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Biomass Studies on Pyrolysis of Sugarcane Bagasse and Cashew Nut Shell for Liquid Fuels

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Abstract

The fast pyrolysis of biomass is one of the most recent renewable energy processes, which offers the advantages of liquid product, 'bio oil' that can be used as potential fuel after modification. This liquid has a number of special features and characteristics, which negatively affect the fuel properties, like, high oxygen content, incompatibility with conventional fuels, heating value half that of hydrocarbon fuels, high viscosity causing difficulties in atomization, low pH causing corrosion, and high instability due to polymerization. The inorganic elements present in the ash are believed to be catalyzing the polymerization reactions and thereby increase the viscosity. The pretreatment of bagasse for deashing have taken up for the present study and investigated for the effect of deashing on product distribution and characteristics, specially on oil. Three different types of pretreatment, like water leaching, leaching with 5M HCl solution and with HF solution of different concentrations have been investigated. The study shows substantial increase in oil yield upon pretreatment. The leachates resulting from water leaching processes have also been found to be a potential source of ethanol, which in turn can be used for making the oil stable over time.

Key words: *Biomass, Sugarcane bagasse, Cashew nut shell, Bio oil, Deashing, Pyrolysis, Pretreatment, Stability.*

1. Introduction

Sugarcane is the world's largest agricultural crop. Located in tropical and subtropical areas of the world, over 1200 sugarcane factories in 80 countries grind and process nearly 800 Mt of Sugarcane [1-5]. Crushing 1000kg of sugarcane produces approximately 255 kg of moist bagasse (50 wt.% moisture content) – the waste left after the cane is crushed. India is the second largest producer of sugarcane next to Brazil with a production of 299.33 million tonnes of sugarcane in 1999-2000 [6-9]. Besides Brazil and India, Australia, South Africa, Cuba, China, tropical and subtropical countries also are major contributors to world production of sugarcane. Thus, sugarcane bagasse has a strong potential in displacing fossil fuels and can be extensively used in boilers, turbines and furnaces for power generation. Generating power by direct combustion of sugarcane bagasse in boilers has a maximum efficiency of about 26%. Combustion systems with low efficiency are traditionally used in sugarcane plants [10-12]. In populated areas, bagasse-fired boilers can be one of the major health hazards due to airborne fly ash. Recently, the overall efficiency of the process is being greatly improved by cogeneration, wherein; the improvement comes from the proper utilization of heat and effective waste heat recovery. On the other hand, pyrolysis offers an effective utilization of the "fuel energy" itself giving energy dense liquids (easier to handle, store and transport), charcoal (improved solid fuel) and gaseous fuel. The ability to decouple the fuel production from the application is unique to pyrolysis liquids and is a major advantage over gasification and combustion, which must use the energy products immediately and cannot store or transport them. Thus, transformation of bagasse into high-density renewable fuels, like charcoal and bio-oil, can significantly increase the profitability of sugarcane plantations [13-18]. Sugarcane bagasse pyrolysis has been referred to by many authors. The role of parameters like peak temperature and tar yield has been investigated [19-23]. Total condensates of the order of 40-60% have been reported on dry bagasse basis. Vacuum pyrolysis of sugarcane bagasse has been reported first by venu et.al. [24-28]. The recent studies reported by Perez et al. gives a very elaborate and extensive understanding about vacuum pyrolysis of sugarcane bagasse particularly, in the assessment of the yield and the product characteristics. The ageing tests reported therein gives an insight to the ageing process of the bagasse oil. This is of interest, particularly in its application as a fuel. The major deterring factor in the wide usage and acceptability of the bio oils are its change of physico-chemical characteristics during storage along with the corrosively in the commonly used storage medium. The oil undergoes polymerization, thus resulting in an increase in viscosity with time. During ageing, etherification and etherification reactions occur between hydroxyl, carbonyl and carboxyl group components [29-32]. It is also mentioned in literature [33-37] that this instability might be attributed to the presence of alkali metals in the ash, which are being carried over/entrained by the char particles with the vapors. These alkali metals catalyze the polymerization reactions and thereby increasing the viscosity. Moreover, these alkali metals in ash, form deposits in combustion applications, where the damage potential is considerably high. Therefore, the present study aims at understanding the influence of this ash on the stability as well as pyrolysis product yields upon vacuum pyrolysis of sugarcane bagasse.

2. Biomass Properties

The proximate and ultimate analysis of the sugarcane bagasse studied herein are given in **Table 1**

Table 1 Properties of sugarcane bagasse

Proximate analysis		**Ultimate analysis**	
Wt. % (on dry basis)		Wt. % (on daf* basis)	
Volatile Matter	84.83	C	56.32
Fixed Carbon	13.28	H	7.82
Ash	1.89	N	0.89
		O (By difference)	27.54

2.1 Effect of different deashing treatments on sugarcane bagasse

Each of the deashing treatments, processes detailed in **Table 1** is associated with mass reduction in the original biomass feedstock. The degree of deashing as well as the mass reduction of the original biomass associated with each process is found to be different. The average mass reduction on dry bagasse basis, for water treatments I, II and III was 18-20%, 26-27% and 26-27% respectively. The acid leaching caused higher mass reduction with as high as 50 wt. % for treatment IV and an average mass reduction of 29-32% for treatment. To understand the nature of change in the chemical composition in sugarcane bagasse due to different pretreatments the chemical composition (extractives, hemicelluloses, cellulose and lignin) of all biomass (untreated and treated) was also carried out by Technical Association of Pulp and Paper Industries methods. The results may be interpreted as follows. In case of water leaching, the extractives are being washed out and thereby reducing the wt.% of extractives (for I hour water leaching) from 25.8 to 13.3. This is attributed to the fact that unlike woody biomass, extractives of sugarcane bagasse comprise largely of starch, sugars, phenolic tannins, and are washed out in simple water leaching. Leaching with 5(M) HCl solution hydrolyses the hemicelluloses fraction to large extent, leading to a drastic reduction of hemicelluloses and extractives with a resultant increase in the cellulose percentage. Similarly, in case of HF treatment apparent percentage of cellulose increases as a result of reduction in hemicelluloses and extractives. It is appropriate to mention here that the reduction in the extractives depends on amount of extractive present originally, which in turn depend on bagasse generation process. It is seen that, the HF treated samples do not show any significant changes in HHV with respect to untreated sample, while the water leached and 5(M) HCl treated sample have a slight lower value. This is attributed to the higher cellulose to lignin ratio for water leached and HCl treated sample, compared to normal and HF treated bagasse, as it is believed that high lignin content translate into high energy content fuels [38-43].

The elemental composition of ash was obtained by Inductively Coupled Plasma Atomic Emission Spectra instrument (ICP-AES) and presented in **Table 2**. In accordance to reported literature [44-48], simple water leaching washes out the alkalis like Na &K wherein, 5M HCl leaching further removes other alkali metals like Mg, Ca, Al etc., but HF treatment removes almost all the ash elements.

Table 2 Elemental composition of untreated and treated sugarcane bagasse

Elements	Untreated bagasse	Water leached (1 hour) bagasse	5(M) HCl treated bagasse	3% HF treated bagasse
Na	0.01160	0.00310	0.00750	-
K	0.17510	0.01430	0.02000	-
Ca	0.08700	0.01700	0.04000	0.02
Mg	0.43740	0.31000	0.28450	0.002
Al	0.00250	0.00190	0.00400	-
Fe	0.00370	0.00270	0.00500	-
Zn	0.00070	0.00100	0.01400	-
Cr	0.00360	0.00005	ND	-
Cu	0.00060	0.00092	0.03000	-
Mn	0.14070	0.00025	ND	-
Ni	0.00030	ND	0.00030	-
P	0.01430	0.00300	0.00210	-
Si	0.91100	0.86150	1.69900	-
S	0.04240	0.02600	ND	-

3. Effect of deashing treatments on pyrolysis product distribution of sugarcane bagasse

The effect of deashing treatments on sugarcane bagasse pyrolysis product distribution (char, gas, and total liquid) and on oil yield (based on treated bagasse basis) has been shown in Figure 1 represents the same on original bagasse basis, i.e. all values are inclusive of mass reduction during leaching [49-52]. As expected, the pyrolysis product distribution relates well to the chemical composition of bagasse as follows. In case of water leaching, the extractives are being washed out and thereby reducing the wt.% of extractives from 25.8 to 13.3 (even for 1 hour water leaching). This is attributed to the fact that unlike woody biomass, extractives of sugarcane bagasse comprise largely of starch, sugars, phenolic tannins, and are washed out in simple water leaching. By virtue of above, though the actual amount of cellulose is not changed, the relative percentage of the same has increased in the water leached bagasse. Leaching with 5(M) HCl solution hydrolyses the hemicelluloses fraction to a large extent leading to a drastic reduction of hemicelluloses and extractives with a resultant increase in the apparent cellulose percentage in the treated bagasse. In this case, the percentage mass reduction is so high (~50%) that the reduction in ash is not manifested, rather it actually seems to be increasing. Therefore, the percentage increase in the oil yield is marginal and is not as high as it should have been, had the ash been removed completely [53-57].

This aspect is confirmed by leaching with HF solution. Leaching with HF not only increases the relative percentage of cellulose in the treated bagasse by removing extractives and hemicelluloses, but also completely removes ash elements. The increase in the oil percentage in this case is by about 69 % (based on original untreated biomass) and by ~145 (based on treated bagasse basis). A special reference has to be made to explain the increase in the oil percentage for treated bagasse. It is well reported that upon deashing, both the amount of volatiles and the rate of their evolution increase [59-60]. In the presence of ash elements, the volatiles escaping undergo secondary cracking and form a soot deposit on the residual char. The oil fraction, which consists of mainly the condensates of primary vapors from cellulose, lignin etc., increases only when the absolute values of cellulose and lignin increase and / or when the secondary cracking of oil (catalysed by the ash constituents) to give lighter organic fraction decreases. Similar increase in oil percentages has been reported by venu et al. [61] for other biomass also wherein reduction of ash is considerable during pretreatment. At this juncture, it is worth comparing the oil percentages as well as the oil to liquid ratios for two bagasse samples having similar composition. It is well known that the bagasse composition changes with the source and origin. Same composition may be arrived by pretreatment or may occur naturally. The bagasse composition used by venu et al. [62-64] matches with the composition of 1 hour water leached bagasse in the present study. The oil yield obtained in this study for the above mentioned pretreated bagasse is ~32%, which is nearly similar to the oil yield of 34.2% reported by venu et. al. (Table 3). Thus, the removal of extractives and the higher percentage of cellulose are capable of giving high yields of oil, which can be obtained by either water leaching of the high extractive content bagasse or by using a bagasse with high cellulose content.

Table 3: Comparison of oil and liquid yield by two processes

	Biomass Components			Total liquid yield (% daf)	Oil yield (% daf)	Ash (% dry)
	Hemicellulose + Extractives	Cellulose	Lignin			
i) Present method (in-situ separation after pretreatment)	35.3	43.4	21.2	62.4	32	1.89

ii) Perez et. al (after processing of liquid)	35.8	43.1	21.1	62.0	34.2	1.6

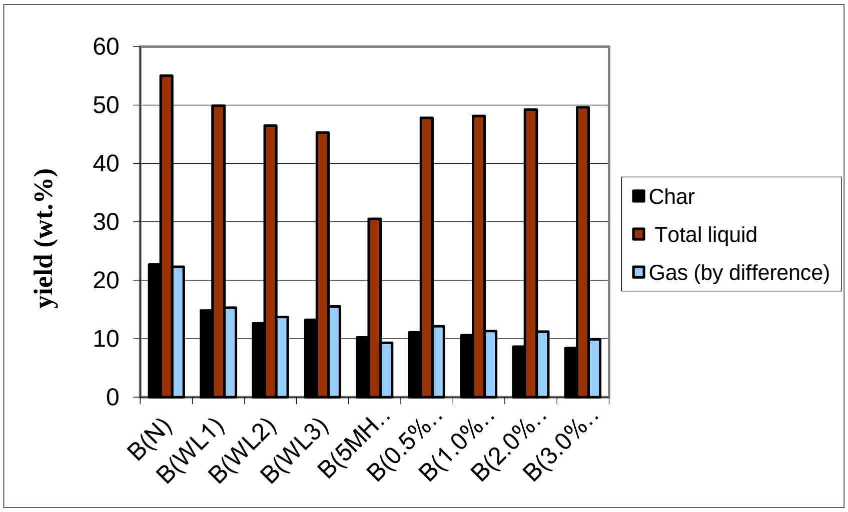


Figure 1: Pyrolysis product distribution of untreated and treated bagasse (original bagasse basis)

3.1 Effect of deashing treatments on pyrolysis product characteristics of sugarcane bagasse

The products obtained on pyrolysis of untreated and treated bagasse have been subjected to different analysis for their properties. The effect of deashing treatments on the pyrolysis product characteristics is reported in the following section.

3.2 Higher heating value (HHV), pH and moisture content of sugarcane bagasse pyrolysis oil

Higher heating value, moisture content and pH of bagasse and treated bagasse oil, is given in **Table 4**. There is no note-worthy variation in calorific value of the oils, however, it is seen that moisture content of the oil gradually decreases from untreated to water leached and to acid treated oil. It is obvious to attribute this to the removal of ash from bagasse, on pretreatment. The removal or and absence of ash reduces the occurrence of ash catalysed lignin decomposition reactions forming char and water [65-66]. The partial pressures of water vapors in the vapor stream being low, reduce the amount of water condensed in the first few collecting bottles maintained at and above 60^0C, and thereby, reducing the moisture content in the oil fraction. It may however, also be mentioned here that, the increase in the amount of primary vapors condensed as oil also contributes to the subsequent reduction of moisture % in the oil content.

Table 4: Properties of sugarcane bagasse pyrolysis oil

Biomass pyrolysis oil	HHV (Higher heating value) MJ/kg	pH	Moisture Content
Normal bagasse pyrolysis oil	23.3	2.6	12.0
Water leached bagasse pyrolysis oil	22.2	2.5	11.2
HCl treated bagasse pyrolysis oil	21.6	2.3	8.3

HF treated bagasse pyrolysis oil	23.2	2.4	7.4

3.3 Bagasse pyrolysis oil-characterization

Characterization of pyrolysis oil in terms of polar and non polar fractions has been carried out by means of solvent extraction. Untreated as well as all the treated bagasse pyrolysis oil was successively extracted during 20 minutes in a separating funnel with 100 ml of the following solvents: hexane, benzene, dichloromethane, ethyl acetate and methanol. Solvents were removed in a rotary evaporator and the recovered bio-oil fractions were weighed and analyzed. The percentage miscibility of untreated and pretreated bagasse pyrolysis oil in different solvents ranging from non-polar hexane to highly polar methanol is presented in **Table 4.** The comparative results of miscibility show that moving from untreated to water leaching the percentage solubility in non polar solvents decreases with gradual increase in solubility in polar solvents. In case of acid treated bagasse pyrolysis oil, there is a drastic decrease in non-polar fraction with an equal increase in the polar fraction of oil. The bagasse pyrolysis oil (untreated and treated) as well as the fractions obtained by solvent extraction, as mentioned above, was subsequently analyzed for their compounds using FTIR and GC/MS techniques [64-66]. The major compounds identified are presented in the **Table 4**. The GC/MS plots and corresponding FTIR of untreated bagasse pyrolysis oil are presented in **Figure 2** and **Figure 3** respectively. Some typical observations are as follows.

i) There is an increase in the oxygenated–polar (Dichloromethane and Ethyl acetate soluble fractions) for treated bagasse pyrolysis oil.

ii) The major compounds present in the water leached bagasse are very similar to that reported by Perez et. al for vacuum pyrolysis of bagasse of similar composition under same reaction conditions. The moisture content and the calorific values are also comparable. Thus, it is seen that not only the product distribution but the nature of pyrolysis oil composition also depend largely on the relative composition of bagasse feedstock. This has been exploited and explained in detail by venu [58].

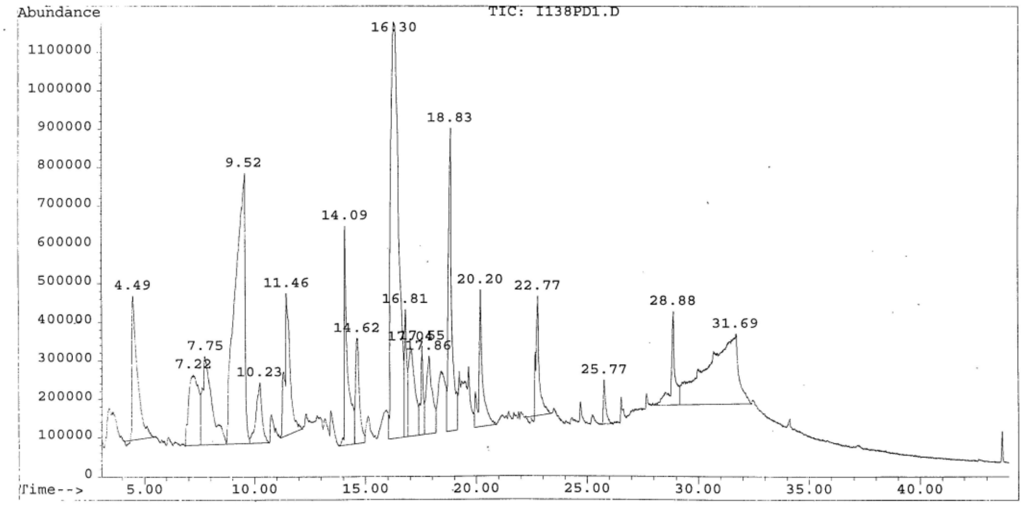


Figure 2: GC/MS of untreated bagasse pyrolysis oil (whole)

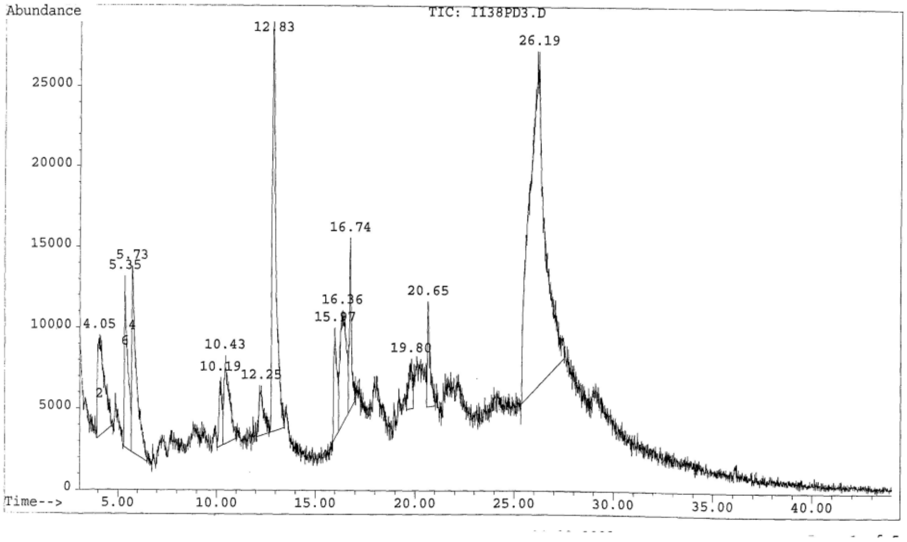
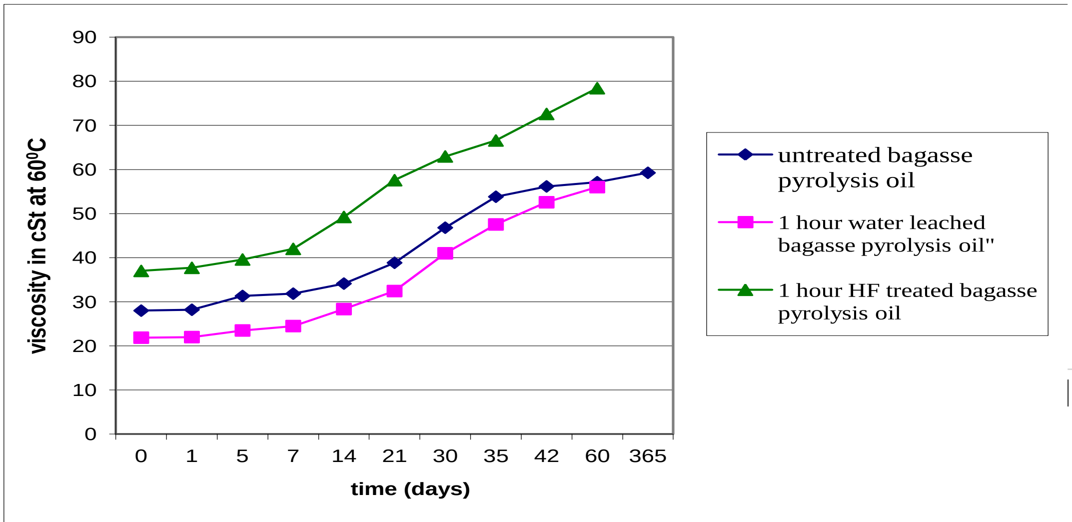


Figure 3: GC/MS of methanol soluble fraction

3.4 Stability characteristics of sugarcane bagasse pyrolysis oil

The variation in viscosity has been monitored for untreated and treated bagasse pyrolysis oils, both stored



at room temperature (**Figure 2**) as well as at 60^0C (**Table 3**). The viscosity is measured at 60^0C for the oils stored at room temperature, while for the oil stored at 60^0C, viscosity was measured at three different temperatures. It is seen that, compared to untreated bagasse oil, initial viscosity as well as rate of change of viscosity of acid pretreated bagasse oil is higher. This is attributed to the more polar fractions present in treated oils as shown in **Table 4**. Pretreatment hydrolyses cellulose and hemicelluloses which results in the increase of more acidic as well as polar fraction in the oil leading to higher rate of increase of viscosity in the pretreated oil compared to untreated pyrolysis oil. The effect is more severe in case of acid pretreatment which leads to accelerated polymerization giving more viscous and more acidic oil having less water.

Figure 4: Viscosity variation of untreated and treated bagasse pyrolysisoil with time (Stored at room temperature)

4. Conclusion

Pre-treatment of bagasse with water, dilute HCl solution and dilute HF solution shows a remarkable change in the pyrolysis product distribution by virtue of a combination of a change in the organic constituents and the selective removal of inorganic ash elements. Mild HF solution is effective in reducing the ash content of the biomass to a negligible amount. Moreover, this treatment effectively increases the oil yield by ~ 69% (on the basis of original wt. of bagasse before treatment) ~145% (on treated bagasse basis) compared to oil obtained from untreated bagasse. Acid pretreatment hydrolyses the hemicelluloses and cellulose into smaller molecules resulting in the increase of acidic as well as polar fractions in the oil and leads to higher rate of increase of viscosity in case of pretreated oil compared to untreated pyrolysis oil. Thus, HF treatment though removes the ash elements completely, yet does not help in improving the stability. Moreover, the leachates from this pretreatment cause environmental concerns and is not advisable to use. Similarly, treatment with 5M HCl leads to a marginal increase in oil yield but results in an increase in viscosity of pyrolysis oil. On the other hand, simple water leaching with least possible water for longer period, is effective enough to reduce the extractives (depends on amount of extractive present originally, which in turn depend on bagasse generation process) along with some selected ash components in bagasse. Water leaching, by virtue of selective removal of ash as well as organic constituents increases the oil yield (increase in the oil yield vary with the reduction in extractives and total mass) by as much as 55.4 % (on original basis) and 113.3 % (on treated basis) on vacuum pyrolysis. The calorific value of oil however is not affected much and is in the range of 22-24 MJ/kg**.** The pretreatment also produces char with a higher adsorptive capacity, thereby adding value to the char obtained from pyrolysis.

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