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Alteration of The Properties of Spent Eucalyptus Biomass from Steam Distillation

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ABSTRACT

Steam distillation is a common method to extract the eucalyptus oil from eucalyptus biomass. The spent biomass of the steam distillation process could be used as biofuel feedstock. This study aims to investigate the effect of eucalyptus biomass steam distillation time on the alteration of the spent biomass. The size of the raw spent biomass used in the study was 1 cm x 1 cm. The properties studied include carbon retention, fuel properties namely proximate and ultimate analyses as well as grindability. Those properties were investigated from 5 to 180 minutes of the distillation time. The results show that the spent biomass has a better grindability than the raw biomass. Whereas, fuels chemistry of spent biomass were little affected by the steam distillation process. Thus it is quite promising to use the spent biomass of steam distillation as a feedstock for bioenergy production as the spent biomass does not undergo a significant change during steam distillation.

Keywords: *alteration fuel properties, eucalyptus leaf, grindability, spent biomass, steam distillation*

1. INTRODUCTION

To prevent dryland salinity, eucalyptus trees were planted in the affected areas in the outback of Western Australia. Thus, eucalyptus biomass is a byproduct of dryland salinity management in Western Australia (WA) (Bartle et al, 2007; Harper et al 2009). Its production as an energy source has been proven to be economic, of large scale and low energy and carbon footprints (Wu et al, 2007; Bartle & Abadi, 2009). The economic energetic performance of mallee biomass could be further improved by producing value-added products such as eucalyptus essential oil (mainly 1.8-cineole) which is abundant in eucalyptus leaf and widely used in various fragrance and pharmaceutical industries (Goodger et al, 2007).

The eucalyptus essential oil is commonly extracted via hydrodistillation and/or steam [Hernandez, 2000]. The distillation processes are environmentally friendly and also relatively safe to operate (Romdhane and Tizaoui, 2005). Also, these processes are suitable for either batch or semi-continuous configuration and thereby easy to scale up (Teranishi et al, 1980). Previous studies showed that eucalyptus essential oil can be easily extracted by both hydrodistillation (Wu et al, 2011) and steam distillation (Li and Madden, 1995). Compared with hydrodistillation which possibly extracts inappropriate components from the leaf, steam distillation offers several key advantages. First, eucalyptus oil extracted by steam can be easily condensed in water after cooling and naturally separated with water, which is able to minimize impurities being extracted (Teranishi et al, 1977). Second, steam distillation protects the extracted material from oxidation by displacing of atmospheric oxygen with steam (Krell, 1963). Last, steam distillation consumes lesser water than hydrodistillation. This is of particular importance in WA due to the lack of freshwater in the state (Hamilton et al, 2005).

Steam or water vapour has been used by several workers in distillation for the isolation of volatile constituents of plant materials to provide essential oils. During steam distillation of thyme (Thymbra spicata), it was found that the yield of the volatile oil was higher at a shorter distillation time with a higher flow rate of steam (Hanci et al, 2003). It was also found in the research that the oil yields obtained for the ground samples were lower than unground samples. Borges and Pino (1993) investigated the isolation of volatile oil from cumin seeds. Their result showed that the 93% of oil yield can be reached in 5 h distillation time. Boutekedjiret at.al (2003) showed that steam distillation gave three times better in

yield compared to hydrodistillation during the isolation of rosemary essential oil.

After oil extraction, the spent leaves can be used as a bioenergy feedstock (. However, to the best of the authors' knowledge, there are no reports on the fuel properties (such as grindability and ash chemistry, etc.) of spent Mallee leaf biomass after steam distillation process. Such data are believed to be critical in applying spent leaves for bioenergy production (Wu et al, 2011). Therefore, the aims of this paper are to investigate the effect of eucalyptus steam distillation on fuel properties of its spent biomass. Eucalyptus leaves were steam distilled for eucalyptus oil (mainly 1.8-cineole) extraction, after which the spent biomass was analyzed by various analytical methods to assess key fuel properties such as grindability and fuel chemistry.

2. METHODS

2.1 Material

Eucalyptus leaf biomass (*E. polybractea*) were used in this study. In order to reduce the moisture content to ± 5%, the biomass was dried in an oven at 40 °C. This leaf biomass was cut to the size of 1 cm x 1 cm and stored in a freezer at -4 °C to avoid microbial degradation and air-dried before subjected to steam distillation.

2.2 Steam distillation extraction

The steam distillation apparatus consists of an extraction column, a flask, a heating mantel and a condenser, as shown in Figure 1. In order to maintain steam condition inside the column, heating tape and insulation were applied to maitain the temperature inside of the extraction column around 105 °C to avoid steam condensation. The heating flow rate of heating mantle was adjusted based on appropriate steam flow rate produced. Milli Q water was heated continuously in the flask until the steam production was stable. Then approximately about 10 g of the sample was fed into the column and the distillation process was continued until a certain distillation time (from 5 min to 180 min) was achieved. Finally the flask and the distillation column were separated as soon as the steam distillation process completed.

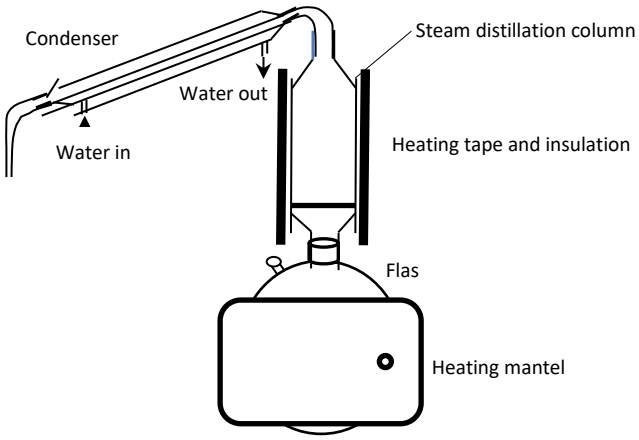


Fig. 1. Schematic diagram of steam distillation apparatus

2.3 Quantification of total carbon in liquid.

The total carbon concentration in liquid (distilled and waste water) were analysed using a TOC analyser (TOC, model: Shimadzu TOC-VCPH).

2.4 Fuel Properties and Characterization of Inorganic Species in Raw and Spent Biomass

2.4.1 Proximate and Ultimate Analysis of Biomass Samples.

In order to quantify of moisture and fixed carbon of biomass sample, a thermogravimetric analyser (TGA, model : METTLER) was applied based on ASTM E870-82. The amount of carbon, hydrogen and nitrogen in biomass samples were determined using a CHNSO elemental analyser (model: Perkin Elmer 2400 Series II). Water leaching process was conducted to extract Chlorine from biomass then injected to IC to quantify its chlorine content. The sulphur concentration was analysed using the Eschka18 combining with combustion process. The oxygen (O) content was determined by the difference.

2.5 Grindability and Morphology of Raw and Spent Biomass.

Laboratory ball mill was used to grind raw and spent biomass samples for grindability assessment. The grindability assessment has been used for assessing and benchmarking grindability for various biomass in previous study (Wu et al, 2011). Briefly, the raw or spent leaf samples with size 1cm x 1 cm were ground in a Retsch Mixer Mill MM400 laboratory ball mill at a frequency of 15 Hz with various grinding time length from 0 to 15 min. The ground samples were then mixed with sodium hexametaphosphate solution (10%w/v) in order to aid dispersion of fine particles before loading into the measuring chamber and analyzed using a laser-diffraction particle size analyzer (Malvern Mastersizer 2000,

Worcestershire, U.K). The particle size distributions (PSDs) were then constructed and the results were reported as cumulated volume percentage curves for biomass grindability assessment. It is important to note that in this study ball milling is only used as a tool at a laboratory-scale for assessing and benchmarking the grindability of various biomass materials.

Cross sectional area of the raw and spent biomass samples were prepared and then examined using a Philips XL30 Scanning Electron Microscope (SEM) for structural characterization. To prepare the sample specimen, the dried raw and spent biomass samples were arranged longitudinally and then set in the epoxy resin which was then solidified, polished, dried in the oven (~50 ^{o}C) overnight and coated with carbon for SEM observation.

3. RESULTS AND DISCUSSION

3.1 Carbon Balance during eucalyptus Steam Distillation

The release of carbon during steam distillation from 5 min to 180 min distillation time is presented in Figure 2. Carbon appears to be released significantly during steam distillation up to 60 min. After 5 min distillation, 0.4% carbon was released. The carbon released from biomass increased considerably from 3.2% at 30 min distillation to 5.1% at 60 min distillation. However, at distillation time of 90 min, the carbon released was only 5.9%. Above 60 minutes, carbon released during steam distillation of the eucalyptus leaf seemed to be relatively constant.

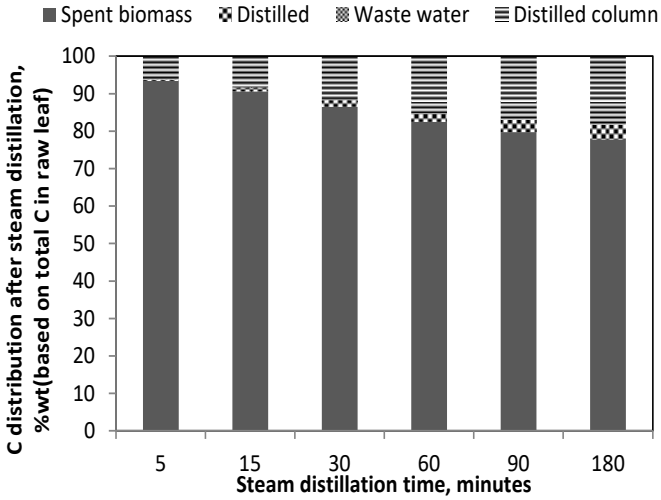


Fig. 2. Distribution of carbon after steam distillation in distillation column, waste water in boiler, distillate and spent biomass.

Carbon retained after distillation process was measured in waste water in the boiler, distillate and spent biomass. Whereas carbon retained in distillation column was measured by subtracting total carbon with carbon retained in waste water, distillate and spent biomass. The carbon distribution after distillation is shown in Figure 2. Generally, there was a significant increase in carbon released in the spent biomass with increase in distillation time. It also can be seen that carbon released from the leaf (biomass) was mostly retained in the column. After the optimum time of the steam distillation of eucalyptus leaf achieved (60 minutes), carbon was still released from the biomass. This suggests that carbon which was not carbon of eucalyptus oil origin, was also released during steam distillation above 60 minutes of distillation time. The evidence that the carbon was still released above 60 minutes of distillation time indicates that parts of biomass materials were destructed and retained inside the column. Figure 2 clearly shows that distribution of C retained increase significantly after 60 minutes steam distillation time.

3.2 Effect of Steam Distillation on Fuel Properties of Spent Biomass

Fuel properties, namely proximate and ultimate analysis, of raw eucalyptus leaf and spent leaf after steam distillation of various distillation times are presented in Table 1. It can be seen from Table 1 that both proximate and ultimate analyses of raw and spent biomass of various distillation times did not vary significantly; steam distillation time has a little effect on biomass fuel properties. This is also the case for the hydrodistillation of mallee leaf (Wu et al, 2011. Thus, it is quite promising to use the spent biomass of steam distillation as a feedstock for bioenergy production. Mass energy density (calorific value) of fuel is one of the main indicator elements in application of energy sources. In this work, the higher heating value (HHV) was estimated based on the result of ultimate analysis with the range of ± 5% error (Abdullah and Wu, 2009). It can be seen from Table 1, that steam distillation process does not affect energy content of biomass during the process. It is interesting to see from Table 1 that the ash content of spent biomass was not significantly different from the raw material, steam

distillation might extracts slightly inorganic compounds during the process. From the ultimate (elemental) analysis, it can be seen that the retention of C and H in spent biomass decreased slightly with increase in steam distillation time. While Cl and S contents of spent biomass were relatively unchanged with increase in distillation time. Although previous study (Wu et al, 2011) showed that Cl and S were water soluble, it seems that both Cl and S were not soluble in water vapour, thus they were left almost unchanged in the spent biomass.

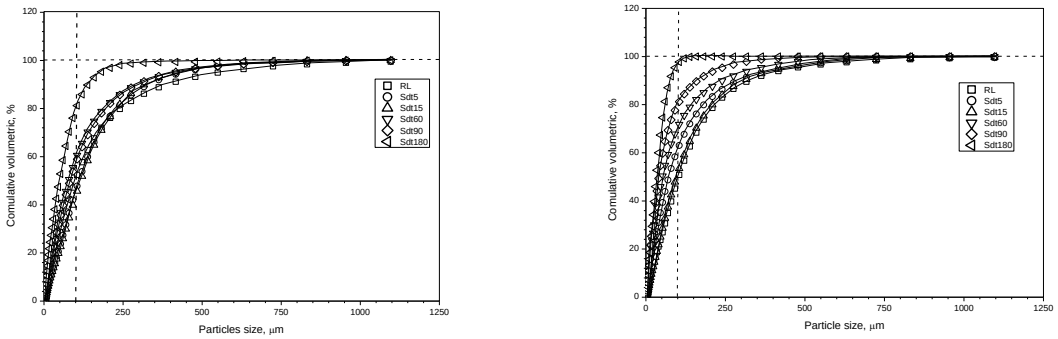
Table 1. Fuel properties of raw and steam-distillation-spent biomass

	Proximate analysis, wt % db				Ultimate analysis, wt % daf					HHV[e], MJ/kg	
Sample	M(ad)	ash	VM	F C	C	H	N	S	Cl	O[d]	
RL	5.0	5.3	77.7	17	54.5	7.4	1.25	0.77	0.24	30.55	21.89
SD5	4.9	5.3	77.8	16.8	54.3	7.6	1.35	0.80	0.20	30.50	21.93
SD15	4.4	5.0	78.4	16.7	54.0	7.2	1.37	0.83	0.18	31.37	21.65
SD30	4.0	5.7	78.4	15.9	52.4	7.4	1.32	0.80	0.20	32.26	21.24
SD60	3.9	5.5	77.4	17.1	52.2	7.3	1.44	0.79	0.19	32.57	21.18
SD90	3.8	5.0	78.4	16.5	52.3	7.2	1.40	0.83	0.19	33.08	21.14
SD180	3.3	5.6	77.4	16.9	51.9	7.2	1.34	0.82	0.17	32.87	21.06

"RL = raw leaf, SD=spent biomass after steam distillation, 5,15 etc = steam distillation time (minutes), M= Moisture, VM = Volatile matter, FC=fixed carbon, HHV= High Heating Value; ad=air dried, db=dry basis, daf=dry ash free, d = by difference, e = by calculation".

3.3 Grindability of Spent Biomass after Steam Distillaiton

Grindability is also an important parameter on the selection of a material as a fuel. Biomass usually posses poor grindability due to its fibrous structure. In this study, the grindability of raw biomass and spent biomass after steam distillation in various times were measured their cumulative volumetric of particle size in various grinding times. The results of the grindability measurement were presented in Figure 3.a and Figure 3.b. It can be seen from the figure that generally for all of data presented, the grindability, which is characterised as cumulative volumetric of the raw leaf is slightly poorer than the spent leaf after distillation. For example, for 8 min grinding, the cumulative volumetric of 100 μm size of raw leaf was only 52.4 %, while after 60 min and 180 min distillation time were 60.5% and 95.3%, respectively. From the figure, it can be seen that an increase in distillation time gave significant effect on the grindability of the spent biomass up to particle size of 500 μm. It also can be noted that the cumulative volumetric of raw leaf and spent biomass relatively increase with longer grinding time, this could be related to the longer the grinding time, the finer particles are (Abdullah and Wu, 2009). This research shows an important finding that the grindability of the spent biomass is better than the raw biomass and the grindability difference is significant. This suggests that the utilisation of spent biomass as a fuel is promising and might offset the grinding cost.



(a) (b)

Fig. 3. Particle distribution of raw leaf and spent biomass for (a) 5 and (b) 8 minutes grinding time.

4. CONCLUSION

Steam distillation process seems to have no significant of chemical and physical properties negative effect on spent leaves as a source for bioneregy production. Alteration of AAEM content was a little affected during the extraction process. Whereas, steam distillation increased the grindability of spent biomass significantly.

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