

1 **Title Page:**

2 Fuel Moisture Influences on Fire-altered Carbon in Masticated Fuels: An Experimental Study

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18 **Text:**

19 **Abstract:**

20

21 Biomass burning is a significant contributor to atmospheric carbon emissions, but may also  
22 provide an avenue in which fire-affected ecosystems can accumulate carbon over time, through  
23 the generation of highly resistant fire-altered carbon. Identifying how fuel moisture, and  
24 subsequent changes in the fire behavior, relates to the production of fire-altered carbon is  
25 important in determining how persistent charred residues are following a fire. Additionally,  
26 understanding how mastication (mechanical forest thinning) and fire convert biomass to black  
27 carbon is essential in understanding how this management technique, employed in many fire  
28 prone forest types, may influence stand-level black carbon in soils. In this experimental study  
29 fifteen masticated fuel beds, conditioned to three fuel moisture ranges, were burnt and  
30 production rates of pyrogenic carbon and black carbon were evaluated. Pyrogenic carbon was  
31 determined through elemental analysis of the post-fire residues (e.g. charcoal), and black carbon  
32 was quantified using thermo-chemical methods. Pyrogenic carbon production rates ranged from  
33 7.23 to 8.67% relative to pre-fire organic carbon content. Black carbon production rates averaged  
34 0.02% in the 4 to 8% fuel moisture group, and 0.05% in the 13-18% moisture group. A  
35 comparison of the ratio of black carbon to pyrogenic carbon indicates that burning in fuels  
36 ranging from 13-15% moisture content resulted in a higher proportion of black carbon produced,  
37 suggesting that the precursors to black carbon were indiscriminately consumed at lower fuel  
38 moistures. This research highlights the importance of fuel moisture and its role in dictating both  
39 the quantity and quality of the carbon produced in masticated fuel beds.

40

41 **Introduction:**

42

43         Forested terrestrial ecosystems have recently received attention for their potential role in  
44 sequestering carbon to help offset global greenhouse gas emissions from fossil fuel and biomass  
45 burning [Malhi *et al.*, 2002]. As a result of this attention, policy and management efforts are  
46 currently being encouraged to include carbon at the stand scale [Canadell and Raupach, 2008].  
47 Carbon cycling in fire-prone forests is inherently complex due to numerous factors acting on the  
48 stand over time [McKinley *et al.*, 2011]. In a given forest, biomass accumulation periods (C sink  
49 as above ground carbon pools grow) are often followed by a disturbance processes, e.g., land  
50 conversion, harvesting, fire, insects, wind, etc. These disturbances can rapidly deplete existing  
51 carbon and transition biomass from live to dead pools, where slow decomposition continues to  
52 release carbon into the atmosphere [McKinley *et al.*, 2011]. Given enough time, the stand may  
53 successfully regain its pre-disturbance carbon stocks and when considered over broad temporal  
54 and spatial scales, this gain/loss carbon cycling may reach equilibrium [Harmon, 2001; Kashian  
55 *et al.*, 2006]. A potential key contributor to long-term carbon storage in fire-affected ecosystems  
56 is the production of fire-altered charred residues remaining after a fire, commonly referred to as  
57 BC [DeLuca and Aplet, 2008]. This thermally altered BC (BC) have the potential to persist in  
58 terrestrial soils for thousands of years, thereby slowly increasing forest soil carbon stocks over  
59 multiple fire events [DeLuca and Aplet, 2008].

60         Throughout the forests of the Western United States, successful fire exclusion policies  
61 have led to large increases in biomass accumulation [Agee and Skinner, 2005]. In many of these  
62 fire-affected forest systems, thinning and prescribed fires are commonly used to reduce excessive  
63 build-up of fuels [Agee and Skinner, 2005]. These management practices actively remove above-

64 ground carbon in order to maintain ecosystem function and increase stand resilience to future  
65 disturbance, an increasingly important goal in the face of climate change and increased fire  
66 frequencies [Westerling *et al.*, 2006]. Mastication is an increasingly common forest thinning  
67 treatment in which a tracked or wheeled excavator mounted with a rotating head or drum grinds  
68 down smaller trees and shrubs, effectively reallocating aerial fuels to the forest floor [Battaglia  
69 *et al.*, 2010]. Oftentimes prescribed fire treatments are employed following mastication to reduce  
70 surface fuel loadings, and to further reduce the threat of high-intensity wildfire [Agee and  
71 Skinner, 2005].

72         The impacts of these management activities on carbon stocks vary widely by ecosystem  
73 type [McKinley *et al.*, 2011; Mitchell *et al.*, 2009]. Although fuels reduction activities may serve  
74 to reduce short-term stand-level carbon stocks, studies have found that fuels reduction may  
75 actually increase stand-level carbon over time, as large diameter trees store a disproportionately  
76 large amount of above-ground carbon [Hurteau and Brooks, 2011]. At the landscape level it is  
77 expected that forest carbon will generally decline with thinning and burning efforts [McKinley *et*  
78 *al.*, 2011; Reinhardt and Holsinger, 2010]. Given these contrasting results, carbon sequestration  
79 in managed forests should be considered an “ancillary benefit,” and some have suggested that  
80 future management efforts designed for carbon retention should allow for continuous disturbance  
81 processes to occur over time [Hurteau and Brooks, 2011]. BC and other fire-altered residues  
82 have yet to be effectively incorporated in these stand-level carbon budgets, yet this may  
83 contribute to a slowly growing passive carbon pool in forest soils through the continued  
84 application of prescribed burning [DeLuca and Aplet, 2008; Hurteau and North, 2009].

85         Carbon in fire-altered residues exists on a continuum, ranging from partially-charred  
86 plant material to re-condensed soot and BC [Masiello, 2004; Preston and Schmidt, 2006]. The

87 ultimate end of the continuum is incombustible mineral ash, which results from the complete  
88 combustion of the charred residues [Smith and Hudak, 2005; Smith et al., 2005]. In the present  
89 study the term “charred residues” or “post-fire residues” simply refers to the material following a  
90 fire ( $\text{g m}^{-2}$ ). Due to inconsistencies in fire-altered carbon quantification procedures, the present  
91 study differentiates between pyrogenic carbon and black carbon, in order to distinguish the  
92 highly recalcitrant portion of the fire-altered carbon (Figure 1) [Keiluweit et al., 2010]. The term  
93 “pyrogenic carbon” (pyrC) refers to all fire-altered carbon in the post-fire residues, regardless of  
94 its resistance to degradation mechanisms. BC is defined as the highly resistant portion within the  
95 pyrC. Due to its thermally altered chemical structure, black carbon is believed to be highly  
96 resistant to degradation mechanisms once incorporated into forest soils [Preston and Schmidt,  
97 2006]. Furthermore, the degree of alteration in the chemical structure of the black carbon is  
98 dependent upon primarily fire temperature but heating duration also plays a role at lower  
99 burning temperatures, combined these factors are also known as charring intensity [Czimczik and  
100 Masiello, 2007; Keiluweit et al., 2010].

101 Charring intensity dictates the chemical structure of BC, and has been related to  
102 degradation rates of BC in laboratory settings [Nguyen et al., 2010]. During burning both  
103 aromatic (e.g. lignin) and non-aromatic (e.g. cellulose) compounds contribute to the formation of  
104 pyrogenic carbon [Shafizadeh, 1984]. However, fuels with high lignin-to-cellulose ratios  
105 produce greater amounts of BC probably as a result of the precursor having a similar chemical  
106 structure to the pyrogenic carbon [Czimczik et al., 2005]. While much research on black carbon  
107 formation has been conducted in laboratory settings, research conducted in wildfire and  
108 prescribed fire settings is limited, with very little research available accurately characterizing the  
109 mechanisms associated with the its formation, such as pre-fire fuel conditions and fire intensity

110 measurements [*Czimeczik and Masiello, 2007*]. This knowledge gap is likely due the inability to  
111 isolate and test variables in the field which likely affect the formation of fire altered carbon.  
112 Further experimental research is warranted to better account for the mechanisms driving BC  
113 production in the fire-altered residues in wildland fire settings [*Forbes et al., 2006*].

114         While BC is present in all fire-affected ecosystems, BC research has primarily focused  
115 within the boreal regions, temperate savannahs, and tropical rain forests, with very few studies  
116 evaluating BC production in the temperate forest types of the Western United States [*Donato et*  
117 *al., 2009; Tinker and Knight, 2001*]. In these forest types, fuel moisture is a key component  
118 dictating fire behavior, fire severity, and influencing post-fire ecological effects [*Reinhardt et al.,*  
119 *1991*]. To our knowledge no studies exist that evaluate the effects of fuel moisture on BC  
120 production, or the production of BC in masticated wildland fuels in dry temperate forests. Given  
121 that BC formation and the degree of thermal alteration is driven by charring intensity, it is likely  
122 that fuel moisture in wildland fuels will influence both the quantity of BC formed, as well as  
123 resistance to degradation of the BC produced. Research is warranted that accurately quantifies  
124 the conversion rates of biomass to BC in both managed and naturally occurring fuel types, as  
125 well as how specific particle characteristics (e.g. fuel moisture) influence the conversion process  
126 and ultimately the longevity of the post-fire charred materials generated in the fire event.

127         The current study evaluates how fuel moisture affects the conversion of masticated  
128 biomass to pyrC and BC. Previous carbon studies in masticated fuels have focused primarily on  
129 how fuel treatments influence changes in above-ground carbon pools, and found little change in  
130 soil carbon following treatments [*Stephens et al., 2009*]. However, these studies did not  
131 incorporate the conversion of biomass to BC, and acknowledge that BC may increase a stand's  
132 soil carbon pool over time [*Hurteau and North, 2009*].

133 In response to these needs, an experiment was designed to evaluate the role of moisture  
134 content on the production of BC in masticated fuels, representative of a mixed conifer stands  
135 commonly found throughout the interior Western United States. We conditioned 15 masticated  
136 fuel beds to simulate the range of fuel moistures (4-8%, 10-12%, and 13-16%) that exist in  
137 temperate dry mixed conifer forests. The objectives of this study were to (1) identify changes in  
138 fire behavior characteristics as a function of fuel moisture, (2) quantify the pyrC and BC in  
139 masticated fuels under the three pre-set fuel moisture groups, and (3) evaluate the relative degree  
140 of resistance to degradation of the fire-altered carbon.

## 141 **2. Methods**

### 142 **2.1 Fuel Bed Construction**

143 An 8-ha mixed conifer stand, comprised of white pine (*Pinus monticola*) and Douglas-fir  
144 (*Pseudotsuga menziesii*) ingrown with lodgepole pine (*Pinus contorta*), on the Clearwater  
145 National Forest (Latitude 46.801 N, Longitude 119.47 W) in Idaho was masticated in June 2009.  
146 Mastication thinning was implemented in the stand to reduce canopy bulk density and wildfire  
147 risk as well as improve stand health for the remaining trees. Masticated particles were primarily  
148 chipped into smaller diameter (< 7.6 cm) particles, although large-diameter >7.6 cm particles  
149 were also present. Fuel loading from this stand was established to ensure our laboratory created  
150 fuel beds closely resembled conditions in the field. The stand was sampled for fuel loading using  
151 square frame methods adapted from Hood and Wu (2006). Using a random start location within  
152 the stand, a quadrat (0.37 m<sup>2</sup>) was placed at distances of 5, 10, 15, 20, and 25 m from the starting  
153 point in the four cardinal directions for a total of 20 plots. Fuel bed height measurements were  
154 taken at the four corners of the quadrat and once in the center; and fuel bed bulk density was  
155 determined by dividing the dry weight of fuel within the quadrat by the volume of the fuel bed.

156 Fuels within the quadrat were removed to mineral soil, placed in a bag, and transported back to  
157 the University of Idaho's Fire Laboratory. Due to their high degree of spatial variability, fuels  
158 >7.6 cm in diameter were excluded from the experiment. Fuels from each of the 20 plots were  
159 then individually sorted into five different size classes (7.6 - 2.5 cm, 2.5 - 1.3 cm, 1.3 - 0.6 cm,  
160 and 0.6 - 0.3 cm, and needles), dried at 100°C until weights remained constant, and weighed.  
161 Fuel loadings for these fuel beds averaged 58.35 Mg ha<sup>-1</sup>, which were slightly lower than to  
162 masticated fuel loadings in mixed conifer stands measured in Colorado [Battaglia *et al.*, 2010].

163 In addition to samples taken for fuel loading determination, masticated fuels were  
164 collected *en masse* from the site and also sorted in a similar manner for the construction of the  
165 experimental fuel beds. Fifteen fuel beds were assembled by recombining the sorted particles,  
166 using estimates of average dry weights of each size class. Replicates of five fuel beds were  
167 designated to one of three pre-determined moisture levels of 3-8%, 10-12%, and 13-16%; fuel  
168 moisture was measured as a proportion of the dry weight.

169 The pre-constructed fuel beds were stored in a chamber with average fuel moistures  
170 ranging from 10-12%. The five fuel beds to be dried to 3-8% were taken from the control  
171 chamber the day of the burn trial and were dried in a convection oven for 4 hours at 37.5 °C, to  
172 simulate the temperature conditions similar to those of a hot summer day in the Northern  
173 Rockies when a wildfire might occur. The five fuel beds to be burnt at the 13-16% level were  
174 exposed to ambient conditions during the spring months of March and April to simulate  
175 conditions which might exist during prescribed fire settings. Prior to ignition for each burn, fuel  
176 moisture samples were taken from a small bin of excess fuel particles allowed to condition  
177 similar to the fuel beds (n = 10 for each size class of the fuel bed).

178 Samples of masticated wood particles and pine needles were analyzed for organic carbon  
179 (OC) content. The samples were milled in a grinder, dry sieved through a 500  $\mu\text{m}$  mesh screen,  
180 and analyzed for C and N through processes described below. Pre-fire organic carbon content for  
181 the organic matter was calculated by multiplying the %OC data for the needle and wood by the  
182 mass of the fuel bed ( $\text{g m}^{-2}$ ). This study only included surface organic matter and not the mineral  
183 soil horizon, as we did not wish to contaminate our samples with mineral soil.

## 184 **2.2 Active Fire Measurements**

185 The fuel bed was thoroughly mixed in a large bin for one minute, spread onto the burn  
186 table over the  $0.37 \text{ m}^2$  area, and compressed to achieve the average bulk density observed in the  
187 field. Temperature and relative humidity readings were recorded every five minutes throughout  
188 the trial with a Kestrel<sup>®</sup> 3000 pocket weather meter to validate that ambient conditions were not  
189 responsible for differences in fire behavior (Table 1). A dual band spectrometer was placed over  
190 the fuel bed and fire radiative power (FRP,  $\text{W m}^{-2}$ ) was logged at five second intervals throughout  
191 the burn trial, as a proxy for fire temperature. In addition, the fuel beds were burnt on a Sartorius  
192 EB Series scale (Goettingen, Germany), synchronized with the spectrometer to log mass loss  
193 throughout the burn period. To minimize the effects of unwanted conductive heat transfer  
194 through the surface of the scale a  $0.37 \text{ m}^2$   $3000^\circ\text{F}$  Ceramic Board (Cotronics Corp, Brooklyn,  
195 NY) was placed over the scale. Masticated fuels were placed directly on this board for burning to  
196 ensure that post-fire residue measurements were not contaminated by in-situ soil, unlike prior  
197 studies which were burnt on sand [Wooster *et al.*, 2005]. Measurements from the dual-band  
198 spectrometer sensor and scale were then initiated and allowed time to calibrate to ambient  
199 conditions. For ignition, a small amount of lighter fluid was added to a strip of excess material  
200 surrounding the area of observation, which was then ignited and allowed to carry the flame

201 through the area of observation. Once ignited, flame heights, flaming time, and smoldering time  
202 were recorded for each experimental trial, with the burn trial considered complete once mass loss  
203 ceased.

### 204 **2.3 Post-Fire Residue Analysis**

205 Several measurements were used to characterize the post-fire residues. Residue mass (g)  
206 was first established by weighing post-fire materials immediately following the burn trial.  
207 Residues were then dry sieved into 3 size classes (< 1mm, 1-6mm, and > 6mm) and weighed, as  
208 the composition of each size class was likely to vary as a function of parent materials [*Nocentini*  
209 *et al.*, 2010]. Two samples were then taken from each size class for further analysis, one for pyrC  
210 and the other for BC analysis (Figure 1). The concentrations of OC and nitrogen (N) in all  
211 samples were measured via high temperature combustion (ECS 4010, Costech Analytical,  
212 Valencia, CA). Carbon to Nitrogen (C:N) ratios were calculated by molar ratio. The stable  
213 isotopic compositions of OC ( $\delta^{13}\text{C}$ ) were determined on samples using high temperature  
214 combustion coupled with isotope ratio mass spectrometry (Delta Plus XP, Thermofinnigan,  
215 Bremen) [*Brenna et al.*, 1997; *Qi et al.*, 2003]. Isotopic reference materials were interspersed  
216 with samples for calibration. Contribution of  $\text{O}^{17}$  was corrected by the IRMS software using the  
217 Santrock correction [*Santrock et al.*, 1985]. PyrC concentration (%C) of the residue was  
218 multiplied by the mass ( $\text{g m}^{-2}$ ) of the post-fire residue to calculate pyrC production for each of  
219 the fuel beds ( $\text{g m}^{-2}$ ).

220 The second sample was analyzed for BC using an adapted two-step thermo-chemical  
221 method outlined in *Gustafsson et al.*, [1997] and *Hatten and Zabowski* [2009]. Samples were  
222 dried at 100°C, weighed, spread evenly across the bottom of a 35-mm ceramic crucible, and  
223 placed in a pre-heated muffle furnace at 375°C for 20 hours to remove any organic, non-

224 recalcitrant carbon. Care was taken to maintain a sample thickness of < 1mm to prevent  
225 additional pyrolysis from occurring in the muffle furnace. Samples were then treated with 6N  
226 HCl to remove any carbonates that may remain in the sample. Following the thermo-chemical  
227 treatment, the samples were elementally analyzed, using the same methods as described above  
228 for pyrC, BC and black nitrogen (BN) concentrations (%), and  $\delta^{13}\text{C}$  isotope determination. BC  
229 production ( $\text{g m}^{-2}$ ) was then calculated by multiplying the proportion of the sample resistant to  
230 thermal oxidation by the proportion resistant to chemical oxidation, and then by the mass of the  
231 post-fire residue.

232         The ratio of BC to pyrC then provided a proxy to determine the relative biologic  
233 recalcitrance of the post-fire carbon [*Hatten and Zabowski, 2009*]. Results were also normalized  
234 by pre-fire organic carbon content to determine pyrC and BC production rates. Inclusion of  
235 isotopic and nitrogen data provide insight to the elemental processes occurring during thermal  
236 degradation at different fuel moistures.

## 237 **2.4 Statistical Analysis**

238         One-way ANOVA with Tukey post-hoc analysis was used to test for differences across  
239 the three moisture groups for: (1) post-fire residue mass, (2) pyrogenic and BC concentrations,  
240 (3) pyrC production, (4) BC production, (5) the BC:pyrC ratio, (6) pyrC and BC isotopes, and (7)  
241 pyrC:N and BC:BN ratios. All statistical analysis was conducted using IBM SPSS predictive  
242 analytics software, version 19.

## 243 **3. Results and Discussion**

### 244 **3.1 Fire Characteristics**

245         Flame heights decreased from 30 cm in the driest fuels to 12 cm in the 13-16% fuel  
246 moisture group, visually indicating increased fire intensity at lower fuel moistures (Table 2).

247 Furthermore, duration of flaming combustion was shorter in the 4-8% (6 min 54 sec) and 10-  
248 12% (10 min 02 sec) relative to the 13-16% moisture group (14 min 44 sec), as flames rapidly  
249 advanced across the table at lower fuel moistures. FRP data corroborate the flaming combustion  
250 results, and show much higher peak radiant flux values in the 4-8% moisture group compared to  
251 the 13-16% moisture group (Figure 2).

252 Peak FRP flux recordings occurred when the fuel bed was entirely engaged in flaming  
253 combustion. Differences in peak FRP flux times were apparent between the two moisture groups,  
254 with the dry fuels producing peak FRP values around 500-600 seconds after ignition, and the wet  
255 fuels peaking at approximately 1000-1100 seconds after ignition. While smoldering combustion  
256 times were highly variable and yielded no significant results, radiant heat release dropped below  
257 the detection levels in the 4-8% fuel moisture group at 2000 seconds, and close to 2500 seconds  
258 in the 13-16% moisture group, indicating sustained low intensity smoldering combustion in the  
259 13-16% fuel moistures. The differences in FRP values between wet and dry fuels in the present  
260 study correspond well with surface soil temperature data measured using thermocouples in  
261 masticated fuels evaluating lethal soil temperatures, which indicate peak soil temperatures of  
262 773-873°K occurring early in the burn when fuel moistures were near 2%, and peak temperatures  
263 of 673-773°K occurring later in the burn when fuel moistures were 16% [Busse *et al.*, 2005].

### 264 **3.2 Carbon Concentrations**

265 Carbon concentrations for pyrC ranged from 15.37 to 74.45%, with the < 1mm particles  
266 displaying the lowest pyrC concentrations, and the > 6mm particles containing the highest  
267 concentration (Table 3). The lack of pyrC in the < 1mm size class may be a function of the high  
268 ash content in this size class [Nocentini *et al.*, 2010]. Trends across fuel moisture, regardless of  
269 residue size, show a slight increase between 4-8% and 10-12%, with the least amount of pyrC

270 being contained in the 13-16% moisture group. In contrast, BC concentrations were the greatest  
271 in the 13-16% moisture group (ranging from 0.49 to 2.65%), regardless of residue size. Although  
272 the 13-16% moisture groups contained the smallest pyrC concentrations, a greater percentage of  
273 the residue was converted to BC. Also, in the > 6mm size class in the 13-16% fuel moisture  
274 group the concentration of BC was an order of magnitude greater than the other moisture groups  
275 or any other size classes (Table 3).

### 276 **3.3 Post-fire Residues**

277         Once fire had established in the masticated fuel beds it sustained the combustion reaction  
278 until nearly 90% of the fuel bed was consumed regardless of moisture group (Table 4). Weight  
279 of the total post-fire residues, and residues by size class also did not vary as a function of fuel  
280 moisture (Figure 3a). Dry biomass conversion to post-fire charred residues and ash averages  
281 were 9.60 (2.73)%, 8.78 (1.95)%, and 10.22 (3.38)% for the 4-8%, 10-12%, and 13-16% fuel  
282 moisture ranges, respectively. Despite sampling differences and fire behavior, these values are  
283 very similar to conversion rates of 8.0% measured volumetrically by *Tinker and Knight* [2001],  
284 who only measured charred residues in coarse woody debris in Yellowstone National Park. Other  
285 studies measuring charcoal production via gravimetric and volumetric means are difficult to  
286 compare to, as they tend to use approximate carbon values for the post-fire residues [*Fearnside*  
287 *et al.*, 1999; *Forbes et al.*, 2006]. Although total fuel bed consumption did not significantly differ  
288 in our statistical analysis as a function of fuel moisture, the average post-fire residue weight in  
289 the 13-16% fuel moisture group was slightly higher than the average weight of the two dryer fuel  
290 moisture groups. Within the post-fire residues, the majority of the residue was allocated in the <  
291 1mm and > 6mm size classes, perhaps indicating that the majority of the pine needles were either  
292 volatilized or fragmented to <1mm in size. The 4-8% and 10-12% fuel moistures contained the

293 majority of their mass in the <1mm size, while the 13-16% fuel moistures contained the largest  
294 amounts of post-fire mass in the >6mm size class. These results suggest that at higher fuel  
295 moistures the masticated woody particles only partially combusted and did not fully consume or  
296 fragment to smaller sizes, unlike masticated fuels burnt under drier conditions.

### 297 **3.4 Pyrogenic Carbon Production**

298 Average pyrC production in the post-fire residues for the three moisture classes ranged  
299 from 200.63 to 252.32 g m<sup>-2</sup> (Table 4). Although there were no significant differences between  
300 fuel moisture groups, pyrC content generally increased with fuel moisture, which is a function of  
301 the slightly higher residue weight (Figure 3b). Conversion rates of the pyrC relative to pre-fire  
302 OC range from 7.23 to 8.54% (Table 4). When rates are normalized by total pre-fire dry weight,  
303 pyrC production ranges from 3.52 to 4.22% (data not shown), which compare well with  
304 conversion rates of 4.8% observed in a slash and burn study in a temperate deciduous forest,  
305 utilizing elemental analysis of the post-fire residues [Eckmeier *et al.*, 2007]. Similar to the slash  
306 and burn fuel type measured in Eckmeier [2007], masticated fuel beds in the present study also  
307 contained a large proportion of small diameter woody materials. Production rates are slightly  
308 higher than those observed in South American rain forests of 1-3%, which is likely a function of  
309 the large woody component in the masticated fuels [Fearnside *et al.*, 1999; Fearnside *et al.*,  
310 2001].

311 Similar to the trends in concentration, most of the pyrC produced was allocated in the >  
312 6mm size class charred particles, while the least amount of pyrC located in the < 1mm residues.  
313 Pyrogenic carbon slightly decreased in the small size fraction with increased fuel moisture, but  
314 increased with fuel moisture in both the 1 - 6mm and > 6mm size fractions.

### 315 **3.5 Black Carbon Production**

316 Similar to the pyrC production, total BC production significantly increased from 0.02%  
317 to 0.05% between the 13-16% moisture group and the two drier fuel moisture groups (Table 4).  
318 This increase in BC production as a function of fuel moisture was observed in all size classes,  
319 although the medium-size residues showed no significant change between the moisture classes  
320 (Figure 3c). In contrast to the pyrC results, the majority of BC was allocated in the fine (< 1mm)  
321 size fraction (0.27, 0.29, and 0.90 g m<sup>-2</sup> for the 4-8%, 10-12%, and 13-15% fuel moisture groups,  
322 respectively), suggesting that at least some of this material was likely BC formed through the  
323 recondensation of volatiles, and highly resistant to our methods of thermal and chemical  
324 degradation. These results also appear contrary to *Nocentini et al.* [2010] who suggest that the  
325 coarsest size fraction contains the largest amount of highly aromatic structures. However, their  
326 elemental analysis was limited to what this study defines as pyrC, and they did not quantify BC  
327 as this study does. Additionally, *Nocentini et al.* [2010] limited their largest size class to > 2mm,  
328 which we included in our middle size class. The coarse charred material, while high in pyrC,  
329 contained the least amount of BC (from 0.002 to 0.007 g m<sup>-2</sup>). These results suggest the  
330 possibility that the fire temperature required to convert the organic carbon to BC did not achieve  
331 high enough temperatures for conversion towards the interior of the macro-size woody particles.

332 These results appear somewhat counter-intuitive compared to other studies which find  
333 that BC production increases with fire severity in pine needles [*Hatten and Zabowski, 2009*].  
334 These results suggest that under lower fuel moisture levels (e.g. increased fire intensities) BC is  
335 preferentially volatilized, as overall fuel bed consumption did not vary.

### 336 **3.6 Black Carbon to Pyrogenic Carbon Ratio**

337 The ratio of black carbon to pyrogenic carbon reflects the relative degree of resistance to  
338 biological degradation in the fire-altered residue [*Hatten and Zabowski, 2009*]. Similar to the

339 pyrogenic and BC production, the ratio for the total fuel bed significantly increased with fuel  
340 moisture, indicating a greater resistance to chemical and thermal degradation at higher fuel  
341 moistures (Figure 3d). Similar to BC production, the highest ratio was observed in the fine (<  
342 1mm) sized particles (averaging 0.007, 0.007, and 0.03 for 4-8%, 10-12% and 13-16% moisture  
343 groups, respectively, Table 4). These results suggest that that the majority of BC created in  
344 masticated fuels exist in the > 1mm size class. The < 1mm size class represented the majority of  
345 the post-fire residues regardless of moisture. It also maintained the highest BC:pyrC ratio,  
346 suggesting that this size class is important in the formation of BC in masticated fuels. The  
347 smallest BC:pyrC ratios were observed the coarse (> 6mm) size residues, indicating that while  
348 large quantities of pyrC are created in this size class, most of it will be subjected to future  
349 degradation.

### 350 **3.7 Composition of Fire Altered Organic Residues**

351 The largest pyrC:N ratio was observed in the > 6mm size class, with smaller ratios in the  
352 two smaller size classes (Table 5). These trends are similar to the unburnt fuel C:N ratios,  
353 suggesting that fragmentation of the woody particles was not a significant contributor to pyrC:N  
354 ratios. If even a small amount of the fuel particles were fragmented from a larger size class (e.g.  
355 > 6mm) size fraction to one of the smaller size fractions (e.g. 1 - 6mm) there would have likely  
356 been a large increase in the C:N ratio of the smaller size classes. This hypothesis is supported by  
357 a lack of any substantial trend in the  $\delta^{13}$  pyrC isotope data, and variation in the isotope data is  
358 likely due to variability in the parent material [*Bird and Ascough, 2012*].

359 Regardless of size class, pyrC:N ratios were less than unburnt C:N ratios, indicating that  
360 regardless of fuel moisture, C was preferentially volatilized relative to N (Table 5). Increased  
361 fuel moisture decreased fire intensity, thus leading to changes in the overall composition of the

362 post-fire residues. Fuel moisture had a significant effect on pyrC:N in the > 6mm and < 1mm  
363 size classes, where lower C:N ratios were found at higher fuel moistures. Our data support the  
364 concept that the presence of N in organic matter is important in the production of highly  
365 recalcitrant black carbon. Previous work has suggested that nitrogen provides stability to chars at  
366 temperatures under 450°C, and C:N ratios increase with temperatures above this threshold  
367 [Knicker *et al.*, 2008; Knicker, 2010]. These trends are attributed to the thermal stability imparted  
368 by N and a preferential combustion of low-N containing organic matter [Knicker *et al.*, 2008].  
369 However, the N associated with pyrogenic materials is less recalcitrant than the associated C [*de*  
370 *la Rosa and Knicker*, 2011]. Despite increased resistance to thermal degradation, the presence of  
371 greater amounts of N may eventually lead to higher microbial decomposition rates of the  
372 organic residue than would be expected for a residue with a high C:N.

373         The stable isotopic composition of the <6mm fractions was lighter than the unburned  
374 material suggesting that lignin (typically ~3 ‰ lighter than cellulose) was preferentially  
375 preserved or transformed into pyrC. The slightly heavier, yet statistically significant, stable  
376 isotopic composition of the <1mm fraction burnt at the highest fuel moisture suggests that more  
377 carbon from cellulose was preserved relative to the higher intensity treatments. We hypothesize  
378 that heavier isotopic composition and lower C:N ratios was a result of N thermally stabilizing  
379 pyrC at this higher fuel moisture and low burn intensity. A possible mechanism could be that  
380 products of cellulose pyrolysis (i.e. depolymerization and release of anhydro sugars) reacted with  
381 N containing compounds in the Maillard reaction forming thermally stable compounds such as  
382 pyridinies, pyrazines, and pyrroles.

383         At low fuel moistures it appears that more indiscriminate combustion of all compounds  
384 occurred, as C:N ratios trended closer towards unburned materials. A Pearson correlation

385 analysis between the BC:pyrC ratio and C:N ratio, produced a moderate negative correlation of  
386  $R = -0.602$ , ( $p = < 0.001$ ), supporting the idea that the presence of N is important in the  
387 generation of fire altered residues with high BC:pyrC ratios.

388 BC to black nitrogen ratios are much lower than pyrC:N ratios, further supporting the  
389 concept that N plays a strong role in thermal stabilization of C [Knicker, 2010]. Since more N  
390 may be available to react and form BC, the selective preservation of N in the higher fuel  
391 moisture may be causing the increased rates of BC production in the 13-16% fuel moisture  
392 group. BC  $\delta^{13}\text{C}$  data in the  $> 6\text{mm}$  size class again support the theory that indiscriminate  
393 combustion is occurring at lower fuel moistures, with lignin and cellulose being preferentially  
394 being removed at higher fuel moistures.

#### 395 **4. Conclusions**

396 The production of BC and elemental composition of our post-fire residues varied as a  
397 function of fuel moisture, with highest BC production rates and BC:pyrC ratios in residues  
398 burned at 13-16% fuel moisture. Fire intensity differences (as controlled by fuel moisture) are  
399 likely a key driver in BC production and/or BC volatilization in masticated fuels, where in dry  
400 fuels pre-cursors to BC were indiscriminately consumed at high radiant intensities early on in the  
401 combustion process. Fuels burnt at higher fuel moistures tended to preserve these precursors for  
402 the eventual conversion to BC. The threshold fuel moisture between BC production and  
403 volatilization in our masticated fuel beds occurred between 12-13%. Fuel moisture is an easily  
404 measurable variable and often dictates fire management decisions, especially in prescribed fires.  
405 Management strategies that emphasize carbon retention should consider burning when fuel  
406 moistures are greater than 12% if possible, to maximize the BC conversion and minimize  
407 volatilization, though this should be validated with field measurements. With low production

408 rates of 0.02 to 0.05% (relative to pre-fire OC), BC is likely not an important contributor to  
409 stand-level soil carbon in the short term (e.g. one fire event). Over time, and multiple fire events,  
410 BC may slowly increase a stand's long-term carbon pool if it can be sufficiently insulated from  
411 degradation mechanisms. However, it remains unclear whether or not multiple fire events create  
412 more BC, or reduce BC levels through the burning of unprotected BC [Rovira *et al.*, 2009].

413         The experimental approach used in this study allowed for a unique opportunity to control  
414 and replicate burns under fixed fuel moistures. The utilization of the dual-band thermal infrared  
415 sensor allowed for accurate characterization of radiant heat release throughout the entire burn  
416 period. This sensor, or similar sensors, should be considered for future studies evaluating fire  
417 behavior and subsequent effects in conjunction with thermocouples for correlation between fire  
418 temperature and FRP, as these "remotely sensed" data may be scaled up to stand and landscape-  
419 level prescribed fires and wildfires. Future BC production studies should attempt to accurately  
420 characterize both the pre-fire fuel characteristics as well as fire behavior, as both have been  
421 shown to play a critical role in determining the post-fire composition of the residues. Moreover,  
422 studies evaluating the persistence of fire-altered carbon should include both experimental  
423 combustion studies as well as field studies in a broad range of fuel types, as well as field  
424 validation of our results.

425         Future research to characterize fire behavior in masticated fuel beds and the associated  
426 fire-effects, including BC formation, are essential. This is especially needed in stands managed  
427 for multiple objectives where both carbon retention and fuels reduction are favored. Recent  
428 events such as the Lower North Fork Fire escape in Colorado, 2012, highlights the lack of  
429 knowledge into predicted fire behavior, including duration of smoldering combustion beneath the  
430 surface, within such masticated fuel beds. Improved understanding is needed to characterize the

431 behavior and effects under varying bulk densities and wind speeds. As noted earlier, very little  
432 research has been conducted in wildfire and prescribed fires to test how specific fuel properties  
433 and weather characteristics influence fire behavior and subsequent production of BC. Given the  
434 inherent challenges of accurately measuring coincident pre-fire loadings, fire behavior  
435 measurements, and post-fire residues; an experimental approach similar to that conducted herein  
436 may prove useful.

437

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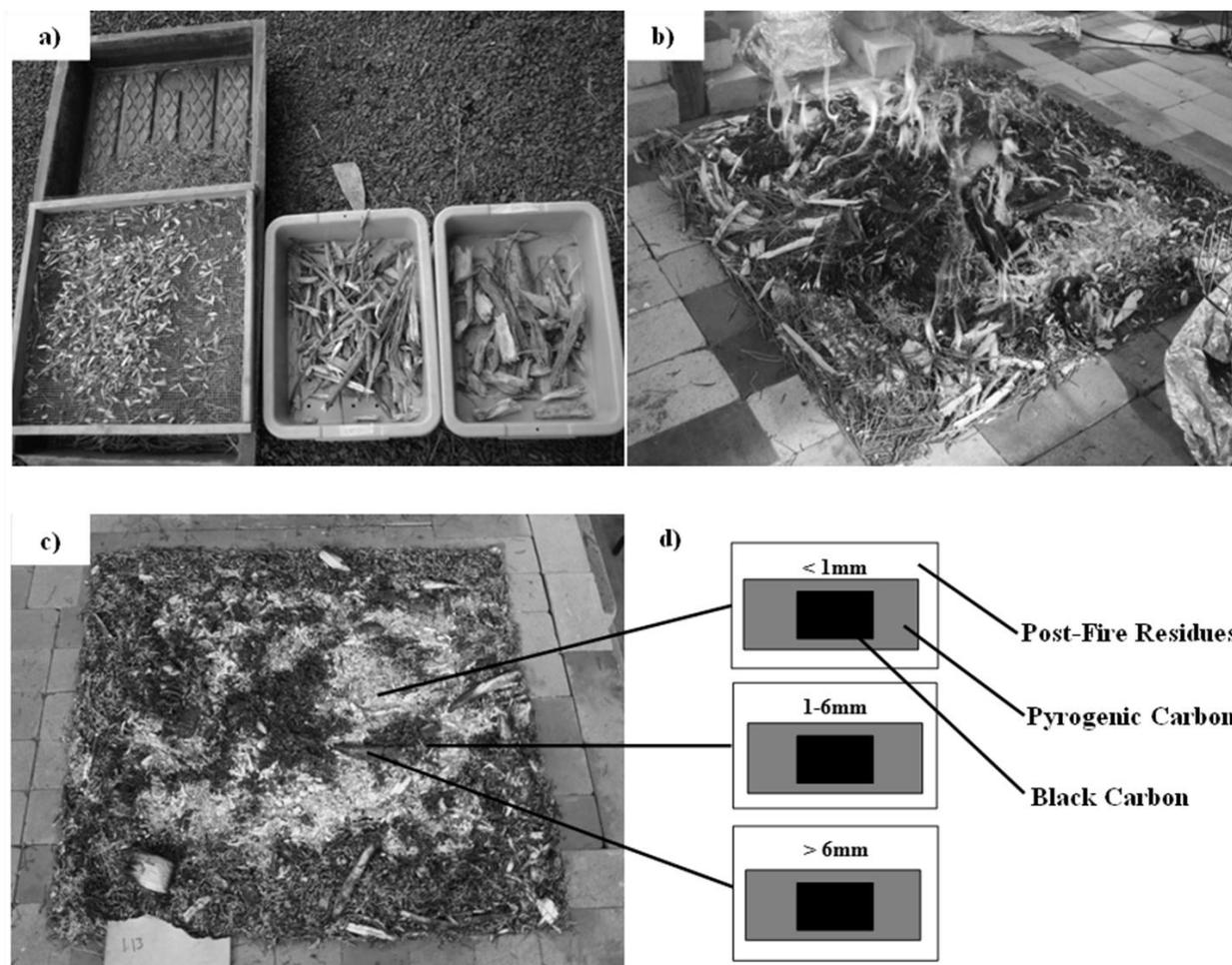
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574 **Figures:**



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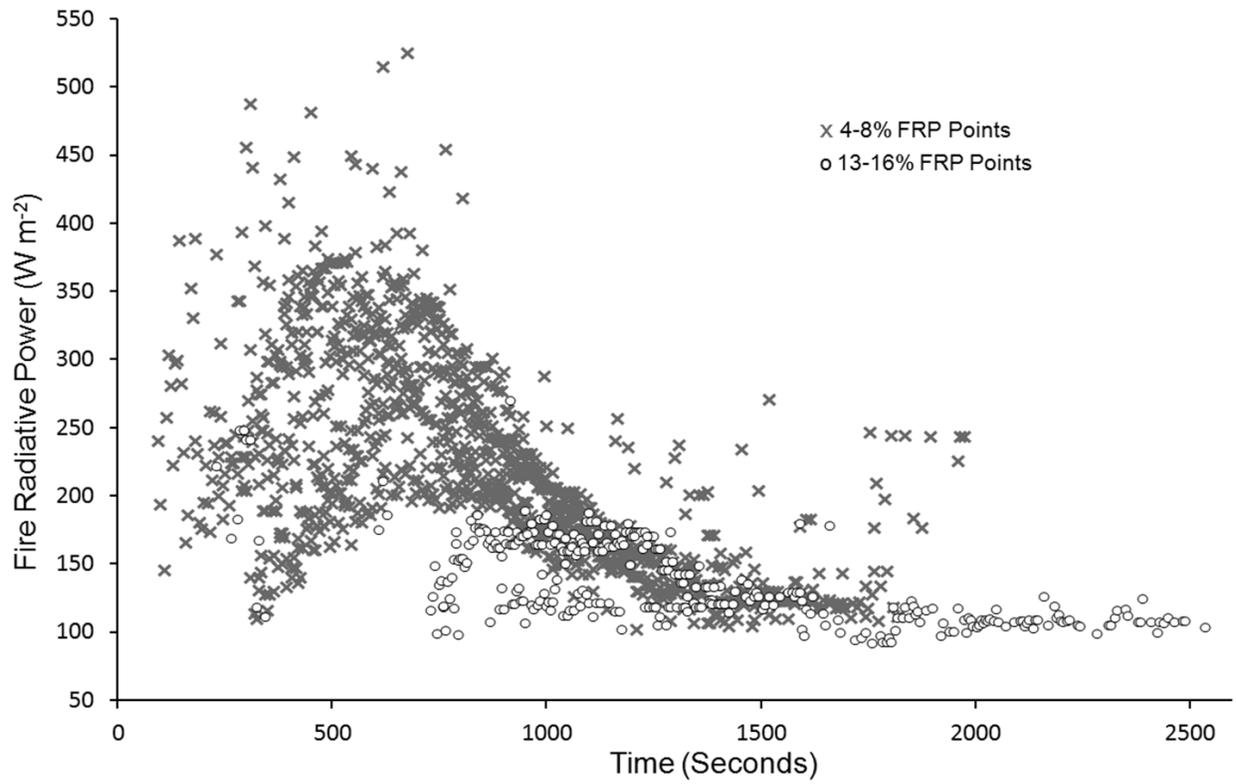
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577 **Figure 1:** Images of: a) fuel bed construction of the individual size classes, b) a masticated fuel

578 bed during combustion, c) post-fire residues, and d) a conceptual diagram of the carbon

579 components associated with the residues after sieving.

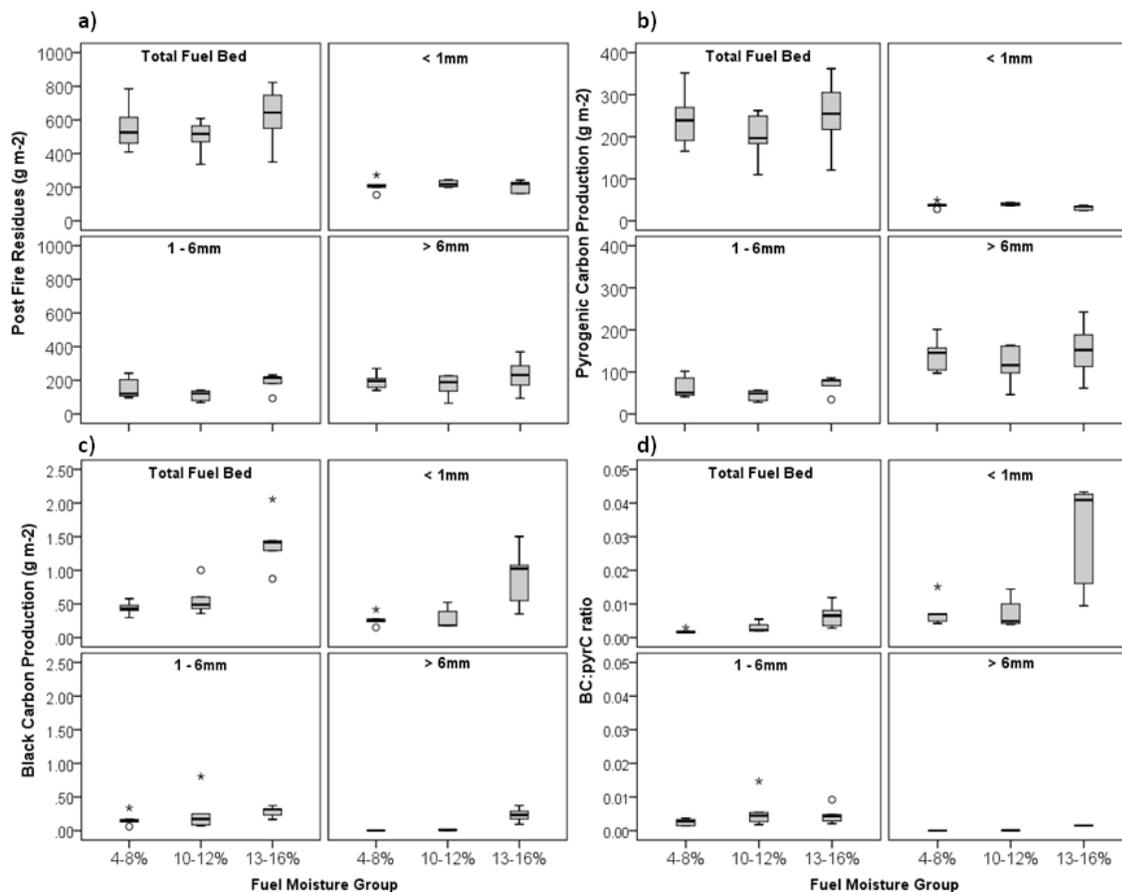
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582 **Figure 2:** Fire Radiative Power (FRP) flux points for the 4-8% and 13-16% fuel moisture groups  
583 (n = 5 for each moisture group).

584



585

586 **Figure 3:** Post fire components by moisture group and size class, a) residue weight, b) pyrC  
 587 production, c) BC production, and d) The BC to pyrC ratio. Outlier values  $>1$  and  $>2$  standard  
 588 deviations away from the median are shown by \* and  $^{\circ}$  respectively.