

**HYDROTHERMAL AND VAPOTHERMAL
CARBONIZATION OF AGRICULTURAL
WASTES TO PRODUCE SOLID FUELS**

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by

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TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENTS	ii
TABLE OF CONTENTS	iii
LIST OF TABLES	vii
LIST OF FIGURES	viii
LIST OF ABBREVIATIONS	xi
ABSTRAK	xii
ABSTRACT	xiii
CHAPTER ONE : INTRODUCTION	
1.1 Research background	1
1.2 Problem statement	4
1.3 Objectives	5
1.4 Scope of works	5
1.5 Thesis outline	6
CHAPTER TWO : LITERATURE REVIEW	
2.1 Overview	7
2.2 Biomass energy	8
2.3 Hydrothermal processing	11
2.4 Hydrothermal carbonization	13
2.4.1 Effects of hydrothermal carbonization on the biomass materials	15
2.4.1.1 Improve of fuel quality and energy densification	16
2.4.1.2 Improve of dewaterability and hydrophobicity	21

	Page
2.4.1.3 Transformation of the microstructure	24
2.4.1.4 Improvement in combustion characteristic	27
2.4.2 Yield from hydrothermal carbonization	28
2.4.3 Hydrothermal carbonization of lignocellulosic biomass	30
2.5 Vapothermal carbonization	33
2.6 Summary	37
CHAPTER THREE : METHODOLOGY	
3.1 Overview	39
3.2 Biomass materials preparation	39
3.3 Experimental setup	41
3.3.1 Hydrothermal carbonization	43
3.3.2 Vapothermal carbonization	43
3.3.3 Thermal carbonization	46
3.4 Experimental procedures	46
3.5 Parameters setting for the treatment processes	48
3.6 Analysis of the hydrochar and performance characterization of the carbonization process	50
3.6.1 Determination of higher heating value	50
3.6.2 Proximate analysis	52
3.6.3 Combustion characterization	53
3.6.4 Scanning electron microscopy (SEM)	55
3.6.5 Other indicators	56

	Page
 CHAPTER FOUR : RESULTS AND DISCUSSIONS	
4.1	58
4.2	59
4.2.1	59
4.2.2	63
4.3	66
4.3.1	66
4.3.2	68
4.4	72
4.4.1	73
4.4.2	82
4.5	89
 CHAPTER FIVE : CONCLUSIONS	
5.1	93
5.2	94

	Page
REFERENCES	96
APPENDICES	

LIST OF TABLES

		Page
Table 1.1	Quantity of agricultural wastes produced in Malaysia in 2007	2
Table 2.1	Lignocellulose composition of biomass materials	32
Table 3.1	Summary of process parameters of carbonization processes done in the project	49
Table 4.1	Properties of biomass materials before and after hydrothermal carbonization in phase 1	62
Table 4.2	Comparison of properties of biomass materials which undergoes hydrothermal carbonization with different water to biomass ratio	64
Table 4.3	Properties of raw EFB and vapohtermal treated EFB in different configurations	67
Table 4.4	Properties of biomass materials before and after vapohtermal carbonization	71
Table 4.5	Comparison of raw biomass materials and carbonized biomass materials from hydrothermal and vapohtermal carbonization	74
Table 4.6	Characteristic combustion parameters of the fuels	78
Table 4.7	Comparison of raw EFB and carbonized EFB from various carbonization methods	84
Table 4.8	Characteristic combustion parameters of EFB and carbonized EFB	88
Table 4.9	Comparing the effects of immediate releasing reactor pressure or not after the holding period end of hydrothermal carbonization and vapohtermal carbonization	90

LIST OF FIGURES

	Page	
Figure 2.1	Classification of biomass conversion methods	10
Figure 2.2	Typical temperature and pressure ranges for different hydrothermal processing products from various biomass materials	12
Figure 2.3	Typical chemical transformations and their direction in a van Krevelen diagram	18
Figure 2.4	Proposed reaction mechanism of hydrothermal carbonization from D-glucose	25
Figure 3.1	Flowchart of the investigation	40
Figure 3.2	The setup of reactor that carry out the hydrothermal carbonization and vapothermal carbonization in this project	42
Figure 3.3	Schematic diagram of the carbonization reactor system used in this project	42
Figure 3.4	Schematic diagram of hydrothermal carbonization	43
Figure 3.5	Schematic diagram of dual chamber vapothermal carbonization	44
Figure 3.6	Schematic diagram of single chamber vapothermal carbonization	45
Figure 3.7	Flowchart showing the experimental procedures	46
Figure 3.8	Bomb calorimeter	51
Figure 3.9	Thermogravimetric analyzer (TGA)	52
Figure 3.10	Sample result from TGA and how mass percentage of fixed carbon, volatile matter, ash and moisture are determined	54
Figure 3.11	Measurement of ignition temperature	55
Figure 3.12	Scanning electron microscope	56

	Page
Figure 4.1	Pictures of dried biomass materials after hydrothermal carbonization in phase 1 comparing to the raw materials (smaller pictures) (a) Oil palm empty fruit bunches. (b) Oil palm mesocarp fibers. (c) Palm kernel shells. (d) Rubber seed shells. 60
Figure 4.2	SEM pictures of hydrothermal carbonized products (a) Oil palm empty fruit bunches. (b) Oil palm mesocarp fibers. (c) Palm kernel shells. (d) Rubber seed shells 62
Figure 4.3	Pictures of dried biomass materials after hydrothermal carbonization in phase 2 comparing to its counterpart from phase 1 (smaller pictures) (a) Oil palm empty fruit bunches. (b) Oil palm mesocarp fibers. (c) Rubber seed shells. 64
Figure 4.4	Pictures of dried biomass materials after vapothermal carbonization comparing to the raw materials (smaller pictures) (a) Oil palm empty fruit bunches. (b) Oil palm mesocarp fibers. (c) Palm kernel shells. (d) Rubber seed shells. 69
Figure 4.5	Comparison of [LEFT] water collected outside the container and [RIGHT] condensate collected inside the container 69
Figure 4.6	SEM pictures of vapothermal carbonized products (a) Oil palm empty fruit bunches. (b) Oil palm mesocarp fibers. (c) Palm kernel shells. (d) Rubber seed shells. 71
Figure 4.7	Comparison of energy content of raw biomass materials and carbonized biomass materials 76
Figure 4.8	Comparison of energy yield from hydrothermal and vapothermal carbonization of various biomass materials 76
Figure 4.9	Comparison of composition of raw biomass materials and carbonized biomass materials 77
Figure 4.10	DTG Curves of raw OPMF and carbonized OPMF 79
Figure 4.11	DTG Curves of raw PKS and carbonized PKS 79
Figure 4.12	DTG Curves of raw RSS and carbonized RSS 80
Figure 4.13	[LEFT] Tar deposited during hydrothermal and vapothermal carbonization [RIGHT] Tar deposited during torrefaction 84

	Page
Figure 4.14 Comparison of energy content of raw EFB and carbonized EFB	86
Figure 4.15 Comparison of energy yield from various carbonization of EFB	86
Figure 4.16 Comparison of composition of raw EFB and carbonized EFB	87
Figure 4.17 DTG Curves of raw EFB and carbonized EFB	88
Figure 4.18 A thin carbon rich layer are formed at the surface of water in hydrothermal carbonization	92

LIST OF ABBREVIATIONS

DTG	Differential thermogravimetric analysis
EFB	Oil palm empty fruit bunches
HHV	Higher heating value
HTC	Hydrothermal carbonization
OPMF	Oil palm mesocarp fibers
PKS	Palm kernel shells
RSS	Rubber seed shells
TC	Thermal carbonization
TGA	Thermogravimetric analysis
VTC	Vapothermal carbonization

PENGGARBONAN HIDROTERMO DAN WAPTERMO SISA PERTANIAN UNTUK MENGHASILKAN BAHAN API PEPEJAL

ABSTRAK

Kajian telah dijalankan untuk mengkaji kesan pengkarbonan hidrotermo dan waptermo dalam memproses beberapa sisa-sisa pertanian. Sebuah reaktor dengan dua ruang telah direka untuk menjalankan kedua-dua proses pengkarbonan berkaitan. Dari eksperimen, pemadatan tenaga tertinggi yang dicapai adalah 1.280 untuk pengkarbonan waptermo dan 1.145 untuk pengkarbonan hidrotermo dalam memproses tandan kosong kelapa sawit. Perbandingan hidrochar yang dihasilkan daripada kedua-dua proses menunjukkan bahawa bahan-bahan daripada pengkarbonan waptermo mempunyai nilai pemanasan dan kandungan karbon tetap yang lebih tinggi berbanding dengan bahan-bahan daripada pengkarbonan hidrotermo. Penurunan kandungan bahan volatil kira-kira 20% dan kenaikan kandungan karbon tetap sebanyak 70-95% dapat diperhatikan dalam produk pengkarbonan waptermo berbanding dengan bahan mentah. Dengan sokongan daripada imej SEM dan carta DTG, dapat disimpulkan bahawa mekanisme tindak balas adalah berbeza untuk pengkarbonan hidrotermo dan waptermo. Pembebasan bahan volatil merupakan sebab utama peningkatan tenaga kandungan dalam pengkarbonan waptermo manakala pembentukan 2,5-HMF yang memberikan kemuncak dekat 340°C dalam carta DTG dicadangkan sebagai sebab peningkatan tenaga kandungan untuk pengkarbonan hidrotermo. Selain itu, projek ini menunjuk bahawa nisbah air kepada biojisim yang lebih rendah memberikan pemadatan tenaga yang lebih tinggi. Perbandingan proses pengkarbonan hidrotermo, waptermo dan termo menunjukkan bahawa pengkarbonan hidrotermo dan waptermo mampu menindaskan pemendapan tar atas permukaan dinding reaktor semasa proses pengkarbonan.

HYDROTHERMAL AND VAPOTHERMAL CARBONIZATION OF AGRICULTURAL WASTES TO PRODUCE SOLID FUELS

ABSTRACT

An investigation was carried out to study the effects of hydrothermal and vapothermal carbonization on various agricultural wastes. A dual chamber reactor was designed to carry out the hydrothermal and vapothermal carbonization processes. From the experiments, the highest energy densification achieved was 1.280 in vapothermal carbonization and 1.145 in hydrothermal carbonization for oil palm empty fruit bunches. Comparison of the hydrochar produced from both processes shows that vapothermal carbonized materials have higher heating values and higher fixed carbon contents as compared to those from hydrothermal carbonization process. Volatile matter of vapothermal carbonized materials have drops about 20% while their fixed carbon content are increased by 70-95% compare to the raw feedstock. With the supports of the SEM pictures and DTG curves of the products, it was concluded that the reaction mechanism during the hydrothermal and vapothermal carbonization are different. Devolatilization was found to be the main reason that increases the overall energy content in vapothermal carbonization, while formation of 2,5-HMF which gives a sharp peak at around 340 °C in the DTG curves was suggested as the reason that led to the increase in energy content in hydrothermal carbonized materials. Other than that, this project also showed that lower water to biomass ratio led to higher energy densification. A comparison of torrefaction, hydrothermal and vapothermal carbonization processes also showed that hydrothermal and vapothermal carbonization are capable to suppress the deposition of tar on the reactor wall during the carbonization process.

CHAPTER ONE

INTRODUCTION

1.1 Research background

The world energy consumption has been increasing from year to year as a result of increasing world population and the advancement of technology. It is expected to be increased from 524 quadrillion Btu in year 2010 to 820 quadrillion Btu by year 2040. Fossil fuel such as coal, natural gas and oil have been our main energy resources and contributed more than 80% of the total energy consumed (United States Energy Information Administration, 2013). The main disadvantage of using fossil fuel as energy source is the environmental issues it causes. Global warming and climate change are examples of environmental impact caused by burning of fossil fuel. Hence, renewable energy has become a more sustainable choice as it can be naturally replenish in relatively shorter period and it is much cleaner as compared to fossil fuel.

Bioenergy is considered the most important renewable energy option due to its potential to provide cost effective and sustainable supply of energy around the globe in the future (Balat and Ayar, 2005). One of the sources of biomass material are agricultural wastes. In Malaysia, agricultural plantation is one of the main economic activities, plantation of the five main crops (palm oil, rubber, paddy, coconut and coco) have taken up around 7 million hectares of land (Department of Statistic Malaysia, 2014). Plantations of agricultural crops have been producing a huge amount of by-product and residues yearly. Instead of being discharged to landfill or open burning, these agriculture wastes can be utilized in producing bio-fuel and used to generate about 55,000 GWh of electricity (Shafie et al., 2012).

Table 1.1 shows the amount of agricultural wastes produced in Malaysia in 2007. From the table, the wastes from oil palm industry have made up most of them. If these biomass wastes can be utilized in energy production, our dependency on fossil fuels could be reduced and it also provides a more sustainable way in discharging these wastes at the same time.

Table 1.1 Quantity of agricultural wastes produced in Malaysia in 2007 (Mekhilef et al., 2011)

Types	Quantity (ktonnes)	Moisture content (wt %)	Dry Weight (ktonnes)
Oil palm fronds	46,837.0	60.0	18,735
Empty oil palm fruit bunches	18,022.0	65.0	6,308
Oil palm fibers	11,059.0	42.0	6,414
Oil palm shells	4,506.0	7.0	4,190
Oil palm trunks	10,827.0	75.9	2,609
Paddy straw	880.0	11.0	783
Rice husk	484.0	9.0	440
Banana residues	530.0	10.7	473
Sugarcane bagasse	234.0	50.0	117
Coconut husk	171.0	11.5	151
Pineapple waste	48.0	61.2	19

Direct combustion, thermochemical conversion and biological conversion are common conversion methods in utilizing biomass as energy source (Woods et al., 2001). Direct combustion of biomass for heat generation is not a satisfying option as high moisture and oxygen content of raw biomass will reduce the combustion temperature and increase the emission of carbon monoxide (Liu et al., 2013). Comparing thermochemical conversion and biological conversion processes, thermochemical processes show several advantages such as shorter processing time, higher product yield, ability to convert a variety of biomass feedstock, ability to produce a diversity of fuel, usually do not require chemicals addition and do not require sterilization process. These made thermochemical conversion processes more favorable in treating large amount and diversify feedstock (Brown, 2011; Chen et al.,

2014; Liu et al., 2013). Pyrolysis, gasification, and liquefaction are commonly used biomass thermochemical conversion processes.

Hydrothermal treatment is another thermochemical conversion process that have gain attention from researchers due to its ability to treat wet or green biomass material directly. Hydrothermal treatment is a process to treat biomass in hot compressed water, hence avoiding energy-intensive pre-drying process (Zhao et al., 2014). The water acts as the reaction medium during the process. Main research directions in hydrothermal treatment processes are hydrothermal gasification to produce hydrogen or methane, hydrothermal liquefaction to produce bio-crude and phenols, and hydrothermal carbonization to produce carbonaceous materials (Kruse et al., 2013).

In this project, the investigation would be focused on hydrothermal carbonization of agricultural wastes in producing solid biofuels. Several researches have shown that hydrothermal carbonization were capable to improve the quality of biomass wastes in term of their fuel quality, hydrophobicity and storage advantage. Further review on the literature will be elaborated in Chapter 2.

Another process that would be investigated in this project is vapothermal carbonization. It is a derivative from hydrothermal carbonization processes. It occurs when the biomass material is subjected to saturated vapor instead of submerged in liquid water during the carbonization process (Titirici et al., 2015). The differences between hydrothermal and vapothermal carbonization are the reaction medium. In hydrothermal carbonization, the reaction medium is liquid water while in vapothermal carbonization, the reaction medium is saturated steam. This project aims to compare

the effectiveness of hydrothermal and vapothermal carbonization in upgrading the fuel quality of agricultural wastes.

1.2 Problem statement

Hydrothermal carbonization is one of the thermochemical conversion technique that gained attention from researchers due to its capability to treat wet biomass materials directly. Several researches have shown that hydrothermal carbonization is effective in upgrading the fuel properties of biomass materials, but hydrothermal carbonization of several materials such as oil palm mesocarp fiber, palm kernel shell, and rubber seed shells have not been studied.

Other than that, two distinct methods have been employed in other researches in order to provide the reaction conditions for hydrothermal carbonization. Most of the experiments are conducted by submerging the biomass material in the water while the other supply the water required for the reaction in the form of saturated steam. The difference between them are the phase of reaction medium (liquid water/saturated steam) that is in contact with the feedstock during the carbonization process, and the latter process is then known as vapothermal carbonization. Up to date, only two literatures on comparing these two methods were found. Both the literatures show that there are significant differences between the two processes but it is difficult to observe a clear trend in comparing the effects of these two methods on the product formed, hence more work need to be done to study the differences in effects between hydrothermal and vapothermal carbonization processes. In one of the literatures (Minaret and Dutta, 2016), the experimental configuration used for the vapothermal carbonization might not be suitable as it mixed the biomass materials with the water

and there is no way to make sure all the water in the system will become saturated steam during the process. Further details will be discussed in the literature review.

1.3 Objectives

The objectives of this project are:

- 1) To investigate the effects of hydrothermal and vapothermal carbonization process on the fuel quality, composition, combustion behavior and microstructure of the biomass materials.
- 2) To compare the performance of hydrothermal, vapothermal and thermal carbonization processes in improving the fuel quality of biomass material.

1.4 Scope of works

The project started with the designing, fabricating and setting up of the hydrothermal reactor system. This reactor was used to carry out the hydrothermal and vapothermal carbonization processes of several agricultural wastes. The agricultural wastes used in this project were oil palm empty fruit bunches (EFB), oil palm mesocarp fibers (OPMF), palm kernel shells (PKS), and rubber seed shells (RSS). The investigation of the project focuses on the solid yield from the process, the effect of hydrothermal and vapothermal carbonization processes on the heating values, composition, combustion behaviors and microstructure of the materials. Comparison between hydrothermal and vapothermal carbonization of various agricultural wastes

were carried out. Other than that, comparison between hydrothermal, vapothermal and thermal carbonization processes were also done for treating EFB.

1.5 Thesis outline

The thesis consists of 5 chapters. Chapter 1 gives a brief introduction about the topic as well as the objectives and scope of this project. The literature review related to the topic are presented in Chapter 2. This chapter summarizes some works done by others on hydrothermal treatment processes, especially on hydrothermal carbonization which produce solid biofuel. The chapter also provides some information on bioenergy and lignocellulosic materials that are useful to this project. Chapter 3 delivers the methodology employed in this project. This includes the procedures and methods of how the investigation are done. Results from the investigations are presented in Chapter 4 along with the data analysis and discussion on the results. Chapter 5 concludes the whole project and states the significant findings of this project.

CHAPTER TWO

LITERATURE REVIEW

2.1 Overview

This chapter summarizes and reviews some literatures that related and could be useful to this project.

In Section 2.2, introduction to biomass energy are presented. This section gives some basic knowledge on the natural of biomass energy. The advantages, potentials and problems in utilizing biomass as an alternative fuel source are included in this section. This section also gives some information on the biomass conversion methods and categorization of these processes.

Section 2.3 provides some insights into hydrothermal processing and the categorization of hydrothermal processing while Section 2.4 focuses on the hydrothermal carbonization process, which is one of the category of hydrothermal processing. Section 2.4.1 summarizes some important effects of hydrothermal carbonization on the treated biomass materials. Section 2.4.2 summarizes the factors that affecting the yield from the process. Section 2.4.3 provides some basic knowledge regarding the lignocellulosic materials and summarizes some works on hydrothermal carbonization of lignocellulosic materials.

Lastly, Section 2.5 presents information about vapothermal carbonization. Definition of a vapothermal carbonization process is presented in this section. Some works that compared hydrothermal and vapothermal carbonization are also summarized in this section.

2.2 Biomass energy

Biomass are organic materials derived from living organisms which mainly composed of carbon, hydrogen and oxygen. All water and land-based vegetation and tress, or virgin biomass, and all waste biomass such as municipal solid waste, municipal bio-solids (sewage) and wastes are examples of biomass materials. Biomass energy is the only renewable source of fixed carbon and it is considered as a clean energy source as it produces no net emission of carbon dioxide throughout the bio-cycle (Balat and Ayar, 2005). The plantation of new crops consumed the carbon dioxide produced during the combustion of biomass for their photosynthesis process, hence they are acclaimed as a “carbon neutral” fuel (Chew and Doshi, 2011). By switching from the dependency on fossil fuel towards biomass fuel, it could reduce the greenhouse gas emission and harmful emission such as oxides of nitrogen and oxides of sulfur from the combustion (Acharya et al., 2015).

Biomass is the fourth largest energy source after coal, oil and natural gas. Together with its potential to be processed into different energy carriers, it has become the largest and most important renewable energy option at present (Ladanai and Vinterbäck, 2009). Utilizing waste biomass for biochar production is a reasonable option as these waste feedstock do not have any economic value and it does not create competition for land with food crops (Kambo and Dutta, 2015). Biomass possess the potential to substitute fossil fuels but several properties of biomass materials have restraint its utilization as the main energy source.

In its untreated form, biomass has relatively low energy density and high moisture content compared to fossil fuel. To generate the same amount of energy from fossil fuel, massive amount of biomass will be needed (Chew and Doshi, 2011). High

moisture and volatile matter content are the main disadvantages of burning biomass, as these lead to poor combustion efficiency and high harmful emissions when directly combusted (Kambo and Dutta, 2014).

Other than that, biomass feedstock can differ considerably in term of their physical, chemical and morphological characteristics due to their heterogeneous natural. They are usually bulky and easily subjected to fungal attack or biodegradation as well. All these characteristics of biomass lead to a higher complexity and cost for feedstock preparation, handling, transportation and storage (Chew and Doshi, 2011). To overcome these problems, biomass is usually converted into various forms of energy carriers such as ethanol, biodiesel, syngas, methane and solid biofuels which are higher in energy density and can be handled and stored with relative ease.

As shown in Figure 2.1, biochemical and thermochemical conversions are two major routes for enhancing biomass properties. In biochemical conversion, biomass materials are broken down by microorganisms and enzymes into smaller molecules while thermochemical conversion process uses heat and catalysts to transform biomass into fuels, chemical, or electric power. Biochemical conversion process are time consuming but usually does not required external energy input (Basu, 2013a; Brown, 2011).

Compared to biochemical conversion methods, thermochemical treatment has shown several advantages such as shorter processing time, higher conversion efficiency, ability to convert a variety of biomass feedstock, ability to produce a diversity of oxygenated and hydrocarbon fuel, lower cost of catalysts, ability to recycle catalyst and does not require sterilization process (Brown, 2011; Chen et al., 2014; Liu et al., 2013). All these characteristics of thermochemical conversion method make it

more viable in treating large amount of feedstock and enable maximum utilization out of the biomass feedstock.

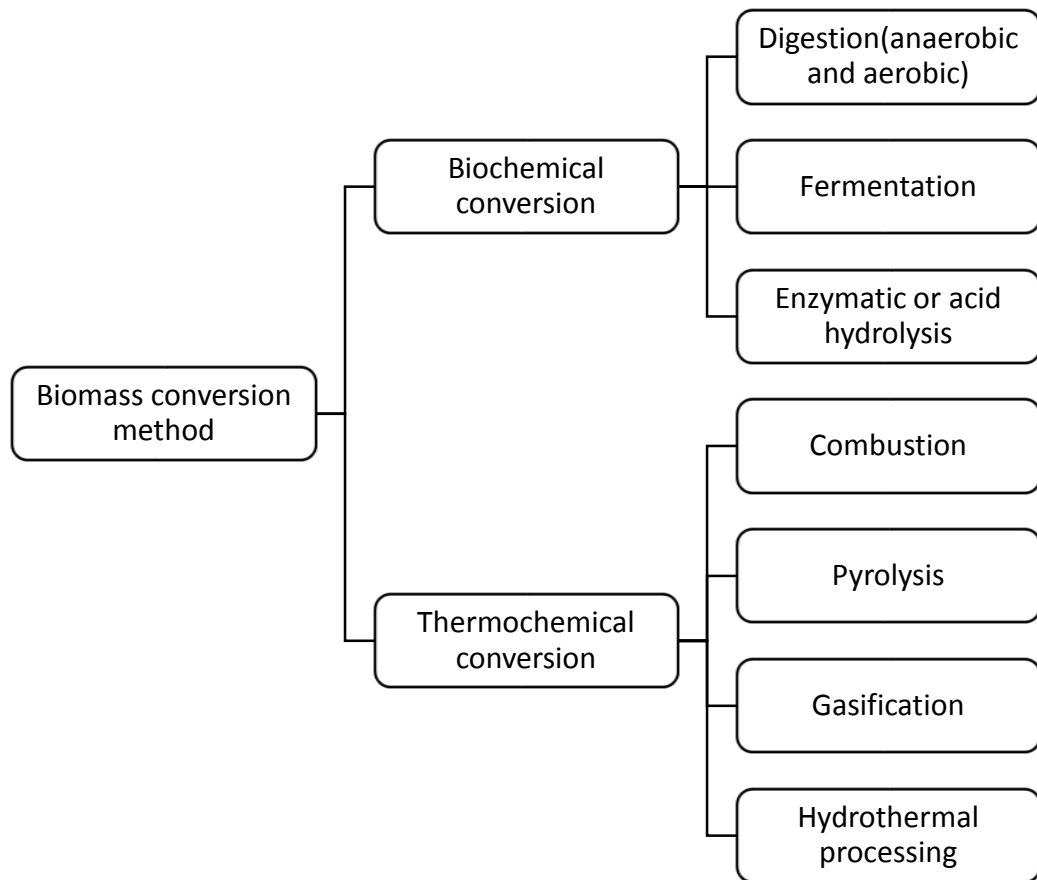


Figure 2.1 Classification of biomass conversion methods

Direct combustion, pyrolysis, gasification and hydrothermal processing are some biomass thermochemical conversion processes. Pyrolysis is thermal decomposition of organic matter under oxygen starved conditions. It usually takes place at temperatures between 400 °C and 600 °C over a short period to produce gas and liquid products and leaves a carbon rich solid which is usually known as char (Anli Geng, 2013; Demirbas, 2009). In gasification, organic matter is converted into syngas at elevated temperature in an environment lack of oxygen. Syngas is a flammable gas mixture of carbon monoxide (CO), hydrogen (H₂), methane (CH₄), nitrogen (N₂), carbon dioxide (CO₂) and some hydrocarbon. In conventional gasification, the reaction