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The Effect of Pd-Doped λ -Al₂O₃ on the Low-Temperature Thermal Decomposition of Corn Cobs and Leaves in Bio-oil Production

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Abstract:

Recently, biomass has become one of the most important renewable energy sources. Corn cobs, stalks, leaves, and other agricultural residues are produced in large quantities annually. However, only few studies have considered the use of these cheap materials as a source of biomass. In the current study, lignocellulose from dry corn cobs and leaves was thermally decomposed at low temperatures (150-250 °C) for different pyrolysis time intervals (30-120 min) in an in-house assembled reactor. The pyrolysis process was conducted in the absence and presence of Pd-doped -Al₂O₃ catalyst. The bio-oils produced were characterized and classified using gas chromatography/mass spectrometry (GS/MS). The GS/MS results showed that the bio-oils produced contained: carboxylic acids, alcohols, hydrocarbons, guaiacols, miscellaneous oxygenates, and multicomponent compounds. The results also showed that the presence of the catalyst increases the bio-oil yield by at least 50%. In addition, in presence of the catalyst, the leaves produced more bio-oils than the cobs.

Keywords: Conventional pyrolysis, Pd-doped -Al₂O₃, corn cobs, corn leaves, unextracted lignocellulose, thermal decomposition, bio-oil, char, retention time, catalyst

1. Introduction

The global energy demand along with the current trends in legislative restrictions and environmental concerns regarding CO₂ emission have stimulated the search for other energy sources that are environmentally friendly, cheap, and renewable (Antal, 1978). Among the alternatives to fossil fuels, biomass has become one of the most important renewable energy sources due to its abundance, carbon-fixing, and carbon-neutral properties (Bridgwater, 2012; Maggi & Delmon, 1994; Pattiya, Titiloye, & Bridgwater, 2008). However, the applications of raw biomass in the industry are limited because it is highly hygroscopic, has high moisture content, and has low volume and energy density (Chen & Lu, 2003; Chen & Wu, 2009; Fiaschi & Carta, 2006; Uemura, Omar, Tsutsui, & Yusup, 2011). Biomass is considered the largest renewable and most economical energy source that is utilized worldwide in underdeveloped, developing, and developed countries (Werther, Saenger, Hartge, Ogada, & Siagi, 2000). It is more abundant than most renewables such as wind and hydropower (Chen & Kuo, 2011). In addition, biomass offers significant life-cycle and greenhouse gases (GHG) savings, compared to fossil fuels. The CO₂ produced from burning biomass is captured during plant growth and therefore, it is perceived to be carbon-neutral and able to reduce the net carbon dioxide emissions when it displaces the use of fossil fuel (Deng, Wang, Kuang, Zhang, & Luo, 2009).

Parallel with the growing interest in biomass as an alternative source of fuel and chemical feedstock, there is an increased demand for innovative technologies for the conversion of biomass to bio-oils (Chen & Kuo, 2011; Deng et al., 2009). Among the currently used conversion processes are: pyrolysis, anaerobic digestion, hydro-carbonization, incineration, and combustion under controlled atmosphere.

Fast pyrolysis of biomass is a thermal decomposition process that takes place in the absence of oxygen that converts biomass into liquid products (bio-oils) along with non-condensable gases and solids (chars) (Chiaromonti, Oasmaa, & Solantausta, 2007; Czernik & Bridgwater, 2004). The major products of the pyrolysis depend on both the temperature and residence time. High temperatures along with long residence times usually produce gases. On the other hand, moderate temperatures and short residence times are recommended to produce liquids [<http://www.sciencedirect.com/science/article/pii/S0961953411000638>]

The use of biomass as an alternative energy source has also raised the controversial issue of depletion of food crop harvests as well as natural forests. This issue can be solved by using farm wastes. Corn is one of the most important crops worldwide and as such, a

considerable amount of corn cob wastes are produced annually (Lu & Chen, 2014). However, only very few studies have considered the use of corn cob and other agricultural wastes as sources of biomass. The utilization of these “wastes” for the production of bio-oils and chemical feedstock offers a route to economic diversification which can enhance the standard of living for the Nigerians.

Bio-oil is considered to be a promising inexpensive and clean alternative fuel that can replace petroleum fuels for use in various thermal devices for the generation of heat and power (Bridgwater & Peacocke, 1999; Mohan, Pittman, & Steele, 2006). Generally, bio-oils are viscous, acidic, thermally unstable, and contain a high proportion of oxygenated compounds. The quality of bio-oils can be improved by the addition of catalysts into the pyrolysis process. The catalyst is expected to enhance the cracking reactions of the heavy molecules in pyrolysis products leading to the production of less viscous bio-oils, reduce the formation of carboxylic acids in order to make bio-oil less corrosive, enhancing the formation of more valuable products, like hydrocarbons, that can increase the heating value of bio-oil.

The current study aims at utilizing the waste of the corn plant, which is extensively grown in Nigeria, to generate bio-oils. Since bio-oil can be produced from indigenous wastes, it can have a significant impact on both the economy and the environment by creating local jobs and saving the cost of importing energy.

To the best of our knowledge, there are only few reports in the literature on the study of low temperature pyrolysis of corn cobs and leaves. Moreover, none of these reports investigated the effect of a catalyst on the quantity of the bio-oil produced. In the current investigation, lignocellulose from dry corn cobs and leaves was thermally decomposed at low temperatures (in the 150-250 °C range) for different pyrolysis time intervals (up to 2 hours) in an in-house assembled reactor. The pyrolysis process was conducted in the absence and presence of Pd-doped $\text{-Al}_2\text{O}_3$ catalyst. The bio-oils produced were characterized, using gas chromatography/mass spectrometry (GS/MS) and then classified.

2. Experimental

2.1. Preparation of Crushed Corn Cobs and Leaves

Dry corn cobs and leaves were randomly collected and sampled from some local farms in the city of Jos, Nigeria. The samples were fractionated, washed in water, and dried in a Gallenkamp vacuum oven at a temperature of 105 °C for 2 hours to remove the moisture. The samples were, then cut individually into smaller pieces and crushed using a Bico pulverizing machine. The crushed samples were sieved through a mesh size of 250-300 μm , in readiness for pyrolysis.

2.2. The Reactor

The reactor used in the study was designed and assembled in house. Fig. 1 shows a schematic representation of the reactor. As shown in the figure, the reactor consisted of Pyrex tubes (16 cm length and 1.5 cm internal diameter). The heating medium was a cylindrical brass block of 9 cm height and 7 cm diameter into which three thermal wells were drilled at approximately equal distance apart. Each well had a depth of 7 cm and 1.7 cm diameter. The block was electrically heated, using a hotplate equipped with a temperature regulator.

A thermometer was placed in one of the wells to monitor the temperature of the system. Pyrolysis samples contained in two different test tubes were dipped into the remaining two thermal wells. The tubes were connected, via rubber stoppers and glass tubes, to water-filled, inverted burettes placed in a trough for the collection of the gas evolved in the reaction (Ekpenyong, 2005).

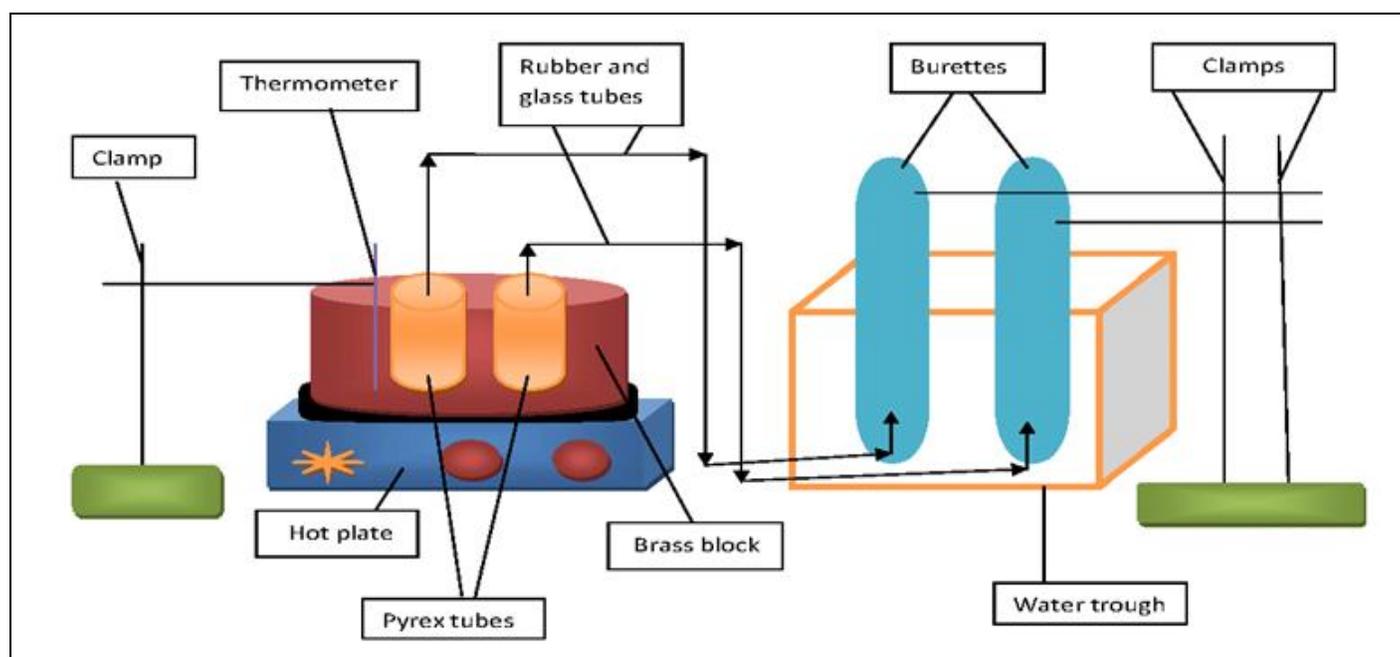


Figure 1: A schematic representation of the reactor

2.3. Pyrolysis in the Reactor

Sieved 3.00 g samples each, of corn cobs and leaves were placed individually in 2 Pyrex tubes. The tubes were, then, placed in the wells of the already heated metallic heating block. Each pyrolysis was carried out at 150 °C, 200 °C, and 250 °C. In the catalyzed pyrolysis, Pd-doped $\text{-Al}_2\text{O}_3$ was ground and sieved to 250-300 μm . The catalyst (3.00 mg per 3.00 g of the sample) was then homogenized in a mortar with the crushed corn cobs and leaves individually. The blend was then placed in the test tube in readiness for pyrolysis. Two samples of the same material were used in each pyrolysis, and the average volume of the bio-oil produced was determined and recorded.

2.4. The Extraction Process

After pyrolysis, each sample was allowed to cool in the test tube. The cooled sample was, then, introduced into a 500-mL Soxhlet extractor. The extraction was carried out in n-hexane for 2 hours in each case. The solvent was evaporated leaving the bio-oil. The volume of the bio-oil produced was determined using a syringe.

2.5. Detection of Bio-Oil Compounds, Using GC/MS

To identify the components of the bio-oil, GC analysis was performed using a GC/MS as follows: a 0.20 g of each bio-oil sample obtained was dissolved separately in 10.0 mL of methylene chloride. Then, a sample of 2.4 mL of each solution was injected separately into the GC/MS and allowed to run for 67 min. The components of the bio-oil sample then passed into the mass spectrometer for detection. The compounds detected by the GC/MS were identified from the MS library and characterized.

3. Discussion

3.1. Uncatalyzed Pyrolysis

The products of biomass pyrolysis include biochar, bio-oil, and gases. The relative yield of products varies depending on the thermal environment and the pyrolysis temperature. At low temperatures, mainly biochar is produced. At temperatures higher than 800 °C, the pyrolysis yield mainly gases. At intermediate temperatures, bio-oil is the major product.

In the current study, the pyrolysis was conducted for both the corn cobs and corn leaves at selected temperatures (up to 250 °C). Figs. 2 and 3 show the volumes of bio-oil produced in the uncatalyzed pyrolysis of corn cobs and leaves at 200 °C and 250 °C, respectively. At 250 °C (Fig. 3), more bio-oil volumes were produced by both samples with the bio-oil yield for both samples is about 50% more than that produced at 200 °C. Moreover, Figs. 2 and 3 show that at both temperatures, the cobs produced more bio-oil than the leaves. In both samples, bio-oil volumes were recorded at a 30 min retention time. In all the experiments, it was observed that the bio-oil yield increased with increasing retention time.

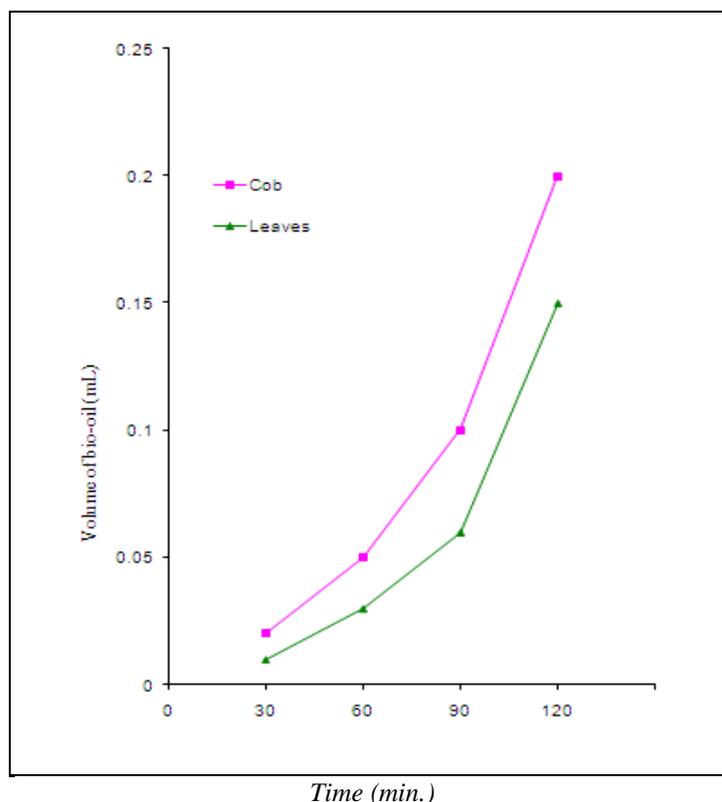


Figure 2: Variation of the volume of bio-oil produced in the pyrolysis of corn cobs and leaves vs. time at 200 °C

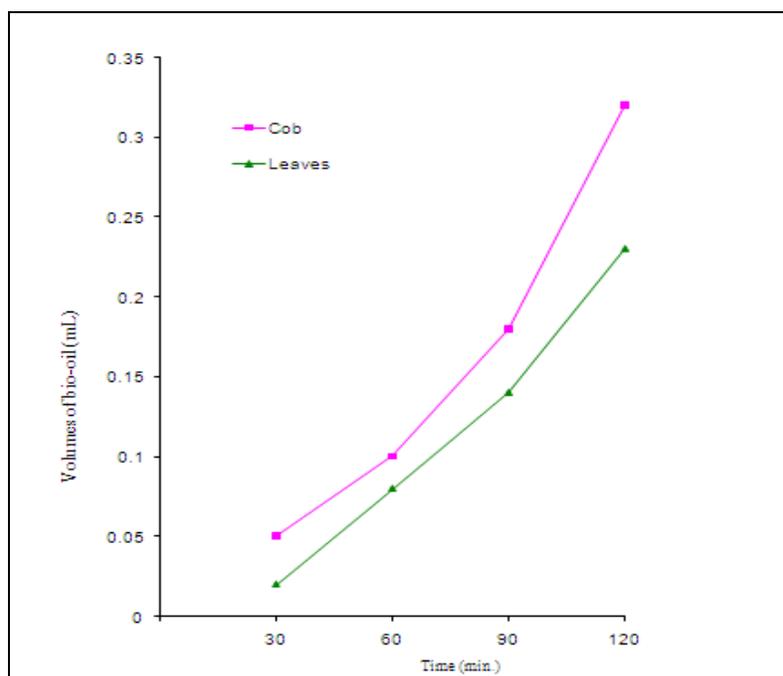


Figure 3: Variation of the volume of bio-oil produced in the pyrolysis of corn cobs and leaves vs. time at 250 °C

3.2. Catalyzed Pyrolysis

Fig. 4 shows the effect of Pd-doped $\text{-Al}_2\text{O}_3$ on the volumes of bio-oil produced in the pyrolysis of corn cobs and leaves at 150, 200, and 250 °C. In general, the volumes of the bio-oil produced from the catalyzed pyrolysis were larger than those produced from the uncatalyzed pyrolysis. In addition, the leaves produced more bio-oil than the cobs for all studied temperatures.

It is interesting to notice that small volumes of bio-oil were formed when the catalyzed pyrolysis was carried out at a temperature as low as 150 °C. At 200 °C, the bio-oil volumes recorded in the catalyzed pyrolysis are higher than those recorded in the uncatalyzed pyrolysis at 250 °C at the maximum retention time. At 250 °C, relatively higher volumes of bio-oil were recorded. In this experiment, the pyrolysis was stopped at 2 hours retention time. The bio-oils obtained were oily liquids with color ranging from light brown to darker brown, depending on the pyrolysis temperature.

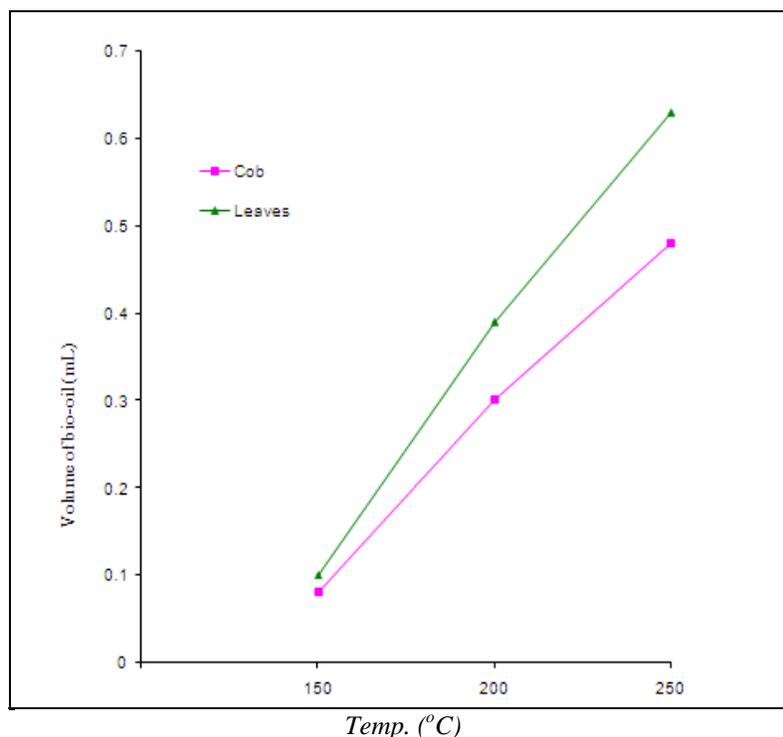


Figure 4: Effect of the pyrolysis temperature on the volume of the bio-oil produced in the catalytic pyrolysis of corn cobs and leaves. The pyrolysis time = 120 min

3.3. Determination of percentage conversion of biomass to bio-oil

Knowing the density of the corn stover bio-oil (1.075 g/mL) and the volume of bio-oil obtained from the catalyzed corn leaves, the percentage of bio-oil formed was determined (Hill, 1984; Kotz, 1991; Robinson, 1992). The percentage of conversion of the catalyzed biomass to bio-oil is about 23% at 250 °C. Taking into consideration the relatively low pyrolysis temperature, this % conversion is acceptable.

3.4. Classification of the bio-oil compounds detected in the catalyzed and uncatalyzed pyrolysed corn cobs and leaves

The GS/MS spectra revealed the compounds present in the bio-oil produced from the corn cobs and leaves. Some peaks were readily assigned while others were assigned with the help of the MS library. The data are summarized in Table 1. In the uncatalyzed samples, the most abundant compounds detected are: dichloroacetic acid and n-hexadecanoic acid. Compounds like amylene hydrate, tetradecanoic acid, pentadecanoic acid, octadecanoic acid and 1,2 benzenedicarboxylic acid, diisooctyl ester were detected in all the catalyzed and uncatalyzed pyrolysed samples. However, dodecanoic acid and 3-isopropyl-2-methoxy-5- methylbenzoic acid were detected in the catalyzed and uncatalyzed corn cob samples while benzaldehyde,4- hydroxy-3,5-dimethoxy was detected in the catalyzed and uncatalyzed pyrolysed corn leaves samples only. Dammar-22-en-3-ol,20,24-epoxy-24-methylacetate(3 β ,24s)-, 1-piperidinecarboxaldehyde, ethanone, 1-(4-hydroxy-3-methoxy phenyl)- and ethanone, 1-(4-hydroxy-3, 5-dimethoxy phenyl)- were detected in the bio-oil produced from the pyrolysis of uncatalyzed corn leaves only.

Retention Time, t_R (min)	Library Match	Peak Area (%)			
		Catalyzed Corn Cobs	Uncatalyzed Corn Cobs	Catalyzed Leaves	Uncatalyzed Leaves
2.10	Ethaneperoxoicacid,1-cyano-4,4-dimethyl-1-phenylphenyl ester		1.38		
2.17	Dichloroacetic acid	1.29		0.45	
3.32	Dammar-22-en-3-ol, 20,24-epoxy-24-Methylacetate(3 β 24s)-				0.38
3.68	Amylene hydrate	0.90	0.33	3.83	0.23
6.17	Methyl cyclohexane-	1.38		2.73	0.39
20.98	1-Piperidinecarboxaldehyde				0.34
30.66	Ethanone,1-(4-hydroxy-3-methoxy phenyl)-				0.44
32.34	Dodecanoic acid	0.24	0.52		
34.77	Benzaldehyde,4- hydroxy-3,5-dimethoxy			4.27	0.38
35.66	3-Isopropyl-2-methoxy-5-methylbenzoic acid	0.69	0.35		
36.41	Ethanone,1-(4-hydroxy-3,5-dimethoxy phenyl)-				0.41
36.98	Tetradecanoic acid	1.13	2.64	2.38	4.06
39.60	Pentadecanoic acid	0.26	0.45	0.14	0.69
41.37	n-Hexadecanoic acid				
45.17	Octadecanoic acid	4.48	6.51	8.88	10.14
51.44	1,2-Benzenedicarboxylic acid, diisooctyl ester	1.99	2.19	1.67	2.83

Table 1: List of the bio-oil compounds detected in the catalyzed and uncatalyzed pyrolysed corn cobs and leaves at 250 °C

It is interesting to notice that the detected acids in the catalyzed pyrolysed samples had smaller peak area than the acids detected in the uncatalyzed pyrolysed samples. Also, the peak area of the hydrocarbon detected in the catalyzed samples was larger than that of the uncatalyzed samples. This shows that the catalyst employed was able to reduce the formation of carboxylic acids in order to make bio-oil less corrosive and enhance the formation of more valuable products, such as the hydrocarbons, that can increase the heating value of the bio-oil. The detected compounds were classified into: acids, alcohols, hydrocarbon, miscellaneous oxygenates, and multicomponent compounds (Table 2). The bio-oil compounds detected in this work are similar to those detected in corn stover pyrolysis works done previously (Zhu, Zhu, Guo, & Zhu, 2006) Bio-oil from corn stover contains several carboxylic acids, multi-ring aromatic compounds, a few aromatic hydrocarbons, polar compounds (phenols and its derivatives) as well as multiple component compounds (Li, Xu, Lu, & Liu, 2005; Yao, Chen, Zhang, Li, & Wan, 2000; Zhu et al., 2006).

Class	Compound
Carboxylic acids	Dodecanoic acid
	Tetradecanoic acid
	n-Hexadecanoic acid
	Octadecanoic acid
	Pentadecanoic acid
Alcohols	Amylene hydrate
Hydrocarbons	Cyclohexane, methyl-
Guaiacols	Ethanone, 1-(4-hydroxy-3-methoxy phenyl)-
	Ethanone, 1-(4-hydroxy-3, 5-dimethoxy phenyl)-
	Benzaldehyde,4-hydroxy-3,5- dimethoxy-
Miscellaneous oxygenates	1, 2-Benzenedicarboxylic acid, diisooctyl ester
	Dammar-22-en-3-ol,20, 24-epoxy-24- methylacetate(3 β ,24s)
	3-Isopropyl-2-methoxy-5-methylbenzoic acid
Multicomponent mixtures	Dichloroacetic acid-
	1-Piperidinecarboxaldehyde
	Ethaneperoxoicacid,1-cyano-4,4-dimethyl-1-phenylphenyl ester

Table 2: Classification of bio-oil compounds detected in the catalyzed and uncatalyzed pyrolysed corn cobs and leaves at 250 °C

During the bio-oil production, a large number of reactions take place. These include hydrolysis, dehydration, isomerization, dehydrogenation, aromatization, retro-condensation, and coking (Diebold, 2000; Shafizadeh, 1982). More than 400 organic compounds have been found previously in bio-oil (Shafizadeh, 1982). These compounds can vary by more than one order of magnitude. The multicomponent mixtures are derived primarily from depolymerization and fragmentation reactions of the three building blocks of lignocellulose: cellulose, lignin, and hemicellulose. The guaiacols and syringols are formed from the lignin fraction, whereas the miscellaneous oxygenates (sugars and furans) are formed from the cellulose and hemicellulose. The esters, acids, alcohols, ketones, and aldehydes are likely formed from the decomposition of the miscellaneous oxygenates, sugars and furans (Shafizadeh, 1982). Syringols that are normally formed during lignin decomposition were not detected in the analyzed bio-oil samples produced in the current study. This is mainly because the low temperatures used in the study (250 °C or lower) are not high enough to break down lignin extensively.

4. Conclusion

Fast pyrolysis at 450-500 °C has the potential of contributing to the world's need for liquid fuels and will ultimately be used for chemical production. However, smaller volumes of bio-oil can also be produced by low-temperature conventional pyrolysis using a simple reactor. In the current investigation, at temperatures of 150, 200, and 250 °C, corn cobs and leaves were pyrolysed in the absence and in the presence of Pd-doped -Al₂O₃ as a catalyst. The results showed that in the uncatalyzed samples, the cobs produced higher volumes of bio-oil volumes than the leaves. However, when the catalyst was used, the leaves produced about double the volume recorded in the uncatalysed samples. Catalyzed pyrolysed leaves were found to have ~22.5% conversion of biomass to bio-oil at 250 °C. The bio-oil samples obtained were characterized using a GC/MS and found to contain esters, acids, alcohols, phenols, alkanes, guaiacols, multicomponent compounds, and miscellaneous oxygenates. The bio-oils samples obtained are comparable with those produced by other processes.

5. References

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