Biogeosciences, 18, 1–17, 2021 https://doi.org/10.5194/bg-18-1-2021 © Author(s) 2021. This work is distributed under the Creative Commons Attribution 4.0 License.

# Experimental production of charcoal morphologies to discriminate fuel source and fire type: an example from Siberian taiga

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Abstract. The analysis of charcoal fragments in peat and lake sediments is the most widely used approach to reconstruct past biomass burning. With a few exceptions, this method typically relies on the quantification of the total char-<sup>5</sup> coal content of the sediment. To enhance charcoal analyses for the reconstruction of past fire regimes and make the method more relevant to studies of both plant evolution and fire management, the extraction of more information from charcoal particles is critical. Here, I used a muffle <sup>10</sup> oven to burn seven fuel types comprising 17 species from boreal Siberia (near Teguldet village), which are also com-

- monly found in the Northern Hemisphere, and built on published schemes to develop morphometric and finer diagnostic classifications of the experimentally charred particles. I then 15 combined these results with those from fossil charcoal from a
- peat core taken from the same location (Ulukh-Chayahken mire) in order to demonstrate the relevance of these experiments to the fossil charcoal records. Results show that graminoids, *Sphagnum*, and wood (trunk) lose the most
- <sup>20</sup> mass at low burn temperatures (< 300 °C), whereas heath-land shrub leaves, brown moss, and ferns lose the most mass at high burn temperatures. This suggests that species with low mass retention in high-temperature fires are likely to be under-represented in the fossil charcoal record. The charcoal</li>
  <sup>25</sup> particle aspect ratio appeared to be the strongest indicator of the fuel type burnt. Graminoid charcoal particles are the most elongate (6.7–11.5), with a threshold above 6 that may be indicative of wetland graminoids; leaves are the shortest and bulkiest (2.1–3.5); and twigs and wood are intermediate (2.0–30 5.2). Further, the use of fine diagnostic features was more
- successful in separating wood, graminoids, and leaves, but it was difficult to further differentiate these fuel types due to overlapping features. High-aspect-ratio particles, dominated

by graminoid and Sphagnum morphologies, may be robust indicators of low-temperature surface fires, whereas abun- 35 dant wood and leaf morphologies as well as low-aspect-ratio particles are indicative of higher-temperature fires. However, the overlapping morphologies of leaves and wood from trees and shrubs make it hard to distinguish between high-intensity surface fires, combusting living shrubs and dead wood and 40 leaves, and high-intensity crown fires that have burnt living trees. Distinct particle shape may also influence charcoal transportation, with elongated particles (graminoids) potentially having a more heterogeneous distribution and being deposited farther away from the origin of fire than the rounder, 45 polygonal leaf particles. Despite these limitations, the combined use of charred-particle aspect ratios and fuel morphotypes can aid in the more robust interpretation of fuel source and fire-type changes. Lastly, I highlight the further investigations needed to refine the histories of past wildfires. 50

### 1 Introduction

Wildfires are the most common disturbance type in boreal forests, triggering gap dynamics or stand-scale forest replacement depending on intensity (temperature of fire) and frequency (Goldammer and Furayev, 1996). Ongoing and anticipated increases in the intensity and frequency of wildfire in boreal forests is raising concerns about its impact on forest composition as well as climate (Jones et al., 2020). Although the largest area of boreal forest globally is in Siberia (Goldammer and Furayev, 1996), its extent and access restrictions mean there are only a few datasets recording changes in wildfire activity, especially from a longer-term perspective (Marlon et al., 2016). Long-term records of wild-

fire activity are vital to understanding how fire regimes vary with changes in climate and human–vegetation interaction, as well as the impacts of fires on boreal forests.

Wildfires can reach temperatures of up to 1800 °C. Char-5 coal, however, is an inorganic carbon compound resulting from the incomplete combustion of plant tissues, which typically occurs at temperatures between 280 and 500 °C (Rein, 2014). Charcoal particles vary with respect to size and form, and characteristics, such as edge aspects, surface features,

- <sup>10</sup> cleavage, lustre, or anatomical details (e.g. tracheids with border pits, leaf veins, and cuticles), that can be used to determine their origin are often preserved (Ward and Hardy, 1991; MacDonald et al., 1991; Scott, 2010; Enache and Cumming, 2006; Jensen et al., 2007; Courtney-Mustaphi and Pis-
- <sup>15</sup> aric, 2014a; Hubau et al., 2012, 2013; Prince et al., 2018). Although macroscopic charcoal analysis, typically counting charcoal pieces or charcoal area per unit sediment volume from a small sediment volume (1–2 cm<sup>3</sup>), is widely done, there has been less focus on the reconstruction of fuel
- 20 sources. However, this is a crucial factor in determining fire type (i.e. the burning of surface fuels in low- or high-intensity fires) or distinguishing between surface and crown fires At a minimum, this requires a better characterisation of charcoal morphology to indicate the nature of the plant material
- <sup>25</sup> burnt (Courtney-Mustaphi and Pisaric, 2014a, b; Feurdean et al., 2017; Hawthorne et al., 2018). The determination of fire type is not only helpful for palaeo-fire reconstructions but could also provide an accessible tool for ecosystem managers and modellers as well as for assessing and mitigating
- <sup>30</sup> the risks of fires that might impact settlements and infrastructures (Moritz et al., 2014).

Ongoing efforts have advanced the utility of charcoal morphological analyses for fuel-type identification and fire regime reconstruction. Umbanhowar and McGrath (1998),

- <sup>35</sup> Crawford and Belcher (2014), and Pereboom et al. (2020) conducted morphometric measurements of the length, aspect ratio (length / width), and surface area of charcoal particles by burning known plant materials originating from American prairie, tropical, and arctic
- <sup>40</sup> ratory. They concluded that longer fragments correspond to graminoids, whereas shorter fragments originate from wood, shrubs, and leaves. Nichols et al. (2000) and Crawford and Belcher (2014) additionally found that charcoal morphometry is generally preserved during transportation by water.
- <sup>45</sup> Other studies have focused on the effects of burning conditions, i.e. open-flame ignition, muffle furnace experiments (Umbanhowar and McGrath, 1998; Orvis et al., 2005), and combustion calorimetry (Hudspith et al., 2018), on charcoal production. In a laboratory study, Belcher et al. (2005, 2015)
  <sup>50</sup> investigated whether fire can be ignited by thermal radiation and may have been the reason for major extinction events in deep geological times, with results giving little support for this hypothesis. Jensen et al. (2005) and Courtney-Mustaphi and Pisaric (2014a) examined subtler diagnostic
  <sup>55</sup> features (morphology, surface features, lustre) of laboratory-

produced charcoal morphotypes of a small number of North American grasses and leaves of coniferous and deciduous trees. Enache and Cumming (2006, 2007) and Mustaphi and Pisaric (2014a) classified charcoal morphologies in Canadian lake sediments based on particle shape, aspect ratio, and sur- 60 face features, and linked these morphometric characteristics to fuel types. Courtney-Mustaphi and Pisaric (2014a) also discussed the potential for categorising charcoal morphologies to explore relationships with taphonomic processes and fuel types. A number of recent studies have attributed fossil 65 charred particles to specific fuel and fire types based on published morphotype categorisations (Walsh et al., 2010 Daniau et al., 2013; Aleman et al., 2013; Leys et al., 2015; Courtney-Mustaphi and Pisaric, 2014a, b, 2018; Feurdean et al., 2017, 2019, 2020; Feurdean and Vasiliev, 2019; Unkel- 70 bach et al., 2018).

This paper presents the first results of laboratory-produced (muffle oven) charcoal morphologies spanning a range of fuel types from 17 Siberian species, with the aim of characterising the diversity of charcoal morphologies produced by 75 boreal understorey and forest vegetation to facilitate more robust interpretations of fuel sources from this region. Specifically, this work (i) evaluates whether there are morphological distinctions (morphometrics and finer anatomical features) between species or fuel types, and (ii) explores the effect of 80 burning temperature on the mass, morphometrics, and finer anatomical features of charred plant material. This combination of factors has not been widely tested in the laboratory, so this study has the potential to advance our understanding of the link between sedimentary charcoal morphologies and fire 85 types and, as the species occur across most of the Northern Hemisphere, refine wildfire histories in boreal regions.

#### 2 Material and methods

#### 2.1 Laboratory analysis

Plant materials used for laboratory burning experiments were <sup>90</sup> identified in the field, stored in plastic bags for transportation, and air-dried. Selected materials include a range of fuel types (graminoid, trunk wood, twigs from trees and shrubs, leaves from coniferous and deciduous trees, shrubs, forbs, and ferns, and fern and moss stems with leaves) from the most common <sup>95</sup> tree, shrub, herb, fern, and moss species around a forested bog near Teguldet village, Tomsk district, Russia. This light boreal taiga forest is primarily composed of *Pinus* and *Betula*. Additionally, needles and twigs of *Picea abies* were collected from Taunus, near Frankfurt am Main, Germany (Table 1). All plant material was collected from living plants, except trunk wood, which was taken from a dead tree.

To determine the mass, morphometrics, and finer diagnostic features of residual charred plant material as well the effect of increasing temperatures on all these characteristics, <sup>105</sup> the dried remains of individual plant species were placed in

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**Table 1.** List of plant materials burned. All plants are from Siberia except for *Picea abies*, which originated in Taunus, Germany. All plant material was air-dried before combustion in the muffle oven. Leaves of deciduous trees and shrubs include veins and petioles. The term "twig" was only used for woody species (i.e. deciduous and coniferous trees and shrubs), and no distinction was made between soft young wood.

Plant type	Scientific name	Family	Common name	Plant burned
Trees				
Conifer tree	Pinus sylvestris	Pinaceae	Scots pine	Needles
Conifer tree	Pinus sylvestris	Pinaceae	Scots pine	Twigs
Conifer tree	Pinus sylvestris	Pinaceae	Scots pine	Dead wood
Conifer tree	Pinus sibirica	Pinaceae	Siberian pine	Needles
Conifer tree	Pinus sibirica	Pinaceae	Siberian pine	Twigs
Conifer tree	Picea abies	Pinaceae	Norway spruce	Needles
Conifer tree	Picea abies	Pinaceae	Norway spruce	Twigs
Deciduous tree	Betula pendula	Betulaceae	Silver birch	Leaves
Deciduous tree	Betula pendula	Betulaceae	Silver birch	Twigs
Shrubs				
Shrub	Vaccinium myrtillus	Ericaceae	Bilberry	Leaves
Shrub	Vaccinium myrtillus	Ericaceae	Bilberry	Twigs
Shrub	Oxycoccus palustre	Ericaceae	Cranberry	Leaves
Shrub	Oxycoccus palustre	Ericaceae	Cranberry	Twigs
Shrub	Empetrum nigrum	Ericaceae	Crowberry	Leaves
Shrub	Empetrum nigrum	Ericaceae	Crowberry	Twigs
Shrub	Ledum palustre	Ericaceae	Wild rosemary	Leaves
Shrub	Ledum palustre	Ericaceae	Wild rosemary	Twigs
Shrub	Chamaedaphne calyculata	Ericaceae	Leatherleaf	Leaves
Shrub	Chamaedaphne calyculata	Ericaceae	Leatherleaf	Twigs
Herbaceous				
Graminoid	Eriophorum vaginatum	Cyperaceae	Cotton grass	Leaves
Graminoid	Calmagrostis CE4	Poaceae	Reed grass	Leaves
Graminoid	Carex spp.	Cyperaceae	Sedge	Leaves
Forb	Cnidium dubium	Apiaceae	Leaves	
Forb	Rubus spp.	Rosaceae	Raspberry	Leaves
Fern	Polypodium	Polypodiaceae	Fern	Leaves
Fern	Equisetum palustre	Equisetaceae	Horsetail	Stem
Moss	Sphagnum spp.	Sphagnaceae	Peat moss	Stem and leaves
Moss	Polytrichum commune	Polytrichiaceae CE5	Hair moss	Stem and leaves

ceramic crucibles, weighed, covered with a lid to limit oxygen availability and avoid mixing of the charred particles, and heated for 2 h in a muffle oven (preheated for 1 h); they were then roasted at 250, 300, 350, 400, or  $450 \degree$ C (File S1 in

- <sup>5</sup> the Supplement). No burning experiments were conducted at higher temperatures because all plant material turned to ash, i.e. a solid residue mostly composed of minerals that crumbled apart into soot and fly ash (Rein et al., 2014). The effect of mixing plant material in known ratios on charred mass
- <sup>10</sup> and morphometrics at an intermediate temperature (300 °C) was also tested. For this experiment, the plant material was combined in volumes to approximate the predominant fuel mixtures for low-intensity surface fires (with a ratio of 75 % graminoid and moss to 25 % shrub, and 50 % graminoid to 50 % mean and fem) intermediate to high intensity surface

 $_{15}$  50 % moss and fern), intermediate- to high-intensity surface

fires (with a ratio of 25 % graminoid and moss to 75 % shrub, and 50 % graminoid and moss to 50 % shrub), and highintensity crown fires (with a ratio of 50 % graminoid, shrub, and moss to 50 % wood and leaf). The experimental temperatures were chosen based on the range of temperatures reported in the literature (250–500 °C; Umbanhowear and Mc-Grath, 1998; Orvis et al., 2005; Jensen et al., 2007; Pereboom et al., 2020). Although dry roasting in a muffle oven approximates some aspects of the heating conditions of vegetation in a natural fire, it does not explore the impact of how long the material was at specific burning temperature and oxygen conditions (Belcher et al., 2015; Hudspith et al., 2017[ISS]). Dry roasting also reduces the influence of flame dynamics and turbulent airflow; therefore, plant tissue is more rapidly reduced to ash than in natural fires. After cooling, the remain-30 ing charred mass of each sample was weighed and the ratio of charred to pre-combustion mass was calculated. Charred samples were then split into two subsets. The first was left intact and stored as reference material. The second subsample

- $_{5}$  was gently disaggregated with a mortar and pestle to mimic the natural breakage that charcoal particles incur over time in sediment (Umbanhowar and McGrath, 1998; Crawford and Belcher, 2014; Belcher et al., 2015) and was then washed through a 125 µm sieve to remove smaller fragments. This
- <sup>10</sup> second subsample was used to make morphometric measurements and to characterise finer diagnostic features. Morphometric measurements of individual charred particles were obtained from photographs taken at  $4 \times$  magnification with a digital camera (Kern DXM 1200FCEG). On average, more
- <sup>15</sup> than 100 charcoal particles larger than 150  $\mu$ m were automatically detected in most samples, except for those burnt at higher temperatures, where particles were more prone to breaking up. The major (*L*) and minor (*W*) axes, along with surface area (*A*) of each particle were measured following
- <sup>20</sup> the algorithm given in Appendix A1, and the aspect ratio L/W was calculated. Finer diagnostic features such as shape, surface features (e.g. reticulates, tracheids with border pits, leaf veins, the arrangement of epidermal cells, and cuticles with stomata), and cleavage were characterised at  $4 \times 1000$
- <sup>25</sup> by inspection of microphotographs or observations of the charred particles themselves under a microscope or stereomicroscope.

To demonstrate the applicability of these experiments to fossil records, seven samples with high charcoal content were

<sup>30</sup> selected from a sediment core from Ulukh-Chayakh mire near Teguldet village (Feurdean et al., 2021 [155]). Sample preparation followed Feurdean et al. (2020) and included bleaching overnight and washing in a 150 µm sieve. The results were compared to pollen and plant macrofossils data
 <sup>35</sup> from the same core.

### 2.2 Numerical analysis

The medians and standard deviations of charcoal morphometrics (L, L/W, and A) were aggregated for each species, fuel type, and burn temperature, and are displayed as box <sup>40</sup> plots. A two-tailed Mann–Whitney test was used to test whether the medians of the charcoal morphometrics of various fuel types were equal (File S2). This test does not assume a normal distribution, only similar distributions in both groups.

### 45 **3 Results**

### 3.1 The influence of temperature on charred-mass production

Only needles and shrub leaves were greenish or brownish in colour at 250 °C, but plant materials of all species were 50 black with a typical charcoal appearance after burning at 300 and 350 °C. A few fuel types (graminoid, *Sphagnum*, and some twigs) turned to ash at 400 °C, whereas all other types of plant tissue became ash at 450 °C (File S1). Most of the charred materials remained intact and retained all of their morphological characteristics. However, samples burnt <sup>55</sup> at higher temperatures tended to break easily during sample manipulation.

The average percentage of charred mass retained at 300 °C decreased as follows (Table 2; Fig. 1): brown moss and fern (50%) > shrub twigs (46%) > shrub leaf (44%) > forb leaf (42%) > needles (41%) > tree twigs (40%) > graminoid (29%) > *Sphagnum* (22%) > trunk wood (11%). This trend in mass loss was similar at all temperatures, although charcoal mass showed a marked decline from 38%–84% at 250 °C to 0.2%–23% at 400 °C across all fuel types. The charred mass of mixed-fuel samples at 300 °C was lowest for samples with high contents of graminoid and *Sphagnum* (33%–35%) and highest for samples with greater proportions of shrub material (38%).

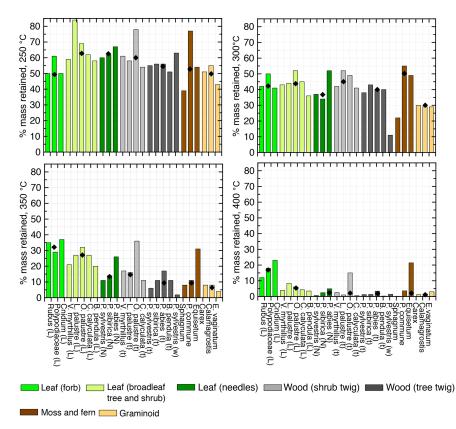
### **3.2** Fuel-dependent variations in length, aspect ratio, 70 and surface area

Graminoid charcoals burnt at 300 °C (L/W = 11.5; Fig. 2b, f; Table 2) were consistently more elongate than those of twigs (shrub, 5.2; tree, 3.8), moss and fern stems (4.6), and leaves (2.7) (Table 2). Charred needles were more elongate 75 (3.1) than those of leaves from heathland shrubs (2.4) and broadleaf trees (2.1) (Table 2). The Mann-Whitney test confirmed that (i) the median aspect ratio of graminoids was significantly different from those of all other fuel types (p < p0.001), (ii) those of all types of wood were different from 80 those of leaves (p < 0.001) and moss (except at 350 °C), and (iii) those of leaves were different from those of moss (p < 0.001; Table S2). The lengths (major axis, L) of charred particles from different fuel types, however, were less clearly differentiated (Fig. 3a, c; Files S2, S3). The surface area (A) <sup>85</sup> varied greatly between individual taxa and fuel types; nevertheless, fragments of shrub leaves tended to be larger than all other fuel types (Fig. 3b, d; Table 2; Files S2, S4). The morphometrics of mixed-fuel samples showed that charcoal from samples with abundant graminoids and moss was more elon- 90 gate (higher L/W) than charcoal from samples with higher proportions of shrubs, wood, and/or leaves (Fig. 2h). Similarly, the longest charcoal particles (higher L) were from samples with greater proportions of graminoids and moss (Fig. 3e), whereas the charcoals with the largest surface ar-95 eas were from samples with more abundant shrubs and leaves (Fig. 3f). The aspect ratios and lengths of individual taxa and fuel types changed slightly with temperature, but the general trends were similar across all temperatures (Figs. 2, 3a, c; File S3). In contrast, relative surface areas varied more with 100 temperature changes (Fig. 3b, d; File S4).

ace area ( $\mu$ m <sup>2</sup> ), and mass retained (%) of charcoal produced in the muffle oven for individual plant species and fuel types	t name, see Table 1. Please note that mean values are shown using bold font.
<b>Table 2.</b> Summary of the mean aspect ratio, length ( $\mu$ m), surface area ( $\mu$ m <sup>2</sup> ), and mass retained (	ole 1. Please note

Fuel types	Species	4	Aspect ratio at	T/ <mark>€№8</mark> °C			Length	Length at $T/^{\circ}C$			Surface area at $T/^{\circ}C$	a at T/°C		Mas	Mass retained at $T/^{\circ}C$	d at $T/^{c}$
Temperature		250	300	350	400	250	300	350	400	250	300	350	400	250	300	350
Graminoid	Eriophorum Calmagrostis Carex <b>Mean</b>	7.2 ± 5.0 7.4 ± 4.6 7.4 ± 5.1 <b>7.3 ± 0.1</b>	$11.1 \pm 6.4$ $12.9 \pm 11.8$ $10.6 \pm 7.0$ $11.5 \pm 1.2$	5.8 ± 3.7 6.0 ± 3.8 8.3 ± 6.0 <b>6.7</b> ± <b>1.4</b>	N/A N/A N/A	$521 \pm 545$ $620 \pm 622$ $487 \pm 499$ $543 \pm 64$	797 $\pm$ 496 951 $\pm$ 712 841 $\pm$ 646 <b>862</b> $\pm$ 79	$635 \pm 74$ $440 \pm 370$ $690 \pm 709$ <b>588 \pm 131</b>	N/A N/A N/A N/A	37 665 82 058 32 165 <b>50 629</b>	42 744 99 009 82 947 <b>74 900</b>	51 820 30 244 51 982 <b>44 682</b>	N/A N/A N/A N/A N/A	42.6 54.7 50.9 <b>49.4</b>	29 29.2 <b>29.5</b> <b>29.2</b>	3.6 8.1 7.9 <b>6.5</b>
Moss and ferns	Equisetum Polytrichum Sphagnum Mean	4.8 ± 3.3 4.8 ± 3.1 3.4 ± 1.8 <b>4.3</b> ± <b>0.8</b>	4.5 ± 2.8 4.2 ± 2.6 5.2 ± 6.2 <b>4.6</b> ± <b>0.5</b>	3.1 ±2.2 4.2 ±2.6 3.2 ±1.6 <b>3.5</b> ± <b>0.6</b>	4.8±3.1 4.7±2.9 N/A <b>4.7±0.1</b>	$506 \pm 501 \\ 673 \pm 665 \\ 612 \pm 574 \\ 598 \pm 84$	$286 \pm 233 \\ 461 \pm 408 \\ 524 \pm 636 \\ 423 \pm 123 \\$	655 ± 510 459 ± 476 319 ± 217 <b>477</b> ± <b>168</b>	$530 \pm 424 \\ 572 \pm 535 \\ N/A \\ 551 \pm 29$	71 381 123 311 109 185 <b>97 346</b>	25 955 84 632 77 224 <b>62 739</b>	166741 68170 24724 86545	75 098 99 522 N/A <b>87 310</b>	54.4 77.0 38.3 <b>56.6</b>	49.4 55.5 21.7 <b>42.2</b>	31.0 111.4 8.3 <b>16.6</b>
Wood (trunk)	Pinus sylvestris	$2.0 \pm 0.9$	$4.9 \pm 2.8$	$2.5\pm0.9$	N/A	$408 \pm 347$	$391 \pm 300$	$482 \pm 384$	N/A	105 057	57 028	97 673	N/A	63.5	11.1	1.8
Wood (tree twig)	Betula pendula Picea abies Pinus sibirica Pinus sylvestris <b>Mean</b>	2.8 ± 1.4 2.3 ± 1.2 2.1 ± 1.3 2.5 ± 0.4	4.5 ± 3.1 3.1 ± 1.9 3.1 ± 1.9 3.5 ± 2.0 <b>3.8</b> ± <b>0.8</b>	2.0±0.8 2.5±1.3 2.6±1.3 2.9±1.7 <b>2.5±0.3</b>	N/A N/A N/A 2.9±1.5	459±402 435±371 450±379 593±558 <b>469±72</b>	$361 \pm 236$ $439 \pm 303$ $318 \pm 248$ $379 \pm 318$ $377 \pm 44$	$347 \pm 85$ $598 \pm 575$ $427 \pm 343$ $407 \pm 295$ $452 \pm 95$	N/A N/A N/A N/A 407 ± 350 N/A	120 539 92 767 119 815 140 004 <b>115 639</b>	37 630 92 107 15 058 74 797 <b>55 051</b>	54 180 199 556 83 461 74 929 <b>102 049</b>	N/A N/A N/A 88 944 N/A	50.8 56.5 55.2 <b>54.5</b>	40.2 40.1 43.3 38.1 <b>40.2</b>	10.9 16.8 11.0 5.7 <b>11.2</b>
Wood (shrub twig)	Chamaedaphne Oxycoccus Ledum Vaccinium <b>Mean</b>	3.5 ± 1.9 3.8 ± 3.0 4.4 ± 2.4 5.0 ± 3.1 <b>4.2</b> ± <b>0.7</b>	6.3 ± 3.5 6.2 ± 3.5 5.4 ± 2.8 3.0 ± 2.6 <b>5.2</b> ± <b>1.5</b>	3.7 ±2.1 4.7 ±3.3 2.9 ±1.3 3.7 ±2.5 <b>3.8</b> ±0.7	N/A 4.1 $\pm$ 2.3 4.0 $\pm$ 2.7 N/A <b>4.0 <math>\pm</math> 0.7</b>	387 ± 340 674 ± 669 333 ± 197 461 ± 446 <b>463 ± 150</b>	$347 \pm 288$ $590 \pm 419$ $458 \pm 288$ $818 \pm 714$ <b>553 \pm 202</b>	$525 \pm 528$ $591 \pm 580$ $342 \pm 266$ $441 \pm 306$ $474 \pm 107$	$\begin{array}{c} 521\pm555\\ 1053\pm700\\ 617\pm580\\ N/A\\ 730\pm283 \end{array}$	61 732 157 873 57 191 72 214 <b>87 252</b>	73 382 51 906 56 836 291 023 <b>118 061</b>	131 063 94 418 254 696 75 212 <b>136 597</b>	N/A 249 197 131 860 N/A N/A	56.8 78.8 56.6 60.8 <b>62.7</b>	41.3 49.4 52.5 42.2 <b>46</b>	11.4 35.9 15.6 16.9 <b>20</b>
Needles	Picea abies Pinus sibirica Pinus sylvestris <b>Mean</b>	2.6 ± 1.6 4.0 ± 2.3 4.0 ± 2.5 <b>3.5</b> ± <b>0.8</b>	2.2 ± 1.1 3.6 ± 1.8 3.7 ± 4.0 <b>3.1 ± 0.8</b>	2.3 ± 0.9 4.7 ± 2.9 3.5 ± 2.9 <b>3.5</b> ± 0.1	N/A N/A N/A N/A	549±535 606±587 690±660 <b>613</b> ± <b>71</b>	342 ± 276 385 ± 292 445 ± 376 <b>390 ± 52</b>	608 ± 692 432 ± 414 492 ± 368 <b>510</b> ± <b>90</b>	N/A N/A N/A N/A	118 375 111 068 134 504 <b>121 303</b>	86728 46629 86639 <b>73332</b>	242 364 52 880 83 649 <b>126 297</b>	N/A N/A N/A N/A N/A	67.2 62.3 59.7 <b>58.3</b>	52.3 34.0 37.3	26.2 14.3 10.6
Broadleaf (tree)	Betula pendula	$2.1 \pm 1.3$	$2.1\pm0.9$	$2.0\pm0.8$	N/A	493 ± 498	$335 \pm 234$	$354 \pm 185$	N/A	156 591	67 692	57 934	N/A	58.3	<b>3</b> 5.6	20.3
Broadleaf (shrub)	Chamaedaphne Oxycoccus Ledum Vaccinium <b>Mean</b>	2.4 ± 1.3 2.1 ± 0.9 2.3 ± 1.0 2.6 ± 2.0 <b>2.3 ± 0.1</b>	2.3 ± 1.7 2.3 ± 1.4 2.7 ± 1.4 2.1 ± 0.9 <b>2.4</b> ± <b>0.2</b>	1.8±0.7 2.2±1.0 2.7±1.6 3.8±2.8 <b>2.6±0.8</b>	$\begin{array}{c} N/A \\ 1.9 \pm 0.8 \\ N/A \\ 2.1 \pm 1.4 \\ \textbf{2.0 \pm 0.4} \end{array}$	633 ± 493 730 ± 486 728 ± 698 441 ± 334 <b>563 ± 98</b>	$347 \pm 289$ $590 \pm 419$ $393 \pm 322$ $414 \pm 309$ $385 \pm 71$	<i>5</i> 71 ± 395 410 ± 343 568 ± 586 442 ± 304 <b>426 ± 101</b>	$\begin{array}{c} N/A \\ 260 \pm 108 \\ N/A \\ 401 \pm 250 \\ \textbf{330} \pm 100 \end{array}$	176 620 222 081 281 751 88 832 174 636	73 382 184 196 67 913 105 547 <b>87 725</b>	216473 94939 219788 88333 <b>106408</b>	N/A 31 142 N/A 75 360 <b>53 251</b>	61.8 69.1 84.5 59.1 <b>68.5</b>	44.7 52.8 47.4 43.4 <b>46.7</b>	27.2 32.0 31.9 21.4 <b>28</b>
Leaf (forb)	Rubus Cridium Polypodium <b>Mean</b>	$1.9 \pm 0.7$ 2.3 $\pm 1.2$ 2.1 $\pm 1.0$ <b>2.1 <math>\pm 0.2</math></b>	2.2 ± 1.1 4.9 ±2.6 2.9 ±2.0 <b>3.3 ± 1.4</b>	2.1±1.0 2.6±2.2 2.5±1.3 <b>2.4±0.3</b>	N/A N/A N/A N/A	$\begin{array}{c} 398 \pm 355 \\ 466 \pm 384 \\ 521 \pm 399 \\ 466 \pm 61 \end{array}$	$354 \pm 237$ $372 \pm 263$ $464 \pm 292$ $418 \pm 65$	$368 \pm 315$ $549 \pm 598$ $383 \pm 284$ $466 \pm 117$	N/A N/A N/A N/A	99 109 108 610 155 561 <b>132 085</b>	79877 34207 106241 <b>70224</b>	88 927 176 828 60 473 <b>118 650</b>	N/A N/A N/A N/A	49.8 50.3 61.4 <b>56.6</b>	50.6 50.6 <b>44.3</b>	35.2 37.3 29.4 <b>33.6</b>

6



**Figure 1.** The percent of charred mass retained after burning known plant species from Siberia in a muffle oven at 250, 300, 350, and 400  $^{\circ}$ C. The abbreviations used in the figure are as follows: L – leaf, N – needles, t – twig, and w – wood. The median mass retained for similar fuel types (identified by the same colour) is reported as black diamonds.

### **3.3** Finer diagnostic features of the charcoal morphologies of various fuel types

### 3.3.1 Graminoid charcoal

Graminoid (*Carex, Calamagrostis*, and *Eriophorum vagina*-<sup>5</sup> *tum*) charred particles were consistently flat, rectangular, and elongated (Files S5a, S6a). They mostly broke parallel to the long axis when pressured, resulting in highly elongated pieces with straight margins. They can also appear as featureless long, thin filaments. Charcoal produced at higher <sup>10</sup> temperatures (350 °C) often had more irregular, zigzag, or denticulate margins. The most commonly preserved surface features were rectangular epidermal cells or contained oval voids, reticulated or mesh patterns, and/or isolated veins.

### 3.3.2 Wood charcoal (trunk, tree twigs, and shrub twigs)

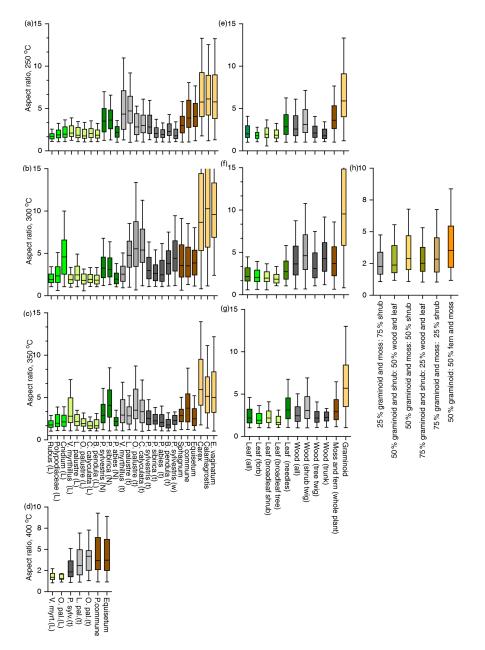
Wood charcoal pieces from tree trunks (*Pinus sylvestris*) were blocky and quadrilateral with 90° corner angles (Files S5b, S6b). Wood charcoal from tree (*P. sylvestris*, *P. sibirica, Picea abies*, and *Betula pendula*) and shrub <sup>20</sup> (Ericaceae) twigs showed both quadrilateral and polygonal shapes. The edges of both trunk and twig charcoal were

smooth, serrated, or denticulate, and surface textures were smooth, foliated, or striated (File S5b). Trunk charcoal of *P. sylvestris* showed rows of brown, open pits in the tracheid walls. Under the microscope, trunk charcoal fragments were shinier and darker than twig charcoals. Large charcoal pieces often broke parallel to the long axis, producing many tiny, elongated pieces (trunks) or pieces of various forms (twigs).

### 3.3.3 Leaf charcoal (needles, deciduous tree and shrub, forb, and fern)

Charred needle fragments were elongated and rectangular (corner angles of 90°; Files S5c, S6a). Their edges were smooth but became serrated and denticulate when broken. Surface features included visible venation and ridges. Charcoals from the leaves of deciduous trees (*Betula*), heathland <sup>35</sup> shrubs (*Oxyccocum*, CETO *Ledum*, *Camadaphne*, and *Vaccinium*), herbaceous plants (*Rubus*), and ferns (Polypodiaceae) were polygonal. Only those of *Cnidium* leaves were elongated, reflecting their needle shape. Edges were mostly undulate but were sometimes smooth or denticulate. Surface textures were generally smooth (featureless) but sometimes included visible venation and ridges. When broken, they showed voids, reticulated mesh patterns, and curly fi-

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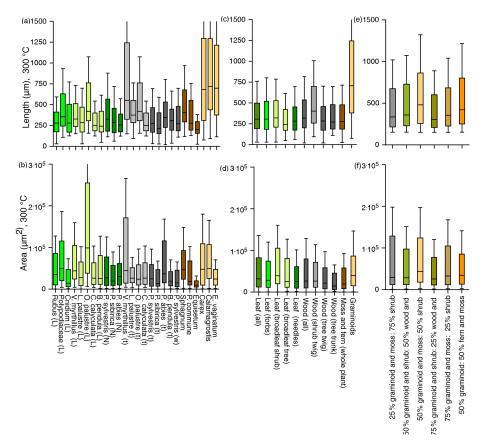


**Figure 2.** The median aspect ratios of charred particles from  $(\mathbf{a}-\mathbf{d})$  the individual measurements burned at 250, 300, 350, and 400 °C respectively, and  $(\mathbf{e}-\mathbf{g})$  fuel types at burning temperatures of 250, 300, and 350 °C respectively, as well as from  $(\mathbf{h})$  mixed-fuel samples burned at 300 °C. The fuel mixtures are arranged in order of increasing proportions of graminoids. Box plots represent the distribution of data as follows: the horizontal line in each box denotes the median, the upper quartile is the median value of the upper half of the data points, the lower quartile is the median value of the lower half of the data points, and whiskers represent the respective minimum and the maximum values. The abbreviations used in the figure are given in the caption of Fig. 1. The individual taxa  $(\mathbf{a}-\mathbf{c})$  belonging to a fuel-type group  $(\mathbf{d}-\mathbf{g})$  are indicated by the same colour.

bres. Birch leaves produced visible charred veins with three branches diverging from a node. When pressured and broken, small leaf pieces had fracture lines radiating out at a variety of angles.

### 3.3.4 Moss and fern stems

*Sphagnum* produced two types of charcoal morphologies. Stems produced elongate particles with ramifications (scars) where leaves branched from the stem. Leaves preserved the anatomical features of the unburned leaves, i.e. a mesh-like appearance (Files S5a, S6a). *Polytrichum* produced several 10



**Figure 3.** The median lengths ( $\mu$ m) and surface areas ( $\mu$ m<sup>2</sup>) of charred particles from (**a**, **b**) individual taxa, (**c**, **d**) fuel types, and (**e**, **f**) fuel mixtures at 300 °C. The abbreviations used in the figure are given in the caption of Fig. 1. See the caption of Fig. 2 for a description of the box plots and colour coding.

charcoal morphologies (quadrilateral, polygonal, or curved with angular edges) with generally featureless surfaces, although some showed mesh patterns. This charcoal type often splits along the main axis. *Equisetum* leaves and stems 5 were generally quadrilateral with straight, undulate, or denticulate margins, and oval voids and reticulated mesh patterns on their surfaces.

### **3.4** Morphometrics and finer diagnostic features of fossil charcoal

<sup>10</sup> The average aspect ratio of the seven sediment samples varied between 2.8 and 11.1, whereas the average length varied between 403 and 742 (Table 3). Samples with higher aspect ratios contained abundant morphologies of graminoids, *Equisetum*, and moss (Table 3). The average surface area varied <sup>15</sup> from 16.565 to 248.287, where samples with greater surface area have higher number of leaves.

### 4 Discussion

note the remarks at the end of the manuscript.

fine diagnostic features, and charcoal production) for 17 <sup>20</sup> plant species belonging to seven fuel types from boreal Siberia CEII. This dataset expands the geographical coverage of fuel types researched, demonstrates the applicability of charcoal morphology assemblages to fossil records, and improves the interpretation of fire types based on charcoal <sup>25</sup> morphologies.

## 4.1 The influence of combustion temperature on charcoal production: implications for charcoal-based fire reconstructions

Knowledge of the charred mass is critical for determining <sup>30</sup> biases in charcoal production to biomass quantity and fire temperature (Walsh and Li, 1964 (1510)). Results from these burning experiments clearly show that the effect of temperature on charcoal production is fuel dependent. Graminoid, *Sphagnum*, and trunk wood produced the lowest amounts of charcoal per unit biomass and lost their mass more rapidly with increasing burning temperature, i.e. from 40 %–63 % at 250 °C to 0.2 %–3 % at 400 °C (Fig. 1; Table 2). Leaves of heathland shrubs, forbs, and ferns (Polypodiaceae), as well as fern stems with leaves (*Equisetum*), produced the most <sup>40</sup>

**Table 3.** The mean aspect ratio, length, surface area, and the number of each charcoal morphotypes in the Holocene samples ranging from 185 to 6750 cal yr BP (35–303 cm) from Ulukh-Chayakh mire. The local to regional vegetation is represented by the percentages of the main pollen types, whereas the local vegetation is represented by the plant macrofossils (values in parentheses are presented as numbers except for the wood remains, which are presented as percentages).

Depth (cm)	25						
• • •	35	84	85	172	248	268	303
Aspect ratio	3.2	3.0	3.0	4.0	11.1	4.3	2.8
Length (µm)	742	555	723	488	403	515	431
Surface $(\mu m^2)$	248.287	122.599	219.413	49.673	16.565	53.354	53.698
Charcoal morpho	ologies (pla	nt macrofos	sil)				
Wood	23 (6 %)	13 (5%)	6 (5%)	11 (15%)	1 (0%)	8 (0%)	6 (0%)
Leaf	11 (0)	7 (0)	3 (0)	2 (0)	0 (0)	4 (0)	4 (0)
Needle	0(7)	1(0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
Graminoid	21 (30)	9 (70)	1 (70)	11 (45)	4 (65)	19 (70)	3 (0)
Equisetum	15 (10)	3 (5)	1 (0)	0(15)	0 (10)	5 (5)	0 (90)
Moss	10 (30)	0 (5)	0 (5)	1 (5)	1 (5)	3 (5)	1 (0)
Pollen (%)							
Trees	90	92	92	94	97	92	97
Shrub	1.1	3.4	3.4	3.5	0.2	0.5	0
Graminoid	5	6.3	6.3	3	13	8.6	9.5
Equisetum	0.5	3.4	3.4	1.2	6.7	9	18
Moss	0	0.2	0.2	0	0	0.3	0

charcoal per unit biomass and retained the greatest mass at higher temperatures from 50 %–84 % at 250 °C to 4 %–24 % at 400 °C (Fig. 1). The charred mass of mixed-fuel samples burnt at 300 °C also changed according to the domi-5 nant fuel type. Pereboom et al. (2020) burned plant tissue

- from similar taxa occurring in the Alaskan tundra in a muffle oven (at 500 °C) and likewise found that graminoids have a lower charred-mass retention (25 %–27 %) than shrubs (up to 33 %). However, they did not test the leaves and wood <sup>10</sup> of shrubs separately and also did not investigate the effect
- of different temperatures on charred mass. Burning experiments on American forest steppe plants in a muffle oven (at 350 °C) and under open-flame conditions showed that the mass retention of grass and deciduous leaf charcoal de-<sup>15</sup> creased more rapidly with temperature compared with wood charcoal (Umbanhowar and McGrath, 1998), which is in partial agreement with the findings of this study. A calorimetric combustion study of various fuel types from mostly tropical

plants found that the charred mass of wood, needles, and *Eq-*<sup>20</sup> *uisetum* was greater that of other leaf types, due to the higher bulk densities and fuel load (Hudspith et al., 2017

Although oven experiments do not fully replicate the burn conditions of natural wildfires, these experimental findings have practical implications for charcoal-based fire recon-<sup>25</sup> structions. First, fuel types with low charred-mass retention at higher temperatures are likely to be under-represented in the sedimentary charcoal record. Cyperaceae (sedges) are the most common graminoids in fens and meso- and eutrophic bogs worldwide, and *Eriophorum* (sedge) and *Sphag*- num (moss) are common in oligotrophic bogs. These fuel 30 types will turn to ash even in relatively low-intensity fires (< 300 °C) and, thus, may leave little or no trace of charcoal in sediments. Second, the woody biomass of typical heathland shrubs from oligotrophic bogs such as Vaccinium, Camadaphne, Oxycoccus, and Ledum as well as 35 brown moss (Polvtrichum commune), the latter of which is common in all boreal habitats, preserve almost half of their biomass when burnt at temperatures up to 300 °C, but their mass declines strongly (11%-15%) at higher temperatures  $(> 350 \,^{\circ}\text{C})$  CE12. This suggests that charcoal from these fuel 40 types is likely to be preserved only when fires are of low to intermediate intensity. Third, the leaves of shrubs, forbs, and ferns as well as stems with leaves of Equisetum are more likely to persist as charcoal (27 %-38 %) after hightemperature fires and, thus, may contribute disproportion- 45 ately to sedimentary charcoal. Mineral constituents can slow pyrolysis (thermal decomposition of plant material producing volatile products and a solid charred residue), and this may explain why Equisetum stems, with high silica content, produce more charcoal. Fuel with higher lignin content, 50 such as wood, might also be expected to produce more charcoal than fuels higher in cellulose and hemicellulose, such as leaves (Yang et al., 2007). However, this did not appear to be the case in the current experiments where leaves retained a higher charred mass than wood with increasing burn 55 temperature. This suggests that there is a need for further research on the quantitative relationship between temperature

and charcoal mass retention for fuels with various structures, chemistry, and bulk density.

### 4.2 Fuel-dependent variability in charcoal morphometrics: implications for the reconstruction of fuel type and transportation by air and water

The consistency of the morphometrics of charred fragments between species in the same genus or family suggests that these measurements are useful for fuel-type identification. Graminoid charcoal particles are at least twice as elongated 10 (6.7–11.5) as all other charcoal types and differ the most from leaf charcoal across all temperatures (Fig. 2; Table 2; File S7). Highly elongated and narrow graminoid charcoal is thought to reflect the occurrence of conspicuous veins parallel to the long axis (Umbanhowar and McGrath, 1998; Craw-

- 15 ford and Belcher, 2014). Charred fragments of leaves (2.0-2.7 b leaves; 3.1–3.5 needles) are also markedly more circutation those of other fuel types. However, there is some degree of overlap between the aspect ratios of woody twigs (2.5–5.2) and those of moss and fern stems (3.5–4.75;
- <sup>20</sup> Table 2). In agreement with Crawford and Belcher (2014) and Umbanhowar and McGrath (1998), these experiments showed that smaller particles have a lower aspect ratio (i.e, more circular particles). Although larger charcoal fragments may be more suitable to categorise fuel type, there is no ob-
- 25 vious threshold for determining what particle size should be used. Charcoal fragments from mixed-fuel samples preserve the aspect ratio of the dominant fuel type; particles with highest aspect ratios (3.5) were found in samples with greater proportions of graminoids and moss. Length and mean surface
- <sup>30</sup> area do not appear to distinguish between fuel types reliably, except for the slight tendency for charred shrub leaf particles to be larger than those of all other fuel types (Fig. 3; File S4; Table 2). The larger shrub leaf fragments may be explained by the arrangement of leaf venation, with frag-35 ments breaking along the three branching veins that diverge from nodes (Umbanhowar and McGrath, 1998; Jensen et
- al., 2007). The measured aspect ratio of graminoids as well as shrub and forb leaves from this study are quite similar to those from the Alaskan arctic, where graminoids (Erio-40 phorum vaginatum and Carex bigelowii) show aspect ratios
- ranging from 5.46 to 8.09 (mean 6.77), and shrubs (Ledum palustre, Salix pulchra, Betula nana, Rubus chamaemorus, and Vaccinium vitis-idaea) show aspect ratios ranging from 2.09 to 2.50 (mean 2.42, Table 4; Pereboom et al., 2020). 45 Considerably shorter graminoid particles (3.62) were obtain from American steppe forests (Umbanhowar and Mc-Grath, 1998), although the aspect ratios obtained for leaves (1.91) and wood (2.13) were closer to those obtained in this study. Crawford and Belcher (2014) produced charcoal

separate graminoids (aspect ratio < 2.0) from shrubs (> 2.0; Aleman et al., 2013). However, Daniau et al. (2013) in- 55 terpreted an increase in the aspect ratio as an indication of the increased proportion of burning of the grass fuel. Courtney-Mustaphi and Pisaric (2014a) also observed that burning monocotyledons from boreal Canada in the laboratory generally produced more elongated charcoal morpholo- 60 gies than other fuels. In term of surface area, Umbanhowar and McGrath (1998) show a surface area of  $65.630 \,\mu\text{m}^2$ (56.737 herein) for graminoids,  $50.150 \,\mu\text{m}^2$  (103.138 herein) for wood, and  $64.946 \,\mu\text{m}^2$  (versus 114.952 herein) for leaves, which are values comparable to Pereboom et al. (2020), 65 who found little differentiation between average surface area for shrub  $(88.246 \,\mu\text{m}^2)$  and graminoid  $(87.474 \,\mu\text{m}^2)$  species. The present study suggests that the morphometric values are generally preserved for all fuel types over the range of temperatures explored, whereas Umbanhowar and Mc-70 Grath (1998) found that burn temperature marginally reduced the aspect ratios of wood.

The combined results from this study and the published literature suggest that, despite some variability in morphometrics of charcoal assemblages from similar fuel types, the 75 aspect ratio decreases from graminoids to wood and leaves. Differences in the aspect ratio might allow the distinction of graminoids from other fuel types in a consistent way. Although there is a wide range of individual measurements, the mean aspect ratios of the three graminoid species (6.7-11.5) 80 suggests that a threshold aspect ratio of 6 could be used to discriminate graminoids. This threshold value appears to be most consistent for wetland graminoids (mean 6.77 for arctic Alaska) but may be too high for graminoids from temperate grasslands (3.8–4.66; Table 4; File S7). Although there 85 is also a good consistency in the aspect ratio of laboratoryproduced wood (2.1-4.5) and leaf (2.0-3.5) charcoal particles across studies, these values overlap, suggesting that it is not possible to specify a threshold value at which charcoal particles are indicative of wood or leaves. Therefore, the use 90 of charcoal morphologies in fuel-type identification requires the use of fine anatomical features (see Sect. 4.3) or validation from other sources, such as anthracological analysis as employed in archaeobotanical studies (Hubau et al., 2012, 2013, 2015; de Melo Júnior, 2017). 95

Particle shape affects the behaviour of charcoal during transportation by air (Clark and Hussey, 1996; Clark, 1998 [1513]) and water (Nichols et al., 2000). Models, assuming a uniform spherical particle shape, and empirical data of transportation by fume indicate that the amount of 100 charcoal particles is greatest near the fire source (Clark et al., 1998 TS14; Clark and Royall, 1995 TS15; Tinner et al., 2006 [1516; Higuera et al., 2007; Peters and Higuera, 2007). However, recent models accounting for different shapes, sizes, and densities of charcoal show that non-spherical par- 105 ticles have lower settling velocities than spherical particles and produce a spatially more extensive and heterogeneous particle-size distribution pattern – i.e, dispersal distances for

Table 4. Comparative results of the aspect ratio from plant species analysed in this study with those from literature. "*Pinus sylvestris* (wood)" sums the mean aspect ratio of wood from trunk and twig; "Wood (total)" sums the mean aspect ratio of wood from trees and shrubs (trunk and twig); "Broadleaf" sums the mean aspect ratio of leaf from trees and shrubs, whereas "Leaf (total)" averages the mean aspect ratio of all leaf types. [IS12]

Fuel type	250 °C	300 °C	350 °C	400°C	500/ <u>CE13</u> 550 °C	Open flame	References
Graminoid (boreal)	7.3	11.5	6.7	_	_	_	This study
Graminoid (arctic)	-	-	_	-	6.7	-	Pereboom et al. (2020)
Graminoid (forest steppe)	-	-	3.6	-	_	4.8	Umbanhowar and McGrath (1998)
Graminoid (grass)	-	-	_	-	3.7	-	Crawford and Belcher (2014)
Pinus sylvestris (wood)	2.7	4.1	2.7	2.9	-	-	This study
Pinus sylvestris (wood)		-	_	-	2.8	-	Crawford and Belcher (2014)
Wood (total)	3.4	4.5	3.1	4.0	_	-	This study
Wood (forest steppe)	-	-	2.1	-		2.3	Umbanhowar and McGrath (1998)
Shrubs (wood and leaf)	-	-	-	-	2.4		Pereboom et al. (2020)
Broadleaf	2.2	2.2	2.3	2.0	_	-	This study
Needles	3.5	3.1	3.5	_	-	_	This study
Leaf (total)	2.5	2.7	2.6	2.0	-	_	This study
Leaf (forest steppe)	-	-	1.9	-	_	2.1	Umbanhowar and McGrath (1998)
Leaf	_	_	_	_	2.2		Crawford and Belcher (2014)

spherical and aspherical particles greater than 150 µm could be up to 20 km apart (Vachula and Richter, 2018). Similarly, Clark and Hussey (1996) derived a velocity index for sedimentary charcoal particles and found that non-spherical par-

<sup>5</sup> ticles have lower setting velocities and a higher residence time into the atmosphere than elongated particles. Based on these studies, it appears that non-spherical charcoal particles (elongated) such as those of graminoids, moss, and ferns are likely to have a more heterogeneous distribution and be de-

<sup>10</sup> posited farther away from the origin of a fire than the rounder, polygonal leaf particles. Erosion during hydrological transportation can also change the shape of buried (sedimentary) charcoal and can be an important consideration when interpreting charcoal morphometrics (Patterson et al., 1988[1517];
 <sup>15</sup> Nichols et al., 2000; Scott et al., 2000[1515]).

Laboratory experiments simulating fluvial transportation found that the surface area of leaf charcoal decreases and circularity increases with transportation, whereas changes in the shape of woody particles is less evident with trans-20 portation, and grassy charcoal preserves a high aspect ratio during transportation (Crawford and Belcher, 2014). However, Nichols et al. (2000) found a slight rounding of sharpangled edges of wood and a greater propensity for breakage of charcoal produced at higher temperatures, the lat-25 ter of which was also found in this study. These findings give further support to the fact that the typical appearance of graminoids as elongated particles and of leaves as circular is preserved during transportation. Nevertheless, other studies using sedimentary charcoal records suggest that erosion during transport accounts for the rounding (degree angles are eroded) of robust charcoal types such as wood, whereas fragile pieces of leaves and grass may break (Vannière et al., 2003; Courtney-Mustaphi and Pisaric, 2014b; Courtney Mustaphi et al., 2015). The differential transportation by air and the fragility of sedimentary charcoal morphotypes calls for investigations into the influence of particle shape on charcoal transportation and into strategies targeting coring locations for generating robust quantitative data for palaeo-fire interpretations.

### **4.3** Finer diagnostic features of the charcoal morphologies for fuel-type identification

Results from fine diagnostic features of charcoal particles show that these can be used to attribute charcoal particles to certain fuel types with some confidence. Apart from the extremely elongated shape that differentiates graminoid 45 charred particles from all other fuel types, graminoids are further distinguished under both microscope and stereomicroscope by their flat appearance and breakage into thin filaments (Files S5a, S6a). Rectangular epidermal cells, reticulate meshes, and oval voids of former epidermal stomata 50 are also good diagnostic features of graminoids (Grosse-Brauckman, 1974). The graminoid charcoals produced in this study are most similar to types C4, C6, D1, D2, and D3 described by Courtney-Mustaphi and Pisaric (2014a) and Enache and Cumming (2006). Comparative studies on 55 graminoid charcoal originating from Poaceae (grass) and Cyperaceae (sedge) would further improve the identification of fuel types given the ecological differences of the two groups, with sedges growing in wetlands<sup>CE14</sup> and grass in drier habitats. 60

A distinct feature of woody charcoal is that they are layered with foliated or striated textures and break into many tiny particles when pressured (Files S5a, S6b). This is due to the abundance of fibres and xylem, which leads to charcoals splitting at various angles (Vaughan and Nichols, 1995). Additionally, conifer wood charcoal presents distinct rows of

open pits in the tracheid walls (Schweingruber, 1978). Attempts to distinguish between charred trunk and twig particles were less successful, although charred trunk particles are blockier. Foliated charred wood fragments also share ap-

<sup>5</sup> pearance with moss and fern stems. These woody charcoals are most similar to types A1, B1, B2, and B3 (Courtney-Mustaphi and Pisaric, 2014a; Enache and Cumming, 2006).

Typical features of charred deciduous leaves are their polygonal shapes with surfaces characterised by void spaces

- <sup>10</sup> or undulated surfaces (Files S5c, S6a). Netted venation is also sometimes visible, mostly with three branches diverging from a node. In contrast, conifer needles are elongated, often show ramification, and can have a wood-like appearance. The deciduous leaf charcoals found here are most sim-
- <sup>15</sup> ilar to morphologies A2, A3, A4, A5, and A46, and conifer needle charcoals are most similar to morphologies C1, C2, and C3 (Courtney-Mustaphi and Pisaric, 2014a; Enache and Cumming, 2006).

Charred *Sphagnum* leaves preserve the meshed pattern of <sup>20</sup> fresh plant material (Grosse-Brauckman 1972). Often, stems contain ramification, likely scars of former leaves (Files S5a, S6a). Both *Sphagnum* and *Polytrichum* charcoals present curvy fragments not seen in other fuel types. However, stems of *Sphagnum* and *Polytrichum* can be easily mistaken for <sup>25</sup> shrub twigs. Burnt *Equisetum* can resemble graminoid char-

coal. Charred moss is similar to morphologies C4 and C7 (Courtney-Mustaphi and Pisaric, 2014a; Enache and Cumming, 2006).

## 4.4 The morphometrics and morphologies of fossil <sup>30</sup> charcoal particles: implications for fuel-type identification

Charcoal fragments from Holocene samples ranging from 6700 to 180 cal yr BP at the Ulukh-Chayakh mire preserved the aspect ratio of the dominant morphologies, i.e. particles 35 with the highest aspect ratios (4-11) were found in samples with a greater proportion of graminoids, Equisetum, and moss (Table 3). Likewise, a greater surface area was found in samples with a higher number of leaves. Comparative results from fossil charcoal morphologies and morphometrics 40 to those from pollen and plant macrofossils from the same depths show a partial agreement. For example, the pollen record indicates that the percentages of tree were > 90 %and the percentages of shrub were up to 3% = g the entire period, whereas the abundance of woody charcoal mor-45 phologies increased infrequently. This suggests that although there was a continuous source of woody fuel to burn, the production of wood charcoal from high-intensity fire only occurred occasionally. There is, however, a better agreement between samples with greater aspect ratios and morpholo-50 gies of understorey vegetation (i.e. graminoids, Equisetum, and moss) and the proportion of these plants in the pollen and plant macrofossil records (Table 3). These findings are in line with the Siberian wildfire behaviour of predominantly lowintensity, surface fires, fuelled by graminoids, forbs, ferns, and mosses, or intermediate-intensity surface fires (shrubs) <sup>55</sup> and only infrequently as high-intensity crown fires (Anderson, 1982[1519]). Another practical application of this finding is that the morphometrical and morphological characterisation of fossil charcoal is more representative of fuel type (what plant types were burning), compared with the pollen <sup>60</sup> data and plant macrofossils that reflect plant types growing regionally and locally.

### 4.5 Applications for fuel- and fire-type reconstructions

The physical and chemical characteristics of fuel are key factors influencing ignition and fire propagation. The ma- 65 jor chemical components of fuels are cellulose, hemicellulose, and lignin, and the minor components include terpenes, resins, and minerals (Planas and Pastor, 2014). Fuels rich in cellulose and hemicellulose (i.e. leaves) pyrolyse at a lower and narrow temperature range (200 and 400 °C), whereas 70 those rich in lignin (i.e. wood) pyrolyse at a higher and wider range of temperatures (160-900 °C; Yang et al., 2007). Fuel types rich in terpene and resins (conifer wood, needles, and Ericaceae) burn faster and at higher temperatures, whereas those rich in mineral components (graminoids) burn 75 less efficiently and at lower temperatures (200 °C) (Plana and Pastor, 2014). Results from the current burning experiments and fossil charcoal samples suggest that the combined use of morphometric and morphological features and charred mass can help distinguish some of the predominant 80 fuel sources. Knowledge of the fuel source may, in turn, provide clues on fire type, i.e. the combination of fire intensity (temperature) and severity (effect on vegetation). Fossils samples dominated by graminoid morphotypes show a high aspect ratio (4-11), which is in line with the elon- 85 gated shape of graminoid charcoal found in burning experiments (> 6.7-11.5; Tables 2, 3). As graminoid charcoal typically preserves at lower temperatures, it likely suggests a graminoid fuel source and, therefore, a lower-intensity fire (Fig. 4). Fossil samples with abundant leaves and wood mor-90 phologies showed considerably lower aspect ratios (3-3.2), in agreement with values from laboratory-derived morphologies of leaves (2.1-3.5) and wood (2.0-5.2). Thus, shorter, and bulkier charcoal particles likely indicate the increased prevalence of leaves and wood as a fuel source (Figs. 2, 4). 95 Because the morphometrical and morphological characteristics of leaves, and wood from trees and shrubs overlap, it is hard to distinguish between high-intensity surface fires, combusting living shrubs and dead wood and leaves, and highintensity crown fires that have burnt living trees. Neverthe- 100 less, the fact that the past fires may have been of higher intensity at times of leaf and wood charcoal dominance than during the graminoid charcoal dominance is additionally suggested by the increased abundance of morphologies of Equisetum and Polytrichum, which are taxa found to remain as 105 charcoal after burning at high temperature.

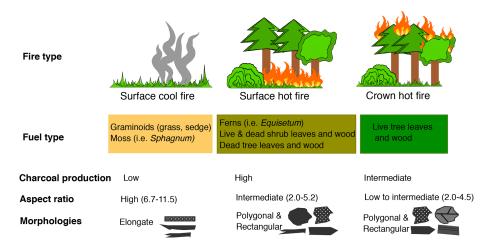


Figure 4. Schematic representation of fire types, and the potential link with fuel types burnt and predominant charcoal morphometrics (aspect ratio) and morphologies as well as charcoal production.

In summary, the consistency of results from this study with those from the literature on various vegetation types (boreal, temperate, and tropical woodlands; and grasslands) suggests the potential of charcoal morphometrics and mor-

- <sup>5</sup> phologies in palaeoecology. For example, the expansion of open habitats during deep geological times or with human impact, the recession of latitudinal and elevational treelines, or the predominant occurrence of surface fires is likely to be reflected in an increase in aspect ratio and graminoid mor-
- <sup>10</sup> phologies relative to total biomass burning. Conversely, the closing up of the forests, shrub encroachment, or the predominance of crown fires may show itself in a decreased aspect ratio of particles and increased bulky morphologies derived from leaves and wood. Results could also provide forest man-
- <sup>15</sup> agers with the range of fire types that key boreal species experienced in the past, which is useful when aiming to make choices for prescribed burning to remove fuel and prevent large fires or select species that will be fit to cope with future fire regimes. Answering all of these questions, however, will
- <sup>20</sup> require further investigations in order to relate the proportion of charcoal morphotypes to the quantity of biomass and extend the morphometric and morphological characterisation to key species of interest.

### 5 Conclusions and recommendations

- <sup>25</sup> This study presents the first results on the morphometry and other diagnostic features of charred particles produced in the laboratory from seven fuel types comprising 17 plant species from boreal Siberia and demonstrates the applicably of these experiments to interpreting fossil charcoal records. The use
- <sup>30</sup> of a higher number of fuel types from species with broad geographical coverage combined with an exploration of various combustion temperatures improves the link between charcoal morphologies, fuel types, and fire characteristics. Re-

sults show a distinct effect of temperature on fuel types, suggesting that species with low mass retention (graminoid, 35 Sphagnum, and trunk wood) during high fire temperature are likely to be under-represented in the fossil charcoal record. The aspect ratio was the strongest indicator of fuel type. Graminoid charcoal particles are more elongate (6.7–11.5) than all other fuel types, with a threshold above 6 that may 40 be indicative of wetland graminoids; leaves are the shortest and bulkiest (2.1-3.5); and twigs and wood are intermediate (2.0-5.2). Other diagnostic features can be used to separate wood, graminoids, and leaves, but not to make further distinctions within these fuel types. Distinct particle shape 45 may influence charcoal transportation, with elongated particles (graminoids, moss, and ferns) potentially having a more heterogeneous distribution and being deposited farther away from the origin of a fire than the rounder, polygonal particles (leaf). 50

Despite these limitations, the combined use of particle aspect ratio and charred morphotypes should allow more robust interpretations of changes in fuel source and fire type from charcoal records. Future efforts to determine fuel sources based on analyses of small charcoal fragments will require 55 (i) a more detailed examination of plant anatomy; (ii) investigation of the proportion of particular charcoal morphotype to the quantity of biomass; (iii) quantification of the relationship between the chemical composition of fuels, combustion temperature, and charcoal production; (iv) determination of 60 the influence of particle shape on differential transportation and fragility; and (v) the use of image-recognition software to collect data on charcoal characteristics such as roundedness, reflectance, and others features that could improve the estimation of fire temperature and erosion during transporta-65 tion.

### Appendix A: The watershed algorithm used to calculate morphometrics of charred particles

The algorithm for automatic detection of morphometrics is based on functions from the "skimage" Python module 5 (watershed algorithm). First, the picture is converted to a greyscale image. A Sobel gradient of the picture is then calculated, which results in an elevation map. To use the watershed algorithm to detect the charcoal particles, a map of markers in the grey picture with grey values higher than 140

- <sup>10</sup> is then created. These are the starting points of the watershed region fill algorithm. Finally, any holes in the watershed regions are filled with the help of a binary fill method (Soille and Vincent, 1990). The detected particles are subject to the calculation of morphometrics such as surface area and
- $_{15}$  lengths along the major and minor axis via supported functions. Particles with a major axis length less than  $150 \,\mu\text{m}$  are excluded from these calculations. The pixel area is calibrated at the micrometre scale, and the results are scaled accordingly.

#### A. Feurdean: Experimental production of charcoal morphologies to discriminate fuel source and fire type

*Data availability.* A limited amount of burnt plant material can be made available from the author upon reasonable request.

*Supplement.* The supplement related to this article is available online at: https://doi.org/10.5194/bg-18-1-2021-supplement.

5 *Competing interests.* The author declares that there is no conflict of interest.

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<u>note the remarks at the end of the manuscript</u>

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