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QUANTIFYING THE CONVERSION OF VEGETATION TO ASH FOR SOIL CARBON FINGERPRINTING

**Milestone 2.3.4 Final report on advances in soil carbon
fingerprinting**

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ABSTRACT

Samples of leaves, twigs and bark representing typical surface fuels from forests and woodlands were systematically heated and combusted under controlled conditions. Very little biomass, carbon (C) and nitrogen (N) was lost when heated at low temperatures to 200 ° and greatest losses occurred between 400 and 600 °C, regardless of the type of fuel burnt. Losses of C and N varied considerably with temperature. Carbon was lost when fuels were heated at temperatures of 300 °C or more. Nitrogen was relatively more abundant when heated at 400 °C, albeit at very low levels (less than 5%). When heating time was varied there were noticeable differences in patterns of weight loss and changes in proportions of C and N. This indicates that both fire intensity and residence time is likely to be important in understanding losses of C and nutrients during fire, particularly during low intensity prescribed burning.

The use of colour of residues after heating surface fuel has the potential to determine fire severity. Existing technology such as near infra-red scanners can measure ash colour, not only indicating fire severity but also, by association, C and N losses from fire. Combustion studies done in a well-controlled laboratory environment could be used to interpret fireground conditions in relation to fire intensity and residence time, according to the nature and amount of charred material, charcoal and ash that remains after fire.



END-USER STATEMENT

Dr Felipe Aires, *NSW National Parks and Wildlife Service*

This report presents the background research for a potentially powerful tool that can be used by land managers for ground truthing post-fire severity. While considerable effort and advances have been made in mapping of fire severity at a landscape-scale using remote sensing imagery and, more recently, Unmanned Aerial Vehicle imagery, the need for on-the-ground assessment remains. Many of the metrics used for ground-truthing fire severity are subjective and categorical. This research has the potential to add more layers of information to how fire severity can be measured reliably and accurately using near infra-red (NIR) scanner technology. As the technology associated with NIR spectroscopy advances, the cost and robustness of suitable measuring devices will decrease and increase, respectively.

The simple, yet systematic approach taken in this study provides valuable data that can be used to develop a greater understanding of carbon and nutrient losses during combustion that will better inform modelling associated with disturbance to ecosystem processes, specifically carbon and nutrient cycling due to prescribed fire and bushfires.

As a final point, the research presented in this report showcases the important contribution that student projects can make to bigger research activities with the additional benefit of improving the educational and training capacity of the Bushfire and Natural Hazards CRC.

1. INTRODUCTION

Fires in forest and woodland ecosystems can cause changes in carbon (C) and nutrient pools, firstly, through heating of plant biomass resulting in volatilisation of gases, followed by combustion at higher temperatures. After fire, these physical processes can create imbalances in C and nutrient cycling (Certini, 2005; Caon *et al.*, 2014; Butler *et al.*, 2017; Zhang and Biswas, 2017; Pellegrini *et al.*, 2018). More often than not, estimates of gaseous emissions and changes in aboveground C and nutrient pools assume that surface and near surface fuels are completely combusted and effectively removed during burning regardless of whether the fire was a low intensity fuel reduction burn or a high intensity bushfire. As such, there have been a number of publications reporting losses of C and nutrients associated with fire, but they have generally only considered the effect of complete combustion of surface fuel (Volkova and Weston, 2013; Possell *et al.*, 2015; Volkova *et al.*, 2015; Volkova and Weston, 2015; Jenkins *et al.*, 2016; Gharun *et al.*, 2018; Karunarante *et al.*, 2019).

An important step in C accounting associated with fire is determining the conversion rate of biomass to charred material, ash and charcoal as C may be moved or transformed to another pool and not completely lost to the atmosphere in gaseous form such as carbon dioxide (CO₂) and carbon monoxide (CO). Understanding the conditions under which C and nutrients in aboveground biomass are affected by heating and combustion will provide information relevant for post-fire management including potential for movement of ash due to runoff, nutrients available for plant regeneration and better estimates of C loss as emissions.

This laboratory-based study quantified C and nitrogen (N) losses during fire and conversion of biomass to combustion products under controlled conditions. A preliminary study performed by Aerts and Bell (unpublished data and see Parnell *et al.*, 2019; Milestone 2.1.4), investigated the loss of biomass and nutrients after systematic heating of different fuel fractions collected from heathy woodlands in Victoria. Minimal changes in biomass, C and N were evident when heated at temperatures less than 200 °C; most fuel fractions had the greatest losses of biomass, C and N between 300 and 500 °C.

This study aims to quantify the conversion of C in typical surface fuels from forests during heating and combustion to determine changes in pools of C and N after fire. In addition, we will quantify C found in residues such as ash and char to account for C and N that is not lost to the atmosphere during burning.

2. METHODS

2.1 SAMPLE COLLECTION

Bulked samples of three dominant types of surface fuels (leaves, twigs and bark) were collected from various locations. The three broad types of fuels were further separated according to physical features. Leaves were classified as 'curly' (*Eucalyptus saligna*), 'flat' (a mixture of leaves from dry sclerophyll forest), and two types of 'needle-like' leaves (Table 1). Twigs were separated into size classes: 0-2.5, 2.5-5.0 and >5.0 mm diameter (thin, medium and thick, respectively). Different types of bark were collected from several tree species based on varying thickness and density (Table 1).

An additional set of fuels were collected from the field from three unburnt plots from Rocky Waterholes in the Blue Mountains (34°19'12.0"S, 150°28'12.0"E) (see Bell *et al.*, 2020). The forest sampled is broadly classified as a Dry Sclerophyll Forest. Replicate fuel samples were collected using a circular sampling ring (0.1 m²) and were separated into leaves, twigs, bark, soil and fine fuel. Fuel samples were oven-dried for 48 h at 60 °C and then passed through a 9 mm sieve. Soil samples were sieved using a 2 mm sieve to remove any roots and rocks.

Table 1. Collection location, species and descriptions of surface fuel samples collected for the study. All collection sites were in New South Wales.

Category	Collection location	Species	Descriptive name
Leaves	Richmond	<i>Eucalyptus saligna</i>	Curly leaves
Leaves	Hornsby	Assorted – <i>Angophora costata</i> , <i>E. saligna</i> , <i>Syncarpia glomulifera</i>	Flat leaves
Leaves	Cobbitty	<i>Pinus radiata</i>	Pine needles
Leaves	Eveleigh	<i>Allocasuarina cunninghamiana</i>	Casuarina phyllodes
Twigs	Hornsby	Assorted – <i>A. costata</i> , <i>E. saligna</i> , <i>S. glomulifera</i>	0-2.5 mm (thin)
Twigs	Hornsby	Assorted – <i>A. costata</i> , <i>E. saligna</i> , <i>S. glomulifera</i>	2.5-5.0 mm (medium)
Twigs	Hornsby	Assorted – <i>A. costata</i> , <i>E. saligna</i> , <i>S. glomulifera</i>	>5.0 mm (thick)
Bark	Eveleigh	<i>Melaleuca quinquenervia</i>	Paperbark
Bark	Hornsby	<i>A. costata</i>	Smooth bark – Red Gum
Bark	Hornsby	<i>E. saligna</i>	Smooth bark – Blue Gum
Bark	Hornsby	<i>S. glomulifera</i>	Rough bark – Turpentine
Leaves	Blue Mountains	Assorted species	Leaves
Bark	Blue Mountains	Assorted species	Bark
Twigs	Blue Mountains	Assorted species	Twigs (2.5-5.0 mm)
Fine fuel	Blue Mountains	Assorted species	Fine fuel (<0.9 mm)
Soil	Blue Mountains	Assorted species	Soil (<0.2 mm)

2.2 SAMPLE PREPARATION AND HEATING

All samples were oven dried at 70 °C for 3 days. Dried samples were kept in a desiccator when removed from the oven and prior to heating in the muffle furnace to prevent absorption of moisture from the atmosphere. Subsamples of each fuel type (approximately 2.0 g, n = 8) were weighed into foil trays and heated at 100, 200, 300, 400, 500 and 600 °C for 60 min. Samples were cooled until they could be handled safely and reweighed to determine weight loss then stored for later chemical analysis. The control samples (referred to as 'unburnt') represented fuel that was not heated or combusted and were held at 25 °C.

To determine the effect of timing of heating, a second study used representative leaf, twig and bark samples (i.e. flat leaves, thin twigs and bark from Blue Gum; Table 1) which were heated for 5, 15, 30 and 60 min at varying temperatures (100, 200, 300, 400 and 500 °C).

2.3 ELEMENTAL ANALYSIS

Subsamples of surface fuels after heating and combustion were finely ground and C and N content (% dry weight) was measured by combustion analysis (Elementar Vario Max CNS, Analysensysteme GmbH, Hanau, Germany).

2.4 COLOUR ANALYSIS

It was important to find a means of consistently classifying residues after heating and combustion as the colour varied by species, type and treatment at different temperatures. The Munsell Colour System is a useful tool for classification of numerous materials by colour scheme (Munsell Colour Company, 1950). It is a system that is already widely adopted in soil science for classification of diverse soil types. Past studies have also adopted this method to classify ash from countless species of plants heated at different temperatures (Ubeda *et al.*, 2009; Dūdaitė *et al.*, 2011; Balfour, 2013; Bodí *et al.*, 2013). Under fluorescent light, burnt, ground material from samples representing the range of surface fuel types was compared to the Munsell Soil Colour Chart to determine the associated hue, value and chromas at each temperature.

3. RESULTS AND DISCUSSION

3.1 CHANGES IN BIOMASS

In general, proportional weight loss of fuel samples was greatest when combusted at temperatures between 300 and 500 °C (Figure 1). Minor differences in weight resulted from heating to 200 °C with weight loss due mainly to removal of any remaining vestiges of water from cell walls and cavities and volatilisation of volatile organic compounds with low boiling points (Matthews, 2010). There was little additional weight loss when samples were combusted at temperatures greater than 500 °C when thermal decomposition was complete.

For all fuel types, minimal changes in sample biomass were evident at 100 and 200 °C (Figure 1). Leaf material had greatest losses of biomass at these two temperatures, with flat leaves losing up to 12% of the initial sample weights and curly leaves losing up to 11% when heated at 200 °C (Figure 1). This contrasted with twigs and bark, which only had weight losses of between 4-6% and 3-9%, respectively.

Bark was the most variable in weight loss, particularly at 300°C when thermal decomposition generally begins. Bark from Turpentine (*Syncarpia glomulifera*) and Red Gum (*Angophora costata*) had the greatest weight loss of up to 85% of initial sample weight (Figure 1). At this temperature, cellulose and hemicellulose begin to pyrolyze and decompose into char, gases and aerosols (Mettler *et al.*, 2012). A study investigating the thermochemical conversion of cellulose, hemicellulose and lignin in plant material found the greatest biomass losses (90, 70 and 60%, respectively) occurred within temperature ranges of 250-360 °C (Pasangulapati *et al.*, 2012).

Surface fuel fractions collected from the field in the Blue Mountains showed similar patterns of biomass loss (Figure 2). Soil had smaller losses of biomass (less than 10%), but this is not surprising as much of the soil matrix is non-organic material (e.g. silica, carbonate). Total biomass loss for fine fuel was also lower than other fuels (80% loss of biomass) probably because of the inclusion of some soil into this fraction despite every effort to prevent this.

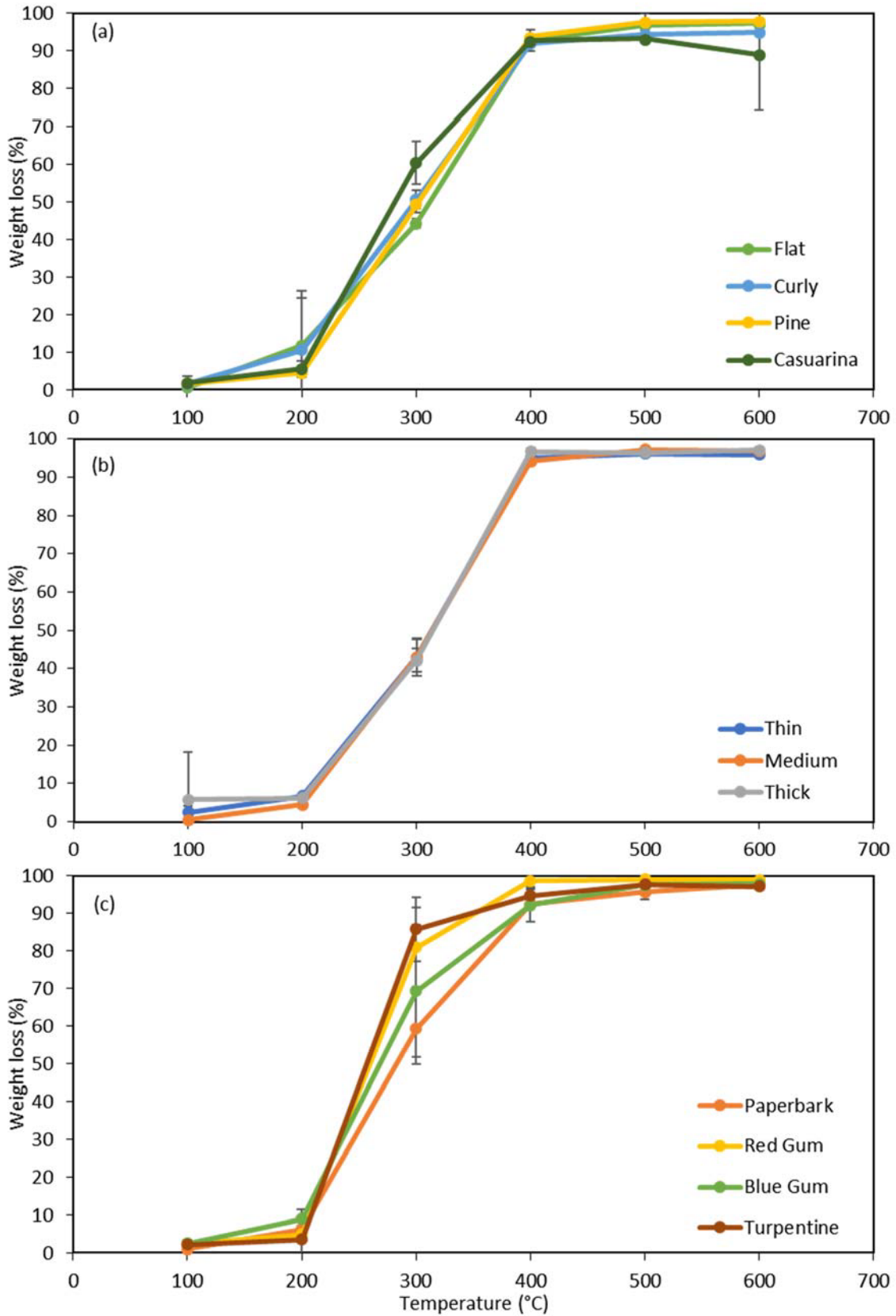


Figure 1. Proportional weight loss (%) of three dominant types of surface fuels; (a) leaves, (b) twigs and (c) bark. Points represent mean \pm standard deviation (n = 8).

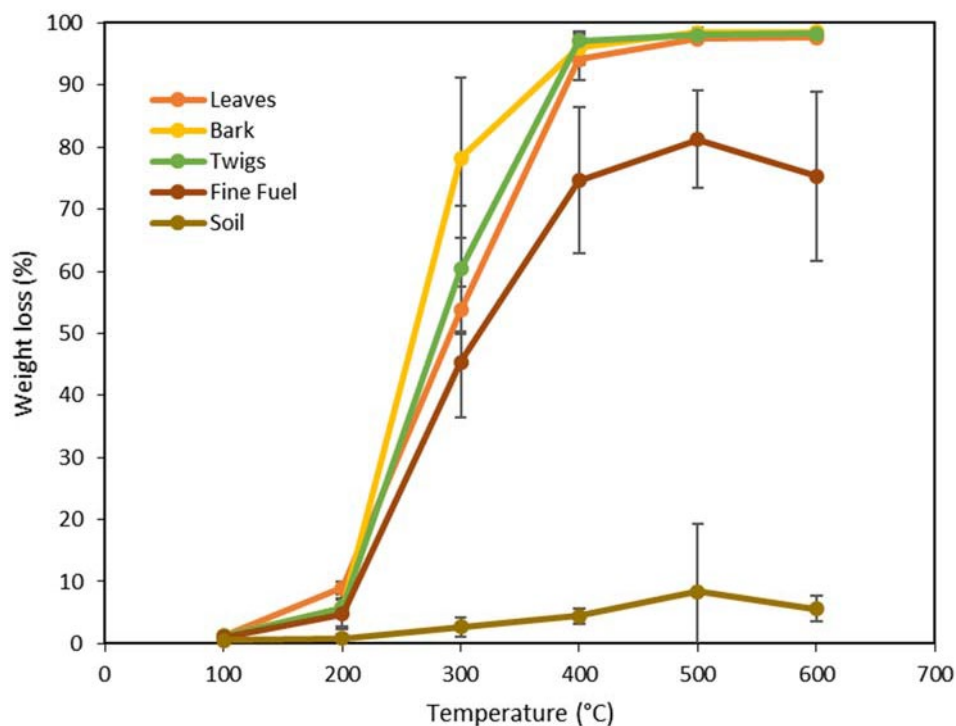



Figure 2. Proportional weight loss (%) of surface fuels (leaves, bark, twigs (2.5-5.0 mm diameter), fine fuel (<0.9 mm) and soil from Dry Sclerophyll Forest in the Blue Mountains; Points represent mean \pm standard deviation ($n = 3$).

3.2 CHANGES IN CARBON AND NITROGEN

Samples were analysed to examine changes in C and N of surface fuels when heated or combusted at six different temperatures (Figure 3). The change in relative C content was consistent for all fuel types. The C content of unburnt (25 °C) samples and those heated at 100 and 200 °C was within the range of 48-58% (Figure 3a, b, c). At 300 °C, the proportion of C in many of the different fuel types ranged from 59-75%, except for flat leaves, Paperbark, medium and thick twigs which all had 50% C or lower. At this temperature, most samples appeared to have been charred completely, which is indicative of a higher ratio of C in these samples compared to other elements in the original material.

When combusted at higher temperatures (400-600 °C), varying proportions of C was present depending on the fuel type. This was not unexpected as wood of different densities has been found to form charcoal with different physical qualities (Belcher *et al.*, 2015). Small amounts of C (generally less than 10%) remained in all samples after combustion at 500 and 600 °C (Figure 3a, b, c). Paperbark has the least amount of C remaining (1-2%) in comparison to the other types of fuel, while bark from Turpentine had the most C present after combustion at 600 °C (13%). Minimal C remained in the ash residue, what was left is likely to be comprised of inorganic salts or silica (non-combustible materials).



Prior to heating, N content was low (less than 2%) in all fuel types (Figure 3d, e, f). Each fuel type differed in N content when heated or combusted, however, there was a consistent increase in N at 400 °C, followed by losses of N during combustion at 500 and 600 °C. Pine needles had the greatest increase in N at 400 °C (3.4%), followed by bark from Turpentine (3.1%) and Paperbark (2.5%) (Figure 3d, e). Nitrogen in leaf material steadily decreased in N content when heated to 200 °C (Figure 3d).

Surface fuels collected from the site in the Blue Mountains (Figure 4) showed similar patterns of loss of C with heating and combustion, albeit at lower starting values for biomass, fine litter and soil alike (i.e. 50% C). Nitrogen in biomass was also slightly lower in fuel samples collected from the field (i.e. less than 2%) reflecting the different age and state of decomposition of materials collected from the forest floor compare to newly fallen material used in the experimental trial. As expected, soil was extremely low in N initially and was all but lost with heating (evaporation of solutes) and combustion (thermal decomposition of organic matter) (Bauhus *et al.*, 1993; Certini, 2005).

3.3 VARIATION IN TIME OF HEATING

An investigation of the effects of duration of burn time and temperature on surface fuels was done as a standard time of 60 mins had only been used previously (i.e. Aerts and Bell, unpublished data and a pilot study using cardboard, paper and wood). The 60 min burn duration was derived from the 'Standard Test Method for Ash in Biomass', whereby material is burned at 575 °C in a muffle furnace for 3 hours to generate ash (American Society for Testing and Materials (ASTM International, 2015). Different times for heating and combustion of samples were therefore tested to determine how long fuel needs for complete combustion. For this study, representative types of fuel were chosen; flat leaves (mixed species), thin twigs (0-2.5 mm diameter) and bark from Blue Gum.

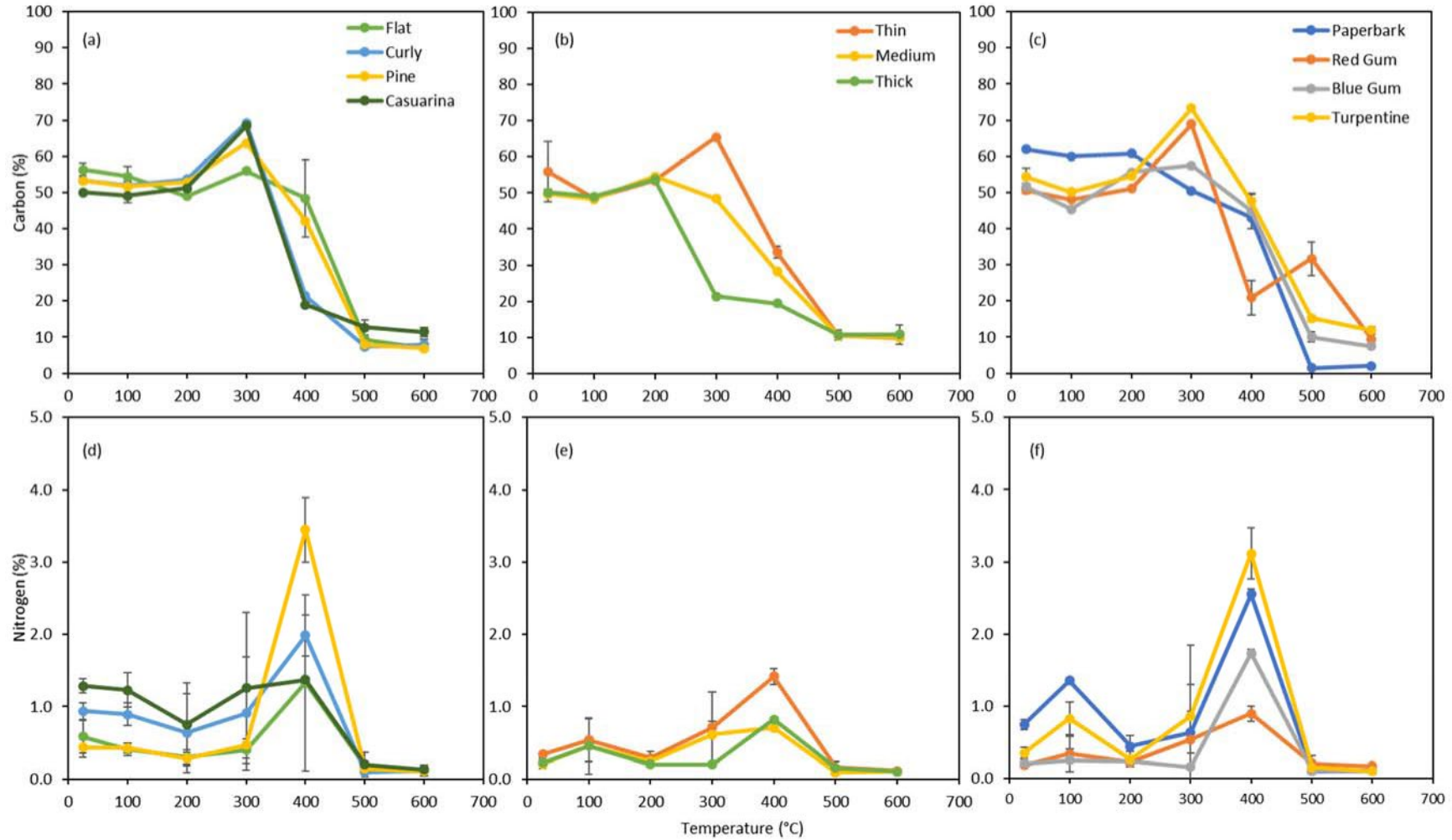


Figure 3. Carbon and nitrogen content (%) of residues at each temperature for three types of surface fuels; (a, d) leaves, (b, e) twigs and (c, f) bark. Points represent mean \pm standard deviation ($n = 8$).

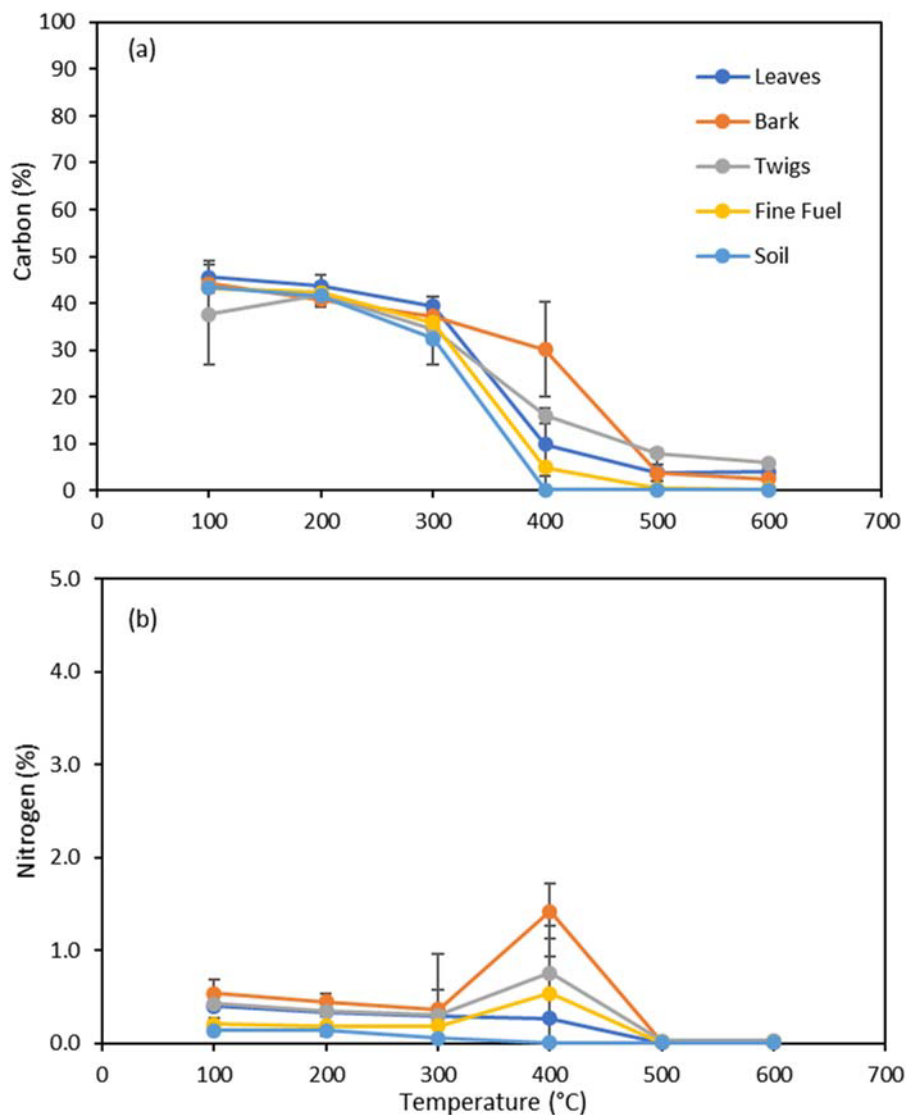


Figure 4. Carbon and nitrogen content (%) of residues at each temperature for surface fuels (leaves, bark, twigs (2.5-5.0 mm diameter), fine fuel (<0.9 mm) and soil from Dry Sclerophyll Forest in the Blue Mountains; Points represent mean \pm standard deviation (n = 3).

3.3.1 Biomass

Heating time had a minimal effect on weight loss at lower temperatures of 100 and 200 °C (Figure 5). At 300 °C, a temperature that results in at least partial thermal decomposition of most surface fuels, timing of heating had an important effect, most noticeably for bark from Blue Gum.

Overall, the longer the duration of heating or combustion, the more weight was lost from each of the fuels (Figure 5). For all fuel types, the greatest weight loss was recorded when samples were heated or combusted for 60 min. This suggests that under field conditions, both residence time of fire and heat output will influence the degree to which fuel is combusted.

3.3.2 Carbon and nitrogen

The C and N content of each fuel type was assessed to determine if duration of heating or combustion had an effect on the amount remaining in residues. For heating or combustion of 30 minutes or less, C content was relatively consistent at all temperatures, remaining at 49-58% (Figure 6a, b, c). At temperatures above 300 °C, combustion for 30 minutes was enough to lower C content by 20-30%. However, greater losses of C were found when samples were in the muffle for a longer period of time, but this varied for fuel type (i.e. final C content was 10% for bark, 14% for leaves and 29% for twigs).

The longer the burn and the hotter the temperature, N content in surface fuels was reduced (Figure 6d, e, f). As seen earlier, N content was low in all fuel types (less than 2%), however, heating samples at 400 °C for 30 minutes or more resulted in an increase in N content for leaf and bark material. An increase in N did not occur with heating or combustion at lower temperatures (100, 200 and 300 °C) or for shorter periods (5 and 15 minutes).

3.4 ASH COLOUR

After heating or combustion, ground samples were a variety of different colours (Figure 7). There are potential links between ash colour, C loss and fire severity (temperature of burns). Between 100 and 200 °C, there was no discernible colour change when compared to the original unburnt material (Figure 7a, b). When combusted at 300 °C, samples showed obvious signs of charring (Figure 7c) and by 400 °C ash had formed (Figure 7d). By 500 and 600 °C each sample had reached final combustion, by turning into complete ash (Figure 7e, f).

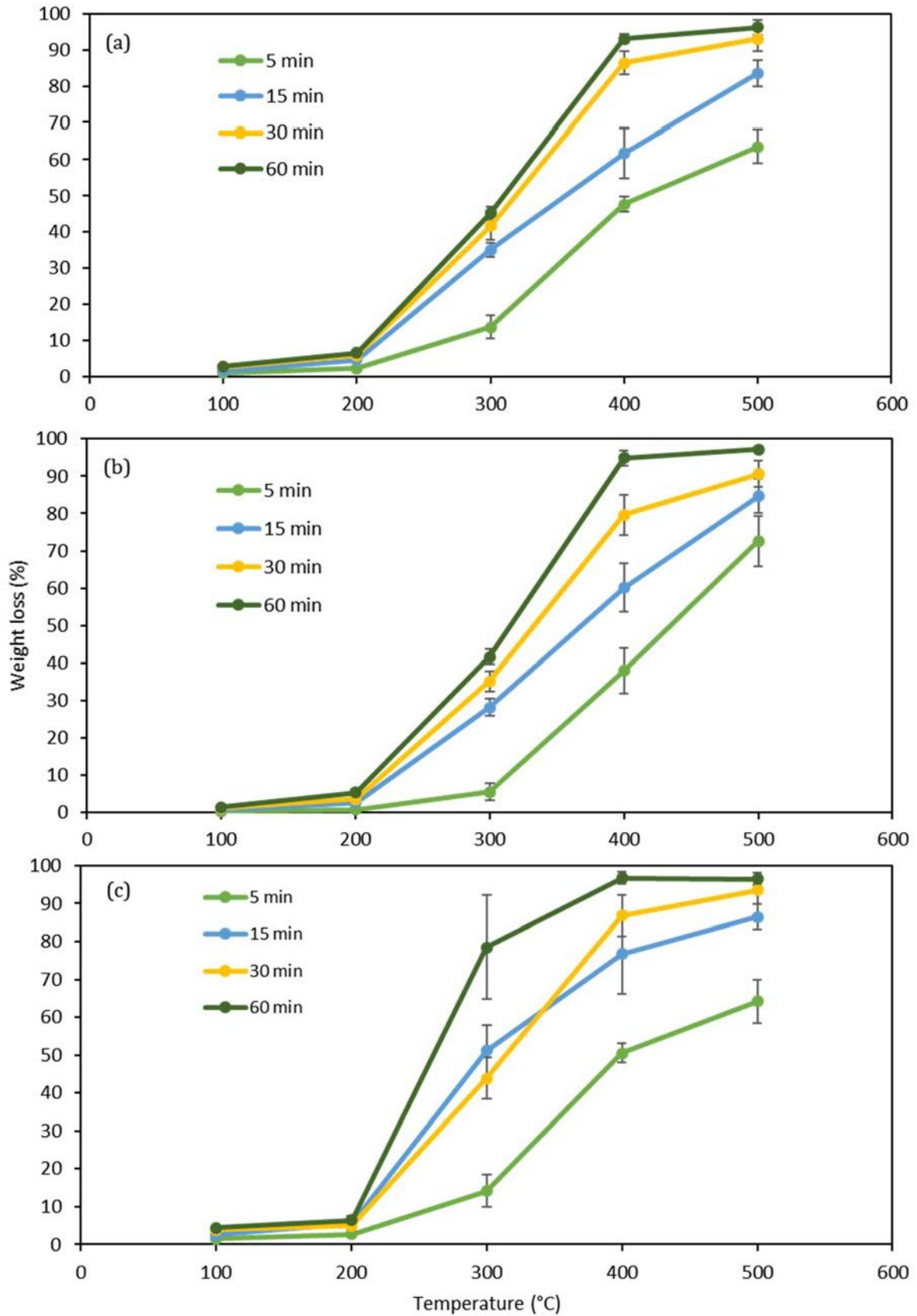


Figure 5. Proportional weight loss (%) of three types of surface fuels with systematic heating of (a) leaves, (b) twigs and (c) bark at various temperatures. Points represent mean \pm standard deviation ($n = 8$).

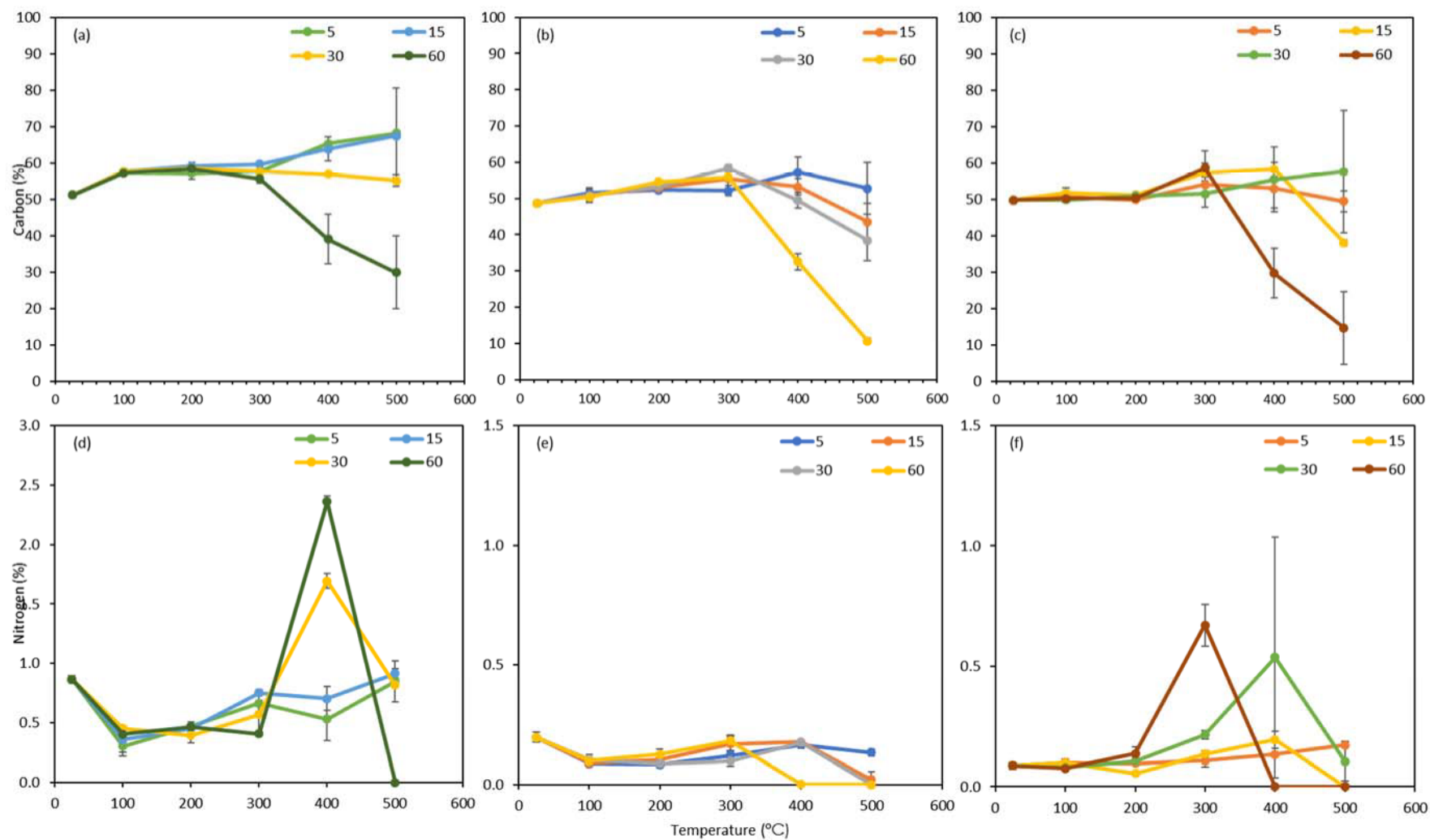


Figure 6. Carbon and nitrogen content (%) at each temperature for three types of surface fuels; (a, d) leaves, (b, e) twigs and (c, f) bark. Points represent mean \pm standard deviation ($n = 8$).

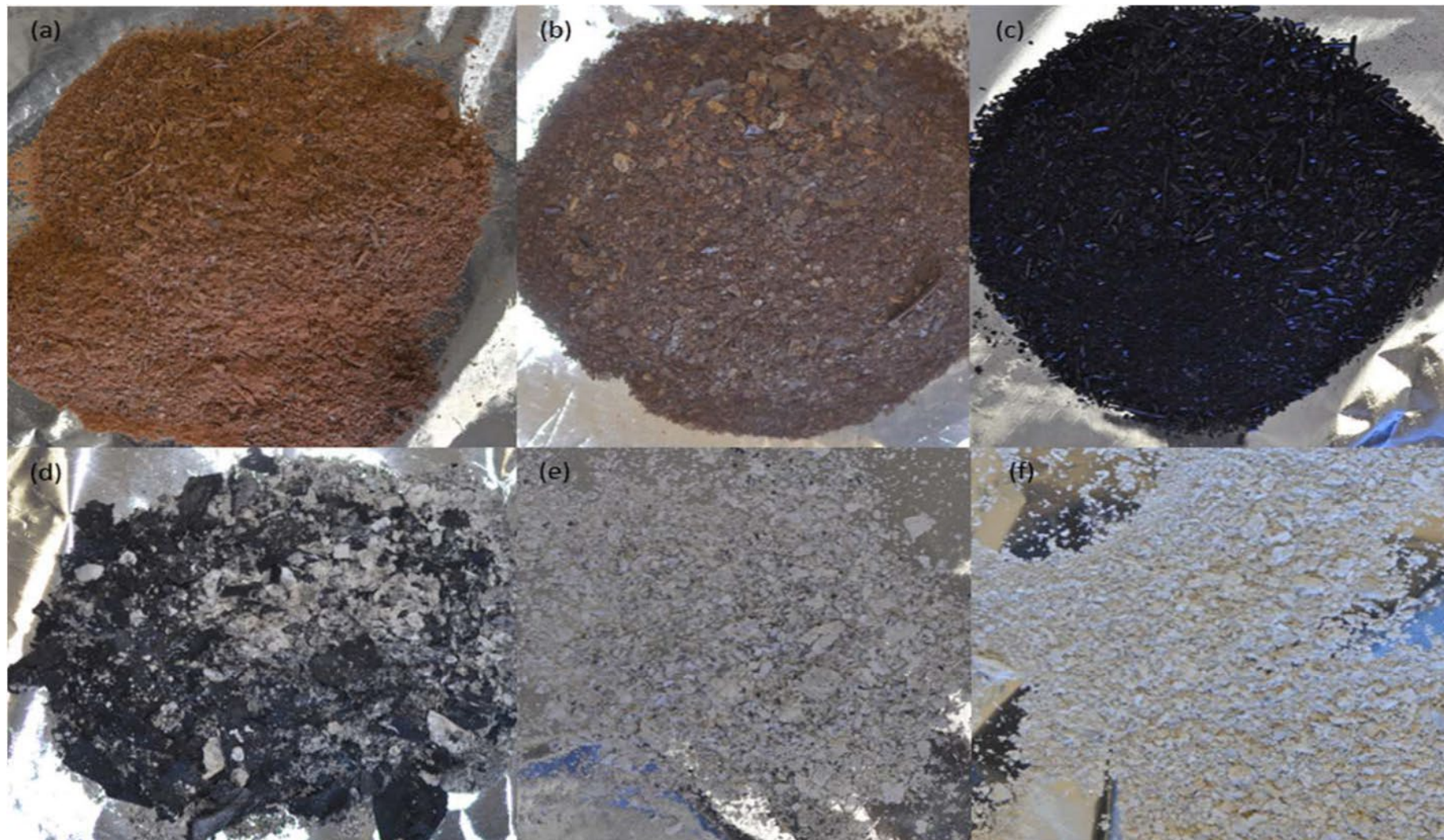


Figure 7. Example of the colour range of ground samples of flat leaves systematically heated at temperatures of (a) 100 °C, (b) 200 °C, (c) 300 °C, (d) 400 °C, (e) 500 °C and (f) 600 °C.



After heating in the muffle furnace for 60 minutes, the resulting residues had yellow or yellow red undertones at lower temperatures (100 and 200 °C) (Table 2). After heating at 300 °C, fuel samples began to char and were predominantly black. The subsequent hues of these residues were in the 'gley range'. At 400 °C, there was a marked change in colour of many of the samples as they turned into grey ash indicating full combustion. Exceptions were curly leaves and pine needles, and bark from Paperbark, Blue Gum and Turpentine, which remained a dark black ash colour at this temperature (Table 2). Some of the samples had a 'mottled' effect where portions of the sample had completely combusted into white or grey ash, while other parts were only charred and remained black (Figure 7d). In instances like these, Munsell colour scores were determined by the predominant colour in the sample. For example, samples of flat leaves heated at 400 °C were not uniform in colour, however, after sifting through the samples, it was evident that majority of the underlying material was a lighter grey colour. Samples were then classified as 2 Gley 8/10BG, which is an off white/grey colour (Figure 7d; Table 2).

Table 2. Descriptions of colour for each of the fuel types heated or combusted at six different temperatures using the Munsell Soil Colour Chart (Munsell Colour Company, 1950). The Munsell colour notation is as follows: R = Red, YR = Yellow-Red, Y = Yellow, B = Blue, PB = Purple-Blue and N = neutral.

Sample	100 °C	200 °C	300 °C	400 °C	500 °C	600 °C
LEAVES						
Flat	10YR 6/4	2.5Y 3/1	1 Gley 2.5/N	2 Gley 8/10BG	1 Gley 8/10Y	1 Gley 8/10Y
Curly	2.5Y 5/4	2.5Y 3/3	1 Gley 2.5/N	2.5Y 2.5/1	2.5Y 5/4	2.5Y 7/2
Pine	10YR 4/6	10YR 3/2	1 Gley 2.5/N	1 Gley 2.5/10Y	2.5Y 8/1	1 Gley 8/10Y
Casuarina	2.5Y 5/3	2.5Y 5/3	1 Gley 2.5/N	1 Gley 7/N	1 Gley 8/10Y	1 Gley 8/10Y
TWIGS						
Thin	10YR 3/4	10YR 3/2	1 Gley 2.5/N	1 Gley 8/N	1 Gley 8/N	1 Gley 8/10Y
Medium	10YR 6/4	10YR 3/2	2.5Y 2.5/1	1 Gley 7/N	2 Gley 8/5B	1 Gley 8/10Y
Thick	10YR 3/2	10YR 2/2	1 Gley 2.5/N	2 Gley 8/5BG	1 Gley 8/10Y	2 Gley 8/5B
BARK						
Paperbark	10YR 8/3	10YR 4/4	1 Gley 2.5/N	2.5Y 2.5/1	2.5Y 7/3	2.5Y 8/2
Red Gum	10YR 6/6	10YR 2/2	1 Gley 2.5/N	2 Gley 8/5B	1 Gley 8/10Y	2 Gley 8/5PB
Blue Gum	7.5Y 4/6	10YR 2/1	2.5Y 2.5/1	1 Gley 2.5/N	1 Gley 8/10Y	2 Gley 8/5B
Turpentine	7.5Y 2.5/3	2.5Y 3/2	1 Gley 2.5/N	1 Gley 2.5/N	1 Gley 8/10Y	2 Gley 8/10B

4. CONCLUSIONS AND FUTURE WORK

Greatest weight loss occurred when samples were heated at temperatures between 400 and 600 °C, regardless of the type of surface fuel burnt. All material followed similar patterns, with minimal weight loss (<10%) at 100 and 200 °C, and maximum weight loss (90-100%) at a higher temperature range of 400 to 600 °C. The exception, however, was bark, which had variable weight loss at 300 °C, ranging from 55-90%. At the same temperature, leaves and twigs lost anywhere between 40-60% biomass. It is likely that the variability in weight loss among different fuel types is due to the amount of plant cell wall constituents – cellulose, hemicellulose and lignin. Losses of C and N losses also varied considerably with temperature. Increased availability of N when heated to 400 °C may be related to the thermal decomposition of cellulose, hemicellulose and lignin. In comparison, C in surface fuels began to decrease when heated above 300 °C.

There were clear differences in patterns of loss of biomass and changes in C and N when heating time and temperature were varied systematically. The greatest changes in both C and N occurred at higher temperatures and longer burn durations, as expected. This indicates that both fire intensity and residence time is important in understanding losses of C and nutrients during fire, particularly during low intensity prescribed burning. Better knowledge of C and nutrient losses during prescribed burning will lead to more precise modelling of C and N cycles in forest and woodland ecosystems.

There was also considerable variation in the colour of residues after heating all types of fuel. With existing technology such as near infra-red scanners, which can measure colour, ash colour has the potential to be an indicator for fire severity and, by association, C and N losses from fire. We have shown that combustion studies done in a well-controlled laboratory environment could be used to interpret fireground conditions in relation to fire intensity and residence time, according to the nature and amount of charred material, charcoal and ash that remains after fire. Our next stage of research will investigate this possibility.



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