CHARCOAL PRODUCTION FROM FOREST RESIDUES

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ABSTRACT: The carbonization behaviors of spruce wood and its forest residue at different conditions were studied in this work. A standard proximateanalysis procedure delivered the lowest charcoaland fixed-carbon yields. Sample sizeconsiderably affected the charcoal and fixed-carbonyields. In this work, for spruce wood andits forest residue, an increase in sample size from10 mg to 130 mg increased the charcoal yield at950°C from 18.60 to 21.49 wt% and 26.78 to28.12 wt%, respectively. For all TGAmeasurements, both charcoal and fixed-carbonyields are significantly enhanced when a closedcrucible is employed, in comparison to an opencrucible. The highest charcoal and fixed-carbonyields obtained in this work were offered by ahigh pressure TGA at 8 bar. These findings showthe influential role of secondary char formingreactions. Conditions that improve or prolongcontact of pyrolytic vapors with the charcoalmatrix enhance the fixed-carbon yield.
Keywords: Charcoal, forest residues, wood, pyrolysis.

1 INTRODUCTION

Forest residues, as an abundant low cost biomass resource, is gaining interest and entering the market for a wide range of applications. However, direct utilization of forest residues is usually challenging considering its heterogeneous properties in terms of biological origin andcomposition, high moisture content, large variety in size and shape, etc.[1]. Carbonization is a promising way to convert low-grade forest residues into high quality fuel for combustion to generate heat and/or power, or as a high quality reductant for metallurgical industry [1]. Several operational parameters influence the carbonization process, which affect the yield and properties of the products as well. Among these factors are the peak temperature, the pressure and the particle size, and they all play an important role in determining both charcoal yield and fixed-carbon content of the produced charcoal. In general, the charcoal yield decreases as the peak temperature increases[1]. On the other hand, increase of peak temperature lead to enhancement of fixed-carbon yield. The pressure gives positive effects on the carbonization process and increases both charcoal and fixed-carbon yield [2,3]. Particle size of biomass feedstock also has certain effects on charcoal and fixed-carbon yield. As reported in our previous study, the particle size has a strong influence on both the charcoal and fixed-carbon yields at pressures in the range of 0.1 to 2.7 MPa [4]. For a carbonization process occurring under pressure and with large particle size feedstock, the increase in charcoal and fixed-carbon yields is mainly attributed to prolonged residence time of tarry vapors in the char matrix. It enhances secondary reactions and recondensation of vapors into solid carbon [5]. Carbonization behaviors of different biomass materials have been studied. However, few studies have been done to investigate charcoal production from forest residues. Theprimary aim of this work is to evaluate carbonization behaviors of spruce forest residueand examine the effects of confinement of the pyrolysis vapors, sample size and pressure on values of charcoal yield $y_{charcoal}$ and fixed-carbon yield y_{fc} for spruce wood and its forest residue.

2 MATERIALS AND METHODS

2.1 Samples

In this work, forest residue (contains mainly tops and branches) collected from a representative Norway spruce tree in a forest located in southern Norway was studied. For comparison purpose, stem wood from the same tree was chipped and studied. Both the spruce stem wood chips and forest residue were dried at 105 °C for 72 hours and ground in a cutting mill mounted with a 1 mm sieve, before further carbonization use.

2.2 Equipment and storage testing

Two atmospheric pressure thermogravimetric analyzers (TGAs) were employed in this work: TA Q600 of TA Instruments and Mettler Toledo TGA/SDTA 851e (MT-TGA). Another TGA (Linseis STA HP-TGA) was also used, and can provide mass loss data at pressures from vacuum up to 50 bar. Table 1 summarizes the geometry and depth of the crucibles used in each TGA. All TGA runs employed nitrogen (99.999% pure) as purge gas with a flow rate of 100 mL min^{-1}. For measurements carried out at atmospheric pressure with TA Q600 and the HP-TGA, 10 mg sample was loaded in the crucible before each experiment. Additionally, some experiments were also performed with the HP-TGA at 8 bar pressure. The sample loaded in a crucible was purged at room temperature, followed by 30 min of drying at 105 °C, and then heated up to 950 °C at a heating rate of 10°C/min. The temperature program is summarized in Table 1. For experiments conducted with the MT-TGA, around 130 mg sample was loaded in a 900 µl crucible and pyrolyzed with the sameprogram as mentioned above. For some experiments carried out with TA Q600 and the MT-TGA, a lid was used to cover the crucible with the loaded sample. These runs are identified as "closed crucible" experiments. The charcoal yield y_{char} was calculated by dividing the final sample massby the mass measured at the end of the drying period at 105 °C.

Table I: Temperature programs used in present work

Pyrolysis method					
Step	dynamic	isothermal	time (min)	heating rate (K/min)	temperature (°C)
1		-	30		25
2	-			jump	25→105
3		-	30		105
4	-			10	105→950

Table II: Specifications of instruments and crucible

Instruments	Crucible/pan volume(µl)	Crucible geometry (dxh mm)
TA Q600	Crucible (90 µl)	6 x 4
HP-STA	Crucible (120 µl)	6 x 4.5
MT-TGA	Crucible (900 µl)	12 x 10

3 RESULTS AND DISCUSSION

Table III shows the proximate analysis of spruce wood and its forest residue. The forest residue has considerably higher ash content than the spruce wood. The proximate analysis can be considered as a kind of carbonization procedure, and we can calculate its fixed-carbon yield y_{fc}. For the forest residue, it offers a yield of 21.19 wt%, which is about 5 wt% higher than the y_{fc} of the spruce wood.

Table III: Proximate analysis and fixed-carbon yield of feed materials

	Proximate analysis (wt %)[a]			
	VM	fC	ash	y_{fc} (wt %)
Spruce	83.43	16.01	0.56	16.10
Forest residue	76.84	20.66	2.50	21.19

Figure 1 and 2 display mean values of three determinations of charcoal yields, measured by the three different TGA instruments. For spruce wood and its forest residue, the increased sample mass have augmented the charcoal yield. A 10 mg sample spruce wood in an open crucible offered an average charcoal yield of 18.6% that increase to 21.49% with a 130 mg sample. By using a closed crucible, the charcoal yield of a 10 mg spruce wood sampleincreased to 21.44%. Likewise, in open crucibles, the average charcoal yield of a 10 mg forest residue sample was 26.78%,and increase to 28.12% for a 130 mg sample, whereas the charcoal yield realized with a closed crucible increased to 28.44% and 30.77%, respectively.

Figure 3 displays the influence of pressure in open crucibles on charcoal yields for spruce wood and its forest residue in the HP-TGA. Increasing pressure substantially enhances the charcoal yield. For the open crucible, 10 mg spruce wood powder gave an average charcoal yieldof 19.78% that increased to 32.96% at 8 bar pressure. As shown in Figure 3, the carbonization behavior of the forest residue is similar to that of spruce wood. The average open-crucible charcoal yield of the forest residue increased from 27.32% at 1 bar to 34.75% at 8 bar. In addition, increase of charcoal yield at elevated pressure is more evident for the spruce wood.

As mentioned above, fixed-carbon yield is considered as a more meaningful metric to evaluate conversion efficiency of the ash-free organic matter in the feedstock into relatively pure ash-free carbon [1]. Table 4 displays the proximate analysis of charcoal produced in the MT-TGA following the ASTM D1103 standard. It allows us to calculate the fixed-carbon yields obtained from different TGA instruments.

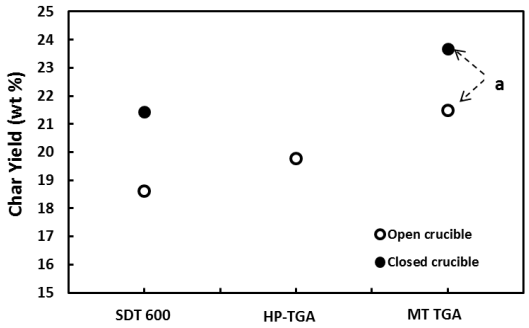

Figure 1: Effects of open vs closed crucible on spruce wood powder charcoal yield.(a)Representing charcoal yields from around 130 mg samples realized at atmospheric pressure (0.1 MPa) in the MT-TGA

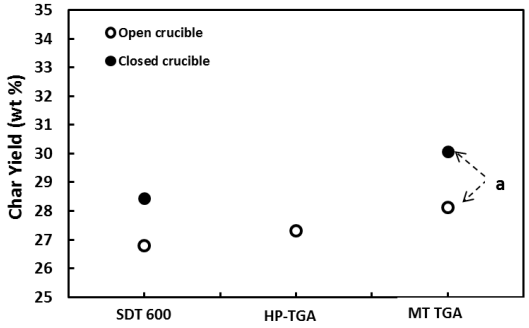

Figure 2: Effects of open vs closed crucible on forest residue powder charcoal yield. (a)Representing charcoal yields from around 130 mg samples realized at atmospheric pressure (0.1 MPa) in the MT-TGA

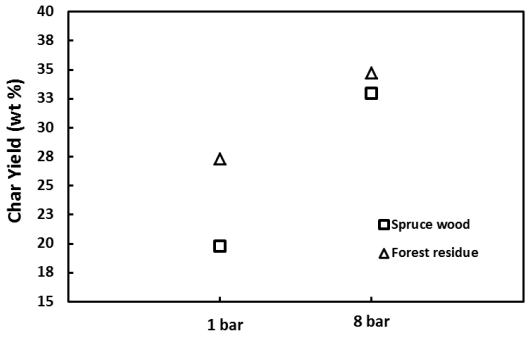

Figure 3: Effects of pressure on spruce wood and forest residue powder charcoal yield

Table IV shows proximate analysis results of charcoal produced in the MT-TGA. It is interesting to see that the volatile contents of the forest residue charcoal are evidently higher than those of spruce wood charcoal, regardless production in open or closed crucibles. Table 5 and 6 summarize charcoal and fixed-carbon yields realized in the atmospheric and pressurized TGAs. All

calculated values exceed the comparable y_{fc} obtained by the proximate analysis procedure. For both spruce wood and its forest residue, larger sample size offered enhanced estimated fixed-carbon yields. In addition, use of a closed crucible increased the fixed-carbon yield by 5-10%. The highest fixed-carbon yields are realized at 8 bar pressure in the HP-TGA. For spruce wood, the value substantiallyincreases, from 18.94 to 30.86%.

The forest residue offered higher charcoal yield than that of spruce wood. One explanation for this could be the high ash content of the forest residue. As reported in other studies, higher charcoal yields can be obtained from biomass materials with higher ash content [1]. The ash forming elements, including calcium, potassium, sodium and phosphorus, might play a catalytic role during the carbonization process and enhance charcoal formation [4]. The forest residue is a mixture of tree tops and branches, including twigs and needles. All these species are biological active and keep growing. During the growing process, more nutrients like calcium, potassium and sodium will transport from the root of a tree to these biological active parts and support their growth [6]. Therefore, concentrations of alkali and alkali earth metals in the forest residues are considerably higher than those in the stem wood [7]. It partially explains the higher ash content measured in the forest residues. Presence of these ash forming elements in the forest residues will also contribute to higher charcoal yield.

Results of the present work showed that increase of sample mass can improve both charcoal and fixed-carbon yield substantially. In addition, in agreement with previous studies, the charcoal and fixed-carbon yields increase as the carbonization take place in a vessel covered with a lid.Enhancement of charcoal and fixed-carbon yields when utilizing large sample massare mainly related to conditions that improve and prolong contact of vapor-phase pyrolysis species with the char matrix, leading to conversion of tarry vapors to solid carbon through secondary char forming reactions [2-3].A catalytic nature of the charcoal surface/structure can also play a role during formation of secondary char. As a large sample is used, the tarry pyrolysis vapor takes longer time to leave the char matrix, and might crack and recondense on solid char surfaces. The charcoal and fixed-carbon yields were also enhanced as observed in the present work. Using a lid restricts release of tarry vapors out from the vicinity of the hot char matrix and increases the extent of formation of secondary charcoal and fixed-carbon content as well [4]. Under pressurized conditions, the escape of tarry vapors from the solid char residues is slowed down and prolong the residence time of the volatiles in the char residues as well. Consequently, the tarry vapors have more chances to crack and repolymerize into solid carbon. Furthermore, saturation temperature and pressure of liquid tar will increase under elevated pressure. This will hinder conversion of tarry vapors and promote liquid-phase coking reactions that enhance solid charcoal formation. Furthermore, elevated pressure increases the concentration and partial pressure in the charcoal pores and structures, thereby enhancing the coke formation through vapor phase secondary reactions [5]. In this way, the elevated pressure increases charcoal and fixed-carbonyields as the samples were carbonized in the HP-TGA under 8 bar pressure.

Table IV: Proximate analysis of charcoal produced in the MT-TGA

		Proximate analysis (wt %)[a]				
Sample	TGA exp	VM	fC	ash	y_{char}	y_{fc}
Spruce powder 130 mg	open crucible	4.04	94.97	0.99	22.26	21.31
Spruce powder 130 mg	closed crucible	4.28	92.92	2.80	24.14	22.60
Forest residue powder 130 mg	open crucible	8.99	85.39	5.62	28.73	24.53
Forest residue powder 130 mg	closed crucible	8.19	85.06	6.75	30.81	26.47

Table V: Charcoal and fixed-carbon yields realized at atmospheric pressure (0.1MPa) in the MT-TGA

	y_{char} (wt %)[a]		y_{fc} (wt %)[a.b]	
	Open crucible	Closed crucible	Open crucible	Closed crucible
Spruce wood[c]	18.60	21.44	17.81	20.07
Spruce wood[d]	21.49	23.68	20.58	22.16
Spruce wood[e]	19.78	-	18.94	-
Forest residue[c]	26.78	28.44	22.89	24.43
Forest residue[d]	28.12	30.07	24.01	25.83
Forest residue[e]	27.32	-	23.35	-

[a] Percent of dried feed material. [b] Fixed-carbon yield = charcoal yield × (100-% volatile matter-% char ash)/(100-%feed ash). Here the volatile matter for char produced at 950°C and ash content measured from the MT-TGA produced charcoal are used. [c] Charcoal and fixed-carbon yields realized at atmospheric pressure (0.1 MPa) in the TA Q600. [d] Charcoal and fixed-carbon yields realized at atmospheric pressure (0.1 MPa) in the MT-TGA. [e] Charcoal and fixed-carbon yields realized at atmospheric pressure (0.1 MPa) in the HP-TGA.

Table VI: Charcoal and fixed-carbon yields realized at atmospheric pressure (0.1 MPa) and 0.8 MPa in the HP-TGA

Sample	TGA exp	y_{char}	y_{fc}
Spruce powder 10 mg 1 bar	open crucible	19.78	18.94
Spruce powder 10 mg 8 bar	open crucible	32.96	30.86
Forest residuepowder 10 mg 1 bar	open crucible	27.32	23.35
Forest residuepowder 10 mg 8 bar	open crucible	34.75	29.85

4 CONCLUSIONS

The carbonization behaviors of spruce wood and its forest residue under different conditions were studied in this work. A standard proximate analysis procedure delivered the lowest charcoal and fixed-carbon yields. Sample size considerably affected the charcoal and fixed-carbon yields. In this work, for spruce wood and its forest residue, an increase in sample size from 10 mg to 130 mg increased the charcoal yield at 950°C from 18.60 to 21.49 wt% and 26.78 to 28.12 wt%, respectively. For all TGA measurements, both charcoal and fixed-carbon yields are significantly enhanced when a closed crucible is employed, in comparison to an open crucible. The highest charcoal and fixed-carbon yields obtained in this work were offered by a high pressure TGA at 8 bar. These findings show the influential role of secondary

char forming reactions. Conditions that improve or prolong contact of pyrolytic vapors with the charcoal matrix enhance the fixed-carbon yield.

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7 PROJECT LOGO