

**INTERNATIONAL JOURNAL OF SCIENCE FOR GLOBAL SUSTAINABILITY****Hydrothermal Liquefaction of Lignocellulosic Biomass to Liquid Biofuels: A Review**Nasir Anka Garba<sup>1</sup>, Jafar Usman<sup>1</sup> and Musa Muhammad Anka<sup>2</sup><sup>1</sup>Department of Biochemistry, Federal University Gusau, Zamfara, Nigeria<sup>2</sup>Department of Science Laboratory Technology, College of Agriculture and Animal Science Bakura, Zamfara State, Nigeria\*Corresponding Author's Email & Phone No.: [nasiranka@fugusau.edu.ng](mailto:nasiranka@fugusau.edu.ng)+2348034248181

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**ABSTRACT**

The realization that fossil fuels are fast depleting has accelerated the search for an alternative energy source that is renewable, economical, and environmentally friendly. The situation has been exacerbated by global warming which has brought it home to all that there is a need to conserve energy and reduce collectively man's global carbon footprint. The instability in the regions from which the fossil fuels are sourced is another caused for concern, also higher oil price threatens the independence and security of nation states all this are unacceptable, hence there is need to search for more stable alternative sources of energy which is CO<sub>2</sub> neutral fuels from biomass, Biomass is a renewable; CO<sub>2</sub>-neutral source of energy. Lignocellulosic biomass a biomass from grass, wood, and other non-food components is being considered by biofuel industries as a good agent for biofuel production, several methods are being used for this conversion, Hydrothermal liquefaction found to be the most suitable for this process, this is because the products from the hydrothermal liquefaction are insoluble in water, more deoxygenated, and have higher energy densities which are beneficial to their uses as the liquid fuels. The bio-crude produced by the hydrothermal liquefaction has also shown high stability in accelerated aging tests. This article reviews on the liquid biofuel produce from the hydrothermal liquefaction process of lignocellulosic material, including the material sources and composition and the mechanism of the process.

**Keywords:** HTL; Lignocellulosic; Biomass; Biofuel; Pyrolysis**1.0 INTRODUCTION**

Global energy demand had been reported to growth rapidly every year, this is due to increase in population, economic growth and industrial expansion, The limited availability of fossil fuels and concern about environmental pollution such as greenhouse gases atmospheric concentration, toxic pollutants emission into the atmosphere and the fact that fossils fuel been projected to vanish by the year 2100 Stephens *et al.* (2010) have led the scientific community to look for alternative energy resources, renewables and safe for the environment, Hence, there is an increasing demand for renewable fuels commonly termed as CO<sub>2</sub> neutral fuels, Biomass is a renewable; CO<sub>2</sub>-neutral source of energy.

The Current main biomass sources for producing alcohol and diesel are based on food crops (such as maize, sugarcane, soybeans, rapeseed, and palm oil). Processing of this biomass requires long biological fermentation time and is criticized for

threatening food security Naik *et al.* (2010). Alternative to food crops, biomass from grass, wood, and other non-food components is being considered by biofuel industries. These components contain primarily cellulose, hemicellulose, and lignin and are called lignocellulosic biomass. Fast pyrolysis has been used to convert the lignocellulose biomass to useful products such as bio-oils. Nevertheless, so far the produced bio-oils by the fast pyrolysis have not been good enough to serve as transportation fuels due to their high oxygen content, low energy density, thermal instability, and high corrosiveness. Such properties lead to difficulty of ignition, coking upon heating, and degradation and clogging of engines and fuel filters Czernik and Bridgwater (2004). The bio-oils also show increases of viscosity and phase separation during storage Lu *et al.* (2009), posing difficulties for further application. Hydrothermal liquefaction (HTL) is a

thermochemical conversion of biomass into liquid fuels by processing in a hot, pressurized water environment for sufficient time to break down the solid biopolymeric structure to mainly liquid components. Hydrothermal technologies are broadly defined as chemical and physical transformations in medium-temperature (200–400°C), with high-pressure (5–40 MPa) water Peterson *et al.* (2008). HTL is similar to the pyrolysis but is carried out in water. It produces a bio-crude that is distinct from the bio-oil produced by fast pyrolysis. Generally, the products from the HTL are insoluble in water, more deoxygenated, and have higher energy densities Xiu and Shahbazi (2012), which are beneficial to their uses as the liquid fuels. The bio-crude produced by the hydrothermal liquefaction has also shown high stability in accelerated aging tests Elliott *et al.* (2015), alleviating some problems associated with the bio-oil by the fast pyrolysis. Furthermore, the process is also favored for converting wet biomass,

as much less energy is needed than that in the pyrolysis process in which more energy must be required to vaporize the inherent moisture Vardon *et al.* (2012). Experimental work also shows that water enhances oil yields, showing a potential of the HTL of lignocellulosic biomass in producing liquid biofuels and value-added chemicals Pütün *et al.* (2002). The HTL of total biomass yields a range of different products including bio-crude oil, aqueous dissolved chemicals, solid residue, and gas. HTL product yields and properties vary significantly according to the feedstocks processed as well as the reaction conditions employed Jazrawi *et al.* (2015). Although the product distribution in bio-oil varies with the composition of the raw material and process conditions, the same groups of compounds are detected in almost all HTL bio-oils Pavlovič and Škerget (2013). The liquid oils contain a range of chemicals including carboxylic acids, alcohols, aldehydes, esters, ketones, sugars, lignin-derived phenols, furans, etc. Stöcker (2008).

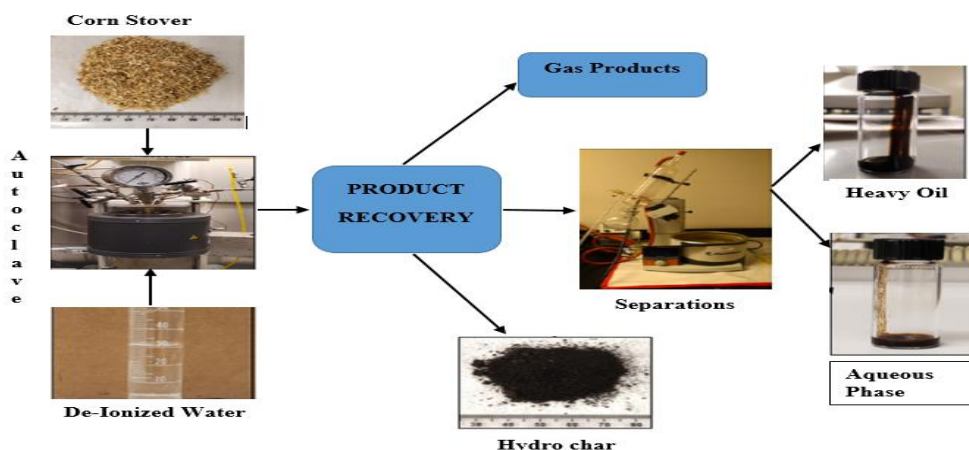


Figure 1: Hydrothermal liquefaction process from corn stover

## 2.0 SOURCES OF LIGNOCELLULOSE FEEDSTOCK

Lignocellulosic materials or feedstock refers to plant dry matter, so called lignocellulosic biomass. It is the most abundantly available raw material on the Earth for the production of biofuels, mainly bio-ethanol. It is composed of carbohydrate polymers (cellulose, hemicellulose), and an aromatic polymer (lignin). These carbohydrate polymers contain different sugar monomers (six and five carbon sugars) and they are tightly bound to lignin. Lignocellulosic biomass differs from other alternative energy sources because the

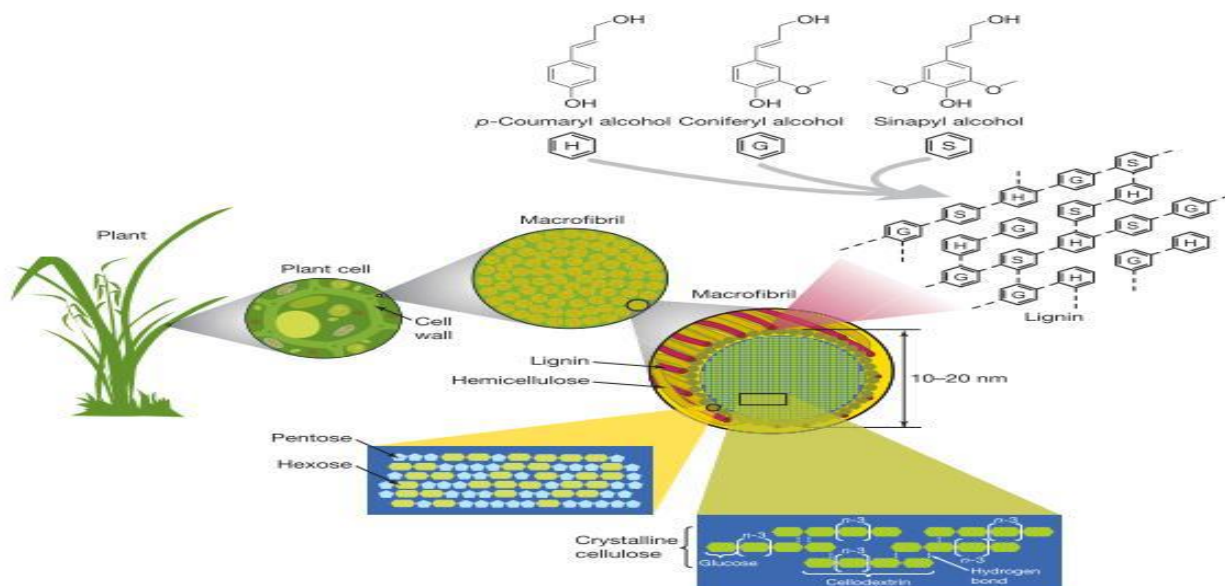
resource varies and their utilization to product stream are through many conversion processes Demirbaş (2001). In the context of lignocellulosic materials, it constitutes the most abundant biochemicals on earth Peterson *et al.* (2008). According to Kang *et al.* (2014) approximately 150EJ/J of energy are available in lignocellulosic materials, therefore, they could provide a sustainable source for low-cost biofuel production Sun (2002); Nagy (2009). Lignocellulosic biomass can be broadly classified into forest residue, Agricultural residue and energy crops. Forest residue includes all naturally occurring

terrestrial plants such as trees, bushes and grass. Agricultural residue is produced as a low value byproduct of various industrial sectors (corn stover, sugarcane bagasse, straw etc.) and forestry (saw mill and paper mill discards). Energy crops are crops with high yield of lignocellulosic biomass produced to serve as a raw material for production of second-generation biofuel; examples include switch grass (*Panicum virgatum*) and Elephant grass. Plants exhibiting C4 photosynthesis are amongst the most promising dedicated energy crops as they possess tremendous intrinsic efficiency in converting solar energy to biomass. Weijde *et al.* (2013) provide an excellent overview of the potential of five C4 grasses from the Panicoideae clade (maize, *Miscanthus*, sorghum, sugarcane, and switchgrass) as lignocellulosic feedstock for the production of biofuels. The authors discuss yield potential, biomass quality and genetic improvement of dual-purpose food and energy cultivars and dedicated energy cultivars through plant breeding and also highlight several research needs. Wang *et al.* (2013) review carbon partitioning in sugarcane, which has a source-sink system that creates high concentrations of easily extracted and economically valuable stem sucrose. Nonetheless, the majority of carbohydrate in sugarcane is lignocellulose. A detailed understanding of the molecular, and physiological processes underlying the partitioning of carbon assimilates will provide targets to manipulate the balance between sucrose and lignocellulosic biomass as carbon sinks. These include sugar transport and localization and the engineering of novel sugar sinks. This classic example of a possible dual-purpose crop can also be improved through breeding and genetic engineering of cell wall properties to further optimize biofuel production. Slavov *et al.* (2013) review the current knowledge about cell wall genetics, chemistry and structure in *Miscanthus*. This includes the prospects of developing detailed molecular genetics and biochemical models of pathways relevant to

biomass conversion efficiency. The life history and genome complexities exhibited by the *Miscanthus* species in question dictate that genome wide association studies are a necessary approach toward the genetic dissection of these traits. Potential targets for biomass improvement include cell wall regulatory genes, intercellular trafficking, and microtubule organization. Opportunities exist to functionally test gene-trait associations for cell wall quality in this bioenergy crop, short-term progress toward understanding of the molecular underpinnings of cell wall quality traits in *Miscanthus* will be driven by research in model grasses. *Setaria viridis* is a rapid cycling C4 panicoid grass with several attributes that make it an excellent model for bioenergy grasses. Petti *et al.* (2013) describe the composition and saccharification dynamics of *S. viridis* aboveground biomass as similar to sorghum, maize, and switch grass, confirming its potential as model species for panicoid translational genomics. Another grass proposed as a model for energy grasses, forage grasses and cereals is *Brachypodium distachyon*. Rancour *et al.* (2012) present chemical composition data of cell walls from distinct organs and developmental stages. Results indicate similar cell wall composition to those previously determined for a diverse set of C3 forage grasses and cereals, highlighting the usefulness of *B. distachyon* as a model for temperate grasses. As with *S. viridis*, the authors report some differences for particular wall traits.

### 3.0 Lignocellulosic biomass Composition

The chemical composition of lignocellulosic biomass varies depending on the specific needs of the plants. Cellulose, hemicellulose and lignin are the three main components of lignocellulosic source, and the proportion of these components in a fiber depends on the age, fiber sources and the extraction conditions used to obtain the fiber Reddy and Yang (2005).



**Figure 2: Structure of lignocellulosic materials; comprised of cellulose, hemicellulose and lignin adapted from Genomics of cellulosic biofuels Rubin (2008).**

Cellulose is the major component of lignocellulosic biomass, and almost half of the organic carbon in the biosphere is estimated to be present in the form of cellulose. Cellulose is the structural basis of plant cells and the most important and abundant natural substance (Zheng *et al.* (2013) which usually exists as long thread like fibres called microfibrils. The amount of cellulose influences the properties, economics of fibre production and the utility of the fibre. For example, higher cellulose content in fibres are more preferable for textile, paper and other fibrous applications. Meanwhile, fibre with higher hemicellulose content would be beneficial in producing ethanol and other fermentation products because hemicellulose is relatively easily hydrolysable into fermentable sugars. Therefore, the value for the cellulosic materials are largely determined by their end use applications Reddy and Yang (2005). However, it is difficult to convert the cellulose into end product because cellulose is insoluble in water and in most organic solvents under mild conditions. Cellulose is insoluble in water because of its low-surface-area crystalline form held together by hydrogen bonds Yu *et al.* (2008). Furthermore, since cellulose contains various kinds of C–O and C–C bonds, the precise cleavage of specific C–O and/or C–C bonds for

the production of specific chemicals is a challenging task Deng *et al.* (2015).

Hemicellulose is the second most common polysaccharide in nature and accounts for about 15–35% of lignocellulose, depending on the biomass type and source Pavlovič *et al.* (2013). Hemicellulose was estimated to account for approximately 22% of softwood, 26% of hardwood and 30% of various agricultural residues Ganesh (2012). Compared to cellulose, hemicellulose is short, highly branched polymer consisting of five-carbon (C5) and six-carbon (C6) sugars monomer such as glucose, xylose, mannose, galactose and arabinose and uronic acids Pasangulapati *et al.* (2012). Due to its structure and branched nature, hemicellulose is amorphous and relatively easy to hydrolyse to its monomer sugars compared to cellulose Yu *et al.* (2008). Hemicellulose is insoluble in water at low temperature. However, hemicellulose hydrolyse at a temperature lower than cellulose; which at elevated temperature their solubility is rendered Harmsen and Huijgen (2010). Lignin is widely found in plants and its production ranks after cellulose, making it the most abundant renewable organic resources with aromatic characteristics in nature Cao *et al.* (2018). Lignin is a complex and non-crystalline three-dimensional network of phenolic polymers, which widely exist in higher plant cells. Lignin is the most invasive component of the plant cell wall, given its role in providing tensile strength to the plant. It is prevalently bonded to hemicellulose, which helps

stabilize all three biopolymers, and thereby enhances the rigidity of the wall Laskar *et al.* (2013). Compared to other biopolymers, lignin is a polymer network resulting from the dehydrogenated radical polymerisation of monolignols (e.g. p-coumaryl-, coniferyl- and

sinapyl-alcohols), connected through carbon-carbon and ether connections Boeriu *et al.* (2004). Lignin accounts for about 15–40% of the dry weight of lignocellulosic biomass and represent as one of the main components in plants Cao *et al.* (2018).

**Table 1: Cellulose, hemicellulose and lignin composition in common lignocellulosic materials adapted from Lee et al. (2014).**

Types of biomass	Lignocellulosic substrate	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Agriculture waste	Corncoobs	45	35	15
	Wheat straw	30	50	15
	Barley straw	33–40	20–35	8–17
	Corn stover	39–42	22–28	18–22
	Nut shells	25–30	25–30	30–40
Energy crops	Empty fruit bunch	41	24	21.2
	Switch grass	45	31.4	12
Forestry waste	Hardwood stems	40–55	24–40	18–25
	Softwood stems	45–50	25–30	25–35
	Leaves	15–20	80–85	0
Industrial waste	Waste papers from chemical pulps	60–70	10–20	5–10
	Organic compound from wastewater solid	8–15	0	0

#### 4.0 HTL MECHANISM FOR BIOFUEL PRODUCTION FROM LIGNOCELLULOSIC BIOMASS

The mechanism of the hydrothermal liquefaction has not been elucidated much in the literature. The pathway of HTL comprises three major steps, depolymerisation followed by decomposition and recombination, biomass is decomposed and depolymerized in to small compounds. These compounds may be highly reactive, thus polymerizing and forming bio crude, gas and solid compounds is essential. Further the critical process parameters such as temperature, residence time, the process of repolymerization, condensation and decomposition of the components from the different phases may vary accordingly. Since biomass is a complex mixture of carbohydrates, lignin, proteins, and lipids, the reaction chemistry and mechanisms of biomass liquefaction are consequently also complex McKendry (2002). Depolymerisation of biomass is a sequential dissolving of macromolecules through utilization of their physical and chemical properties. The intrinsic hemicellulose and cellulose biopolymers contributes positively towards the thermal stability of the biofuel Jae *et al.* (2010). Depolymerisation process overcomes the difficulties and obstinate properties of lignocellulose biomass that mimics the natural geological processes of producing fossil

fuels. The decisive parameters temperature and pressure changes the structure of the long chain polymers consisting of hydrogen, oxygen, and carbon to shorter chain hydrocarbons Zein and Winter (2000). Further, the energy contents of the organic materials are recycled in the presence of water. Decomposition step involves the loss of water molecule (dehydration), loss of CO<sub>2</sub> molecule (decarboxylation) and removal of amino acid content (deamination) Jena *et al.* (2015). The dehydration and decarboxylation facilitate the removal of oxygen from the biomass in the form of H<sub>2</sub>O and CO<sub>2</sub> respectively. Biomass comprising macromolecules are hydrolysed to form polar oligomers and monomers Martin and Kornülle (2011). Water at high temperatures and pressure breaks down the hydrogen bonded structure of cellulose and causes the formation of glucose monomers. Because fructose is more reactive than glucose, it rapidly degrades in plethora of products by the different types of reactions, including isomerization, hydrolysis, dehydration, reverse-aldol defragmentation, rearrangement and recombination reactions Zhang and Wilson (2016). The majority of the degradation products such as polar organic molecules, furfurals, glycoaldehydes, phenols and organic acids are highly soluble in water. Recombination and repolymerization of reactive fragments This is the third step in which

the reverse to the initial processing steps are happening due to the unavailability of the hydrogen compound. If hydrogen is freely available in the organic matrix for the liquefaction process the free radicals will be capped yielding the stable molecular weight species. At conditions the unavailability of the hydrogen or the concentration of the free radicals are excessively large, the fragments are recombined or repolymerized to form high molecular weight char compounds otherwise noted as coke formation Shah (2015). HTL is a biomass to bio-liquid conversion route carried out in water at moderate temperature of 280–370 °C and high pressures (10–25 MPa) that has a liquid bio crude as main product along with the gaseous, aqueous and solid phase byproducts Toor, (2010). The product stream of this process

have high energy content and unique feature of enhanced heat recovery in comparison to other processes Choudhary, (2011). Many complex reactions take place during the transformation of biomass into crude oil like products Gollakota *et al.* (2016). In HTL of lignocellulose biomass the reactor design in the hydrothermal processing can be either batch or continuous. Continuous reactors require feeding system that operate under pressure and include either slurry based pump or lock and hopper systems for large particles. The feedstock requires pre-treatment especially the woody biomass for the reasons of reducing the particle size, removing the contaminants, and alkaline treatment to obtain a stable slurry for easy pumping. The feedstock is then processed via HTL at temperature of around 350 °C.

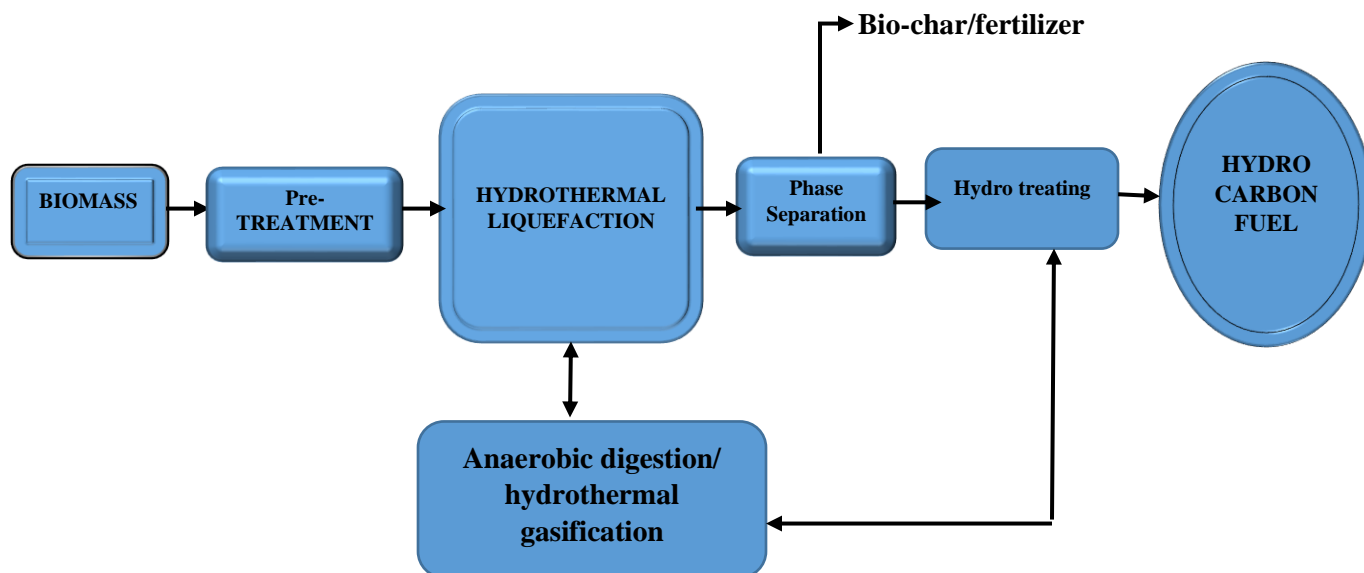


Figure 3: Hydrothermal liquefaction mechanism of lignocellulose biomass

## 5.0 CONCLUSION

This review has demonstrated that HTL process as a promising technique and excellent conversion method for lignocellulosic biomass to produce biofuel and useful chemicals and valuable products

such as carboxylic acids, alcohols, aldehydes, esters, ketones, sugars, lignin-derived phenols, furans. And compare with the other methods of conversion HTL produces oil with higher stability which is one of the problem when using pyrolysis

as a method of conversion, Furthermore, the process is also favored for converting wet biomass, as much less energy is needed than that in the pyrolysis process in which more energy must be required to vaporize the inherent moisture

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