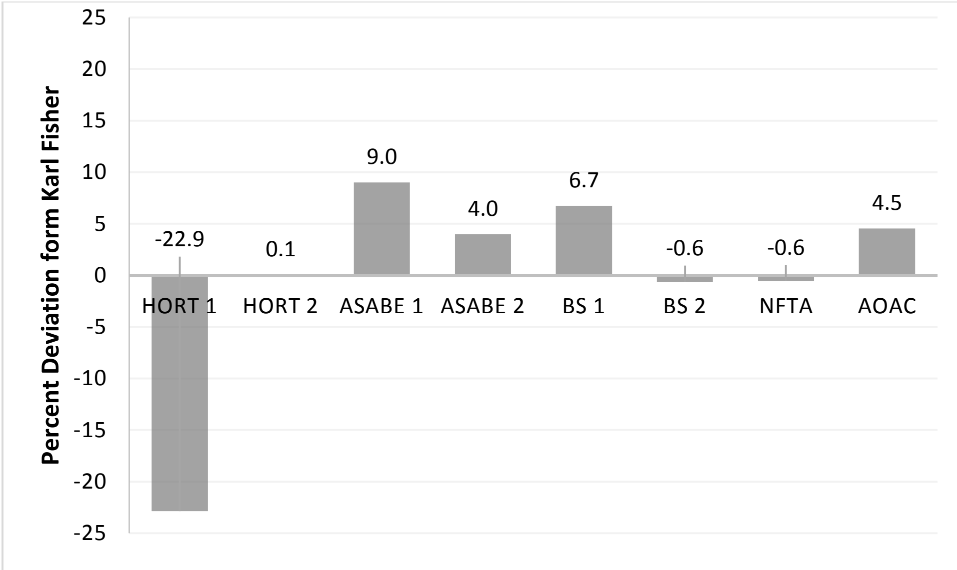

Figure 4. Percent deviation of moisture content for each method compared to Karl Fischer (KF).

Temperature, drying time and form experiment

Analysis on SAS indicated significant interactions of all three treatments factors. The results of the analysis are summarized in Table 5. Since the three-way interaction was significant, Bruin (2006) proposed the comparison of all the levels of one factor and fixing the other two factors. Summary of this analysis is as tabulated in Table 6 and is restricted to display only the significant interactions. For the interaction of all factors over all levels of size, significant interactions were observed for all drying temperatures at two hours indicating high variability and less accuracy in comparison with KF moisture content measurement. In terms of the levels of time, the lack of significant interaction for ground samples indicates that drying time was a factor influencing the results for whole and cubed samples at certain temperatures. . Significant interaction for only two combinations of factors indicates that the effect of drying temperature was not as apparent as the other factors. The permutations of this analysis can be seen graphically in the following charts.

Table 5. ANOVA table output for the analysis of the three main effects.

Source	DF	Type III SS	Mean Square	F Value	Pr > F
Temp	3	319	106	7.24	0.0003
Time	2	22136	11068	753.15	<.0001
Temp*Time	6	830	13	9.41	<.0001
Size	2	8814	4407	299.89	<.0001
Temp*Size	6	430	72	4.88	0.0003
Time*Size	4	15006	3751	255.28	<.0001
Temp*Time*Size	12	1079	90	6.12	<.0001

Table 6. Test of two factors over every level of the other factor.

Temp*Time*Size Effect Sliced by Temp*Time

Temp	Time	DF	Sum of Squares	Mean Square	F Value	Pr > F
60C	24h	2	385	192	13.08	<.0001
60C	2h	2	7822	3911	266.12	<.0001
80C	2h	2	6570	3285	223.53	<.0001
103C	2h	2	5590	2795	190.19	<.0001
130C	2h	2	4819	2409	163.95	<.0001

Temp*Time*Size Effect Sliced by Temp*Size

Temp	Size	DF	Sum of Squares	Mean Square	F Value	Pr > F
60C	Cube	2	5033	2516	171.23	<.0001
60C	Whole	2	7902	3951	268.86	<.0001
80C	Cube	2	2753	1376	93.66	<.0001
80C	Whole	2	7691	3845	261.68	<.0001
103C	Cube	2	2248	1124	76.5	<.0001
103C	Whole	2	6898	3449	234.71	<.0001
130C	Whole	2	6203	3101	211.03	<.0001

Temp*Time*Size Effect Sliced by Time*Size

Time	Size	DF	Sum of Squares	Mean Square	F Value	Pr > F
2H	Cube	3	1801	600	40.85	<.0001
24H	Whole	3	437	146	9.91	<.0001

Figure 5 summarizes the results obtained from this experiment and displays the different distribution of the results based on the type of sample preparation that was carried out. Evidently from the boxplot in Figure 5, the ground samples were found to have less variation for all drying times (2, 24, 48 hours) across the different drying temperatures (60^{o}C, 80^{o}C, 103^{o}C, 135^{o}C). The range of the means was 3% moisture content, where the lowest mean was 76% moisture content and the highest was 80%.

Conversely, the distribution for cubed and whole were significantly more dispersed compared to the ground samples. For the cubed samples, the range of the means was calculated to be 14% moisture content. For whole samples, the range was 11% moisture content. Overall, based on the observation and comparison of the mean moisture contents between the three boxplots, whole samples have the tendency to under-dry hence the lower means of moisture content observed as compared to slight over-drying observed for the comparatively higher collection of moisture content means for the ground samples. Since the cubed samples also displayed the characteristics of under-drying which is undesirable, for all drying times at temperatures below 103^{o}C, the ground samples were observed to be consistently close to the KF measurement of 74.8% moisture content.

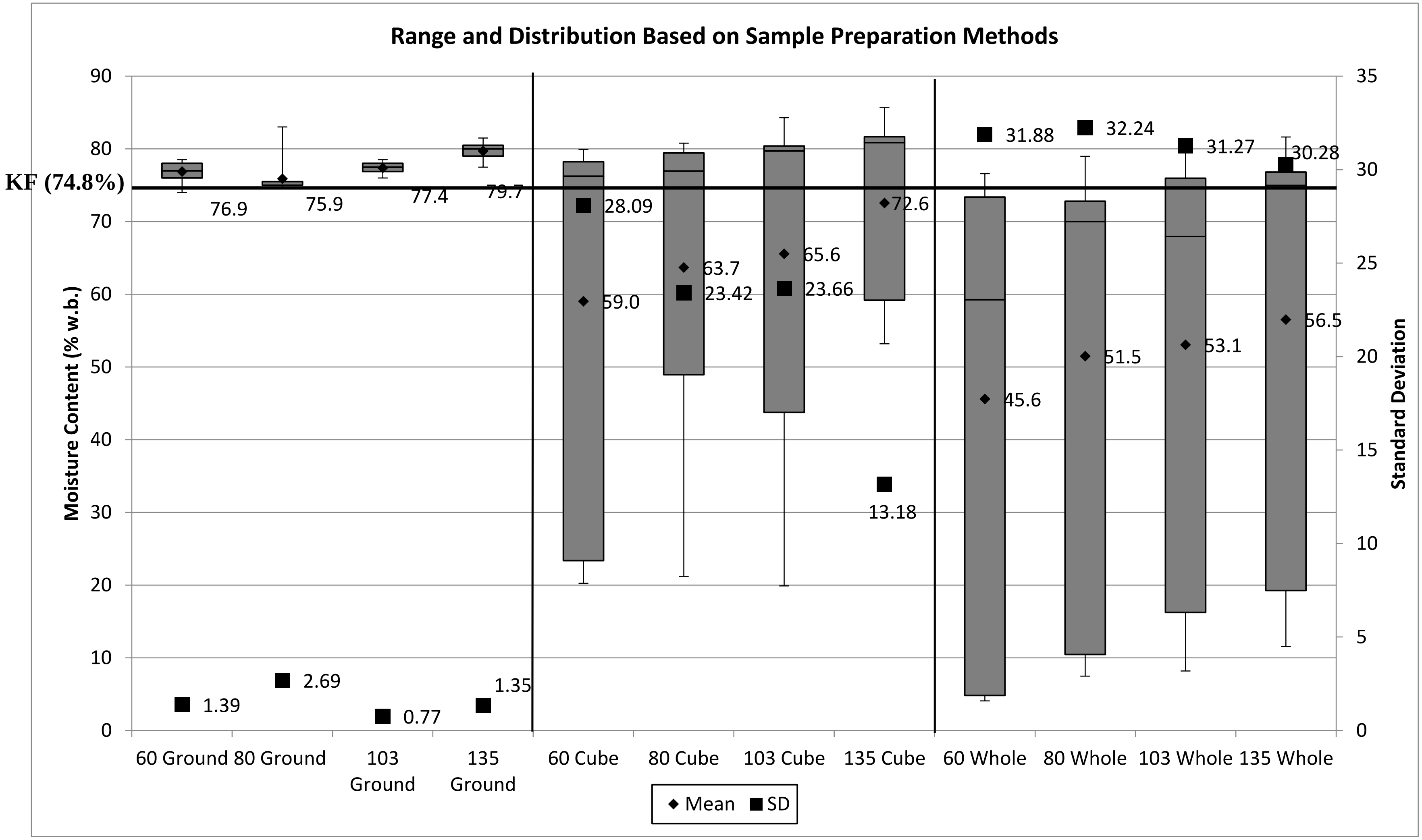

Figure 5. Boxplots of the range and distribution of moisture content observed for different sample preparation types across all drying times.

When comparing the distribution of moisture content with respect to different drying time considering all sample preparation types, the observed moisture content offers further insight into the influence of the different treatments used in the experiment. From Figure 6, assessing the results of 2-hour drying time, the means for all three sample preparation types vary greatly where the highest calculated mean was 76% moisture content for 2-hour ground samples and 10% moisture content for whole samples. The range was calculated to be 66% moisture content. For the 24-hour drying time, the observed variation was less than the 2-hour drying set with a range of 11% moisture content between the highest mean of 80% moisture content and lowest mean of 69% moisture content. The 48-hour drying time was found to be the most consistent where the range was calculated to be 3% moisture content.

Based on these observations, a 48-hour time period allows the samples to dry sufficiently with the caveat of slight over-drying the samples as seen in the upper end of the boxplot whiskers exceeding 80% moisture content for all sample preparation types. For the 24-hour set, except for the whole samples, the results observed for ground and cubed samples were almost equal to the previous set, indicating that whole samples require longer time for sufficient drying to occur or certain drying temperatures for it to sufficiently dry the samples within a 24-hour timeframe. Evidently, all ground samples were comparatively consistent for all drying times. However, the higher moisture content observed in the 24-hour and 48-hour set indicates that higher temperatures may cause some over-drying.

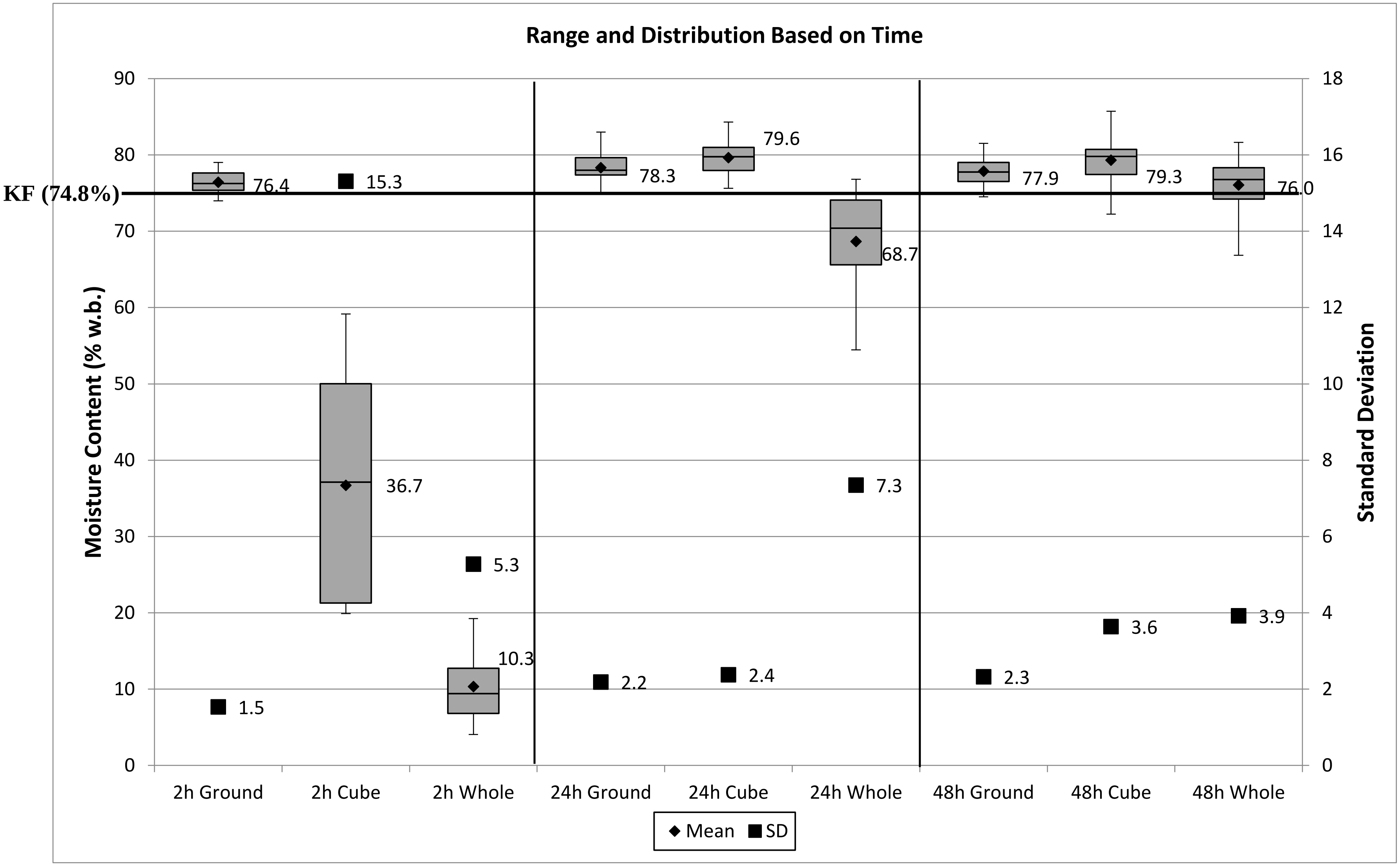

Figure 6. Summary of range and distribution of moisture content across all drying temperature for different drying times.

In terms of the percent deviation of the moisture content measured compared to the results measured by KF test, further details of the influence of the treatment levels can be seen more clearly. Referring to Figure 7, although the ground samples seemed to be the best option based on the observations made from Figure 2 and 3, drying at 135^{o}C for 24 hours or more resulted in a comparatively higher percent deviation compared to other drying temperatures and times. Cubed samples were observed to have very big deviation for drying time of 2 hours regardless of the temperature and the least deviation at 60^{o}C and 80^{o}C for drying times of more than 24 hours. Drying at 135^{o}C for more than 24 hours resulted in higher percent deviation compared to 103^{o}C for drying time more than 24 hours. Similarly, whole samples also have very big deviations for 2-hour drying periods across all drying temperatures. However, percent deviation was found to be reduced significantly for drying temperatures of 103^{o}C and 135^{o}C for more than 24 hours of drying time. Significantly, percent deviation was the least for the combination of 103^{o}C for 48 hours and 135^{o}C for 48 hours.

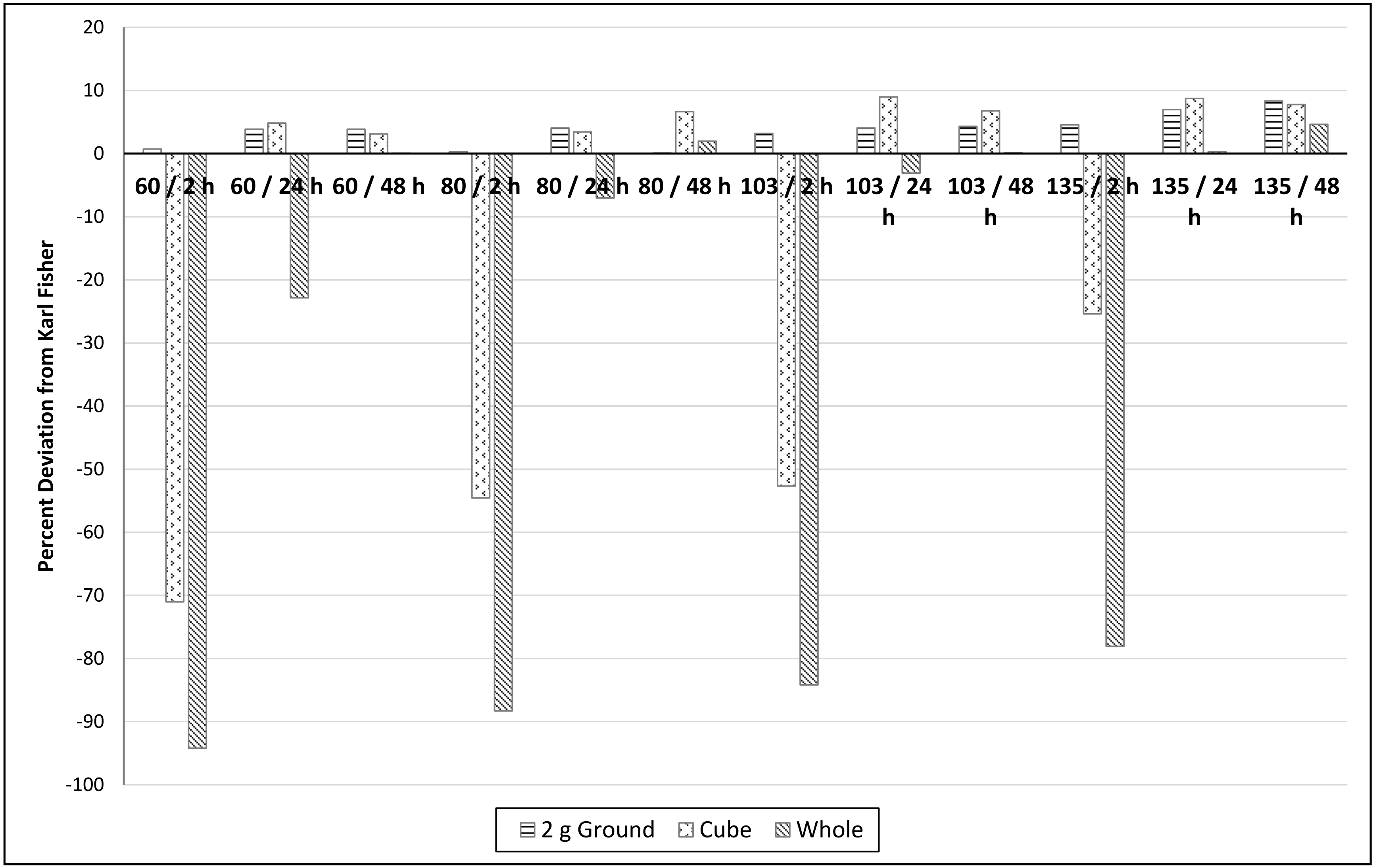

Figure 7. Percent deviation for all observed treatment levels.

Conclusions

HORT 2, BS 2, and NAFTA were found to be the best recommended methods that are capable of producing high accuracy of moisture content determination. These methods produced high reproducibility and the least percent deviation from the standard KF method. Comparison of these three methods indicates that HORT 2 method can serve as the easiest and simplest of the three and should be given precedence.

Because whole and cubed samples achieved similar accuracy as ground samples, suspicion of the thorns projecting different drying attributes compared to the inner parts can be rejected. Thus, with adequate drying time and temperature, the whole or cubed samples can be used to simplify processing and handling of the rind during testing.

Based on statistical and graphical analyses, interaction of all three factors was found to be significantly influencing the measurement of moisture content. Ground samples were found to be more robust in producing accurate readings at different drying times and temperatures. Depending on the process optimization needs, ground samples dried for two hours (at 60°C, 80°C, 103°C, 135°C) can be applied for high turn-around labs, or if handling of the tough rind is an issue, then the use of whole rind dried at 103°C for 48 hours or 135°C for 24 hours can be applied instead.

Acknowledgement

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CHAPTER 4. PHYSICAL AND CHEMICAL CHARACTERIZATION OF DURIAN WASTE FOR THERMOCHEMICAL CONVERSION APPLICATIONS

A paper to be submitted to *The Journal of Biomass and Bioenergy*

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Abstract

Heterogeneity of the physical and chemical characteristics of biomass is seen as a major challenge for wider application and utilization of biomass resources. Understanding the characteristics and make-up of the many biomass resources in Malaysia is vital in order to fully harness its potential. Existing techniques used in the physical and chemical characterization of biomass were applied to durian biomass. Low ash, sulfur and nitrogen content as well as comparable calorific value found in durian biomass makes it an attractive option for co-firing with coal. For pyrolysis applications, the high oxygen content found may result in unfavorable products and discrepancies found in lignocellulosic fraction tests indicates further testing is needed. Carboxylic acids and phenols were identified as the two highest compound groups.

Introduction

Sustainability, diversification, flexibility and emission reduction are key aspects of the New Energy Policy (2011-2015) under the 10th Malaysian Plan (2011-2015). The focus of this policy is to ensure energy security, economic efficiency and improving environmental and social standards (EPU, 2010). Development of renewable sources such as biomass, hydroelectric and biofuel is seen as a vital building block to achieve these targets (Ahmad, 2010).

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Utilization of biomass resources is very attractive and is currently spearheaded by utilization in cogeneration plants such as those under the state organized Small Renewable Energy Power program (SREP) (Ludin et al., 2004) as well as joint state-private initiatives such as BioGen and Chubu Electric Power. At this moment, palm oil biomass is a major contributor to these initiatives, where total available palm oil biomass as of 2010 is estimated to be about 87 million Mg, which is equivalent to a total potential energy of 1554GJ (KeTTHA, 2011). By 2020, biomass is targeted to achieve at least 800 MW of electricity generation capacity, of which 500 MW is derived from palm oil biomass (KeTTHA, 2011). This can pave the way for adoption of other sources of biomass, where it can take advantage of the infrastructure and management structure established through palm oil biomass utilization.

Agricultural products such as sugar, rice and fruits generate high waste-to-product ratios. In South East Asia, most lumber and agricultural practices generate residues in the range of 20 to 70% of raw material input ((Lacrosse and Shakya, 2004). The high waste-to-edible portion ratio of fruits like durian, where the edible portion is only about 15-30% (Brown, 1997) of the total fruit weight, creates a large waste stream especially in post-harvest processing centers. Moreover, due to the climacteric nature of most tropical fruits such as durian, mangosteen, jackfruit and mango, ripening of the fruit occurs rapidly after harvest thus decreasing its edible and economic value in a short selling window. Coupled with the seasonal nature of these fruits, oversupply or glut of fruit production severely decreases farmers' income and creates waste problems. Therefore, utilization of fruit biomass is essential to not only regulate fruit prices through diversification of products derived from these fruits and its waste, but also reduce environmental issues of waste disposal and utilizing its potential as a renewable energy feedstock.

Total Malaysian fruit production for the year 2011 was 1.6 million Mg from a total crop area of about 230,000 ha (DOA, 2012). Durian, banana and rambutan were the top three major fruit crops in year 2011, with productions of 360,000 Mg, 306,000 Mg and 63,000 Mg respectively (DOA, 2012). The Department of Agriculture (DOA) and the Federal Agricultural Marketing Authority (FAMA) are two state agencies that provide technical resource, marketing and logistical support to local fruit producers. Both agencies have many operation hubs throughout Malaysia and most of their activities including collection and distribution of commodities and most importantly dissemination of information and knowledge are channeled through these facilities. Since availability and logistical constraints are two of the major challenges to the development of biorefineries (Caputo et al., 2005), this problem can be potentially alleviated through this existing network. Furthermore collection of waste as renewable feedstock can be easily implemented in the same network as well.

Conversion of fruit biomass can be done through thermochemical processing routes other than combustion through cogeneration with coal in an electricity generation plant. Thermochemical processing can be summarily defined as the conversion of plant polymers in biomass into fuels, chemicals, or electric power through the application of heat and catalysts. There are five main thermochemical processing routes: Combustion, gasification, fast pyrolysis, hydrothermal processing, and hydrolysis to sugar, where each is classified by its processing temperatures and applications (Brown, 2011).

Fast pyrolysis is the thermal decomposition of biomass in the absence of oxygen at temperatures typically around 400-600°C that involves very high heating rates (0.5-2s) followed by rapid cooling and condensation of the vapor products. Fast pyrolysis yields gas, solid and liquid product streams that distinguish it from other thermochemical methods such as combustion and gasification. Biomass composition as well as heating rate and heating duration influence the product distribution and the ratio between gas, liquid and solid produced. The liquid stream or bio-oil is of particular interest due to its potential as a feedstock for conversion into drop-in or advanced biofuels through bio-oil upgrading (Mohan et al., 2006; Czernik et al., 2002; Elliott, 2007; Marker et al., 2005). Furthermore, as a form of liquid feedstock, bio-oil has the potential to improve energy density and ease of transportation thus improving the overall efficiency and viability of a biorefinery.

The high oxygen content in most plant carbohydrate polymers makes biomass significantly different from conventional energy sources in terms of its chemical composition. The main constituents of biomass are celluloses, hemicelluloses, lignin, organic extractives and inorganic minerals, where the weight percent of each components are unique to each type of biomass (Mohan et al., 2006). Compared to information available on other contemporary feedstock such as corn residues, perennial grasses and softwoods, thermochemical studies on durian or other tropical fruit biomass is almost non-existent. With regards to other potential feedstock in Malaysia, there have been a number of pyrolytic studies done on palm oil (Abdullah and Gerhouser, 2008; Abdullah et al., 2010) and these studies have found comparable results of bio-oil yields from palm oil empty fruit bunches (EFB).

Chemical composition is highly critical in understanding the thermal degradation characteristics of lignocellulosic materials in biomass (Antal, and Varhegyi, 1995; Liou et al., 1997). Furthermore, chemical composition also affects the pyrolysis process where high nitrogen and ash concentration negatively affect hydrocarbon yields (Sanderson et al., 1996). The objective of this paper is to meet the overarching need to identify and characterize these traits that will ensure further analysis of the material such as yield prediction, mathematical models, process optimization and economic analysis can be carried out. This will eventually lead to the ability to streamline the process making it more efficient and feasible so that it can supplant conventional fuel and energy sources. Investigation of the energy content available is also crucial for thermal and electrical applications, where it is normally reported in a standardized heating value (HV) (Vargas-Moreno et al. 2012).

Therefore, for tropical fruit biomass like durian rind to be considered as feedstock, it is imperative that its physical and chemical characteristics first be established and compared with other potential feedstock before other analysis can proceed. Due to the scarcity of material available, bench-scale pyrolysis was not possible. However, preliminary pyrolysis product identification can be ascertained through a micropyrolyzer-mass spectrometer system that will allow identification of monosaccharides and phenolic monomers that can be used as a basis for understanding the thermal degradation of durian biomass.

Materials and methods

Material

Frozen *Mon Thong* or Golden Pillow (*Durio zibethinus Murray*) durians imported from Thailand were used in this experiment. The fruits were purchased from a local Asian market in Ames, Iowa where the fruits were refrigerated at -18°C (0°F) during transport and storage. It is sold in 12-kg (25-lb) boxes containing about four to five fruits. Each fruit was individually placed in plastic nettings and packed in corrugated carton boxes.

The materials used in this experiment were the rinds collected from the fruits. The frozen fruits were thawed at room temperature for at least 12 h before separation of the edible pulp and the rind was done. Each fruit was cleaned by brushing its surface to remove dirt or other foreign contaminants. Separation was done manually using a sturdy cleaver or knife to pry open the fruit. Once open, the pulp was scooped out by hand and the rinds were then dried at 60°C for 48 hours in a forced air oven (Heratherm 51028115H, 60L, 120 VAC 60 Hz, Thermo Fisher Scientific Inc, USA). The dried rinds were then ground using a knifemill (Heavy-Duty Cutting Mill SM 2000, Retsch GmbH) fitted with a 5- mm particle size screen. Samples were sent to the University of Missouri Agricultural Experiment Station Chemical Laboratories (ESCL) to determine its pre-drying moisture content. Moisture content was determined using the Karl Fischer reagent test (AOCS Ca 2e-84).

Characterization of durian biomass

Proximate analysis was done using a thermogravimetric analyzer (Mettler-Toledo, International Inc., Columbus, OH, USA) following the ASTM D7582 - 12 method (ASTM, 2012). Ultimate analysis was done using a TRUSPEC-CHN elemental analyzer (TruSpec Micro CHNS, LECO Corporation, St. Joseph, MI, USA). Oxygen content was calculated as a difference in relation to the other elements. Metals and inorganic component analysis were performed at University of Missouri Agricultural Experiment Station Chemical Laboratories (ESCL) using Inductively Coupled Plasma - Optical Emission Spectroscopy (ICP-OES) (AOAC Official Method 985.01). Samples were also sent to this lab to determine the lignocellulosic content of the rind, where acid detergent fiber (ADF: AOAC 973.18), lignin (ADL: AOAC 973.18) and neutral detergent fiber (NDF: JAOAC 56, 1352-1356) were determined to calculate fraction of cellulose, hemicellulose and lignin. Samples were analyzed in duplicates.

Higher heating value (HHV) was estimated using correlation developed by Sheng and Azevedo (2005) for different biomass fuels that is based on the ultimate analysis of the material. This correlation was found to provide the highest accuracy and the estimation of HHV is done using the composition of the main elements of the proximate analysis: carbon (C), hydrogen (H) and oxygen (O). The correlation used is as follows;

$$HHV = -1.3675 + 0.3137[C] + 0.7009[H] + 0.0318[O]$$ (Sheng and Azevedo, 2005)

<u>Analytical pyrolysis</u>

A Frontier Lab Double Shot Micropyrolyzer 20202iS (Frontier Laboratories Ltd, Japan) coupled to a Varian 450 GC (Agilent Technologies, Inc. Santa Clara, CA, USA) was used in the pyrolysis experiment and product identification. Sample weights in the range of 250 ± 5 mg were used and pyrolysis tests were performed at temperatures of 500^{o}C. Product identification was done using the software accompanying the mass-spectrometer. The peaks were compared to those from the National Institute of Standards and Technology (NIST) mass spectral data library in order to determine its identity.

Results and discussion

Proximate and ultimate analysis

Table 7 summarizes the results of the proximate and ultimate analyses done on the durian samples. Values for corn residues and other fruit processing wastes are also listed for comparison. In general, the properties of durian biomass are comparable to other biomass, with regards to low ash content and almost similar volatile content and fixed carbon. Similar to palm oil biomass, untreated durian waste has high moisture content and significant amount of drying must be done to reduce the moisture content to less than 50% for most thermal conversion (Mckendry, 2002). High moisture content in biomass may influence its pyrolytic behavior as well as the physical properties and quality of the bio-oil. Therefore depending on the intended aim of the process to either generate more bio-oil or char, the initial moisture content is a crucial factor (Demirbas, 2004).

Furthermore, high moisture content of biomass can also cause ignition and combustion problems, as well as flame stability during combustion. In electrical generation, this issue can be solved by co-firing of biomass with coal (Demirbas (2), 2004). Therefore, sufficient drying must be done prior to thermal conversion and due to climatic conditions in Malaysia; this can be done by drying the biomass in the sun for a specific time. The values of volatile content and fixed carbon serve as an indication of how well the biomass can be utilized as energy source. These values measure the reactivity or how easily it can undergo thermal degradation (McKendry, 2002; Vamvuka et al., 2011). Comparing the volatile contents of durian and the other biomass shows that it is more thermally reactive than palm EFB but less reactive compared to some other biomass.

Table 7. Proximate and ultimate analyses of durian rind and other biomass samples, in comparison with coal.

Proximate analysis (% d.b.)								
	Durian	Palm EFB[a]	Apple jus residue[b]	Orange juice residue[b]	Sugarcane bagasse[b]	Corn stover[c,d]	Corn Cobs[c,d]	Bituminous Coal[e]
Moisture content	74.8[K.F.]	67.0	8.5	8.5	9.2	10.6	12.1	11
Ash content	4.1	7.7	2.3	3.3	0.69	3.7	1.0	9
Volatile content	83.0	79.6	85.4	76.5	82.6	78.7	86.5	35
Fixed carbon	17.0	20.4	14.6	23.6	17.4	17.6	12.5	45
Ultimate analysis (% d.b.)								
C	42.6	45.0	53.4	44.9	45.0	46.6	47.4	73.1
H	6.32	6.4	7.9	7.1	6.1	5.0	5.9	5.5
N	0.59	0.25	1.8	1.4	0.3	0.8	0.7	1.4
S	0.07	1.06	<0.01	0.1	<0.01	0.2	0.2	1.7
O	50.45	47.3	36.85	46.3	48.6	40.1	38.1	8.7
O:C ratio	1.2	1.05	0.69	1.03	1.08	0.86	0.80	0.12
H:C ratio	0.15	0.14	0.15	0.16	0.14	0.11	0.12	0.08

[K.F.] Moisture content determined through Karl Fischer method
[a] Omar et al. 2011; Ma and Basiron, 2005
[b] Virmond et al., 2012
[c] Demirbas, 1996
[d] Mullen et al., 2010
[e] McKendry, 2002

In terms of the ultimate analysis results comparison, as with other biomass, durian samples contain relatively higher oxygen content compared to coal. The other components such carbon, hydrogen, nitrogen and sulfur are comparable to other biomass. Most crucially it has very low levels of sulfur and nitrogen compared to most other biomass. These two components are important factors in the formation of pollutants such as NO_x and SO_x, as well as the occurrence of fouling and slagging in combustion equipment (Jenkins et al., 1998). The O:C and H:C ratios can be used to evaluate the energy content in biomass. The higher proportion of oxygen and hydrogen relative to carbon indicates less energy available due to the lower energy contained in carbon-oxygen and carbon-hydrogen bonds, as compared to carbon-carbon bonds (McKendry, 2002). This relationship can be illustrated using a Van Krevelen diagram (Figure 8) that shows increasing heating value moving diagonally upwards from right to left.

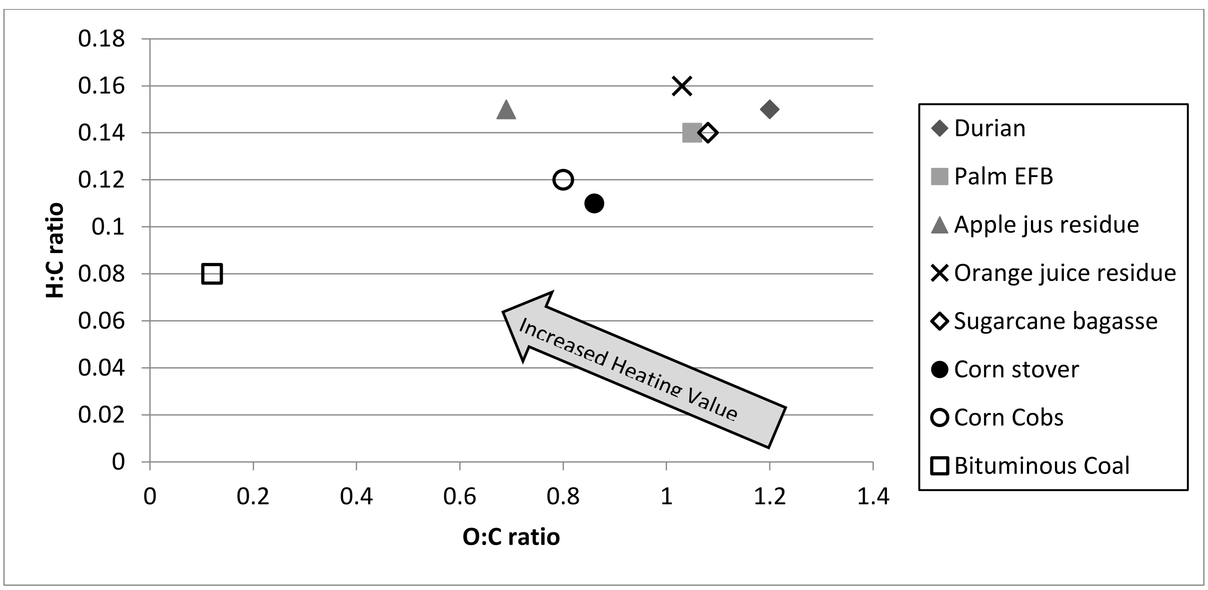

Figure 8. Van Krevelen diagram adapted from McKendry (2002) illustrating the relationship between carbon, hydrogen and oxygen atomic ratios and heating value for various solid fuels.

The Van Krevelen diagram in Figure 8 that was plotted from the O:C, and H:C ratios summarized in Table 7, the relationship between both ratios and the magnitude of heating values can be seen. Durian samples have the highest O:C ratio (1.2) and an average H:C ratio (0.12) compared to other biomass and coal, and from Figure 8, it can be seen that its heating value is the lowest among the others mainly due to its high O:C ratio. Interestingly, this value is not far off from palm EFB, which is being used extensively in boilers and co-firing plants in Malaysia. For co-firing application, determination of suitable fraction of durian biomass and coal is essential in order to account for the high O:C ratio.

Thermal decomposition of durian exhibits different rates of degradation. There are three distinct decomposition zones in relation to the peaks of the derivative thermogravimetry curve as seen in Figure 9. The three zones relate to moisture evolution, degradation of hemicellulose-cellulose and degradation of lignin. For pyrolysis of biomass, hemicellulose degradation occurs at temperatures below 350°C, cellulose degrades between 250°C and 500°C, while degradation of lignin was observed at temperatures above 400°C (Bo et al., 2009; Parthasarathy and Narayanan, 2013). The TGA profile (Figure 9) of the durian samples seems to follow this convention. The first peak around 30-103°C marks the first zone and this corresponds to the evolution of moisture and light volatile compounds in the sample. This is also an indication of the moisture in the sample (2.6% w.b.) that is left in the sample after drying the sample at 55°C for 48 hours. The second zone can be seen between 200°C and 350°C where the two peaks indicates the degradation of hemicellulose-cellulose. The lignin degradation zone can be seen to occur at 538°C. The final peak at 914°C refers to the combustion of remaining materials to produce ash.

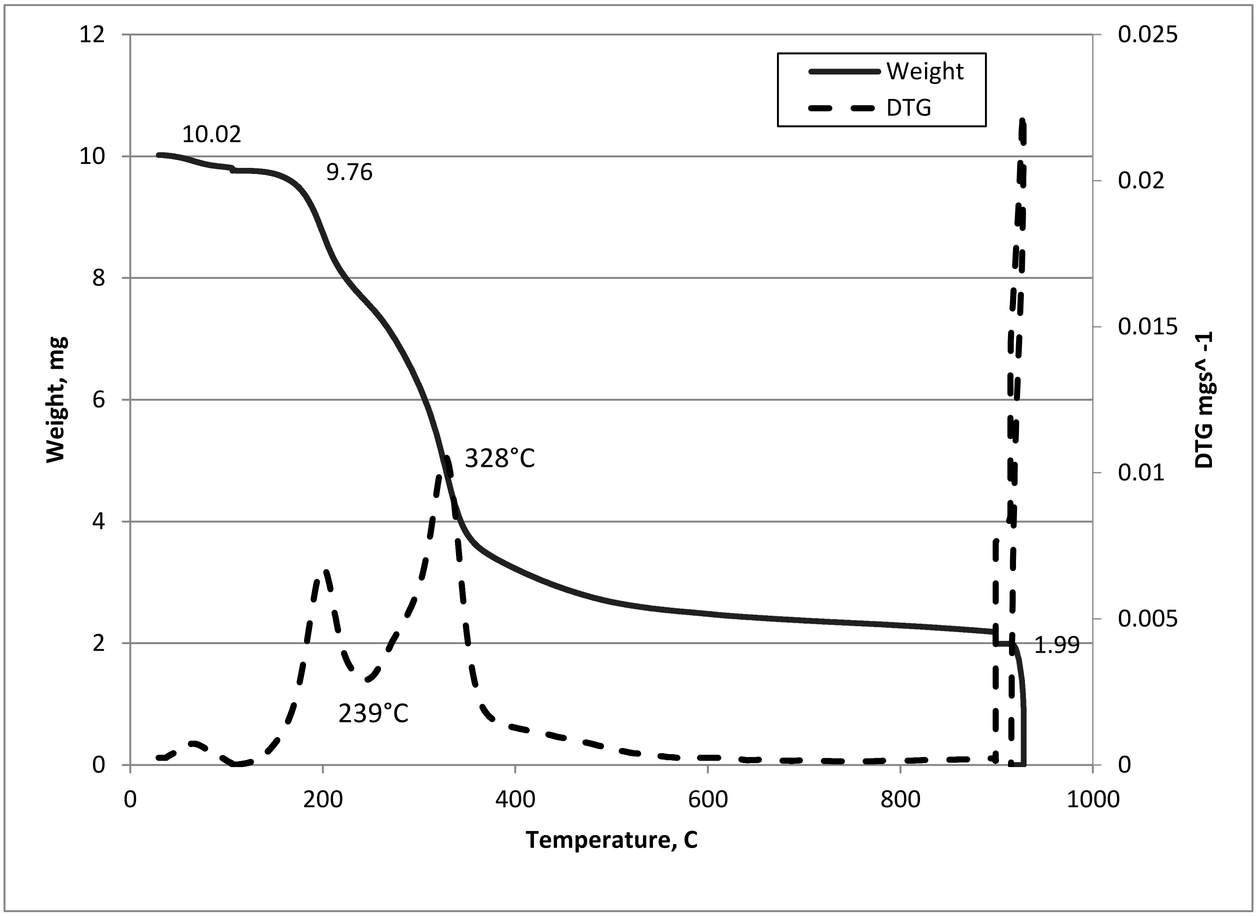

Figure 9. Thermogravimetric analysis (TGA) and its derivative curve (DTG) for durian rind samples dried at 55°C

The profile also indicates three other vital features of the burning profile. The thermal reactivity of the sample is normally represented by its peak burning temperature. The ignition temperature for the durian sample was recorded at 239°C (512K) and the peak temperature was observed to be 328°C (601K). These numbers are comparable to those observed for pine cones, colza seed and cotton refuse as summarized in Table 8 (Haykırı-Acma, 2003). The maximum combustion rate for durian was found to be substantially lower than the other biomass. The higher ignition temperature, peak temperature and maximum combustion rate indicates that the durian samples have fewer components that are easily combusted, thus making it less reactive (Vamvuka et al., 2011).

Table 8. Comparison of burning characteristics of durian sample with other biomass (Haykırı-Acma, 2003).

Sample	Ignition temperature (K)	Maximum combustion rate (mg/min)	Peak temperature (K)
Durian	512	0.66	601
Sunflower shell	417	5.50	573
Colza seed	423	2.80	535
Pine cone	463	5.20	565
Cotton refuse	423	3.70	598
Olive refuse	438	3.40	537

Lignocellulosic analysis and heating value

The total lignocellulosic fraction of the rind amounts to about 40% of the total dry weight. Based on the method used to determine this fraction (AOAC 973.18), the digestible fraction or the extract contains proteins, starch, sugars, organic acids and pectin that are soluble in a neutral detergent. The remaining lignocellulosic compounds are retained in the raffinate. The low value of the lignocellulosic fraction in the durian samples (Table 9) indicates that it has higher fraction of digestible components more suitably used as feed. Although Hokputsa et al. (2004) found that durian rind contains high amounts of pectin, these numbers are lower compared to those reported by Khedari et al. (2004). This discrepancy may be due to the frozen durian used in this experiment, as compared to fresh durian used by Khedari et al., Our durian samples underwent several freeze-thaw cycles and this occurrence is presumed to cause significant changes in the structure of the cell walls due to ice formation (Burke et al., 1976), thus some water-soluble cellulose might have leached out when the fruit thawed, resulting in the skewed data. When comparing the available result and the one reported by Khedari et al., the hemi-cellulose and lignin contents are low compared to corn residues.

Referring to the heating values presented in Table 9, the calculated heating values for durian samples are comparable to those of other agricultural post-harvest biomass. It is almost similar to palm EFB and higher than corn residues, but it is almost half the heating value of bituminous coal. However, these values are well within the range of energy content found in biomass, which is between 17 and 21 MJ/kg (Mckendry, 2002).

Table 9. Summary of lignocellulosic components and heating value of durian rind and other biomass.

	Lignocellulosic (% d.b.)								
	Durian	Durian[j]	Palm EFB[a]	Apple juice residue[h]	Orange juice residue[i]	Sugar-cane bagasse[d]	Corn stover[c]	Corn Cobs[c]	Bitu-minous Coal[g]
Cellulose	10.3	60.5	23.7	7.2	9.2	45.4	51.2	52.0	n.d
Hemi-cellulose	19.2	13.1	21.6	n.d.	10.5	7.8	30.7	32.0	n.d
Lignin	8.8	15.5	29.2	23.5	0.84	23.4	14.4	15.0	n.d
	Higher heating value, HHV($MJkg^{-1}$)								
	18.0[e]	n.d.	19.0[f]	22.1[b]	15.3[b]	15.9	17.8	17.0	34

[a] Omar et al. 2011; Ma and Basiron, 2005
[b] Virmond et al., 2012
[c] Demirbas, 1996
[d] Pereira et al., 2011
[e] Sheng and Azevedo, 2005
[f] Yang et al., 2004
[g] McKendry, 2002
[h] Dhillon et al., 2012
[i] Rivas et al., 2008
[j] Khedari et al., 2004
n.d. (not determined)

Inorganic analysis

The composition of alkali and alkaline earth metallic species in biomass is of great concern particularly in thermochemical conversion of biomass. Significant amounts of elements such as K, Na, Mg and Ca may affect reactor operations or may inversely affect economic feasibility of the conversion process (Mohan et al., 2006). Williams and Horne (1992) also found that metal salts can cause lower temperatures during thermal degradation and increased production of char at concentrations of more than 1%. Based on the results tabulated below (Table 10) the amounts of K, Na, Mg and Ca for durian samples are low compared to most biomass. It has considerably lower metal salts compared to palm EFB. These metals are also catalytically active and can cause secondary cracking reactions that might result in loss of yield and phase separation of the bio-oil produced (Brown, 2011). The total ash content of this sample (4 % weight, d.b.) is slightly higher than the theoretical limiting value of 2.5 % (weight, d.b.) ash content that can negatively affect the catalytic processes (Brown, 2011). Washing and pretreatment of durian biomass with dilute acid can feasibly remove ash content and improve processing capabilities.

Heavy metal concentrations are also negligible and this is preferred considering the effects of having dangerous heavy metal compounds being released in the product streams and possibly causing significant health and environmental hazards.

Table 10. ICP-OES analysis of common inorganic elements in durian biomass.

W/W%= grams per 100 grams of sample in dry basis	**Durian**	**Palm EFB**[a]	**Apple juice residue**[b]	**Orange juice residue**[b]	**Sugarcane bagasse**[b]	**Corn stover**[c]	**Corn Cobs**[c]
Calcium	0.2	6.5	13.4	22.2	6.2	0.02	0.3
Phosphorus	0.2	1.1	18.5	10.7	6.9	0.1	0.2
Sodium	0.001	1.3	0.9	0.3	0.5	0.01	.02
Magnesium	0.2	n.d.	9.0	6.3	8.0	0.06	0.2
Copper	0.001	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Potassium	1.5	12.8	31.3	31.6	23.4	1.0	0.4
Iron	0.002	1.3	5.9	2.9	0.5	0.008	0.2
Zinc)	0.001	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Manganese	0.001	n.d.	0.2	0.1	0.1	0.001	0.09
Chromium	0.0001	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cobalt	0.00002	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Molybdenum	0.00003	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Silicon as SiO2	0.05	49.1	6.6	3.2	48.7	0.5	2.8
Barium	0.001	n.d.	n.d.	n.d.	n.d.	0.01	0.002
Thallium	0.00001	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Vanadium	0.00001	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Titanium	0.00002	n.d.	2.5	0.2	0.2	0.0003	0.04
Antimony	0.000003	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Arsenic	0.0002	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Aluminum	0.0012	0.5	n.d.	n.d.	n.d.	0.02	0.5
Nickel	0.00002	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Mercury	0.00001	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cadmium	0.0001	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Lead	0.0001	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

[a] Omar et al. 2011 [b] Virmond et al., 2012 [c] Mullen et al., 2010
n.d. (not determined)

Analytical pyrolysis

Characterization of pyrolysis product is essential in predicting the characteristics of bio-oil produced from the pyrolysis of durian biomass. Bio-oil is a dense liquid product of pyrolysis that can increase the overall energy density of the biomass as well as having a potential application in upgrading processes to produce biofuels or biochemicals (Brown, 2011). Tables 11 and 12 summarize the broad approximation and identification of products that can be derived from pyrolysis of durian samples at 500°C. Based on the results, carboxylic acids and phenols were found to be the top two highest compound groups. High percentage of acids produced is also expected due to the high oxygen content of the biomass. Removal of organic acids such as acetic acid in bio-oil is essential in avoiding corrosion of metals and storage instability (Agblevor, 1995; Diebold and Bridgwater, 2002). Phenols can also be extracted to produce high value products such as resins and adhesives. Ether soluble furans and ketones were the next two highest compound groups. It was interesting to note the lack of the highly valued carbohydrate derivatives, specifically levoglucosan in the list of compounds found. This is expected due to the low percentage of carbohydrate in the biomass.

Table 11. Compounds identified from the volatiles generated by durian biomass pyrolysis.

Compounds	**Formula**	**Area (%)**
Acids		
Acetic acid	C2H4O2	9.599
n-Hexadecanoic acid	C16H32O2	5.672
Propanoic acid, 2-oxo-, methyl ester	C4H6O3	5.516
Tetradecanoic acid	C14H28O2	1.681
Pentadecanoic acid	C15H30O2	1.002
Alcohols		
Cyclopropyl carbinol	C4H8O	4.907
Ketones		
2-Cyclopenten-1-one, 2-hydroxy-	C5H6O2	4.836
3',5'-Dimethoxyacetophenone	C10H12O3	2.602
1,2-Cyclopentanedione, 3-methyl-	C6H8O2	2.394
2-Propanone, 1-(acetyloxy)-	C5H8O3	1.749
Furans		
2-Furancarboxaldehyde, 5-(hydroxymethyl)-	C6H6O3	5.086
Furfural	C5H4O2	4.343
2(5H)-Furanone	C4H4O2	1.747
2-Furanmethanol	C5H6O2	1.217

Table 11continued

Phenols		
Phenol, 2,6-dimethoxy-	C8H10O3	3.945
2-Methoxy-4-vinylphenol	C9H10O2	2.355
Phenol, 2-methoxy-	C7H8O2	1.983
Phenol, 2,6-dimethoxy-4-(2-propenyl)-	C11H14O3	1.938
Phenol, 2-methoxy-4-methyl-	C8H10O2	1.458
Phenol, 2-methoxy-4-(1-propenyl)-	C10H12O2	1.444
Phenol	C6H6O	1.168
Phenol, 3-methyl-	C7H8O	1.085
Nitrogen Compounds		
Cyclobutylamine	C4H9N	4.98
Aldehydes		
Undecanal	C11H22O	2.839
Nonanal	C9H18O	1.78
Butanedial	C4H6O2	1.503
Esters		
1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester	C16H22O4	0.988
Aromatics		
1,2,4-Trimethoxybenzene	C9H12O3	1.326
Misc. Oxygenates		
4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl-	C6H8O4	1.702
5,9-Dodecadien-2-one, 6,10-dimethyl-, (E,E))-	C14H24O	1.235
2,4-Imidazolidinedione, 5-methyl-	C4H6N2O2	1.167
2-Methyliminoperhydro-1,3-oxazine	C5H10N2O	1.02
Tricyclo[2.2.1.0(2,6)]heptan-3-ol, 4,5,5-trimethyl-	C10H16O	0.911

Table 12. Summary of compound group fraction derived from durian biomass pyrolysis.

Compounds	Total area (%)
Acids	23.5
Phenols	15.4
Furans	12.4
Ketones	11.6
Aldehydes	6.1
Misc. Oxygenates	6.0
Nitrogen Compounds	5.0
Alcohols	4.9
Aromatics	1.3
Esters	1.0

Conclusions

Based on the characterization results, durian biomass was found to be similar to other biomass such as corn residues in terms of its chemical characteristics. The high moisture content of post-harvest durian biomass is seen as a major stumbling block for thermochemical conversion. However, there is potential of sun-drying the material due to the favorable climate in Malaysia.

If the issue of high moisture content can be resolved, durian biomass is an attractive feedstock option due to its low ash and comparable calorific value, especially compared to palm EFB which is widely used in Malaysia. This feature is attractive in the application of this material in the production of electricity through co-firing with coal. High ignition temperature, peak temperature and maximum combustion rate indicates that the durian samples have fewer components that are easily combusted, making it less combustible and thermally reactive.

For pyrolysis applications, there are still some uncertainties in terms of its high oxygen content and the discrepancies with other published results on the fraction of lignocellulosic compounds. More detailed analysis must be done to study the pyrolysis properties of durian biomass which include, larger scaled pyrolysis testing and determining the yield and properties of bio-oil and char produced. Based on available results, carboxylic acids and phenols were found to be the two highest compounds identified from the micropyrolyzing experiment. Future studies can also include identifying suitable pretreatment processes to improve its processing parameters, yield and overall economic viability.

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CHAPTER 5. COMPOSTING OF DURIAN RIND FOR GREENHOUSE MEDIA APPLICATION

A paper to be submitted to *The Journal of Bioreseource Technology*

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Abstract

Composting is a viable, simple and comparatively inexpensive method of waste disposal. Greenhouse media application for compost is an attractive option that can replace peat and also provide necessary organic matter and nutrients as well as containing plant growth regulators and pathogenic inhibitors for optimal plant growth. Weight change, CO_2 production rate and temperature profiles were observed for different levels of C:N ratios and sample preparation methods. Size of composting volume affected the temperature profile, where thermophilic heat rate was not achieved. The combination of 30:1 and 50:1 ratios, with Fitz and knife mill preparation methods seemed to produce better composting conditions as well as producing better quality compost.

Introduction

With agricultural activities being a major economic driver in Malaysia, there are sizable amounts of biomass from various sources that can potentially be utilized for the generation of energy, fuel or other usable form. Malaysia's major agricultural crops are rubber (39.7%), oil palm (34.6%), rice (12.7%), cocoa (6.8%) and coconut (6.3%) from an estimated 4.9 million

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hectares (14.9% of Malaysian land mass) of agricultural land (Basiron, 2007). These practices generate residues in the range of 20 to 70% of the raw material input (Lacrosse and Shakya, 2004) indicating the vast amount of biomass that would otherwise be discarded and pose environmental issues. Crucially, open burning of agricultural residues is a prevalent practice among South East Asian countries, which leads to the yearly-occurrence of smog and haze across the region and resulting in decreased air quality (Afroz et al., 2003).

Tropical fruits play an important role in the nation's agroindustry with durian being the most highly valued commodity. Total Malaysian fruit production for the year 2011 was 1.6 million Mg from a total crop hectarage of about 230,000 ha (DOA, 2012). Durian, banana and rambutan are the top three major fruit crops in year 2011, with productions of 360,000 Mg, 306,000 Mg and 63,000 Mg respectively (DOA, 2012). Due to its seasonal and climacteric nature, tropical fruits like durian face seasonal glut issues that cause oversupply and substantial loss of income to growers. This creates large volumes of waste from unsold and over-ripened durians and improper disposal results in environmental issues. Despite increased diversification of value added products derived from these fruits, post-harvest processing also creates a considerable waste stream as only 15-30% of the fruit is edible (Brown, 1997). Therefore, identifying a cost-effective way of managing this waste or preferably finding useful applications of this waste stream is paramount to the national agenda.

Composting is a viable, simple and comparatively inexpensive method of waste disposal. Composting can replace landfilling and incineration of waste by transforming organic matter into a stable product through a biological oxidative process that naturally occurs in soil (Bertoldi et al., 1983). By definition, it is the exothermic biodegradation of organic waste involving a complex interaction of biochemical reactions and microbial catabolism (Paul and Clark, 1996). Here, carbonaceous substrates or organic matter in the waste materials are hydrolyzed and oxidized to produce carbon dioxide and heat, and subsequently conversion into compost. In this stable and mature form, compost is widely applied in agricultural activities such as agronomic and horticultural crops in both field and greenhouse settings, forest soil amendment (silvicultural), and land reclamation applications. (Shiralipour et al., 1992). More recently, compost is also evaluated for its use as highway embankment erosion control (Glanville et. al, 2004; Bakr et al., 2012) and as a treatment measure for bio-security purposes in response to large scale emergency livestock mortality (Glanville and Trampel, 1997; Glanville et al., 2006).

Its application as growing media as a substitute for peat is an attractive proposition due to increasing cost and the inherent environmental impact of peat extraction (Herrera et al., 2008; Benito et al., 2005). Compost as growing media also benefits the plant where it provides the necessary organic matter and nutrients as well as containing plant growth regulators and pathogenic inhibitors (Atiyeh et al., 2002; Tilston et al., 2002).

Composting is a mature process and this is reflected in the vast amount of prior studies done on a multitude of subjects that range from municipal solid waste to kitchen wastes, and animal carcasses. However, to the best of the author’s knowledge, composting study on tropical fruit waste or durian biomass has not been done. In order to capitalize on the potential of durian biomass and tropical fruit waste in general, ideally it is vital to understand its composting characteristics. Therefore, this study will evaluate key composting characteristics such as temperature profiles and carbon dioxide (CO_2) production rates observed during the composting of durian rind at different levels of carbon and nitrogen (C:N) ratios and sample preparation methods. The compost products will then be evaluated through relevant chemical and physical tests to gauge its viability for greenhouse media applications.

Materials and methods

Material

Frozen *Mon Thong* or Golden Pillow (*Durio zibethinus* Murray) durians were used in this experiment. The fruits were purchased from a local Asian market in Ames, Iowa. These fruits were harvested before maturity and post-harvest ripening was carried out in storage at room temperature. Once ripe, the fruits were frozen at -40°C (-40°F). Then each fruit was individually placed in plastic netting and packed in corrugated carton boxes. Each box weighs about 12 kg (25 lb) and contains four to five fruits. The fruits were refrigerated at -18°C (0°F) during transport and storage.

The materials used in this experiment were the rinds collected from the fruits. The frozen fruits were thawed at room temperature for at least 12 hours before separation of the edible pulp and the rind was done. The fruits were cleaned by brushing their surfaces to remove dirt or other foreign contaminants. Separation was done manually using a sturdy cleaver or knife to pry open the fruit. The collected rinds were then randomly divided into three portions. Each portion was then processed to reduce its size according to its randomly assigned sample preparation method. The samples were then poured into one-gallon round wide-mouth corrosion resistant HDPE jars (S-17077, Uline, Pleasant Prairie, WI), where each jar was filled with approximately 800g of durian rind samples.

Ultimate analysis was done using a TRUSPEC-CHN elemental analyzer (TruSpec Micro CHNS, LECO Corporation, St. Joseph, MI, USA) to determine the C:N ratio of the durian sample, which was found to be 75:1. Moisture content of the durian rind was determined by oven drying at 60°C for 48 hours. Moisture content was found to be 65% (w.b.).

Experimental set-up

The C:N ratio is an essential factor in composting. By influencing microbial cell growth and functioning, the process is slowed down if nitrogen is a limiting factor and loss of nitrogen in the form of ammonia or other compound will occur if the nitrogen content is in excess. Depending on the material, optimum range of C:N ratio is reported to be around 25-40 (Golueke, 1991). Addition of manure or food scraps are usual sources of nitrogen in common compost processing. For this particular experiment, in order to simulate conditions in Malaysia and eliminate possible variation in the characteristics of manure sourced locally compared to those available in Malaysia, urea is chosen due to is ubiquity and explicit nitrogen content that will aid replication in Malaysia and calculation of C:N ratios. The different C:N ratios will enable the observation of the effect of different nitrogen content on the compost process. The experiment was designed to test three types of sample preparation methods and three C:N ratios, with three replications.

Due to the recalcitrance and rigidity of the durian rind structure, a suitable method of size reduction is essential with regards to the process management, cost and particularly how it will affect the composting process. Particle size of the material is essential for microbial activity and airflow, where smaller sizes will result a in larger surface area, therefore enabling the microbes to access more substrates and oxygen (Cooperband, 2000). Particle size selection is usually a compromise between achieving high rates of aeration to facilitate the aerobic process and at the same time ensuring adequate surface area for the aerobic decomposition to occur. Based on several studies, the optimal particle size should be between 3 to 50 mm (Gray and Biddlestone, 1974; Hamoda et al., 1998; Snell, 1991; Wilson, 1993).

The three methods chosen for this study incorporate common methods and equipment that can easily be applied in Malaysia. For the three sample preparation methods selected for this study, the first method (cube) was to manually cut the rind into 25-mm (1-inch) cubes. The second method (knifemill) was to use a cutting mill (Heavy-Duty Cutting Mill SM 2000, Retsch GmbH) with a 20-mm screen, and lastly (Fitzmill) a comminutor mill (Fitzpatrick 5 HP Stainless Steel Fitz Mill, Fitzpatrick Co.) with a 30-mm screen.

The three levels of C:N ratios selected were 10:1, 30:1, and 50:1. Different levels of C:N ratios were achieved by adding urea (46-0-0) calculated using the following formula;

$$Ratio = \frac{Q_{durian}(C_{durian} \times 100 - M_{durian}) + Q_{urea}(C_{urea} \times 100 - M_{urea})}{Q_{durian}(N_{durian} \times 100 - M_{durian}) + Q_{urea}(N_{urea} \times 100 - M_{urea})}$$

Where:

Q = mass of material

C = carbon (%)

N = nitrogen (%)

M = moisture content (%) of the material

Moisture content for urea was assumed to be negligible (0 % w.b.) and by plugging in the mass of durian samples used (10-kg) and the respective carbon, nitrogen and moisture contents, the mass of urea needed for 10:1, 30:1, and 50:1 C:N ratio levels was calculated to be 200g, 50g, and 13g respectively.

Experimental Chamber

Composting needs to occur at a specific temperature and moisture content to enable thermophilic microbes to breakdown the organic materials. Optimal conditions for composting were found to be in the range of 52-60°C ((Bach et al., 1984; McKinley and Vestal, 1984), with moisture content of the material at 50-60% (Tiquia et al., 1998; McKinley et al., 1986; Suler and Finstein, 1977). Temperature and relative humidity is generally high throughout the year in Malaysia. Annual temperature range is around 23-33°C with a relative humidity range of 70-90% (McGinley, 2011). In order to simulate climate conditions similar to those found in Malaysia, as well as providing an optimal composting environment, a controlled environment chamber model IUF3050A (Puffer-Hubbard, MI) was used to house the compost jars. The temperature in the chamber was maintained at 27°C ±2 (80F ±3) using a fan powered space heater (Sunbeam SFH442-WM1, Sunbeam Products, Inc.). Relative humidity was maintained at 80% ± 5 using an evaporative humidifier (Essick Air MA0800 Digital Humidifier, Essick Air Products, Inc. AR, USA).

Temperature and carbon dioxide production rate

During the composting process, temperature and carbon dioxide production rate was measured every two days after the jars were thoroughly shaken. Duration of the composting process is dependent on when it achieves stability and maturity. Evaluation of stability and maturity are based on the metabolic activity of the microbes involved in the degradation of the organic material that can be determined by a drop in compost temperature (Flynn and Wood 1996) and a decline in carbon dioxide respiration rate (Switzenbaum et al., 1997; TMECC, 2002). Temperature was recorded using temperature recorders (TRIX-8 LogTag Temperature Data Recorder, MicroDAQ.com, Ltd., NH 03229 USA) that were placed in each jar and in the experimental chamber to observe the respective temperature profiles. Carbon dioxide production rate was recorded using a carbon dioxide sensor (PASPORT Carbon Dioxide Gas Sensor, PASCO Scientific, CA 95747 USA). CO_2 production rates (ppm/s) were recorded by placing the probes in the plastic jar for two minutes and the rate determined by the slope was recorded.

Analysis of compost

Compost samples were sent to the Iowa State University Soil and Plant Analysis Lab to determine pH, electrical conductivity, and water-dissolved macronutrients (phosphorous, potassium, calcium, and magnesium). Statistical analysis was done on SAS v9.2 (SAS Institute, Cary, NC). Removal of outliers for percent Zn was necessary to compare treatment means.

Results and Discussion

Temperature

Metabolism and population dynamics of microbes are highly dependent on temperature during composting (Namkoong and Hwang, 1997; Joshua et al., 1998). For the composting process to be effective, a minimum temperature must be attained to achieve high rates of decomposition (Finstein and Morris, 1975; Finstein et al., 1986; Miller, 1992). Based on several studies, a temperature range of 52-60 °C is optimal for the composting process (MacGregor et al., 1981; Bach et al., 1984; McKinley and Vestal, 1984). However, it is also worth to note that optimal composting temperature varies with different composting material type and process control and it is possible to achieve high microbial activity at lower temperatures (Suler and Finstein, 1977). Furthermore, Miller et al. (1990) found that composting at moderately thermophilic temperatures (47-54 °C) also produces good quality compost.

Figure 10(A) to (D) summarize the temperature profiles for all three C:N ratios and control that were observed during the experiment. The temperature in the chamber was set at 30°C throughout the experiment and any increase in temperature is assumed to be induced by the heat generated from the composting process. In general, all four groups showed positive deviations from the chamber temperature up to the 11th day, which indicates that heat was generated in experimental compost jars. The highest temperature observed in all groups was 38.2°C which is well below the optimal range of 52-60 °C or the more moderate thermophilic range of 47-54°C. Based on these observations, it seems that the initial mesophilic stage (10-45°C) was allowed to commence but the pile did not reach the next stage of process, where

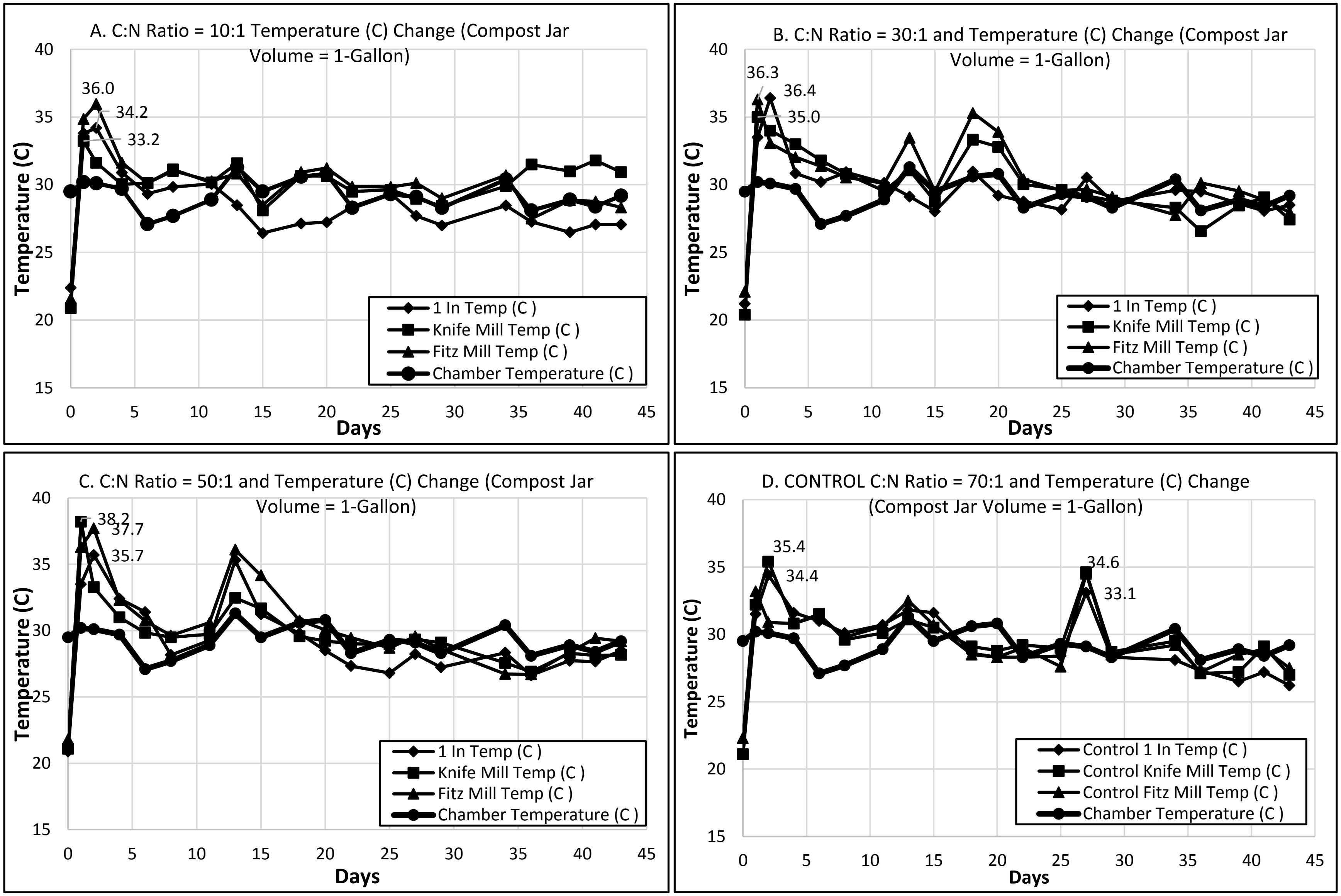

Figure 10: Temperature profiles and highest temperature achieved during composting process.

oxidation of carbon compounds will produce heat to move the process to the thermophilic stage (45-75°C). The initial volume of the compost pile in the experimental jar was about 6.7 cm^3 ($6.7x10^{-3}$ m^3) and this is considerably smaller than the critical volume of 0.76 m^3 for compost piles to heat up and retain heat (Cooperband, 2000). Therefore, the lower maximum temperature observed may be caused by the combination of reduced organic material, microflora, and thermal insulation as seen in bigger piles.

When comparing the temperature profiles of the different C:N ratios and the control, the 10:1 (Figure 10(A)) was observed to have just one temperature spike compared to the other groups that had multiple spikes. Nitrogen supply to the compost will either cause nitrogen uptake by the microbes through nitrogen immobilization or release through mineralization in the form of ammonia gas. Mineralization occurs at a C:N ratio of 20:1 or lower (Cooperband, 2000). A strong ammonia stench was observed in the chamber during the initial two weeks of the experiment and this seemed to confirm that some portion of nitrogen was lost and the decomposition process was relatively inhibited in the 10:1 ratio group. The multiple temperature peaks in the 30:1 and 50:1 ratio group may also indicate that nitrogen availability to the microbes was limited and due to rigorous mixing and shaking of the jar, where some pockets/clusters of material came into contact with the urea later in the experiment. As seen in Figure 11, the carbon dioxide evolution profile seemed to concur with this observation due to the lower overall carbon dioxide reading in the jar.

Particle size affects moisture retention, free air space (Jeris and Regan, 1973) and porosity of the compost mixture (Naylor, 1996). Based on the results, the different profiles may indicate some effect of the different preparation methods used in this experiment. For the other

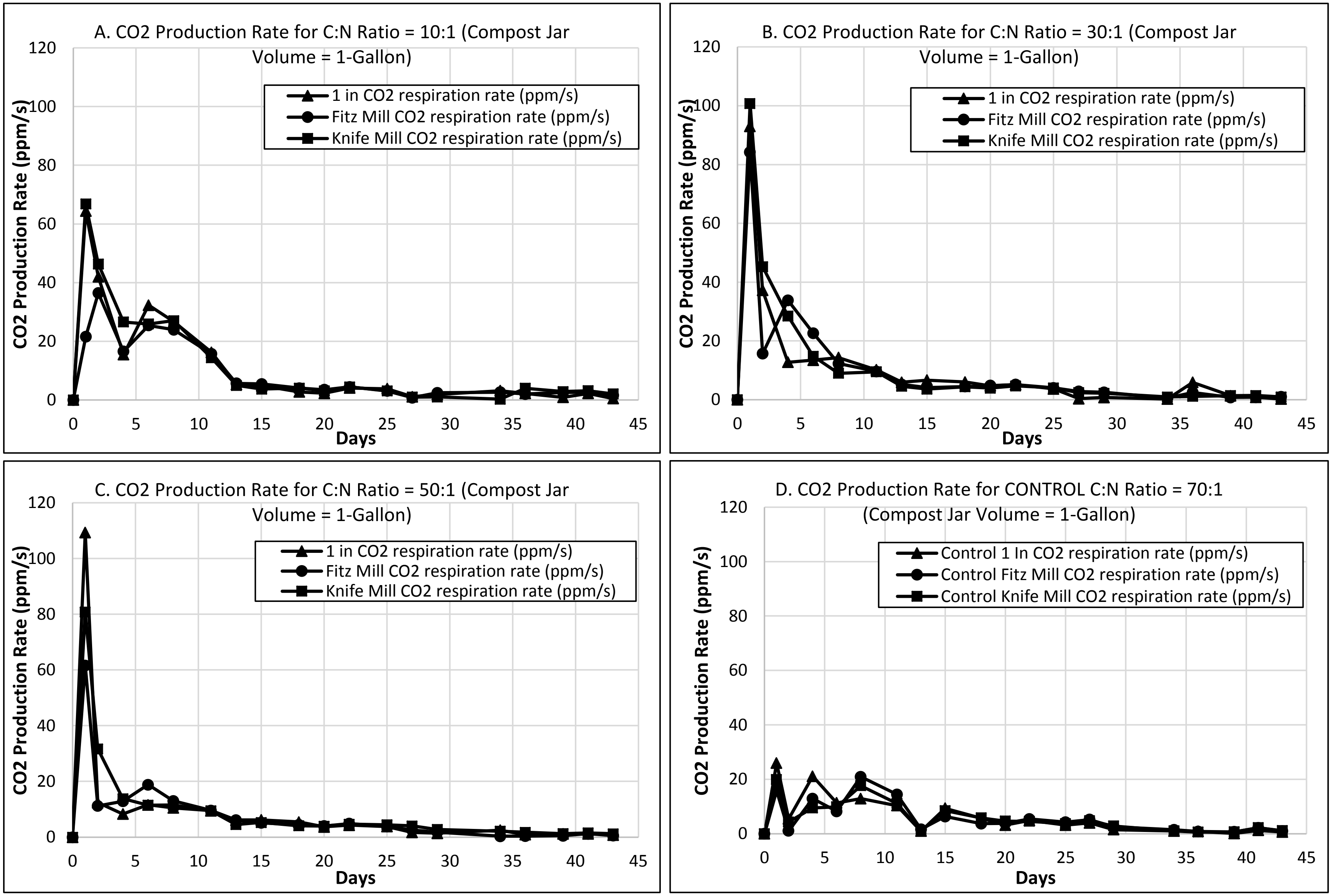

Figure 11. CO_2 production rate profiles for all C:N ratio groups.

ratio groups, the 1-in cube method was observed to have consistently lower temperature profiles. This may be due to the relatively larger size and less surface area for microbial activity. It is also possible that the larger particle size increased aeration and porosity within the pile and this may have negatively affected its capability to insulate heat and negatively affect the composting process. The knife mill method produced the highest temperature profile among the three methods. Comparing the cutting process for the knife mill and the Fitz mill, the knife mill was able to shred the materials more finely than the Fitz mill. Thus, the higher temperature profile seen was probably influenced by increased microbial activity due to relatively higher surface area produced.

CO_2 production rate and weight change

The decline of microbial activity resulting in the decrease in carbon dioxide production during composting is an indication of stabilization and maturation of the compost. Tracking of carbon dioxide production not only indicates the suitable time to stop the composting process, but also provides a measure to quantify microbial activity during composting. Carbon dioxide production rate also provides a general indication of microbial activity observed in the composting vessel. Based on the results summarized in Figure 11 and 12 (A) to (D), for all C:N ratio groups, the carbon dioxide production profile seemed to confirm the results shown in Figure 9, where the highest readings occurred during the first 13-days of the experiment, indicating that the carbon dioxide peaks are most likely due to mesophilic microbial activity. The 30:1 and 50:1 ratio groups were found to have the highest peaks, while in the control group; carbon dioxide evolution rates were more erratic and considerably less than the other three groups. Comparing the three sample preparation methods, all three methods produced profiles

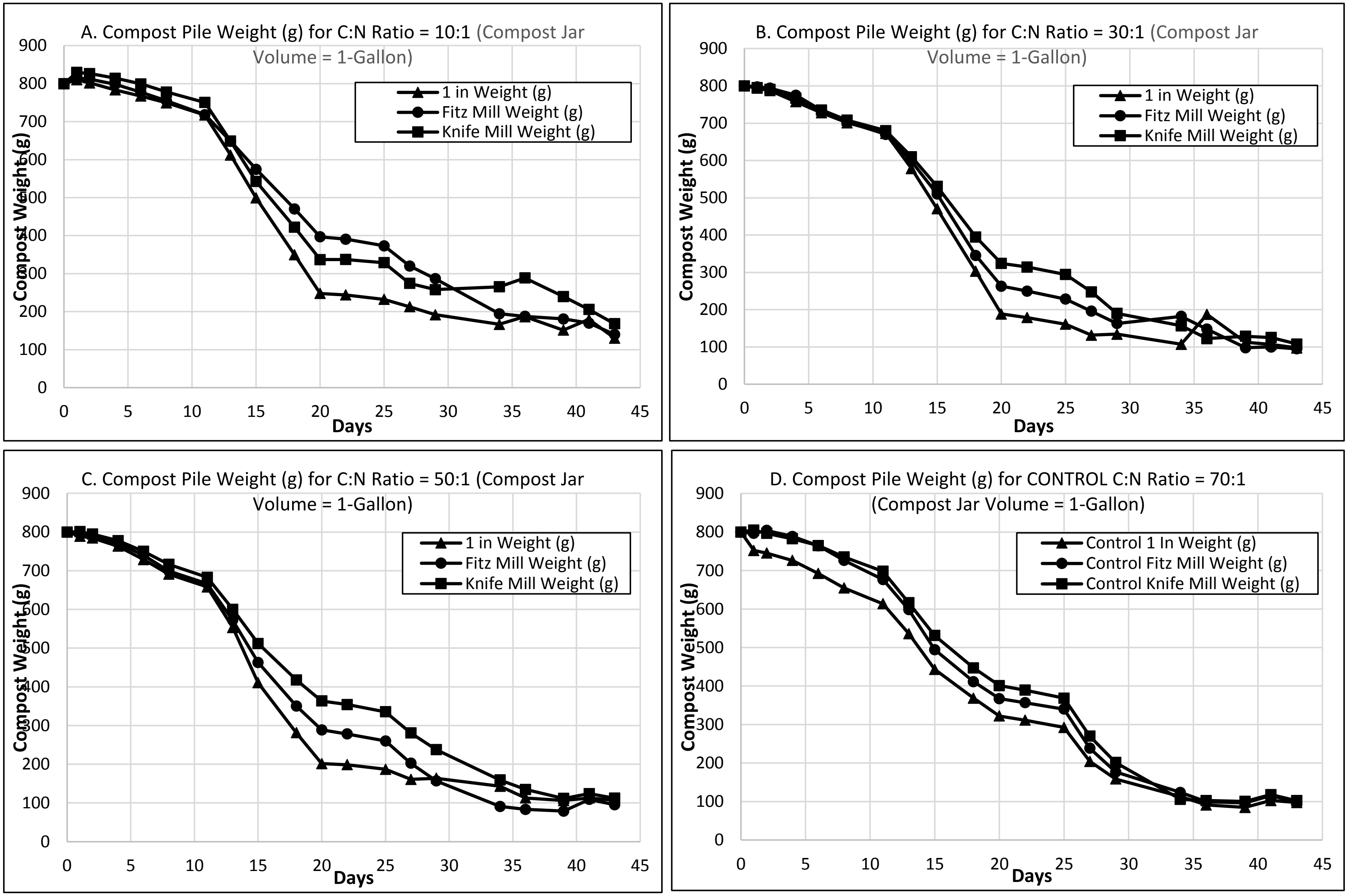

Figure 12. Weight loss profiles for all C:N ratio groups.

that generally follow the same trends albeit with marginal peak height in some cases.

Viewing the weight change profile in Figure 12, the pile weight started to level off after day 34 for all groups. In general, all three C:N ratio groups and control group did not differ much in terms of weight change during the composting process. However, it is hard to discount the influence of drying during the composting process unless periodical moisture content sampling was conducted. Cubed samples were found to lose weight more rapidly than other methods. Based on the carbon dioxide production rate in Figure 11, this may be due to higher aeration rates within the pile and possible drying out of the material.

Analysis of Compost

Depending on its use, compost characteristics such as pH, moisture content, soluble salt content and nutrient content are vital indicators of its quality and suitability (US Composting Council, 2001). This information will be used to determine if further adjustment or addition of supplemental nutrients should be done to promote optimal plant growth.

Compost contains different minerals that a plant needs in different amounts. It is normally divided into three categories: primary macronutrients (N, P and K), secondary macronutrients (Ca, Mg, Na and S) and micronutrients (B, Cl, Cu, Fe, Mn, Mo and Zn). Typically, macronutrients are required in greater amounts than micronutrients (Brinton, 2000). Table 1 lists the mean water soluble macronutrients available in the compost compared to the recommended levels suggested by CEC (1986). Due to its solubility in water, it is more accessible and more readily absorbed by the plant roots to supplement its growth.

Looking at the results obtained in Table 13, the amount of nutrients available to the plants are generally lower than the amount recommended by the CEC. This low amount may affect the overall growth of the plants and may result in stunted growth (Morgan 1998) or deformation of flowers (Eysinga et al., 1980). Lower amounts of macronutrients were also reported by Kala et al. (2009) in their study on oil palm wastes composting. It was suggested that addition of other materials such as sludge compost can increase or improve these numbers. Nutrient supplement is essential in compost application as the levels of nutrients in most compost are not sufficiently high to provide adequate nutrients for horticultural plants (Chaney et al., 1980; Hue and Sobieszczyk, 1999).

Results for the quantities of nutrients were not expected to be different because only durian rinds were used except for the addition of urea. From Table 13, some variability was found in the amount of nutrient content for K, Mg and Zn for the different treatments. This discrepancy may possibly be caused by the difficulty in preparing the sample for chemical analysis due to the rigid and recalcitrant structure of the durian rind. Because the analysis requires washing the compost with distilled water and quantifying the nutrient absorbed by the filtrate, the cubed samples were harder to breakdown and this resulted in less surface area for contact with the distilled water. Hence, values obtained for the cubed samples were generally lower than the other treatments. Furthermore, different parts of the rind may contain different amount of macronutrients as observed by Kala et al. (2009) where different parts of the oil palm waste were found to have different amounts of macronutrients.

Table 13. Mean water soluble macronutrients for all treatment levels compared to the recommended CEC (1986) values.

C:N	Size	P (%)	K (%)	Ca (%)	Mg (%)	Zn (%)
10:1	**Cube**	0.1[a]	1.0[c]	0.002[c]	0.026[c]	0.001[c]
10:1	**Fitz Mill**	0.1[a]	1.6[bc]	0.003[abc]	0.047[abc]	0.003[ab]
10:1	**Knife Mill**	0.1[a]	1.5[bc]	0.004[abc]	0.050[ab]	0.001[ab]
30:1	**Cube**	0.1[a]	1.6[bc]	0.003[abc]	0.047[abc]	0.003[ab]
30:1	**Fitz Mill**	0.1[a]	3.3[a]	0.004[ab]	0.048[abc]	0.002[ab]
30:1	**Knife Mill**	0.1[a]	2.4[abc]	0.005[a]	0.054[a]	0.002[ab]
50:1	**Cube**	0.1[a]	1.5[bc]	0.002[bc]	0.028[bc]	0.001[ab]
50:1	**Fitz Mill**	0.1[a]	2.5[ab]	0.003[abc]	0.037[abc]	0.003[ab]
50:1	**Knife Mill**	0.1[a]	2.4[abc]	0.003[abc]	0.041[abc]	0.005[ab]
Control	**Cube**	0.1[a]	1.2[bc]	0.002[bc]	0.022[c]	0.001[ab]
Control	**Fitz Mill**	0.1[a]	2.1[abc]	0.003[abc]	0.036[abc]	0.002[ab]
Control	**Knife Mill**	0.1[a]	3.1[a]	0.005[a]	0.057[a]	0.008[a]
Recommended		0.5	0.3	2.0	0.3	n/a

Means with the same letters are not significantly different

For application as greenhouse media, essential quality indicators such as pH, electrical conductivity, moisture content, particle size, stability and maturity of the compost must be in a certain range (US Composting Council, 2001; Stoffella and Kahn, 2001). Table 14 lists values of pH, electrical conductivity and final moisture content for the compost produced from this experiment, compared to the recommended range suggested by the US Composting Council (2001). Based on the results summarized in Figure 10 to 12, the compost is assumed to be stable and mature except for the treatment controls, where it is suspected that based on the result profiles, sufficient decomposition was not achieved. In terms of the particle size of the compost, all samples except the cubed samples passed the ½-inch screen test.

The final pH range is highly dependent upon materials used, addition of amendments and the composting process itself (Stoffella and Kahn, 2001). Very high or low pH can negatively affect plant growth and development, as well as injuring the roots. In general, the pH values were found to be on the high side for all treatments. Only the first treatment combination (10:1, Cube) was found to be within the recommended range. Compost obtained from 10:1 C:N ratios were found to be lower than other C:N ratio groups. Conversion of urea into ammonia and its release as described earlier may have caused the lower pH value. To condition this compost for application as greenhouse media, high levels of pH can be reduced with the addition of elemental sulfur (S), but it must be in a very fine form in to ensure its effectiveness (Marfa et al., 1998).

Electrical conductivity (EC) is used to measure the ability of the soluble ions to conduct electricity. This will indicate the amount of soluble salts available in a compost sample. Excessive salt content, particularly sodium and chloride, can be phytotoxic to the plant (US Composting Council, 2001). Based on the results in Table 14, the soluble salts are low in the compost samples as indicated by the low values of electrical conductivity in all samples.

Moisture content affects handling, storage and transportation of the compost. High moisture compost can increase transportation cost due to reduced bulk density and also cause handling issues, where dry compost (<35% moisture) can be dusty and irritating, and wet compost (>55% moisture) can cause clumpyness. From Table 14, moisture content observed for all compost samples, except the first two treatment combinations (10:1, Cube; 10:1, Fitz Mill) were found to be within the recommended range.

Table 14. Summary of means of essential compost quality parameters compared to the range recommended by US Composting Council (2001).

C:N	Size	pH	Electrical Conductivity (dS/cm)	Moisture (% w.b.)	Particle Size
10:1	**Cube**	7.9[d]	0.04[c]	28.0[c]	No
10:1	**Fitz Mill**	8.8[bc]	0.06[ab]	28.9[c]	Yes
10:1	**Knife Mill**	8.5[cd]	0.06[ab]	45.1[ab]	Yes
30:1	**Cube**	9.3[ab]	0.07[ab]	46.6[a]	No
30:1	**Fitz Mill**	9.7[a]	0.10[a]	48.5[ab]	Yes
30:1	**Knife Mill**	9.3[ab]	0.08[ab]	37.2[ab]	Yes
50:1	**Cube**	9.6[a]	0.06[ab]	34.3[ab]	No
50:1	**Fitz Mill**	9.8[a]	0.09[a]	36.8[ab]	Yes
50:1	**Knife Mill**	9.6[a]	0.06[ab]	35.4[ab]	Yes
Control	**Cube**	9.8[a]	0.06[ab]	50.0[ab]	No
Control	**Fitz Mill**	9.6[ab]	0.09[ab]	42.3[ab]	Yes
Control	**Knife Mill**	9.8[a]	0.11[a]	51.9[a]	Yes
Recommended for greenhouse media		5.5 to 8.0	< 3.0	35 to 55	Pass through 1/2–inch screen
Means with the same letters are not significantly different					

Based on the criteria recommended by the Composting Council (US Composting Council 2001), comparison of the results in Table 14 is illustrated in Table 15. Each treatment combination was assessed based on the recommended range. Attributes that fell within the range were assigned with check marks (✓). Conversely, ‘x’ marks (✗) were assigned to attributes that were outside the recommended range. This tabulated result can then be used in relation to the observations gathered from the temperature (Figure 10), carbon dioxide evolution (Figure 11) and weight (Figure 12) profiles to narrow down plausible treatment combinations that can produce good composting process and product. The assessment can then proceed with elimination of treatment combinations that indicate retarded composting process with the most amount of attributes outside the recommended range.

Table 15. Assessment of compost quality parameters based on the recommended range.

C:N	Size	PH	Electrical Conductivity (dS/cm)	Moisture Content (% w.b.)	Particle Size
10:1	**Cube**	✓	✓	×	×
10:1	**FitzMill**	×	✓	×	✓
10:1	**KnifeMill**	×	✓	✓	✓
30:1	**Cube**	×	✓	✓	×
30:1	**FitzMill**	×	✓	✓	✓
30:1	**KnifeMill**	×	✓	✓	✓
50:1	**Cube**	×	✓	×	×
50:1	**FitzMill**	×	✓	✓	✓
50:1	**KnifeMill**	×	✓	✓	✓
Control	**Cube**	×	✓	✓	×
Control	**Fitz Mill**	×	✓	✓	✓
Control	**Knife Mill**	×	✓	✓	✓

The control treatment was omitted from Table 14 due to the suspicion that it did not achieve maturity and stability based on the temperature profile and the carbon dioxide evolution profiles in Figure 10 and 11. Assessing the remaining treatment combinations in terms of the C:N ratios, all 10:1 ratio treatment combinations can also be rejected based on the lower profile trends observed in Figure 10 and 11 indicating lower levels of microbial activity and less rigorous composting process. Comparing the sample preparation methods, the cubed samples were found to have a higher weight loss rate that may be caused by higher pile porosity and higher aeration rates. This is undesirable in a compost pile since it will reduce insulation of heat generated within the pile and thus lowering pile temperature. Furthermore, the cubed samples did not pass the recommended particle size limit. Therefore, the options can now be narrowed down to treatment combinations involving C;N ratios of 30:1 and 50:1 for both Fitz mill and knife mill. This is also beneficial from a cost and logistics standpoint as the high C:N ratio indicates that addition of manure or other nitrogen source is not critical in obtaining good compost. Furthermore, the inherent concern of pathogenic risk with the use of manure or other biological waste is also eliminated and lessens the concerns related to safety and health regulations.

Conclusions

Stability and maturity are key factors that ensure successful application of compost. Temperature and carbon dioxide production profiles can be used to monitor and evaluate the composting process of durian rind. Decomposition and breakdown of the durian rind was observed in all treatment combinations at different rates. Temperature and weight change profiles between the C:N ratios were found to follow similar trends. However, comparing the profiles for the different sample preparation methods, the cubed samples were found to have lower temperature and weight change profiles compared to the other two. Although tested processes did not achieve thermophilic heat rates, this is not crucial as thermophilic temperature is essential in the destruction of pathogens particularly from manure or human waste, both of which are not used in this experiment. Carbon dioxide production rate profiles seemed to indicate that the high C:N ratio seen in the control as unfavorable to providing optimal conditions for high microbial activity. Similarly, using the same profiles and based on observations during the experiment, the 10:1 ratio was also found to be unfavorable to the composting process.

The nutrient content observed from the analysis of the compost indicates that durian rind compost is suitable for greenhouse media application. However, pH conditioning, nutrient supplement, and particle size reduction are needed to amend and improve its quality. Selection of the best treatment combination is difficult due to uncertainties seen during composting. However, based on the observed temperature, weight change and carbon dioxide evolution profiles, as well as comparison of treatment combinations that fall within the recommended product attributes, the 30:1 and 50:1 ratios for both Fitz Mill and knife mill methods are seen as the most plausible treatment combination.

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CHAPTER 6. GENERAL CONCLUSIONS

The diversification of energy sources through the development of renewable resources in line with the guidelines of the current Malaysia plan can be seen as a step in the right direction in securing energy security and environmental conservation. However, a more aggressive stance in terms of utilizing the abundant biomass resources needs to be pursued. Implementation of biomass utilization methods can be facilitated by the existing framework within relevant governmental agencies such as FAMA, DOA, MARDI (Malaysian Agricultural Research and Development Institute) and PORIM (Palm Oil Research Institute Malaysia).

When compared to Karl Fischer reagent test which was designated as the reference or standard in this study, HORT 2, BS 2, and NAFTA were found to be the best methods for producing accurate moisture content tests. These methods produced high reproducibility and the least percent deviation from the standard KF method. Comparison of these three methods indicates that HORT 2 method can serve as the easiest and simplest of the three and should be given precedence.

The three factors chosen (drying time, drying temperature and sample preparation method) were found to be significantly influencing the accuracy of a moisture test. Ground samples were found to be more robust in producing accurate readings at different drying times and temperatures. Cubed and whole samples require adequate drying time and temperature to remove moisture from the samples. Depending on the process optimization needs, ground samples dried for two hours can be applied for high turn-around labs, or if handling of the tough rind is an issue, then the use of whole rind dried at 103°C for 48 hours or 135°C for 24 hours can be applied instead.

Durian biomass was found to display similar physical and chemical characteristics to other established biorenewable feedstocks such as corn stover or oil palm biomass. Although the high moisture content of post-harvest durian biomass is seen as a major stumbling block for thermochemical conversion, the favorable drying climate in Malaysia can potentially be applied as a cost effective pretreatment method. In terms of its chemical properties, durian biomass is an attractive feedstock option due to its low ash and comparable calorific value, especially compared to palm EFB which is widely used in Malaysia. This feature is attractive for the application of this material in co-firing with coal to produce electricity. For pyrolysis applications, there are still some uncertainties in terms of its high oxygen content and the discrepancies with other published results on the fraction of lignocellulosic compounds. However, the results show that it is comparable to corn and the bio-oil product was chemically similar to other biomass based on the identification of pyrolysis products.

Temperature and carbon dioxide production profiles can be used to monitor evaluate the composting process of durian rind, as well as a method of compost maturity indication. A controlled environment was erected to mimic conditions in Malaysia and found to provide an adequate environment for composting. Different rates of decomposition and breakdown of the durian rind was observed in all treatment combinations. The results indicate that higher C:N ratios and smaller particle size are key parameters that ensure good compost production. The nutrient content observed from the analysis of the compost indicates that durian rind is suitable for greenhouse media application. However, pH conditioning, nutrient supplement, and particle size reduction are needed to amend and improve its quality.

Future work

One of the biggest goals in this study was to fill the fundamental gaps in understanding the potential of utilizing durian biomass as potential feedstock for biorenewable resources. As acknowledged earlier, this study aims to be the starting point by extending the knowledge and techniques acquired from this exploratory study of durian biomass to other tropical fruit in Malaysia. Since there a myriad of other fruits available and some are indigenous to a specific region in Malaysia, characterization of other tropical fruits will not only further add to the fundamental insight but also enable the identification of the most suitable conversion route that can fully utilize a particular feedstock type at the highest efficiency and economic viability.

Extension of this knowledge can also include application of the moisture content test for durian biomass established in this study for other tropical fruit biomass. It is vital to confirm if the same test can be applied without considerable loss in accuracy or if it requires a different set of processing parameters in order to obtain test results with the highest accuracy. The latter scenario would also include determining if the same levels of treatment can be applied or if it requires a different approach should its characteristics differ greatly than those considered in this study.

Future studies can also include further optimization of feedstock selection by way of assessing different blend or fractions of biomass in order to minimize the effect of a particular undesirable trait. This include blending fruit biomass with high oxygen content with other fruit biomass or other types of biomass in order to reduce its O:C ratio. Blending can also be a form of pretreatment to reduce the overall moisture content of the blend by blending dryer biomass. By studying its thermochemical processing and product properties, favorable results will point towards avoiding the necessity of drying and its inherent high cost.

Composting of durian biomass in a larger scale can further extend the understanding of the composting parameters as well as assessing the compost produced. Composting of other tropical fruit biomass can also be carried out and assessment of composting different types of fruit biomass blend can also be done. Food processing and livestock research are two other major research activities carried out in MARDI. Therefore, future research can also include studying the composting attributes of different blend of tropical fruit biomass with food processing waste and/or animal waste. As seen in the results of the compost analysis discussed earlier, blending tropical fruit biomass with these two waste streams can potentially improve its overall quality such as its nutrient content.

Expansion of this research area will also include assessing the potential of utilizing this feedstock from an economical perspective. The viability of logistical and processing parameters and how it converges with its economic feasibility is normally done through techno-economic analyses. It will be interesting to gauge the performance of utilizing this feedstock either individually or in lieu with oil palm EFB, and comparing it with existing resources. This will also provide vital indications of the avenues for optimization in order to fully integrate this feedstock into the energy mix.